A Guide to the characterisation of dielectric materials at RF and microwave frequencies

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THE PREPARATION OF THIS GUIDE

This Good Practice Guide on RF and microwave dielectric measurements has been prepared by the National Physical Laboratory and the Institute of Measurement and Control with funding from the National Measurement System Policy Unit (NMSPU) of the Department of Trade and Industry.

It was the intention of the NMSPU and the compilers of the Guide that its usefulness should be extended to as many workers in the field of RF and Microwave dielectrics as possible. The initial step in its preparation was therefore to consult those working in the field upon its content. This activity was greatly facilitated by a special discussion session on the prospective Guide at an open meeting of the National Measurement System's EMMA-Club – the Electromagnetic Materials Measurements and Applications Club - in December 2001. The compilers of the Guide are very grateful for all of the advice that was received from Club members at that time and for all other advice received from industrial and academic sources since then.

With this information to hand, a Technical Panel of specialists was established to advise on the scope and structure of the Guide. The persons listed below served as members of the Panel:

Bob Clarke	NPL	Chairman and Editor of the Guide.
Andrew Gregory	NPL	
David Cannell	Filtronic Comtec	
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Significant technical and editorial contributions to the Guide itself were made by Panel members and also by Gareth Jones, Jemeela Quraishi, Philip Merewood, Kevin Lees, Sara Fletcher and Karen Hood, all at NPL. The compilers of the Guide would also like to acknowledge the following for their help and advice:

Dr Stan Evans, Prof. Alan Preece (Bristol Oncology Centre), Prof. Jerzy Krupka (Technical University of Warsaw, Poland), Dr Richard Wylde (Thomas Keating Ltd), Dr Tom Cross (Nottingham University), Dr Bryan Kibble and the following NPL staff members: Geoff Orford, Stephanie Bell, John Gallop, Shakil Awan, David Adamson and Stuart Pollitt.

The compilers of the Guide would like to express their sincere thanks to all of these contributors, and especially the Panel members, for their valuable inputs and would also like to thank David Kent and Michael Yates of the Institute of Measurement and Control for their help and guidance in the preparation of this document.

NOTES ON THE GUIDE

This Guide refers to other publications that provide information or guidance. The editions listed are believed to be current at the time of publication, but reference should be made to the latest editions.

This Guide is subject to review by the responsible technical panel of the Institute of Measurement and Control. The Institute welcomes all comments on the Guide and requests that these be addressed to the Institute.

Users of this Institute of Measurement and Control Guide shall be responsible for its correct application.

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Foreword

This Guide is one of a series devoted to practical metrology commissioned by the National Measurement System Policy Unit of the Department of Trade and Industry. It is comprised of contributions by experts, including experts from UK companies concerned with dielectric measurements and measuring instrumentation. These contributions have been compiled at the National Physical Laboratory in accordance with a scope that was agreed by a technical committee. It is published by The Institute of Measurement and Control.

This Guide is aimed at all engineers and scientists who make dielectric measurements at RF and microwave frequencies. Although it focuses on the measurement of dielectric parameters, the basic principles presented here and much of the advice and information given should also be of value to those who are measuring other electromagnetic materials parameters such as magnetic permeability.

The aim of the compilers has been to develop an approach that will help readers to achieve greater confidence in the claimed traceable accuracy of dielectric measurements, no matter how modest that accuracy might be. A sound understanding of basic principles is all-important both for devising ways of attaining the levels of accuracy required in standards laboratories and for the ‘fit for the purpose’ measurements in industrial and other fields. Measurements of the latter type are often governed by economic considerations; the methods and instruments must be affordable and easy to implement. These considerations have also been central to the formulation of the advice given in this Guide. Beyond this, the major objectives have been to assist the reader in understanding particular measurement methods and in making choices between the many different measurement techniques that are available.

The reader’s attention is drawn to the Glossary (Section 10). This is not intended to be merely a dictionary of terms to be consulted when unfamiliar ones are met in the main text but to be also a compendium of basic concepts, the correct understanding of which is necessary to perform reliable measurements.

Finally, my thanks go to the many individuals and companies that have contributed to the production of this Guide, with particular thanks to Bob Clarke of the National Physical Laboratory who has been a significant driving force in the production of this guide and a major contributor to it. I am confident that engineers and scientists, and indeed anyone who has to make a measurement of dielectric properties, will find this Guide useful, and that attention to the principles and advice given will enhance the quality of products and processes, improve industrial competitiveness and foster innovation.

Dr S Pollitt
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September 2003

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SECTION A: BACKGROUND

1. Introduction and Scope

1.1 Introduction

This Guide is concerned with practical dielectric measurements. It aims specifically to provide guidance for readers who wish to determine the radio-frequency and microwave (RF & MW) electromagnetic properties of materials. It focuses chiefly on the measurement of complex permittivity, ε^*, but also provides advice on the measurement of other parameters, including complex magnetic permeability, μ^*. Even though restricted, as here, to RF & MW frequencies, the field of dielectric measurements is very wide and, as can be seen from the Contents pages, a large number of measurement techniques and material types must be surveyed to cover the field. We have therefore taken it for granted in compiling this Guide that readers with specific measurement problems will want to be selective in their reading of it. There are, though, a number of good practices in dielectric metrology that apply across the field and we can all benefit from using them. There is also a good deal of common background material of a theoretical nature that we need to appreciate in order to understand the recommendations made throughout the Guide. We have therefore split the Guide into two main sections, the first of which, 'Section A: Background', is potentially of interest to *all* readers and the second, 'Section B: Practical Advice', is designed to be read *selectively*. There is also a 'Section C' containing useful supporting material such as worked examples and references. In general, we have tried to structure the Guide to make it easy for readers to pick out a route through its pages that is relevant to their own particular requirements.

There is a process of decision-making that one should go through even before any measurements are attempted and the Guide aims to help us with this too. We start with the assumption that you, the reader, want to know the dielectric properties of one or more materials. One of the first questions to ask must be *'Do I really need to measure the material at all?'* - adequate information may already be available in the scientific or manufacturers' literature. Assuming that such information is not available, and measurements are indeed necessary, should one perform the measurement oneself or approach a test laboratory to perform the measurements instead? This decision must depend upon one's own experience in the field, upon the costs and the funds available and upon the likelihood of success in the conditions that prevail in one's own particular working environment. Advice is offered here on making all of these preliminary decisions, but the main concern of the Guide is with the steps to be followed should we decide to go ahead and measure the material ourselves. Which technique should we use? Indeed, should we use more than one technique? How should we prepare and handle the material specimens? What level of *uncertainty* do we *need* in our measurements and what level can we *achieve* by any particular technique? These last two questions about uncertainty are very important. They address major criteria that should influence our choice of measurement techniques. To choose the wrong method, e.g. one that cannot achieve our desired uncertainty, may result in much lost time and money.

It is one thing to perform a measurement, but to have *confidence* in the results of that measurement is an entirely different matter. It is surely a waste of our time and funds to perform a measurement and to quote our results without having any grounds for confidence in those results! So, having set up our measurement system, we should ask ourselves how we know that it is operating correctly. The Guide describes a number of ways in which we can gain confidence in our measurements, e.g. by comparing two or more different techniques or by measuring dielectric reference materials that already have well-characterised properties. Closely associated with these issues, and equally essential for attaining confidence, are the principles of *calibration* and *traceability*. If our measurement system is not calibrated and if the quantities that we are measuring are not traceable to international standards, we are effectively cut off from the rest of the world of measurement and the rest of the world can, strictly speaking, make no practical use of our results! Issues of *uncertainty*, *confidence*, *calibration* and *traceability* are so important that we have dedicated a whole chapter to them - Chapter 4.

Such are the major issues addressed in this Guide, we will now explain how it is organised.

1.1.1 A Guide to the Guide

The step-by-step approach to practical dielectric measurement described in the second paragraph above actually starts in this guide with Chapter 5 and ends with a short chapter on the presentation of measurement results - Chapter 8. Chapters 1 - 4 provide a theoretical context and rationale for the practical recommendations that follow in Chapters 5 – 8, because it is part and parcel of 'Good Practice' to be clear about the scientific theories that support such recommendations. Chapters 9 – 11 provide supporting material. Here are the details:

Section A: Background: Chapters 1 - 4. Following this introductory chapter, Chapter 2 on *'Basic Principles'* provides the scientific and engineering background to good practice in dielectric measurement. For the sake of succinctness we have provided only enough information to make our reasoning clear. However, references are given to the scientific literature that contains the full theoretical background. Chapter 3 provides a brief survey of existing international standard dielectric measurement methods - i.e. approved and documented measurement techniques for characterising dielectrics. It is often a good idea to follow these standards, but sometimes it is better *not* to follow them exactly and Chapter 3 tries to provide advice to help us to decide which track to follow. Chapter 4 is concerned with calibration, traceability, uncertainty and confidence in measurement.

Section B: Practical Advice: Chapters 5 - 8. Chapter 5 commences the second part of the Guide, which focuses on practical advice. It starts with *preliminaries*: decisions to be made and steps to be taken before measurements commence. It then proceeds to guidance on our choice of techniques to suit the various physical types of dielectric materials (solid, liquid, powder, etc.) that we may have to measure. Chapter 6 covers issues that are common to many, sometimes all, dielectric measurement methods, such as the measurement of specimen size and of Q-factor, while Chapter 7 presents the Guide's 'core' survey of nineteen basic dielectric measurement methods. Advice on choosing between these methods, and advice on measurements in the production environment is also presented in Chapter 7. Clear presentation of measurement results is an important function of good practice and it is considered briefly in Chapter 8.

Section C: Support Material: Chapters 9 - 11 and References. Chapter 9 contains worked examples to illustrate techniques and issues covered elsewhere in the Guide. The Glossary of Chapter 10 may be consulted for the definition and clarification of many of the technical terms used in this Guide. It attempts to resolve ambiguities in nomenclature and also to discourage poor terminology. Chapter 11 provides advice on obtaining further information on RF & MW dielectric measurements, because clearly, we cannot cover everything here. The Guide ends with a comprehensive list of References which complement and explicate its contents.

1.1.2 General Observations.

(1) RF & MW dielectric measurements often prove to be more difficult to perform without significant error than one might expect - a fact that the members of the Guide's Technical Panel will readily attest! They are *in general* very challenging, so please do not underestimate the problems *or* the uncertainties involved when you embark upon such measurements.

(2) In a field as diverse as electromagnetic materials measurement, 'Good Practice' must always remain to some extent a matter of experience and opinion. Certainly, many of the practices advocated here would take their rightful place in any good practice guide on measurement, but others arise from the accumulated experience of the contributors to the Guide. Final decisions as to which course to follow, however, must always be taken by the person 'on the ground'. Here lies the importance of knowing the theory and the scientific thought that lies behind the decisions that have to be made. We cannot guarantee that all of the advice given here will work every time but we do hope that the combination of practical advice and theoretical background presented in this Guide will help you on your way to successful measurements.

Do's and Don'ts: In order to emphasise the most important principles that we recommend in this Guide, we have inserted '*Do's and Don'ts'* lists at appropriate points in the text, usually at the end of sections and chapters. We make no apology for repeating some of their content in a number of places in order to emphasise important points. We start here with our most basic *Do's and Don'ts*, some of which were touched upon above.

► ***Do's and Don'ts:***

> ***Do*** start with a good idea of the *measurement uncertainty* that you require: all other decisions that you need to make will depend upon this. If you *don't* know what level of uncertainty you need, you may end up wasting considerable time and money on inappropriate techniques, either because you are using an expensive technique that is too accurate for your needs or because you are using an inadequate technique that cannot give you the information you need. Guidance on uncertainties is given in Chapter 4.
>
> ***Do***, in general, ask (i) '*Do I need to perform a measurement at all?*', (ii) If '*Yes*', then ask: '*Should I perform it myself or ask a test laboratory to perform it*', (iii) if you proceed with the measurement, then ask: '*What technique(s) should I use?*', (iv) then ask: *'How can I achieve confidence in my measurement results?*' and '*What is my uncertainty?*', then ask: (v) '*How should I present my results?*'. See Chapters 5 - 8.

Do think, before you start, about which parameters you actually need to measure. Do you need to measure both the *real* and *imaginary* parts of the permittivity? Could your dielectric material be magnetic (i.e. could it have a complex permeability significantly different from 1.0)? If so, you may need to measure magnetic permeability as well as permittivity. Do you really need measurements over a wide band of frequencies, or will just one frequency suffice? On this last point, bear this in mind: for a low-loss material like polyethylene, if you do not need to know its loss, just one provably-accurate value of real permittivity measured at one frequency could apply all the way across the spectrum from 1 kHz to 150 GHz because the real permittivity of low-loss materials varies so slowly with frequency, see Section 2.5 and 5.3.2.

Don't underestimate the difficulty of performing RF & MW dielectric measurements accurately - those who set out to use a new measurement technique often do. *Easy* measurements often prove to be bad measurements! Guidance is given in Chapters 5 - 7.

Don't underestimate the uncertainty that can be achieved with any given method. Try to estimate likely uncertainties before you start. See Chapter 4.

Don't use the term '*Dielectric Constant'*. It encourages the wrong attitude towards the dielectric properties of materials! The relative permittivity of materials is *not* constant[†]. It can vary significantly with temperature, humidity, concentration, preparation method, level of contamination, polarisation, etc. See the Glossary, Chapter 10, and Section 2.1 for preferred usage.

[†] The only real 'dielectric constant' is ε_0 - the *Permittivity of Free Space*, otherwise known as the *Electric Constant*. This is the constant that relates ε', the *relative* permittivity of a material, to its *absolute* permittivity, $\varepsilon_0\varepsilon'$. See Section 2.1. $\varepsilon_0 = 8.8542 \times 10^{-12}$ F.m^{-1}.

1.2 The Scope of the Guide

An enormous range of methods has come into use in laboratories around the world for measuring the electromagnetic properties of materials. To cover the whole field of dielectric measurement would therefore require a substantial multi-volume book, rather than a Guide such as this. We have therefore found it absolutely necessary to limit the *scope* and *depth of detail* in this Guide.

For example, while we focus here on RF & MW, we recognise that there is also a need for practical guidance on dielectric measurements at low frequencies (LF) below 1 MHz. This would include, for example, detailed advice on how to characterise charge transport mechanisms or on how to characterise high-field electrical breakdown in dielectrics (see Section 6.6.3). But such LF and high-field measurements form specialist subjects in their own right and we will have to leave it to experts in those fields to guide us on these matters.

Since 1954 the most widely known and comprehensive standard work on RF & MW dielectric measurement has been that of Von Hippel [1]. It has deservedly stood the test of time and remains one of the most useful treatises on RF & MW dielectric measurements, but there have been significant technical advances since it was published. A number of reviews (e.g. [2] and [3]), books with details on measurement (e.g. Anderson [4], Ch. 2) and conference proceedings (e.g. [5]) have been published since that time which can help to bring us up to date but the RF & MW community may still benefit from a more comprehensive guide that can take recent advances into account. The Technical Panel that has been drawn together to produce this Guide therefore comprised experts who were well placed to take account of these advances in measurement and in the use of novel materials and their applications. At the same time, core and well-established techniques that deserve to be promoted because of their proven general utility and long-term value have not been ignored. A number of methods which are not used so often these days but which deserve re-appraisal in the light of modern technological developments are also included.

So, bearing in mind the current industrial and academic requirements for dielectric measurements and also the need to limit the content of the Guide, the Technical Panel decided upon the scope printed on the next page. They should cover the most important issues in the technical field of RF & MW dielectric measurements today.

The Scope of the Guide:

- The Guide provides advice on the characterisation of dielectric materials in the RF & MW frequency range 1 MHz – 150 GHz. Less detailed coverage is provided down to 1 kHz and above 150 GHz.

- As part of this task, the Guide presents a survey of many of the RF & MW dielectric material measurement techniques in present-day use – see Section 7.1.

- The Guide is written for scientists and engineers who possess at least a basic knowledge of the theory of dielectrics and of electrical engineering at RF & MW frequencies.

- The Guide is chiefly concerned with the measurement of complex permittivity, ε^*, at RF & MW microwave frequencies. This includes the measurement of real permittivity, ε', imaginary permittivity, ε'', and loss tangent, $\tan\delta$, where $\varepsilon^* = \varepsilon' - j\varepsilon''$ (with $j = \sqrt{-1}$) and $\tan\delta = \varepsilon''/\varepsilon'$. Please see the Glossary, Chapter 10, for a guide to the terms and symbols used here.

- It chiefly aims to cover the measurement of those materials having ε' lying between 1 and 200. In some places measurements of higher values of ε' (e.g. as in ferroelectrics) are discussed.

- Measurement of dielectrics with low loss ($\tan\delta < 3\times10^{-4}$), medium ($3\times10^{-4} \le \tan\delta < 3\times10^{-2}$), and high loss ($\tan\delta \ge 3\times10^{-2}$) is covered.

- The measurement of complex magnetic permeability, $\mu^* = \mu' - j\mu''$, is discussed *only* in connection with those methods for which it is a natural extension of a dielectric measurement technique.

- Measurements in the temperature range -40ºC to 400 ºC are considered but, in general, discussions refer to measurements at ambient temperatures unless otherwise explicitly stated.

- Measurements on both solid and liquid dielectrics are covered, as are material types ‘in between’ such as viscous liquids and soft and malleable solids. Measurements on composite and heterogeneous materials are discussed, as are measurements on powders.

- The measurement of dielectric anisotropy is covered, together with recommended techniques for performing it.

- The Guide describes the basic principles of dielectric metrology, providing an understanding as to why one class of technique should be preferred to another for a given material and frequency range.

- The importance of dielectric reference materials and measurement comparisons for dielectric metrology is emphasised.

- Methods for estimating and reducing measurement uncertainty are covered, as is the basic concept of uncertainty.

- Documented national and international-standard dielectric measurement methods are briefly surveyed in the Guide and their benefits and limitations are presented.

- Detailed descriptions of *Measurement Equations* (see Glossary) are, in general, *not* included - they are catered for by references to the relevant literature - the limited space available in this Guide allows no other policy. However more detailed descriptions are used where they illustrate important metrological principles or else where they are essential for the full understanding of a method or practice.

- While the above scope describes the main focus of the guide, related areas of metrology are catered for, as far as is possible, by references to the literature.

2. Basic Principles

The theoretical treatments presented here cover only those aspects of dielectric and electromagnetic theory that bear upon the practical business of RF & MW dielectric measurement. In all cases, more fundamental treatments can be found by following up the references in the text. Please see standard textbooks for formal introductions to the concepts and quantities we introduce here [1, 4, 6, 7, 8, 9, 10, 11, 12, 13, 14].

2.1 Basic Definitions

Please note that many of the technical terms that we introduce and use both here and elsewhere in the Guide, are also defined or explained in the Glossary, Chapter 10.

The fundamental quantity with which we will be concerned in this Guide is the *complex relative permittivity.* This is often given the symbol ε_r in the theoretical literature and it is converted to the *absolute* permittivity by multiplying it by the *permittivity of free space*, $\varepsilon_0 = 8.8542 \times 10^{-12}$ F.m^{-1}. Note that *absolute* permittivities in the SI system have units of *farads per metre*, whereas *relative* permittivities are dimensionless quantities. In the practical world we are concerned almost exclusively with relative permittivities, so we can, without ambiguity, drop the subscript '*r*' in this Guide for convenience and we instead give the complex relative permittivity the symbol ε^*, where the '*' signifies a complex quantity. As it is a complex quantity, ε^* has two components:

$$\varepsilon^* = \varepsilon' - j\varepsilon'' \qquad \ldots (2.1)$$

where, if we assume that the dielectric is placed between plane-parallel electrodes to form a capacitor, as in Figure 1, ε', the *real part of the permittivity*, characterises the *capacitative* part of the admittance, *Y*, of the capacitor and ε'' characterises the *conductive* or *lossy* part of the admittance. In all materials, ε' and ε'' depend upon ambient parameters such as the temperature, relative humidity, etc, as well as the frequency.

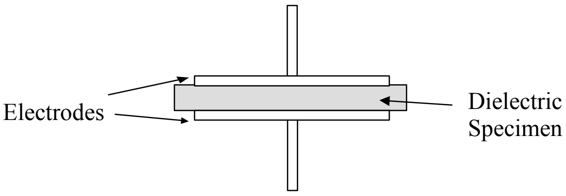


Figure 1. A dielectric specimen in a plane-parallel-electrode admittance cell.

The electrical properties of the dielectric specimen in the rudimentary admittance cell of Figure 1 can be captured in simple *equivalent circuits*, such as those shown in Figures 2 (a) and (b) in which the resistive and conductive components *R* and *G* represent the dielectric loss of the specimen. It is usually better to use the parallel equivalent circuit of Figure 2 (b) for dielectric materials because *C* and *G* are proportional to ε' and ε'' respectively, as discussed below. Note that we are concerned with practical measurements here, so we are following the Electrical Engineers' practice of using the symbol '*j*' for the square-root of minus one, $j = \sqrt{-1}$, rather than the '*i*' of the Theoretical Physicist.

Figure 2. Simple series, **(a)**, and parallel, **(b)**, equivalent circuits of the dielectric specimen in the admittance cell of Figure 1. *R* and *G* take account of the dielectric loss in the specimen. *R* is the equivalent series resistance and *G* is the equivalent parallel conductance of the dielectric.

In some circumstances it is better or more conventional to quote the *loss tangent*, $\tan\delta$, to quantify the conductive or *lossy* part of the complex permittivity, rather than ε'':

$$\tan\delta = \varepsilon''/\varepsilon' \qquad \ldots (2.2)$$

For convenience, ε', the real part of the relative permittivity, is often referred to as just 'the permittivity', while ε'' is often called 'the loss factor'. The loss tangent, $\tan\delta$, is often spoken of as *'tan-delta'*. There can be no objection to such usage as long as it is employed in contexts where confusion and ambiguity cannot occur. For small losses it is often convenient instead to refer to the *loss angle*, δ, measured in *radians*, which is the arctangent of $\tan\delta$. As $\delta \approx \tan\delta$ for small angles, they can be numerically equivalent for most practical measurements. The advantage of this is that one can use the convenient unit '*microradian*' (μrad) to quantify small losses (Similarly '*milliradian*', mrad, can be used for medium losses).

Note the *minus sign* in Equation 2.1. This sometimes causes confusion because a number of texts define ε^* with a plus sign at this point. In practical metrology, the minus sign is to be preferred because it ensures that both ε'' and $\tan\delta$ are *positive quantities* and can be tabulated as such when reporting results. To understand where the minus sign comes from, refer again to Figure 1. First, assume that we have removed the dielectric specimen from the region between the plane-parallel capacitor electrodes – but that they nevertheless remain physically fixed in space in the same positions. The admittance cell then has only a vacuum (or air, see Note (1) below) as its dielectric. Assume that its total capacitance in such circumstances is C_v. Its admittance is then $Y_v = j\omega C_v$, where $\omega = 2\pi f$ and f is the frequency in hertz (see Note (2) below). By the definition of complex relative permittivity given in the Glossary (and ignoring fringing-fields for the moment, see Section 7.1.3), we see by analogy that when the specimen is again replaced between the two electrodes in such a way that the capacitor so formed is *completely* filled with the lossy dielectric, as shown in Figure 1, the admittance becomes $Y = j\omega\varepsilon^* C_v = j\omega(\varepsilon' - j\varepsilon'')C_v = j\omega\varepsilon' C_v + \omega\varepsilon'' C_v$. The first, *imaginary*, term in this last expression shows, as expected, that the *capacitance* of the loaded capacitance cell (C in Figure 2 (b)) is $\varepsilon' C_v$, while the second, *real*, term shows that its *conductance (G),* is $+\omega\varepsilon'' C_v$. Note that this conductance is a *positive* quantity - since ε'' is defined to be a positive quantity, as the conductance must be for all passive materials. The conductance characterises the *power loss* in the dielectric - if it were negative the dielectric would be generating power!

Two important notes on the last paragraph:

(1) We have implicitly assumed (as we do for convenience throughout this Guide) that the relative permittivity of air is the same as that of a vacuum, namely 1.0000. This is not quite true. At typical ambient temperatures and relative-humidities in a laboratory we have $\varepsilon'_{air} \approx 1.0006$. In most RF & MW measurements and for most applications this difference is insignificant, but it may have to be taken into account in some cases.

(2) We have also introduced the *frequency*, f. This is a good place to point out that we generally assume that we are working in the *Frequency Domain* in this Guide and not in the *Time Domain*. Broadly speaking, *'working in the frequency domain'* means that we are measuring parameters (like ε' and ε'') as a function of *frequency*, whereas *'working in the time domain'* means that we are measuring them as functions of *time*, (see [15, 16], for a fuller explanation). Time Domain (TD) concepts are touched upon at a number of points in the text, however, most notably (i) in Section 7.1.19, which is explicitly concerned with TD measurements, and (ii) in Section 2.5, where constraints on the possible values of ε' and ε'' are deduced from a TD argument. Most RF & MW dielectric measurements are performed in the FD (which is why we concentrate on it in this Guide), however, TD measurements have their advantages - see Section 7.1.19.

The *magnetic* parameter that corresponds to the *dielectric* parameter ε^* is the *complex relative magnetic permeability*, μ^*, which may be defined and treated by analogy with ε^*:

$$\mu^* = \mu' - j\mu'' \qquad \ldots (2.3)$$

By convention in this Guide we refer to materials for which *for all practical purposes* μ^* is effectively equal to ($1.0 - j0.0$) as 'non-magnetic materials' or just simply as '*Dielectrics*'. Do bear in mind, though, that some materials that we may be tempted to think of as non-magnetic may actually be magnetic! If in doubt, it is best to check by measurement.

The introduction of μ^* brings us to a suitable point for considering issues relating to the *linearity* of the dielectric and magnetic response of materials to applied electric and magnetic fields. At sufficiently high field-strengths, dielectric materials will always respond non-linearly to applied fields, see e.g. [1, 17] and Section 6.6.3 of this Guide. However, for typical applications at RF & MW frequencies, one is more likely to encounter this phenomenon when dealing with magnetic properties (e.g. hysteresis) than with dielectric properties. Strictly speaking, the values of both ε^* and μ^* for any material are functions of *both* the applied *static* field (E or H as appropriate) *and* the corresponding *RF & MW* field magnitudes. However, the physics of dielectrics is such that for many RF & MW applications we can assume that we are working in a *low-field* approximation, i.e. that the fields are sufficiently low for the non-linear effects to be negligible. But this is, in general, *not* the case for magnetic materials.

In *optical* measurements and in *quasi-optical* measurements at and above millimetre-wave frequencies (Sections 7.1.17, 7.1.18) it is often convenient to express the dielectric properties in terms of the *complex refractive index:* $n^* = n - jk$ [18]. This quantity is related to ε^* and μ^* by:

$$n^* = n - jk = \sqrt{\varepsilon^* \mu^*} \qquad \ldots (2.4)$$

For non-magnetic materials, we have the following relations

$$\varepsilon^* = n^{*2} = (n - jk)^2 , \qquad \text{so} \quad \varepsilon' = n^2 - k^2 \text{ and } \varepsilon'' = 2nk \qquad \ldots (2.5)$$

Rather than using k to quantify the loss, it is more conventional in optical and quasi-optical systems to employ the *power absorption coefficient, α_p,*

$$\alpha_p = 4\pi f k / c \qquad \ldots (2.6)$$

where f is the frequency and c is the speed of light and α_p is the *power* absorption coefficient per unit length of signals transmitted through the medium. (Note that α_p is *not* the same as the α which appears as the real part of the propagation constant, $\gamma = \alpha + j\beta$, in a dielectric or transmission line, see Equation 2.10, below. In fact $\alpha_p = 2\alpha$). α_p is conventionally measured in the units *nepers per metre* (Np m^{-1}). As 1 Np = 8.69 dB, the power attenuation of a signal passing through the dielectric may also be expressed in decibels as $8.69 \times \alpha_p$ dB m^{-1}.

We can express the loss tangent of the material in terms of the (quasi-)optical parameters n and α_p as follows:

$$\tan\delta = \frac{8\pi f n c \alpha_p}{(4\pi f n)^2 - (c\alpha_p)^2} \qquad \ldots (2.7)$$

which for low-loss materials having $\alpha_p << 4\pi f n / c$ reduces to $\tan\delta \cong c\alpha_p / 2\pi f n$.

2.2 Basic RF & Microwave Measurement Theory

In addition to the above quantities relating to the *intrinsic properties of dielectrics*, ε^* and μ^*, we must concern ourselves also with the *extrinsic* quantities we encounter in our measurements. In this respect, dielectric measurement methods and measurement cells largely fall into two broad classes:

> (1) Those in which the dielectric properties are measured as an impedance, Z, as in Figure 2 (a), or more commonly, as an admittance, Y, as in Figure 2 (b). These may collectively be called *Lumped-Impedance Methods* and are largely used at low frequencies (LF) and in the RF region of the spectrum up to 1 GHz.
>
> (2) Those in which the dielectric is considered to be interacting with *travelling* and *standing electromagnetic waves* - these may collectively be called *'Wave Methods'.*

Both lumped-impedance and wave techniques can be employed in *resonators.* Resonators are measurement cells with *resonating* electromagnetic fields inside them that are used to obtain high sensitivity for measuring the loss of low-loss dielectrics. *Lumped-Impedance*, *Wave* and *Resonator* methods are discussed respectively in the three sub-sections that follow: 2.2.1 – 2.2.3.

2.2.1 Lumped-Impedance Methods

In these methods we need to use an impedance/admittance analyser or bridge (Section 6.1.2) or a Frequency Response Analyser (Section 6.1.3) to perform the measurements on the cell. We measure quantities, see Figure 1, such as the *cell impedance* $Z = R + jX$, where R is the equivalent *series resistance* and X is the equivalent *series reactance*. Both are measured in units of *ohms* (symbol 'Ω'). Or else we may measure the *cell admittance* $Y = G + jB$, where G is the equivalent *parallel conductance* and B is the equivalent *parallel susceptance*, both measured in units of *siemens* (symbol 'S' - formerly called 'mho'). By definition, for any lumped component we have $Y = 1/Z$. Both Y and Z are *complex* quantities, while R, X, G and B are (normally) *real* quantities. Reactances are often modelled as series inductances: $X_{LS} = j\omega L_S$, where $\omega = 2\pi f$, or capacitances, $X_{CS} = 1/j\omega C_S$, while susceptances are likewise modelled as parallel inductances: $B_{LP} = 1/j\omega L_P$ or capacitances, $B_{CP} = j\omega C_P$. The corresponding equivalent circuits for lossy capacitances are shown in Figure 2 (a) and (b), respectively.

In lumped-impedance analysis one treats continuous dielectric media, and specimens thereof, in terms of *lumped equivalent-circuits*, i.e. circuits containing discrete components: inductors, capacitors and resistors. This is perfectly acceptable as long as their physical dimensions are very small compared with the wavelength, λ, of the radiation. It is this requirement that eventually limits the usefulness of lumped-impedance methods as one moves up through the frequency spectrum. This limitation can itself be illustrated by an equivalent circuit. Assume that we have an admittance cell (see 7.1.1), such as that of Figure 1, with capacitance C between its electrodes (with or without the specimen in it - it does not matter for this illustration). The connections between the impedance analyser and the cell will give rise to a '*residual*' (i.e. *unwanted* but *small*) series resistance, R, and inductance, L, and so we end up with the equivalent circuit shown in Figure 3 for their combination.

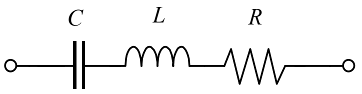


Figure 3. Equivalent circuit of a capacitance cell, including residual series inductance and resistance in the leads between the cell and the measuring instrument.

This has impedance $Z = R + j\omega L + 1/j\omega C$, so we have for the cell:

$$Y = \frac{1}{R + j\omega L + 1/j\omega C} = \frac{j\omega C}{[(1-\omega^2 LC) + j\omega RC]} \qquad \ldots (2.8)$$

If we did not initially know about the residual elements L and R in the circuit, we would find by measurement that the *apparent capacitance* of the cell as a function of frequency $C'(f)$ would be

$$C'(f) = \frac{C}{[(1-\omega^2 LC) + j\omega RC]} \qquad \ldots (2.8a)$$

Ignoring the term with R in for the moment, we see that the apparent capacitance C' of the cell rises with frequency from its d.c. value C. If $\omega^2 << 1/LC$, the rise goes as the square of the frequency, which is typical of the effects of residuals in RF measurements. If the frequency rises until $\omega^2 = 1/LC$, the admittance cell actually resonates. Now return to the R-term and the case where $\omega^2 << 1/LC$. When we multiply out the expression in Equations 2.8 and 2.8 (a), we find that it gives rise to a *conductance* term that also rises in magnitude with the frequency. In strictly lumped-circuit measurements these extra residual terms give rise to errors of measurement that limit the useful frequency range of lumped-circuit models. One can, however, extend the lumped-impedance theory to take account of the *second-order* terms like $\omega^2 LC$. One class of admittance bridge - the *Twin-T Bridge*, examples of which are the *Woods Bridge* [19] and the *Williams Bridge* [20] - is used with just such a theory up to about 200 MHz, but the equivalent-circuit model of the Woods Bridge, for example, has to take account of *87 residuals!* So in the interests of simplicity most metrologists prefer to turn to *wave methods* instead at higher frequencies, as described below, because they can take care of these effects in a more simple, direct and comprehensive way. Before looking at them in detail, however, we will stand aside and ask ourselves a more general question.

What Distinguishes the LF, RF and Microwave Regions of the Spectrum? Measurements at microwave frequencies usually differ from electrical measurements at lower frequencies because they are *conceived of* differently: they deal with *waves* rather than with *impedances* and *admittances*. This conceptual difference leads to one possible definition of the word 'microwave' which runs as follows:

> *A microwave is an electromagnetic wave whose wavelength, λ, is comparable in linear dimension to the significant functional parts of the equipment that is used to guide, manipulate or measure it.*

Traditionally, this definition would have picked out a well-defined region of the spectrum, say between 1 GHz and 150 GHz, in which components like waveguides, for example, do indeed have transverse-dimensions close to λ. More recently the advent of optical fibres would force us to admit that even light waves can be 'microwaves' in this sense! In this Guide we therefore retain a *frequency range* definition of 'microwaves' as being waves in the frequency range 1 - 150 GHz, but the *way of thinking* about microwaves encapsulated in the above definition is very important in metrology.

At LF one is typically using equipment (e.g. cells, interconnections) that is significantly smaller than λ in its linear dimensions. One can therefore work in the *long-wavelength limit*. That is to say that approximations can be used in the theory of measurement and in the measurement equations that allow one to ignore the wavelength of the signals employed. At LF one ends up precisely with the non-wave, lumped-impedance approach discussed above. This is now seen to be an *approximation* having an upper frequency limit of validity somewhere below the frequency range at which λ approximates to the typical equipment size. In fact, in accurate measurements λ should ideally be at least *ten times larger* than this size for lumped approximations to work. Likewise, in the millimetre-wave region, *above* microwave frequencies, quasi-optical methods are used (Sections 7.1.14, 7.1.17, 7.1.18) and as the frequency rises further through sub-millimetre, infra-red and up to light frequencies we find that we can work in the *short-wavelength limit* - again availing ourselves of simplifying assumptions in measurement. The concepts of the long-wavelength and short-wavelength limits are illustrated in Figure 4.

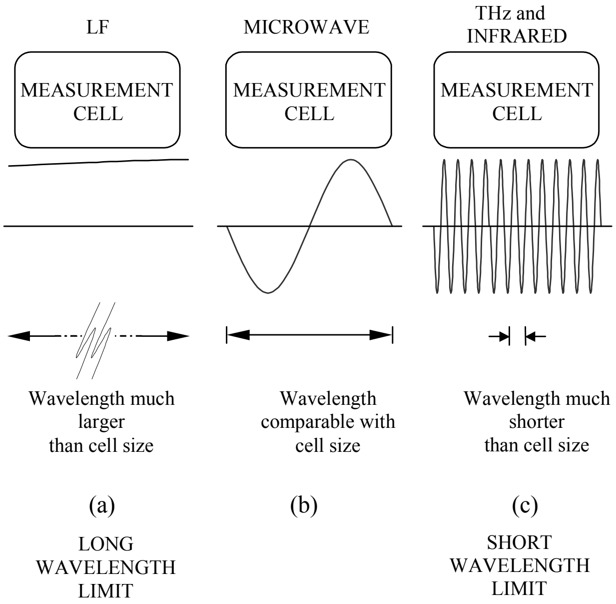


Figure 4. (a) Long-wavelength limit, **(b)** Microwave, **(c)** Short-wavelength limit.

The need to adapt equipment dimensions at microwave frequencies to match the wavelength gives rise to the requirement for many differently-sized measurement cells in this region of the spectrum. At LF and from sub-millimetre-wave frequencies upwards (above approx. 300 GHz), a single instrument/cell combination may cover many decades of frequency. For example, a single Frequency Response Analyser (Section 6.1.3) and its admittance cell can cover 5 decades of frequency at LF, while a single DFTS interferometer (Section 7.1.18) can cover 5 decades in the sub-millimetre to infra-red regions of the spectrum. While it is true that modern microwave measurement equipment, if fitted with coaxial connectors on its measurement ports (e.g. network analysers, Section 6.1.1), can also cover decades of frequency - this is *not* generally true of the microwave dielectric measurement cells or specimens themselves. Even broad-band techniques, such as the coaxial transmission-line method (Section 7.1.10), only have a frequency range of about two decades of frequency, beyond which their sensitivity drops dramatically. Sadly, many resonance techniques can operate at one spot-frequency only!

It is these distinctions that should be borne in mind when we distinguish 'LF', 'RF' and 'Microwave' in this Guide. At LF the long-wavelength limit assumptions are valid for practical measurements. RF encompasses the region of the spectrum in which long-wavelength assumptions can validly be made at the low frequency end, but they are breaking down and compromising accuracy at the high frequency end. In microwave measurements no simplifying 'short-wavelength' or 'long-wavelength' assumptions can be made at all.

2.2.2 Travelling-Wave Methods

Wave methods in general may be classified in two contrasting ways:

(1) They may be *travelling-wave* or *standing-wave* (resonant) methods. The former are discussed in detail in this Section while the latter are covered in 2.2.3. However, the basic principles of both are similar and are described in this Section.

(2) They may employ a *guided-wave* or a *free-field* propagation medium. Coaxial, metal and dielectric waveguide, microstrip, co-planar waveguide and optical-fibre transmission lines are examples of *guided-wave* media while propagation between antennas in air uses a *free-field* medium. Most of the discussions in this Guide are couched in terms of guided-wave media but many of the basic concepts can easily be extended to free-field media. Sections 7.1.14, 7.1.17 and 7.1.18 cover free-field methods in detail and they give guidance on those features that are unique to such measurements: namely the existence of diverging and converging beams and of beam polarisation, the use of lenses and the problems caused by diffraction.

In *guided-wave travelling-wave* methods the properties of the measurement cell are measured in terms of *Scattering Parameters* or 'S-parameters' [12], which are the *reflection* and *transmission coefficients* of the cell, defined in relation to *a specified characteristic impedance,* Z_0. This is usually the characteristic impedance of the transmission lines connecting to the cell. In metrology Z_0 is usually 50 Ω for coaxial transmission lines but some telecommunication systems use 75 Ω. Note that S-parameters are coefficients that refer to travelling *voltage* or *current* waves - *not* to power waves - and consequently S-parameters are *complex-number* parameters in the frequency domain which take account of the *phase* as well as the *amplitude* of the travelling waves. For a two-port test device, see Figure 5, with an input, 'Port 1', and an output, 'Port 2', the reflection S-parameters are usually given the symbols S_{11} for the reflection coefficient at Port 1 measured when Port 2 is matched to Z_0, and S_{22} for the reflection coefficient at Port 2 measured when Port 1 is matched to Z_0. S_{21} is the forward transmission coefficient from Port 1 to Port 2 and S_{12} for the reverse transmission coefficient from Port 2 to Port 1, both measured when the connections to Ports 1 and 2 are matched to Z_0. (N.B. in transmission-line theory, a *matched* port is one that has a device with input impedance Z_0 attached to it).

It is good practice in such measurements to ensure that as much of the measurement system as possible is matched to Z_0, because mismatches produce reflections and *multiple* reflections that can seriously reduce the accuracy of measurement. The well known formula for deriving the reflection coefficient, Γ, from an impedance Z which terminates a transmission-line of characteristic impedance Z_0 [21] is:

$$\Gamma = \frac{Z - Z_0}{Z + Z_0} \qquad \ldots (2.9)$$

This can equally well be taken as giving us the input reflection S-parameter of the impedance Z. Thus, $S_{11}(Z) = \Gamma$, and it shows us that if the value of Z is anything other than Z_0 - i.e. if we have a mismatch - we will also have a reflection of finite magnitude. In the transmission-line methods of Sections 7.1.10 and 7.1.11, all of the transmission line is ideally matched except the portion containing the specimen. This type of wave-analysis also applies in free space and in many other wave systems. Note that in free space $Z_0 \approx 377$ Ω for plane waves.

The propagation of electromagnetic waves through any uniform isotropic medium, or any uniform transmission-line containing such a medium, is governed by two parameters: the *characteristic impedance* Z_0, already introduced, and the *complex propagation constant*, γ, of the medium [10]. Both are functions of the complex permittivity, ε^*, and permeability , μ^*, of the medium. The propagation 'constant' governs both the wavelength and attenuation of waves moving through the medium:

$$\gamma = \alpha + j\beta = 2\pi f\sqrt{\varepsilon_0\varepsilon^*\mu_0\mu^*} \text{ or, because } c = 1\big/\sqrt{\varepsilon_0\mu_0}\,,\quad \gamma = 2\pi f\sqrt{\varepsilon^*\mu^*}\big/c = 2\pi\sqrt{\varepsilon^*\mu^*}\big/\lambda_0 \quad \ldots (2.10)$$

Where c is the speed of light in free space and λ_0 is the free-space wavelength, α is called the *attenuation constant* and β is the *phase constant*. For low-loss dielectrics in which it is assumed that $\alpha \approx 0$, β is sometimes itself called the propagation constant. Note that, from the metrological point of view, all of these so-called 'constants', just like 'dielectric constant', are misnomers because they are *not* constant: they depend upon the material medium, temperature, humidity, frequency, etc! But that is the convention and so we must accept it!

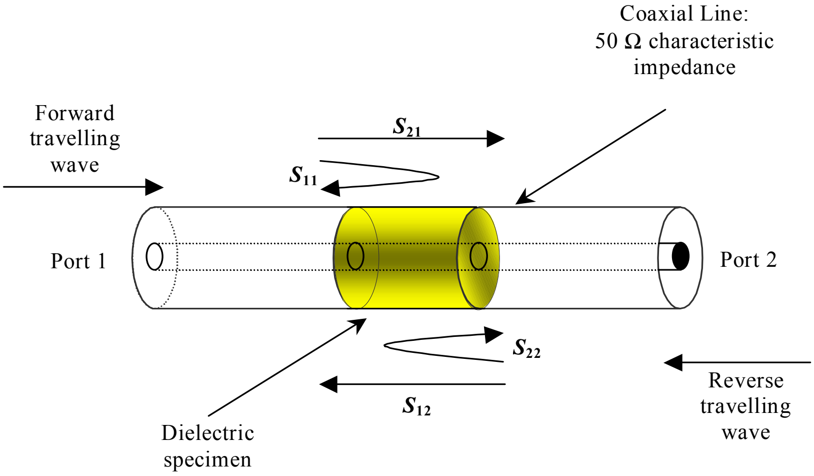


Figure 5. Electromagnetic travelling waves in a matched coaxial measurement cell that contains a dielectric specimen (see Section 7.1.10 for details of this measurement method). The cell is placed in a measurement system in which there are waves travelling in both directions, designated *forward* and *reverse* waves. The figure shows the reflection coefficients of the specimen S_{11} and S_{22}, for the forward and reverse wave respectively, and the corresponding transmission coefficients, S_{21} and S_{12}. The S-parameter subscripts refer to the measurement ports (i.e. the two ends) of the coaxial cell.

In non-magnetic materials, $\mu^* = (1.0 - j0.0)$ and

$$\gamma = \frac{2\pi f}{c}\sqrt{\varepsilon^*} = \frac{2\pi}{\lambda_0}\sqrt{\varepsilon^*} \qquad \ldots (2.11)$$

then by suitable manipulation of equations we can derive the following useful relationships ([22], Ch 1)

$$\alpha = \frac{2\pi}{\lambda_0}\sqrt{\frac{\varepsilon'}{2}(\sqrt{1+\tan^2\delta} - 1)}\,, \qquad \beta = \frac{2\pi}{\lambda_0}\sqrt{\frac{\varepsilon'}{2}(\sqrt{1+\tan^2\delta} + 1)} \qquad \ldots (2.12)$$

and

$$\lambda = \lambda_0\left[\sqrt{\frac{\varepsilon'}{2}(\sqrt{1+\tan^2\delta} + 1)}\right]^{-1} = 2\pi/\beta = \lambda_0\frac{\beta_0}{\beta} \qquad \ldots (2.13)$$

where λ and β are the wavelength and the propagation constant in the dielectric medium respectively and β_0 is the propagation constant in the absence of the dielectric (i.e. for vacuum dielectric).

In a low-loss dielectric α is small ($\alpha << \beta$) and we find:

$$\lambda \approx \lambda_0/\sqrt{\varepsilon'}\,,\ \beta \approx 2\pi\sqrt{\varepsilon'}/\lambda_0 \text{ and } \alpha \approx \pi\sqrt{\varepsilon'}\tan\delta/\lambda_0 \text{ or } \alpha \approx \pi f n\tan\delta/c\,,$$

so $\tan\delta \approx c\alpha/\pi fn$ and referring back to the discussion of Equation 2.7, we find $\alpha = \alpha_p/2$. This relationship actually follows more fundamentally from the definitions of α and α_p. The first is the exponential coefficient for decay of *field-strength* as the wave propagates through the medium, the second describes the decay of *power*, so the relationship $\alpha = \alpha_p/2$ is valid even if α is not small.

As dielectric metrologists we can readily see from all of these equations that the measurement of *propagation* or *transmission* parameters, like β, γ and λ, provides a means for deriving dielectric parameters. Many dielectric measurement methods are based upon this principle. The same is true of the measurement of *reflection* parameters, Γ, S_{11}, S_{22}, etc. Reflections at plane interfaces between two uniform media are governed by the *intrinsic wave impedances* of the media, η_1 and η_2, respectively, where $\eta_1 = \sqrt{\mu_1 * / \varepsilon_1 *}$ and $\eta_2 = \sqrt{\mu_2 * / \varepsilon_2 *}$, where μ_1*, μ_2*, ε_1* and ε_2* are the *complex relative permeabilities* and *permittivities* of the two media respectively. By analogy with Equation (2.9), the reflection coefficient in Medium 1 of a wave *at normal incidence* (i.e. at 0° angle of incidence) on Medium 2 is:

$$\Gamma = \frac{\eta_2 - \eta_1}{\eta_2 + \eta_1} \qquad \dots (2.14)$$

Note that such reflections are dependent upon the *ratio* of μ* and ε*, whereas the transmission parameters defined in Equation 2.10 are governed by the *product* of μ* and ε*. This has important consequences for the measurement of the permeability of magnetic materials by wave methods: in order to separate μ* and ε* we can measure the specimen *both* in transmission and reflection. This is readily achieved by measurements in transmission cells, such as the one shown in Figure 5, in which all four S-parameters are easily determined. Further details are given in Section 7.1.10. If we are sure that we have a non-magnetic material however, we have $\mu^* = (1.0 - j0.0)$ and we can measure ε* *either* by transmission *or* reflection methods. The choice should normally be made on the basis of which is the most accurate, see Section 7.1.10. For such non-magnetic materials, we see that the reflection from a dielectric interface between two media with permittivities ε_1* and ε_2* travelling at normal incidence from Medium 1 to 2 is:

$$\Gamma = \frac{\sqrt{\varepsilon_1 *} - \sqrt{\varepsilon_2 *}}{\sqrt{\varepsilon_1 *} + \sqrt{\varepsilon_2 *}} = \frac{n_1 * - n_2 *}{n_1 * + n_2 *} \qquad \dots (2.15)$$

where n_1* and n_2* are the complex refractive indices of the two media as defined in Section 2.1. For free-field measurements in which the angle of incidence is not necessarily normal these equations may be generalised for any angle of incidence by use of Fresnel's Equations [23, 10]. In the free-field (see Section 7.1.14) it is also important to take account of whether the electric field ('E-field' for short) polarisation of the incoming radiation is *parallel* to the dielectric surface or in a plane *perpendicular* to the surface. There are two sets of Fresnel's equations – one each for the parallel and perpendicular cases.

<u>A Note on Terminology</u> In dielectric metrology travelling-wave methods are often referred to as *Single-Pass* or *Double-Pass* methods. *Single-pass* implies that the wave that interacts with the dielectric specimen passes through it once: i.e. that one is using a *transmission method*, either in a transmission-line or in free-space. *Double-pass* implies that the wave passes through the specimen twice, as in many *reflection methods*. Note, however, that with very high-loss materials the signal may be absorbed before it is reflected back from the rear face of the specimen. The measured reflection is then from the front face only. Then one determines ε* purely from the measured mismatch and the wave cannot strictly be said to pass through the specimen at all.

Much of the important theory of E.M. travelling-wave metrology is contained in the wide range of readily available books, guides and manuals that explain the use of Automatic Network Analysers (ANAs) – please see Section 6.1.1 and, e.g., [11, 12]. The important concept of *ANA Software Error Correction* is nowadays central to all accurate RF & MW measurements by wave methods. In dielectric measurements it is the means by which we are easily able to correct for the imperfections of connectors, leads and many residuals - i.e. it can specifically correct for the circuit imperfections that limit the accuracy and frequency range of measurement.

2.2.3 Resonance, Cavity, Standing-Wave and 'Multi-Pass' Methods.

Resonance methods are generally used for measuring the loss of low-loss dielectrics (Section 5.3.2). The cell in such measurements is commonly referred to as a *cavity* or *resonator*. In such cells the real permittivity, ε', is typically determined by measuring the change in resonant frequency when the specimen is inserted into the resonator or else it can be measured by changing the length of the resonator to return it to resonance at the same frequency. These contrasting methods are known respectively as the *frequency-change* and *length-change* methods for determining ε'. It is possible to measure ε' by the length-change method because the wavelength in the dielectric medium, λ, differs from that in free space (Equation 2.13). The determination of dielectric loss, ε'' or tan δ, in such resonators usually proceeds via the measurement of *Quality-Factor*, otherwise known as *Q-factor* or *Q*: see Section 6.4.

Both lumped-impedance and wave techniques can be employed in resonators. In the former case an admittance cell can be resonated with an external inductor. This RF '*dielectric-test-set'* method (Section 7.1.4) was first used for measuring low-loss dielectrics in the 1930s. At higher frequencies where wave methods are more appropriate, the resonator is often modelled using *standing-wave* equations rather than a *travelling-wave* analysis. Note, however, that wave resonance methods are also often referred to as *'multi-pass techniques'* because a travelling wave in the cell, in bouncing backwards and forwards between the two ends of the resonator, will pass through the dielectric specimen many times before it is absorbed. This concept helps to explain why resonant methods are more sensitive to low losses than single and double-pass techniques. Note also, though, that this multi-pass travelling-wave picture and the standing-wave picture are formally mathematically equivalent: they predict the same E- and H-fields in the resonator. Further details of standing-wave and multi-pass methods are to be found in Sections 5.3.2, 6.1.9, 7.1.5 - 9 and 7.1.15 -17.

2.3 Loss Processes: Conduction, Dielectric Relaxation, Resonances and Nonlinearities.

Section 2.2 has covered many of the basic definitions and concepts that we need to use elsewhere in the Guide. In this Section we turn to *the physics of dielectric response behaviour*.

The most relevant physical processes here are those that give rise to power loss in bulk dielectrics. In the RF & MW region of the spectrum such power losses arise largely from four clearly distinguishable physical processes. It is not our major concern in this document to explain the theory of these processes in any great detail - for this see [24, 25, 26] or [4] and [14] for an introduction - but we do need to understand their basic features because they affect our approach to measurement. If we are trying to understand, as metrologists, the loss processes present in a dielectric, we should choose a measurement technique appropriate for the physical processes that are of interest.

The four physical processes associated with mechanisms for generating loss (i.e. conversion of electromagnetic energy into other forms of energy - see Glossary) are commonly called '*loss processes*'. They are: (1) *Electrical Conduction*, (2) *Dielectric Relaxation*, (3) *Dielectric Resonance* and (4) *Loss from Non-Linear Processes*. They are explained in the following paragraphs.

(1). *Electrical Conduction* in which charge carriers (electrons, ions or holes) in a material medium are free to move physically through the medium under the influence of an electric field. The ease of conduction is quantified by the *conductivity* of the medium, σ, measured in siemens per metre (S m^{-1}). In general, σ will depend upon frequency, temperature, concentration of carriers, etc, though in many materials σ will change only slowly with frequency in the RF & MW range. If there is uniform conduction in a uniform medium this process will not affect the value of the real permittivity ε' of the material. However, if uniform conduction is prevented by the interpenetration of insulating regions in the dielectric, a *Maxwell-Wagner Effect* [4, 24, 7] can occur, which can give rise to behaviour that mimics a *dielectric relaxation* (see below) in which ε' falls with frequency. In complex structured materials and conductor/insulator composites, the charge carriers may migrate by a *percolation* process [27], which can also result in dielectric behaviour similar to that of dielectric relaxations (see below).

(2). *Dielectric Relaxation* refers to the response of the *electric dipoles* in a material medium to the applied alternating electromagnetic fields. Many different types of dipoles can be present in dielectric media. Some materials have *permanent* molecular dipoles inside them and they are called *polar materials.* Their molecules are referred to as *polar molecules*. Materials in which the dipoles are induced only by the application of the electric field itself are called *non-polar materials*. Polar molecules typically exhibit a number of different relaxation processes. If they are in a liquid phase they can rotate bodily to try to align themselves with the field, giving rise to *rotational polarisation.* Otherwise portions of large molecules can move with respect to each other, giving rise to one or more *distortional polarisation* processes, each with its own relaxation behaviour. As mentioned in the last paragraph, any interfaces in a material that prevent or inhibit the passage of charge carriers will have dipolar layers set up across them when the E-field is applied and they give rise to *interfacial polarisation*, a phenomenon, essentially similar to the Maxwell-Wagner Effect, which exhibits its own relaxation behaviour. The membranes in the cells of biological tissues typically exhibit this behaviour [28]. In a complex medium, many or all of these processes may be present, giving rise to very complex relaxation behaviour. Each process will have its own *strength,* which is a parameter that measures the extent to which it contributes to the total magnitude and behaviour of ε' and ε''.

Seen in the frequency domain, the characteristic feature of any relaxation process is the response of its dipoles to the applied alternating E-field. The net polarisation of the dipoles attempts to ‘keep up’ with the applied alternating field, but as the frequency increases it increasingly fails to do so and therefore it lags behind the field in phase. In the simpler models of relaxation, each process is therefore characterised by a relaxation time, τ_r, characterising the speed at which it is able to respond. For the metrologist working in the frequency domain, it is perhaps more useful to refer to a *relaxation frequency* f_r, where $f_r = 1/2\pi\tau_r$. This is the practice we follow in this Guide.

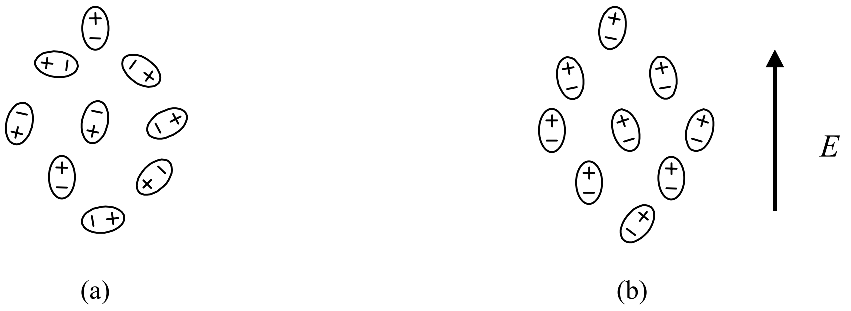


Figure 6. The effect of applying an electric field (E-field) to an ensemble of rotationally free molecular dipoles (e.g. polar molecules in a liquid). **(a)** When no field is applied thermal excitation ensures that the dipoles are randomly orientated. **(b)** When an electric field is applied the dipoles tend to orientate themselves parallel to the field, imparting an overall polarisation to the ensemble.

The reader is referred to the literature [26, 24, 25] for formal scientific models and explanations of dielectric relaxation, but the following simplified phenomenological description of the behaviour of ε' and ε'' as a function of frequency is broadly characteristic of any relaxation process. It is illustrated in Figure 6. We consider three frequency ranges:

(i) At low frequency (LF), well below f_r, the dipoles easily respond to the applied E-field and readily align themselves with it. They vibrate or rotate almost in phase with it. The polarisation will achieve the maximum value that the physics of the system will allow, and this allows the aligned dipoles to make a full contribution to the value of ε'. The portion of the total value of ε' contributed by this process (as opposed to the contributions from other processes) is therefore high at LF. Electromagnetic energy is stored in the polarisation of the molecules, but little is converted to heat, so ε'' is low at LF. Now, in fact, the description just given and Figure 6 (b) are a little too simplistic. For all but very high E-fields, the situation is always more complicated! A more exact picture is as follows. At ambient temperature, and for the typically low-magnitude applied E-fields that one normally applies in the RF & MW laboratory, the level of polarisation is completely overwhelmed by the random thermal motion and rotations of the molecules. Application of the E-field therefore merely induces a *statistical preponderance* of polarisation in one direction rather than the other, but this process can nevertheless respond quickly enough to the LF signal to produce the net effect described above. If *very high* E-fields (over 10 kV m^{-1}) are applied, however, *polarisation saturation* can occur [1], which is actually the situation shown in Figure 6(b) where the dipoles are virtually completely aligned. In this case, the E-field-induced polarisation dominates over the thermal effects, and this can give rise to *high-field non-linearities* in polarisation (and so in ε') that are discussed further in Section 6.6.3.

(ii) At frequencies close to the relaxation frequency, f_r, the molecular dipoles struggle to keep up with the applied alternating RF field, so their net polarisation magnitude is lower than that of the LF response and their vibrations typically lag in phase. The net result is that their contribution to ε' is lower than at LF, so ε' falls with frequency, while ε'' peaks at f_r, giving rise to significant dielectric loss.

(iii) At frequencies well above f_r, the dipoles are so relatively sluggish that they find it very difficult to respond to the applied alternating field at all. As the frequency rises they are able to do so less and less effectively and so the contributions to ε' and ε'' from the relaxation process fall away towards zero.

A typical response is shown in Figures 7 and 8 for the simplest model of dielectric relaxation behaviour, known as *Debye Relaxation* (see below). This behaviour is exactly analogous to the behaviour of the equivalent circuit shown in Figure 9 with $f_r = 1/2\pi RC_1$. An illustration of this Debye behaviour is given in

Section 9.1 - which looks at an equivalent circuit for the relaxation of liquid deionised water.

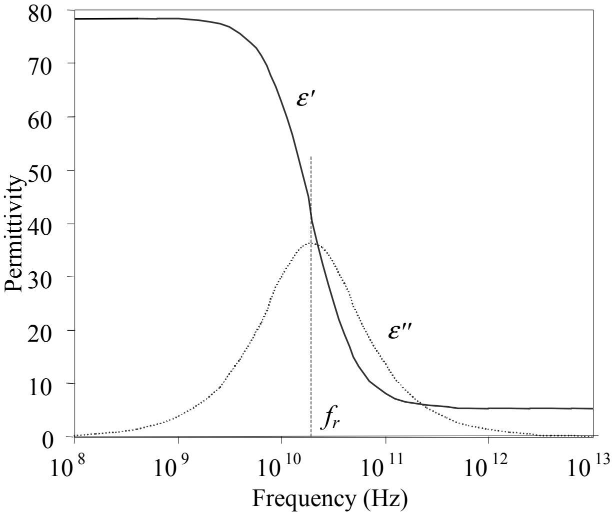


Figure 7. A typical Debye Relaxation response, see Equation 2.16. This plot is for deionised water, see further details in Section 9.1. The diagram omits the effects of all other loss processes in the water. The use of a logarithmic scale demonstrates just how slowly dielectric properties change with frequency when governed by a relaxation process.

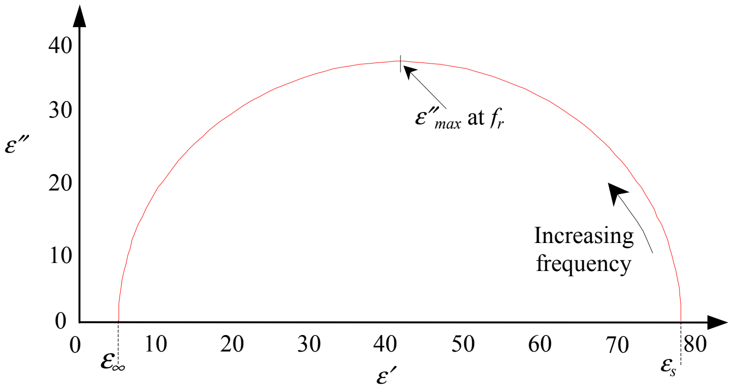


Figure 8. A ‘Cole-Cole Plot’ of the same data given in Figure 7, in which ε'' is plotted against ε' giving a semi-circular trace that is characteristic of a Debye Relaxation. Frequency increases as one moves anticlockwise around the trace from ε_s to ε_∞. See Equation 2.16 (below) for explanation of ε_s, ε_∞ and f_r.

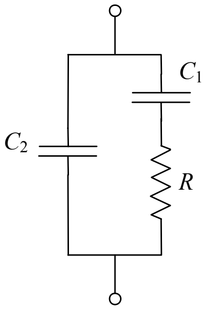


Figure 9. An equivalent circuit of the Debye Relaxation of Figure 7.

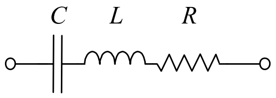


Figure 10. A simple equivalent circuit of a dielectric resonance.

(3). Dielectric Resonance. Dielectric polarisation *resonance* should not be confused with *dielectric relaxation*. A simple equivalent circuit of a resonance is given in Figure 10, which may be compared with the circuit for relaxation in Figure 9. The physics of relaxations and resonances is completely different and the two should not be confused. A resonance may appear as a sharp or broad feature in the frequency domain, depending on its Q-factor (see Section 6.4 for a discussion of Q-factor) whereas relaxations *always* exhibit *very broad* spectral features. Furthermore, a resonance gives rise to a frequency dependence for ε' (or indeed a frequency dependence for μ' in magnetic resonance) in which ε' (or μ') can either *fall* or *rise* with frequency, whereas in relaxation behaviour ε' (or μ') can only *fall* with frequency.

In general, at RF & MW frequencies the physics of *linear*, *homogeneous*, *non-composite solid* and *liquid* dielectrics does not allow a resonance to occur. The molecules of such materials, because of their close proximity to one another, interact to such an extent that all potential RF & MW resonances are damped effectively into non-existence. Therefore …

> *(i) any sharp features that we appear to measure in the spectrum of ε' and ε'' at RF & MW frequencies for linear, homogeneous, non-composite solid and liquid dielectric materials, and ...*
>
> *(ii) any apparent increases in ε' with frequency ...*
>
> *are invariably caused by non-intrinsic effects, usually imperfections in our measurement cells!*

There *are* however, genuine dielectric resonances in the *infra-red* region in the spectra of solids and liquids. At these frequencies the energy in the electromagnetic (E.M.) radiation is high enough to excite resonant polarisation of the electron clouds around atoms and molecules. This phenomenon is called *electronic polarisation*. There are also resonances at these frequencies associated with the *ions* in a lattice of an ionic material. Both of these processes give rise to *small but measurable* effects at RF & MW, see (i) below.

As just described, the *resonance frequencies* of the *intrinsic* resonance processes in *homogeneous*, *linear*, *non-composite solid* and *liquid* dielectrics do not lie in the RF & MW region of the spectrum, but this does *not* mean that we do not encounter *some* effects of material-related resonances in this region of the spectrum. We should, in fact, recognise the following important RF & MW phenomena relating to dielectric resonance:

> (i) Low Frequency Resonance Tails. The infra-red resonances *do* produce effects at RF & MW frequencies. In many pure non-polar materials dielectric *relaxation* behaviour can be insignificant at RF & MW frequencies. Such non-polar materials therefore tend to exhibit very low relaxation loss, but this allows other loss processes, though small, to become dominant for such materials. The infra-red resonances caused, for example, by electronic polarisation, have *low frequency tails* which can dominate over relaxation losses in many pure inorganic crystals. Typically in the MW range one can measure loss angles of a few tens of microradians from this cause, generally rising slowly with frequency.
>
> (ii) Gases. If their pressure is suitably low, gases, as opposed to solids and liquids, *do* exhibit sharp spectral lines in the RF & MW region of the spectrum caused by molecular resonances [29]. These resonances form the basis of the science of the *microwave spectroscopy* of gases. The damping away of these resonances that occurs in solids and liquids does not occur in low-pressure gases where the interaction between molecules is low. As the pressure is increased, however, and the gas density increases, molecular interactions also increase and one experiences the phenomenon of *pressure broadening of spectral lines* [29], effectively a lowering of Q-factor, and ultimately, in the limit as one approaches the density of liquids and solids, the complete damping away of the resonance
>
> (iii) Resonances in Composite Materials. An important parameter associated with *composite materials* (Section 5.3.7) is a *structure-length, s,* corresponding to the typical linear dimension of the component particles or structures that make up the material. If the frequency is such that the e.m. radiation wavelength in the material is close to *s*, resonances can occur within the meso- or macroscopic *particles* and *structures* that make up the material. This is particularly prominent if one component of the material is a high-permittivity low-loss component, such as a sintered ceramic. The particles of such materials can effectively act as if they were dielectric resonators (Section 7.1.15) because they can store e.m. energy through the mechanism of total-internal-reflection. Bear in mind that if the free-space wavelength is λ_0, the wavelength in the material will be $\lambda_0/\sqrt{\varepsilon'}$ (Section 2.2, Equation 2.13). So if $\varepsilon' \cong 1000$, resonances can occur in the millimetre-wave region of the spectrum even if *s* is as small as 0.1 mm. It is usually this phenomenon that limits the useful frequency range of dielectric composites, see

Section 5.3.7. Of course, such resonance effects can also be employed *deliberately,* as they are in Frequency Selective Surfaces (FSSs) and Photonic (or Electronic) Band Gap (PBG or EBG) materials.

(iv) While we do not expect dielectric resonances to occur in homogeneous solid materials in the RF & MW range, we can expect *magnetic resonances* to occur in this range. This will be evident from the response of μ' and μ'' as a function of frequency. Ferrites, for example, exhibit *ferrimagnetic resonances* in the RF & MW region of the spectrum [30, 31, 32, 33]. See also [13], Ch. 15.

(4). *Loss from Non-Linear Processes.* It is well known that *hysteresis* in magnetic materials leads to loss [34]. Ferroelectrics [1, 7, 35] exhibit much the same phenomenon, leading to an independent source of electrical loss. The relative magnitude of these non-linear effects usually rises with the amplitude of the applied fields.

Having listed the four common sources of loss in dielectrics we return to *Dielectric Relaxation* (2) and look at it in more detail because it is much the most characteristic of loss processes in the RF & MW region of the spectrum and so it deserves further consideration.

Dielectric Relaxation Models. As already stated, the *Debye Relaxation* is our simplest relaxation model [4, 17, 24], see Figures 7 & 8. It corresponds to the equivalent circuit in Figure 9. Three *real-number* parameters (corresponding to the three lumped components in the equivalent circuit) are used to characterise the frequency response of the relaxation: (i) the *'static' permittivity*, ε_s, i.e. the value of ε' at very low frequencies, (ii) the *relaxation frequency*, f_r, discussed above, and (iii) the *high-frequency permittivity limit,* ε_∞, i.e. the value of ε' at high frequencies, well above f_r. All three parameters are functions of temperature. The *Single-Debye* dielectric response is then described by the equation:

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + jf/f_r} \qquad \ldots (2.16)$$

The consequent behaviour of ε' and ε'' is shown in Figures 7 and 8. Equation 2.16 describes the relaxation equation corresponding to just *one* loss process, so the quantity (ε_s - ε_∞) in the equation should be interpreted as the *strength* or the *contribution* to the total complex permittivity from this one process. What happens when more than one loss process is occurring in a material can be illustrated by the *Double-Debye Equation* which has two dielectric relaxations:

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_h}{1 + jf//f_{r1}} + \frac{\varepsilon_h - \varepsilon_\infty}{1 + jf//f_{r2}} \qquad \ldots (2.17)$$

Where f_{r1} and f_{r2} are the relaxation frequencies of the two relaxations and ε_h is the notional high frequency permittivity limit of the lower frequency relaxation. For most practical purposes at RF & MW frequencies, the relaxation behaviour of *deionised water* can effectively be modelled by the *Single Debye Relaxation* formula (see the worked example in Section 9.1). However, *Double* and *Multiple Debye* behaviour is also an important phenomenon for dielectric metrologists because it is typically exhibited by the low-molecular-weight alcohols [36, 37], e.g. propanol and butanol, that are used as dielectric reference liquids, see Section 4.2.2.

Many years of study of the phenomena of dielectric relaxation by means of dielectric spectroscopy (see e.g. [25]) have led to the development of a range of more detailed relaxation models, of which the Cole-Cole, Cole-Davidson, Havriliak-Negami relaxations are typical:

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{1 + (jf/f_r)^{1-\alpha}} \qquad \text{(Cole-Cole [38])} \qquad \ldots (2.18)$$

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{(1 + jf/f_r)^{1-\beta}} \qquad \text{(Cole-Davidson [39])} \qquad \ldots (2.19)$$

$$\varepsilon^* = \varepsilon_\infty + \frac{\varepsilon_s - \varepsilon_\infty}{(1+(jf/f_r)^{1-\alpha})^{1-\beta}} \qquad \text{(Havriliak-Negami [40])} \qquad \ldots (2.20)$$

Where α and β are additional fixed parameters and where the Havriliak-Negami formula, Equation 2.20, captures the features of both the Cole-Cole and Cole-Davidson models. All three models require more parameters to characterise them than the Debye Relaxation, and their relaxations are even broader in the spectral domain than the Debye Relaxation.

Over the years attempts have been made to understand all but the Debye relaxation behaviour *phenomenologically* in terms of *distributions* of relaxation times [24, 25] but it is nowadays possible to present models that capture more of the *physics* of dielectric behaviour at a molecular level. These physical models include those that take account of co-operative behaviour between different-sized regions in the dielectric, e.g. the Dissado-Hill relaxation model [41]. Other types of model are used to characterise charge-carrier transport in complex composites via percolation in fractal structures [27]. Some of these processes give rise to *very broad* relaxations in the spectral domain, Figure 11 [25, 41]. But one should not over-generalise the utility of any one of these relaxation models: there are many different physical causes of dielectric relaxation and just as many models have to be used to describe them. Metrologically, the main thing to remember about all of these relaxations is that their response in the spectral domain is *very broad*, and if ε' and ε'' are to be plotted against frequency it is often best to plot against log (*f*), rather than *f*, if one is to exhibit all relevant features of their relaxation - see Figure 11 and Section 8.

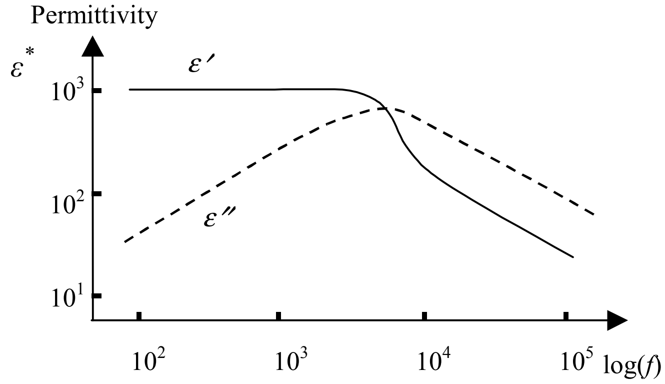


Figure 11. A typical broad relaxation from a complex dielectric material.

It can readily be shown from these relaxation models that the ε' contribution from dielectric relaxations *always falls* with frequency (or else, in the limit, at very low and high frequencies, it may asymptotically be approximately constant). Given (i) that d.c. conductivity, σ, has no effect upon ε', (ii) that intrinsic dielectric resonance does not occur in homogeneous non-composite dielectric solid and liquid materials in the RF & MW region and (iii) that dielectric behaviour in this region is normally dominated by relaxations, we can make the following general statement:

> *In the RF & MW region of the spectrum ε' always falls with frequency (or, in the limit, remains stationary) in homogeneous solids and liquids that exhibit a linear response to the applied fields.*

But as we have seen in the discussion labelled (3) on dielectric resonances, above, this statement does not necessarily apply to (i) gases, (ii) composite materials with a structure-length close to the E.M. wavelength in any one of the components in the material, (iii) non-linear materials.

If, on the other hand, one is measuring linear, homogeneous, non-composite dielectric solid and liquid materials and one actually finds ε' to be *rising* with frequency, *one can be sure that one's measurements are in error* and that the effect is almost certainly associated with the measuring instruments that one is using, or perhaps with the mathematics one is using! It is possible that there are exceptions to this rule, but we are not aware of them.

► ***Do's and Don'ts***

Do remember that for homogeneous, non-composite, solid and liquid materials with a linear response to voltage at RF & MW frequencies any *measured* sharp changes of properties with frequency, especially resonance, are invariably instrumental artefacts, i.e. caused by systematic errors.

Don't forget that in such materials in the RF & MW range, ε' invariably falls with frequency (or in the limit remains constant).

2.4 Ambiguity in the Definition of Conductivity, Loss Factor and Loss Tangent.

In this section, we issue a warning. As we have seen in Section 2.3, there are a number of physical processes - *conductivity*, *relaxation*, *resonance*, *hysteresis* etc. - that give rise to loss in dielectrics. However, when we measure the loss of a dielectric at a single frequency we cannot, in general, distinguish between them. *Phenomenologically* they all give rise to just one measurable quantity – namely *the total measured loss*. This fact leads to an *ambiguity of terminology* in the field of dielectric measurements which afflicts *all* loss parameters, but most notably *conductivity* σ, *loss factor* ε'', *loss tangent* $\tan\delta$ and *loss angle* δ.

There are two different conventions that are followed:

(i) *The Physical Convention or Usage*, in which σ, measured in siemens per metre, describes *only* the conductivity arising from transport of free charge carriers (as discussed in (1) in Section 2.3) and in which ε'', $\tan\delta$ and δ describe *only* dielectric (i.e. dipolar) behaviour (discussed in (2) & (3) in 2.3). Thus a *distinction* is made between the two different types of physical process. If the dielectric loss factor is ε'', then the analogous 'loss factor' for the conductivity of the charge carriers expressed in the same dimensionless units is $\sigma/2\pi f\varepsilon_0$ and the *total effective loss factor* (i.e. the analogue of ε'' that characterises the *total* loss in the dielectric) is $(\varepsilon'' + \sigma/2\pi f\varepsilon_0)$.

(ii) *The Phenomenonological Convention or Usage*, in which only the *total* loss is quoted, and it is expressed *either* by the single parameter ε'' *or* by the single parameter σ, *irrespective of the physical cause*. In this case ε'' and σ are *interchangeable* via the relation $\sigma = 2\pi f\varepsilon_0\varepsilon''$, i.e. they present two different ways of quantifying the same phenomenon: namely the *total measured loss*. (Loss tangent, $\tan\delta$, may be used in this way too, with its usual definition: $\tan\delta = \varepsilon''/\varepsilon'$). The convention of using the loss factor ε'' (or $\tan\delta$) to quantify total losses, including conduction losses is used in many publications, including this Guide, but in *biomedical studies* the parameter σ is commonly used for total loss rather than ε'', as in, e.g. [42].

Unfortunately, when reading the published literature, one is often not sure which convention is being followed.

Recommendation: One would like to recommend in this Guide that only one of these conventions should be followed, but both are so entrenched and in their own ways so convenient and useful, that this would be futile. One can only recommend that in reporting one's results one must be *explicit*, through the use of words or equations, as to which convention one is following. One option may be to refer to *dc conductivity* for charge transport conductivity when using the physical convention and *ac conductivity* or *total effective conductivity* in the phenomenological convention, but these terms are not universally accepted.

To follow our own recommendation we should make it clear that *in this Guide, unless otherwise stated, we follow the Phenomenological Convention:* i.e. ε'' and $\tan\delta$ are used here to describe the *total* loss measured in the dielectric, irrespective of its physical cause. This is more appropriate in a metrological context as only total losses can actually be measured. The physical convention can only be used if one has a *physical model* that can distinguish between the loss caused by the passage of charge carriers and the loss caused by dielectric (dipolar) processes. Such models were introduced in Section 2.3, from which one can see that one needs to perform measurements across a broad band of frequencies to gain enough information to implement such a physical model.

2.5 The Relationship between ε' and ε'' - The Kramers-Kronig Relations.

(Note: much of the following is taken from *EMMA-Club Technical Note 1*, see [43]. The Technical Note provides more information on the formulae that are used).

Consider a homogeneous dielectric specimen that is linear in its electrical response. In the frequency domain its response to an applied electric-field is characterised by *two* parameters: $\varepsilon'(f)$ and $\varepsilon''(f)$, where f is the frequency. These are the two parts of the complex parameter ε^* discussed in Section 2.1. Viewed in the *time domain*, however, the relationship between the voltage, V, applied across a capacitor formed from this dielectric as in Figure 1, and the current that passes through it, I, will be given by a *single* function of time, t. The fact that both $\varepsilon'(f)$ and $\varepsilon''(f)$ are derived from a *single real function*, $I(t)$, of a *single real parameter* (t) implies that their values must be intimately related to each other. They are, in fact, related by the *Kramers-Kronig Relations* [25, 44]. The significance of these relations is that if $\varepsilon'(f)$ is known over a sufficiently wide frequency range, $\varepsilon''(f)$ can be computed directly from it. Likewise, if $\varepsilon''(f)$ is known over a sufficiently wide frequency range, along with ε_∞, the high frequency real-permittivity limit (see Section 2.3), $\varepsilon'(f)$ can be computed directly from it.

The quantities ($\varepsilon'(f)$ - ε_∞) and $\varepsilon''(f)$, in fact, form a *'Hilbert Transform Pair'*. So, if we have measured the values of both $\varepsilon'(f)$ and $\varepsilon''(f)$, these quantities can be used to check each other: that is to say that the internal *consistency* of our measurements can be checked. The mathematically inclined can use the full Kramers-Kronig relationships in exactly this way and this expedient can be recommended when we have a considerable body of data available to us over a wide frequency range. See for example [25], [41] or [44]. But for a simple, cost-effective check upon measurements over a limited frequency band, the full Kramers-Kronig relationships are rather inconvenient to use and are unnecessarily complicated to apply as a simple check of consistency.

A simplified equation. In 1971 Arnold Lynch derived and published [45] a simplified equation for relating changes in ε', $\Delta\varepsilon'$, to $\tan\delta$ when the electrical response of the material is determined by dielectric relaxation behaviour. The formula is:

$$\Delta\varepsilon' / \varepsilon' \approx m \ \tan\delta \log_{10}(f_2 / f_1) \qquad \text{... (2.21)}$$

Here $\Delta\varepsilon' = (\varepsilon'_1 - \varepsilon'_2)$, is the difference in permittivity measured at frequencies f_1 and f_2 respectively. If we make $f_1 < f_2$, we will have $\varepsilon'_1 > \varepsilon'_2$, and with the definition just given, $\Delta\varepsilon'$ will be a positive quantity representing the *fall* in ε' between f_1 and f_2. Note that dielectric relaxation always results in a fall in ε' with frequency, as explained in Section 2.3. The value of the numerical parameter m in Lynch's derivation depends upon assumptions made about the nature and frequency spread of the relaxation processes which are giving rise to the loss, $\tan\delta$, and to the changes in ε'. It is generally the case that $1.0 \le m \le 2.3$, however, and as Lynch noted, setting $m \approx 1.5$ provides an equation which holds within a factor of 1.5 for most practical cases. Expressions of this general sort were known to the dielectrics community well before 1971 [46, 47] but Lynch's contribution was to note just how useful this equation could be for checking the consistency of dielectric measurements.

How to use Equation 2.21 for checking dielectric measurements. The approximate equation, Equation 2.21, applies to *homogeneous*, *linear* dielectrics over frequency ranges at which $\tan\delta$ can reasonably be construed to have a meaningful 'average' value. Reference to Lynch's paper [45] should be made for an idea of what 'reasonable' might mean in this context. Changes by a factor of 10 in $\tan\delta$ from f_1 to f_2 will clearly not give a well-defined 'average' from which an accurate check of $\Delta\varepsilon'$ can be derived. However, $\tan\delta$ usually changes very slowly with frequency in typical dielectric relaxations and a change by a factor of 2 from f_1 to f_2 will generally give agreement for $\Delta\varepsilon'$ within Lynch's allowance factor of 1.5, provided that $\tan\delta$ is measured at a median 'centre' frequency between f_1 and f_2 measured on a logarithmic scale of frequency. The worked examples in Section 9.2 show how this formula can be applied in this way in practical measurements.

When the formula cannot be applied. Lynch showed that Equation 2.21 is reasonably accurate if the dielectric response is dominated by relaxation behaviour, i.e. if $\tan\delta$ is varying slowly with frequency. One should not expect it to hold at much higher frequencies (e.g. infra-red) where sharp dielectric resonances, rather than relaxations, can occur and it will not apply either in composite dielectrics that are resonating internally (see Section 2.3). Care in its application must also be taken with *conducting* materials, i.e. materials with free charge carriers. Consider such a material with free-charge conductivity σ_p, whose behaviour, for all practical purposes in the frequency range of interest, can be described by the equivalent circuit of Figure 2 (b). As noted in Section 2.4, when measuring at a single frequency we usually cannot distinguish the *true* dielectric loss, caused by a relaxation (which, in fact, is absent in this example) and the *apparent* dielectric loss caused by the effects of conduction. We will obtain an *equivalent* (phenomenological) loss tangent, given by $\tan\delta_e = \sigma_p / 2\pi f\varepsilon'\varepsilon_0$, where

ε_0 is the permittivity of free space. So there is an apparently *finite* value for tan δ to insert into Equation 2.21, giving rise to a *finite* predicted $\Delta\varepsilon'$. But in fact $\Delta\varepsilon' = 0$ for such a material. Thus, unless additional information is available which allows one to distinguish physical conductivity losses from relaxation losses, or unless it is known that the conductivity loss, though finite, is small compared with the relaxation losses, Equation 2.21 cannot be expected to hold in materials known to have free charge carriers. For moderately conducting materials, however, the effects of pure conduction can be subtracted from the value of tan δ before Equation 2.21 is applied, so that tan δ must cover only dielectric relaxation loss and not loss caused by the conductivity of free carriers, σ_p. For highly conducting materials the use of Equation 2.21 is not advised.

Care should also be taken with anisotropic specimens (see Section 5.3.11). If the specimen is measured with the electric field in different directions at f_1 and f_2 respectively, a spurious change in ε' may be attributed to the specimen. It may be thought that this difference is caused by the change in frequency, but in fact it would have been caused by the change in the E-field orientation in the specimen. Use of the formula with ferroelectrics should also, in general, be ruled out.

Finally, note that Equation 2.21 can only be applied if the measurement uncertainties for $\Delta\varepsilon'$ are small compared with the quantity on the right hand side of Equation 2.21.

3. Standard Measurement Methods for Dielectrics

3.1 Background

Internationally agreed standard methods of measurement provide a way of sharing good practice and ensuring the compatibility of measurements that are made in different laboratories. They facilitate international trade, which requires that basic materials properties be determined in an agreed manner. That said, however, methods outlined in such standards often lag behind the state of the art in metrology. This is inevitable as test houses and product-control laboratories cannot be expected to be as up-to-date or as well equipped as calibration laboratories or those that have the task of developing new measurement methods for new types of materials.

Standard methods can be read as a guide to the art of measurement and they clearly must be followed, where possible, in many commercially-oriented measurements. But there are many areas of metrology where effective written standards do not exist. In these areas one often has to fall back on the scientific literature. We hope that this Guide can also help to fill the information gap.

International standards are developed by panels of experts and then submitted for technical approval by the standards organisations of the principal nations involved in the field. Each standard contains the date of publication (or last revision) in its title, which can act as a guide to the rate of development in the field. The gestation period for standards is often very long – many have taken over ten years to develop from the first draft to full approval, so it is not surprising that many of them have a rather ‘old-fashioned’ air about them. Many existing standards do not take account of the advantages of automation and computing, for example. This is just one reason why it is often better to develop one’s own techniques if there is no specific commercial requirement to follow the standards. But one is advised to do this whilst following the spirit of the available standards.

For the metrologist another problem that is sometimes encountered with standard methods is that they do not provide sufficient practical advice on just how measurements should be performed. Furthermore, they rarely treat the pitfalls and difficulties of measurement in any depth and are often uninformative about uncertainties. One of the main aims of this Guide is to provide the additional information required, where possible.

Standard methods are of particular importance when specifying a procedure for the determination of the properties of a material for a defined end-use. The wide range of specific applications leads to the production of a large number of standards. There are, for example, over 250 standards in the *American Society for Testing and Materials* (ASTM) catalogs with comparable numbers in the *International Electrotechnical Commission* (IEC) and *Comité Européen de Normalisation* (CEN) systems. *British Standards* (BS) are nowadays subsumed into the IEC and CEN ranges. This is an example of *harmonisation* of standards – it is obviously in no-one’s interest for international standards bodies to set up standard methods that are incompatible with each other and today all such bodies collaborate to produce consistent harmonised standards wherever possible. Note that CEN *electrical* standards are normally referred to as ‘CENELEC’ standards.

3.2 Overview of standard methods.

Clearly we cannot cover all of the hundreds of standard methods for EM materials here. We will rather mention some of the more general ones and a number that have proved useful to the compilers of this Guide. Some other specific standards are also referred to at appropriate points elsewhere in the Guide. This overview covers measurements on both liquids and solids. The LF, RF and microwave standards covered treat materials of low, medium and *high* permittivity ($100 < \varepsilon' < 10000$) and dielectrics with low, medium, and high loss.

3.2.1 Standards for Dielectric Liquids

There are two principal commercial applications for which dielectric liquids are measured:

> At low frequencies (typically 50 - 60 Hz) [48, 49] liquids are used as electrical insulation e.g. transformer oil and the impregnation or filling of high-voltage cables.
>
> At RF *Tissue Equivalent Materials* (t.e.m.s) are employed for Specific Absorption Rate (SAR) [50] measurements when determining the energy dissipated in a phantom model of the human head exposed to the radiation from a mobile phone, typically at frequencies in the band 400 MHz – 2.45 GHz [51, 52].

Standards in these two areas address very different frequency and loss regimes. At LF the cleanliness of the measurement cell is of particular importance and there is thus much to be said for demountable cells that permit thorough cleaning. In the assessment of low-loss liquids at mains frequency attention is focussed in ASTM D924 on the design of the test cell, and on methods of sampling and temperature control. For transformers and cable systems that operate at elevated temperatures the standard specifies that at 100 ^{o}C any two properly conducted determinations of the loss should not differ by more than 5% of their mean if they are to comply with the standard. It is often difficult to make precise measurements at exactly the mains frequency and thus reference is made to Appendix X3 of ASTM D150-98 [53] for the effects of frequency. Two RF methods for t.e.m. and SAR liquids are given in EN 50361-2001 [51] and IEEE Standard 1528 [52]. Three methods are outlined in the latter: slotted line, open-ended coaxial probe and TEM-line (i.e. transmission line in which TEM-mode E.M. waves propagate). The standard is mainly concerned with procedures for the determination of SAR in the human head and so the procedures for dielectric measurement are given in an appendix.

3.2.2 Measurements on Dielectric Solids

A crucial parameter for dielectric measurements on solids is specimen thickness. ASTM D374M-99 [54] outlines the methods for materials between 0.2 and 5.0 mm in thickness. Thinner materials are discussed in ASTM E252-84 [55], which considers the use of weighing as a method for thin plastic and metallic films.

A method that offers advantages at LF (typically 1 MHz) when measuring the permittivity of thin polymer films is ASTM D1531-01 [56] - the *'fluid immersion'* method, see Section 7.1.2. This method permits measurement of both the dielectric properties and film thickness by measuring the capacitance and loss of a specimen in a three-terminal electrode system using two fluids, e.g. air and silicone oil. This can either obviate the problems associated with measuring the thickness of thin compressible materials, or, provide a cross-check on the consistency of measurements by comparing the electrical thickness with that measured directly, or by weighing.

At RF two key source documents [53, 57] are ASTM D150-98 and IEC 60250, both of which are generic documents covering frequencies from 15 Hz to 300 MHz. The former is particularly of value for the range of general advice provided, formulae for the calculation of inter-electrode and edge capacitance for a range of electrode configurations and a discussion of the choice of apparatus. No mention is made, however, of the use of automated or computer-controlled equipment which is likely to be the first choice for today's metrologists.

IEC60377-1, 'Part 1: General' [58] gives an introduction to the terminology of dielectric properties and to the wave and geometrical aspects of microwave measurements. IEC 60377-2, 'Part 2: Resonance Methods' [59] covers several resonator configurations including closed cylindrical cavities and re-entrant (or hybrid) cavities, TE_{01}–mode cavities, dielectric resonators, TM_{010}–mode cavities and open-resonators, but none of these are discussed in depth and the standard has not been adapted to the use of ANAs. At 300 MHz and above IEC 60377-02 considers a range of techniques including the re-entrant cavity (see Section 7.1.4 of this Guide, useful from 100 to 1000 MHz), the coaxial cavity resonator (1 to 3 GHz) and TM_{010} and TE_{01}–mode cavity resonators for 1 to 30 GHz. The open-resonator (also called the *'confocal'* resonator, Section 7.1.17 of this Guide) is the preferred method for low-loss materials above 30 GHz. Measurements on dielectric resonators are also covered in standard IEC 61338-1-3 [60], see Section 7.1.15.

A specialised area of interest to ceramicists, dielectric measurement at high temperatures, is covered by two standards [61, 62] ASTM D2149-97 and D2520-01. The first method covers the determination of the permittivity and loss of solid ceramic dielectrics from 50 Hz to 10 MHz over a range of temperatures from -80 to 500 °C. The second method covers a much wider range of frequency (500 MHz to 50 GHz) and temperature (ambient to 1650 ^{o}C). Although these methods may be used over the specified frequency range each octave increase in frequency usually requires a smaller test waveguide or resonant cavity.

Finally, reference is made to ASTM D3380-95 [63], which permits the rapid measurement of apparent relative permittivity and loss tangent of metal-clad polymer-based circuit substrates in X-band (8 to 12.4 GHz). It is suitable for testing PTFE (polytetrafluorethylene) impregnated glass cloth and similar materials having a nominal specimen thickness of 1.6 mm, but other thicknesses are also considered.

3.3 Further Information

CEN, IEC, BS and ASTM [64] are the principal organisations that develop the standard methods that are used in Europe and the UK. Bearing in mind that there are several hundred standard methods that fall into this category, and that new standards are continually being developed, readers are advised to visit the web-sites given in the references to find standards that are directly relevant to their material, frequency and application of interest.

4. Measurement Uncertainty, Traceability and Confidence

We observed in Section 1 that measurements can only be of value if we can have confidence in their results. The concepts of *calibration*, *traceability* and *uncertainty* are central to this aim and it is the object of this chapter to explain them and to explain how they help us to attain confidence in our measurements. In doing this we will also be going some way towards explaining why the 'good practices' recommended in this Guide are 'good'. We start with a general introduction to the science of *metrology*.

4.1 An Informal Introduction to Metrology

The most important concepts and terms to understand are the following:

1. *Metrology* is the science and craft of accurate measurement.

2. *Standard*. The word 'standard' is ambiguous because it has many different meanings in the English language. Even in the context of metrology it is used to signify two completely different concepts:

> (a) A piece of equipment or a reference artefact that is used in a laboratory to allow calibrations to be performed - the word is used in this way when we refer to 'national standards' or 'travelling standards'.
>
> (b) A written document that describes an approved method or protocol for carrying out measurements.

In the French language these two functions can easily be distinguished: the equipment or artefact can be referred to as an *étalon*, while the written method is referred to as a *norme*, but English does not serve us so well so we should clarify our terminology. In this part of the Guide (Section 4) we are concerned with *physical artefact* standards, (a), while Section 3 reviews (b), the *written standard methods*.

3. *Calibration.* Calibration [65] is the comparison of a measuring instrument against a reference or a standard instrument or artefact to discover errors (see below) in the values indicated by the instrument and to allow us to compensate for them. With simple instruments, e.g. a voltmeter, this may be achieved simply by determining and applying just one correction factor. In the case of more sophisticated instruments, many correction factors may have to be determined and applied. One example is the Automatic Network Analyser (ANA - see Section 6.1.1) in which typically up to 12 complex-number correction factors can be computed and applied automatically by the software in the ANA itself. In general *all instruments that play a significant role in a measurement should be calibrated traceably (see below) to international standards*. Only if the instruments are calibrated and traceable can we be justified in having confidence in the results that they give us.

4. *Measurement Traceability and Traceable Measurements.* A calibration should in general be part of a *chain* of calibrations. For example, the measuring instrument used for the calibration should itself have been calibrated, against one or more laboratory standards. The laboratory standards themselves should have been calibrated against other, more accurate, standards, typically maintained in accredited calibration laboratories [66]. In the UK calibration laboratories are accredited by UKAS – the *United Kingdom Accreditation Service*. These standards, in turn, will have been calibrated against national standards maintained by National Measurement Institutes (NMIs, otherwise known informally as National Standards Institutes) – NPL is the NMI for the UK. The NMIs, in turn, are responsible for comparing their national standards against each other internationally to ensure compatibility. Thus, any calibrated measurement in a laboratory should be connected by a *chain* of calibrations to the standards of the international measurements community. This chain is referred to as a *traceability chain* and any measurement which lies in such a chain is said to be *traceable*. It is a basic principle of good measurement practice that *all significant measurements must be traceable*. Traceability through the chain is not necessarily of the same measured parameter, however. We may be interested in the parameter *complex permittivity,* ε^*, but microwave dielectric measurements are normally traceable to international standards via the parameters of *length*, *frequency* and *attenuation* for which national standards are maintained. At RF they are usually traceable via the parameters *capacitance* and *resistance* to national standards of those quantities. The relationship between the parameter of interest – ε^* in this case – and these other, measured, parameters is established via *measurement equations* (see the Glossary). As most measurements these days make use of computer programs to apply these equations, it is clearly important that they should do so correctly – and so *validation of software* becomes an important part of traceability in the modern laboratory – see Section 6.9. Finally, (1) it is an important principle of measurement traceability that *all traceable calibrations should be carried out to a stated measurement uncertainty*, (2) *traceability can only be established if adequate measurement records are kept.*

5. *Errors and Uncertainties.* In metrological usage both errors and uncertainties are quantities – i.e. their magnitudes can be expressed numerically - but the two terms must not be confused. An error is the difference between the measured value of a parameter and its 'true value'. In some cases, a good approximation to the 'true value' may available to us, e.g. if we measure an accurately calibrated reference specimen. Then the error in the measurement would be the difference between the value measured by the measurement system and the known value of the reference specimen. This can then be used to apply a *correction* to measurements made by the system. However there are many situations where the 'true value' is not known, for example if temperature or humidity is known to have an effect on the measurement but, because of insufficient information, the error cannot be calculated with sufficient accuracy to make reliable corrections. In this case an *estimate* should be made of the range within which we expect the 'true value' to lie, using whatever information is available to ensure that this range encompasses the 'true value' (to a stated degree of confidence). This estimated range is usually referenced to the measured value and is called the *uncertainty* of the measurement, as in e.g. '*20.2 dB ± 0.3 dB at a confidence level of 95%*, where '*± 0.3*' is the uncertainty in dB, see Figure 12. The stated level of confidence is essential for us to be able to interpret what the uncertainty statement actually means - see Section 4.3 for more details.

Typically, each measurement is subject to many different sources of error and uncertainty. It is therefore necessary to estimate an uncertainty for each source of error that we can identify, these estimates can be summarised in a table which is commonly referred to as an *Uncertainty Budget* and the *total uncertainty* of the measurement is derived by combining all of the contributions in the budget in the correct mathematical way (see Section 4.3.2). The errors and uncertainties that we have just been speaking of should therefore be seen as the *total errors* and *total uncertainties* obtained when we combine all of these contributions.

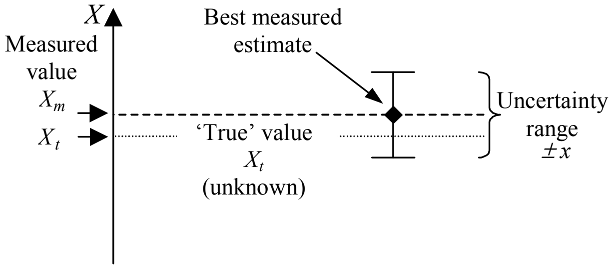


Figure 12. The interpretation of a measurement result and its uncertainty. The measured value of a parameter X is X_m and its estimated uncertainty is $\pm x$. The reported result will be '*$X_m \pm x$ at 95% confidence level'*. This statement means that we estimate that there is a 95% chance that X_m is separated from the 'true' value of X, namely X_t, by less than $|x|$ (as shown). This should be the case if the uncertainty has been assessed correctly and if there are no significant but unaccounted-for sources of error affecting the measurement.

6. *'Accurate' and 'Precise' Measurements.* These terms generally describe the comparative *quality* of a measurement and are not usually taken to be quantities, but again, they do have clearly distinct and different meanings. An *accurate* measurement is a measurement with a small *total* uncertainty. A *precise* measurement, by contrast, is a measurement taken with fine discrimination [65]. A measurement can be *precise* but not *accurate* if it is subject to a large systematic error! However the term '*precise*' is *not* a preferred term in metrology as it is too often mistakenly taken to mean '*accurate*'! One should refer instead to the '*resolution'* of a measurement which *quantifies* the smallest difference that can meaningfully be distinguished in the measurement. Regrettably, another mistake that is commonly made in measurement to take the *resolution* of a measurement to be its *total uncertainty*! In fact, the resolution of a measurement is *always* just one contribution to an uncertainty budget that tells us what the total uncertainty is.

7. *Types of Error and Uncertainty*. Some errors are caused by random processes and so their effect can be reduced by taking the average of a set of repeated measurements - uncertainties associated with them are called *random uncertainties* [65]). Other errors are caused by a *bias* or *offset* in a measuring instrument. They are called *systematic errors* and their associated uncertainties are *systematic uncertainties.* One common cause of systematic error is the use of uncalibrated measuring equipment! But even if you are using calibrated equipment for measurement you should remember that not all errors can be accounted for by applying calibration corrections, some will remain uncorrected even after the calibration. These are referred to as the *residual errors* and they will give rise to systematic errors in all measurements subsequently performed. An uncertainty contribution to account for these residual errors may be included in your uncertainty budget if it can meaningfully be estimated. Much of the business of performing accurate measurements is concerned with the

issues of how to *estimate*, *correct for* and *reduce* systematic errors. This is particularly important in RF and Microwave measurements where systematic errors usually *dominate* over random errors (i.e. they are usually significantly larger in magnitude). Further information on systematic errors in dielectric measurement is given in Section 4.3.1.

> ***A Note on Terminology:*** '*Random*' and '*Systematic*' are traditional terms which remain in common use to describe two types of uncertainty. However, in some situations where a more formal approach to estimating uncertainties is required – for example in international measurement comparisons and in calibration-laboratory accreditation - one comes across the terms '*Type A*' and '*Type B*' in reference to uncertainty evaluations [65]. 'Type A' evaluations are, by definition, carried out by statistical methods and so generally cover treatments of random and repeatability uncertainties, whereas 'Type B' evaluations are carried out by non-statistical methods, and so cover treatment of most systematic uncertainties. In this Guide we retain the traditional terminology ('*Random*' and '*Systematic*') because it is more familiar to most people.

8. Repeatability and Reproducibility. If measurements are repeatedly carried out on the same dielectric specimen throughout one measurement session, a spread of results is obtained – the *Repeatability* of the measurements is a measure of the closeness of agreement between these results. As we cannot usually be sure what the cause of the variations is (it may be due to small temperature drifts, for example, or to the specimen being placed in a slightly different position each time), it is usual to treat repeatability as a random error of measurement, so its uncertainty is computed statistically from the spread of results obtained. The *Reproducibility* of measurements on a specimen, by contrast, is the closeness of agreement of measurements obtained *under changed conditions*, e.g. on two different measurement systems or, perhaps, on the *same* equipment after it has been recalibrated and when measurements are carried out on another day. It is always good practice to estimate the repeatability of a measurement, and to include an entry for it in the uncertainty budget, as this gives one useful information about the reliability of one's equipment. Equally, it is important to report figures for the reproducibility when one has been carrying out a programme of measurements on the same specimen in different measurement systems.

9. Reference Materials. A reference material for permittivity is a material with a value of permittivity that is already known. The 'known' value should have been measured in a traceable way on a calibrated instrument. Such measurements will often have performed in a standards laboratory. Reference materials are used for three main purposes in metrology: (i) for checking the correct operation of measurement systems, (ii) for helping one to estimate measurement uncertainties, and (iii) to provide measurement traceability. In the latter case the reference material is itself used to calibrate the measurement system. The use of reference materials is common in dielectric metrology and is to be recommended for many reasons, see Section 4.4.2. Note that the 'known' value should have its own uncertainty, specified by the laboratory that performed the reference measurements.

4.2 Confidence in Measurement

4.2.1 Confidence - Why do we want it and how do we achieve it through measurement traceability?

It is the *documented traceability* of a measurement, together with good measurement practice in one's own laboratory, which allows one to have confidence in a measurement. As explained above, measurement traceability ties one in with the international measurements community. International compatibility of measurement underlies our confidence in international trade. Similarly, the world scientific community wishes to ensure that its measurements can be meaningfully compared with one another to promote scientific progress. It is presumably because we wish to be members of these trading or scientific communities that we are performing our measurements in the first place. We want these communities to have confidence in our measurements, so traceability should be important for us.

Measurement traceability and the taking of uncertainties seriously are two principles that have been widely accepted by the RF and Microwave community in general, but they have not always been so clearly recognised in electromagnetic materials metrology. The opinion is sometimes expressed that traceable calibration can be very expensive and that one can get by without it! This is one reason why it is important to know the level of uncertainty required in one's measurements – there is no point in paying a large sum of money to a calibration laboratory to have calibrations performed at a much higher level of accuracy than is needed. Traceable calibration can often be performed cost-effectively and traceably *in-house* by oneself. Before embarking on any set of measurements one should think about the level of traceable uncertainty that is required and how it can be attained cost-effectively. For example, traceability can in some cases be demonstrated very *cost*-effectively by the use of dielectric reference materials (Section 4.4.2). *Good* practice does not have to mean *expensive* practice!

Human Beings (even Metrologists) are fallible, and so mistakes and blunders are sometimes made in measurements. As a result, even documented traceable measurements can sometimes include significantly erroneous results. Such mistakes and blunders should of course be avoided by adoption of a metrological regime that promotes cross-checking of every kind, but when they do happen they are often eventually detected by comparing the measurements obtained on different systems against each other. Measurement comparisons are therefore very important (see Section 4.4.3). Only by tracing back through a *recorded* history of measurement comparisons can one identify when, and hopefully where, the errors occurred. Nothing undermines confidence in measurements so much as not knowing where the cause of an erroneous measurement lies. Keeping good laboratory records and documenting one's traceability are therefore both steps that we can use to recover from mistakes when they have been made, and, of course, they form part of good laboratory practice.

Remember that traceability includes within it the need to estimate measurement uncertainties – so we should know what good practice is in this area too.

4.2.2 How to Quantify Confidence – Confidence Levels and Coverage Factors.

We gave an example above of a statement of a measurement result that contained an uncertainty related to a level of confidence. *'20.2 dB ± 0.3 dB at a confidence level of 95%'*. This is not the place to provide a full treatise on uncertainties and uncertainty distributions - there are other guides readily available that do this (see Section 4.3) - but we do need to understand why a statement of *confidence level* (or its equivalent) is *essential* here. The quoted statement means that (blunders aside) we are confident that there is a 95% chance that the 'true value' of the measured quantity (the 'measurand') lies within the stated uncertainty range, ± 0.3 dB in this case. To put this another way – we estimate that there is a one-in-twenty (5%) chance that the correct result lies *outside* this range. A bald statement that '*the uncertainty is ± 0.3 dB.'* does not give us this information: we could be of the opinion that the chances of the correct answer lying within this range are 2%, 68% ('one standard deviation'), 95% or even 100%, or maybe we simply don't know! This is not good enough, we must give the recipients of our measurements the information they will need to be able to interpret our uncertainty statements correctly.

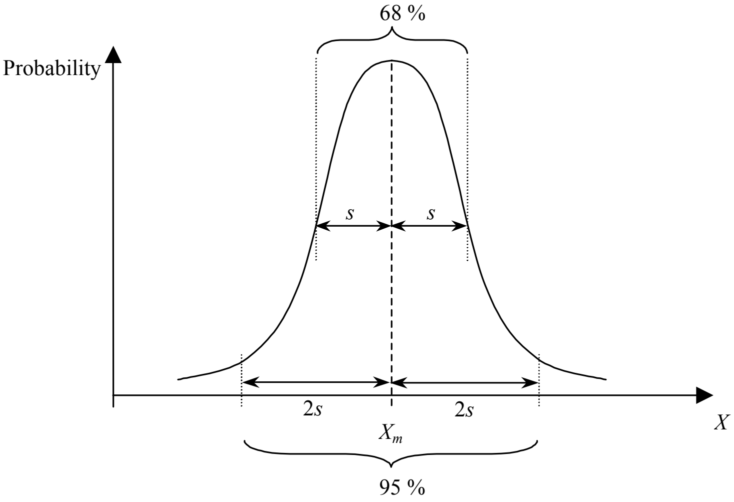


Figure 13. A Gaussian Probability Distribution with standard deviation s. X_m is the measured value of the parameter X. The distribution curve shows our estimated relative probability that X_t, the 'true' value of X, actually has any particular value close to X_m. In this case there is estimated to be a 68% chance of X_t lying within the range $\pm s$ of X_m and a 95% chance of X_t lying within the range $\pm 2s$ of X_m. (The percentages are approximate).

In estimating uncertainties we usually reach the conclusion that it is more likely that the 'true value' lies closer to our stated value (20.2 dB in this case) than further away from it. This notion is captured in the *uncertainty probability distribution*, one example of which is shown in Figure 13. The figure shows a Gaussian Probability Distribution – which commonly crops up in statistical analysis and is often (but not always) the most appropriate uncertainty distribution to apply in our measurements. The distribution width is governed by the parameter s, its *standard deviation*. If our chosen uncertainty distribution truly is Gaussian, there is a 68% chance of the correct value lying within one standard deviation, s, of the stated value (i.e. within $\pm 1 \times s$). There is also a 95% chance of its lying within two standard deviations (i.e. within $\pm 2 \times s$) and a 99.7% chance of its lying within three standard deviations (i.e. within $\pm 3 \times s$). These observations lead to another method for stating confidence levels that is commonly used by accreditation bodies [66] – namely the use of *Coverage Factors*. Again, assuming that we truly are dealing with a Gaussian Distribution, the measurement result *'20.2 dB ± 0.3 dB at a confidence level of 95%'* can equally well be stated as *'20.2 dB ± 0.3 dB for a coverage factor of k = 2'*, which means that ± 0.3 dB corresponds to two standard deviations, equivalent to 95% confidence in this case. It is common practice in the metrological community to work with 95% confidence levels (or a coverage factors of $k = 2$). Lower confidence levels give rise to a significant chance that the correct value lies outside the stated limits, whereas higher levels can give rise to very large uncertainty ranges that are often less useful. For example, we can be 100% sure that the magnitude of the reflection coefficient, $|\Gamma|$, of a passive termination on a coaxial transmission line will lie between 0 and 1.0. To say that the reflection coefficient magnitude is *'0.5 ± 0.5 at a confidence level of 100%'* is something we can do with great confidence but it doesn't tell us any more than we already knew before we carried out the measurement! 95% appears to be the level that is most useful to the RF & MW measurements community most of the time, though where measurements relate to health and safety, for example, one may wish to work with higher confidence levels.

We must be careful not to give the impression that the last paragraph covers all there is to say about the computation of confidence levels and uncertainties. Please bear in mind that: (i) some uncertainty probability distributions that we will have to deal will not be Gaussian: for example the 'U-shaped' distributions encountered in mismatch-error uncertainties [66] are very far from Gaussian. (ii) Even if our distribution *is* Gaussian, the numerical multiplying factors used above, i.e. 2 and 3 in as in $2 \times s$, $3 \times s$, above should normally be *Students-T* factors [67] – generally higher than 2.0 & 3.0, respectively, depending on the 'degree of freedom' in each measurement. Please refer to the Guides listed in the next paragraph for the recommended practice here.

4.3 Uncertainties: Their Estimation and Reduction.

To acquire a good foundational knowledge of uncertainties in measurement, readers are recommended to read another NPL Good Practice Guide, *'The Beginner's Guide to Uncertainty of Measurement'* by Stephanie Bell [65]. For practical measurements the UKAS document M3003 *'The Expression of Uncertainty and Confidence in Measurement'* [66] describes the required practices for all accredited UKAS laboratories: its practices are followed at NPL and they should meet the requirements of most scientists and engineers who need to measure dielectrics at RF & MW frequencies. The main international guide on uncertainties is the ISO *Guide to the Expression of Uncertainty in Measurement*, usually referred to as the '*GUM*' or '*The Guide*' for short [68]. '*The Beginner's Guide'* and M3003 are both written to harmonise with the GUM.

Reference to these guides should be made for the estimation, reduction and treatment of *random* uncertainties (e.g. repeatability), as the methods used are common to all fields of metrology. Similarly, general treatments on *uncertainty budgets* and the combination of uncertainties to give *total uncertainties* will be found there. However, it is the purpose of Section 4.3 of this Guide to highlight aspects of uncertainty estimation that are specific to *dielectric* measurements. Avoidance and reduction of *systematic* uncertainties, in particular, is very subject-specific so we need to look in detail at systematic uncertainties in the dielectrics field. This is done in Section 4.3.1. Worked examples on how uncertainty budgets are put together can be very helpful – one such is given in Section 4.3.2, while a useful method for calculating uncertainties is described in Section 4.3.3.

4.3.1 Systematic Uncertainties in RF & MW Dielectric Measurements

We will start with the general points and progress towards more specifically dielectric issues:

(a) As with most RF & MW measurements, systematic uncertainties in RF & MW dielectric measurements generally dominate over random uncertainties and so attempts to reduce them are important.

(b) All calibrated equipment used in measurements will have an uncertainty allocated to its measurements by the calibration laboratory. This should be included in our uncertainty budget – it is effectively a systematic uncertainty contribution which has already had its value estimated for us. If we are reduced to estimating instrumental uncertainties from instrument *manufacturers' specifications*, rather than from a calibration certificate, it probably means that our traceability is inadequate!

(c) An important approach to detecting, and perhaps reducing, systematic errors is to measure a specimen by a number of different methods. If only one method is available *in-house*, it is good practice to send a reference specimen for measurement in another laboratory from time to time – ideally a laboratory that can itself perform traceable measurements. Measurement comparisons between a number of laboratories are invariably very informative and also help one to estimate systematic uncertainties. See Section 4.4.3.

(d) One of the best ways of reducing systematic uncertainties is to rely as little as possible on the calibration of complicated measurement equipment. Use of null and substitution methods can enable some calibrations to be dispensed with completely, along with their associated uncertainties – see Section 4.4.1.

(e) Besides the systematic errors originating in measurement systems, dielectric measurements commonly suffer from systematic errors caused by the *imperfection of specimens* - and these can be dominant. For example, measurement equations typically assume that the specimen has a well-defined shape and size and that it is homogeneous. In practice the specimen may be (i) warped or twisted, (ii) of variable thickness or the wrong size, and/or (iii) inhomogeneous. Do take care to ensure that such sources of error are minimised by making the effort to prepare specimens properly, see Section 5.4. If such faults cannot be avoided (e.g. because the specimens have been delivered from elsewhere for you to measure), do take steps to estimate the uncertainties that may result from these imperfections.

(f) The following actions can help in the estimation of systematic error and in improving accuracy:

(i) Measure a number of specimens of the same material but of different thickness – see Section 6.2.

(ii) Measure specimens over a broad band of frequencies, even if you only need the result at one frequency – see Section 6.2.

(iii) Use reference materials to help you to estimate uncertainty magnitudes – see Section 4.4.2.

(g) As mentioned above, the relating of measured values (capacitances, micrometer readings) to derived quantities (permittivities) makes use of *measurement equations* which, these days, are usually incorporated into computer programmes. One should ask oneself just how closely these measurement equations actually model the physical measurement cell. If they fail to capture all the significant features of the measurement system (e.g. the effect of residual capacitances), they will give rise to systematic errors. One needs to estimate these errors and to include them in the uncertainty budget. Specific steps should be taken to *validate* such models and the software in which they are encapsulated – see Section 6.9.

(h) Commercial software packages for uncertainty analysis are available on the market and they may be able to help you. But do ask their manufacturers whether these products are properly validated and whether they conform to the spirit of the GUM [68] before using them.

Estimation of systematic uncertainty is certainly not easy. We may follow recommendation (c), above, for example, but the magnitude of the *discrepancies* between the different measurements will not immediately tell us the magnitude of the systematic uncertainty to be entered into the uncertainty budget. For example, let us say that we have measured a permittivity value of $\varepsilon' = 25.3$ for a reference material and that a calibration laboratory tells us that its permittivity is $\varepsilon' = 24.7 \pm 0.4$ at 95% confidence level. Our discrepancy of $\Delta\varepsilon' = 0.6$ does not mean that we should take our systematic uncertainty to be ± 0.6 at 95% confidence level, though it might mean that it could be roughly of that magnitude. To get a good estimate of systematic uncertainty, evidence from such comparison measurements must be put together with that from other measurements, also from our experience and from our understanding of the systems involved. We may be able to estimate a systematic uncertainty of one system, for example, by realising that it must be close to that of another that operates in a similar way.

It is usually best to estimate such uncertainties conservatively when first starting with a new measurement system. As one gains experience one may be able to reduce the uncertainties associated with it as one becomes more familiar with the magnitude of the errors that it is likely to produce. One may also be able to make practical improvements in instrumentation or in working practices to reduce uncertainty in the light of experience. Keeping records of a history of measurements on a number of reference specimens can help here. Unfortunately, accumulated experience sometimes forces one to *increase* one's estimates of uncertainty - one cannot always be right first time! It is difficult to give exact advice here, other than this: do not be tempted to *underestimate* systematic uncertainties (for whatever reason, perhaps to make our measurements appear better than they actually are?) – it usually causes trouble in the long-run and can turn out to be embarrassing!

4.3.2 Evaluation of an Uncertainty Budget – A Simple Example.

We can illustrate some of the principles of uncertainty estimation here by taking a specific example – a measurement by the *Equivalent-Thickness Method* of the real permittivity ε' of a laminar specimen in an admittance cell – see Sections 6.5 and 7.1.1 and Figure 14 for further details. Simple as it is, this example will illustrate many of the features that commonly occur when one is estimating uncertainties in dielectric measurements. The important measured parameters are:

t_s the specimen thickness
M_i the cell micrometer reading when the specimen is in the cell
M_o the cell micrometer reading when the specimen is removed from the cell
$t_x = M_i - M_o$ the displacement of the cell micrometer

where M_o and M_i are chosen to give the same total cell capacitance with and without the specimen present. Then:

$$\varepsilon' = \frac{t_s}{t_s - t_x} \qquad \ldots 4.1$$

which is our *measurement equation* for this method (see the Glossary). Let us look at the most likely sources of uncertainty in each parameter. For the sake of illustration let us say that the mean measured thickness, t_s, is 1.000 mm and that $t_x = 0.750$ mm, so that our first estimate for the permittivity of the specimen is $\varepsilon' = 4.0$.

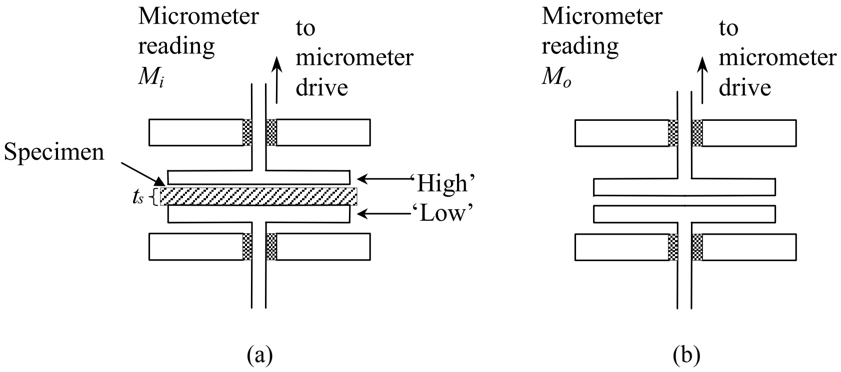


Figure 14. Measurement of a dielectric specimen in an admittance cell by the Equivalent-Thickness Method. **(a)** With the specimen in the cell and a small air gap above the specimen, the micrometer that positions the top electrode reads M_i. **(b)** The specimen has been removed from the cell and the electrode separation has been reduced to produce the same total capacitance as in (a), the micrometer now reads M_o.

Let us assume that t_s is measured by a hand-held or bench micrometer (see Section 6.3.1). The micrometer is calibrated and the stated uncertainty from the calibration laboratory for a reading of approximately 1.0 mm is ± 0.002 mm at 95% confidence level (C.L.). This is Entry 1 in our uncertainty budget (U.B.) – see Table 4.1. The *resolution* of the micrometer is ± 0.0005 mm with 100% confidence, so this is Entry 2 in the budget – it has a rectangular probability distribution, see M3003 [66]. (Note that accumulated experience may show us that some of these contributions to our U.B., such as micrometer resolution, are *always* negligible so we may validly drop them on a day-to-day basis – but we are trying to illustrate a point here).

Now the micrometer may not read the actual thickness of the specimen (see Section 6.3.1). Consider two cases.

(a) We have a *soft* or *malleable* specimen (e.g. a polymer like polyethylene) so the specimen will be compressed to some extent by the micrometer anvils: the specimen thickness is actually *larger* than that measured! We should try to estimate the extent to which this happens.

(b) We have a very *hard* specimen like a sintered ceramic. At *some* scale, the surface of the specimen will be rough. The micrometer anvils will rest on the protruding parts of the surface and so the mean thickness will be *smaller* than that measured!

Let us say we are dealing with case (a) – that we have a soft specimen and we know from experience that the likely compression of our specimen is 0.003 mm ± 0.001 at 95% C.L. The best way to deal with this is *not* to add in a systematic uncertainty of ± 0.003 – we can do better than that. Our knowledge that a compression occurs for such a specimen tells us that we have a bias in the thickness measurement that we can *correct for*. The correction to the value of t_s, in fact, is, let us say, + 0.003 mm ± 0.001 mm. The ± 0.001 mm is Entry 3 in our U.B. But we now also have a corrected estimate for t_s – namely $t_s = 1.003$ mm, and we should recalculate ε' using Equation 4.1 to give us our new best estimate for ε' as 3.96.

Now, we stated that t_s was the measured *mean* thickness. If the specimen is measured at say 8 points across its face, standard statistical techniques [67] can be used to compute the mean thickness and its standard deviation. With a good quality specimen the thickness variation across its face should not be too great. A typical soft polymer, if machined on a lathe or well-moulded, will probably be uniform to within a few tens of micrometres, but of course, the *mean* thickness can be computed more accurately than this if we take enough measurements. Let us say that our (uncorrected) measured mean value of t_s was 1.000 mm ± 0.003 mm for a coverage factor of $k = 1$, i.e. the uncertainty range is the uncertainty in the *mean* thickness for one standard deviation.

Note that we have already reached Entry 4 in our table and so far we have covered only one parameter: $\underline{t_s}$!

It might be though that, as the actual measurement involves two readings on the measurement-cell micrometer, namely M_i and M_o, we should adopt a full error analysis for both of these readings, but this is not so. The *absolute* micrometer reading may have a large *zero-error* – this may be of the order of millimetres if the micrometer is not set to zero when the electrodes of the cell just contact (see Section 6.1.5). However both M_i and M_o share this same zero-error in common. In statistical terminology the absolute zero-errors in both M_i and M_o are 100% *correlated* and, in fact, they cancel completely once the latter is subtracted from the former. All that we need to consider here is therefore the uncertainty in *differences* in readings on the cell micrometer - i.e. the uncertainty in t_x – which is considerably less than the uncertainty in absolute micrometer readings, see Section 6.1.5. Let us say that this amounts to ± 0.001mm at 95% C.L. This is Entry 5 in the U.B. Now this illustrates two very important principles in metrology – (i) operate in such a way that uncertainties cancel in measurements and (ii) don't then treat the uncertainties that *are compensated* as if they are *uncompensated* when compiling the uncertainty budget - this would give us an unnecessarily large uncertainty estimate! It may be noted in passing that this compensation of errors is precisely the reason why the equivalent-thickness method is such a good technique. We can add two more uncertainties, Entries 6 and 7, for the *setting resolution* of the micrometer when it is taking the readings of M_i and M_o. We assume the value, ± 0.0005 mm with a rectangular distribution and a 100% C.L. in each case, as we did for the micrometer used to measure specimen thickness.

We should measure t_x a few times, as we did for t_s, say 5 times in this case, and compute a mean and a standard deviation of the mean for the repeatability: say ± 0.002 for a coverage factor of $k = 1$. This gives us Entry 8 for our U.B. (Note that in this particular measurement method, we could, and perhaps should, also perform some *reproducibility* testing, by starting with different values for M_o, i.e. using different air-gap thicknesses above the specimen when it is in the cell – see Section 7.1.1. – but we will not consider this further here).

Finally we should consider Equation 4.1 and ask whether it is actually *valid* for the measurement undertaken. It will only be valid if certain conditions hold (see Section 7.1.1 for more detail). For example: it is valid only if (i) the specimen faces and electrode faces are all flat and parallel to each other; or (ii) if the electric field between the electrodes is perpendicular to these faces at all significant points in the cell and if there are no fringing-fields. In fact none of these conditions holds exactly, so we should ask just how closely the mathematical model from which Equation 4.1 is derived captures the situation that actually obtains in the cell. This is not the place to go into the technical details for this particular cell, so we shall say that long experience has taught us that the uncertainties are best expressed as an additional uncertainty in t_x which in this case (again, we suppose, from experience) is likely to be of the order ± 0.004 at 95% C.L. This is Entry 9 in the U.B. (This is, in fact, very plausible way of treating such systematic uncertainties in this particular measurement technique).

All of these uncertainties are summarised in Table 4.1, below, which is in the form used in M3003 [66]

Table 4.1 An Uncertainty Budget for the Equivalent-Thickness Method for ε' in an Admittance Cell.

	*C*1	*C*2	*C*3	*C*4	*C*5	*C*6	*C*7	*C*8
Entry	Sym-bol	Source of Uncertainty	Value (mm)	Probability Distribution	Div-isor	c_i (mm^{-1})	$u_i(\varepsilon')$	v_i
1	$u(t_s)_1$	Micrometer calib. - t_s thickness meas.	0.002	normal	2	11.67	0.012	∞
2	$u(t_s)_2$	Micrometer resolution - t_s thickness meas.	0.0005	rectang.	√3	11.67	0.003	∞
3	$u(t_s)_3$	Thickness correction to t_s	0.001	normal	2	11.67	0.006	∞
4	$u(t_s)_4$	Calculating the mean from 8 t_s thickness measurements	0.003	normal	1	11.67	0.035	7
5	$u(t_x)_1$	Cell micrometer displacement t_x	0.001	normal	2	15.73	0.008	∞
6	$u(t_x)_2$	Resolution for reading M_i	0.0005	rectang.	√3	15.73	0.005	∞
7	$u(t_x)_3$	Resolution for reading M_o	0.0005	rectang.	√3	15.73	0.005	∞
8	$u(t_x)_4$	Repeatability: calculating the mean of 5 readings of t_x	0.002	normal	1	15.73	0.032	4
9	*u(eq.)*	Error in the model of the cell expressed in Eq. 4.1 expressed as an uncert in t_x	0.004	normal	2	15.73	0.032	∞
	$u(\varepsilon')$	Computed standard uncertainty		normal $k = 1$			0.06	v >100
	$\boldsymbol{U(\varepsilon')}$	**Expanded uncertainty** (95% C.L.)		normal $k = 2$			**0.12**	v >100

The measurement result together with the uncertainty derived from this table may be stated as:

$\varepsilon' = 3.96 \pm 0.12$ at 95 % confidence level.

It must first be said that there are many details in Table 4.1 that cannot be fully explained without recourse to document M3003 [66], but the important points are as follows.

Column *C*1 allocates symbols to the uncertainty sources, as it is often convenient to refer to them in this way.
Column *C*2 describes them in accordance with the discussion in the paragraphs above, stating the measured quantities to which the uncertainty applies, in this case either t_s or t_x.
Column *C*3 is the uncertainty range as specified for t_s or t_x in the text above. In this example we are expressing our uncertainties in absolute units (millimetres). It is also possible to build up uncertainty budgets from uncertainties expressed as percentages of measured values.
Column *C*4 is the assumed shape of the uncertainty probability distribution – 'normal' being a term used for Gaussian probability distributions.
Column *C*5 is a factor that corresponds to the stated *confidence level* or *coverage factor* of the uncertainty contributions described in the paragraphs above. All of the uncertainties in the table have to be combined at the *same* confidence level or coverage factor before one can combine them to give the total uncertainty and it is usual to do this by converting them to one standard deviation (or $k = 1$ coverage factor). Thus, for entries with *Gaussian or normal probability distributions* where the uncertainty is expressed for one standard deviation, the divisor is just 1, while for 95% C.L. (or coverage factor $k = 2$) the divisor is 2. *Other* probability distributions require different divisors (see M3003 [66]). The appropriate divisor for a rectangular distribution is √3.
Column *C*6 is a column of so-called *sensitivity coefficients*, c_i. They show just how sensitive the measurement result, ε', is to changes in the measured quantities, t_s and t_x, that appear in the *Measurement Equation* 4.1. Formally the value of c_i that appears against Entries 1 to 4 in the table is the partial derivative $\left|\partial\varepsilon'/\partial t_s\right|$, while in Entries 5 to 9 it is $\left|\partial\varepsilon'/\partial t_x\right|$. Both are calculated for the measurement conditions that obtain, i.e. for the measured t_s and t_x values, using Equation 4.1. (See Section 4.3.3 on a convenient method for calculating the c_i values)
Column *C*7 shows the effect upon ε'– the quantity being measured - of each uncertainty contribution. These are the standard uncertainties in ε', i.e. they are computed for a coverage factor of $k = 1$. For each entry, Column 7 is computed from other columns in the table as follows:

$$C7 = \frac{C3 \times C6}{C5} \qquad \ldots 4.2$$

Column *C*8 is the number of *degrees of freedom* associated with each measurement uncertainty. Please consult M3003 [66] for details. However, we can say that (a) in *repeatability measurements* or in *any other measurements* where *n* measurements are taken and averaged to obtain a mean, the number of degrees of freedom is $(n - 1)$; (b) for many systematic uncertainties estimated in the light of experience, the number of degrees of freedom may be taken as ∞. This column becomes significant when one computes the *Total Expanded Uncertainty* of the measurement for ε' in the last row of the table – see below.

The significance of the last two rows of the table is as follows.

In the *penultimate row* of the table we calculate the total *Standard Uncertainty*, $u(\varepsilon')$, in ε' for a coverage factor of $k = 1$ by squaring and adding all of the entries in Column *C*7, then taking the square root. This process is sometimes referred to as 'adding the uncertainty contributions in quadrature' and is valid if they all account for mutually uncorrelated sources of error, see M3003 [66]. The resulting entry is placed in Column *C*7 because it is an uncertainty for ε'.

In the *final row* of the table we calculate the *Expanded Uncertainty*, i.e. the uncertainty, $U(\varepsilon')$, for ε' at a coverage factor of $k = 2$ or for 95% C.L. In general $U(\varepsilon') = T \times u(\varepsilon')$. Our example has been carefully chosen so that the multiplying factor, T is just 2.0 because the computed number of degrees of freedom for $u(\varepsilon')$ and $U(\varepsilon')$ in Column *C*8 is very high. However, T is actually a 'Student's T-factor' which can be significantly greater than 2.0 if the number of degrees of freedom is small [66, 67].

For example, let us assume that we carry out another measurement on a second specimen and that the measurements and uncertainties are all the same *except* that the specimen has very rough surfaces, such that the value for Entry 4, Column 3 is ± 0.010 mm. This is now the dominant contribution to the uncertainty budget (all of the other contributions become almost negligible beside it) and $u(\varepsilon')$ becomes ± 0.11, as can be seen by recalculating the table. But v, the number of degrees of freedom for $u(\varepsilon')$ is close to 7 – as calculated by using the rules in M3003 for the case where there is one dominant uncertainty. Note now that $v \approx 7 = (n - 1)$, where $n = 8$ is the number of measurements we took to obtain the mean value of specimen thickness, t_s. The Student's T-factor for 7 degrees of freedom and 95% C.L. is 2.43 – so the total *Expanded Uncertainty* in ε' at 95% C.L. for the specimen with the rough surface will be $U(\varepsilon') = 2.43 \times (\pm 0.11) = \pm 0.27$, which is quite large.

This illustrates an important point applicable to all uncertainties that can be reduced by averaging. If we average over more measurements, not only does the estimated uncertainty fall because the uncertainty of the mean becomes smaller but *the corresponding Student's-T factor falls as well*. Thus, for this particular specimen, if we double the number of thickness measurements to 16, the total uncertainty for ε', i.e. $u(\varepsilon')$, may fall to ± 0.08 mm, but the Student's T-factor for $v = (n - 1) = 15$ degrees of freedom falls to 2.18 and so the total expanded uncertainty for 95% C.L. will be $U(\varepsilon') = 2.18 \times (\pm 0.08)$. = ± 0.17 in ε', which is significantly smaller. The purpose of this paragraph is not to *explain* why this should be so (see M3003 for that) but to point out that whenever a dominant uncertainty can be reduced by averaging it is often worth the effort to do the job properly and average a larger number of measurements. If such an error is not dominant, it may not be so necessary to do so. We can see that our understanding of the basic theory of uncertainties is central to our making the right decisions here. Of course, the number of repeat measurements undertaken must also depend upon the time taken, the time available and the economic cost of our time. Our judgement must always be used in these matters

Table 4.1 also serves to illustrate another point: only three of the entries in the table, Entries 4, 8 and 9, contribute significantly to the total uncertainty. It is the process of squaring the contributions in Column *C*7 before adding them (i.e. adding them in quadrature) that renders the others virtually negligible. There may be a temptation to assume that it will always be these three entries that will dominate, but it would be a mistake to make this assumption. Dielectric materials vary so much in their physical and electromagnetic properties (see Section 5) that it is unwise to generalise in advance about which uncertainty contributions will dominate in any particular case. Each case must be investigated separately in the light of our practical experience with each material. One consequence of this is that one often does not have a ready answer to the question *'How accurate is this measurement technique?'*. In most RF & MW measurements one can give a specific reply but in the case of dielectrics one has to say '*It depends upon the properties of the material and specimen'*. One can, however, usually quote *best* uncertainties for a given technique, on the assumption that one has an ideal specimen.

This section has illustrated a number of issues that arise when uncertainty budgets are calculated. Please do refer to the other guides cited above for the rationale behind the numerical computations employed here.

4.3.3 A Shortcut for Computing Uncertainties.

Column 6 of the U.B. table above contains *sensitivity coefficients*, c_i, which show how sensitive the permittivity, ε', is to the uncertainties in the input parameters t_s or t_x. As described in the last section, these coefficients are partial differentials, such as $|\partial\varepsilon'/\partial t_s|$, derived by differentiating Equation 4.1, which is the measurement equation for this method. Now Equation 4.1 is a relatively simple measurement equation. One can easily differentiate it algebraically to derive the desired coefficients, but many measurement equations are far more complex and, indeed, many measurements these days require a computer program to derive the parameter of interest from the measured quantities: it is difficult to differentiate a computer program! Fortunately, there is a much easier way to achieve the desired aim. Computation of uncertainties generally proceeds on the basis of *first-order approximations*, so one can sacrifice mathematical sophistication and exactness for convenience by using *piecewise differentiation*.

Only two sensitivity coefficients appear in Table 4.1: the magnitudes of $\partial\varepsilon'/\partial t_s$ and $\partial\varepsilon'/\partial t_x$ We replace both of them with first-order approximations $\Delta\varepsilon'/\Delta t_s$ and $\Delta\varepsilon'/\Delta t_x$, where the Δ's are small *finite* differences and where $\Delta\varepsilon'$ is calculated by computing the measurement equation twice using slightly different values of t_s (or t_x). Thus, using the values of t_s, as corrected, and t_x from the last section:

$$\varepsilon' = 1.003\ /(1.003 - 0.0750) = \ 3.96443 \qquad \varepsilon' + \Delta\varepsilon'|_{ts} = (1.003 + \Delta\ t_s)\ /((1.003 + \Delta\ t_s) - 0.0750)$$

where $\Delta\varepsilon'|_{ts}$ signifies a small change in ε' caused by a change in t_s. Let us take Δt_s to be + 0.001 mm for the purposes of this differentiation, which is typical of the uncertainty magnitudes we are dealing with, so

$$\varepsilon' + \Delta\varepsilon'|_{ts} = 1.00\underline{4}\ /(1.00\underline{4} - 0.0750)\ =\ 3.95276 \ \text{ and }\ \Delta\varepsilon'|_{ts} =\ (\varepsilon' + \Delta\varepsilon'|_{ts}) - \varepsilon' = 3.95276 - 3.96443 = -0.01167$$

Thus $\Delta\varepsilon'/\Delta t_s$ = -0.01167/0.001 = -11.67 as shown in Table 4.1. Note that the minus sign is not needed in the table as all uncertainties are expressed as ± quantities.

Likewise, taking Δt_x to be + 0.001 mm, we have $\varepsilon' + \Delta\varepsilon'|_{tx} = 1.003\ /(1.003 - 0.075\underline{1})\ =\ 3.98016$

$\Delta\varepsilon'|_{tx} =\ \varepsilon' + \Delta\varepsilon'|_{tx} - \Delta\varepsilon' = 3.98016 - 3.96443 = 0.01573$ and $\Delta\varepsilon'/\Delta t_x = 0.01573/0.001 = 15.73$, as in the table.

Note that one needs to use at least four decimal places to preserve accuracy here – in fact one needs greater accuracy than is specified for the actual measurement result! But this is just a mathematically necessity to calculate the required gradients accurately enough. This technique can be extended to all sensitivity coefficient computations and can easily be automated on a computer.

Another computer technique that can be very informative is to use a *Monte Carlo* method [69] to compute uncertainties, as follows. Referring back to Table 4.1, Columns *C*3 and *C*4 define an input probability distribution for each entry in the table. One can use a random-number generator in a computer and a little mathematics to generate a large number (typically ~ 1000) of input values (in this case for t_s and t_x) that have been prepared to have exactly this distribution. One then sees what distribution for ε^* one ends up with. This method is useful for investigating co-variances between measured and computed parameters [69].

► ***Summary on Uncertainties.***

► ***Do's and Don'ts for Uncertainties:***

Do set up an uncertainty budget table for your measurement system.

Do consider cost-effectiveness of uncertainty analyses.

Do operate in such a way that uncertainties *cancel* in measurements. ***Don't*** then include such uncertainties in the budget as if they were uncorrelated, otherwise your uncertainty estimates may be too large!

Do, wherever possible, check measurement uncertainties by carrying out measurement comparisons and by using reference materials and specimens.

Don't forget to document all of the measurements and calibrations that provide your traceability. *There is no such thing as undocumented traceability*!

Don't waste time with unnecessarily formal mathematics when computing uncertainties – first-order approximations should suffice in virtually all cases.

Don't forget that if you are performing measurements for someone else (e.g. a customer), *they* can influence your uncertainties through the way that they prepare their specimens. ***Do*** help them to get better results by telling them about the optimum requirements for specimens in your measurement system.

Don't confuse significant property variations in inhomogeneous materials with your measurement uncertainties. Carry out enough measurements to check on specimen inhomogeneities and, if possible, quote the variation range separately from the measurement system uncertainties.

4.4 Good Practices

In this section we look at four important examples of good practice in dielectric measurements. All four aim to help us to reduce the uncertainties of our measurements and to improve our confidence in them.

4.4.1 Null and Substitution Methods

Let us assume that we wish to measure a physical quantity, say a capacitance. We can measure it directly on an impedance meter. This may be convenient, but it is often the case at RF & MW frequencies that *more accurate* measurements can be performed by using a *Null* or *Substitution* method. Such methods can be recommended if they can deliver better uncertainties than direct meter measurements. Both *Direct Substitution* and *Indirect Substitution* methods can be used. Impedance Bridges (e.g. a Wheatstone Bridge) provide a good example of the latter. Both can be used in dielectric metrology and so we will look at a few common features here.

(1) *Direct Substitution Methods* can best be explained by an example: the measurement of the capacitance, C_d, of a dielectric specimen. We can measure this directly on an impedance analyser or meter, but it may be more accurate, as illustrated in Figure 15, to use a calibrated variable capacitor, C_v, to do so, as follows. (i) Preset the capacitor C_v to a known value of capacitance, C_{v1} (NB. $C_{v1} > C_d$), and 'measure' its capacitance C_m on the meter. (ii) Place C_d in parallel with C_v and readjust C_v so that the meter again reads C_m. Read the re-adjusted value of the variable capacitor as C_{v2}. Then we have $C_d = (C_{v1} - C_{v2})$. This null method is worth using if the impedance analyser has *high resolution* but *poor accuracy,* see Paragraph 6 in Section 4.1. In fact, with this technique, the impedance analyser can remain *completely uncalibrated* as long as it is stable and as long as the variable capacitor *is* calibrated. In fact, the meter does not even have to be a recognised impedance meter - any meter which changes its reading whenever the capacitance is changed should suffice - provided it is stable enough and has adequate resolution. Note that such a method can be classed as a *null* technique because one adjusts C_v to give a null change in the reading C_m. In this particular example the substitution method may well be more accurate than trusting the impedance analyser at RF & MW frequencies. If there are long coaxial leads between the capacitors and the impedance analyser they can introduce impedance residuals which give rise to measurement errors (see Section 2.2.1, Equation 2.8), but C_v and C_d can potentially be placed physically very close together.

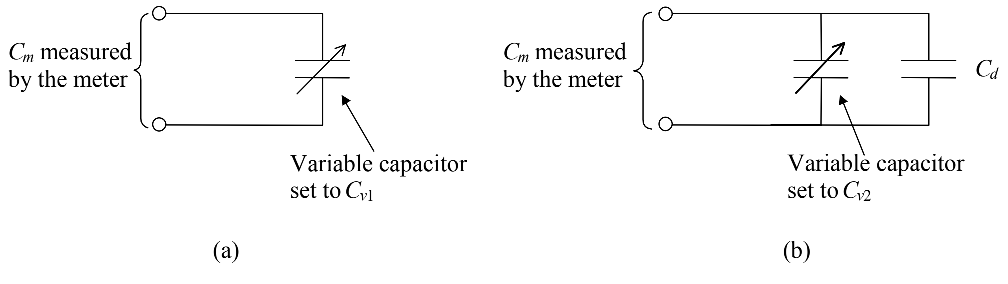


Figure 15. A simple substitution measurement. The 'unknown' capacitance C_d is measured against a calibrated variable reference capacitor. In **(a)** the capacitance measured on the meter is C_m when the reference capacitor is set to C_{v1}. In **(b)** the 'unknown' capacitance C_d is placed in parallel with the reference capacitor, which then has to be reset to C_{v2} so that the meter again reads C_m. Clearly $C_d = C_{v1} - C_{v2}$. This measurement can be carried out on an uncalibrated but stable meter because there is no change in the capacitance it measures.

(2) *Indirect Substitution Methods* including bridge techniques can be somewhat more complicated, but nevertheless are well worth using if they deliver better accuracy than the available alternatives. A good example of this is the 'equivalent-thickness' approach to permittivity measurement described in Sections 6.5, 4.3.2 and 7.1.1. In this method we substitute an air dielectric capacitor of thickness t_a for a dielectric specimen of thickness t_s in such a way that both produce the same capacitance when measured. The permittivity, ε' can be computed from t_a and t_s, but it is determined *indirectly* via dimensional measurements

Null and substitution techniques are generally more complicated to use than direct methods and are often more costly both in time and the resources needed. So, clearly, in order to know *when* it is worth our while to turn to such a method, rather than use a convenient direct method, we must have a good grasp of the uncertainties of both techniques and also of the uncertainty we actually *need*. Once again, this illustrates that knowledge and understanding of uncertainties is part of good measurement practice.

4.4.2 The Use of Dielectric Reference Materials

How can we check that our dielectric measurement instrumentation is functioning correctly? How can we demonstrate that our measurements are *traceable* to international measurement standards? Given the very wide range of techniques that are used to measure dielectrics, see Section 7.1, there can no single answer to these questions. However, use of *Dielectric Reference Materials* can go a long way towards boosting our confidence that our measurements are sound. Such materials can also act as traceable calibration standards in many cases.

Dielectric reference materials should ideally meet the following criteria:

(a) They should be well-defined both in their physical form, their chemical composition and their impurity content. It follows that they must be homogeneous and of uniform density. These criteria are essential for the reproducibility of the properties of standard materials, and they effectively exclude very many materials, including most solids, from acting as reproducible dielectric reference materials at all!

(b) Their properties should have been measured accurately in a way that guarantees traceability to international measurement standards. Measurement uncertainties should have been estimated and supplied in accordance with international practices [68]. They should ideally have been measured in a number of reputable standards laboratories, whose measurements should agree to within their estimated uncertainties.

(c) For maximum convenience, they should have been characterised over a wide range of temperature and frequency.

(d) They should be readily machinable or deformable into the specimen shapes demanded by the many different measurement systems that are used for dielectric measurements.

(e) They should be readily available and reasonably cheap to obtain.

(f) They should be easy to use and difficult to contaminate.

(g) They should be completely safe to use and have a low level of toxicity and flammability.

In fact, none of the reference materials currently available for RF & MW dielectric measurements meet all of these criteria. Virtually all materials that meet criterion (a) are liquids and none of these liquids meet criterion (f). The second part of criterion (b) - that the materials should have been shown to have *consistent* measured properties across a number of laboratories - is arguably only met by *deionised water* [70]. Organic liquids can be very useful reference dielectrics but many do not meet criterion (g). These limitations do not make reference materials impossible to use, but one must be fully aware of the problems presented by each material before it is used, otherwise one runs the risk of compromising accuracy rather than improving it!

Full recommendations on the use of dielectric reference materials are made in other NPL publications [37, 71]. They may be summarised as follows:

<u>Solid Dielectric Reference Materials</u> suffer from the problem that one cannot guarantee that one batch of a material will have the same dielectric properties as another, even if they are obtained from the same manufacturer. One cannot therefore usefully *publish* the properties of such materials so that they can be available to all workers as a reference. There are a few exceptions, all of which are pure *crystal* materials - quartz, YAG (yttrium aluminium garnet) and sapphire, for example - that are sufficiently reproducible to act as reference materials for real permittivity, ε', but they generally exhibit far too low a loss to act as useful references for loss in most cases.

A workable practice for solids is rather to maintain a single batch of (stable, homogeneous, etc.) reference specimens that have been calibrated traceably in a reference laboratory and to use them to check or calibrate one's own measurements. Ideally one should try to employ specimens with dielectric properties close to those of the materials to be measured. It will not always prove possible to use the very same specimens that the reference laboratory has used – it may employ a measurement system that requires specimens of a different shape from one's own measuring system, for example. In that case, a number of specimens of both required shapes should be machined from a single batch of homogenous material, ideally from *contiguous* portions of the same sample of material. The reference laboratory should measure a number of appropriate specimens to demonstrate consistency of properties, and then one can use the other specimens to check one's own measurements.

Liquid Dielectric Reference Materials are very widely used and, if uncontaminated, can more easily meet criterion (a) above, though generally not (f) and, sometimes, not (g) either. *High-loss* dielectric liquids, such as deionised water and n-alcohols, are readily commercially available in sufficiently pure grades to be regarded as reproducible and their cost is not excessive. They are widely used both as check and calibration standards in accurate and traceable measurements but it is nevertheless all too easy to misuse them. *Low-loss* reference liquids (see [72] for an international comparison on such liquids) are best used only as a check on *real* permittivity measurements as they are generally too easily contaminated to reproduce their loss reliably under all but the most stringent measurement conditions. For high-loss measurements, by contrast, NPL has carried out a measurement programme to characterize a range of reference liquids (mostly n-alcohols) across the RF and low microwave region up to 5 GHz [37]. **A warning:** There are, as yet, no internationally agreed values for the RF & MW complex permittivities of reference liquids and no formal international measurement comparisons on high-loss reference liquid have been carried out to boost confidence in the published NPL data. However, the NPL measurements are traceable to international standards and they are compatible with good quality data published elsewhere. The published complex permittivities of these liquids [37] are used at NPL for calibrating and checking high-loss dielectric measurements.

Practical Use of Dielectric Reference Materials Good practice guidelines for storage and handling of dielectric reference materials are exactly the same as those for other materials to be measured – as described in Section 5.4.2. However, in the case of solid reference specimens, special care ought to be taken in handling and storing them as they may be needed for many years to come. Special care ought also to be taken not to contaminate reference liquids, see Section 5.3.3. Used reference liquids should, in general, not be re-used as they may have been contaminated during measurement.

4.4.3 Measurement Comparisons

As highlighted at a number of points in this Guide (e.g. Sections 4.2.1, 4.3.1) measurement comparisons form a major weapon in our arsenal for identifying and quantifying systematic errors and uncertainties. They are also invaluable for detecting mistakes in our laboratory practices. Comparisons should ideally be carried out by independent measurement methods on the *same* specimens, see e.g. [73]. If that is not possible (e.g. because two methods require differently shaped specimens) the specimens in the comparison should at least be taken from the same batch of material as described in 4.4.2. Opportunities for undertaking such comparisons should be welcomed for the metrological benefits that they can deliver. Measurement comparisons are regarded as very important internationally by the National Measurement Institutes (NMIs), like NPL, that are responsible for maintaining National Standards of measurement. These laboratories participate in a regular programme of comparisons on all basic quantities, e.g. length, mass, current, microwave power and impedance, to ensure that measurements are consistent across the world. NMIs also participate in international comparisons on derived units like dielectric permittivity [74, 72, 75].

4.4.4 Check-Lists, Written Procedures, Measurement Records, etc.

General good laboratory practices should, of course, always be followed in dielectric measurements. These include the maintenance of *written measurement procedures* and *protocols* and the keeping of *adequate records*.

It is all too easy for experienced metrologists to work without written procedures, but when the time comes to pass working practices on to others, important points sometimes get forgotten. Make sure that all important steps in a measurement protocol are always taken into account by maintaining (i) an uncertainty check-list, (ii) written procedures and check-lists for the practical actions to be undertaken in the measurements, (iii) measurement records that are available to others – most importantly, others must know how to access and interpret your records. Whenever something metrologically notable happens in a measurement, record the fact. Others may benefit at a later date from your experience of it.

All such procedures help to promote a better understanding of uncertainties and also help us to reduce our uncertainties and achieve confidence in our measurements.

SECTION B: PRACTICAL ADVICE

5. An Introduction to Practical Dielectric Measurements

As stated in the Introduction to this Guide, Chapters 5 to 8 cover practical aspects of the characterisation of dielectric materials at RF and Microwave frequencies. This chapter provides an introduction, a survey of material types and an overview of the best approaches to characterising different types of materials.

5.1 Options: To Measure or not to Measure?

We can clearly save ourselves time and money by *not* measuring the properties of materials if we can possibly avoid it. We can, for example, pursue the following options:

(1) Consult manufacturers' data sheets - *but how comprehensive are they and how trustworthy is the data?*

(2) We can consult tabulated and published data in the scientific literature. For example, NPL, through its *Electromagnetic Materials Measurements and Applications Club* (EMMA-Club) [76] has published Technical Notes (e.g. [43, 70]) and has been setting up a database [71] to point us towards tabulated data on RF & MW dielectric properties of materials - *but can the original data be trusted? What are the uncertainties in the tabulated data and how can we be sure that the data applies to the particular dielectric material we wish to characterise?*

Should we rely on published data? The answer usually depends upon the stage we have reached in the work we are doing. If we are developing an RF & MW application – perhaps a radar system, an antenna, a telecoms receiver or a measurement system - and we need to select a dielectric material for use in that system, then it is probably best to consider two different stages in our development work: (a) the *design* stage (b) the *end-use* application.

(a) *The Design Stage.* In system design it will often be sufficient and generally more cost-effective to consult manufacturers' data or a dielectrics database if they are available. It is largely with design applications in mind that such databases are assembled. But we can encounter a host of problems here. Many data sheets present data only over a limited range of frequencies and temperatures, and typically not at microwave frequencies! If a suitable data sheet *can* be obtained, it should at least apply to the material obtained from that manufacturer, but if instead we obtain data from a general database, can we be sure that it applies to the grade or variety of the material that we wish to use? For example, the dielectric properties of all ceramics depend critically upon how they are prepared and sintered, so there is no such thing as a 'standard' or 'representative' batch of sintered alumina. The best approach is to gain some background knowledge about the type of ceramic we are using - its method of preparation and its purity - and then to seek data on a similar material in the database. A similar approach should be used in the case of polymers. A material like 'PVC' is far from being a uniquely defined material – polyvinyl chloride is prepared in many different ways with different amounts of plasticiser and other additives admixed with it. Both the additives and the processing of the polymer give rise to a whole range of different 'PVC' polymers with varying dielectric properties, so the uncritical adoption of data on 'PVC' from a database will not do. Again we should be prepared: learn beforehand about the grade of material to be used and look for the best match to that in the database. We will sometimes have to conclude that the available tabulated data is not adequate for our purposes.

(b) *End-Use Application of a Material.* When developing engineering and electronic systems it is often the case that we need to know the properties of our materials more accurately for their end-use application than for the initial design stages of system development. At this final stage is usually prudent to measure the properties of the dielectric materials to be sure that they are adequate for the intended application. Of course there are exceptions: we may have used the materials before and have practical experience of them, or else we know someone else who has used them, or we may conclude that for some suitably well-defined materials the information obtained from a database is adequate. However, the two main questions to be asked here are: (i) how accurately do we need to know the properties of the materials, (ii) what is the uncertainty of the data we can obtain on the material: either from the manufacturer, from a database or by measuring it? Our next steps depend upon our answers to these two questions.

Note that certain well-defined classes of materials, like polyethylene and certain grades of alumina, exhibit a quantifiable relationship between their density and their permittivity. So in some cases, a density measurement may be all that is needed [77] to characterise the permittivity of the material - it will almost certainly be less costly than a dielectric measurement. See Section 6.8.

If, now, we decide that the material must be measured, we have two further options:

(3) We can request a calibration laboratory or test house to carry out the measurements on our behalf - *but will this be more cost-effective than carrying out the measurements ourselves?*

(4) We can carry out the measurements ourselves.

The choice between (3) and (4) will largely be based on (i) cost-effectiveness, (ii) time taken for the measurements, (iii) whether or not there are other suitable laboratories available.

Usually, if we have a ‘one-off’ measurement job, unless we already have a suitable measurement system available to us, it will be better to go for option (3), assuming that we can find a suitably accredited or experienced laboratory to perform the measurements. Calibration and test laboratories should already be aware of the pitfalls and uncertainties that arise in dielectric measurements and if we start from scratch, we have to spend time, through trial-and-error, to learn about them the hard way. If we have never carried out a particular type of measurement before we can often underestimate the difficulties. Experience shows that this is particularly the case with RF & MW dielectric measurements.

If we have a long-term interest in measuring or testing dielectric materials we may well decide to set up a measurement system ourselves, but even in this case it may be more cost-effective to seek advice from an experienced laboratory at first. The cost savings that can be achieved by learning from experience in measurement should never be underestimated. In fact, one of the best pieces of general advice that can be given to help us to choose between all of the above options is to *seek out experienced advice*. One of the best ways of doing this is to join a measurement club, like the EMMA-Club mentioned above [76], whose members join the club precisely to help each other in this way. Clubs can also help to inform us about which manufacturers supply relevant equipment. See Section 11 for details of other clubs and societies.

► ***Do's and Don'ts: To Measure or not to Measure:***

Do be aware that a common material name like ‘PVC’ can cover a whole range of different materials with very different dielectric properties (particularly loss tangents).

Do make sure that you know the grade of material you are using so that you can access the appropriate data in databases.

Do be aware of the level of uncertainty to which you need to know the properties of your dielectric materials.

Do try to assess the accuracy of published data to decide whether it is adequate for your needs.

Do join a measurement club if you have a long term-interest in measurements so that you can learn from more experienced people.

Do seek the advice of experts if you decide to carry out measurements yourself by techniques you are not familiar with.

Do consider whether you may be able to get the information you need by performing a density measurement, rather than a permittivity measurement (Section 6.8).

Do ask yourself and experts whether the measurements you wish to perform can be carried out on commercially available equipment or whether you should construct your own measurement system.

Don't underestimate the difficulties of performing measurements yourself by a technique that is new to you.

5.2 Varieties of Dielectrics

Dielectric materials come in many different forms, physical phases, shapes and sizes. The wide range of RF & MW dielectric measurement techniques that have been developed over the years reflects not only the need to measure at different frequencies but also the wide range of physical and electrical properties that the dielectric materials themselves exhibit. Consider the following (incomplete) list:

High-loss solids (e.g. RAM, radiation absorbing material)
Low-loss solids (e,g. quartz and other pure crystals, ceramic dielectric resonator materials)
Hard solids (e.g. crystals, ceramics, some polymers, building materials)
Malleable solids (e.g. biological tissues, aerated polymer foams)
High-loss liquids (e.g. ethyl alcohol, polar liquids in general)
Low-loss liquids (e.g. cyclohexane, non-polar liquids in general)
Viscous liquids (e.g. some foodstuffs, some organics, some SAR* calibration fluids)
Non-viscous liquids (e.g. water, some organics)
Toxic materials (e.g. some liquid organics and solid inorganics like beryllia)
Magnetic solids (e.g. ferrites)
Thin films (e.g. windows, coatings, ferroelectrics)
Dielectric resonators (typically sintered ceramics in 'puck'-form).
Substrates (e.g. FR4, alumina, LTCC – low temperature co-fired ceramic)
Materials available only in small quantities (e.g. expensive crystals, trial samples of ceramic)
Materials available in copious quantities (e.g. radome materials, some substrates).
Composite materials (e.g. carbon-loaded materials, some substrates)
Anisotropic materials (e.g. some crystals, polymers with frozen-in stress, fibrous materials, tissues)
Structured materials (e.g. layered materials or materials with a macroscopic lattice structure)
Significantly inhomogeneous materials (e.g. many composites, foodstuffs, human tissues, etc.)
Moist and aqueous materials (foodstuffs, biological tissues, building materials – how moist are they?)
Sintered materials (especially ceramics, but also, e.g. PTFE)
Powders (e.g. cement, pharmaceuticals - are they dry or damp?)
Significantly porous materials (e.g. some building materials, some RAM & ceramics))

(* SAR is the Specific Absorption Rate of RF signal power into biological tissues or other dielectrics, measured in watts per kilogram [50])

It would not be too much of an exaggeration to say that each of these different classes of dielectric requires a different measurement technique. It is true that some techniques can be pressed into service for quite a number of these classes, but it is certainly the case that to measure each class of material *optimally*, e.g with least uncertainty or least measurement time or cost, we would need to use different techniques and measurement equipment. In an ideal world, the measurement method would be chosen, or at least adapted, to match the dielectric to be tested, rather than the other way around! Sheer economics, however, usually force us to carry out measurements on non-ideal equipment, see Section 7.2.

There are too many degrees of freedom here to treat all of these material types in great depth. However, in Section 5.3 we look into special considerations for some of these classes and in Section 7 we survey the available measurement techniques and also give general guidance on which of these classes of material the various techniques are best suited to. It occasionally happens that we are supplied with an unknown material to measure and that we have no idea at all of what its permittivity is, so we need to make a quick preliminary check to allow us to choose the optimum technique. See Section 7.2 also for advice on how this might be done.

5.3 Matching Measurements to Materials

Sub-sections 5.3.1 - 5.3.13 describe special considerations for a wide range of material types. The nature of these considerations ranges as widely as the nature of the materials. One point to remember when choosing suitable measurement methods is that they vary enormously in the measurement uncertainties that they can deliver – so the selected method should be chosen not just on the basis of the *type of material* to be measured but on the basis of *required uncertainties* too. See Section 7.1 for the levels of uncertainty to be expected from each method.

5.3.1 Medium to High Loss Materials.

If the material to be measured is homogeneous, both medium and high-loss materials can usually be measured in the form of small specimens, even as small as a few millimetres on a side, but better accuracy is often obtained if larger specimens are used. At RF, below 1 GHz, the use of admittance bridges and admittance cells is effective. At microwave frequencies, where it is appropriate to think in terms of travelling waves (see Section 2.2.2), the use of *single-pass* or *double-pass techniques* is appropriate.

Consideration of the Kramers-Kronig relations (Section 2.5 [44, 25]) or of Lynch's Formula (Equation 2.21 [45]) shows that the dielectric properties of lossy dielectrics can vary quite rapidly with frequency. Fortunately travelling-wave techniques are well suited to the broad-band measurements that are required for characterising lossy dielectrics. Such measurements are readily performed at microwave frequencies using ANAs (see Section 6.1.1) while admittance measurements on impedance analysers and FRAs (Sections 6.1.2 and 6.1.3) are often more suitable at LF and RF. These methods are discussed in Sections 7.1.1 - 3 and 7.1.10 - 13.

The above methods do not exhaust those available for medium to high-loss materials. A particular class of resonance methods - *perturbation methods* - should also be mentioned. They are especially useful when only a small amount of material is available – the specimen can be used to perturb the resonant frequency and Q-factor of a resonator and ε^* is computed from the change in these parameters when the specimen is inserted into the resonator. Such methods are covered in detail in Section 7.1.16 and also in 7.1.6. Conversely, specimens available in large sheet form can be measured by free-field methods, Section 7.1.14.

5.3.2 Low-Loss Materials.

The real part of the permittivity, ε', can often be measured by the same techniques discussed above in Section 5.3.1 for higher loss materials, but those techniques are often too insensitive for measuring the losses of low-loss materials. For measurements above 1 MHz, one is often best advised to turn to a resonance or 'multi-pass' technique (Section 2.2.3), where, notionally, the multiple passage of the signal through the specimen increases the sensitivity of the measurement to the loss in the dielectric. The loss is then determined by the measurement of Q-factor (Section 6.4). Resonators can be constructed for specimens of virtually any size, but loss measurements are usually more sensitive if larger specimens are used. Thus, the general advice is to use resonator techniques for low losses, but there are some exceptions, e.g. some Frequency Response Analysers (Section 6.1.3) can measure loss tangents as low as 10^{-4} over a broad frequency range at LF and RF.

Resonators come in many different forms (see Sections 7.1.4 - 9 and 7.1.15 - 17) but each is generally restricted to a very limited band of frequencies, sometimes to just one frequency. So to measure the loss of a low-loss material over a broad bandwidth one may need to use a number of resonators. Fortunately, the Kramers-Kronig relations (Section 2.5 [44, 25]) and Lynch's Formula (Equation 2.21 [45]) show that the dielectric properties of low-loss dielectrics vary very slowly with frequency. Therefore, one may need only measure the loss once per octave of frequency to characterise a dielectric adequately, e.g. use a logarithmic scale of frequencies, such as 1, 2, 4, 8, 16 …GHz. The same considerations show that real permittivity, ε', for a low-loss material changes even more slowly with frequency than the loss tangent. For a homogeneous polymer like polyethylene, for example, with $\varepsilon' \approx 2.3$ and a loss angle of less than 300 μradians in the RF & MW band, the value of ε' should fall by less than 0.006 across the whole spectrum from 1 MHz to 100 GHz.

One can take advantage of this fact to reduce effort in measurements. For example, one can measure ε' at just one frequency, ideally a frequency at which it can be measured accurately and/or cost-effectively, and then use resonators across the required band to measure $\tan\delta$ only. In the RF range 100 MHz - 800 MHz, re-entrant cavities (Section 7.1.5) can measure low loss adequately but (without employing numerical analysis, see Section 6.9) such resonators cannot generally measure ε' very accurately (± 2% uncertainty or worse at 95% C.L. is typical). But one does not need to rely upon that determination of ε' if one has measured ε' more accurately at a lower frequency, e.g. in a three-terminal admittance cell (Section 7.1.1). Alternatively one can measure ε' at a higher microwave frequency in a cavity or open-resonator (Sections 7.1.7, 7.1.8 and 7.1.17). In such measurements the uncertainty for ε' may be as low as ± 0.5%, or better, at 95% confidence level.

In general, *one should not touch low-loss specimens with one's fingers*. Use clean polymer gloves or tweezers for this. Specimen contamination can significantly increase the apparent loss of a specimen, and many materials are easily contaminated by human sweat. Take steps to avoid *all* forms of specimen contamination.

5.3.3 Liquids

Measurements on dielectric liquids, in contrast to solids, have their advantages and disadvantages.

Advantages:

(A1): The real permittivity, ε', of a dielectric is effectively defined as the ratio of capacitance between two conducting electrodes when the space between them is *completely* filled with the dielectric, to the capacitance obtained when it is *completely* filled by a vacuum. (This definition is readily generalised to complex permittivity – see the Glossary). Liquids have the property that they can readily fill the complete volume of a measurement cell, whereas solid specimens often leave unfilled air-gaps between themselves and the electrodes. There are also 'fringing-fields' around the solid specimens which pass through air, especially in two-terminal admittance measurements (see Section 7.1.3, and Figure 28 (b)). The presence of significant electric fields in the air-dielectric surrounding a solid dielectric specimen can give rise to significant errors of measurement. Liquid measurements need not suffer from such errors. For solids the errors can be reduced by applying corrections, as in the methods described in Sections 7.1.3, 7.1.4, 7.1.10 and 7.1.11, or else by explicit field modelling. They can also be mitigated by applying the liquid-immersion techniques for solids described in Section 7.1.2, but none of these approaches detracts from the fact that dielectric measurements on liquids can often be more accurate than those on solids when they are measured by similar techniques. Now at LF or at RF one often determines ε' by measurement of inter-electrode capacitances as described in Sections 7.1.1 – 4. These arguments clearly apply to such methods, but, by extension, they also apply to other measurement methods across the spectrum and to measurements of loss as well: if the liquid can be made to fill the measurement cell such that there are no liquid/air boundaries at points where significant e.m. fields are present, the *measurement equations* (see Glossary) for the cell can be simpler and they will describe the measurement geometry more exactly than for solid specimens filling only part of a cell.

(A2) As discussed in Section 6.2, paragraph (c), systematic errors of measurement on a material can often be reduced by a set of measurements on specimens of different thickness or size. This is readily achieved with liquids, simply by changing the distance between electrodes or the length of a cell or resonator. In the case of solid dielectrics, a whole set of specimens may have to be machined or moulded to achieve this, which can be costly and time-consuming. Once again, better accuracy can be obtained more readily with liquid dielectrics.

(A3) Liquids can be more easily temperature-controlled *if* they fill the cell. Solid specimens may be somewhat thermally isolated in their cells, e.g. dielectric resonators in a cavity cell, see Section 7.1.15.

Disadvantages:

(D1) To counter (A1), we should note that liquids always require a container and it may prove necessary for this to be placed *inside* the measurement cell. Thus in transmission line measurements, Sections 7.1.10 – 11, the liquids must be contained between *windows* which will affect the measurement significantly and will often increase the measurement uncertainties. However, this requirement can be turned into a benefit – the windows can be used to *match* the travelling waves into the liquid and perhaps *improve* the measurement uncertainty (Section 7.1.10). This possibility can be implemented by numerical modelling prior to one's designing of the measurement cell. (Note also, in this particular case, that the liquid fills the whole cross-section of the cell, making it unnecessary to make corrections for air-gaps around liquid specimens that are always present around solid specimens, see Figure 37, another advantage for liquids).

(D2) In unsealed cells, liquids, especially volatile organic liquids, will evaporate and thereby change their temperature and this will change their dielectric properties

(D3) Liquids are more easily contaminated than solids, e.g. by moisture, vapours and gases in the air, and by contaminants in the measurement cell. For this reason too one should generally reseal a source bottle of liquid as soon as the liquid has been delivered from it.

(D4) Liquid mixtures may separate out into their components if they are not stirred before measurement.

(D5) Toxic liquids can affect or contaminate the experimenter, especially if they are volatile.

(D6) To avoid (D2) - (D5) one usually has to set up a sealed apparatus for introducing liquids into measurement cells, whilst avoiding contamination in either direction. This can make liquid measurements more costly and difficult than measurements on solid dielectrics.

(D7) In spite of attempts to avoid contamination of the liquid, it is often necessary to dispose of a liquid after it has been measured because gradual contamination is unavoidable (this is always recommended for dielectric *reference* liquids, for example, See Section 4.4.2), so the cost of the measurement may mount because an adequate quantity of liquid has to be purchased and/or manufactured, whereas solid specimens can often be re-used many times (if they too are protected from contamination, see Section 5.4).

(D8) Some liquids are flammable! Especially volatile organics!

(D9) Some liquids may be corrosive or may be absorbed into dielectric components in the measurement system, e.g. into flexible plastic tubing, from which they can *leach out* contaminants.

(D10) When measuring *conducting* liquid dielectrics at LF and low RF one can encounter the *Electrode Polarisation Effect*, which can easily give rise to erroneous values for ε' and ε'', see the next paragraph.

The Electrode Polarisation Effect. This is predominantly an LF effect. In a conducting liquid the ions and electrons that carry the electrical current meet a potential barrier when they reach the electrodes of a measurement cell and they find that they cannot pass into the electrode [78]. Consequently they can 'pile up' there and in admittance cells this gives rise to a spurious *series admittance* which can easily dominate over and overwhelm the admittance of the liquid being measured. An equivalent circuit is shown in Figure 16. The magnitude of the effect is very difficult to predict and so in dielectric measurements the effect is best avoided. Four approaches for avoiding it may be mentioned (see also Section 7.1.19 for time domain measurements):

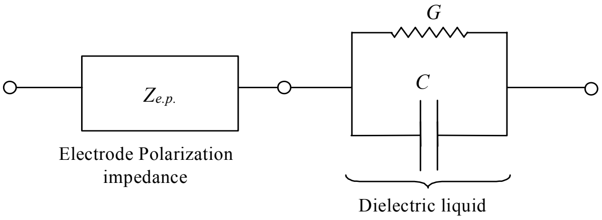


Figure 16. An equivalent circuit showing the effect of electrode polarisation in LF measurements upon conducting liquids. The parallel G/C combination on the right represents the dielectric liquid as in Figure 2(b). $Z_{e.p.}$ is the series impedance introduced by the polarisation effect at the electrode/liquid interfaces. Its presence can give rise to significant measurement errors at LF.

1. Use of an electrode material in which the polarisation effect is small. This amounts to using an electrode with a large *microscopic* surface area (i.e. a *rough* surface on the micro-scale). Application of platinum black to electrodes is a traditional method for reducing the effect, otherwise one can purchase an electrode material, such as platinum-iridium, specifically manufactured to have such properties.

2. The second method is simply to work at higher frequencies since the polarisation effect falls off roughly as the square of the frequency. When measuring the *static permittivity* (Section 2.3) of conducting alcohols such as methanol at NPL it was found that, while the polarisation effect dominates at frequencies below 1 kHz, it became almost negligible at 10 kHz in the particular admittance cell that was used [79, 37].

3. A third method which works well only at LF is to use the full benefits of the *four-terminal measurement system* [80] offered by impedance analysers (Section 6.1.2.). The current electrodes are placed at either end of the liquid cell and the polarisation effect occurs at those electrodes, while the voltage electrodes are interposed in the cell *in between* the two current electrodes and, because insignificant current is drawn through the voltage electrodes, errors from charge build-up there are simply avoided. The method becomes less effective as the frequency rises to RF because of *residuals* in the cell, see Section 2.2.1.

4. The fourth approach dispenses with electrodes altogether! Currents, specifically eddy currents can be induced in conducting liquids by inductance coils and if two such coils are used instead of electrodes the electrode polarisation effect simply does not occur [81]!

Safety Note: In general, it cannot be emphasised too greatly that one should take extra safety care when dealing with liquids. Always consult COSHH [82] guidelines on liquids that you intend to use or measure.

► ***Do's and Don'ts for Liquid Dielectrics:***

Do take special care to avoid contamination in either direction - *into* the liquid and *by* the liquid.

Do re-stopper bottles of liquids immediately after use to prevent atmospheric contamination.

Do check that you are measuring the liquid at the right temperature if it could be evaporating - measure the temperature of the *liquid*, not the *cell* in which it is placed - they may be different!

Do consult COSHH [82] (Health and Safety) data sheets on the liquids to be measured so that you can take the correct safety steps to handle them appropriately. If someone else supplies you with an unknown liquid to measure, you should *refuse* to handle it until they have also supplied the appropriate COSHH data for it.

Do consult COSHH guidelines and other chemical data resources to decide whether a liquid to be tested could affect or corrode your measurement apparatus.

Do dispose of liquids safely in accordance with COSHH guidelines.

Do consult safety experts on the handling of chemicals.

Do warn the customers for your measurements that it may be necessary to dispose of their liquids after measurement if they are liable to contamination.

Don't pour liquids that may have been contaminated during measurement back into their source bottle or reservoir; you may thereby contaminate perfectly good uncontaminated liquid stock!

Don't underestimate the ease with which solvents can absorb contaminants: e.g. moisture from the atmosphere, contaminants from human contact (e.g. sweat and salts) and contaminants from surface films in measurement cells which have not been adequately cleaned. Low-loss liquids can easily increase their loss by orders of magnitude through these processes. Hygroscopic liquids like alcohols and alcohol mixtures can change their composition over periods of minutes in moist atmospheres.

Don't forget about the *Electrode Polarisation Effect* at LF when measuring conducting liquids: it can easily give rise to large measurement errors for complex permittivity.

5.3.4 Thin Films and Small Specimens

What *counts* as a 'thin-film' or a 'small specimen' depends upon one's application and the dielectric properties of the specimen. A 'thin' specimen may vary from sub-μm for ferroelectric films to, say, 200 μm for window materials or substrates. The main requirement is that the specimen should give rise to a measurable change in a measured parameter when the specimen is introduced into the cell. This could be a change in capacitance and/or conductance, ΔC or ΔG, in admittance methods, or of resonance frequency, Δf or Q-factor, ΔQ, in resonance methods. Note that, as ever, better sensitivity may well be achievable from a resonator technique, especially if the film is placed at a maximum (antinode) of the standing-wave of the electric field in the cell.

If an appropriate change in the measured parameter is achieved, one can only derive ε' and $\tan\delta$ for the specimen if its *size* or *thickness* is known. The uncertainty in thickness may well be the dominant uncertainty for ε' and $\tan\delta$. One may therefore need to use more accurate dimensional measurement techniques than those given in Section 6.3 to achieve this.

Taking these thoughts into account, we consider here, first, *lumped-impedance* admittance methods (see Section 2.2.1) at RF, then *travelling-* (2.2.2) and *standing-wave* (2.2.3) methods for microwave frequencies, then finally *coaxial* and *waveguide probes* (7.1.12).

Measurements on Thin Films in Admittance Cells at RF. For lower permittivity materials ($\varepsilon' < 30$), the best approach for thin films when using the methods described in Sections 7.1.1 to 7.1.3, is to use a *fixed cell geometry* so as to minimise repeatability errors. Measure the small changes in capacitance and conductivity, ΔC and ΔG, when the specimen is introduced into the cell. Use the air-gap *Capacitance Method* (Section 7.1.1). Optimise the setting of the electrodes in the cell to maximise sensitivity, subject to your being able to insert and remove specimens without disturbing anything else in the measurement set-up. For higher permittivities this technique may also be appropriate but one can also use the metallised specimen technique, if metallization does not destroy the specimen (Section 7.1.1). Optimise the sensitivity of the impedance analyser by appropriate selection of instrument settings and by using a high level of averaging. *Always* repeat the measurement a number of times and take the mean result.

Better sensitivity may be available by using the resonant techniques of Section 7.1.4. The measurement procedures will be similar those of the standing-wave resonator methods described below, because they are all based upon Δf or ΔQ measurements.

For *homogeneous thin films* in the methods described in Sections 7.1.1 - 7.1.4, sensitivity can be improved by *stacking* a number of nominally identical specimens, if available. Indeed, if a number of such specimens are available it is *good practice* to do so to check for consistency and systematic errors.

Small Diameter and Small Cross-Section Specimens, i.e. those with cross-sectional dimensions smaller than the electrodes. Such specimens can be measured in admittance cells, as shown in Figure 17 with appropriate measurement equations. One has to use a theoretical model and measurement equations that take account of the parallel air-capacitance around the specimen (for which one has, of course, to know the areas of the specimen and the electrodes). Ideally one must also take account of the fringing-fields, which are always present in this case. Methods and formulae for such measurements on cylindrical and circular laminar specimens are given in a number of written standards, notably [53]. One can also use electromagnetic modelling (Section 6.9) to improve uncertainties in such measurements.

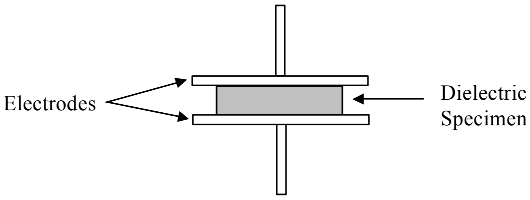


Figure 17. The configuration to be used for measuring a small diameter dielectric specimen in a plane-parallel-electrode admittance cell – compare Figure 1.

Travelling- and Standing-Wave Methods for Thin Films at Microwave Frequencies. The travelling-wave transmission and reflection measurement techniques of Sections 7.1.10 and 7.1.11 are generally too insensitive to measure the properties of thin films and small specimens. More appropriate methods are the standing-wave *resonator techniques* discussed in Sections 7.1.7, 7.1.8, 7.1.16. and 7.1.17. One seeks *appropriate* changes in the resonant frequency and Q-factor, Δf and ΔQ, respectively, on introducing the specimen to the resonator. Changes or perturbations that are too large or too small cannot be measured accurately - or possibly at all!

Perturbation Methods, Section 7.1.16, use first-order approximations. So if $\Delta f/f$ or $\Delta Q/Q$ produce percentage changes of more than about 10%, the perturbation may well be too large, and a smaller (or thinner) specimen may then be recommended. For the other methods, larger changes can be acceptable provided *exact*, and not *approximate* or perturbation measurement equations are used. Note that Q-factor changes of less than 0.5% may be below the useful resolution of the measurement cell.

In principal, very small changes in frequency Δf (e.g. better than parts in 10^6) can be measured very accurately. Bear in mind, though, that the Δf–value measured in dielectric measurements is that of a *resonance frequency* and the accuracy with which resonance peaks can be defined depends upon the Q-factor and can also easily be compromised by temperature drifts and noise in detectors. Ultimate resolutions for $\Delta f/f$ therefore often turn out to be similar to those for $\Delta Q/Q$, i.e. of the order ± 1% and certainly no better than ± 0.1%. Consult Section 6.4.5 for a method suitable for measuring small *changes* in Q-factor accurately.

The basic principle of the resonance techniques for measuring thin films that have been described so far is that *nothing* should be allowed to affect the measurement set-up, and thereby generate changes in f and Q, *apart from* the presence or absence of the specimen. One should therefore, in principle, work with a *fixed* measurement geometry in these methods. However, if the source frequency cannot be changed with sufficient resolution, or if $\Delta f/f$ is large, greater than 5%, say, it may be better to *keep the frequency fixed* and *change the length of the resonator with a micrometer*. The length of the resonator, L, is to be changed in such a way that it will resonate at the *same* frequency with and without the specimen. Subsequently, the measurement equations are used to compute ε' from ΔL. (NB. L here may actually be *any* cell dimension that can be used to control the resonance frequency, e.g. an electrode-spacing). Such a *change-of-length* approach can be just as sensitive as a *change-of-frequency* approach for ε', and can be more accurate if Δf is large compared with f, because sources of loss in resonators (e.g. metal losses, coupling losses) will change with frequency, giving rise to systematic errors when measuring tan δ. In principle, in a micrometer-driven cell, one can use both methods to see if they are consistent with each other – and this can help one to estimate systematic errors.

Coaxial and Waveguide Probes for Measuring Thin-Film Coatings. These probes (see Section 7.1.12) are very sensitive to the dielectric properties of any material that is placed immediately up against their face. Their sensitivity falls off rapidly with depth into the dielectric. They can therefore be used as sensitive probes for the properties of surface coatings, especially for measuring variations across a flat surface. Three important points must be borne in mind, however:

(i) The probe itself will not be able to distinguish between complex permittivity variations in the surface films and variations in their thickness.

(ii) It is essential that good gap-free contact to the surface is made, otherwise *large errors* will result. The method is probably therefore best used with malleable specimens.

(iii) If the coating is delicate the probe may scratch or destroy it.

5.3.5 Magnetic Materials Compared with Non-Magnetic Materials

In this Guide (see Section 2.2.1) we refer to materials for which the permeability, μ^*, is equal to (1.0 - j0.0) for all practical purposes as 'non-magnetic materials' or just simply as 'dielectrics'. *Magnetic materials* are those materials for which μ^* differs significantly from (1.0 - j0.0) at some frequencies of interest. A number of methods discussed in this Guide can be used for magnetic material measurements. The transmission line methods of Sections 7.1.10 and 7.1.11 being among them. In such methods, three approaches may be taken:

(i) ε^* and μ^* of a single specimen can be determined from a single measurement in a transmission cell if transmission coefficients and reflection coefficients are measured [83]. It is the fact that both reflection, S_{11} (or S_{22}), and transmission, S_{21} (or S_{12}), coefficients are measured here that provides one with sufficient data to compute both of the material parameters ε^* and μ^*, see Section 2.2.2, Equations 2.10 and 2.14.

(ii) ε^* and μ^* of a suitable single specimen can be determined in a one-port reflection cell if the specimen is moved to different positions in the standing wave in the cell. A traditional method is to place a specimen firstly hard up against a short-circuit at the end of the transmission line (as in the Roberts & Von Hippel method [84]) then to move it away from the short by $\lambda/4$ and measure it again [1].

(iii) A variation on (ii) is to use at least two or more specimens of significantly different thickness.

Free-field equivalents of these transmission line methods above can also be used, see Section 7.1.14.

The *resonator perturbation method* is also effective for measuring the properties of magnetic materials including ferrites [31, 32]. In fact, it has been the traditional method for determining the parameters of ferrimagnetic resonances for many years; please refer to Section 7.1.16 for details.

Whichever method is used, one must find a way to distinguish between effects due to ε^* and those due to μ^*. This may require two measurements with the specimen placed in different parts of a measurement cell. Method (ii), above, works, for example, because the antinodes of the E- and H-fields alternate in the standing wave along the length of the transmission line.

Three important points relating to magnetic materials should be made:

(1) If one is in doubt as to whether an unknown material is magnetic or not, one ought to use one of these (or related) techniques to check.

(2) RF & MW values of both μ' and μ'' can be significantly affected by the presence of *static magnetic fields*. Thus a small bar-magnet can change the microwave complex permeability of a ferrite material *significantly*! This can easily be demonstrated in the laboratory, for example in a coaxial transmission-line measurement, see Section 7.1.10. Not only are these values changed by the presence of these fields, they are rendered

anisotropic (if they were not so already in the absence of the static H-field).

(3) When RF & MW properties of ferrites are *intentionally* being measured in the presence of strong static magnetic fields, typically in studies of ferrimagnetic resonance, small *spherical specimens* are used because only in spherical specimens is the internal static H-field uniform when the externally applied H-field is of uniform field-strength [31, 32]. The frequency of the ferrimagnetic resonance that is being studied is dependent on the field-strength of the static H-field, so this is an important consideration.

5.3.6 Ferroelectrics and High Permittivity Dielectrics, $\varepsilon' > 100$.

The RF & MW properties of ferroelectrics ([13], [14] Chapter 12, and [35]) are most easily measured by the thin film methods of Section 5.3.4. Very high permittivity ferroelectric films may only be measurable if they are of sub-micron thickness, so special thickness measurement techniques must be used. Thicker specimens may be measured in admittance cells at RF frequencies if their permittivity is not excessively high (e.g. if $\varepsilon' < 3000$, depending on frequency and specimen size) by applying metal electrodes to both surfaces, see Section 7.1.1.

5.3.7 Inhomogeneous, Composite and Structured Materials in General.

It is always important to be aware of the physical nature of the material under test and how it influences the dielectric properties of the material. This can be at the bulk scale, but one should also consider the microstructure and even the chemical structure of the material. Particular consideration should be given to determining whether the material under test is inhomogeneous and/or anisotropic. It is important to realise that inhomogeneity and anisotropy may be intentional or unintentional. Sections 5.3.8, 5.3.10 and 5.3.11 discuss some specific cases. The purpose of this section is to make some general observations.

First some definitions. Materials are *inhomogeneous* if their properties (in this case ε^*) are a function of spatial position within the material. For example, one would consider a mixture of two dissimilar materials (such as chopped carbon fibres in a polymer) inhomogeneous. However, an inhomogeneous material need not be anisotropic. A material is *dielectrically anisotropic* if its properties are different for different directions of imposed E-field. If the chopped carbon fibres are uniformly dispersed in a polymer with a random distribution of fibre orientations then the inhomogeneous composite material remains isotropic. However, if the processing method instils some alignment of the fibres, the composite material becomes anisotropic. In this case, it is important (where possible) to measure test specimens extracted from the bulk material in orthogonal orientations consistent with the expected anisotropy.

Some of the details of the theory of the permittivity of random composite materials is given in [14] Chapters 4 – 7. This includes mixture formulae for particles of different shapes in composites. When it comes to measuring such inhomogeneous materials, it is important to consider the *structure-length* of the inhomogeneity both in relation to the size of the test specimen and the measurement wavelength (see also Section 2.2.3 paragraph 3 (iii), and Section 7.1.17 (g)). In the first instance, the test specimen should provide a good representation of the bulk material. For example, if the test specimen contains chopped carbon fibres that are even half the size of the test specimen, then there are not likely to be many such fibres in an individual test specimen and a high proportion of those that are within the specimen are likely to be damaged or broken. Secondly, recall that permittivity is intended to be an *intrinsic* material property. That is, the permittivity of a material should be independent of the geometry of the test specimen (and the orientation of the test specimen for isotropic materials). This is particularly important when measuring isotropic inhomogeneous materials - a widely used class of materials - since they offer significant benefits for tailoring electrical, dielectric, magnetic and electromagnetic performance for a host of applications. To aid material design, it is useful to assume that the properties of composite materials are intrinsic since the geometry of the material in the final product or component design is then independent of the geometry of the test specimen. The properties of composite materials can be considered intrinsic when the structure-length of the inhomogeneity is small compared to the wavelength. ‘Small’ in this context usually implies less than a tenth of the wavelength (though it can be smaller than this in a resonant system).

This point is worth emphasising, since there has been a recent increase in the use of materials at RF and microwave frequencies which are inhomogeneous on a scale which is *not* small compared to the wavelength. Examples include *photonic bandgap* [85] and negative refractive index [86] materials. These materials can nevertheless still be isotropic. Most researchers use ε^*, μ^* (or refractive index, n) as the parameters to characterise these materials and use predicted or measured values of these parameters when solving Maxwell’s equations for application scenarios. However, much care must be taken when using ε^* values measured in one specimen geometry in design studies in which the material geometry or orientation is different.

Random composites in which the intended structure-length is small compared with wavelength can also exhibit other unintended larger-scale inhomogeities. Many, for example, have *surface layers* that may have a different composition to the bulk material, while moulded specimens may have a higher density near their surfaces than in the bulk. In measuring such specimens one has to decide whether mean values for ε^* of the whole sample are required or whether one needs to machine separate surface layer and bulk dielectric specimens from the sample so that they can be characterised separately.

Density is an important parameter for composites in general - in the case of ceramic and most other composite materials it can be important to specify the specimen density as well as geometrical properties if comparisons are to be made with materials from more than one source. See Sections 6.8 and also 5.3.10.

Note that most random composites consisting of components of very different permittivity will have an *upper frequency* limit of usefulness that is determined by their structure-length and the permittivity of the constituents Scattering from particulate components will occur if the wavelength in the dielectric is close to the structure-length - see 7.1.17 (g) and 2.3 paragraph 3 (iii) for more details.

5.3.8 Foodstuffs

We include foodstuffs here not just because there is direct interest in their measurement to facilitate microwave cooking but because they illustrate a whole range of problems that can also arise with other significantly inhomogeneous types of material, e.g. measurements on biological tissues in general – perhaps for medical purposes (see Section 5.3.13).

One is usually interested in dielectric measurements over a range of food materials. This requires a technique that can be used on a variety of materials over a range of frequencies. Experience has shown that the most flexible method for making dielectric measurements on food materials is the coaxial probe (Section 7.1.12), as discussed below. However, over a more limited frequency range, waveguide transmission cells (Section 7.1.11) have also been used. The latter can have the advantage for composite foodstuffs (e.g. sauces containing solid chunks) of ‘averaging’ complex permittivity across a volume of material, though the exact nature of the mathematical averaging may remain rather indeterminate (see Section 5.3.10).

Food materials can be divided into three main groups; liquids, soft solids and solids. Examples of soft solids are gels, mousses and mashed potato. Examples of solid foods are beef, vegetables and pasta. Many foodstuffs can cover a range of states depending on the temperature, as some foods will heat-set as the temperature rises (e.g. eggs) or cools (e.g. jelly). The probe technique of Section 7.1.12 is adaptable to these changes.

The main factors which need to be taken into account when making dielectric measurements on foodstuffs are:

(i) Temperature
(ii) Bio-variability and Anisotropy
(iii) Specimen presentation

In each of these areas there are specific problems but before any measurements can take place the measurement instrument must be calibrated.

Calibration Whichever method is used, a calibration should ideally be carried out at each of the temperatures that the product is to be measured. However this is not always possible if measurements need to be made over a large range of temperatures in materials such as the soft solids and liquids. Consider the use of a coaxial probe, for example. The measurements must be made with the probe face under the surface of the material. At temperatures below the freezing point of the foodstuff the probe is physically frozen into the material and cannot be removed. In practice, though, it has been found that if a calibration is carried out at a higher temperature (e.g. 15 ºC to 20 ºC) and measurements made at the lower temperatures (e.g. at –15 ºC), then the results obtained are not significantly different to those obtained if calibrations are carried out at the lower temperatures.

Temperature Measurements are usually required over a range of temperatures and care has to be taken to ensure that it is the food material that is being measured rather than an artefact (e.g. air or steam bubbles in the food). Accurate recording of the temperature at the point of measurement can be made by using a fibre optic temperature probe attached to the measurement cell. Some of the problems involved with temperature measurement can be illustrated by considering examples of each of the three different types of foods.

> *Liquids:* These are usually measured from a frozen to heated state as the materials can super-cool. The measurements are generally taken from the lowest temperature to the highest after the probe has been frozen into position. If measurements are required when the material is cooling down, care has to be taken to ensure that effects of super-cooling are taken into account. This can give rise to a hysteresis effect at the lower temperatures with the liquid having higher ε' values when cooling down but the frozen solution having lower values when the product is frozen, the values increasing as the product is heated and starts to thaw.
>
> *Soft Solids:* Probes should also, if possible, be inserted under the surface when measuring soft solids, especially if there is the possibility of the top surface drying out. Usually with gels, measurements are taken as the temperature decreases and the product changes from liquid to solid. It is also advisable to stir the product during cooling to ensure an even temperature throughout the material during the cooling process. In some cases however stirring is not advisable, especially in materials which have different properties depending on the rate at which the material is stirred.
>
> *Solids:* With solid materials the coaxial probe is placed on the surface of the material and care should be taken to ensure a good contact. When frozen materials are being measured the temperature of the probe should be close to that of the material being measured. This ensures that the heat from the probe itself does not thaw a small area of the material, resulting in a measurement of a thin layer of ice or water on the surface of the foodstuff under measurement. As the food thaws there is a possibility that any free water present will be pressed out of the food material and again cause problems by collecting as a layer underneath the probe. There are also problems caused at the solid/liquid phase change. An example of this is a chicken breast or fillet. The temperature at which the meat freezes is –3.5ºC and at this temperature ε' at low microwave frequencies can rise from approximately 10 to 50 and ε'' from 5 to 30, depending upon the amount of ice present. The surface of the product can look completely thawed but the ice can be present below the surface. At higher temperatures the structure of the material could change as the food 'cooks'. The surface of the material becomes dryer and it may be necessary to cut away the surface due to effects such as case hardening.

Anisotropy and Specimen Preparation. Anisotropy of dielectric properties and conductivity is present in any foods such as meat that have a layered or fibrous structure. The permittivity along the grain can be completely different to that measured across the grain (see Section 5.3.11 on anisotropic materials). Specimen preparation is also very important, as can be illustrated by an example. Consider a filled pasta such as ricotta cheese and spinach tortellini in a tomato sauce. In this case three food types are used in combination. Should the various components be measured individually or should the complete mix be measured? If the components are measured individually the following problems could arise:

> *Sauce* – the sauce need not be a smooth sauce but could contain chunks of tomato, onion, herbs and spices which could all have an effect on the dielectric properties. The quantity of herbs and spices present could be very small but have a large effect on the dielectric properties.
>
> *Pasta* – measurements made on the pasta alone will be different from those made on a combination of pasta and filling. When dealing with fresh pasta care has to be taken to ensure the pasta does not dry out as the water content can have an effect on the dielectric properties. If the filled pasta is being measured then the amount of filling can vary at different points and this can also have an affect on the values being measured.
>
> *Filling* – If the filling is being measured on its own then care should be taken to ensure that the filling does not dry out and that the mix of, e.g., cheese and spinach is uniform.

A bulk dielectric measurement can be obtained by liquidising the product but again the results can vary depending upon how long the mixture has been liquidised for. Some materials such as mousses are aerated (See Section 5.3.10) and this can also cause difficulties in making valid measurements as the amount of air present can have a large affect on the results, especially if the air bubbles are not distributed uniformly through the system. If one can remove the air from the material then measurements can be made on the de-aerated material and mixture formulae (Section 5.3.10) applied to obtain the dielectric values for aerated mixes. As stated above, it may also be possible to use a waveguide cell (7.1.11) to obtain an average permittivity for such products.

For materials such as breadcrumbs which are dry, the crumb can be pressed into a sample cake of known density and measurements taken over a range of densities. The dielectric parameters of the crumb can be determined from these results. See also Section 5.3.10.

Biovariability This can have the effect of causing large variations in the values of both permittivity and loss. Peas from the same pod can have values that vary by as much 10% to 15% and the variation between different pods can be even larger. The variety of fruit and vegetable can also make a difference and the permittivity of a *Cos* lettuce is different from that of an *Iceberg* lettuce. The time at which the food is harvested can also make a difference with foods picked before being ripe and then allowed to ripen having different values to those that are allowed to ripen *in situ*. This has been used commercially, for example with water melons, where dielectric measurements have been used to identify the time at which the fruit is ready for harvesting. Some foodstuffs can degrade very quickly. Ripe food can become mouldy within a matter of hours at room temperature and care has to be taken to ensure that the food is fresh when measured unless the moulding process is itself being studied.

Moisture Measurement in Foodstuffs Besides measuring the dielectric properties of foodstuffs to help with their processing or cooking, another very important metrological area is the measurement of moisture in unprocessed foodstuffs. This can have enormous financial importance. For example, millions of tonnes of newly harvested grain are produced in the USA every year. Its cost is based upon its weight but the higher the moisture content the higher the weight of the grain! As the purchasers of the grain wish to pay for the grain and not the moisture, measurement of the latter is very important. It is performed by dielectric measurements [22]. Such measurements extend even to the measurement of the dielectric properties of single kernels and seeds, see [22], Ch. 12. Moisture measurement of *fish* is also important to determine its freshness and has been performed dielectrically by using a microstrip proximity transmission cell [87]. A complementary technique has also been developed to determine the quantity of *fat* in fish [22], Ch. 27 (Kent *et al.*).

5.3.9 Moisture Measurement in Dielectrics.

As mentioned in the last paragraph, moisture measurement by dielectric means can be ‘big business’ in the food industry. Its applications extend beyond foodstuffs, however. Such measurements have long been the subject of a series of conferences and workshops [88], one of which, an IEEE Workshop in 1993, gave rise to the book ‘*Microwave Aquametry’* [22] edited by Andrej Kraszewski. There is much useful information on moisture measurement in this book, many of the techniques discussed are dielectrically based. It also presents a 15-year bibliography of literature in the field, covering almost 600 publications.

Almost any of the techniques covered in the present guide can be adapted for moisture measurement. We need only note that resonance techniques, as usual, will often be more sensitive. Given that water has both high permittivity and high loss at microwave frequencies, however, its presence is easily detected in normally low-loss or low-permittivity materials, so that non-resonant admittance cells and single- or double-pass methods are often sensitive enough. Measurement of moisture content is best achieved by calibration against reference specimens containing known amounts of water, as analytical computation of permittivity vs. water content can be quite a complex affair (Section 5.3.10). Another important point to note is that moisture may not be the only component of a composite dielectric giving rise to loss. For example, presence of dissolved salts can also cause significant loss, especially at LF & low RF, and so can act as a confounding factor. In this case it may be necessary to perform measurements over a broad band of frequencies to distinguish the *dielectric* loss arising from moisture from the *conduction* loss arising from dissolved salts, see Section 2.4.

5.3.10 Powders, Mixtures and Aerated Products

Powders are an important class of materials, however they pose a major measurement challenge. The source of the challenge stems from the fact that it is difficult (if not impossible) to measure a single particle to obtain the permittivity of the particle material. Therefore, one needs to construct a test specimen from the powder that suits one’s test method. There are a number of possibilities, either the powder can be packed into or flow through a suitable container, or the powder can be formed into a solid, free-standing test piece by pressing or mixing it with a binder material. In all cases, the added complexity comes from the dependence of the measured permittivity upon the particle shape, the concentration of the powder and the influence of the particle arrangement (microstructure) on the measured value. These additional parameters affect the properties of aerated or porous materials and of dielectric mixtures in general. It is assumed here that one generally wishes to extrapolate the measured permittivity to that of the bulk material (i.e. the value at 100 vol %), or else to estimate the properties of a mixture at a different volume concentration (or porosity level) from that tested.

First of all, some guidance (derived from experience) can be provided for the preparation of test specimens from powders. Since powders often constitute an increased health and safety risk – they can become airborne more easily and thereby ingested - COSHH [82] and risk assessments should be completed before handling the powder. Ideally, specimens should be prepared at a volume concentration close to that to be used for intended applications, this requires knowledge of the bulk density of the powder. This can be measured by Archimedes method, otherwise known as '*Archimedean Weighing'*, [89] using a density bottle or a pycnometer. The porosity of pellets pressed directly from the powder can then be determined by measuring the density of the pellet (by measuring the physical dimensions and mass of the pellet). The volume fraction of any retained porosity is given by:

$$P = \left(1 - \frac{\rho_{pellet}}{\rho_{powder}}\right) \qquad \ldots 5.1$$

The optimum pressure to be used when pressing powders may need some experimentation. A pressure in the range 130-260 MPa is suggested as a starting point. The dwell time over which the pressure is applied may also be varied (for example, in the range 60 - 300 s). Heat may also be used to aid consolidation, if required. Nevertheless, many powders may be too hard to form a sufficiently consolidated pellet. In these cases a binder must be used. It is important for the binder to have sufficiently different (often lower) permittivity compared to the powder under test and for the binder to have low loss. Even so, it will be difficult to give an accurate measure of the loss for low-loss powder materials using this method. Fortunately, those powders which are too hard to be pressed themselves are likely to have permittivities which are significantly higher than many convenient binders such as polymers and plastics. Some insulator ceramics may also be considered as binders.

Paraffin wax is a versatile choice since it melts at relatively low temperature (~ 60 °C) or can be dissolved in a range of organic solvents. Nevertheless, care must be taken when blending the powder to achieve a homogeneous dispersion. Consideration should also be given to the accuracy at which quantities of powder and binder are combined as this will directly affect the accuracy of the powder volume concentration. Specimens are readily pressed from the resulting mixture once it has been allowed to cool to room temperature, using the least severe of the conditions recommended above. The volume fraction of powder, V, in the resulting test specimen can be estimated as follows by measuring the specimen density, knowing the densities, ρ, of the constituents and assuming no retained porosity:

$$V = \frac{\rho_{sample} - \rho_{binder}}{\rho_{powder} - \rho_{binder}} \qquad \ldots 5.2$$

This method is particularly well suited to measuring the properties of metal and other conductive powders.

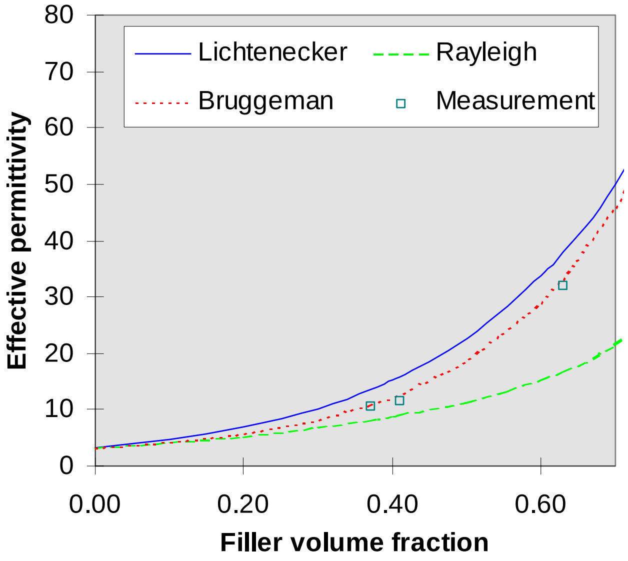


Figure 18. Comparison of mixture formulae for calcium titanate filler in a polymer binder. The measurements demonstrate that the Bruggeman formula best fits the measured data in this case (from Youngs' Thesis [90], with permission).

The final stage of the process is to extrapolate the measured properties to those of a fully dense material, or to some other powder concentration for design purposes. The tools to be used for this are *mixture laws*, also known as *effective medium theories*. This topic deserves a Good Practice Guide of its own, and, indeed, there is a vast scientific literature and a number of books on the subject. The reader is referred to the following references, as a starting point: [14, 91, 92] and, in the particular context of moist mixtures, to A Sihvola [22] Ch. 8. Only a brief discussion of these can be provided here. For uniformly dispersed non-conducting powders at low filler concentrations the Maxwell-Garnett (or Rayleigh) [93], unsymmetric Bruggeman [94] and Lichtenecker [95] mixture laws are commonly used, see Figure 18, but there are many more. The former has certainly been extended for ellipsoidal particles [96, 91] (used for generic shapes such as spheres, rods or flakes) and coated particles [96, 91]. These mixture laws may also be used with care at higher filler concentrations to extrapolate to concentrations that are within 5-10 vol % of the measured concentration. The uncertainty associated with this process will clearly increase the further the extrapolation is taken from the concentration of the test specimen.

For conducting powders, the microstructure of the dispersion becomes more important. If the powder is uniformly dispersed and the particles do not contact each other until the powder is close-packed then the previously stated mixture laws can be used. However, if the particles are randomly dispersed then the mixture can become conductive at powder concentrations well below the close-packed condition. This effect is often referred to as *percolation*. In this case the symmetric Bruggeman model [94] is more representative, although the phenomenological modifications proposed by McLachlan [97] provide for a variable critical powder concentration (the concentration at which the mixture undergoes the insulator-conductor transition: the *percolation threshold*) and a more accurate representation of the critical behaviour as a function of both powder concentration and frequency. The interested reader is referred to the following publications for more detail on the dielectric properties of conducting powder dispersions: [96, 90, 91, 98]. The relative importance of selecting the most appropriate mixture law depends on the difference between the dielectric properties of the constituents and on frequency. This is exemplified by comparing Figures 19 and 20. These figures present the calculated effective conductivity of a composite material as a function of conductor concentration at a frequency of 1 GHz using a wide range of mixture laws. The majority of the mixture laws are for spherical filler particles, although the Lagarkov mixture law for stick-like particles (an aspect ratio of 100 is used) and the spheroidal particle Maxwell-Garnett law are also included to demonstrate the effect of particle shape. The Doyle mixture law provides a means of accounting for clustering of the filler particles. Two types of clustering are considered. The first assumes that all the filler particles reside in clusters at any filler concentration. The second results in an increase in the proportion of filler particles that are in clusters as the filler concentration increases. Figure 19 is for a filler conductivity of 10^6 S m^{-1} and demonstrates the larger contrast between filler and matrix property. Figure 20 is for a filler conductivity of 1 S $m.^{-1}$ In absolute terms, the bounds defining the effective properties of a mixture become larger with increasing contrast between matrix and filler properties. When the contrast is small, as in Figure 20, selecting the most representative mixture law for a given mixture becomes less important.

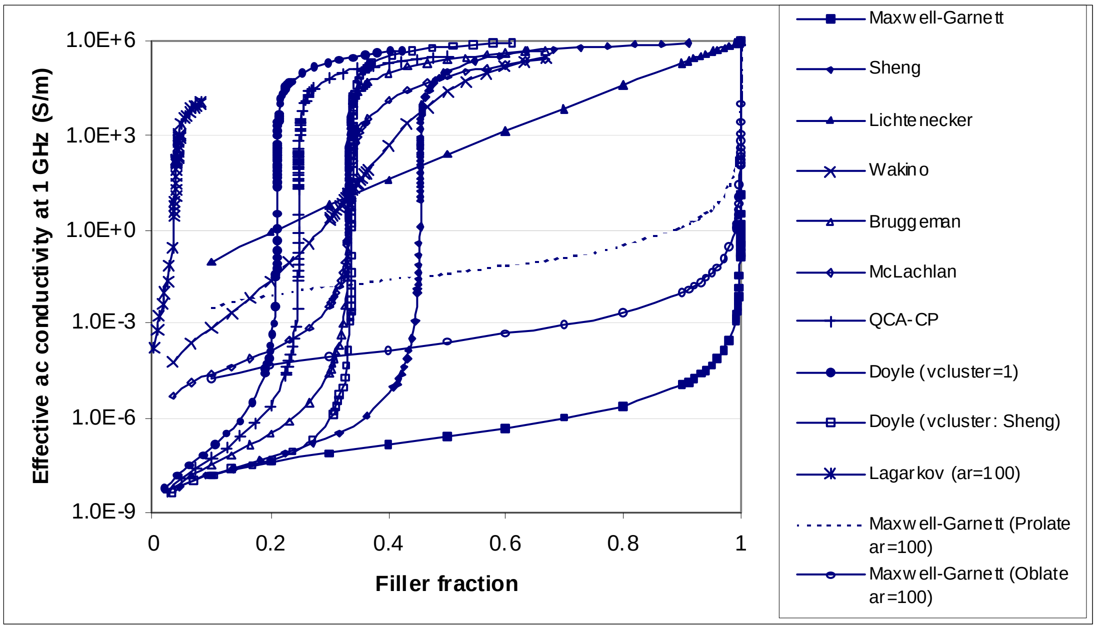


Figure 19. Calculated effective conductivity of a composite material as a function of conductor concentration at a frequency of 1 GHz using a wide range of mixture laws. The filler conductivity is 10^6 S/m (from [90], with permission).

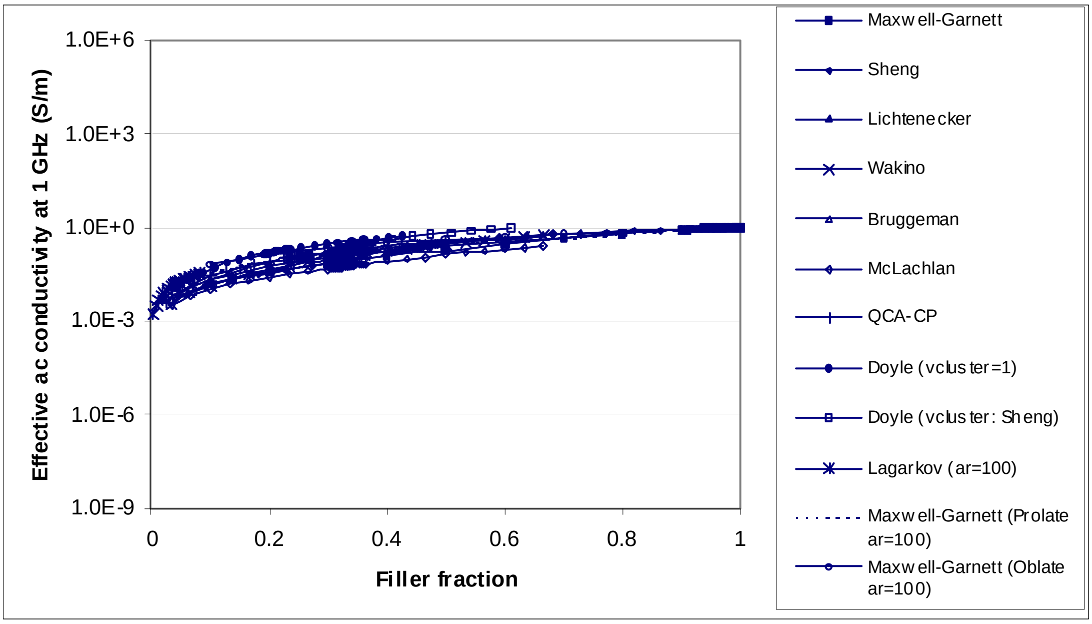


Figure 20. As Figure 19 but for filler conductivity of 1 S/m demonstrating that the choice of mixture law is apparently less important as the conductivity of the filler becomes closer to that of the binder (from [90], with permission).

5.3.11 Anisotropic Dielectrics

As stated in the last section, a material is anisotropic if its properties are different in different directions. In the case of dielectrics this means that either or both ε' and ε'' will take different values in different directions in a material. In fact, a full theoretical treatment [1, 4] recognises that both the complex permittivity, ε^*, and the complex permeability, μ^*, are material properties that are formally represented by *tensors*. We need not go into too many details here but the metrologist has to be aware that is does <u>*not*</u> mean that the value of ε' (for example) changes *gradually* as one rotates the polarisation of the E-field vector in the specimen (although this may *appear* to be the case in some measurement techniques). Rather it means that the material actually exhibits at least two *clearly distinct* values of ε' in different directions, a phenomenon that also gives rise to *birefringence*. This can result, for example, in *two separate resonances* being visible when a specimen is being measured in a high Q-factor resonator such as an open resonator, see Section 7.1.17, Figure 52.

Many purely ionic crystals like quartz and sapphire are well known to have quite marked anisotropy [99]. For example, quartz has permittivities $\varepsilon'_\perp$ = 4.43 and $\varepsilon'_\parallel$ = 4.63 perpendicular and parallel [100] respectively to its optic-axis, while the corresponding values for sapphire are $\varepsilon'_\perp$ = 9.40 and $\varepsilon'_\parallel$ = 11.58 [71]. Not surprisingly, fibrous materials can be significantly anisotropic, either dielectrically or in terms of their conductivity. The LF conductivity of muscle fibres can be 30 times higher along the fibre than across the fibre, for example. *However*, even an ostensibly amorphous polymer may be anisotropic if it has frozen-in stress, while manufacturing methods (e.g. slip-casting) often generate anisotropy in materials (see next Section). Dielectric metrologists must be aware of these facts because to assume a material is isotropic when it is not can actually lead to large measurement errors when using some measurement techniques, e.g. waveguide probes, Section 7.1.12 and open-resonators, Section 7.1.17.

As pointed out in Section 6.6, one needs to measure dielectric anisotropy in the right kind of measurement cell, i.e. one which sets up a linearly polarised electric field in the specimen under test. One then has to perform measurements on the specimen in two orthogonal polarisations to measure its anisotropy (for uniaxial materials, for example). Rectangular (or square) waveguide cells possess the correct linear polarisation, as do free-space and other TEM methods like open-resonators. See Sections 5.3.8, 7.1.12 and 7.1.17 for further discussions on measurements of anisotropy.

Some materials (e.g. sugar solutions) rather than being *linearly* anisotropic are *optically active* and they need to be characterised in cells which set up left or right *circularly polarised* waves, see Section 6.6.1. Methods which use TE_{01}-modes in circular waveguide (e.g. 7.1.7) may be relevant for this purpose.

5.3.12 RAM – Radiation Absorbing Materials

Manufactured RAM typically comes in four forms:

(a) Carbon-powder impregnated polymer foam – typically used at microwave frequencies in pyramidal form for free-field absorption, as in microwave electromagnetic anechoic chambers.

(b) Ferrite or other magnetic material – typically used in ceramic tile form for RF anechoic chambers.

(c) Absorbing rubbers, impregnated with magnetic, conducting or lossy dielectric materials – typically used in gaskets or applied to surfaces or as screening to enhance the *electromagnetic compatibility* (EMC) of electronic equipment.

(d) Laminas, thin films or paints for coating surfaces – typically for similar screening purposes or for absorbing *surface waves*.

For measurements of *intrinsic* dielectric and magnetic properties of RAM, i.e. ε^* and μ^*, each of the four types of RAM requires a different measurement technique, as described below. In many cases, though, users of these materials do not need to know the intrinsic properties. They may be interested only in *extrinsic* properties, like reflectivity or absorption performance, and such measurements are often best done in the free-field by the techniques described in Section 7.1.14. Intrinsic measurements are useful, however, if multilayer matching RAM is desired (as in ‘hybrid’ RAM). Only if the intrinsic dielectric properties are known can the design of the hybrid RAM be optimised for minimum reflection, e.g. by Genetic Algorithm (GA) techniques [101]. For intrinsic measurements of ε^* and μ^* the following methods are suitable:

(a) *Carbon-Powder Impregnated Foam.* Measure them either by the free-field methods of Section 7.1.14, or, at RF, in a suitable TEM cell - see ‘*Other TEM Cells*’ in Section 7.1.10 - or by using a coaxial or waveguide probe, Section 7.1.12. In the latter case, however, take note of the paragraph on measuring *soft materials* in Section 7.1.12 and also take note that such carbon-loaded RAMs are rarely impregnated uniformly, so extensive sampling over a wide area may be necessary to obtain the ‘average’ dielectric properties. Such ‘averaging’ is performed automatically by free-field and large area TEM–cell measurements. Also when using probes, note that carbon impregnated RAM (unless painted on the outside) tends to shed carbon particles, so, if the RAM has been in long use, it may be better to cut into it to an internal region that cannot have shed carbon particles so as to perform measurements that can characterise the bulk of the material.

(b) *Ferrite or other Magnetic Material.* Cut or grind coaxial or waveguide specimens for the methods described in Sections 7.1.10 and 7.1.11. Alternatively, small ferrite specimens can be measured at microwave frequencies by the perturbation method of Section 7.1.16. Indeed this is an established technique for characterising ferrites for all types of microwave applications [31, 32]. At RF entire ferrite tiles can be measured in a suitable TEM cell - see the paragraph on ‘*Other TEM Cells*’ in Section 7.1.10.

(c) *Impregnated rubbers.* For magnetic-material impregnated rubbers, cut or grind coaxial or waveguide specimens for the methods described in Sections 7.1.10 and 7.1.11. If the material is relatively soft and will not retain its shape in the transmission line, it can be sandwiched between two rigid window materials of known permittivity, as one does in transmission line measurements on liquids. If the rubber is non-magnetic, a similar approach can be used or else a probe technique (Section 7.1.12) can be used, as long as the correct numerical analysis is employed if the specimen is in laminar form.

(d) *Laminas, thin films or paints for coating surfaces.* Use the methods advocated in Section 5.3.4 for thin films. If the RAM is a *Surface Wave Absorbing Material* – sometimes referred to as ‘SWAM’, note that a special technique has recently been developed for monitoring fields close to the SWAM using an Optically Modulated Scatterer [102, 103] – see also Section 7.1.14.

5.3.13 Biological Tissue and Biomedical Dielectric Measurements.

The problems that one encounters in the measurement of biological tissues can be very similar to those encountered in the measurements of foodstuffs (see Section 5.3.9). This is not surprising because most foodstuffs *are* biological tissues. Typical measurement difficulties include the effects of biovariability, anisotropy and gross inhomogeneity in specimens, e.g. large differences in permittivity between moist and fatty tissues. Temperatures of interest range from sub-freezing in the study of cryo-preserved tissues stored for use in tissue transplants to the mid-40s Celsius, which are the temperatures used *in vivo* for diathermy and hyperthermia medical treatments of tumour tissues, see e.g. references in [79].

Coaxial probes (see Section 7.1.12) offer one of the most widely-used techniques for measuring tissue *in vivo*, though in medical studies chemical methods may have to be used to sterilise the probes if they cannot withstand high temperature treatments. Some of the problems and errors that can arise in such measurements are described in [22], Ch. 19. 'Non-calculable' reflectometric probes have also been used for medical tissue measurements *in vivo* in order to achieve better measurement penetration into tissues, see Section 7.1.13.

There is already a considerable body of data available on the dielectric properties of human tissues, however, see [42] for example, so that it may not always be necessary to carry out such measurements oneself, though there are circumstances in which they will be always be necessary, e.g., when dielectric properties are being measured for diagnostic purposes.

Many of the dielectric measurements that are carried out in health and safety testing today are not on biological tissues at all, but rather upon *Tissue Equivalent Materials* (t.e.m.s), usually liquids, such as those used in measurement of the SAR (Specific Absorption Rate) levels generated by mobile communication devices [50, 51, 52]. The t.e.m.s are required to have complex permittivities similar to those of actual tissues – hence the reason why dielectric measurements are central to SAR metrology. Again, coaxial probes are the most widely-used tools for this job.

5.4 Specimen Preparation and Handling.

5.4.1 Manufacture and Preparation of Specimens

It is difficult to overemphasise the importance of having a properly prepared specimen if one wishes to obtain the best measurement accuracy. For many techniques, such as admittance-cell methods (see Sections 7.1.1 to 7.1.3) the 'best available' uncertainty is often dependent upon the uncertainty in a critical specimen dimension (usually the thickness). This will be the *best* achievable uncertainty, other factors will usually ensure that the actual uncertainty is worse than this. In general one should inspect the *measurement equations* which relate the measured parameters to the permittivity in order to optimise the specimen dimensions for best accuracy and to discover just how sensitive the result will be to specimen dimensional imperfections, see Section 4.3.

When one *has* to measure specimens that are less than perfect (for example if they are the only specimens available), one may be able to choose a technique that is less sensitive to these imperfections. For example, measurements in a TE_{01}-mode cavity and in open-resonators (see Sections 7.1.7 and 7.1.17) can be rendered relatively insensitive to specimen dimensions by ensuring that all specimen surfaces lie in low electric-field regions, e.g. at a *node* in a standing wave pattern. This will be the case if the specimens are an integral number of half-wavelengths thick (i.e. half-wavelengths *in the dielectric medium*). This has the added advantage that surface contamination is not 'seen' by the measurement.

For many measurements at RF & MW frequencies one would like the dimensional uncertainties to be as small as a few μm if they are not to present the dominant measurement uncertainty. This is, in general, possible to achieve (at a cost) with ceramics and hard specimens by machining or grinding their surfaces. Unfortunately, there are many other materials which cannot be machined to such tight tolerances. Without excessive cost the best achievable uniformity for soft polymers (e.g. polyethylene) is often a few tens of μm. This certainly can compromise measurement accuracy. In these circumstances it is important to take as many steps as possible to reduce other sources of error associated with the specimen shape. Here are a few pointers:

1. Many specimens can be moulded (e.g. polymers) or else sintered (e.g. ceramics) to the 'correct' shape for the measurement. Having done this, it is as well to check that a specimen has indeed achieved the specified shape, as it may have contracted or expanded after removal from the mould. If not, improvements may be possible by machining. For example, by turning or grinding specimen faces flat and parallel.

2. If thickness is critical, always measure it at a number of points across the face of the specimen, see Section 6.3: the faces may turn out to be uneven or non-parallel. Use the *mean thickness* in computations. This should be a *weighted mean* if the electric field magnitude varies significantly across the specimen, as does that of a Gaussian Beam in an open-resonator, see Section 7.1.17.

3. Check that the specimen is not warped or dished. These defects can cause significant uncertainties in some measurement methods. Sometimes these effects can be minimised by mounting a specimen in a specific way, see Section 7.1.17 again, or else by weighing it down at its edges.

4. Even turning of specimens on a lathe can introduce unevenness on specimen surfaces. If not done carefully a small ‘knob’ or protrusion can be left at the centre of the specimen face. This should be removed to improve measurement accuracy, especially if the method requires the specimen to lie flat on a surface. Likewise burrs or lips of the edge of specimens should be removed. Thickness may vary with diameter if the specimen is badly machined. Centreless grinding is often a better way to prepare the surfaces.

5. Remove all burrs and projections, if possible, without contaminating or damaging the specimen!

6. All specimens should be prepared with clean cutting tools. Polymers should usually be prepared at low machining speeds, without cutting fluids. This avoids both contamination and specimen distortion (or melting!) from the heat generated by machining.

7. Specimens of magnetic materials should not be held using magnetic chucks when machining.

8. Vacuum chucks offer an effective means of holding specimens for machining without contaminating them. Unfortunately they cannot be used with all specimens.

As ever, what can be done to improve measurement will depend upon the time and finances available: but remember that the eventual uncertainty of a measurement often varies inversely with the resources that one can put into preparing specimens.

In addition, one may often be required to take account of other aspects of specimen preparation of, e.g. curing or heat treatments. There are so many possibilities here that we cannot cover them all in this general guide. There are two very important issues that we should mention, however: *moisture in specimens* and the more general issue of *specimen contamination*, see also Sections 5.3.2. and 5.3.3.

Regarding moisture, make sure that you know from your ‘customer’, if you do not already know yourself, whether the specimen is to be measured ‘dry’ (i.e. with no moisture uptake in it from the atmosphere) or under ambient conditions. If it is to be measured dry, you will need to know how to dry it and keep it dry. It is often sufficient to place specimens for a period in a bell jar containing a drying agent, but the amount of time it will take to do this varies significantly from one type of material to another, though it is typically a number of days. Provided one’s measurement takes minutes to perform rather than hours, moisture uptake during an actual measurement is usually only significant for hygroscopic liquids,

The contamination of solids by volatile liquids is a condition that may solve itself through gradual drying, provided that the specimen material does not react or dissolve in the liquid. If a specimen is contaminated with other types of substance it may have to be abandoned if their removal could cause more damage than the actual contamination. Sintered ceramics may be an exception because some common contaminants can be removed by heating the material in air on a clean non-interacting ceramic base-plate, e.g. a zirconia setter. The temperature used for cleaning the material should be carefully chosen so that it does not affect the sintering of the ceramic. For refractory materials, such as alumina, a period of one hour at 1000 ^{o}C is usually sufficient. It would be advisable to consult the manufacturer of the material before embarking upon such a treatment, however.

Finally, do remember the significant influence of temperature on dielectric properties and do allow specimens that have been brought into the laboratory from elsewhere adequate time to reach laboratory temperature.

5.4.2 Handling and Storing

The two main policies here are very simple:

A. *Prevent specimens from degrading and from becoming contaminated and damaged*. It is especially important to prevent degradation of reference specimens, as discussed in Section 4.4.2, because they are purposely kept for long periods of time, but it is also important to prevent degradation of *any* specimens that are to be kept or remeasured over a period of days, weeks or months.

B. *Prevent specimens from contaminating or adversely affecting you, your colleagues and your laboratory*! We refer here, of course, to toxic and/or flammable specimens – see the COSHH regulations [82].

Advice on how to follow these policies is scattered throughout this guide in relevant sections, but here are some of the more important general points to remember:

1. *Specimen degradation 1*. Specimens can degrade with time in a number of ways. They can become contaminated, on the one hand, by moisture, but on the other, a specimen that is *intended* to be moist can dry out. Biological tissue specimens will denature and decompose with time. Steps against these eventualities range from storage in adequately sealed containers to freezing – do consult relevant experts.

2. *Specimen Degradation 2.* Many polymers and other dielectrics can degrade when exposed to light, particularly ultra-violet light. Store all specimens in the dark. e.g. in a drawer.

3. *Specimen Degradation 3.* Some materials, even polymers like polyethylene, can oxidise. If such materials are intended for long-term use, they should either be made from material grades that contain anti-oxidants or else they should be stored in the dark in containers which seal them from the atmosphere.

4. *Specimen Degradation 4.* Some specimens, especially thin polymer specimens, can become warped with time (from the effects of 'frozen in' stress). If this is likely to happen, they can be stored under compression to keep them flat.

5. *Cleanliness 1.* Low-loss specimens ($\tan \delta < 0.003$) should always be kept, manipulated and measured in clean conditions. They should *never* be touched by the human hand. Contamination from moisture, dirt, sweat (especially the chemical salts in sweat) can increase dielectric losses significantly in some ceramics. Clean polymer gloves and/or tweezers may be used *with care* for manipulating specimens.

6. *Cleanliness 2*. Make sure the measurement cell is clean prior to insertion of the specimen.

7. *Toxicity, flammability.* See also Section 5.3.3. In general *always* consult materials suppliers on these matters. Take the advice on handling and storage from COSHH [82] datasheets and safety experts. Perform COSHH assessments with help from safety advisors. Be aware of the risks associated with any material *before* you use it and avoid them.

6. Dielectric Measurements: Common Themes

In the following Section of this Guide, Section 7, we will be presenting a survey of a number of techniques used for RF & MW measurements upon dielectric materials. However, there are many features of these techniques that are shared, the use of instrumentation or the measurement of Q-factor, for example, and it is a good idea to consider these features separately. In this section we therefore consider nine 'common themes' in practical RF & MW dielectric metrology. Perhaps the most important sub-section is 6.2, which provides general advice on *all* dielectric measurements. Most of the other sections can be read selectively: readers can pick out from their titles those sections that are most likely to be relevant to their interests.

6.1 Instrumentation

The purpose of this section is to the survey instrumentation and equipment commonly used in RF & MW dielectric measurements and to give guidance on how it can best be put to use for this purpose.

6.1.1 Network Analysers - Automatic Network Analysers (ANAs).

ANAs are the 'work-horses' of today's RF & MW laboratories. We therefore assume a general familiarity with their use and with the concept of ANA calibration on the part of the reader. For those who are not so familiar, there are a number of excellent introductions to their use and theory [11, 12, 104, 105]. Where available, use of *vector network analyers* (VNAs) rather than *scalar network analysers* can be recommended because they can measure the real and imaginary parts of S-parameters. In some techniques, e.g. ANA transmission line methods (Sections 7.1.10 & 11) the ability to do this is essential: we *need* this complex-quantity information when we are trying to measure quantities which also have real and imaginary parts: i.e. the complex permittivity and permeability of materials. More generally though, VNAs are also to be preferred because they provide *more information* than scalar analysers, this information can be put to use in reducing measurement errors and for diagnostic purposes. Scalar analysers are cheaper, however, and can be used for some purposes, e.g. measurement of Q-factor, Section 6.4. ANAs should, of course, *in general* be calibrated prior to a measurement, but full calibration is not always necessary. For example for measurement of high-Q factors, the VNA can be used as a source/receiver combination for which calibration is often unnecessary over a short band of frequencies, see Section 6.4. Nevertheless one does need to know that the ANA is both *stable* and *linear*, so periodic checking and verification of ANAs is advisable.

Some VNA models have a synthesised time-domain facility as one of their features [106]. This can be useful in a number of techniques for *gating* or *de-embedding* the S-parameters of a specimen from those of any mismatched components and imperfect transmission lines that lie between it and the ANA, see Section 7.1.19.

Note *that, unless otherwise explicitly stated, the term 'ANA' is used in this Guide to imply a Vector ANA.*

6.1.2 Impedance Analysers, 'Materials Analysers', Admittance and Impedance Bridges.

Such instruments are available today from reliable manufacturers in fully automated form with a number of calibration regimes built into their software. The use of such instruments can be well recommended, if only because they save considerable time compared with the older manual bridges. They are generally used in the LF and RF ranges up to 1 GHz and their use up to at least 200 MHz is usually to be preferred for impedance and admittance based measurements because better accuracy can often be achieved than with ANAs, though residual impedances, such as inductance in leads (see Section 2.2.1, Equation 2.8), can significantly reduce their accuracy, even at frequencies as low as 10 MHz. The frequency coverage-range of impedance analysers overlaps that of ANAs (which can be used at all RF & MW frequencies). ANAs are generally to be preferred for S-parameter-based measurements especially above 250 MHz where good quality air-line-based standards can readily be used to calibrate them. Some impedance analysers are supplied by the manufacturer with an attachable dielectric measurement cell and they are marketed as 'Materials Analysers'. The manufacturer's recommended calibration and measurement techniques may well be followed and the internal materials-measurement software used with such cells, but better accuracy can sometimes be obtained by following some of the alternative methods described in this Guide. If so used, instruments should be switched to their impedance (or admittance) measurement mode and any internal software that displays or outputs measured dielectric parameters should not be used, as it applies only to manufacturer's own techniques.

Two important issues arise which are concerned with connections between the measurement cell and the analyser. Firstly some bridges have coaxial connections (see, e.g., [107]) but many do not. For example how should one connect an analyser fitted with four-terminal pair connectors (4-T-P) [80] to a three- or two-terminal admittance cell? The answer is given in Section 7.1.1. Secondly, how can these instruments be checked and calibrated? 4-T-P national standards are available up to 1 MHz (shortly to become available up to 10 MHz in the UK at the time of writing this Guide) but such instruments are commonly used up to 30 MHz (and above) with 4-T-P connectors. Where there is doubt, they can be used with a null or substitution method in which a full capacitance calibration is not necessary, see Section 6.5, but traceability for resistance and conductivity can remain a problem and so uncertainties for loss may have to be increased in consequence.

6.1.3 Frequency Response Analysers (FRAs), Dielectric Spectrometers and Analysers

Frequency Response Analysers (FRAs) are purpose-built instruments used for dielectric measurements below 10 MHz and down to frequencies as low as a few microhertz (μHz). Commercially available systems also enable measurements as a function of temperature, time, dc and ac voltage bias. The specimen under test is measured in an admittance cell configured as a parallel-plate capacitor. Representative specimen geometries include discs with diameters in the range 10 to 40 mm and thicknesses in the range 0.5 to 10 mm.

The principle of operation of the FRA is described by Barnikas [108]. Essentially, a sinusoidal voltage is applied to the specimen under test whose admittance is determined by measurement of the in-phase and quadrature components of the voltage across the specimen through the use of an automated comparator circuit comprising a series of resistors and capacitors. The real and imaginary components of the permittivity of the specimen are subsequently obtained from the dimensions of the specimen capacitor (plate separation - d, plate area - A) and the measured capacitance (C) or conductance (G), respectively, following Equation 6.1:

$$\varepsilon^* = \frac{Cd}{\varepsilon_0 A} - j\frac{Gd}{\omega\varepsilon_0 A} \qquad \ldots (6.1)$$

Some FRAs are supplied with additional circuitry in the form of a variable (capacitance) reference standards and an active specimen cell. These features enable more accurate measurement of low-loss materials at low frequencies where the impedance becomes large, and the improved accuracy of phase measurements yields greater loss-tangent resolution [109]. The use of the reference standard eliminates the uncertainty in the comparator circuitry of the FRA. The active specimen cell reduces the effects of cable inductance compared to standard coaxial cable (apparent at frequencies above 100 kHz) by placing the analogue electronics in the specimen cell fixture thus enabling the use of rigid air-insulated lines. The calibration processes are generally described in the equipment manual and involve the measurement of the capacitor with no specimen in it.

It is worth noting the typical performance limits of the instrument. FRAs can have an impedance range of 0.01 to 10^{14} Ω and a loss-angle resolution of < 0.1 mrad. It is recommended that the reader refer to the equipment manual or contact the manufacturer for more details. Schaumburg [110] provides a discussion of best measurement practice, specimen preparation and measurement accuracy. The contributions to measurement uncertainty are the residual system uncertainties, uncertainties in specimen dimensions, effects of bad electrical contact between the electrodes and the specimen, the contribution of edge capacitance and electrode polarisation effects (Section 5.3.3). The residual system uncertainties should be obtainable from the equipment manual. The effects of bad electrical contacts are observed as a lowering of the real permittivity and/or the presence of additional losses. These effects can be mitigated by the metallisation of the specimen faces. Edge effects (see Section 7.1.3.) can be physically eliminated by the use of guard rings or mathematically removed. An example of the latter is given by Equation 6.2 in which D is the diameter of the specimen capacitor, C_0 is the capacitance of the empty capacitor and t is the thickness of the capacitor plates. The size of the edge-capacitance increases with increasing t and decreases with decreasing d/D.

$$\begin{aligned} C^*_{sample} &= C^*_{measured} - C_{edge} \\ C_{edge} &= \frac{2dC_0}{\pi D}\left[\ln\frac{8\pi D}{d} - 3 + z(x)\right] \end{aligned} \qquad \ldots (6.2)$$

where $z(x) = (1+x)\ln(1+x) - x\ln x$ and $x = t/d$ and d is the electrode separation.

6.1.4 Q-meters.

The Q-meter has been the instrument traditionally employed for measuring Q-factor in the RF range, typically from 10 kHz to 70 MHz. It is essentially an L-C resonant circuit with loose coupling between the resonant circuit, the source and the detector. The output of a voltmeter in the detector circuit may thus be calibrated directly in terms of the circuit Q-factor, which is determined from the amplitude of a resonance, see Section 6.4.

Q-meters used to be used to measure the full range of RF components, i.e. resistance, capacitance and inductance. Where available though, impedance analysers (Section 6.1.2) and FRAs (Section 6.1.3) are these days normally to be preferred. Likewise at higher microwave frequencies, ANAs have nowadays subsumed most of the functions of Q-meters so they are no longer a key part of the current measurement repertoire. However, historically Q-meters played a key role in the measurement of low-loss dielectric materials such as polyethylene for submarine telephone cables where a resolution of 0.5 μradians was achieved when measuring LDPE (low density polyethylene) with a loss of 50 μradians at 30 MHz [111]

Two factors are of interest here:

(i) A standard of Q-factor may be provided by use of a carefully designed inductance coil, but this should have been calibrated traceably in a calibration laboratory.

(ii) It may be preferable to use the *Attenuator Series Substitution* method described in Section 6.4.5 for measuring Q-factor. This requires a traceable attenuator, which can be measured to ± 0.001 dB or better at the frequency of measurement. The RF detector in the Q-meter can then be used as a null detector. In this way non-linearities in the detector circuit may be avoided.

6.1.5 Displacement Measurement with Micrometers.

Micrometers generally play two major roles in dielectric measurements: (i) to change the distance between electrodes, mirrors etc. during the measurement and (ii) to measure the size (typically the thickness) of specimens. The latter is a sufficiently important topic to deserve a separate discussion - see Section 6.3. Here we discuss the use of micrometers in measuring instruments.

We first consider non-digital micrometers. When choosing such a micrometer for fitting into a measurement cell, remember that the resolution of the micrometer depends upon the size of its drum. Drums 10 cm in diameter, or larger, can be used to measure displacements of a few mm down to a resolution of a few tenths of a μm. Such resolutions are required to gain the full benefit of some measurement techniques, especially of *equivalent-thickness* methods, Section 6.5. Such resolutions cannot be obtained from small diameter drums.

More conveniently, micrometers are these days available with a digital readout. They can read to ± 1μm even with a small drum diameter and they may be recommended if this resolution is adequate.

In dielectric measurements we frequently need to know the value of the micrometer zero-error, Z, i.e. the correction to the micrometer reading, M, which tells us what the actual gap, D, between the driven electrodes (or equivalent) actually is, i.e. $D = M - Z$. In admittance cell measurements this parameter is often best measured capacitatively, as shown in the worked example in Section 9.5. In re-entrant cavities (Section 7.1.4) and some other cells, it is not possible to measure inter-electrode capacitance because the electrodes are effectively shorted together via the rest of the cell and so it may be necessary to use ball gauges or slip gauges instead.

<u>Backlash.</u> When adjusting a micrometer, always avoid backlash by making sure that the last rotation applied when setting it is in *the same consistent direction* - either clockwise or anti-clockwise. Which of the two is better will depend upon instrumental considerations. For example, if a micrometer is being used to *lift* and *lower* a mirror or electrode it will usually be better to make the final setting motion a *lift*: if there is a little friction between the moving parts, lowering motions can be unsteady, with the device being lowered sometimes becoming temporarily suspended above its support - in this case the micrometer.

Always use a *calibrated* micrometer, and check, from time to time, that its motion remains true by using e.g. calibrated slip gauges. Calibration of micrometers by an accredited UKAS laboratory is not usually very expensive.

6.1.6 Automated Translational Displacement Mechanisms and Displacement Measurement Systems.

Manually adjusted micrometers have the advantage of giving one a confidence-boosting 'hands on' feel in measurements but it has to be admitted that motor-driven automatic displacement transducers and automated translational displacement measuring systems may well be more efficient and time-saving in many cases. The following practices can be recommended for these systems.

(i) Calibrate the measurement systems and check their correct operation regularly.

(ii) In designing automated systems, consider the inclusion of an alternative *manual* displacement/ measurement mechanism that runs in parallel with, and can substitute for, the automated system. This enables checks and displacement-calibrations to be performed easily and cost-effectively *in-house*. For example, an automated system may be based upon a motorised micrometer with an optical displacement measurement system (e.g. laser interferometer or Moiré fringe counter), but the micrometer itself can be calibrated cheaply and can be used to check the automated measurement systems from time to time.

(iii) Furthermore, do not rely upon the automatic translation mechanisms *themselves* to give you the magnitude of the displacement: transducers and motors can fail, e.g. stepper motors can 'miss' a step. Use a *separate* displacement measurement system in parallel with them (e.g. an optical system, as above).

(iv) In designing a measurement system, be sure that the automatic displacement measurement system that you intend to use has adequate resolution - some can only be read to ± 1 μm - this generally means that the *uncertainty* in displacement will be *greater than* ± 1 μm. In some RF & MW measurement techniques we aim for tenth-of-μm resolutions. (see Section 6.5).

6.1.7 Coaxial Connectors and RF Cables.

All *critical* coaxial connectors in your measurement system, i.e. those through which RF & MW signals travel to and from the measurement cell, should be gauged [112, 113] from time to time, especially those at which connections are regularly made and broken. At such points in a measurement system *use connectors which are specifically designed for metrological use* rather than those intended for one-off connections, e.g. use APC-7 or APC-3.5 connectors rather than SMA connectors [*ibid*, 114]. The latter can wear rapidly. Out-of-gauge connectors can easily give rise to poor contacts and to large, varying mismatches and therefore to significant unquantifiable measurement errors. Follow good connector practice in general [112, 113].

Keep RF & MW cables as short as possible. If they are flexed during measurements, use the best quality metrological cables you can afford: flexing of cables gives rise to significant phase changes in travelling waves at microwave frequencies. Check cables from time to time by measuring their S-parameters on an ANA and gauge their connectors – connectors on regularly flexed cables are more likely to go out-of-gauge as a result of usage than those on fixed geometry devices. The effective working lifetime of a coaxial cable is normally inversely proportional to the number of times it is flexed. Consult manufacturers' specifications on the minimum radius-of-curvature to which cables can be flexed. Using smaller radii may produce mismatches in the cable during measurements and is likely to degrade a cable's long-term performance.

6.1.8 Measurement Cells in General.

A number of general recommendations can be made here:

(a) With the exceptions of (i) free-field measurements (See Section 7.1.14) and (ii) resonance measurements (Section 6.1.9), it is usually best to keep the measurement volume of cells as small as possible, consistent with their achieving the required resolution for ε' and tan δ. Also keep connections as small and simple as possible. At RF greater size brings with it residual inductance (see Section 2.2.1) and therefore more complex cell equivalent-circuits have to be employed to model the cell. For admittance cells (Section 7.1.1 – 4) remember that the larger the size, the smaller the usable frequency range. Resonators and cavities invariably have to be built to the size demanded by the physics of the E.M. waves and so this advice may not so be readily followed – but, again, keep connections small and simple.

(b) *Temperature control.* If the cell is to be *actively* temperature controlled, small cell size may also be an advantage because power requirements can be smaller and temperature settling times can be faster. However, in some cases a *contrary* policy may be advantageous. Whenever a high resolution of a measured quantity such as capacitance is required, it can be an advantage to have a cell with a long time-constant, so that the constant temperature cycling of a temperature-control system does not compromise resolution. In such cases use of a 'massive' (large mass) cell can be recommended. Likewise, if a small-thermal-mass cell is used in a temperature controlled laboratory, cycling of the temperature in the room can easily compromise measurement resolution. In this case use *passive temperature control* to increase the thermal time-constant of the cell – e.g. use lagging or simply place the cell in a sealed box. See also Section 6.7.

(c) Be aware of the configuration of the E.M. fields in the cell - this will enable you to chose the optimum size and shape for specimens, see Section 6.6. This is even more important for anisotropic materials. Note, in particular, that E-fields usually pass *through*, i.e. from face to face of, laminar specimens in 'lumped impedance' methods but, by contrast, they usually lie *in the plane* of the lamina in travelling- and standing-wave cells. This fact can be important if the material to be characterised is anisotropic (Section 5.3.11).

(d) Keep cells clean so that they don't contaminate specimens.

6.1.9 Resonators and Cavities

Resonators and cavities form a special class of measurement cell with their own points of special interest. Background theory can be found in [8], [115], [10] and [9]. In general we use resonators for measuring specimens of low loss, and so we need to know how to reduce loss in the resonator itself and in coupling mechanisms – this will increase the resonator Q-factor and increase its resolution for specimen loss. But we also need to distinguish unambiguously between *the loss of the cell* and *that of the specimen under test* – a point that is important in resonator and in measurement-procedure design. However, we should not think that the benefits of resonators arise only in the measurement of dielectric loss - the increased resolution offered by resonator techniques also applies to real permittivity ε': some of the most accurate methods for measuring ε' at microwave and millimetre-wave frequencies are resonator methods.

Common concerns with resonators are:

(i) How can we improve Q-factor?
(ii) What should the magnitude of our coupling coefficients be?
(iii) How do we estimate losses from coupling holes, diffraction, etc.?
(iv) How do we derive the measured values of ε' and tan δ?
(v) How do we measure Q-factor accurately?

The last point is sufficiently important to deserve a section of its own and so it is discussed in Section 6.4. The others are discussed below, but first we should develop a little theory regarding loss in resonators (see also Section 6.4.4). When the dielectric specimen is mounted in the resonator, the Q-factor we measure is called the *loaded Q-factor* or Q_l. (but see the paragraph labelled 'Warning' at the end of Section 6.4.3). Now a simple relationship exists between Q_l and all of the individual sources of loss in the resonator. Each of these can itself be allocated its own Q-factor:

$$\frac{1}{Q_l} = \frac{1}{Q_{specimen}} + \frac{1}{Q_{resonator\ walls}} + \frac{1}{Q_{coupling}} + \cdots \qquad \ldots (6.3)$$

Where $Q_{specimen}$ accounts for the dielectric loss in the specimen – this is the parameter we are trying to measure, $Q_{resonator\ walls}$ accounts for the power lost in the metal walls of the resonator due to conduction losses, $Q_{coupling}$ accounts for power lost through all coupling mechanisms into the resonators: note that power is lost both through output *and* input coupling ports. Now let us examine some of the terms on the right hand side of Equation 6.3 in detail. $Q_{specimen}$ is inversely proportional to the loss tangent of the specimen, tan δ, so we can write

$$\frac{1}{Q_l} = F_f \tan\delta + \frac{1}{Q_{resonator\ walls}} + \frac{1}{Q_{coupling}} + \cdots \qquad \ldots (6.3a)$$

where F_f is the specimen 'filling-factor' in the resonator defined as follows:

$$F_f = \frac{\text{EM energy contained in the specimen } (W_s)}{\text{EM energy contained in the whole resonator } (W_r)}$$

or [116]:

$$F_f = \frac{W_s}{W_r} = \frac{\iiint\limits_{Vs} \varepsilon_s' E.E \mathrm{d}V}{\iiint\limits_{Vr} \varepsilon' E.E \mathrm{d}V} \qquad \ldots (6.4)$$

Where V_s is the volume of the specimen and V_r is the complete volume of the resonator including the specimen and ε_s' and ε' are the corresponding real parts of the permittivity at each point in the corresponding volumes. Thus F_f quantifies the extent to which the specimen effectively *fills* the resonator. In dielectric resonators (Section 7.1.15) it is the body of the dielectric itself that is resonating and so F_f is very close to 1.0. In other resonators (e.g. those described in Sections 7.1.7 and 7.1.17) F_f may be less than 0.1, while in perturbation methods, Section 7.1.16, F_f may be < 0.01. The optimum filling-factor to be used depends on the method, the loss to be measured and a number of other factors, see Section 6.4.4.

Just as we gave a meaning to $Q_{specimen}$ in Equations 6.3a and 6.4, we can do the same for $Q_{resonator\ walls}$. In this case we need to know the *surface resistance* [115, 116] of the metal walls, R_s, and we define a *Geometric-Factor* [116], G_s, such that: $Q_{resonator\ walls} = G_s/R_s$. The surface resistance is connected to the metal conductivity, σ, by the relationship $R_s = 1/\sigma\delta_s$, where δ_s is the skin-depth in the metal, $\delta_s = \sqrt{2/\sigma\mu\mu_0\omega}$, μ is its relative permeability, μ_0 is the permeability of free space and ω is the angular frequency: $\omega = 2\pi f$. With this substitution, Equation 6.3 becomes:

$$\frac{1}{Q_l} = F_f \tan\delta + \frac{R_s}{G_s} + \frac{1}{Q_{coupling}} + \cdots \qquad \ldots (6.3b)$$

G_s is given by:

$$G_s = \frac{\omega \iiint\limits_{V} \mu_0 \, | H |^2 \, \mathrm{d}V}{\iint\limits_{S} | \vec{H} |^2 \, \mathrm{d}S} \qquad \ldots (6.5)$$

where H is the magnetic field at any point in the resonator, and $\vec{H}$ is its tangential component on the conducting surfaces of the resonator, V is its volume and S designates the metal surface of the resonator. The integral in the denominator is taken over all of the cavity walls. G_s is called the *Geometric-Factor* for metal-wall loss in the cavity. Note that G_s is independent of the actual conductivity of the walls, just as F_f is independent of the actual dielectric loss of the specimen. There are some simplifications here. First we are assuming that the conductivity of the metal walls is the same at all points. However, if this is not the case we can modify Equation 6.3b to include multiple terms for wall-loss, each with its own appropriate value for R_s. Another more intractable point is that we have ignored the loss due to *contact resistances* between different bodies of metal in the walls, e.g. between the resonator and any lid or port in it that is needed to insert or remove the specimen. More will be said on this point below.

Equation 6.3 can be further modified: we see that we can take each contributory Q-factor on its right hand side and replace it with a more specific formula based upon a physical model, but we will not pursue this further here. Equation 6.3b contains two of the most important factors in the design of any resonator: the *filling-factor* and the *geometric-factor* (the next term in equation 6.3b – coupling loss - is discussed in Section 6.4.3). We can now return to the questions we asked above and also make some other important points about resonator design.

(a) *How can we improve Q-factor?* For a given resonator geometry we need to minimise all sources of loss in the resonator. Figure 21 illustrates these for an open-resonator (see Section 7.1.17). We can use high conductivity metals, such as copper, to construct the resonator, or if that is not possible we can coat the inside of the resonator with a film of high conductivity metal such as silver. For more details see *'Loss in Metal Walls and Mirrors'*, below. Coupling holes aside, see (b) below, try to ensure that breaks in the metal wall of the resonator are made along physical lines that are *not* crossed by electrical currents. To achieve this you will need to understand the field and current patterns of the resonant mode you are using – see for example [10]). Wherever there are metal-to-metal interfaces, the contacts may be poor or patchy, giving rise to series contact resistances. Not only can this reduce Q-factor but it can also distort field patterns in the resonator significantly, perhaps leading to large systematic errors. In general, minimise the influence of coupling holes, see (b) below. In open-resonator and quasi-optical systems make sure that mirror apertures and specimen diameters lie in sufficiently low fields for the diffraction losses at their edges to be negligible.

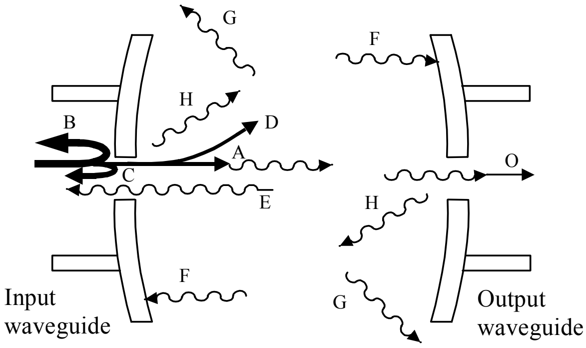


Figure 21. Power transfer and loss processes in a resonator. An open-resonator (see Section 7.1.17) is used as an example. The resonator has two small coupling apertures for input and output from waveguides (shown over-scale for clarity). (A) Power coupled from the input into the resonant mode, (B) input power reflected by mismatch, (C) attenuation of input signal by the 'cut-off' section of the input coupling aperture, (D) power coupled through the input aperture that does not enter the resonant mode, (E) power lost from the resonant mode via the *input* coupling aperture, (F) dissipation in the metal reflectors (conversion to heat) (G) diffraction at reflector edges, (H) scattering (diffraction) from the coupling apertures, (O) power transmitted to the output waveguide. Losses (E) to (O) *load* (i.e reduce) the Q-factor of the resonant mode. (After Clarke & Rosenberg [215]).

(b) <u>*How can we reduce loss from Coupling Mechanisms?*</u> Coupling mechanisms or resonator 'ports', be they mere holes, monopole antennas, or loops, can reduce Q-factor by two different mechanisms:

(i) By coupling power out of the resonator. Note that *output* coupling ports are *designed* to do this, but don't forget that *input* coupling ports also couple power *out* of the resonator just as effectively!

(ii) In open-resonator and quasi-optical structures coupling ports can *scatter* radiation out of the resonant mode and out of the resonator, see Figure 21.

In general, use *small* coupling holes/mechanisms so that less power is coupled out of the resonator (but see Section 6.4.3 for an alternative approach that applies to reflectometric Q-factor measurement). In the specific case of coupling <u>*holes*</u>, see Figure 22, make them as thin (front to back) as possible, by cutting into the back of the wall in which the hole is placed (i.e. cut back the wall *outside* the resonator). Heidinger and his co-workers have shown [117] that by carefully machining thin coupling holes in open-resonators (see Section 7.1.17), smaller diameter holes can be used to achieve the same coupling coefficient. This has the advantage that less power is scattered out of the resonance mode by diffraction from the coupling holes. Heidinger has been able to increase Q-factors considerably by these means.

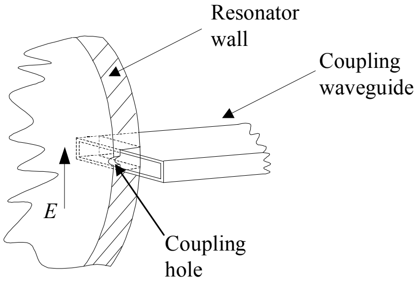


Figure 22. Coupling from waveguide into a resonator using a small coupling hole – reduce the depth of the hole by reducing the thickness of the resonator wall on the *outside* of the resonator.

(c) <u>*What should the magnitude of our coupling coefficients be?*</u> This is discussed in some detail in Section 6.4.3. In general in *transmission measurements* we should use as *low* a coupling factor as possible, consistent with obtaining good signal-to-noise ratio. Resonator insertion losses in the range 40 – 60 dB are generally acceptable for transmission measurements. For coupling <u>*holes*</u> this requires hole diameters in the approximate range $\lambda/20 - \lambda/10$, where λ is the free-space wavelength, depending on the depth of the hole, see (b) above. Note that coupling coefficient changes *very* rapidly with coupling-hole size – as the cube of the diameter for small, thin holes - so it is probably better to design for a small coupling hole size and subsequently open out the diameter carefully by small steps if the coupling coefficient obtained is too small. In reflection measurements, other factors come into play – refer to Section 6.4.3. In general remember that the lower the loading from the coupling, the higher the Q-factor.

(d) *How do we estimate losses from coupling holes, diffraction, etc.?* In principle these can be explicitly computed using suitable field formulae, see, e.g. [118] for coupling holes. However, in *practical* resonators analytical formulae may not so readily be applied. Analysis of coupling through a hole, for example, may assume that the metal wall in which it is situated is infinitesimally thin - in practice it will have finite depth, see Figure 21, and so it will form a *waveguide below cut-off* [119]) and allow less power transmission than a thin coupling hole of the same diameter. In many cases, therefore, to estimate coupling losses, it may be necessary, in practice, to measure them.

(e) *Should we polish metal surfaces in the resonator in order to improve Q-factor?* It is very difficult to give a general answer here. Experience has shown that losses can actually be increased by the process of polishing surfaces because of adherence or inclusion of polishing compounds into the surface. On the other hand, rough or deeply scored surfaces will certainly reduce the Q-factor [120], so it is advisable to use well-machined, shiny surfaces. If the Q-factor obtained is adequate, it may be better to avoid polishing. High tolerance machining, e.g. diamond turning, may be the best solution for preparing a good low-loss surface.

(f) *How do we derive the measured value of ε' and tan δ?* Details are left to the discussion of individual resonators in Section 7 and to the references to be found there.

Computation of Filling-Factors and Geometric Factors The definition of F_f and G_s in Equations 6.4 and 6.5 might make it appear that one should involve oneself in quite deep mathematics to derive values for these parameters. Traditionally, this has always been the case, but the advent of computer-based numerical modelling based upon discretised techniques such as FDTD and FI renders this task much easier. In some modelling packages one can explicitly calculate the average energy stored in a resonator and also in specified *volumetric regions* of a resonator, for example inside the specimen. The ratio $F_f = W_s/W_r$, as in Equation 6.4, can therefore readily be obtained by these means, and this can be invaluable in a number of measurement techniques, e.g. in calculating filling-factors for re-entrant cavities, see Section 7.1.5. See Section 7.1.5 also for a discussion of other important issues relating to filling-factors and metal loss.

Loss in Metal Walls and Mirrors. In general, we want this source of resonator loss to be as low as possible. For this reason copper is used for cavity walls and mirrors for most measurements at ambient temperatures because it has the highest conductivity of commonly available metals [21]. Aluminium can also be used, but only if electric currents set up by the resonance do not have to pass through metal-to-metal contacts in the cavity walls (see (a) above): aluminium makes poor electrical contacts because of its robust surface oxide film. Sometimes it is more convenient or cost-effective to use another, lower-conductivity, metal to manufacture a cavity, e.g. brass, because it is cheaper to use and easier to machine, or else invar to prevent cavity expansion with temperature. In such cases it is advisable to coat, 'flash', deposit or sputter a thin film of higher conductivity metal on the inside walls of the resonator. A silver film is often a good choice. Whatever metal is used it should be deposited to a depth of at least three skin-depths, δ_s in the coating metal. At cryogenic temperatures cavities can be made from a superconducting metal like niobium to reduce the wall losses.

Surface Impedance. Equation 6.3b above demonstrates how the Q-factor of a resonator relates to the *surface resistance*, R_s, of its walls. *Full* characterisation of the interaction of the E.M. field with the metal walls, however, requires the concept of a *surface impedance* Z_s, which has a reactive, as well as a resistive part. Besides the loss, Z_s also takes account of resonance-frequency changes that occur because the E.M. fields penetrate into the metal. Waldron, [115] Ch. 5, provides an account of this phenomenon. In *cryogenic measurements* with *superconducting* cavity walls, it should be remembered that, while superconductors may be effectively lossless at LF, there are physical mechanisms that cause losses in them at RF & MW frequencies. They too have a 'skin-depth', called the *superconducting (or London) penetration depth* [121], and so their resonator wall-loss can also be characterised by a surface impedance, Z_s. Nevertheless, Q-factors of superconducting cavities are invariably much higher than those of ambient-temperature cavities of the same size. At 10 GHz, they may be in excess of 1.10^9, as opposed to ~ 5.10^4, for a typical ambient-temperature dielectric-measurement cavity.

Mode-degeneracy, coincidence and interference. *Mode-degeneracy* is the term used for the case where two or more resonance modes coincide in frequency. In general mode-degeneracy should be avoided in metrology. Resonators and cavities should be designed so that other resonant modes do not overlap in frequency with the mode that is being used for measurements. In some cases degeneracy cannot be avoided, however. For example, the TE_{01}-mode is degenerate with the TM_{11}-mode in cylindrical cavities, see Section 7.1.7. As one wishes to use only the former mode for dielectric measurements in a 'TE_{01}-mode' measurement cavity, the latter has to be removed by a mode-filter. In general, even if it is possible to design a cavity which avoids mode-coincidence when no specimen is in the cavity, there is no guarantee that mode-coincidence will not occur when the specimen is present. The most important practical ways to avoid or to detect the presence of mode-coincidences are (i) avoid distorted resonances, e.g. resonances with two peaks or with inherent asymmetry, (ii) use more than one axial resonant mode, as is possible in TE_{01}-mode cavities and open-resonators, (iii) use two specimens of different thickness. One can also use numerical analysis to check for possible mode-coincidences if one has the relevant numerical or computing tools, see Section 6.9.

The Positioning of Coupling Ports. Coupling ports can be positioned and orientated in resonators so that they preferentially launch and detect the required resonance mode, whilst simultaneously failing to excite (or detect) unwanted modes – especially unwanted modes which are wholly or partially *degenerate* with the wanted mode. One way of achieving this is to place the ports at *nodal positions* of the relevant E- or H-fields of the potentially most troublesome interfering modes, choosing the node of whichever field (E or H) is being used to effect the coupling. Careful consideration of the *direction of polarisation* of the launching mechanism can also preferentially increase the coupling coefficient, β, of the wanted mode, whilst reducing it for the unwanted modes. It is sometimes advantageous, for diagnostic purposes, to depart from this practice (e.g. in *dielectric resonator* metrology, Section 7.1.15, where mode identification is often more effective if *all* modes are temporarily excited during an initial set of measurements), but this practice should ideally be adhered to when the dielectric measurements are actually being performed. In order to make this policy effective, one must be aware of the modal field-patterns that the resonator can sustain, see Section 6.6.

Coupling Ports and the Placing of Specimens. In general dielectric specimens should be kept well away from coupling ports of a resonator because their presence will change the coupling-coefficient of the port. This is easily understood in the case of *coupling holes*. The coupling coefficient, β, of such a hole depends upon the ratio of its diameter to that of the wavelength of the E.M. radiation. In dielectrics the wavelength is smaller than in free space so if the dielectric specimen is placed directly over the coupling hole, this effect will probably cause β to rise. However, the presence of the specimen over the coupling hole will *also* present a *mismatch* to the radiation, which would tend to make β fall. The combination of these two opposing effects is difficult to quantify - all we can say is that the placing of a dielectric over the hole will almost certainly *change* the loading of the cavity, so giving rise to a spurious change in Q-factor, which in turn will introduce a systematic error into the measurement of loss. This effect can be important in measurements on very low-loss specimens as the error may amount to as much as 10 µrad in loss angle.

> ► ***Do's and Don'ts for Resonators and Cavities:*** Some of these points are covered in the text above, others are discussed in greater detail elsewhere in the Guide.
>
> ***Do*** (in general) design for high Q-factor and use high conductivity metals for resonator walls. If necessary coat the walls with a high conductivity metal film.
>
> ***Do,*** where possible, try to design resonators so that metal walls and specimen supports lie in low fields and the specimen in high fields. This can reduce systematic errors. ***Don't*** forget that for metal-loss in the walls of a resonator it is the *H-field* (magnetic field) that matters, not the E-field (see Equation 6.5).
>
> ***Do*** design resonators to increase the ratio of power loss in the specimen to that from all other sources. Even where the latter can ostensibly be removed from the measurement by means of a substitution technique (see Section 4.4.1), residual systematic errors are likely to be lower if the loss of the rest of the resonator is lower. (NB. This point may not apply in resonator perturbation measurements, Section 7.1.16)
>
> ***Do*** think carefully about where to place coupling ports in resonators. You may wish to use points where the surface currents or fields are high if you use a very small coupling mechanism, or else you may choose points where the fields or currents are low, where larger-sized coupling mechanisms may be tolerated. Avoid points where the relevant field or currents of the *desired* resonant mode pass through a node. ***Do*** on the other hand, if possible, select points where the relevant fields (or currents) of potentially troublesome *unwanted* resonance modes are close to their nodal position (especially if they are degenerate with the wanted mode).

Do, if coupling *holes* are used, make sure that they have as small a depth as possible because it may be possible to get adequate coupling with a lower diameter coupling hole, thereby reducing the loss caused by scattering of radiation (diffraction) from the coupling hole

Do consider using *tunable* resonators. Resonators in which one dimension (e.g. the length in a TE_{01}-mode cavity or in open-resonators) can be used to control the resonant frequency are more flexible in their uses. They also offer better opportunities for estimating systematic errors in measurements and will allow *change-of-length* methods to be used instead of, or as well as, *change-of-frequency* methods, see Section 6.4.5

Don't place breaks, apertures, doors, etc. in the metal walls of resonators at points where the surface current of the desired resonance mode will be strong. This reduces the Q-factor and can seriously distort field patterns in the resonator if there are poor metal-to-metal contacts: they can potentially give rise to *irreproducible* systematic measurement errors.

Don't place specimens directly on coupling ports or even near to coupling holes – this can change their coupling coefficient and introduce errors for measurement of loss by changing their loading of the Q-factor.

6.2 General Recommendations on Dielectric Measurements

The following recommendations apply for most RF & MW dielectric measurements:

(a) All significant equipment should be calibrated and the calibration should be traceable to international measurement standards.

(b) The effectiveness of all calibrations (e.g. the calibration of an ANA) should be checked. Once the measurement system has been calibrated and *prior* to the measurements upon the specimens under test, it is good practice to *check* the calibration, either by a measurement upon a *check specimen* that has known dielectric properties or else by measuring a traceable dielectric reference material (Section 4.4.2). In both cases the measured complex permittivity should lie within pre-specified acceptable limits for the calibration to be declared valid. If it does not do so, the calibration may have to be repeated. The purpose of such checks is twofold. *First*, they can demonstrate that the calibration has been performed correctly and so they can improve confidence in the measurements (Section 4.2). *Secondly*, they prevent one from wasting time in performing invalid measurements. Check specimens should be carefully maintained specially for this purpose. They may have been used many times before: a consistent history of measurement helps to promote confidence, so a record of check measurements should be kept. This can be added to, say, once every few months or once a year, depending on the frequency of usage of the equipment. Ideally the properties of check specimens should match closely with those of the specimens under test. If a wide range of materials with differing properties is to be measured on a regular basis, it may be as well to maintain a corresponding range of check specimens.

(c) For laminar specimens, whenever possible, it is good practice to measure a number of specimens of the same material but of different thickness to check for systematic errors. Likewise, in transmission line measurements (Sections 7.1.10, 7.1.11) specimens of different length may be used. A similar principle, suitably adapted, can be applied to many other dielectric measurement techniques.

> However, when the aim of the measurements is to determine the *differences* in the intrinsic dielectric properties of a number of similar specimens, they should all have the *same* size and shape, so as to remove differences caused by systematic errors.

(d) Whenever possible it is good practice to measure specimens across a broad frequency range to check for consistency of properties across that range. Such measurements can also expose effects that give rise to systematic uncertainties, e.g. standing-waves in a cell can be detected as ripples in the value of cell S-parameters across the measured frequency range: they are symptomatic of mismatches in supposedly matched cells. It is therefore good practice *not* to restrict measurements only to the frequency of interest if one can afford to do otherwise. This is usually not a problem with ANA measurements.

> Whenever both the real permittivity, ε', and dielectric loss, $\tan \delta$, of a non-conducting material are being measured across a broad band of frequencies, it is good practice to check for mutual consistency of the behaviour of these two parameters by following the procedure described in Section 2.5.

(e) It is good practice to make sure that all dielectric specimens are individually identified. They should each have a unique name or serial number. In some cases, the specimens themselves can be marked in a way that does not compromise the accuracy of measurement. In others, especially with low-loss materials and liquids, it may not be possible or advisable to do this. In this case each specimen should be allocated a clean container, e.g. jar, bottle, tin, package, polyethylene bag - as appropriate, and the container should be uniquely marked instead. To prevent similar specimens from getting mixed up, *only one specimen at a time should be out of its container*, i.e. each should be returned to its container before the next specimen is removed.

Record the provenance of specimens. If they are taken from a single batch of material or are machined from a single sample, this fact can be recognised by allocating specimen names or serial numbers that explicitly code for the batch or sample. It is sometimes important, especially with inhomogeneous materials, to record the position in the batch or sample from which each specimen is taken.

(f). Dielectric properties can change rapidly with temperature. The temperature of dielectric measurements should *always* be recorded. In the case of ambient temperature measurements, an uncertainty of ± 0.2 ºC or better should ideally be achieved, though this may not be possible at elevated temperatures. A relative humidity of less than 50% is recommended for measurements unless otherwise required by the measurement or its 'customer'. In some cases, especially with low-loss specimens, it will be necessary to record relative humidity at the time of measurement and possibly also during the prior storage of the specimen.

(g). Record *all* relevant information on measurements in a lab book or computer file (see Section 4.4.4). If doubts arise about a measurement at a later date, this may enable you to trace the cause of the problem. On the other hand, if there is nothing wrong with the measurement, the record will help you to demonstrate this fact at a later date and so provide confidence in the measurement.

(h). It is good practice to generate a measurement uncertainty budget following the practices of 'The Guide' (GUM) [68]. In RF & MW measurements it is conventional to state uncertainties for a coverage factor of $k = 2$, corresponding to a confidence level of ~ 95%. See Section 4.3 and 4.3.2 in particular for details.

Regrettably, it will not be possible to apply all of the above principles to all dielectric measurements. In many cases we may not be able to do so because of cost and time constraints. We should, however, in all cases, keep as closely as possible to the spirit of these recommendations.

6.3 Measurement of Specimen Size: Thickness, Diameter, etc.

Most dielectric measurements require an accurate measurement of at least one specimen dimension, such as thickness and diameter, so dimensional measurements are to be regarded as an important part of any dielectric measurement - not to be skimped. Significant dimensional measurements should be performed by the most accurate method that is cost-effectively available. Callipers are *not* normally accurate enough for small laboratory specimens, but micrometers and air-gauging [122] generally are suitable for all but very thin specimens. Care should always be taken not to contaminate or damage the specimen during the dimensional measurement process. Reference may also be made to the standard methods ASTM D374M-99 [54] and ASTM E252-84 [55], which describe methods for thickness measurement of sub-millimetre and thin films.

6.3.1 Use of Micrometers for Thickness Measurement

Bench mounted micrometers are to be preferred to hand-held micrometers because they present less risk for contaminating or of dropping the specimen. As one is often trying to attain a resolution of ± 1 μm or better, the micrometer anvils should be clean and all dust should be removed with a lint-free cloth prior to measurement. An effective way of achieving this is to hold the cloth with a *very light force* between the anvils and then to draw it gently sideways. Heavy force should never be applied between the anvils as this distorts and may damage the micrometer. Most micrometers have a ratchet mechanism that ensures that an appropriate and repeatable force is applied during measurements. Micrometer zero-errors (see Section 6.1.5 for a definition) should be checked prior to *and after* measuring the specimen, or after every few specimens if a batch is being measured, and the mean value used to obtain the thickness – if the 'before' and 'after' values are significantly different, clean the micrometer and repeat the measurements!

Specimen thickness, t_s, should ideally be measured at a representative number of points across the area to be tested in order to check its uniformity. The *relevant* mean thickness across the area to be subjected to dielectric measurement should be computed. The significance of the word 'relevant' here is that the weighting of the measurements to compute the mean value should depend upon the nature of the dielectric measurement. For the Gaussian Beam measurements in open-resonators, Section 7.1.17, for example, higher weighting should be given to points in the centre of the specimen where the highest beam E-field amplitude lies, while for specimens to be used in an admittance cell, uniform weighting across the area between the electrodes is appropriate. The appropriate weighted mean value for t_s should be used in all subsequent computations and its standard deviation should be used to estimate uncertainty in ε' due to uncertainty in t_s. With forethought and practice, these procedures need not take too long and, with care, each specimen can be adequately measured in a few minutes.

Hard Specimens, e.g. ceramics: a calibrated traceable micrometer may be used to measure specimen thickness, t_s, to ± 2 μm at any given point across the face of the specimen. It is usually good practice to use the micrometer constant-force ratchet mechanism in such measurements.

Rough Specimens. Bear in mind that, especially with hard specimens, you are measuring the distance between the highest projections of the surface between the micrometer anvils, so the *mean* surface of the specimen will always lie below these projections. The mean thickness will therefore be systematically smaller than that measured. For rough specimens you will have to judge whether this is significant and, if possible, estimate a correction. Sometimes better measurements and estimates of roughness can be achieved with smaller micrometer anvils.

Soft Specimens. Excessive force will compress or even damage the specimen so it may be necessary to abandon the use of the constant-force ratchet mechanism on the micrometer, which may be too heavy. Use a 'light finger-tight' approach instead. Experiments in another metrological field (the application of torque to RF & MW coaxial connectors) have shown that the human hand can achieve high levels of repeatability just from 'feel', so a little practice should help. Soft specimens often have less uniform thickness than hard specimens (typically about 10 - 30 μm variation), arising from difficulties in machining, etc., so the absolute uncertainties in thickness measurement, even though larger than for hard specimens, may not present the dominant error in the ε' measurement - they may arise instead from *thickness variation* across the surface instead. See Section 4.3.2 for a worked example.

6.3.2 Air-Gauging for Inner and Outer Diameter Measurements

Measurements of diameter are often important and are crucial to the achievement of low uncertainty in coaxial transmission line measurements, see Section 7.1.10. In this technique one is concerned with air-gaps between the specimen and the inner- and outer-coaxial conductors, so one not only has to measure the *outer*-diameter, o.d., of the annular specimen but also the inner-diameter, i.d., of the concentric hole through it by which it is fitted onto the *inner*-conductor (see Figure 37). Micrometers can readily be used for measuring o.d.s, but cannot normally be used for small i.d.s. Mechanical techniques may be used for this purpose, but it has been found that the use of *air-gauging* [122] is cost-effective and particularly convenient for the measurement of i.d.s and o.d.s *both* of annular specimens *and* the inner and outer-conductors of the coaxial line into which they are to fit. Indeed, whenever *specified* diameters, as opposed to *arbitrary* diameters, are to be measured, air-gauging is both accurate and convenient. In the coaxial transmission line method, i.d.s. and o.d.s should ideally be measured with a total uncertainty of ± 1 μm to enable gap corrections to be applied effectively. Air-gauging is able to achieve this – *provided that* (i) the specimens are more than 5 mm in length, (ii) their i.d.s are > ~ 1 mm. Otherwise other techniques must be used. Suitable air-gauging systems are available commercially and are convenient to use and may be cheaper in the long run than other methods. If air-gauging cannot be used, diameters may be measured by other suitable techniques, mechanical or optical, provided the uncertainty is at least as good as that required. For example 'expanding-ball' or 'plug' gauges are inexpensive and can deliver uncertainties of ± 0.01 mm or better for i.d.s.

6.4 Q-factor and its Measurement

Sub-sections 6.4.1 to 6.4.4 explain various aspects of the *theory* of resonator Q-factor measurement and show how they feed into *practical measurements*, which are discussed in Section 6.4.5. Background theory can be found in [7], [8] and [9]. Special note should also be taken of the book by Kajfez [123], which covers Q-factor measurement in much greater depth than is possible here.

Two contrasting approaches for Q-factor measurement will be considered here - note, though, that elements from both of them can be (and commonly are) combined in practical Q-factor measurements:

> (a) A *low-cost* approach, which commonly employs *manual* operation, *amplitude-only* measurement of the resonance using a *Scalar* network analyser (see Section 6.1.1) with a *Resonance-Width* (‘*3-dB point*’ or equivalent) determination of the Q-factor. For convenience, this is referred to as the *Manual Method* below.
>
> (b) An *automated* approach employing a *vector* ANA (see Section 6.1.1) in which a full *complex S-parameter theory* of the resonator is developed and in which the numerical analysis is used to *fit* a parameterised model of the resonator to the measured S-parameter data obtained for the resonance, Q-factor being one of the parameters that is fitted to the data. This is referred to as the *S-Parameter Method* below.

Both approaches have their place. The Sections that follow, however, explain the limitations of the Manual approach (a) and explain why it might be that one might prefer to use the more comprehensive S-parameter approach (b) in some measurements. A number of other approaches are also considered below.

One other very important aspect of Q-factor measurement is the design of the coupling ports of the resonator. The requirements here for transmission- and reflection-based Q-factor measurements are very different, and they are covered in the text below, notably in Section 6.4.3. See also Section 6.1.9.

6.4.1 Introduction: Resonances and Q-factor as a Measure of Loss.

The term *Quality Factor*, *Q*, commonly called *Q-factor* is readily defined for resonating systems in the frequency domain as follows:

$$Q = 2\pi \times \frac{\text{Energy stored in the resonance}}{\text{Energy lost per cycle}} \qquad \ldots (6.6)$$

In the electromagnetic resonators with which we are concerned here the ‘energy stored’ is the *electromagnetic energy* stored in the fields in the resonator, the ‘energy lost’ is the *electromagnetic energy* lost *by whatever means* from such storage and ‘per cycle’ refers to a cycle of the sinusoidal resonating electromagnetic signal at the frequency that is present in the resonator.

Q-factor quantifies the relative rate of loss of electromagnetic field energy from a cell, circuit, enclosure, cavity or resonator that is designed to store that energy. Place a lossy dielectric in such a cell and the Q-factor will fall. This principle forms the basis of many techniques for measuring the loss of dielectrics.

Introductory texts on electrical engineering ([9, 8, 6] also [123]) often use simple equivalent circuits to illustrate the concept of Q-factor and we can do this here by using the circuit in Figure 3 as a simple model of a resonance cell. The relevant Q-factor at resonance frequency f_r is $Q = 2\pi f_r L/R = 1/2\pi f_r CR$. The resonant frequency is readily shown to be $f_r = 1/2\pi\sqrt{LC}$ and the voltage across the capacitor V_c is related to the circuit input voltage, V_i by:

$$\frac{V_c}{V_i} = \frac{1}{(1 - f^2/f_r^2) + jf/Qf_r} \qquad \ldots (6.7)$$

When $f = f_r$, i.e. at resonance, we see that $V_c/V_i = -jQ$ and that the *magnitude* of this ratio is Q. This is significant in dielectric measurements because in such resonators it is the *capacitor* that contains the dielectric specimen under test. As the E-field in the capacitor and the specimen itself is increased by a factor Q, compared with a non-resonant measurement, we see that for high Q-factors the resonant technique can be far more sensitive to dielectric losses than the latter. This is the reason why resonance methods are usually to be preferred for low dielectric loss measurements. The Q-factor can be seen as a *Magnification Factor,* which increases the sensitivity of resonant techniques for the measurement of low dielectric losses.

Equivalent circuits can also show us how Q-factor is related to the width of the resonance in the frequency domain. Let us assume that we have a resonator with a high Q-factor – noting that the whole point of using resonant methods is to achieve a high Q-factor and hence a high sensitivity to dielectric loss. Take the input frequency to be $f_r + \Delta' f$, (the reason for the prime mark ‘′’ will become apparent below) where $\Delta' f \ll f_r$. On expanding and ignoring second-order terms, the denominator of Equation 6.7 becomes $(j(1 + \Delta' f/f_r)/Q - 2\Delta' f/f_r)$.

Now let $\Delta' f = f_r/2Q$ and expand the denominator. We may now also ignore a term proportional to $1/Q^2$, because it must be negligibly small, so we obtain:

$$\frac{V_c}{V_i} = \frac{Q}{(1+j)} \qquad \ldots (6.8)$$

Taking the modulus, we see that the voltage amplitude is reduced by a factor of $1/\sqrt{2}$ compared to that on resonance when the frequency differs from the resonant frequency by $\Delta' f = f_r/2Q$. Note that when the *voltage* is reduced in magnitude by $1/\sqrt{2}$, the *power* in the resonance is halved, so we have a 3-dB reduction in power. We obtain the same relationship if we *lower* the frequency below the resonant frequency, such that $\Delta' f = -f_r/2Q$ Now we will define Δf (Δ with no prime ‘′’) to be the *full* width of resonance between frequencies $f_r + f_r/2Q$ and $f_r - f_r/2Q$, see Figure 23. We obtain $Q = f_r/\Delta f$, which forms the basis for the traditional *Resonance Width* or *Full Width - Half Maximum* (*FWHM*) method for measuring Q-factor, otherwise known as the *3-dB Point* method.

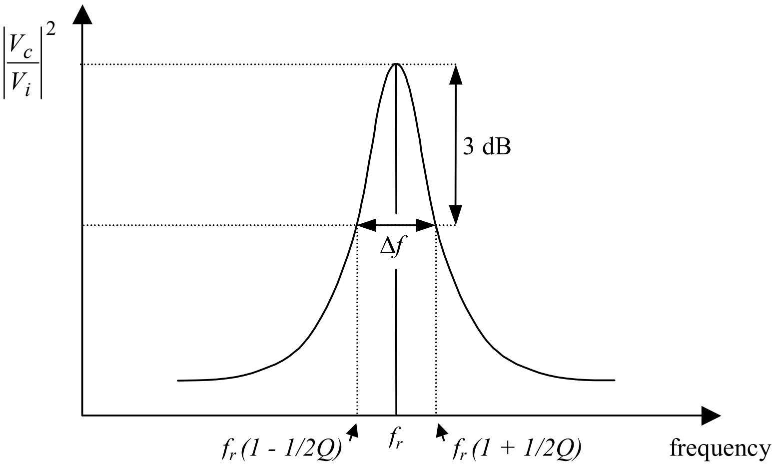


Figure 23. A typical resonance curve, see Equation 6.7, showing how the width of the curve at the ‘half power’ or ‘3dB’ points can be used to measure the Q-factor by the *Full Width - Half Maximum* (*FWHM*) method.

Such is the simple explanation of the classic method for Q-factor determination. But it by-passes a number of important points:

(1) We see that the 3-dB method is only approximate: it only applies if Q is large.

(2) The model is based upon an equivalent circuit in which only one resonance can occur. Typical standing-wave resonators (see Section 2.2) exhibit many resonances at different frequencies Some of these may overlap if their separation is closer together than about $f_r/2Q$ in frequency, where Q in this case is a typical Q-factor for the overlapping resonances. This will give rise to errors of measurement if we erroneously assume that only one resonance is present (see Section 6.1.9). The shape of the resonance can be used to check if other modes are interfering.

(3) We can see in Equation 6.8 from the presence of j in the denominator that the phase of V_c/V_i changes as we sweep the input frequency across the resonance. In using the 3-dB method we ignore this phase change - yet phase is readily measurable these days on instruments such as ANAs. Measurement of both phase and amplitude of a resonance can give us extra information which may help us to detect instrumental faults and to avoid multiple resonances, as discussed in (2), above.

We begin to see that the Resonance-Width method may not present our best approach to Q-factor measurement. A more comprehensive method, the full *S-parameter Method*, based upon measurement of both amplitude and phase of resonances is described in Section 6.4.2,

The 3-dB Resonance Width method for Q-factor has been so ubiquitous that it is sometimes tempting to think that Q is defined in terms of width of resonance. But in fact the concept of Q-factor is not restricted by its definition to single resonances at all. The concept also applies to the storage and loss of energy in multimodal and *mode-stirred chambers* (sometimes called Reverberating Chambers), which exhibit multiple overlapping resonances. These instruments have also been used for dielectric measurements, see the Review in [2].

6.4.2 Q-factor of Resonant Systems as Characterised by Complex S-Parameters.

A more complete theoretical description of the measurement of resonator Q-factor is based upon the determination of its S-parameters (S_{11} or S_{21}) – a measurement usually performed on *vector* ANAs these days (See Section 6.1.1.). If impedance or admittance measurement systems are being used, very similar considerations apply because, for example, Equation 2.9 readily relates reflection coefficient to impedance.

For practical reasons the mathematical model of a resonance based on a full complex S-parameter analysis should take into account one further factor that has not yet been mentioned: non-resonant *leakage* of signals directly from the input to output couplings of the resonator. This leakage may either be internal or external to the resonator, see Figure 24. In many practical resonator designs leakage is a significant factor, particularly when a lossy specimen is inserted into the resonator and the amplitude of the resonance is reduced to a value comparable with that of the leakage signal. It may appear that this is only a problem when the resonator is measured in *transmission*, not *reflection*, but this is not so because in reflection there is effectively a very large 'leakage' signal which is present all of the time - namely the mismatch of the resonator, which is normally so large that the resonator reflection coefficient magnitude, $|\Gamma|$, lies close to unity, $|\Gamma| \approx 1.0$, when the frequency is far away from any resonances. So then, we see that we should, in general, include a fixed leakage signal (or its equivalent) in our model of a resonator. This has the advantage that it also covers leakage at any point in the detection system.

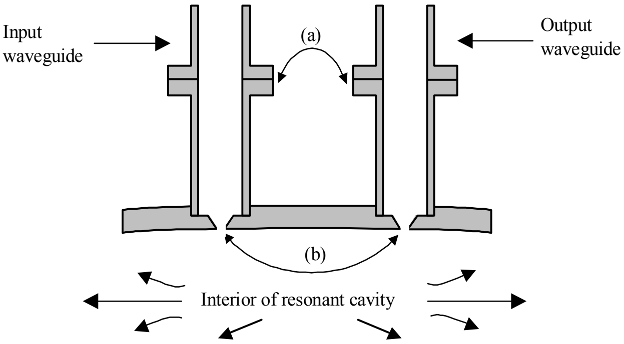


Figure 24. Signal leakage paths between the input and output ports of a resonator. Path (a) between waveguide flanges is *external* to the resonator and could potentially be avoided by use of screening or waveguide shims. Path (b) is directly between coupling holes *inside* the resonator and in some resonator designs this may be unavoidable – e.g. the open-resonator, Section 7.1.17, if input and output couplings have to be physically close.

This point can be clarified if we take Kajfez's general equation for the *reflection coefficient* of a resonator [123]:

$$\Gamma = \frac{jQ_l\Gamma_s t + \Gamma_s + de^{-2j\delta}}{1 + jQ_l t} = \Gamma_s + \frac{de^{-2j\delta}}{1 + jQ_l t} \qquad \ldots (6.9)$$

where $t = 2\dfrac{f - f_r}{f_r}$, Q_l is the 'loaded' Q-factor, f_r is the resonant frequency. Γ_s is the reflection coefficient of the resonator far from resonance, d is a parameter which governs the amplitude of the resonance and $e^{-2j\delta}$ is a phase factor which takes account of phase changes in the transmission lines and other arbitrary phase factors. This equation can be plotted on a polar chart for Γ, showing the response as the frequency is swept. Indeed such a chart can be displayed on an ANA screen, where it looks like Figure 25 (a).

Now when the *transmission coefficient* of a high Q-factor cavity is measured between two coupling ports the same generic equation arises:

$$T = \frac{jQ_l Lt + L + de^{-2j\delta}}{1 + jQ_l t} = L + \frac{de^{-2j\delta}}{1 + jQ_l t} \qquad \ldots (6.10)$$

where T is the transmission coefficient (which can be measured as S_{21} or S_{12}) and L accounts for a constant leakage between input and output ports. Note that, like Γ_s in Equation 6.9, L is a *complex* quantity commonly referred to as a *leakage vector*. Its effect can be seen by comparing Figures 25 (b) and (c). Usually the main difference between L in transmission measurements and Γ_s in reflection measurements is their relative magnitude. For the reason given above (i.e. the mismatch of the cavity), $|\Gamma|$ generally lies close to 1.0, however, resonators are usually designed to minimise L so that $|L| << |T|$ - though this is difficult to achieve with very lossy specimens.

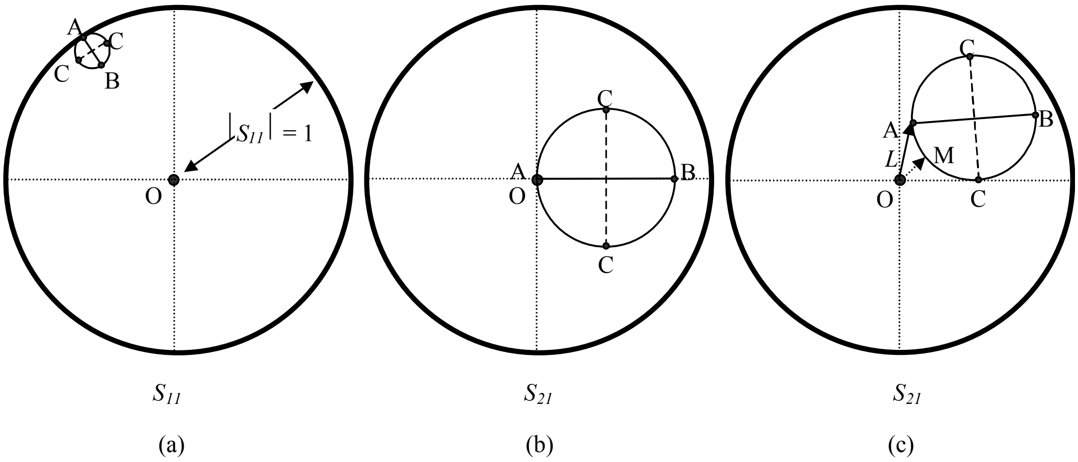


Figure 25. Polar plots of the reflection and transmission coefficients of a typical resonator, showing the circular traces (the small circles, which have diameter d, see Equations 6.9 and 6.10) obtained when the frequency is swept across a resonance. In each case the point 'O' is the centre of the polar chart, representing zero transmission or reflection coefficient. Point 'A' is the *pole* of the resonance i.e. the point reached on the chart both *well above* and *well below* the resonant frequency. 'B' is the *peak* of the resonance at frequency f_r. The two points marked 'C' are the two *3-dB points* which lie one quarter of the way around the resonance circle from 'A' and 'B'. **(a)** A typical trace obtained from a *reflection coefficient* measurement with sub-critical coupling – for critical coupling the circular trace would be larger, with $d = 1$, and 'B' would be coincident with 'O', i.e. the resonator would be matched on resonance. In (a) the circumference of the polar chart represents $|\Gamma| = 1$. **(b)** A plot of the *transmission coefficient* of a resonance showing the ideal case with no leakage. **(c)** A plot of the *transmission coefficient* showing a more typical case – here the *leakage signal*, L, between the input and output ports shifts the whole resonance circle on the chart, causing 'A' to move away from 'O'. The vector between 'O' and 'A' is referred to as the *leakage vector, L*, which can be measured by curve-fitting, see main text. For 'M', see the main text below.

In all three parts of Figure 25, the resonance traces out a circle on the complex S-parameter plane as the frequency is swept across the resonance. The actual diameter, d, of the circle depends upon the *coupling coefficient, β* (Section 6.4.3). In Figure 25 (a) *Critical Coupling* would occur if the reflection coefficient circle passed through the point 'O' at $|\Gamma| = 0$ in the centre of the chart on resonance. The positions of the resonance point, 'B', the 3-dB points, both marked 'C', and the 'pole' of the resonance, 'A' (i.e. that of the measured parameter far from resonance) are plotted on the resonance circles in all three diagrams and can be seen to be evenly distributed at 90° intervals around the resonance circle. Figure 25 (b) shows the transmission coefficient through a 'perfect' resonator with no leakage (L = 0). Figure 25 (c) shows a situation more typical of what is normally found in a practical resonator, though, to make the diagram clear, the magnitude of L is shown as being much larger than would be ideal. The phase of the axis of the resonance (the diameter between the points 'A' and 'B') is effectively *arbitrary* - it depends upon the length of the input air-line and the method of coupling - *unless* the ANA has been calibrated to make this phase zero. The phase of the fixed leakage vector L is also arbitrary and bears no necessary relation to the phase of the resonance axis. This situation can give rise to *apparently* distorted resonances in amplitude-only measurement methods which produce errors in Q-factor measurement if the 3-dB method (Section 6.4.1) is used and the leakage is ignored, see below. Note that as the frequency is swept uniformly across the resonance, the rate at which the S-parameter, traces out the circles in the figures is fastest at the resonance frequency at 'B' and falls away rapidly as the null point 'A' of the resonance is approached.

In practice the *leakage vector*, L, is almost certainly *not* fixed in either amplitude or phase if the frequency is swept, but the point to remember here is that *if* the Q-factor is high, the changes in frequency required to measure Q will be small and, unless the leakage is itself associated with a high-Q resonance, the changes in L with frequency will probably be negligibly small. (Warning: this is not true for *low Q* measurements). Of course, if L is caused by one or more unwanted but overlapping resonances, it will be difficult to isolate it in our measurements and we will have a measurement problem on our hands. As shown in Figure 24, L is generally associated with *direct non-resonant* coupling between input and output coupling apertures, particularly if they are physically close together, or else it may be caused by leakage outside the resonator.

The similarity of Equations 6.9 and 6.10 can be explained if we consider how the transmission coefficient S_{21} of a resonator is related to the reflection coefficient (or S_{11}) in any given resonator. In the special case of a resonator in which leakage in S_{21} is negligible, and the two coupling apertures are *identical in design* and *where they essentially sample the same E.M. field,* the answer is simple and interesting. A good example of this is the open-resonator with two coupling holes at the pole of its concave mirror, shown in Figure 51 (a), Section 7.1.17. *Relative to the input signal*, and with matched source and receiver, the signal coupled out of the output port is, by definition, S_{21}. But the input coupling port is *physically identical* to the output coupling port and is *sampling almost exactly the same field*; therefore *relative to the input signal* the signal that passes out of the input port is also ~ S_{21}. This is *added* to the mismatch signal from the outside of the input port, which we discussed above, and which has the property $|\Gamma_s| = |S_{11\text{-far-off- resonance}}| \approx 1.0$ and so we get $S_{11} \approx \exp(j\theta) + S_{21}$, where $\theta = -2\delta$ is the (arbitrary) phase of Γ_s. in Equation 6.9. Clearly, since $\exp(j\theta)$ is essentially constant in a high Q-factor measurement, S_{11} and S_{21} essentially contain the same information about Q. We may therefore justifiably ask which parameter we should prefer to measure. This is discussed at a number of points below.

Leakage and Apparently Distorted Resonances In a '*scalar*', i.e. *amplitude-only*, visual presentation of transmission coefficient, as measured, for example, on a spectrum analyser or an ANA in *amplitude* display mode, perfect leak-free resonances of the form shown in Figure 25 (b) will exhibit the well-known symmetrical bell-shaped 'Lorentzian' line shape shown in Figure 26 (a). At any given frequency, *f*, the amplitude of the resonance in Figure 26 (a) is given by the *length* of the vector from the centre of the polar chart in Figure 25 (b) to the point on the resonance circle corresponding to frequency *f*. This vector gives us the amplitude of the transmission coefficient of the resonator as a function of *f*. However, one often encounters resonances that appear to be *asymmetrically distorted* when viewed in a scalar amplitude-only format, as in Figure 26 (b). This is entirely explained by the presence of leakage, as can be seen with the aid of Figure 25 (c). As the frequency is swept, it sweeps the point 'M' around the resonance circle. We can see that the vector 'O'-'M' does not reach its maximum length at the peak of the resonance, and also that (as shown in Figure 26 (b)) in one part of the sweep it has a *lower* amplitude than points far away from resonance, i.e. those near to point 'A'! This phenomenon is one of the reasons for preferring a *vector* ANA measurement to a scalar measurement. It is also one of the reasons why the 3-dB method can lead to error. Automated Q-factor methods based upon the full complex formulae for the S-parameters of the resonator, as sketched above, can easily correct for constant or slowly varying leakage as explained in the next sub-section on automatic methods for Q-factor.

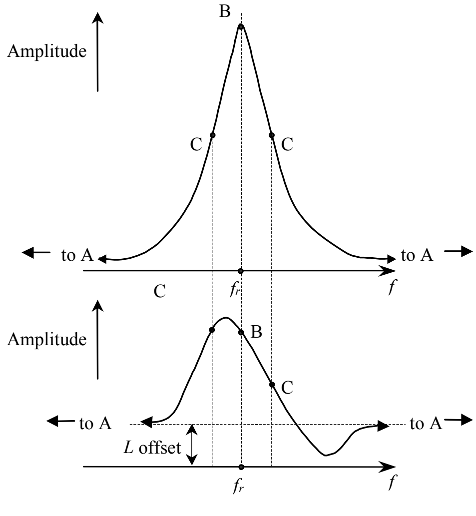


Figure 26. Amplitude-only transmission measurements of a resonance as a function of frequency. Points 'A', 'B' and 'C' correspond to those in Figure 25 (strictly, point 'A' lies at ± ∞ in this figure). **(a)** The perfect Lorentzian line shape obtained when there is no leakage in the resonator – this curve corresponds to the trace in Figure 25 (b). **(b)** This shows the distorted resonance line obtained when significant leakage is present – this curve corresponds to the trace in Figure 25 (c). The 'offset' has the amplitude L of the leakage vector. Taking the '3-dB points' to be 3-dB down from the peak of this measured curve could give rise to considerable errors in Q-factor measurement.

It has been shown that the mere *appearance* of distortion in an amplitude-only presentation of a resonance does not mean that it is *actually* distorted, it may simply be displaced on the complex S-parameter plane by a leakage vector, *L,* as discussed above. However, *genuine* distortions are possible too. They occur, for example, if there is *over-coupling* into a resonator or if resonances overlap or are degenerate (i.e. if they occur at almost exactly the same frequency). These situations are generally to be avoided in measurements, see Section 6.1.9.

Another potential cause of apparently distorted resonance line-shapes is the frequency domain line-width of the RF & MW signal source itself. This should be significantly narrower than that of the resonance to be measured – by a factor of at least 10, ideally more. See the discussion on source-stability and phase-noise in Section 6.4.5.

The S-Parameter Method – An Automatic Method for Measuring Q-factor based upon Numerical Data Fitting. In this method the full complex S-parameter equations for the resonance, Equations 6.9 or 6.10, should ideally be employed, though even data from a *scalar* ANA can be fitted to the *modulus* of these two equations. The best approach is to optimise the following parameters in a non-linear least-squares fitting process, so that the S-parameters predicted by the equations are optimally matched to those that are measured, see Kajfez [123].

(1) The resonant frequency.
(2) The amplitude and phase at the peak of the resonance.
(3) The Q-factor.
(4) For transmission measurements, a complex leakage signal (as described above), which may be independent of frequency, or have a phase change proportional to frequency. For reflection measurements a similar term must be included to capture the effect of the mismatch of the coupling.
(5) Where necessary a transmission line-length parameter should be included to compensate for the fact that the resonator coupling points are displaced along a transmission line from the ANA calibration planes. This will change the phase of the measured S-parameter linearly with frequency. For high Q-factor resonators (Q > 10,000) this correction is unlikely to be significant even for transmission line-lengths of up to a metre, but it will be significant for low Q-factor measurements.

The highest weighting in the fitting should be given to the data points closest to the centre of the resonance, point ‘B’ in Figure 25. Use of data beyond the ‘6 dB’ or ‘quarter-power’ points of the resonance is not recommended because of (i) their lack of sensitivity to Q-factor, (ii) the noise introduced by the lower signal-to-noise ratio and (iii) the greater effects of possible interference from nearby resonances. If another resonance *is* nearby in the frequency domain, it may be necessary to avoid its influence by using only data close to the centre of the resonance. It may be that in this region only the *phase-change* information can be trusted for determining Q-factor - the amplitude changes slowly near the resonance peak - and this is another reason why the full complex S-parameter method is often to be preferred. The weighting of the data points may be performed by any appropriate method. For example, closer frequency spacing can be used near to the resonance peak. If, on the other hand, frequencies are equally-spaced, as is usual on an ANA, the fitting equation must be modified to provide the weighting. The practical aspects of Q-factor measurement in relation to this S-parameter method are discussed in Section 6.4.5.

In spite of the general recommendation here to use a full vector curve-fitting method wherever possible, other techniques may prove adequate and more cost-effective in many circumstances. It is doubtful whether the full S-parameter fitting process produces a significant improvement in accuracy for perfect resonances i.e. those with no leakage or distortion – but it is a considerably more convenient method for Q-factor, is more flexible and is readily automated.

6.4.3 Coupling into Resonators: Q-factor Loading by Coupling Mechanisms.

The background theory here may be found in [8] and [9]. All coupling mechanisms load the Q-factor of resonators to some extent and in any given method, consideration should be given as to whether or not this significantly affects the measurement results. As mentioned in the Glossary and Section 6.1.9, the total Q-factor of a resonator may be analysed as the sum of contributions from all sources of loss in the resonator. Thus, if the sources of loss are s_1, s_2, etc. then we have $1/Q_{total} = 1/Q_{s1} + 1/Q_{s2}$ … etc. Now one such source of loss is the power coupled out of the resonator by both the output *and* input coupling mechanisms, see Figure 21. Whether such loss can introduce an error into the measurement of dielectric parameters, depends upon the measurement technique being used and the amplitude of the coupling coefficients, β_i and β_o, for the input and output couplings respectively. Equation 6.11 describes how the Q-factor is reduced by them [9]:

$$Q_l = \frac{Q_0}{1+\beta_o+\beta_i} \qquad \ldots (6.11)$$

where Q_l is the ‘loaded’ Q-factor, i.e. Q-factor reduced by the effect of the coupling and Q_0 is the unloaded Q-factor, which is the extrapolated value that the Q-factor would have if there were no coupling. Note that one can only ever measure *loaded* Q-factors. Now for a weakly coupled resonator measured in transmission it can be shown that $(\beta_o + \beta_i) = d/(1-d)$, where d is the diameter of the circles in Figures 25 (b) and (c) – i.e. the same parameter given in Equation 6.10 - and so Q_0, the parameter we want to calculate, is easily computed from the measured loaded Q-factor by the formula:

$$Q_0 = \frac{Q_l}{(1-d)} \qquad \text{... (6.12)}$$

This formula assumes that the resonator is weakly-coupled, and that the coupling factors are similar in magnitude [124, 125]. A simple worked example of this correction is given in Section 9.6. Similar corrections can be derived for reflectometric measurements. ***Important:*** Note that use of this formula to correct for the loading of coupling ports requires that the ANA that is being used for the measurements should be *fully calibrated*, otherwise d cannot be properly quantified.

Note that coupling factors do not just depend upon the size of the coupling mechanisms, they also depend upon the impedance of the external circuits that the cavity is coupled to. If the external source impedance or load impedance is low (e.g. connected to a 50-Ω match), the Q-factor will be loaded to a greater extent than if it were high (e.g. as it would be if the measurement cell were connected to a high input-impedance bridge).

It is not always necessary to apply Equation 6.12 to our measurements to calculate the specimen loss accurately if one is using a *substitution measurement technique* (Section 4.4.1). Many traditional resonator methods such as those described in Sections 7.1.4 - 7 and 7.1.16 - 17 follow this principle. The measurement is performed in such a way that the loss through the coupling apertures *is the same* when the specimen is in the cavity and when it is out. One has:

$$\frac{1}{Q_{specimen\ out}} = \frac{1}{Q_{resonator}} + \frac{1}{Q_{coupling}} + \cdots, \qquad \text{... (6.13)}$$
$$\frac{1}{Q_{specimen\ in}} = \frac{1}{Q_{resonator}} + \frac{1}{Q_{coupling}} + \frac{1}{Q_{dielectric}} + \cdots$$

Clearly the Q-factor of the specimen (from which tan δ is computed) can easily be derived by subtracting these two equations - the left hand sides of which are *measured* quantities, while the loss of the coupling mechanisms cancels out. This approach cannot be used so simply, however, in other methods - for example the measurement of dielectric resonators, Section 7.1.15. A substitution technique cannot be used in this case because without the dielectric resonator there is no resonance (or at least, no resonance at a nearby frequency) so $Q_{specimen\ out}$ is strictly-speaking *not defined* and Equation 6.12 must be used to correct for the effects of coupling if they are not to lead to errors in measured tan δ.

Section 6.1.9 introduced the question of the value of *coupling coefficient* that ought to be used. We can revisit this question in the light of the above discussion. In the case of *transmission measurements* the answer is simple: use the *lowest* coupling coefficient value that does not compromise accuracy through resolution and signal-to-noise limitations. Remember that a high coupling-coefficient brings with it significant perturbations to the fields inside the resonator and they might falsify the measurement equations. It also lowers the Q-factor, thereby reducing the sensitivity of the measurement. Furthermore, in Equation 6.11, the higher β_o and β_i are, the more they couple the resonator to the 'outside world', i.e. to the rest of the measurement system. This increases the sensitivity of the measurement to unwanted changes *outside* the resonator – a source of measurement error. It is often possible to use resonator insertion losses as high as 50 dB (i.e. amounting to about 25 dB per coupling port) *or more* to measure Q-factor. Such high insertion losses may be recommended for minimum perturbation to the resonator. Remember that detector resolution and signal-to-noise ratio can generally be improved by the use of an amplifier and by automatic averaging in ANAs.

The case of *reflection measurements* is somewhat different. If the coupling used were as low as just mentioned (i.e ~ 25 dB for one coupling port) one would be forced to measure a small signal (the resonance) in the presence of a large one (the mismatch of the resonator) - which is generally regarded as poor metrological practice. One therefore tends to use larger coupling factors for reflection-coefficient Q-factor measurement. A natural consequence of this is that Q_l becomes significantly smaller than Q_0, so the loading of the coupling must certainly be accounted for. This requires use of a *fully calibrated* ANA. Kwok & Liang describe one method for performing these measurements using a calibrated *scalar* ANA [126]. Another approach is to employ *critical coupling* for which β is equal to 1, and the loaded Q-factor is half that of the unloaded Q-factor (see [123], pp 41, 58). This is also the condition for the resonance circle to pass through the centre of the S_{11} polar-chart, so that $|S_{11}| = 0.0$ on resonance. In this method one can adjust the degree of coupling specifically so that the condition $|S_{11}| = 0.0$ is achieved on resonance, then one knows that $\beta = 1$ and from Equation 6.11 (simplified for one coupling port) $Q_l = Q_0/2$, so one does not have to take any further steps to determine the effect of coupling.

Whether reflection methods are to be preferred to the transmission methods must depend upon local circumstances. If one only has a reflectometer to hand and no means of measuring transmission coefficient, then clearly only the reflection method can be used! Yet for the reasons given above it suffers from a number of faults: field-perturbation, lower loaded Q-factor, and greater interference from the system outside the resonator, to which may be added complexity of the adjustment to achieve the condition $|S_{11}| = 0.0$ if the *critical coupling* method is used. Compared with these difficulties the transmission method is easier to use, though it generally requires more expensive detection equipment.

Warning: There is a degree of ambiguity in the use of the term 'loaded'. It is used both (i) to indicate the effect of *coupling* upon Q-factor *and* (ii) to indicate the effect upon Q-factor when the *specimen* is inserted into the resonator. 'Loading' in this second sense is discussed in Section 6.4.4.

6.4.4 Loading of Resonators by Dielectric Specimens.

As just mentioned above, a resonator is said to be 'loaded' by the specimen when the specimen is inserted into it. Thus Equation 6.3a of Section 6.1.9 may be transformed into:

$$\frac{1}{Q_{specimen\ in}} = F_f \tan\delta + \frac{1}{Q_R} \qquad \ldots (6.14)$$

Where F_f is the filling-factor of the specimen and Q_R accounts for *all* other losses in the resonator (e.g. metal loss, coupling loss, etc). In practical measurements, how should Q_R and F_f be related to the expected value of $\tan\delta$ to be measured? i.e. just how much should the specimen be allowed to *load* the resonator? It appears, after surveying the literature on dielectric measurements in resonators, that, provided *adequate resolution* for $\tan\delta$ can be obtained, almost any regime can be valid. One can pick out four such regimes for special mention, all four have been used successfully for many years:

(i) $1/Q_R > \tan\delta$ or even $1/Q_R >> \tan\delta$, but $F_f \approx 1$. This regime is typical of RF resonators based on inductor coils (Section 7.1.4) and of RF re-entrant cavities (Section 7.1.5). One does not usually *want* to work with low Q-factor resonators but one generally has no choice in the matter at low RF because metal losses are so high. In capacitor/inductor-based resonators F_f is naturally close to 1 and F_f is typically ~ 0.95 in re-entrant cavities. Note that one would not want F_f to be lower in this regime because even more sensitivity would be lost if this were so. In spite of the difficulties, experience shows that although Q_R may be as low as 100, loss tangents as low as 10^{-5} can be measured by using the amplitude method for small ΔQ that is described in Section 6.4..5.

(ii) $1/Q_R < \tan\delta$ or $1/Q_R \approx \tan\delta$ and $F_f \approx 1$. This regime applies to $TE_{01\delta}$-mode dielectric resonators, see Section 7.1.15 [127].

(iii) $1/Q_R < \tan\delta$ or $1/Q_R \approx \tan\delta$ and $F_f < 1$. This regime is usually found in methods where the specimen only fills part of the resonator and in which the resonant frequency can be adjusted by tuning the length of the resonator. Examples are the TE_{01}-mode cavity and the open-resonator, see Sections 7.1.7 and 7.1.17, respectively. Tunability can offer a number of advantages, e.g. it may allow one to check on systematic errors, as discussed at a number of points in Section 7.

(iv) $1/Q_R < \tan\delta$ or $1/Q_R \approx \tan\delta$ and $F_f << 1$. This is the regime followed in resonator perturbation methods, the advantages of which, particularly for small, high-loss specimens, are discussed in Section 7.1.16.

In relation to (iv) we can note that resonators can be *too* sensitive for the perturbation method of Section 7.1.16 if filling-factors are too high. In the case of high-loss specimens, e.g. $\tan\delta > 0.3$, for example, the loading would be much too high if F_f were ≈ 1.0. That is one reason *small* specimens are used in perturbation methods. Clearly, in all cases, one needs a sensitivity that is *appropriate* for the materials to be measured. A sensitivity that is too low will not give us the information we need, but a sensitivity that is too high could render the approximations used in measurements invalid or else make the loaded Q-factor so low that it cannot be effectively measured. In designing or choosing a measurement system for a particular type of dielectric material one clearly must take these issues into account.

6.4.5 Practical Measurement of Q-factor

A number of practical issues are discussed below to help to improve uncertainties and to help in the choice of the best method to be used for any particular measurement application. Where it is not explicitly given below, the rationale for the advice given is to be found in Sections 6.1.9 and 6.4.1 – 6.4.4.

RF & Microwave Signal Sources: Frequency Stability and Phase-Noise. It is important to ensure that the signal source that one is using to perform Q-factor measurements is stable and that it exhibits low phase-noise. This can be checked by viewing its signal in the frequency domain on a good quality spectrum analyser. The signal can be checked both straight from the source (but first ensure that it is not too powerful for the spectrum analyser input!) and also by viewing the signal that emerges from the cavity/resonator as the frequency is swept over the resonance. The spectrum should be stable and *its spectral line-width should be narrow compared with the resonance line-width to be measured*. Most modern synthesised signal sources, of the sort that are used with ANAs, are suitable for measuring the typical Q-factors that are encountered in RF & MW cavities, but it should be remembered that as the frequency rises towards the millimetre-wave region, the Q-factors of the resonators used for dielectric measurements typically get higher, whereas the phase-noise of the signal sources typically gets worse! For example a Q-factor of 2×10^5 is easily attainable at 100 GHz in an open-resonator (Section 7.1.17), while measurements in superconducting resonators at cryogenic temperatures can easily produce Q-factors in excess of $1{\times}10^{9}$. Some types of source may be unsuitable for these measurements, e.g. older-model synthesizers, which produce higher phase-noise, or certain solid-state sources like Impatt Diodes. Phase-locked Gunn diodes or klystrons ought to be ‘clean’ enough to perform the kind of Q-factor measurements discussed here, however. If the source line-width is too broad, erroneous measurements of Q-factor will be obtained.

Q-factors Values In RF & MW dielectric measurements at ambient temperatures, *unloaded* Q-factors of measurement cells (i.e. resonator Q-factors *without* the specimen in the cell) can lie anywhere in the range 10^2 to 10^6. In the RF region of the spectrum, the metal loss (i.e. the conductivity loss in the internal metal walls of the cell, Section 6.1.9) usually limits Q-factors to a few thousand, or less. In fact, in techniques where coil inductors are resonated with the capacitance of the measurement cell (see Section 7.1.4) Q-factors as low as 100 can be used to advantage, even for measurements on tan δ as low as 10^{-5}. This can best be achieved by using the *amplitude-change* (‘magnification factor’) method for Q-factor discussed in the section on the measurement of small changes in Q-factor below. As the frequency rises the effects of metal-loss fall, so unloaded cell Q-factors above 10^4 are readily available in the microwave region, while an unloaded open-resonator Q-factor of above 10^5 is easily achieved at millimetre-wave frequencies. Clearly, better resolution for low loss is achieved with these higher Q-factors. *Loaded* Q-factors will, of course, depend upon the dielectric loss of the specimen and upon the cell’s filling-factor (see Sections 6.1.9 and 6.4.4). With lossy specimens the loaded and unloaded Q-factors may differ by a factor of over 10 and so it is sometimes advantageous to use two different Q-factor measurement methods to achieve best uncertainties.

Increasing Resonator Q-factor For high sensitivity in loss measurement it is usually desirable to use as high a product of Q-factor and filling-factor as possible and so the adoption of methods to improve Q-factor is to be recommended. Higher Q-factor improves resolution for low loss and also reduces systematic errors caused by loss in the measurement cell. See Section 6.1.9 for details of how to increase Q-factor.

Reflection or Transmission? See Sections 6.4.2 and 6.4.3. In some areas of dielectric metrology (e.g. the characterisation of dielectric resonators) the reflection method seems to have become the norm, but one should always ask, before embarking on a programme of measurements, whether it is necessarily the best method. There are, of course, sometimes instrumental considerations that require us to use a reflection method. For example, if the measurement cell is situated in a Dewar or furnace we may wish to minimise heat conduction out of the cell by using only one input/output line. Alternatively we may have only a reflectometer to hand and so cannot implement a transmission method, or else we may simply decide that reflection measurements are cheaper. However, the reflection method does have three disadvantages compared with the transmission measurement of Q-factor. First, (see Section 6.4.3) higher coupling factors usually have to be used, so the making of corrections for resonator loading is generally essential – and we generally need to know what the coupling factor is. By contrast, in transmission methods (especially in substitution methods) the loading of the coupling may have negligible effect on the dielectric-loss measurement. Secondly, a higher coupling factor perturbs the fields in the resonator to a greater extent – leading potentially to systematic errors. Thirdly, it has been found that resonances viewed in reflection tend to show greater distortion than those viewed in transmission. This is probably caused by the fact that the frequency dependence of the mismatch at the coupling point has a first-order effect upon the reflection coefficient but usually only a second-order effect on the transmission coefficient. The main extra cost of using the transmission method may often be the need to use an amplifier to recover signal amplitude if the resonator insertion loss is high. Insertion losses on resonance as high as 60 dB or more are used for minimal field-perturbation in resonators. So if an ANA is being used for detection with such a high insertion loss it may be necessary to use an amplifier to pull the detected signal out of the noise. This should be a low-noise, low-power amplifier to make sure that the signal power entering the ANA does not accidentally exceed the ANA's input power specification. In manual Q-factor measurements, sensitivity can be enhanced by use of Phase Sensitive Detectors (PSDs).

An Automated or a Manual Method? Clearly a cost-benefit exercise should be undertaken before deciding, but note that measurement of Q-factor by automated methods has three advantages: (i) they can readily correct for leakage, (ii) the averaging regimes they use to minimise effects of noise can be more flexible, (iii) they are less time-consuming and far less tedious to implement! Automated methods are, in general, therefore, to be preferred to manual methods. Of the automatic techniques available, the use of a full *vector* ANA is potentially more accurate and more informative – see Section 6.4.2, Equation 6.10 - as it makes use of all of the information available: i.e. full complex S-parameter data - both amplitude and phase. Note that in reflection measurements it may not be clear from the response of a *scalar* ANA whether the resonator is *under*- or *over*-coupled – a special test must be performed to check for this - but on a *vector* ANA the degree of coupling is immediately apparent on inspection of the polar response of the ANA as in Figure 25 (a). In spite of the general recommendation supporting full vector curve-fitting methods wherever possible, manual techniques do prove adequate in many circumstances. With a high-Q-factor resonance, and with good signal-to-noise ratio and no leakage or distortion, manual methods can be just as effective as automated methods. This is because in such ideal measurement circumstances the dominant uncertainty sources for Q-factor usually lie elsewhere – in temperature drifts, for example, or in the repeatability of measurement results when the specimen is placed in and removed from the resonator a number of times.

Traceability and Detector Linearity Whichever method is used, equipment should be adequately calibrated and traceable for all of the relevant measurement parameters. Q-factor measurement normally relies upon attenuation measurement, and in the case of vector-ANA measurements, upon phase measurement also. All attenuators employed should be calibrated and should be traceable to international standards. Non-linearity in a detection system gives rise to errors in measured Q-factor, so non-linearity should be checked for by the use of a calibrated attenuator from time to time. Alternatively, the *Attenuator Series Substitution* method should be used for measurement, which actually incorporates a calibrated attenuator into the measurement system, as described below. ANAs should likewise be checked from time to time for linearity of amplitude measurement by the use of an external calibrated attenuator and for phase measurement by measuring phase through a number of well-matched transmission lines of known length. Note that the linearity range required for any one Q-factor measurement is quite small (one is unlikely to require a range of more than 7 dB). However the detection system itself may have to work over a much wider dynamic range if a large range of Q-factors is to be measured. Remember that detector linearity problems are most likely to occur at high power levels: detectors can under-read when high power levels are measured (an effect known as gain compression).

Automated Measurements on an ANA (or equivalent) Background details are given in 6.4.3, but here are a few practical points to remember:

(1) A polar display of the complex S-parameter data on the vector ANA may be much more informative than an amplitude-only display: e.g. leakage signals can readily be seen on such a display, as in Figure 25 (c).

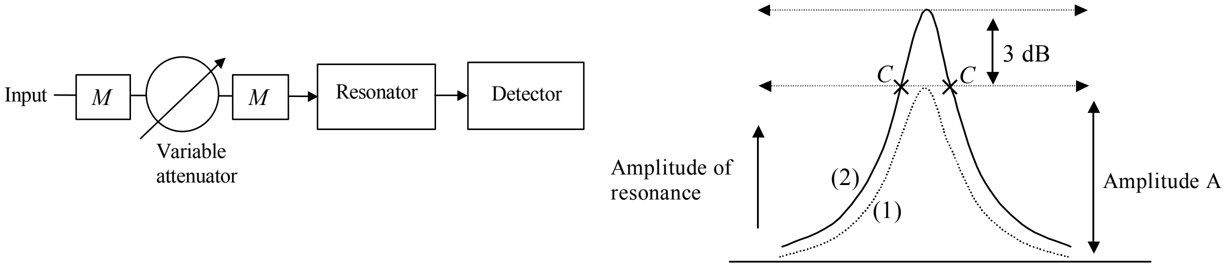
(2) Thought should be given to the optimum degree of automatic averaging in such measurements. Better noise immunity should be weighed against longer measurement times, during which the resonator S-parameters may drift in value, for instance, because of temperature changes.

(3) In full vector complex S-parameter measurements the phase changes in long transmission lines leading to the resonator should be calibrated out, otherwise the fitting of the phase of the resonance will be erroneous. In other words, the ANA measurement plane(s) should be situated exactly on the coupling port(s) to the cavity, this is readily achieved by using vector ANA software.

Manual 'Resonance Width', 3-dB and Related Methods for Q-factor. The *Resonance-Width* method is otherwise known as the *Full Width - Half Maximum* (FWHM) or the *3-dB point* method, see Section 6.4.1, Equation 6.8. The method has been widely used for many years with many different types of source and detector. If a source of adjustable frequency and constant power output, and a voltage or power detector are available a simple measurement system can readily be constructed. If a weak transmission response is to be measured a spectrum analyser can be used as a detector, or a phase-sensitive detection (PSD) system can be used. ANAs are, however, ideally suited - no other instrumentation being required. Either scalar or vector ANAs can be used for this method. Three procedures may be followed in the resonance-width technique to improve its accuracy:

(1) Because of the potential errors discussed above, e.g. from leakage and non-linearity, it is recommended that Q-factor measurement by this method be checked from time-to time by measuring the resonance width at lower or higher attenuation levels compared with the peak amplitude as well as at the 3-dB points, e.g. measure also at the 6-dB or the 1-dB points.

(2) It is recommended that an *Attenuator Series Substitution* method be used, Figure 27 (a), in which a calibrated adjustable attenuator is placed between the RF source and the resonator. This may be a stepped attenuator or any other attenuator (e.g. a rotary vane attenuator) that can adequately reproduce a set of attenuation steps (e.g. 1 dB, 3 dB, 6 dB, etc). A matching component should be placed on either side of the attenuator, otherwise its attenuation steps will be uncalibrated because of mismatch errors [119]. In the 3-dB transmission method, the attenuator is at first set to 3 dB, the resonance is peaked and the peak amplitude, *A*, is recorded. Sweeping the frequency will produce Trace (1) in Figure 27 (b). The attenuator is reset to 0 dB to obtain Trace (2) in Figure 27 (b) and the frequency of the source is tuned in turn to each of the two 3-dB points, marked as 'C' in the figure, which both occur when the detected amplitude is once again *A*. A similar method applies for all other attenuation steps. If such a method is *not* used, the detector itself must be checked for adequate linearity across the attenuation range employed.



(a) (b)

Figure 27. The *Attenuator Series Substitution Method* for determining Q-factor by means of amplitude measurements. **(a)** The measurement configuration. The two components marked '*M*' are matching elements (e.g. isolators or attenuators). They are included in the circuit to ensure that the attenuation of the calibrated variable attenuator is correct. **(b)** The measured amplitude as a function of frequency. Trace (1) is obtained with 3 dB of attenuation switched into the attenuator. Trace (2) is obtained when it is switched out. The points marked '*C*' are the 3-dB points.

(3) Temperature drifts cause drifts of resonant frequency, f_r. It takes a finite amount of time to measure the frequency of one 3-dB point and then the other in sequence. During this time f_r can drift, making it appear that the resonance width is either wider or narrower than it actually is. Therefore the frequencies of the 3-dB points should always be measured a number of times, alternating between the higher and lower 3-dB points so as to detect and correct for the drift.

A Warning: '3-dB Points' in Reflectometric Measurements of Q-factor. **IMPORTANT**: Measured '3-dB points' correspond to the half-power points of a resonance in *transmission* measurements but they do *not* in general do so in reflection measurements. In the latter the relationship between detected reflected signal and the power in the resonator is more complicated than for transmission measurements – the equations are given in Kwok and Liang [126]. As shown in Figure 25 (a), maximum reflection (usually with $|\Gamma| \approx 1.0$, see Section 6.4.2) is obtained at the *pole* of the resonance, 'A' in the figure, at a frequency far away from resonance frequency, and it is simply a mistake to assume that the 3-dB points on the resonance curve occur when the reflected signal is 3 dB below maximum reflection.

Measurement of Small Changes in Q-factor In substitution measurements for the loss of a specimen, we measure the Q-factor of the resonator with and without the specimen, so we are interested in measuring *small changes* in Q-factor, ΔQ, accurately. For low-loss specimens, these changes can be very small. The methods described above for Q-factor – curve-fitting and the 3-dB point method – are ideal for measuring *absolute* Q-factor accurately, but for measuring small Q-factor *changes* it is sometimes *not* the best policy to carry out two measurements of this type consecutively with and without the specimen. Like all measurements, Q-factor measurements are subject to drift and repeatability errors and it may be that the small change in Q-factor we are trying to measure are lost in the repeatability errors of the absolute Q-factor measurements. In such cases it is sometimes better to determine ΔQ by measuring the change of the resonance amplitude, ΔA, with and without the specimen. As discussed in Section 6.4.1, the resonance amplitude is proportional to Q so we have:

$$\Delta Q = Q_0 \Delta A / A_0 \qquad \ldots 6.15$$

where ΔA and A_0 are measured linearly in terms of voltage, *not* power, and A_0 and Q_0 are respectively the resonance amplitude and Q-factor measured without the specimen in the resonator. The full substitution measurements then consists of (i) a measurement of Q_0 by whatever absolute method is deemed to be best, followed (ii) by a measurement of ΔQ by the amplitude change method sketched here. In any particular system, an auxiliary experiment ought to be performed to establish whether this method is better than measuring the absolute value of Q-factor twice in succession (with and without specimen). ANAs normally compensate for source power variations automatically, but if one is not using an ANA, one should also monitor the power stability of the source.

Other methods of Q-factor measurement. These may be appropriate if they can be shown to produce comparable or better uncertainties than those recommended above. Methods include:

(i) Determination of Q-factor by measuring only the phase change of the S-parameter concerned. This method should to be based upon equations such as 6.9 or 6.10. If the Q-factor is very low or if there is a nearby or near-degenerate resonance, it is sometimes better to use this method. The phase changes should be measured only very close to the resonance frequency, as it is here that the rate of change of phase with frequency is greatest and the sensitivity of the method is highest.

(ii) Measurement of Q-factor by changing the *length* of a resonator. A *change-of-length* method may be preferable to a *change-of-frequency* method for low Q-factors if the frequency tuning range of the source is limited. Note also that the Q-factors of unloaded resonators itself changes with frequency, and systematic errors associated with this effect can only be avoided if all measurements are performed at the same frequency. This is usually only significant for low Q-factors.

(iii) Measurement in the time domain, e.g. by rate of decay of a resonant oscillation. Such methods can be useful for very high Q-factors ($Q > 10^6$) such as are found in superconducting resonators [128]

Sources of Uncertainty. Whichever measurement method is used for Q-factor, experience has shown that without significant effort *any* method is unlikely to deliver unloaded Q-factors with uncertainties much less than ± 1% (at a 95% confidence level). The dominant sources of uncertainty may be any of the following:

(i) *Drift* of the resonant frequency – usually caused by temperature changes or temperature cycling in temperature-controlled systems and laboratories. Note that rates of change of temperature can be reduced by passive temperature control – see Section 6.1.8.

(ii) *Phase-noise* on the measured signal can sometimes be quite high, especially at millimetre-wave frequencies. This may be caused by 'microphonics' in the detection system and so can sometimes be reduced by preventing critical equipment from vibrating. If phase-noise is high, it may sometimes be necessary to abandon full vector Q-factor measurement and to rely upon an amplitude only technique for Q-factor. See also the discussion on phase-noise at the start of Section 6.4.5

(iii) *Phase changes in coupling leads*. As the measurement frequency is swept to measure Q, the phase of the transmission coefficient in coupling leads changes. This effect was discussed above. In full vector S-parameter measurements on an ANA, one should calibrate these phase changes out or at least use short lines and establish, by auxiliary experiments, that their effects are negligible.

(iv) *Distorted and overlapping resonances.* These have been discussed above in Sections 6.1.9 and 6.4.2. *Always* check that your measurements are not being affected by distortion and nearby resonances.

(v) *Detector linearity*. Check from time to time that your detectors are linear (even those in ANAs!). Use *calibrated* attenuators to check linearity or else use such an attenuator as part of the measurement process, as in the *attenuator series substitution* method discussed above.

Postscript: A Useful Parameter Metrologists are most often concerned with the reciprocal, $1/Q$, of Q-factors, see for example Equation 6.14, rather than their absolute value. Q is an inconvenient, not to say ungainly, parameter to have in one's measurement equations. For low-loss measurements a practice has been introduced by Dr A C Lynch of defining a parameter $D = 10^6/Q$. This enables loss in resonators to be readily expressed in a manner that is commensurate with the use of loss angles measured in microradians (or loss tangents measured in units of 10^{-6}). This proves to be more helpful in the understanding loss processes than the use of Q-factor itself. However, the use of Q-factor is so generally ubiquitous and familiar that we have decided to use it throughout this Guide.

6.5 The 'Equivalent-Thickness' Approach

It is well worthwhile giving special mention to this method, which can sometimes be used when others cannot, or else when they would deliver significantly greater uncertainties. It is well-suited to the measurement of the real permittivity of laminar specimens and it is a *null* or *substitution method*, as described in Section 4.4.1, in which an 'equivalent' thickness of air, t_a, is substituted for the specimen, of thickness t_s, in such a way as to leave a measured parameter (typically capacitance) unchanged.

The simplest example is the measurement of permittivity in a micrometer-driven parallel-electrode admittance cell, see Sections 4.3.2, 7.1.1 and 7.1.3, and Figure 14. In the alternative, conventional *capacitance method*, see Section 7.1.1, we would place the specimen in the cell, adjusting it so that the electrode just touches both specimen faces. Then, for electrodes of area A, we would measure capacitance $C_i = \varepsilon'\varepsilon_0 A/t_s$ with the dielectric specimen present. Then we would measure the air capacitance $C_a = \varepsilon_0 A/t_s$, after removing the specimen from the cell but *without re-adjusting the electrode spacing* (for simplicity here we ignore the effects of fringing-fields and air-gaps and we assume that the permittivity of air is 1.0). It is then easy to calculate the specimen permittivity as $\varepsilon' = C_i/C_a$. In the *equivalent-thickness* approach, we measure the capacitance, C_i, of the cell as before with the specimen present. Again, $C_i = \varepsilon'\varepsilon_0 A/t_s$, but this time, on removing the specimen, we re-adjust the electrode spacing, reducing the gap between the electrodes, until *we once more measure the same capacitance C_i between the electrodes*. We can use the reading on the micrometer to give us the thickness of the *equivalent air capacitor*, t_a. This is the thickness of air that has the same capacitance as thickness t_s of dielectric. We now compute the permittivity from $\varepsilon' = t_s/t_a$.

What is the point in doing this? It would seem to involve us in extra work! Well, as noted in Section 4.4.1, one advantage is that we don't have to trust to the calibration of our capacitance bridge. More importantly, it is found that for lower permittivity materials (say $\varepsilon' < 5$) this method is often more accurate than the conventional 'capacitance' method and it fits very neatly with 'air-gap' methods (see Sections 7.1.2 and 7.1.3) in which the upper electrode does not touch the specimen. The advantage of air-gap methods is that neither the cell nor the specimen are physically distorted by the pressure of the electrodes upon the specimen – an important requirement when soft specimens are being measured.

The equivalent-thickness method is particularly appropriate in resonance techniques, such as those discussed in 7.1.4, 7.1.7 and 7.1.17. By changing the length of the resonator (or of a capacitance gap within it) the resonator can be measured *on resonance* both empty and loaded (i.e. *with* the specimen in it) *at exactly the same frequency* so that many losses in the resonator (e.g. metal-wall losses, coupling losses) barely change between the specimen-in and specimen-out conditions, thereby reducing systematic measurement errors.

As pointed out in Section 2.2.1 we can divide resonant methods into two classes: those (largely RF methods) based upon lumped-impedance approaches and those (largely microwave methods) based upon standing-wave methods. In the former, one is generally concerned with measurement of capacitances and conductances. The equations given above are characteristic of the equivalent-thickness approach in such measurements. However, in travelling-wave methods the relationship between permittivity and dimensions, is different. In a totally matched system, equivalent thickness will vary inversely with $\sqrt{\varepsilon'}$ rather than ε'. But resonators (e.g. Section 7.1.7 and 7.1.17) are *not* matched (typically they have a 'short-circuit' at each end) so the relationship is even more complicated since it must take account of standing-waves in the system. Nevertheless in resonators like TE_{01}-mode cavities and open-resonators, the relationship between ε' and equivalent-thickness is explicit and so the method can be used with all of its usual benefits, with the help of computer analysis.

Equivalent-thickness methods, and their analogues, can fruitfully be applied in a number of other techniques, e.g. the transmission line method of 7.1.10, but always, and only, if they provide metrological benefits through improved uncertainties.

6.6 Electromagnetic Fields and Specimens

6.6.1 Field Geometry and Specimens

Section 2.2.2 introduced the distinction between the *lumped-impedance* and *wave* approaches to dielectric measurement. One of the most significant differences between them is the *direction* and/or *polarisation* of the E-field in the dielectric specimen. Let us take measurements upon laminar specimens as an example. In lumped-impedance methods one is usually measuring the admittance (capacitance and conductance) *through* the specimen from face to face, so the E-field is *perpendicular* to the faces, whereas in travelling- and standing-wave techniques the E-field is usually transverse to the direction of wave propagation through the specimen and so is also transverse to the specimen, *parallel* its the faces. In other words, in lumped-impedance methods the E-field is perpendicular to the face of the specimen, whereas in wave methods it generally lies in the plane of the specimen. This is an important consideration if the specimen is anisotropic (See Section 5.3.11).

In dielectric metrology one typically uses lumped-impedance methods and their extensions at frequencies below 1 GHz and wave methods in the microwave range above that. If the specimen *is* anisotropic and if it is being measured across the spectrum, it can therefore sometimes appear that its permittivity has a step in its value in this region of the spectrum!

If one is *absolutely* sure that the material is completely isotropic these considerations may be unimportant, but bear in mind that with unknown specimens and materials one is usually *unsure* of this. Note that:

(i) Polymers can have ‘frozen-in’ stress giving rise to anisotropy

(ii) Ceramics may have microstructural physical alignments within them associated with the method with which they are manufactured, see Section 5.3.7.

(iii) Many crystals and structured materials have *intrinsic* anisotropy [99].

(iv) ‘Thin’ specimens and laminas often have a different permittivity in a direction perpendicular to the plane of the specimen from that in the plane of the specimen because of the way they were manufactured.

One has to use one’s judgment, based on knowledge of the material and on one’s desired uncertainty, to decide whether these considerations are likely to be significant in any given case. If in doubt, *test for* these effects.

The linearly-polarised open-resonator (Section 7.1.17) can measure anisotropy in the plane of a specimen, whereas many cylindrical cells that support TE-modes, e.g the TE_{01}-mode cavity (Section 7.1.7) and conventional $TE_{01\delta}$-mode dielectric resonators (Section 7.1.15) are circularly polarised and so cannot permit such measurements. See further details on anisotropic materials in Section 5.3.11.

6.6.2 E.M.-field considerations for optimising sensitivity and reducing systematic errors.

There are (at least) two important principles to follow here (though these comments do not necessarily apply to the resonator perturbation method described in Section 7.1.16). Where possible:

(1) Try to make sure that there are *strong* fields in the part of the cell that the specimen is placed.

(2) Try to make sure there are *weak* fields elsewhere (if the physics of the cell allows this) and especially in regions of the cell that contain elements that may introduce systematic errors into the measurement.

By these means one *maximises* the sensitivity of the measurement to the desired parameters and one *minimises* the sensitivity to undesirable effects and their associated systematic errors. Thus, from (2) any physical dielectric supports required to keep the specimen in place should ideally lie in low field regions. This principle is followed, for example in dielectric resonator measurements, which use a quartz support to keep the specimen away from cell walls (Section 7.1.15), and this is normally placed in a low field region. Principle (2) may also be applied to fringing-fields in admittance cells, which ought to be minimised in magnitude - hence the preference for three-terminal (Section 7.1.1) rather than two-terminal (Section 7.1.3) admittance cells. These principles can only be followed if one is aware of the field patterns in resonators and the current patterns on their walls. Many standard text books contain these details (see, e.g. [10] and Cook, pp. 12 – 27 in [5]).

The same two principles apply if we are trying to select out *regions of a specimen*. Thus in TE_{01}-mode cavities (Section 7.1.7) and open-resonators (Section 7.1.17), one can choose the specimen thickness to be of an integral number of half-wavelengths, thus arranging for an *antinode* (i.e. maximum) of the E-field to lie in the centre of the specimen, and a *node* (i.e. a field minimum) to lie on both of its plane surfaces. In this way one does not 'see' any contamination that might be present on the specimen surface.

6.6.3 Non-Linearity in Dielectrics

The class of *functional* materials covers a number of materials that are *intrinsically* non-linear. They include ferroelectrics, and also some thin-films, devices like doped and biased semiconductors, 'weak links' in superconductors (Josephson Junctions) and material-to-material interfaces (e.g. metal/insulator 'Schottky-Barrier' junctions). It is probably best to consult a solid-state physics text book for information on such materials (e.g. [13]), because detailed advice on their characterisation lies beyond the scope of this Guide. Further information on measurements on ferroelectrics can be obtained from [35] and [129].

However, at sufficiently high field-strength, *any* dielectric can be non-linear. As pointed out in Section 2.3, dielectric behaviour is largely dominated in the RF & MW range by *dielectric relaxation*. Such relaxations, associated as they are with dipoles, are subject to saturation. This can occur, for example, when *all* of the dipoles are aligned in a dipole-rotation process. If large static or alternating fields are applied to materials, they therefore become non-linear, i.e. their complex permittivity will change with field-strength [1, 13, 17, 130]. The effect can be very small for non-polar materials (i.e. those without permanent molecular dipoles in the absence of E-field) - it is typically of the order of one part in 10^6. But it can be quite large for polar molecules, especially long-chain polymers, amounting to more than one part in 10^3 in some cases. This effect will probably not be significant in most low-field dielectric measurements, however, which typically apply up to a few tens of volts to specimens. Usually kilovolts have to be applied to detect an effect at all. But it is worth remembering that the complex permittivity measured in such low-field techniques is the *low-field* permittivity.

If the *intention* of one's scientific study is to *measure* such non-linear effects, remember:

(i) Take all appropriate safety measures to deal with the high voltages you will have to use.

(ii) Build *robust* cells! High fields produce large forces between different parts of the cell that can distort the cell. Capacitance changes from cell distortions may be larger than those to be studied in the specimen!

When *very* strong fields are applied to dielectrics an extreme and irreversible form of non-linearity occurs: they *break down*, see [131]. The study of dielectric breakdown constitutes a metrological field in its own right and cannot be treated in detail here, but a point to bear in mind in such measurements is that the *electric strength* of air (i.e. the highest field-strength that can be applied before breakdown occurs) is approximately 3 V μm^{-1} or 3 kV mm^{-1} and a safety factor of at least three should always be used. Note that breakdown *along* the surface of a material could occur at lower voltages, especially if the surfaces are not clean.

6.6.4 Magnetic Fields and Non-Linearity of Permeability in Magnetic Materials

Referring back to the discussion on electrical non-linearity in Section 6.6.3, similar considerations apply to the measurement of permeability in the presence of strong static magnetic fields. However, here, as pointed out in Section 5.3.5, the non-linearity is generally *much stronger* than the effect of static electric fields upon dielectrics

► *Summary: E.M. Fields and Specimens*

► *Do's and Don'ts regarding E.M. Fields and Specimens*

Do make sure you know how the electric field is oriented in your measurement cell

If you are trying to measure the non-linearity of dielectric materials by applying large fields to them, ***do*** remember that these fields will also set up electrostatic forces across the cell itself and these can lead to the distortion of the cell, affecting, e.g., capacitances between electrodes. The measurement changes caused by this effect may exceed those you are trying to measure in the dielectric.

Do note that for ferroelectrics and thin films and any material for which properties change with field-strength it is important to record and report the field-strength used.

6.7 High and Low Temperature Measurements and Temperature Control

6.7.1 Temperature Control

At ambient temperatures, any methods that can hold the specimen to ± 0.2˚C should be adequate. Try to ensure that the entire specimen is at the same temperature. Attempt to measure the temperature of the *specimen* and *not the surrounding cell*, even to the extent of inserting the temperature sensor into the specimen (if it is a liquid) and removing it just prior to each dielectric measurement. Remember that the temperature of an evaporating liquid will be lower than its surroundings. If temperature sensors and temperature control components are a permanent fixture of the cell make sure that they do not interfere with the electrical measurements. In particular, ensure that they do not effectively 'short-circuit' admittance cells via conducting cables or liquid-filled tubes! Always ensure that all temperature sensors are calibrated prior to measurement. This can often be done cost-effectively, *in-house*, by direct comparison with a mercury-in-glass thermometer that is already traceably calibrated. See also Section 6.1.8 on temperature control of measurement cells.

6.7.2 High Temperature Measurements

Section 7.1.3 describes one method that can be used for high temperature ceramics up to 10 MHz and 1200˚C and this is related to two existing standards: ASTM D 2149-97 [61] and ASTM D150-98 [53]. Both standards give a warning that contacting electrodes on specimens may be unsatisfactory at higher frequencies because of their series resistance, but they do not quantify the effects. Another standard concerned with measurement of ceramics at elevated temperatures is ASTM D2520-01 [62], discussed in Section 3.2.

At higher microwave frequencies there appear to be no recommended cells in the standards literature so one must design them oneself. They should use components that can withstand the high temperatures (e.g. stainless steel waveguide) without reacting with the specimens. The effects of thermal expansion and contraction of the cells should be minimised by design and then either be compensated for in the measurement equations or should be taken into account in the uncertainty budget. It may be found that actively heating the specimen and cell and then taking measurements as they are allowed to cool slowly can provide the best stability.

6.7.3 Measurements below Ambient Temperature

Remember that moisture from the atmosphere will condense on unprotected cells and on specimens at temperatures significantly below ambient, giving rise to systematic measurements errors, especially for loss. This can be avoided, if necessary, by using a vacuum cell. See also Section 5.3.8.

Cryogenic measurements require a well-sealed cell. For low-loss measurements dielectric resonators are often favoured [132], the measurements being performed in a '*cavity*' cell, see Section 7.1.15. Dielectric losses generally fall rapidly as the temperature approaches absolute zero so that Q-factors higher than 10^9 can be obtained at liquid-helium temperatures with suitable materials, e.g. sapphire. Microwave sources must be suitably stable to measure such high Q-factors, see the discussion on signal sources in Section 6.4.5.

6.8 Specimen Porosity and Density

Many materials are porous and their complex permittivity will, of course, depend upon the degree of porosity that they exhibit. Two types of porosity may be distinguished: open and closed. *Open* pores are open to the air and will (eventually) be filled with liquid when submerged. *Closed* pores are isolated in the matrix of the material and will not be filled when submerged. The *Archimedean Weighing* [89] procedure can be used to distinguish between them, after heating the specimen and allowing it to cool in a suitable liquid.

The permittivity, ε', of a porous material depends upon its porosity P, where $P = (1 - \rho_m / \rho_d)$, ρ_m is its measured density and ρ_d would be its full (pore-free) density. If the material is otherwise homogeneous and its porosity does not exceed 10%, Equation 6.16 may be used to relate ε' to ε'_d, the permittivity of the fully dense material:

$$\varepsilon' = \varepsilon'_d\left(1 - 3\frac{\varepsilon'_d - 1}{2\varepsilon'_d + 1}P\right) \qquad \ldots 6.16$$

For higher porosities, it is probably necessary to use more complex mixture formulae, see Section 5.3.10.

The density of compressible materials affects their permittivity. If variations are observed in the ε' values of a set of nominally identical specimens, variations in density may be the cause, and the above formula may be used, to test this hypothesis if it is thought that air-filled pores are causing the variation.

Another formula which is often used for relating the permittivity of low permittivity polymers such as polyethylene and PTFE to density is given in Equation 6.17:

$$\frac{(\varepsilon'-1)}{(\varepsilon'+2)}=K\rho \qquad \text{... 6.17}$$

Where ρ is the density in g.cm^{-3} and K is a constant for each low-loss polymer. For one grade of PTFE one reference [77] gives $K = 0.119$, from which we can compute $\varepsilon' = 2.046$ for $\rho = 2.174$ g.cm^{-3}. Values for other polymers have been given as: $K = 0.3250 \pm 0.0005$ for polypropylene, 0.3255 for HDPE (high density polyethylene), 0.3290 ± 0.0002 for LDPE (low density polyethylene) [133].

6.9 Electromagnetic Field Modelling and its Role in Dielectric Measurements

It was traditional in past decades, to design dielectric measurement systems to be easily calculable. They had simple geometries in which the mathematics employed to describe electromagnetic fields was tractable and, hopefully, delivered *explicit* equations for computing ε' and tan δ. In recent decades computer-based tools have become more accessible and more reliable and so they can now be used as *part of* the measurement process. They can model much more complex geometries and can help us to estimate correction terms, e.g. effects of fringing-fields, in more traditional methods. This is especially true of (i) *modal analysis* in which fields are decomposed into orthogonal modes, as used with e.g. coaxial probes, Section 7.1.12, and (ii) *discretised* methods such as the Finite Element (FE) [134], Finite Difference Time Domain (FDTD) [135], Finite Integration (FI) methods (a particular formulation of FDTD) and Transmission Line Modelling (TLM) [136]. These discretised techniques can, in principle, be used to model almost any complex measurement geometry.

Much of the advice in this Guide assumes that such modelling facilities are *not* available, i.e. that we are restricted to traditional techniques. Mathematical modelling is, after all, expensive - software has to be written, or purchased, and validated. Subsequently, much time and effort is needed the gain the expertise for its proper application. Nevertheless, when these techniques are fully integrated into the measurement process, they can improve long-term convenience and, more importantly, can improve our measurement accuracy. The dielectrics community has been reaping the benefits of these techniques in recent years. Therefore, at appropriate points of the Guide we have given pointers as to where mathematical modelling can help to improve our metrology.

In dielectric measurements, modal (and related) analytical modelling is central to the use of split-post-resonators (Section 7.1.8), coaxial and waveguide probes (7.1.12) and dielectric resonators (7.1.15). Other workers have used modal analysis to render the TM_{01}-mode method an *exact*, rather than a *perturbation* method [137]. FDTD and related FI modelling is ideal for evaluating the effects of fringing-fields in two-terminal admittance and re-entrant cavity methods (7.1.3, 4 & 5) and for computing filling-factors (Section 6.1.9) in the latter. Another useful feature in a number of commercially-available modelling packages (including FI software) is the ability to use an *Eigenvalue-Solver,* which can help one to identify all of the resonant modes of the cell. These can be compared in the frequency domain with those actually measured when one is trying to identify the correct mode to use for measurements in a resonator.

► ***Do's and Don'ts for Electromagnetic Field Modelling***

Do evaluate any software that you have written or have purchased by using it first to model easily analytically calculable geometries; ensure that your calculations agree with analytical methods.

Do, if you can, evaluate the modelling of complex geometries by using two or more independent computational methods and checking that they agree.

Do check numerical models of measurement cells by measuring known specimens and reference materials.

Don't expect that new software will give you 'the right answers' at first use.

Don't trust software unless you have tested it and have gained enough experience to be adept at using it.

7. Measurement Methods

7.1 A Survey of Methods

This survey is, of course, limited in scope, but we hope that we can cover enough ground here to allow the principles of good dielectric measurement to be demonstrated for each method and to show how they can be extended to related techniques. Our main aims for each method are:

(i) To introduce the method.

(ii) To present its metrological principles, strengths and weaknesses.

(iii) To emphasise good practices, especially those not normally mentioned in the scientific literature.

(iv) To describe which types of measurement the method is good for, and which it is inappropriate for.

(v) To provide guidance on typical uncertainty values and how to reduce them.

(vi) To provide the reader with enough information to allow an informed selection between techniques. Further information on this matter is given on this in Section 7.2.

There is unfortunately not enough space here to give *detailed* descriptions of the methods, so *measurement equations* (see Glossary) are generally not given. However, references are supplied to publications in which the appropriate details and equations can be found.

> *Note 1:* Some common themes are covered in more detail in Sections 5 and 6. Cross-references are provided.
>
> *Note 2:* Where uncertainties are given, they should be interpreted as being for 95% confidence level. Uncertainty values are given for the purpose of guidance in the choice of methods, but, in practice, *actual* uncertainties can vary significantly from one material and one specimen to another, so the figures should not be taken too literally.
>
> *Note 3:* The depth of coverage varies from method to method. More details are given for fundamental methods through which important metrological points can be demonstrated. For convenience, summaries of the most important points are given at the end of the longer descriptions.
>
> *Note 4:* The methods are presented roughly in order of increasing frequency, but this scheme sometimes takes second place where similar methods are best grouped together.

7.1.1 Admittance Methods in General and Three-Terminal Cells

Introduction. One of the simplest methods for characterising dielectrics was introduced in Section 2.1: we can carry out a capacitance measurement on a dielectric specimen in a two-terminal parallel-electrode admittance cell, see Figure 1, in which the dielectric under test (d.u.t.) is substituted for an equal thickness of air in the cell. The ratio of measured capacitances gives us the specimen permittivity. In practice, this approach forms the basis of perfectly sound dielectric measurement techniques all the way through the spectrum from a few μHz up to a few GHz in frequency. To determine the loss, one measures admittance, Y, rather than capacitance, hence the term 'admittance method' is to be preferred to 'capacitance method' as it is more general. For loss measurement of low-loss materials the method can be modified for resonance measurements, as discussed in Section 7.1.4, below.

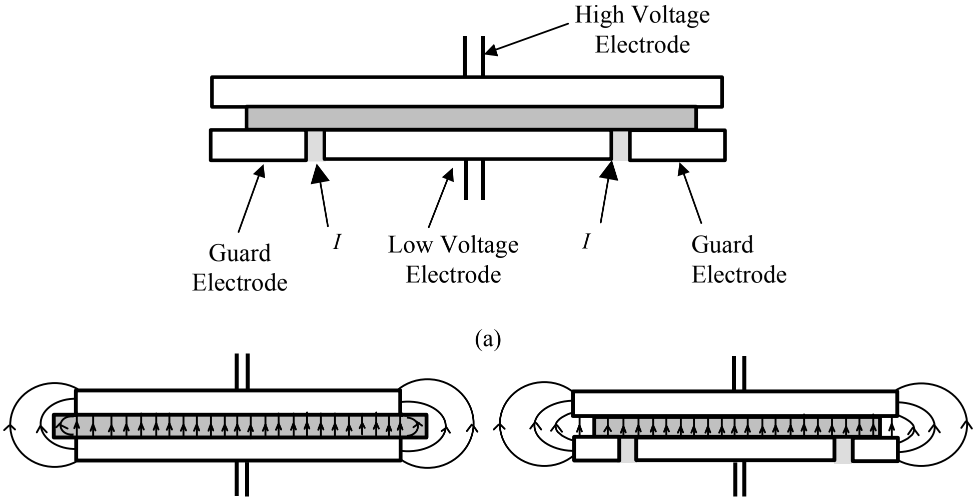
In order to reduce measurement errors, two variations on this admittance technique are used:

> (i) As shown in Figure 28 (a) an earthed *screen* or *guard* electrode can be placed around the low voltage measurement electrode to remove the effect of fringing-fields, see Figure 28 (b). Without this guard, the presence of fringing-fields in the air-dielectric surrounding a solid dielectric specimen can give rise to significant errors of measurement. Cells with such a third electrode are called *Three-Terminal Admittance Cells* (3-T cells) and they are generally much more accurate to use than the simple *Two-Terminal* (2-T) type discussed below in Section 7.1.3 and shown in Figures 1 and 28 (b).

(ii) As briefly discussed in Section 5.3.3, and in the Glossary entry ‘*complex permittivity’* the complex permittivity, ε^*, of a dielectric may be defined by the ratio of the admittance, Y, between two electrodes with and without the dielectric:

$$\varepsilon^* = \frac{(Y \text{ when the space around the electrodes is completely filled with the dielectric})}{(Y \text{ when it is completely filled by a vacuum})} \quad \ldots 7.1$$

Liquids will readily fill the full volume of a measurement cell, whereas solid specimens cannot do so and this gives rise to measurement errors for solids, which are caused by electrode/specimen air-gaps and by fringing-fields. By immersing a solid specimen in a liquid of similar permittivity, these disadvantages can be overcome. This principle is used and further developed in the *Three-Terminal Liquid Immersion Techniques* discussed in Section 7.1.2.



(a)

(b) (c)

Figure 28. **(a)** A three-terminal admittance cell. ‘*I* ’ is a *thin* low-loss insulating ring (it is exaggerated in thickness for clarity in the diagram), which separates the low voltage measurement electrode from the annular guard electrode. **(b)** In a two-terminal cell the E-field lines fringe out around the edge of the electrodes, giving rise to a fringing-field, which can pass partly through the specimen, thereby generating measurement errors. **(c)** In a three-terminal cell the field lines between the high and low voltage measurement electrodes are straight and the field is uniform, so measurement errors caused by fringing-fields are removed.

The admittance cell method is generally suitable in the range from sub-hertz to 1 - 2 GHz (in very small cells) for thin sheets or laminas in both 2-T and 3-T parallel-electrode admittance cells, but the latter with a much-restricted upper frequency limit. It is suitable for low to high-loss specimens, though not usually for strongly conducting materials (but see the section on ‘Air-Gap Methods’, below). Resonance methods are usually best suited for measuring very low losses accurately, however loss angles below 100 μrad may be measured directly on impedance and frequency response analysers (see Sections 6.1.2 and 6.1.3), provided they have sufficient resolution and repeatability.

In this Section, 7.1.1, we cover only *non-resonant* measurements in which the cells are connected to an impedance analyser or a frequency response analyser (see Section 6.1.3). *Resonance* methods are discussed below in Section 7.1.4.

Note that admittance cell methods cannot measure the magnetic properties of magnetic specimens.

Residual Fringing and Stray Fields Even a 3-T guarded electrode cannot completely remove these fields because the guard-electrode cannot be allowed actually to touch the ‘Low’ measurement electrode. There is therefore a gap between them, see Figure 28 (a), that slightly distorts the otherwise uniform E-field. Ideally the capacitance due to these fields will be less than 1% of what it would be without the guard electrode, but for thick specimens, or if there are large gaps between the guard and ‘Low’ electrodes, the reduction in these fields may amount to only 10% or less. See Section 7.1.3 for a description of the fringing and stray capacitances that arise in 2-T systems.

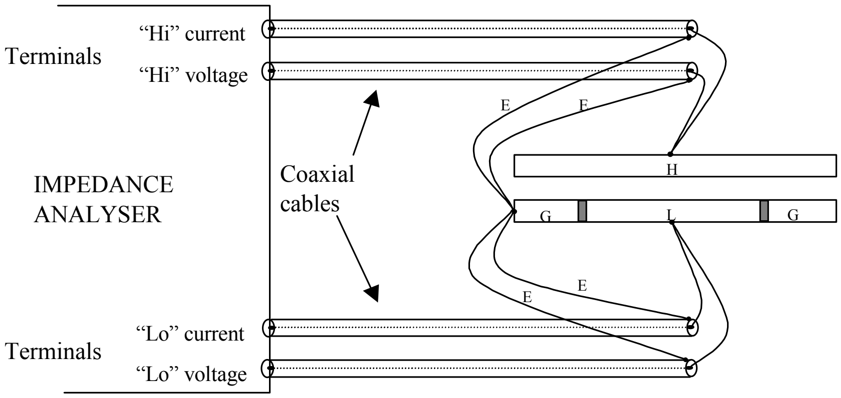


Figure 29. The best way to connect a three terminal (3-T) cell to an impedance analyser fitted with four-terminal-pair (4-T-P) connectors. 'H' is the high voltage measurement electrode, 'L' the low voltage measurement electrode and 'G' the guard-ring electrode. The measurement electrodes are connected, ideally from their centres, to the *inner-conductor*s of the relevant coaxial leads. The connections marked 'E' are between the *outer-conductor*s of the coaxial lines and the guard electrode. They should be kept as short as possible to minimise self and mutual inductance.

Connection of Admittance Cells to Impedance Analysers. Some thought should be given to the best way of connecting impedance analysers to admittance cells. This is not obvious because the cells have either two or three terminals, while the analyser will often be fitted with *four-terminal-pair* (4-T-P) connectors. Consultation of the Impedance Guide [80] is recommended to gain understanding of the principles involved here. Following the details in the Glossary of Reference [80] leads to the following recommendations:

Usually the best way to connect a 3-T cell to a 4-T-P-fitted impedance analyser is shown in Figure 29:

> 'High' voltage and current leads (usually the inner-conductors of coaxial leads) should be connected directly to the *un-guarded* electrode which should be the electrode that is driven by the micrometer.
>
> 'Low' voltage and current leads (usually the inner-conductors of coaxial leads) should be connected directly to the electrode that is surrounded by the guard electrode, because they are both close to the 'Guard' potential.
>
> These connections to the electrodes should be to a point as close to the electrode-centres as possible, to minimise inductive effects associated with the distribution of current in the electrodes themselves.
>
> The screening on coaxial leads should reach as closely as possible to the cell without risk of contact. They should all *individually* be connected to *one single point* on the cell's own guard electrode in a good zero-impedance connection. Any extra screening required to minimise stray capacitances in the system, should also be connected to this point
>
> In 2-T measurements, where there is no guard electrode, the cable screens cannot be connected to the cell but the screening on the cables should be taken to within a few centimetres of the cell and all of the screens connected together in a good zero impedance connection at that point.

So much for 4-T-P connections. In 2-T measurements in which the connection into the impedance analyser or bridge is via *a single coaxial connector*, the analyser may be calibrated for admittance measurements using methods recommended by the manufacturer, or methods similar to those used on ANAs.

Cables should be as short as possible for both 2-T and 3-T measurements to reduce residual inductance: this will increase the measurement frequency range: see Section 2.2.1 on the problems introduced by residual inductances, also consult ASTM D150 [53] on methods for compensating these effects. One exception to the general rule of using the shortest possible-cables is that some impedance analysers allow a special inductance correction to be made specifically for 1-metre length cables. Our experience is that this works effectively, though it may not be advisable to use this correction above 30 MHz.

Whenever *Materials Analysers* (Section 6.1.2) are used with their own proprietary cells the manufacturer generally provides a more direct connection between cell and analyser. Use of this direct connection is to be recommended whenever available.

Methods and Options:

The Use of Air Gaps above the Specimen Contacting a specimen with both electrodes can significantly distort both the cell and the specimen, leading to erroneous results. It can also damage them if excessive force is applied - this is all too easy to do via the *mechanical-advantage* afforded by a micrometer drum and screw thread. After such mistreatment, the mechanical relaxation time for the cell to return to its original condition may cause the zero-error, Z (Section 6.1.5), to drift during a set of measurements: changes of over 10 µm in Z can result from this. Such thoughts have lead to the development of methods in which an air gap is *deliberately* left above the specimen and in which one measures a *series combination* of two capacitances, the capacitance of the air gap and the capacitance of the specimen. The air gap need not be large, 50 µm to 200 µm is typical, and, in fact, repeating the measurement with a number of different air-gaps is a good way of checking systematic uncertainties. A micrometer-driven cell is essential for the use of this method. If the micrometer reading, M_i, its zero-error, Z, and the thickness of the specimen, t_s, are all known, the complex permittivity of the specimen is readily computed. The guidance of existing standards may be followed, in particular BS 7663:1993 Method A, see [138]. For admittance cells, one of the best ways to measure Z is to use a capacitance method, see Section 9.5. The air-gap approach can be used both in conjunction with the *capacitance method* and with the *equivalent-thickness method*, as explained below.

The Capacitance Method. In this 'traditional' method ε' and tan δ are computed from the admittances obtained with the specimen both in and out of the cell, as described in Section 6.5. The method is best suited to specimens with $\varepsilon' > 5$ and also to thin-film measurements, for which the impedance analyser must be adjusted for high resolution, e.g. use a high averaging factor. See [138] for details.

The Equivalent-Thickness Method. This was described in Section 6.5. The basic equation for this method, which takes account of the air-gap above the specimen but not of fringing-fields, is as follows:

Let t_s be the specimen thickness
M_i be the micrometer reading when the specimen is in the cell and when the impedance analyser is reading a capacitance of C_i pF
M_o be the micrometer reading when the specimen is not in the cell, after the electrode gap has been reduced so that the impedance analyser is again reading a capacitance of C_i pF
$t_x = M_i - M_o$

Then:

$$\varepsilon' = \frac{t_s}{t_s - t_x} \qquad \ldots 7.2$$

Note that we do not need to know the value of the micrometer zero-error, Z, (Section 6.1.5) to compute ε'. We *do* need to know Z, though, if we wish to measure tan δ, see [138] for full details and measurement equations for the method. It is well suited to low permittivity specimens with $\varepsilon' < 5$, but it is not appropriate for very thin specimens ($t_s < 0.3$ mm, say), for which the capacitance method above should be used instead. A worked example of the use of this method in a 3-T cell is given in Section 9.4.

Applied Metal Electrodes. The use of applied electrodes is advisable for high-permittivity materials with $30 < \varepsilon' < 3000$ and also for measuring the temperature-dependence of dielectric properties. They are inappropriate, though for measurements of *loss* on low-loss materials, tan δ < 1000 µrad. Such electrodes are most often used on ceramics. Care should be taken to ensure that the deposition of the electrodes does not alter the properties of the material under test and that a low resistivity, non-corroding electrode material is selected. Air-fired palladium-silver is often chosen for this purpose. Either micrometer-driven or sprung electrodes may be used, but with care, and they must make good electrical contact with the metallic coating. Generally films 10 - 20 µm thick will prove suitable. Impedance analysers fitted with four terminal pair (4-T-P) connections can be used to assess the suitability of the metal films by measuring their conductivity: the conductivity of metal coatings which are too thin can introduce significant measurement errors. The 4-T-P analysers can also be used to check that the connection impedance to the electrodes is adequately low. Such measurements may be done at any convenient frequency - not necessarily the frequency at which ε^* is being measured. Loss in both the metallisation and contact resistance can be taken into account. There is a well-established theoretical relationship between the surface resistivity of the electrodes and the observed values of dielectric loss, consult report [139] for further details. The report showed that realistic values of electrode resistance can *double* the observed loss of a nominal loss angle of 100 µrad at frequency of 10 MHz, so care must clearly be taken.

Further Comments on the above Methods For $\varepsilon' < 5$ best uncertainties can usually be obtained by the combined *Equivalent-thickness* method and the air-gap technique. Typical uncertainties for ε' may be ± 0.5% to 2% for a 3-T cell and about ± 5% for a 2-T cell. Metallised specimens are recommended for ε' when $\varepsilon' > 30$. They may be measured between sprung or micrometer-driven electrodes. When measuring *loss*, the presence of conducting electrodes acts as a confounding factor. Conducting electrodes are therefore not advised for loss angles less than 3 milliradians. The air-gap method should be used instead, which may, however, give rise to uncertainties of ± 10% or more in $\tan\delta$. For the most accurate measurement of both permittivity and loss of a material with $\varepsilon' > 5$ both applied-electrode and air-gap methods may have to be used, the first for ε' and the second for $\tan\delta$.

Specimens. Those which are not thin films should have plane-parallel faces and should be flat to within ± 10 µm, they should not be significantly warped, saddled or dished, or have protrusions, voids or inhomogeneities, otherwise measurement errors and uncertainties will probably be significantly increased. Specimen thickness, t_s, should ideally be between 0.2 and 5 mm but not be greater than $D/7$, where D is the diameter of the smallest measurement electrode. The rationale for this lies in the fact that the optimum t_s will lie in a narrow range determined by (i) the large errors caused by the uncertainty in measuring t_s for very thin specimens and (ii) the systematic uncertainties caused by distortions to the E-field, including fringing-fields, for large t_s. Fringing capacitances typically amount to 5% to 12% of the total capacitance of 2-T cells, but less than 1% in 3-T cells. Thicker specimens can be measured more accurately in 3-T cells than in similar 2-T cells. Specimen cross-section may be circular, or if the specimen is significantly larger than the electrodes it can be of any convenient shape. Different considerations clearly apply for *thin films* with thickness in the range 1 µm to 200 µm, for which more accurate measurements of t_s are clearly required, see Section 5.3.4.

Frequency Range and Electrode Size. Because of the effects of residual inductance, it is recommended that it is best when measuring loss to ± 10% at 100µrad to restrict the maximum frequency of measurement, f_{max}, to 1 MHz. Frequencies up to 10 times higher may be useable if cells are connected directly to analysers without the use of leads or if the measurement plane of the bridge can be defined at the cell by means of a calibration process. Note: these are notional limits and measurements with greater uncertainty may be carried out typically up to double these frequencies but be aware that uncertainties tend to rise as the square of the frequency. In general, f_{max} is determined by lead inductances (approx. 1 nH per cm) and cell capacitance (see Section 2.2.1, Equation 2.8). The general rule is that the higher the frequency to be measured, the smaller or shorter should be the components and leads. Better accuracy for ε' and better resolution for $\tan\delta$ will normally be obtained with larger D but this benefit has to be weighed against these frequency limitations.

Sprung Electrodes vs. Micrometer Driven Electrodes Some commercially available admittance cells are supplied with sprung electrodes. Compared with micrometer-driven cells they have two disadvantages: (i) they will compress soft and malleable specimens - usually the force cannot be adjusted and it may be entirely inappropriate for the specimen, (ii) they do not have the flexibility that a micrometer-driven cell does, which facilitates the evaluation of systematic uncertainties, e.g. one cannot measure the empty-cell admittance if the electrodes are sprung. Generally speaking, more accurate measurements can be obtained from micrometer-driven cells, though they may be more time-consuming to obtain. Cells with sprung electrodes may readily be used, however, for measurements upon high permittivity *metallised* ceramic specimens, see below.

Electrode Diameter. In many methods which compute ε' from capacitance and in all measurements of $\tan\delta$, one contribution to the uncertainty budget will be the uncertainty in the electrode diameter, D. In air-gap methods D will be the diameter of the 'High' and 'Low' voltage electrodes of the cell (or the smaller of the two if they are unequal). For specimens with applied metal electrodes D will be the diameter of the applied electrodes - which may also be the diameter of the specimen if it is circular. In the latter case D will be the *same size as*, or *larger than* the diameter of the cell electrodes. In the *Capacitance Method with sprung electrodes* the percentage error in the measured ε' and $\tan\delta$ will be proportional to D^2. Thus if $D = 20\text{ mm} \pm 0.1\text{ mm}$, the corresponding contribution to the total uncertainty in ε' and $\tan\delta$ will be ± 1%. Electrode diameter must therefore be measured as accurately as possible. However, if the air-gap *Equivalent-Thickness Method* and the *Capacitance Method* are employed with *fixed* or *micrometer-driven electrodes,* they do not usually require the diameter of the electrodes to be known accurately for ε' measurement and so are likely to be more accurate for ε', but the specimen diameter D is *always* required for $\tan\delta$ determinations.

Control of Electrode Spacing by Micrometers and Micrometer Zero-Errors See Section 6.1.5 and the worked example on the measurement of zero-error in Section 9.5

► ***Summary: Admittance Cell methods and the 3-T Technique***

Advantages of the 3-T Technique. This can be one of the most accurate of all dielectric measurement methods for laminar specimens, especially for low permittivity specimens: uncertainties better than ± 0.5% are achievable for ε' (for $\varepsilon' < 5$) and ± 5% for tan δ. It is certainly more accurate than 2-T methods described in Section 7.1.3.

Disadvantage of the 3-T Technique. The operational frequency band for a cell is not as great as that of a 2-T cell of similar size.

Admittance Cell Methods: Typical Sources of Dominant Uncertainties:

For ε':
- The uncertainty in specimen thickness
- The uncertainty and repeatability in reading micrometers
- Warped and poorly shaped specimens
- For the *capacitance method* at high frequencies: inductance of long leads.

For tan δ, as for ε', plus:
- Resolution and the systematic errors in the impedance analyser for measuring conductivity.
- For applied metallic electrodes: additional losses in the electrodes.
- At high frequencies: additional losses in coaxial leads.

7.1.2 Liquid Immersion Admittance Cells

This method is for measuring the permittivity, ε', of solid dielectric specimens *only* and is not intended for dielectric loss measurement. There are two existing standards for the liquid immersion method that should be referred to here: BS 7663:1993 [138] and ASTM D1531-01 [56]. The latter gives the basic theory of the method, though it is not, in general, necessary to use the exact design of cell described there unless one is required commercially to follow this standard. At RF up to 10 MHz, this is one of the most accurate methods for ε' of low-permittivity specimens if $\varepsilon' < 5$, though experience shows that it can be useful for permittivities up to 10 with reduced accuracy. As discussed in Sections 5.3.3 and 7.1.1, admittance-cell measurements can be performed much more accurately on liquids than on solids because of the presence of air-gaps and fringing E-fields around solid specimens which pass through air and not the specimen. In the *Single-Liquid Immersion* method, these problems are overcome by surrounding the solid specimen with a liquid of similar permittivity and measuring the capacitance difference when the solid specimen is inserted between the electrodes in the liquid. By using a substitution method the uncertainty for measuring ε' of the solid may be very little higher than that of measuring the liquid alone. A traditional application has been the accurate determination of the permittivity of polyethylene, $\varepsilon' \approx 2.32$ by immersing specimens in 1-cSt (1 centistokes) silicone fluid which has similar permittivity at typical ambient temperatures [140]. If a good permittivity match between specimen and a suitable liquid cannot be obtained, the *Two-Fluid Immersion Method* may be used. The word 'fluid' is used here because either two liquids or one liquid and a gas – usually air - may be used, as appropriate. With either method total uncertainties better than ± 0.5% can be obtained, provided that the solid specimens do not interact with the fluid or have open porosity.

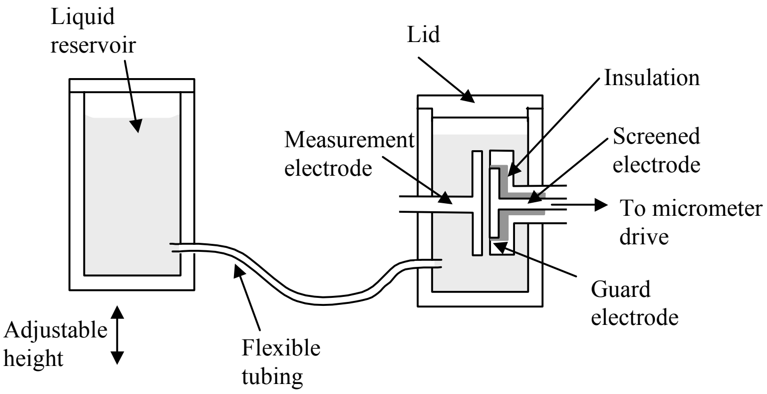


Figure 30. A Liquid Immersion Cell (right), with a separate reservoir for storing the dielectric liquid.

The Measurement Cell. A 3-T micrometer-driven design is recommended, with the electrode faces mounted vertically to allow the specimen to be inserted easily without loss of liquid from the cell. A typical immersion cell is shown in Figure 30. In designing such cells, arrangements should be made for precise temperature control of the immersion liquid to within ± 0.2 ˚C. In Figure 30 the liquid reservoir is itself temperature controlled.

Specimen Materials. Not all materials are suitable for measurement by this method. Materials that may dissolve in or be contaminated by the immersion fluids cannot be measured. For materials with open pores one has to consider whether one wishes to measure specimen permittivity with air or liquid in its pores. If the former, the method may be inappropriate, unless a way can be found for making a correction, or if the error can be shown to be insignificant, e.g. by Archimedean weighing [89], see Section 6.8.

Immersion Liquids. Reference liquids (Section 4.4.2) may be used as immersion liquids. Many of them are lossy, so the specimen needs to be cleaned before it is used again - one should at least be sure that all of the liquid has evaporated from it before re-use. Remember that the liquid permittivity is strongly dependant on frequency and temperature. For $\varepsilon' \approx 80$, de-ionised water may appear to be an attractive immersion fluid, but there is a possibility that the cell may be damaged by water-induced corrosion. Whichever liquids are used, one must know their permittivity for this method to work. If it is not previously known, it is readily measured in the immersion cell itself by the capacitance method described above.

Methods. The *Single-Liquid Method* proceeds along the lines of the *capacitance method* described in 7.1.1, the theory is essentially the same but uncertainties in ε' caused by fringing-fields and by uncertainties in electrode spacing and specimen thickness are *significantly reduced*. Indeed, if the specimen has *exactly the same permittivity as the liquid* there is no capacitance change at all on inserting the specimen into the liquid, so one can know that it has the same permittivity without knowing its thickness and the electrode separation, etc!

If a good liquid/solid match of permittivities cannot be obtained, the *Two-Fluid Immersion Method* is more accurate. It also uses the *capacitance method* but four capacitances are measured in all, with appropriate repeats and suitable averaging to reduce uncertainties - 'Fluid 1' is often air:

C_1 - the cell capacitance with Fluid 1 alone
C_2 - the cell capacitance with the specimen in Fluid 1
C_3 - the cell capacitance with Fluid 2 alone
C_4 - the cell capacitance with the specimen in Fluid 2

The same electrode spacing is used throughout, just enough to allow the specimen to rest between the electrodes. Air and fluid permittivities are readily computed from these capacitances, see [56].

Liquid Immersion: Other Applications. The use of liquid immersion to improve accuracy for solids is a principle that can be applied in other measurement methods. For example, it can be used in transmission line methods, such as those described in Sections 7.1.10 and 7.1.11, and Lynch has described just such a measurement in a waveguide bridge [141]. The method may, in suitable circumstances, be extended to the measurement of loss, provided suitable equations are used and the loss of the liquid is correctly accounted for.

► ***Summary: Liquid Immersion***

Advantage of the Liquid Immersion Techniques. This can be the most accurate of all measurement methods for ε' at low RF (e.g. 1 MHz), especially for low permittivity specimens. Uncertainties better than ± 0.2% are achievable – the method should be more accurate than the 3-T methods described in Section 7.1.1.

Disadvantage of the Liquid Immersion Techniques. The method is not normally suitable for measuring loss or for porous dielectrics or for solids that will dissolve in the immersion liquids.

Liquid Immersion: Typical Sources of Dominant Uncertainties:

For ε', uncertainty in the permittivity of the immersion liquids, e.g. caused by temperature variation.

► ***Do's and Don'ts for Liquid Immersion***

Do always consult COSHH guidelines [82] and safety personnel on liquids that you intend to use, see 5.3.3.

Don't try to measure loss by this technique, unless the method is suitably extended (as mentioned above).

7.1.3 Two-Terminal Admittance Cells

For solid specimens the use of three-terminal (3-T) admittance cells (7.1.1) is normally to be preferred to the use of two-terminal (2-T) cells because the former can render the significant effects of fringing-fields around specimens almost negligible, see Figure 28. 3-T measurements on solids are therefore, in principle, more accurate than 2-T measurements unless full modelling of the fringing electromagnetic fields in the cell is possible (see Section 6.9). Unfortunately, 3-T cells are considerably more limited in their effective frequency range than are the 2-T cells because earthed screening or guard electrodes become ineffective at higher frequencies. The guard on the cell cannot be at the same RF potential as the guard on the impedance analyser because of residual inductance effects in the leads connecting them. Therefore, 3-T cells have to make way for 2-T cells as one moves up the spectrum. This is usually at a frequency in the range 1 MHz and 30 MHz, depending on connection lead length and specimen capacitance. It is certainly possible to use 50-mm 3-T cells at 10 MHz for specimens with capacitances in the 50 pF region, but the deleterious effects of residual inductance (see Section 2.2.1 and [53]) are visible at 30 MHz and only 2-T methods are really viable at 100 MHz. Remember that the magnitude of these effects tends to rise as the *square* of the frequency. The best way to evaluate the upper frequency limit of a 3-T cell is to measure a low-loss reference specimen. Its dielectric properties will certainly not change rapidly with frequency, but if the *measured* ε' or $\tan\delta$ *appear* to be changing rapidly with frequency, you will know that the instrumentation has reached its frequency limit.

Low-loss specimens For low-loss materials the *resonant* lumped impedance admittance methods described in Section 7.1.4 will generally deliver better resolution for loss than non-resonant and bridge methods. 2-T electrode systems are used exclusively in RF resonator cells because it is not feasible to use a driven guard electrodes at frequencies much above 1 MHz.

High Temperature and Cryogenic Admittance Measurements For high-temperature measurements on ceramics up to 1200°C and for low temperature measurements in Dewars, one may prefer a 2-T to a 3-T admittance measurement because of the difficulty in setting up an effective guard electrode in a cell situated at the end of a long cable well away from the impedance analyser. 2-T measurements are easier to implement in these circumstances. They can, in any case, be corrected for fringing-field effects by comparing a *single* accurate measurement on the material in a 3-T cell at room temperature with a measurement in the 2-T cell also at room temperature. Such a pair of measurements enables one to determine the effect on measurements of the fringing-fields in the 2-T cell. If the specimen material has very different complex permittivity at measurement temperature than at room temperature, ε_T*, say, as opposed to ε_R*, one can perform the comparison between the 2-T and 3-T cells with *another* specimen which has a permittivity close to ε_T* at room temperature, if such a specimen is available. See Section 6.7 for further information on high and low temperature measurements.

Methods All of the methods used with 3-T cells and described in Section 7.1.1 are applicable to 2-T cells also. However, one must remember that whenever 2-T methods are used, some attempt should be made to reduce the effects of *fringing-fields* and *stray fields* as explained in the next paragraph

Fringing and Stray Fields in 2-T cells Ignoring for the moment the loss in the specimen and the effect of air-gaps between specimen and electrodes, we can analyse the total capacitance, C_t, of a 2-T parallel-electrode admittance cell when the specimen *completely* fills the space between the electrodes in the following way. Let A be the geometric area of the electrodes, t_s the specimen thickness (also the separation of the electrodes) and D_s its diameter (assumed to be greater than that of the electrodes). Then the following equations hold:

$$C_t = C_g + C_f(\varepsilon', t_s, D_s) + C_s \qquad \text{and} \qquad C_g = \frac{\varepsilon'\varepsilon_0 A}{t_s} \qquad \dots (7.3)$$

C_g is the most important term. It is known as the *geometric capacitance* and is the capacitance that would be measured if the E-fields only passed *directly* through the specimen perpendicularly to the electrodes. C_s is a constant *stray capacitance,* which is present in all 2-T cells - see the paragraph on stray capacitance below for more details. C_f is the *fringing capacitance,* which depends on specimen size and permittivity

It is the fringing capacitance, C_f, as shown in Figure 28 (b), otherwise known as *edge capacitance* (C_{edge}), which seriously limits the accuracy of 2-T measurements. It is a slowly varying function of t_s, increasing more slowly than C_g as t_s is reduced, but it is also a function of the permittivity and the diameter of the specimen. This is because the fringing-fields pass through the outer part of the specimen, as shown in the figure, if it has a larger diameter than that the electrodes. The purpose of the guard electrode in 3-T cells is to render both C_f and C_s negligible, but in 2-T cells this cannot be done, and so we need to know the likely magnitude and variation of C_f.

Kirchhoff's formula for fringing-fields [142] was traditionally used for computing C_f between two co-axial cylindrical electrodes, but it applies to the case without the specimen only and so cannot take account of the effects of the specimen upon C_f. The international standards literature (see Section 3, particularly ASTM D150 [53]) should be consulted for more recent practices in applying corrections for C_f. Another example of a formula used for calculating a fringing-field in a specific measurement cell geometry is given in Section 6.1.3, Equation 6.2. If no correction for C_f is made, a fractional uncertainty approximately equal to C_f/C_t will have to be included in the uncertainty budget for both ε' and tan δ. In the *capacitance method* (see Section 7.1.1) uncertainties as high as ± 5% for an electrode diameter, D, of 50 mm and ± 12% for D = 7 mm may be expected. For low permittivity specimens ($\varepsilon' < 5$) this may be reduced by a factor of two or three, by using the *equivalent-thickness* method because C_f values with and without the specimen are very similar in magnitude in this method. Metrological studies in the 1970s, e.g. [143], showed that if the specimen is large enough in diameter to encompass most of the fringing-field then a correction might not be needed. For example, it was found that for a 50-mm diameter cell, the extra errors due to fringing are insignificant for specimens of 53-mm diameter or larger. However, it is still not clear if this conclusion applies to higher permittivity specimens.

Numerical Modelling of Fringing-fields. Explicit modelling with, for example, discretised methods such as *Finite Difference Time Domain* (FDTD) or *Finite Integration* (FI) software should offer a route towards more accurate 2-T measurements - see Section 6.9. They can model all stray and fringing-fields and calculate their magnitudes if ε' is known. If the specimen and cell are circular in cross-section, many modelling packages will allow the fields to be computed in a much-simplified 2-D model. This saves computer memory and time. Difficulties may be experienced when modelling very high permittivity specimens, however.

Stray Capacitance, C_s. The stray capacitance is normally regarded as a constant capacitance for any given cell. It is formed from the capacitance between those parts of the support structure of the cell that are unavoidably connected to the 'High' and 'Low' voltage terminals of the impedance analyser but which remain fixed in space when the electrode separation is changed. C_s may be higher than 20 pF for large cells that operate up to 10 MHz, but below 1 pF for small 4-mm diameter cells operating up to 1 GHz. See Section 9.5, for a worked example on how C_s can be measured in a 2-T cell. The presence of C_s does not normally present a major metrological problem because substitution techniques (Section 4.4.1) can remove its effects. C_s does, however, dilute the loss sensitivity of 2-T measurements somewhat and it also limits the frequency range of the cell: the higher C_s, the lower will be its upper frequency limit. Metrologically, the most important requirement for the stray fields that give rise to C_s is that they should *remain undisturbed* during a set of measurements. This allows the substitution method to take effect for eliminating C_s. However, in 2-T methods the *metrologist* (i.e the person performing the measurements) is also close to ground potential and can modify C_s if, say, an arm or hand is placed near to an unscreened 2-T test cell. Therefore: (i) the operator should *withdraw at least 0.5 metre away* from the cell when measurements are being taken on an impedance analyser, (ii) the geometry of all components within 0.5 metre of the cell, especially of the cell itself (apart from its moveable electrode) and any coaxial leads attached to it must *remain undisturbed* during each set of measurements.

► *Summary: The 2-T technique*

Advantage of the 2-T Technique. The operational frequency band for a cell is greater than that of a 3-T cell of similar size.

Disadvantage of the 2-T Technique. As traditionally used, it is generally far less accurate for ε' than the 3-T methods described in Section 7.1.1: uncertainties greater than ± 2% must be expected even for low ε'. Computer modelling of the fringing-fields may mitigate this limitation.

Two-Terminal Admittance Cells: Typical Sources of Dominant Uncertainties:

For both ε' and tan δ:

The effects of fringing-fields (if they are not compensated by computer modelling).
All of the effects listed for 3-T methods at the end of Section 7.1.1.

► *Do's and Don'ts for the 2-T Technique*

Do try to estimate errors caused by fringing-fields and correct for them.

Don't perform measurements when you or your hand are touching, or even close to, the cell – this can change the stray capacitance and introduce measurement errors - withdraw at least 0.5 m before taking measurements.

7.1.4 'Dielectric Test Set', 'Hartshorn and Ward' and Related Methods.

As stated in Section 7.1.1, the loss of medium and low loss specimens can readily be measured in admittance cells connected to impedance analysers. Loss angles below 100 µrad may be measured directly on an impedance analyser but better sensitivity for low loss can be achieved, as ever, by using resonance techniques. The resonance techniques discussed in this section all use 2-T electrode systems and so are subject to the large errors caused by fringing-fields that all 2-T systems suffer from (as discussed in Section 7.1.3). However what they lose in accuracy they make up in sensitivity in loss measurements. Just as with non-resonant 2-T cells, the advent of convenient electromagnetic field-modelling packages allows one to get around the problems caused by fringing-fields (see Sections 6.9 and 7.1.3), so the accuracy of these approaches may be improved over what was formerly available.

The Hartshorn and Ward Technique. At frequencies in the range 100 kHz - 100 MHz a resonant 2-T cell method was very widely used for many years from the 1930s onwards [144], this was named the *Hartshorn and Ward (H & W) Technique*, after the two scientists who developed the method at NPL. It was used both for medium and low-loss specimens until the advent of sensitive impedance analysers in the 1970s, which were found to be more convenient and accurate for medium loss specimens. However the method may still be used to advantage for low-loss specimens. The principle of the method is to resonate a 2-T micrometer-driven admittance cell with an inductor, as shown in Figure 31.

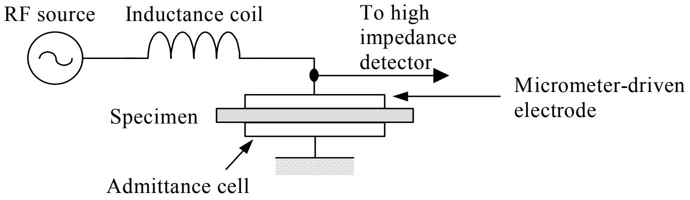


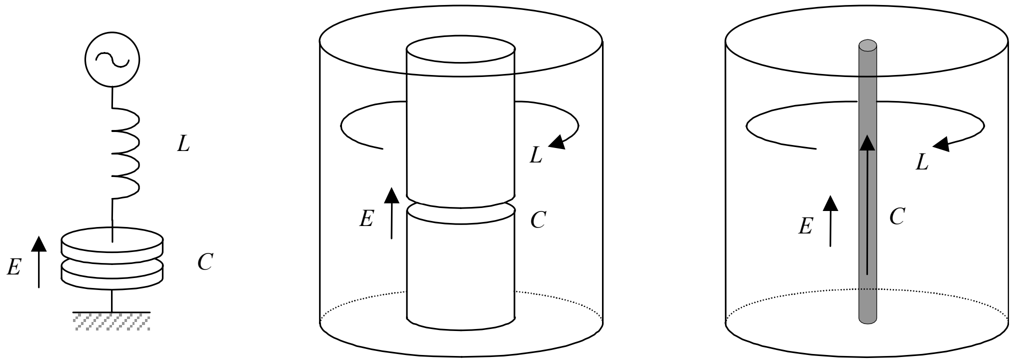
Figure 31. A block diagram showing the basic principles of the Hartshorn and Ward method for low-loss specimens. A number of different inductance coils can be used to cover a band of frequencies.

In the original H & W method mutual-inductance coupling was used to excite the resonance. More recently Q-meters (see Section 6.1.4) or suitable substitutes for Q-meters have been used. The amplitude of the resonance is sampled via a high input-impedance detector, which is designed not to load the Q-factor excessively. Obtainable Q-factors are low - of the order of a few 100 to a few 1000, because of the high loss of the inductors at these frequencies, but techniques have been developed to measure low losses in spite of this - see Section 6.4.5 (Equation 6.15) for the measurement of small changes in Q-factor. As with all resonant techniques one is limited in one's choice of resonant frequencies, but with this type of resonator a range of inductance coils can be used to cover the full frequency range mentioned above. Note that the resonant frequency f_r will be given approximately by $1/\sqrt{LC_t}$, where L is the inductance of the coil and C_t is the total capacitance of the 2-T cell. This will depend upon the capacitance of the specimen, so actual measurement frequencies cannot normally be stipulated beforehand in this method. For low-loss specimens, though, the actual measurement frequencies need not be stipulated - precisely because their dielectric properties change so slowly with frequency (see Section 2.5). The complete H & W system consisting of the 2-T cell, its coils and detectors has often been referred to as a *Dielectric Test Set*.

The full range of methods described in Section 7.1.1, can be used in the H & W method i.e. the *capacitance-change* and *equivalent-thickness* approaches, *air-gap* and *metallised-electrode* approaches. In fact most of these methods were originally developed *for* H & W resonance systems prior to the advent of suitable impedance analysers. The only differences relate to the fact that this is a *resonance* technique, rather than an *analyser-based* technique, so that, for instance, in the *equivalent-thickness* approach one adjusts the micrometer to allow the system to resonate at the same *frequency*, f_r, with and without the specimen, rather than brings the cell back to the same capacitance (although these actions are equivalent, one doesn't actually measure the capacitance in the resonance method). Further details of methods and measurement equations are given in [53].

A Family of Dielectric Measurement Cells. The H & W 2-T cell is the lowest frequency member of a family of resonance cells that can be employed all the way up through the spectrum into the microwave region, see Figure 32. They have in common the feature that the E-field passes directly through the specimen from bottom to top, as shown in the Figure, so for cylindrical specimens, they are sensitive to ε^* in a direction parallel to the axis of the cylinder – a point which is very important to remember if the specimen material is anisotropic, see

Sections 5.3.11 and 6.6.1. In such a resonator $f_r \approx 1/\sqrt{LC}$ so the resonant frequency of measurement cells in this family can be increased *either* by reducing *L or* by reducing *C*, or both. One can reduce *L* by abandoning the coil inductors of the H & W method, Figure 32 (a), and by effectively 'wrapping' the inductance *L* around the capacitor, as shown in Figure 32 (b), This configuration gives one a *re-entrant cavity* or *hybrid* cavity, discussed in detail in the next Section, 7.1.5. Such cavities can be used conveniently in the frequency range 100 MHz - 1 GHz. To resonate at even higher frequencies one can effectively reduce *C* by using a thicker and narrower specimen. One ends up with the TM_{010}–mode (or TM_{020}–mode) cylindrical cavity shown in Figure 32 (c), as discussed below in Section 7.1.6. In such cavities, which typically operate in the range 1 - 10 GHz, the specimen generally takes the form of a rod stretching the full height of the cavity.



(a) H & W cell (b) Re-entrant cavity (c) TM_{010}-mode Cavity

Figure 32. A family of dielectric-measurement resonators. **(a)** The principle of the Hartshorn and Ward method: the admittance cell is resonated with an inductor. **(b)** The *re-entrant cavity* is a coaxial cavity with a capacitance gap in its inner-conductor where the dielectric specimen is placed. The inductance *L* is that of the magnetic field in the region between the inner- and outer-conductors. **(c)** The TM_{010}-mode cavity can, in principle, be seen as a re-entrant cavity that has its inner-conductor fully retracted so that only a cylindrical cavity remains. This reduces the capacitance, *C*, of the specimen, which in the TM_{010} cavity typically takes the form of a rod, as shown.

► ***Summary: The Resonant 2-T technique***

Brief Summary: Main Advantage of the Resonant 2-T Technique. Better resolution for low loss than impedance analyser methods (typically at least 10 times better than good impedance analysers).

Brief Summary: Disadvantage of the Resonant 2-T Technique. Generally far less accurate for ε' than the 3-T methods described in Section 7.1.1, uncertainties greater than ± 2% must be expected even for low ε', though use of computer modelling of fringing-fields can mitigate this limitation.

Resonant 2-T admittance cells: Typical Sources of Dominant Uncertainties:

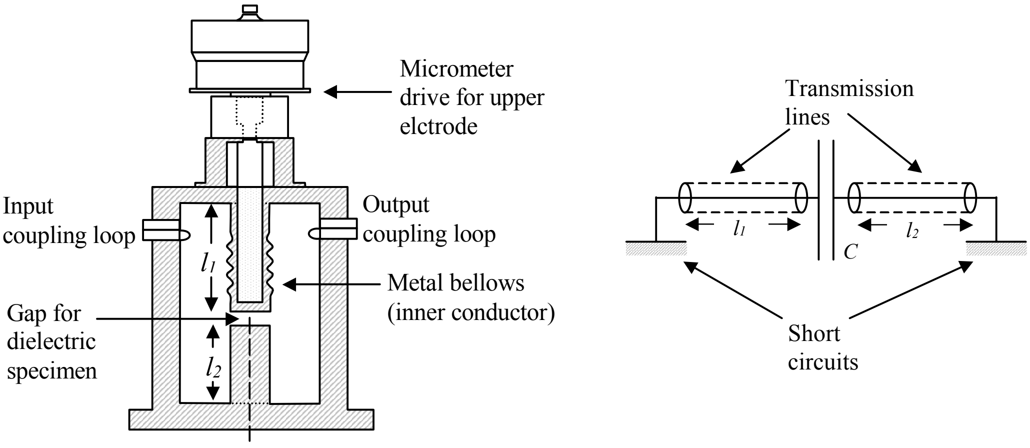
For both ε' and tan δ:

The effects of fringing-fields (if they are not compensated by computer modelling)
All of the effects listed for 3-T methods at the end of 7.1.1

7.1.5 Re-entrant or Hybrid Cavities.

A re-entrant cavity, otherwise known as a *hybrid cavity*, is shown in Figure 32 (b) and in greater detail in Figure 33 (a). These cavities were a natural development from the H & W technique in the 1940s and 1950s (see e.g. [145]), their purpose being to extend that method up to higher frequencies. However, such cavities are still very much in use for low-loss measurements in the frequency range 100 – 1000 MHz, since other microwave resonator designs require exceedingly large resonators at these frequencies. The re-entrant cavity takes the form of a coaxial line short-circuited at both ends (the top and bottom in Figure 33 (a)) with a series air-gap in the inner-conductor where the disc specimen is placed. The cavity requires an aperture in the outer-conductor to allow insertion and retrieval of the specimen – however this does not usually affect its performance or its resonant frequency significantly. Special insertion/retrieval tools are usually required to place laminar specimens concentrically into the capacitance gap of the inner-conductor, whilst avoiding the risk of dropping the specimen inside the cavity.

The cavity is resonated in the TEM-mode. Usually only the lowest order axial resonance is used, and its resonant frequencies can be computed approximately from the equivalent circuit of Figure 33 (b). The size of the specimen gap can be controlled by a micrometer, which requires one part (usually the upper part) of the coaxial inner-conductor to be movable. This can be achieved by making part of it from a metal bellows, as shown, or by having a rigid inner-conductor that contacts the top of the resonator electrically via spring fingers. The bellows approach is probably the better as variable contact resistances are avoided. Coupling into such cavities is usually achieved via small loop antennas connected to coaxial terminals. These couple to the circumferential H-field in the cavity which itself loops around the inner-conductor giving rise to the inductance L, as shown in Figure 32 (b).



(a) (b)

Figure 33. A typical re-entrant cavity: **(a)** details of the cavity, **(b)** a simple equivalent circuit (this ignores, e.g. fringing-fields and the loose coupling).

The size of re-entrant cavities depends upon two factors, the cross-sectional dimensions of the specimens to be measured and the desired resonant frequency. Note that unlike H & W test-sets, which use a set of coils of different inductance to achieve a range of resonance frequencies, the inductance of a re-entrant cavity is fixed by its size, so only one fundamental-mode resonance frequency is possible. To cover the band from 100 to 1000 MHz about three or more resonators of different sizes may be needed. But note that because of the slow variation of dielectric properties with frequency exhibited by low-loss materials, it is unlikely that one would need more than three such resonators – operating, say, close to 150, 400 and 900 MHz - to characterise a low-loss dielectric adequately across this band. As in the H & W method, precise resonance frequencies depend upon the capacitance of the specimen. Inner-conductor diameters between 15 and 50 mm have been used with outer-conductor diameters at least three times as large to give the cavity enough volume to achieve a high Q-factor. There is a tendency to use smaller electrode and specimen diameters than in the H & W technique simply to reduce the specimen capacitance to achieve higher resonance frequencies. Cavity lengths may extend to almost a metre at the lowest frequencies down to 100 mm at 1 GHz. Q-factors typically lie in the range 2,000 to 10,000. Higher Q-factors are normally obtained with larger cavities and at higher frequencies.

Traditionally re-entrant cavities have been used purely for loss measurement because these cavities have high fringing capacitances – up to 15% of the total specimen capacitance and so they are not the most accurate devices for measurement of ε' – typical uncertainties being ± 5% for ε' and ± 10% for $\tan\delta$. However, an uncertainty of ± 10% for $\tan\delta$ is often useful, whereas ± 5% for ε' is typically not! One can get much better uncertainty for ε' in the frequency range 100 MHz to 1 GHz by measuring ε' accurately by other methods at, say, 1 MHz and/or 10 GHz and extrapolating ε' upwards or downwards, respectively, using Equation 2.21, Section 2.5, see also Section 5.3.2. However, this traditional approach to re-entrant cavity usage is being revised in recent years. Computer modelling of the fields inside re-entrant cavities by discretised techniques such as FDTD and FI is relatively simple using commercial modelling packages. There is considerable potential for improving the accuracy for both ε' and $\tan\delta$ by this technique because all of the fringing-fields can be modelled.

Filling-Factors and Metal Loss in Re-Entrant Cavities As mentioned in Section 6.1.9 commercially available FDTD and FI packages are also invaluable for calculating filling-factors and it is appropriate at this point to discuss some important issues relating to these parameters. In the H & W technique, Section 7.1.4, the filling-factor of the resonator has traditionally been taken to be exactly 1 because most of the energy in the system can be regarded as being stored in the capacitor at the peak of the oscillation cycle. At microwave frequencies, however, cavity filling-factors significantly less than 1 are common in many resonator designs – see Section 6.4.4 (this observation does not necessarily apply to dielectric resonators, Section 7.1.15, and to other cases where the specimen fills the whole resonator). Perhaps surprisingly in re-entrant cavity measurements, in spite of the smallness of the specimen compared to the rest of the cavity, filling-factors, F_f, also remain close to 1 for the fundamental TEM-mode. As stated above, this is because most of the energy in the cavity is stored in the capacitance gap and the fringing-fields around it at the peak of the oscillation cycle, as in a typical L/C resonant circuit. Typically, computer modelling shows that $F_f \approx 0.95$ if the specimen is the same diameter as the electrodes (i.e. the diameter of the inner-conductor). However $F_f \approx 0.98$ to 0.99 if the specimen has a diameter close to that of the outer-conductor because then the fringing-fields around the capacitance gap, which store typically ~ 4% of the energy of the cavity, also pass through the specimen. Note that if higher-order axial modes are used to obtain resonances at higher frequencies the filling-factors obtained are much smaller because proportionately more energy is stored in the rest of the cavity. Computer modelling also helps us to understand the metal loss in the cavity. In any attempt to model this, remember that the surface impedance R_s of the bellows section of the inner-conductor will be higher than that of the rest of the cavity. As usual, higher Q-factors are obtained if the cavity is constructed from, or internally plated with, low-conductivity metal (copper, silver) - errors in measurement of tan δ should generally be reduced if this is done.

Methods Measurement methods are extensions of those used in the H & W method (Section 7.1.4) and similar considerations apply regarding specimen shape and size. The air-gap method described in Section 7.1.1 can be used for lower permittivity specimens, typically with $\varepsilon' < 5$. Metallised electrodes can be used for high permittivity specimens but the diameter of metallisation should not exceed that of the electrodes, otherwise fields in the cavity will be distorted. Details of measurement methods are described in [59] and [145].

► ***Summary: Re-entrant or Hybrid Cavities***

Advantages of Re-entrant Cavies. Better resolution for low loss than other RF methods. Re-entrant cavities are considerably smaller in size than other resonators in the frequency range 100 MHz – 1000 MHz.

Disadvantage of Re-entrant Cavities. Generally far less accurate for ε' than other available techniques: uncertainties greater than ± 5% must be expected even for low ε', though use of computer modelling of fringing-fields can mitigate this limitation.

Re-entrant Cavities: Typical Sources of Dominant Uncertainties:

For both ε' and tan δ:
The effects of fringing-fields (if they are not compensated by computer modelling).
All of the effects listed for 3-T methods at the end of 7.1.1.

For tan δ:
Errors in the estimation of filling-factors (unless they are explicitly obtained by computer modelling).
Errors arising from metal losses in the cavity.

7.1.6 $\mathrm{TM}_{010/020}$-Mode Cavities

The TM_{010}–mode cavity, Figure 34 (a), has already been introduced at the end of Section 7.1.4 (see Figure 32 (c)) as one of a family of resonators that also includes the Hartshorn and Ward dielectric test-set and the re-entrant cavity (Section 7.1.5). The last subscript '0' in TM_{010} indicates that the strength of the E and H-fields does not change as one moves from top to bottom along the axis of the cavity. Otherwise the axial E-field is at a maximum on the axis of the cavity and falls away to zero amplitude on the cylindrical side-wall, as required by the electrical boundary conditions there. As in the re-entrant cavity, the H-fields loop around the axis of the resonator and so the coupling arrangements are similar. Specimens normally take the form either of rods [146] or else tubes, which can be used for containing liquid dielectrics [59, 147, 148]. It is usual for specimens to reach from top to bottom of the cavity, but this is not necessary [4]. The frequency of resonance is determined by the radius, a, of the cavity: $f_r = j_{01}c / 2\pi a = 2.405c / 2\pi a$, where j_{01} is the first zero of the Bessel Function J_0 and c is the speed of light. TM_{010}-mode resonators are normally used in the frequency range from about 2 to 10 GHz, but they become inconveniently large at lower frequencies. Unloaded Q-factors in the range 2,000 – 10,000 are typical. Q-factors improve as the cavity length-to-radius ratio l/a increases [149], though ever more slowly when l becomes significantly longer than a. In spite of this, it is common for the length of the cavity to be made comparable with the radius, because very long cavities normally require very long specimens and this can be inconvenient. The cavity length should also be chosen so that the TM_{010}-mode resonance is well separated in the frequency domain from that of other resonances so as to avoid the problem of mode-coincidences and degeneracy [*ibid.*] see Section 6.1.9.

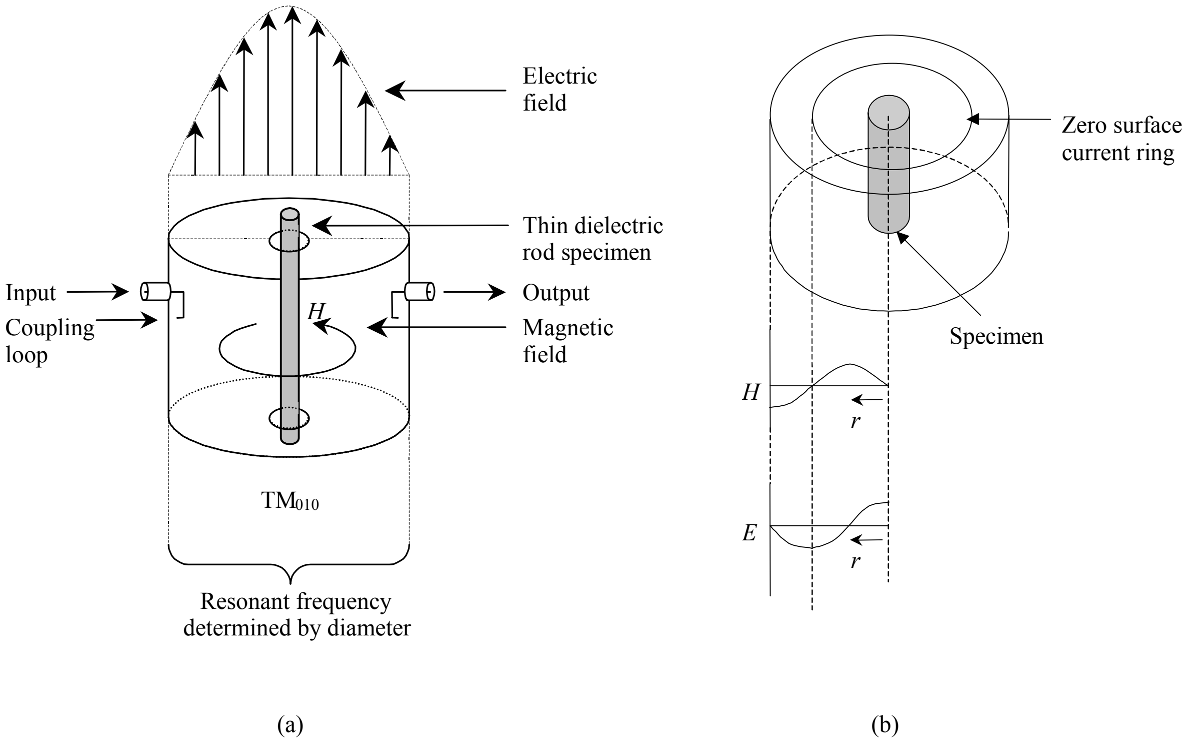


Figure 34. (a) A TM_{010}-mode cavity resonator containing a dielectric rod specimen, which in this case is introduced though two small holes (not shown to scale) on the axis of the resonator. **(b)** A TM_{020}-mode cavity resonator, showing the position of the circle on the top of the resonator where there are no surface currents, this is the best place to make a break in the surface if a lid is needed (After [4]).

From the very beginnings of its exploitation in dielectric measurements [150, 151] this method has been used as a *perturbation method*, see Section 7.1.16. If perturbation computations are used, the diameter of the specimen must be small compared with that of the resonator because the assumption is made that the field-strength E is constant inside the whole volume of the specimen, whereas, in fact, it falls as one moves away from the cavity axis. However, more recently, advantage has been taken of the relatively simple internal geometry of the TM_{010}-mode cavity to employ modal analysis to derive exact equations for the cavity [137]. If this is done, the diameter of the specimen need not be restricted. Similar advantages accrue if discretised FDTD or FI modelling packages are used for the modelling - such packages are available commercially (see Section 6.9).

There remain advantages to using narrow diameter specimens, however. One is that both *medium-* and *high-loss* specimens may be measured by this technique if their diameter is sufficiently small. Furthermore, if a small hole is inserted *on-axis* at the top and/or the bottom of the cavity, as shown in Figure 34 (a), specimens can be introduced through it. This makes it unnecessary to have a door or lid in the cavity to introduce specimens. Small holes in the top and/or bottom of the cavity do not significantly perturb its performance, so this can greatly improve the convenience of the technique. However, perturbations become significant if the hole diameter exceeds about 5% of the cavity diameter. For larger diameter specimens a replaceable door or lid in the cavity becomes essential. However, in TM_{010}-mode cavities, this can introduce a far more serious metrological disadvantage than the mere inconvenience of having to remove the lid to insert or remove the specimen. Specifically, in Section 6.1.9 a recommendation is made that cuts or breaks in the metal wall of the resonators should be put in positions where they are not crossed by the electrical currents of the resonant mode in the walls, otherwise contact impedances reduce Q-factor, and, worse, they tend not to be reproducible. Thus, whenever a lid/door is removed to introduce/remove the specimen, contact impedances can change, introducing measurement errors for tan δ. Unfortunately, with the TM_{010}-mode it is impossible to find *any points or lines* in the walls of the cavity (except the two points on the axis of the cavity) where currents do not flow. There are radial currents, for example, at all points on the flat ends of the cavity, so contact impedance irreproducibility errors are endemic to the method. Fortunately this problem can be avoided by using the *TM_{020}-mode* rather than the TM_{010}-mode, as explained next.

<u>TM_{020}-mode Cavities.</u> The radial field amplitudes of the E- and H-fields in a TM_{020}-mode resonator are shown in Figure 34 (b) – the E-field may be compared with that of the TM_{010}-mode cavity as shown in Figure 34 (a). The resonant frequency of the TM_{020}-mode is given by $f_r = j_{02}c/2\pi a = 5.520c/2\pi a$, where j_{02} is the *second* zero of the Bessel Function J_0 and c is the speed of light in free space [149], so for the same resonant frequency a larger diameter is needed for a TM_{020}-mode cavity than a TM_{010}-mode cavity. It will be seen from Figure 34 (b) that there is one radius in the TM_{020}-mode cavity at which the E-field is zero. There is also a radius at which the H-field is zero and this is also a radius at which there is *no radial current.* If a lid or door having this diameter is placed concentrically in the top face of the cavity, its contact impedances will not affect the currents in the face of the cavity and so the Q-factor will not be affected by contact impedances - see Anderson [4], Ch. 2. This principle was tested by Rosenberg [149] at NPL and found to work well, thus overcoming one major disadvantage of TM_{010}–mode operation.

Designating a cavity as a 'TM_{010}' or a 'TM_{020}'-mode resonator makes it appear that it can only be used in one resonant mode and at one frequency – so that a range of cavities may be required to cover a broad band of frequencies. However (apart from the lid-contact impedance issue), it is clear that a single cavity may be made to resonate in the TM_{010}, TM_{020} …or any TM_{0n0} mode – thereby affording a degree of frequency coverage. The only problem with this working scheme is that cylindrical cavities can resonate in many other modes too. Mode density rises with frequency, which makes it difficult to identify the correct mode, and mode-coincidences can cause significant errors (Section 6.1.9).

<u>Perturbation Equations vs. Full Numerical Analysis of *TM_{010}-mode TM_{020}-mode Cavities.*</u> Details of the advantages of perturbation techniques are given in Section 7.1.16. One advantage of the first-order perturbation theory, however is that relatively simple measurement equations can be used. Thus, with TM_{010}-mode and TM_{020}-mode cavities the following two equations can be used for deriving ε' and ε'':

$$\varepsilon' = 1 - a\frac{\Delta f_r}{f}.\frac{V_0}{V_s} \qquad \ldots 7.4(a)$$

$$\varepsilon'' = b.\frac{V_0}{V_s}.\left(\frac{1}{Q_l} - \frac{1}{Q_0}\right) \qquad \ldots 7.4(b)$$

Where a and b (= $a/2$) are constants that depend upon the mode, f_r is the resonant frequency without the specimen and Δf_r is the change in resonant frequency obtained on introduction of the specimen (note that it must be negative for $\varepsilon' > 1$). Q_0 is the unloaded Q-factor of the cavity without the specimen and Q_l is the loaded Q-factor with the specimen. V_0 is the volume of the cavity and V_s is the volume of the specimen. For the TM_{010}-mode the numerical constants in these equations are [4, 151] $a = 0.539$, $b = 0.269$. For the TM_{020}-mode $a = 0.2316$ and $b = 0.1158$ [149]. The main disadvantage of perturbation equations is that they are *approximations* and will not generally apply if large-diameter specimens are used. In the case of these TM cavities one of the main reasons for this is that the E-field will not be uniform in the specimen, as assumed by these perturbation equations. For specimens that perturb the cavity too much (for example, $|\Delta f_r|/f_r$ and $|\Delta Q|/Q > 10\%$, depending on desired uncertainty) only a full modal or discretised E.M.-field analysis will suffice (see Section 6.9).

<u>Measurements.</u> Irrespective of whether these cavities are used with a perturbation technique or with computer modelling, the measurements to be undertaken are the same – and are similar to those taken in all fixed geometry cavity techniques – i.e. the resonant frequency and Q-factor are measured with and without the specimen present. ε' is computed from the change in resonant frequency and tan δ from the change in Q-factor.

► ***Summary: $TM_{010/020}$-Mode Cavities.***

<u>Advantages of $TM_{010/020}$-Mode Cavities.</u> Unlike most other standard microwave techniques, these cavities are suitable for measuring *rod* specimens. They are also suitable for liquid measurements. Narrow specimens can be introduced easily through a small holes on the axis of the cavity and liquids can be circulated through a tube down the axis of the resonator to measure changes of ε^* in real time. These cavities may therefore be used cost-effectively for production monitoring of materials (see Section 7.2.1). Medium and high-loss specimens can be measured by the perturbation method, provided the specimen diameter is small enough to avoid excessive loading of the Q-factor.

Disadvantages of $TM_{010/020}$*-Mode Cavities.* Unless one actually wishes to measure rod or tube-shaped specimens, specimen shape may be a disadvantage (though shorter specimens can be used [4]). Uncertainties for ε' may be ± > 1% if a perturbation method is used.

$TM_{010/020}$ *Cavities: Typical Sources of Dominant Uncertainties:*

For ε':

Approximations in the theory if a perturbation analysis is used.
Ability to measure Δf_r if specimens are small.

For tan δ:

Approximations in the theory if a perturbation analysis is used.
Ability to measure ΔQ if specimens are small or of low loss.
In TM_{010}–mode cavities, repeatability of contact impedances if a lid or door in the cavity has to be opened to insert specimens. This can be avoided by using a TM_{020}-mode cavity.

► ***Do's and Don'ts for*** TM_{010} ***and*** TM_{020} ***-Mode Cavities***

If using the perturbation theory ***do*** check that that its approximations are valid by measuring a specimen of a dielectric reference material with already-known properties and with roughly the same size and dielectric properties as the dielectrics under test. This should provide useful information that can be entered into the uncertainty budgets for the measurements.

Don't just assume that the perturbation approximations are valid, check them!

7.1.7 TE_{01}-Mode Cavities

The TE_{01}-mode cavity [152, 153], see Figure 35, is used for measurements on laminar low-loss specimens, typically in the 8 – 40 GHz range. Strictly speaking, TE_{01}-mode cavities should be referred to as TE_{01n}-mode cavities, where n is the number of antinodes (i.e. the number of half-wavelengths) of the resonant E-field along the cavity axis on resonance. However, in practice we are not restricted to a single value of n. As measurements using different values of n are often used for cross-checking purposes, the 'n' is often dropped when referring to this method, as we do in this Guide. These resonators have a high resolution for low losses and they are typically used for measuring cylindrical specimens that notionally have the same diameter as the resonator. The technique can be used across a range of temperatures [154]

The electric fields in the resonator are circularly or ('azimuthally') polarised with respect to the resonator (z-) axis, as in dielectric resonators (Section 7.1.15). Tuning can be achieved easily, as shown in the figure, by changing the resonant length of the cavity with a micrometer-driven metal piston. Typically, the cylindrical dielectric specimen sits on the piston during measurements. Electrical contact problems around the circumference of the piston (and the errors in loss measurement they would induce) do not occur because the currents in the cylindrical wall of the cavity are purely circumferential (they have no z or radial components), so there would be no current flowing between the piston and the cylindrical walls of the cavity even if they did touch. Furthermore, as the E-field falls to zero on the outer wall of the resonator, the specimen itself need not be an exact fit to the diameter of the cavity. Typically the specimen can be up to 0.5 mm smaller in diameter in a 50-mm diameter cavity without having a significant effect on the measurement accuracy.

One potential problem with the TE_{01}-mode in a circular cylinder is that it is *always degenerate with* (i.e., see Section 6.1.9, it always resonates at the same frequency as) the TM_{11}-mode. The latter has a very different field pattern that the measurement equations of the method do not take into account, so a method must be found for filtering out the TM_{11}-mode. One of the most effective was implemented at NPL in the 1970s [152, 155]. The cylinder of the cavity was manufactured from *helical waveguide* which had originally been manufactured for long-distance trunk waveguide communications (before this function was usurped by optical fibres). The metal wall of the helical waveguide was formed from a very tightly-wound insulated wire embedded into an external epoxy binder to give it strength. In manufacturing the waveguide, the wire was wound accurately onto a metal mandrel having the same diameter (typically 50 or 60 mm) as the required internal diameter of the finished waveguide. Once the epoxy was set, the mandrel was removed carefully from the finished waveguide cylinder. Such a waveguide can only support circumferential and *not* axial currents in its cylindrical walls and this effectively filters out the TM_{11}-mode. Unfortunately, such waveguides are no longer manufactured on a regular basis. However some stocks of the waveguide from the 1970s still exist.

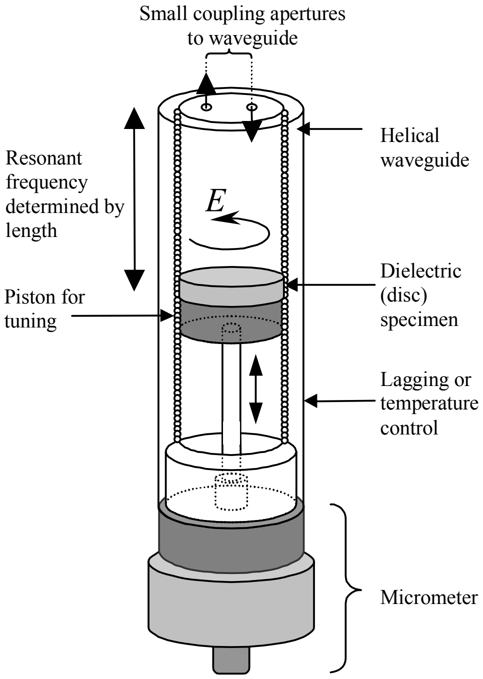


Figure 35. A micrometer-tuned TE_{01}-mode cavity for dielectric measurements.

Coupling into the cavity is typically from waveguide via small coupling holes, as shown in Figure 35, delivering a high insertion loss of the cavity on resonance – the general guidance of Sections 6.1.9 and 6.4.3 can be followed here. In principle, the same cavity geometry can be used over quite a broad frequency band, e.g. the 50-mm-diameter cavities used at NPL have operated from 8 – 35 GHz, but the bandwidth of any one cavity is likely to be limited to a single waveguide band by the coupling mechanism and the size of the coupling holes. As in many resonant techniques ε' can be measured by a length-change/equivalent-thickness method, see Section 6.5. Specimen loss is computed from the change in Q-factor with and without the specimen, see Section 6.1.9. Measurement equations and further measurement details are given in the references cited above.

Though it may be less convenient to use than other resonant methods (e.g. those described in Sections 7.1.8 and 7.1.17), because the specimen has to be placed in and removed from the cavity, the TE_{01}-mode cavity method has proved over the years to be both accurate and sensitive for very low loss specimens which have loss angles below 100 μradians (see, for example, [73]). This is typically because most of the cavity is filled with air-dielectric, so that, for example, unloaded Q-factors may be as high as 60,000 at 10 GHz for the mode with $n = 9$ in a 50-mm diameter cell. Dielectric resonator techniques (Section 7.1.15) are probably the most sensitive and arguably the most accurate for such low losses, but they are optimised for puck-shaped specimens, whereas the TE_{01}–mode cavity is more suitable for laminas. Another advantage that the TE_{01}-mode method has over the dielectric resonator method is that it uses a true substitution technique, so that one can measure the metal losses in the empty cavity *at the same frequency* as that at which the specimen is measured. This is typically not possible in dielectric resonator cavities and can introduce systematic errors in the latter technique. Finally, the fact that the cavity is *tunable* by use of the piston, gives access to a wider range of measurement frequencies and, at any one frequency, also allows the specimen to be measured with more than one axial mode, say both $n = 8$ and $n = 9$, to allow cross-checking of results to improve confidence.

Specimens should ideally be an integral number of half guide-wavelengths thick, not just because this renders the measurements insensitive to surface contamination on the specimen – see Section 6.6.2 – but because it can be shown from the measurement equations that this is the condition that gives best uncertainties.

Experience over many years at NPL with such a cavity operating at 10 GHz shows that resolutions for loss angles may be as low as 10 μradians, while uncertainties for $\varepsilon' < 5$ can be as low as ± 0.5% for 'ideal' specimens (i.e. flat homogeneous low-loss specimens which are an integral number of half wavelengths thick). Permittivities up to 40 can be measured with uncertainties of the order ± 1% or better. The technique can be extended to liquid measurements [155]. The liquid should be contained in a cell in a part of the cavity remote from the coupling holes. A low-loss quartz-crystal window can be used to contain the liquid. The thickness of the liquid can be easily changed if the piston is allowed to move through the liquid [*ibid.*]. This gives one an extra degree of freedom which can help in the elimination of systematic errors.

► ***Summary: TE$_{01}$ –Mode Cavities.***

Advantages of TE$_{01}$–Mode Cavities. High sensitivity for low-loss materials. A substitution technique can be used, allowing metal losses in the cavity to be measured at the same frequency as the dielectric properties (compare dielectric resonators, Section 7.1.15). This is one of the more accurate methods for permittivity at microwave frequencies.

Disadvantages of TE$_{01}$–Mode Cavities. Suitable only for low-loss specimens, typically $\tan\delta < 0.003$. Not suitable for measurements of anisotropy. Specimens must have a circular cross-section close to that of the cavity. Best uncertainties are achieved only if the specimen is an integral number of half-guide-wavelengths thick.

TE$_{01}$–Mode Cavities: Typical Sources of Dominant Uncertainties:

For ε':
- Specimen thickness measurements, especially if the specimen is not an integral number of half-wavelengths thick or if it is uneven or warped.
- The resolution of the measurement of the displacement of the micrometer, particularly if the loaded Q-factor is low.

For $\tan\delta$:
- Contamination of specimen surfaces, especially if the specimen is not an integral number of half-wavelengths thick.
- Contamination of the cavity or uneven conductivity in cavity walls.
- Ability to measure Q-factor.

► ***Do's and Don'ts for TE$_{01}$ –Mode Cavities***

Do remember that the guide-wavelength in the cavity is not the same as in free-space so that a specimen that is the ideal integral number of half-wavelengths thick in free space will not be so in the cavity.

Don't try to use specimens whose diameter is too close to that of the cavity – they may become jammed and so damage the cavity! A gap around the specimen of 0.1 – 0.5 millimetre in a 50-mm diameter cavity will ensure that the specimen cannot get stuck and will have virtually negligible effect upon the measurement.

7.1.8 Split-Post Dielectric Resonators

The split-post dielectric resonator (SPDR), Figure 36, was developed by Krupka and his collaborators [156] and is one of the easiest and most convenient techniques to use for measuring microwave dielectric properties. It uses a fixed-geometry resonant measurement cell for characterising low- or medium-loss laminar specimens, such as substrates and thin films, in the frequency range 1 - 36 GHz. The main drawback with this technique is that each SPDR can only operate at a single fixed frequency. However, the simplicity of the measurement procedure and the relatively low cost of manufacture of the resonator means that it is practicable to measure the same specimen in several SPDRs operating at different frequencies.

The basic geometry of an SPDR is shown in Figure 36. Two identical dielectric resonators (see Section 7.1.15) having the usual cylindrical disc or 'puck' shape are placed coaxially along the *z*-axis so that there is a small laminar gap between them into which the specimen can be placed to be measured. By choosing suitable dielectric materials for the pucks the resonant frequency and Q-factor of the SPDR can be made to be temperature stable. Once a resonator is fully characterised, *only three parameters* need to be measured to determine the complex permittivity of the specimen: its *thickness* and the changes in *resonant frequency*, Δf, and in the *Q-factor*, ΔQ, obtained when it is placed in the resonator.

Specimens are typically 1 mm in thickness or less, but may be thicker at lower frequencies, their diameter depending on frequency. Thus, larger-diameter specimens are needed at lower frequencies, typically over

50 mm at frequencies below 2 GHz but as small as 30 mm at 10 GHz, the minimum diameters being determined by the diameters of the constituent dielectric resonators. The specimen thickness must clearly be less than that of the gap. This is typically 1 mm at 10 GHz but can be as high as 5 mm below 2 GHz.

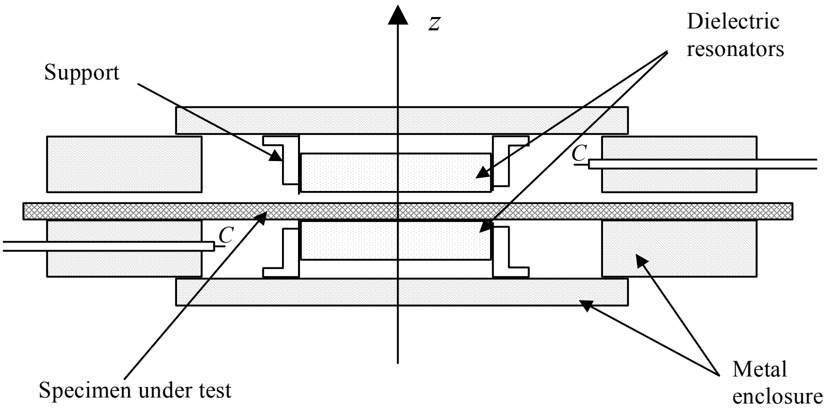


Figure 36. A Split-Post Dielectric Resonator (SPDR) cell for dielectric measurements. Transmission coupling is via both dielectric resonators, '*C*' marks the coupling loops.

The two dielectric pucks resonate together in a *coupled resonance* in the $TE_{01\delta}$-mode and so a circularly-polarised evanescent E-field exists in the gap region between them. If the resonators were to be well-separated and each were resonating alone, the azimuthal field above and below their plane faces would decay *exponentially* with distance from the resonator. However, as the two resonators are very close together – usually the separation is in the range 1 to 5 mm - the field magnitude in the gap between them is approximately *parabolic* as a function of z, with a shallow minimum in the centre of the gap. The variation of maximum to minimum E-field magnitude as one moves vertically in the gap may amount to ~ 10%, but this variation magnitude depends on the resonator design. The numerical analysis that is used to compute the permittivity and loss of the specimen in this system assumes that the specimen is sitting on the lower dielectric resonator and explicitly takes account of this field-magnitude variation. The fact that the field magnitude across the specimen gap varies only by a small amount reduces the need for specimens to be completely flat, which is an advantage, as this can be difficult to achieve with some types of laminar material.

In the specimen gap, the circularly-polarised azimuthal E-field is continuous in value across the air/dielectric interface above the specimen. An attractive feature, which follows from this fact, is that the sensitivity for measuring the properties of thin films can be increased by stacking a number of them together. Their *total* thickness, excluding air-gaps, should be used to compute ε' and tan δ. Another use for this feature is for the measurement of ε^* of a coating or a film on a substrate. Δf and ΔQ are measured both with the substrate alone – to give Δf_s and ΔQ_s - and with the coated substrate – to give Δf_c and ΔQ_c. Then ε^* for the coating can be easily computed from its thickness, by using (Δf_c - Δf_s) for Δf in the subsequent computations and (ΔQ_c - ΔQ_s) for ΔQ. A correction for the variation of E-field magnitude across the gap ought to be made in this case, unless uncertainties of the order ± 10% are acceptable. These features of the SPDR and its general ease of use make it one of the most convenient techniques for laminar and thin-film specimens at microwave frequencies.

The geometry of the system is such that simple analytical models cannot be used to relate ε^* to Δf and ΔQ, so numerical solutions must be employed. The geometry of the SPDR lends itself to modelling by mode-matching or Finite Difference (FD) techniques (see Section 6.9). Note that this modelling can be done comprehensively for quite large values of Δf and ΔQ (i.e. large compared with f and Q, respectively) so the method is *not* inherently a perturbation method (see Section 7.1.16), as the approximations used in perturbation equations can be avoided in the numerical analysis. This feature allows a wider range of specimen complex permittivities to be measured than would be possible with a perturbation technique.

The main limitation of the technique is that its resolution for very low losses is reduced by the Q-factors of the two dielectric resonators it employs, but it can typically be used to measure loss angles down to 100 μradians with reasonable accuracy. The dominant measurement uncertainties are the thickness of the substrate and the accuracy of Q-factor measurements. Typical uncertainties can be ± 1% for ε' up to 10 and ± 5% for tan δ. A conference paper [157] gives details of studies that show that measurements on reference specimens in SPDRs agree well with those carried out by other well-established methods. For further information, see also Section 9.3, which illustrates a typical measurement in an SPDR and discusses some aspects of its uncertainty budget.

► ***Summary: Split-Post Dielectric Resonators (SPDRs)***

Advantages of Split-Post Dielectric Resonators (SPDRs). They provide one of the most convenient and cost-effective methods available for measuring laminar specimens at microwave frequencies: fast measurements are possible, as is easy-access for specimens. The method is suitable for thin films, including dielectric coatings and surface films, and also, to some extant, for warped and non-flat specimens.

Disadvantages of Split-Post Dielectric Resonators (SPDRs). Each SPDR is limited to a single frequency range of measurement and is suitable only for laminas. It is typically not suitable for very low losses < 100 μradians.

Split-Post Dielectric Resonators (SPDRs): Typical Sources of Dominant Uncertainties:

For ε':
    Ability to measure Δf_r if specimens are small.
    Approximations in the theory.

For tan δ:
    Ability to measure ΔQ if specimens are small or of low loss.
    Approximations in the theory.

7.1.9 Substrate Methods, including Ring Resonators.

Substrate and printed-circuit manufacturers need to know the dielectric properties of their substrates and special techniques have been developed to enable them to measure these properties with the facilities commonly at their disposal. Most notable among these are facilities for depositing metal resonant structures lithographically onto substrate surfaces. Such structures include ring [158, 159], T-shaped [160], and stripline [161] resonators, all of which may be used to measure dielectric properties at a number of harmonically related frequencies across a wide band. The main advantage of this approach is that it gives designers of integrated circuits precisely the information that they need - an effective value for permittivity, ε_e', that is appropriate for their applications. This is not necessarily the absolutely 'true' value of ε' for the substrate material because, like all measurements, these measurements are subject to systematic errors. However, if one is using a strip-line technique to determine a parameter, ε_e', which is subsequently to be used for designing similar strip-line circuits, some degree of beneficial compensation of errors must occur.

Metrological problems can arise in these techniques if one is interested in measuring loss. High-temperature processes used in manufacturing can cause deposited metal to in-diffuse into a substrate, so measured properties may differ from those of pure bulk material. The loss tangent of low- and medium-loss substrate materials is generally not measurable by these substrate techniques because radiative and conductivity losses (surface and bulk) in the deposited metal structure tend dominate: in practice the measured Q-factors can be below 100.

► ***Summary: Deposited Resonant Structures on Substrates***

Advantages of Deposited Resonant Structures on Substrates Well suited for providing circuit designers with a parameter, ε_e', which is particularly relevant for circuit design.

Disadvantages of Deposited Resonant Structures on Substrates One needs access to metal film deposition facilities. Measured permittivity may have significant systematic errors if the software analysis on which it is based is only approximate. The method is not suitable for low-loss measurements.

Deposited Resonant Structures on Substrates: Typical Sources of Dominant Uncertainties:

For both ε' and tan δ:
    Imperfect computer models.

For tan δ:
    In-diffused metal in the substrate.
    Radiation losses.
    Metal conductivity losses.
        … all tending give erroneously high apparent loss.

7.1.10 Coaxial Transmission Line

Introduction. This method makes use of *annular* specimens, which should fit closely between the inner and outer-conductors of the coaxial conductors of precision coaxial air-line, as shown in Figure 5. The air-line should ideally be fitted with Laboratory Precision Connectors [112], which allow for a well-matched coaxial connection to an ANA. The complex permittivity and permeability of specimens is computed from the S-parameters of the specimen, as measured by the ANA. Similar techniques apply with other types of uniform air-line - the use of rectangular waveguide for such measurements is very common and is described in Section 7.1.11.

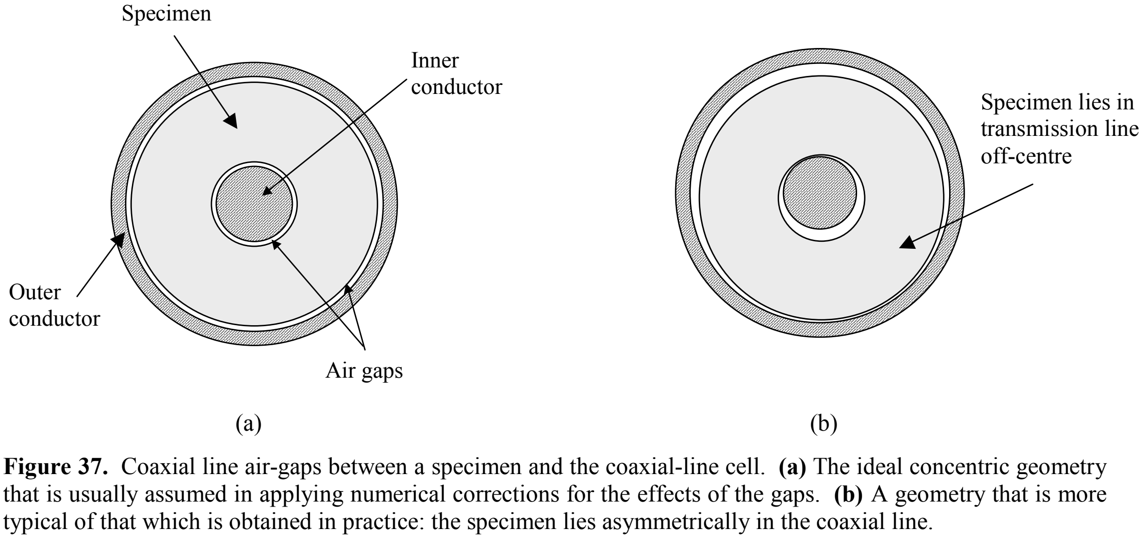
For many years, until the advent of ANAs, air-line measurements were largely restricted to one-port measurements - the Roberts & Von Hippel method is typical [162] - in which the specimen is placed hard up against a short-circuit and its reflection coefficient is measured. Such one-port techniques may still be recommended in many instances (see below) but there are advantages to be gained from measuring both reflection *from* and transmission *through* specimens in a matched transmission line, as illustrated in the worked example in Section 9.7. For purely dielectric specimens typically up to 10 mm in length, one typically finds that reflection coefficient measurements tend to be more accurate at lower frequencies (< 500 MHz), while transmission measurements tend to be more accurate at higher microwave frequencies (although one should not over-generalise here). The combination therefore allows broad-band measurements to be performed on just one specimen from, say, 100 MHz to 18 GHz in a 7-mm diameter air-line, taking advantage of the best uncertainties from both methods. Reference [79] presents a comparative uncertainty analysis of the different measurement options presented here (e.g. transmission in matched line, reflection in matched line, reflection from a shorted line, i.e. Roberts & Von Hippel, etc.). The Reference is concerned with dielectric *liquid* measurements but the analysis is just as relevant for solid dielectric materials. For *magnetic* materials, both transmission and reflection data is required at all frequencies, though reflection-only measurements are possible by moving the specimen in the line, see Section 5.3.5.

Inevitably for solid specimens metrological problems arise from the presence of air-gaps around the specimen that lie between it and the inner and outer-conductors of the line, see Figure 37. They *dilute the apparent permittivity* obtained from the measurement but, more seriously for accurate measurements, they also help to launch *higher order modes* - both effects give rise to significant measurement errors, as explained below. Three practical approaches can be taken to reduce these problems:

(i) Make the gaps as small as possible by means of accurate high-tolerance machining.

(ii) Make corrections for them in the measurement equations. For this, the size of the air-gaps needs to be known. They can be estimated by the use of air-gauging – see Section 6.3.2.

(iii) Remove them by surrounding the specimen with a ‘solder’ - this forms the basis of the ‘UTE’ method described below – or else use a liquid-immersion technique, see Section 7.1.2.



(a) (b)

Figure 37. Coaxial line air-gaps between a specimen and the coaxial-line cell. **(a)** The ideal concentric geometry that is usually assumed in applying numerical corrections for the effects of the gaps. **(b)** A geometry that is more typical of that which is obtained in practice: the specimen lies asymmetrically in the coaxial line.

Air-gap problems do not usually arise for liquid measurements, but a well-designed liquid cell is required instead [79]. The liquid must be contained between solid dielectric windows, as shown in Figure 38, so a multi-layer theory based upon cascaded two-ports is necessary for the S-parameter analysis of the specimen/cell combination.

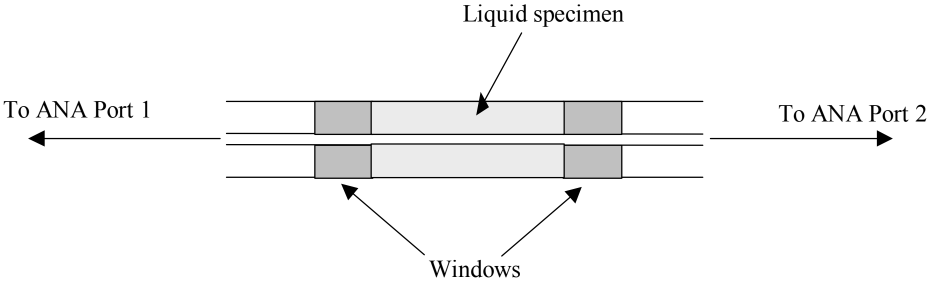


Figure 38. A coaxial-line cell for measuring liquid dielectrics.

These transmission-line methods are often the most cost-effective choice for (i) broad-band measurements, for (ii) magnetic materials, for (iii) medium- to high-loss materials and (iv) for materials that are only available in small volumes. Larger-diameter coaxial lines can be used to improve uncertainty at lower frequencies. For example, 14-mm precision air-line can be used over the range 50 MHz - 6 GHz, while 7-mm precision air line is suitable for 100 MHz to 18 GHz. Uncertainties for ε' can be as low as ± 1% for low-permittivity materials if a correction for air-gaps is made but it may be higher than ± 5% for high-permittivity materials, so use of other methods is advisable if more accurate measurements are required (e.g. those of Sections 7.1.1, 7.1.7, 7.1.15 and 7.1.17). This is a *non-resonance* method, so if *loss* is to be measured, specimens of lower loss (e.g. tan $\delta < 0.05$) are probably better measured by a resonant method (e.g. those of Sections 7.1.1, 7.1.3, 7.1.7 - 7, 7.1.15 and 7.1.17), though the present method *can* be used for measuring ε' for low-loss specimens, see below.

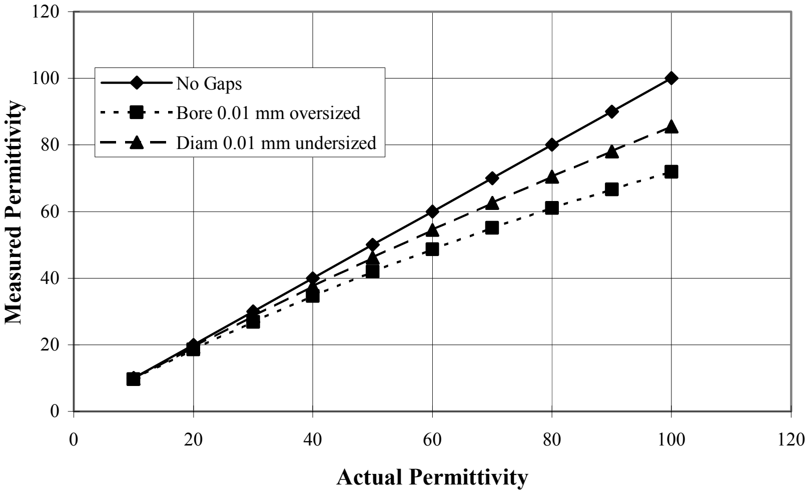


Figure 39. Discrepancies obtained for ε' as measured by the coaxial air-line technique if air-gaps of the type shown in Figure 37 are not compensated for. The data has been computed for 7-mm 50-Ω coaxial line. The two dashed traces correspond respectively to the two cases where (i) the hole bored through the specimen (through which the inner-conductor fits) is 0.01-mm *over*-sized, (ii) the outer diameter of the specimen is 0.01-mm *under*-sized. Note that the errors are greater for the former case.

The transmission line method is best suited to loss tangents above 0.05 and permittivities below 100, though the *resolution* for loss tangent may be as low as 0.02. But it should be emphasised that very poor uncertainties can arise (i) with high values of ε' (above ~ 20), (ii) at the highest and lowest frequencies listed above, and (iii) for low loss tangent. The best uncertainties for permittivity measurements upon solid specimens, below ± 2%, are quite difficult to achieve. The machining of such specimens must be done to the highest level of accuracy to obtain them. This applies especially to the sizing and positioning of the hole for the inner-conductor in the centre of the specimen. Figure 39 demonstrates for 7-mm 50-Ω coaxial line the magnitude of the error that can arise if air-gap corrections are not made. The Figure can also be used to estimate the magnitude of uncertainties introduced when the actual size of the air-gaps is uncertain - see the worked example in Section 9.7. Such machining can be expensive and this may justly be regarded as a disadvantage of the method. As ever, achievable uncertainties depend critically upon specimen properties and specimen preparation (see Section 5.4).

Variable temperature measurements are possible, but the coaxial-line method has a *major disadvantage* if such measurements are to be performed in a temperature-controlled cell: the coaxial metal inner-conductor provides a *mechanically inaccessible* highly thermally-conducting path out of the cell, making uniform temperature control very difficult! This can be overcome by using thin-walled precision stainless-steel air-lines as thermal isolators immediately outside the liquid cell, see [37], but such solutions are expensive. For these reasons one may prefer a waveguide technique for variable-temperature measurements (Section 7.1.11) - at least the metal waveguide outer wall is accessible for thermal control. A *reflection* air-line method is another option - see below.

Theory and Standard Methods. NIST Technical Note 1341 [83] provides an excellent guide to the theory of this method as does the NIST follow-up work by Baker-Jarvis and his colleagues [163, 164]. The paper by Jenkins *et al.* [79] explains how uncertainties can be computed in liquid measurements. With the exception of the uncertainties caused by air-gaps, this treatment can be extended to solids. The NIST Technical Note provides the formulae which correct for air-gaps, based on a simple capacitative model.

There are two published standard methods for the transmission-line technique: ASTM D5568-01 [165] and a UTE (*Union Technique de L'Electricité*) standard from France [166]. ASTM D5568-01 gives a general procedure for transmission-line measurements, including measurements on magnetic materials and measurements in waveguide, see Section 7.1.11. Procedures for the processing of data and practical measurements are also given, but one important issue that is not covered is the influence of *higher-order modes* launched by air-gaps, which can create very large errors, see below. Computations based on those of Nicolson and Ross [167] are cited, but they can be unstable [163], producing erroneous results when the length of the specimen is close to one or more half-wavelengths in the medium of the dielectric. A recent paper [168] presents an explicit algorithm which is believed to be more stable. An alternative recommended approach (as used at NPL) is the *iterative* approach presented below. The UTE standard [166] is for coaxial measurements on thin specimens of lossy, non-porous, materials in a special coaxial-line cell. The method gets over the air-gap problems by using a low-melting-point alloy, referred to as 'solder', to fill the gaps. Specimens are machined to the same length as the cell.

Both air-gap and UTE techniques can have their advantages. Because the UTE method avoids higher-order mode resonances, it should be more accurate for higher permittivity specimens over a wider frequency range for the measurement of ε'. It is likely, however that the air-gap method can have better accuracy for the loss of lower loss specimens: longer specimens can be used and there is no potential additional source of spurious loss which the solder metallisation might introduce. Longer specimens should also provide better uncertainties for lower permittivity. A practical problem is that long specimens may be difficult to insert into and remove from airlines - they may, for example - become wedged in. However, with careful machining, and especially if the material is malleable, a number of short specimens may be introduced end-to-end into the line to make up one long *composite* specimen. The effect of small air-gaps *between* them should be small as long as the length taken for the composite specimen is exactly the sum of the lengths of the individual short specimens, ignoring the length of the air-gaps between specimens. For measurements on porous materials the air-gap method may also be preferred if there is any suspicion of the solder used in the UTE method being absorbed into the specimen.

Calibration of ANAs for Transmission Line Measurements. If conventional transmission lines and connectors are used, two-port ANA calibration may be carried out by the conventional 'TOSL' technique [11, 12, 104, 105] which employs '*Thru*', '*Open*', '*Short*' and '*matched Load*' calibration standards. However, for transmission measurements one also has the option of using a number of other calibration methods. Two of these, the '*Thru-Reflect-Line*' (TRL) and the '*Line-Reflect-Line*' (LRL) methods [104] are particularly appropriate if measurements are performed in non-standard line or in the Free-Field (see Section 7.1.14). Both methods make use of extra lengths of transmission line of the same type that the measurement cell is made from to perform the calibration. The TRL technique is usually programmed-in as a calibration option in modern ANAs, so guidance on its use can normally be obtained from the ANA-manufacturer's literature.

Measurement Equations for Transmission Measurements. Following the principles sketched in Section 2.2.2. the S-parameters of a specimen as shown in Figure 5 can be expressed in terms of its length and its intrinsic material properties: ε^* and μ^*. In measurements, however, we need to perform the converse calculation and derive ε^* and μ^* from the measured S-parameters. One option is to do this explicitly by algebraic manipulation. A number of authors have followed this option. A widely-used set of explicit equations was published by

Nicolson and Ross [167] but, as stated above, they can give rise to unnecessary error for certain lengths of specimen [163] Modified explicit equations, e.g. from [168], may be used if they can be shown to be stable and yield the correct solutions for one's own measurement cell. However, advantages can be obtained by using the *direct* equations that express the S-parameters in terms of ε^* and μ^* instead. They can readily be placed into an *iterative loop*, to obtain ε^* and μ^* from the S-parameters [79]. Usually only 2 - 4 iterations are needed if a Gauss-Newton algorithm is used. The advantages of this iterative approach are fourfold:

(i) The iterative method is stable: experience shows that it yields reliable values for complex permittivity.

(ii) For non-magnetic materials, ε^* for a *homogeneous* specimen can be determined without knowledge of the location of the specimen along the coaxial line by iteration from the S-parameters, as illustrated in the worked example in Section 9.7. This is convenient because measurements of specimen location are not required, and this eliminates a source of uncertainty.

(iii) Perhaps more importantly, for non-magnetic specimens one can chose whether to derive ε^* purely from the *transmission* S-parameters *or* from the *reflection* S-parameters. This can prevent good data from being contaminated by poor data (which the explicit formulae for ε^* tend to do). For example, at microwave frequencies computation of ε^* purely from S_{21} or S_{12} can be much more accurate than computations based on S_{11} or S_{22}, see the illustration in Section 9.7.

(iv) The iterative method also offers a convenient approach for uncertainty calculation.

Whether the specimen is magnetic or not, one should always measure all four S-parameters, if possible. Even if some of them do not contribute to the final result, as in (iii) they can act as a check. In the transmission method both *Forward* and *Reverse* S-parameter measurements should be performed (*Forward*: S_{11} & S_{21}; *Reverse*: S_{22} & S_{12}). If the results do not agree to within expected uncertainties, it may be because of a faulty measurement or else it may be because the specimen is inhomogeneous – so useful information can therefore be gained.

Circumferential Air-Gaps and the Launching of Higher Order Modes. As described in Section 6.3.2, *air-gauging* is an effective way of estimating the size of air-gaps with an uncertainty of a few microns. However, even if capacitative corrections are made for air-gaps there are two significant residual sources of error that they introduce. First, in practice, the specimen never lies symmetrically in the coaxial line as shown in the ideal case of Figure 37 (a) but will lie askew or off-axis as shown in Figure 37 (b). Furthermore, the size of the gap will not remain uniform along the length of the specimen. Geometrical arguments, and the light of experience, may be adduced to support the view that corrections based upon *average* specimen and airline diameters do give the best correction factor. But an allowance must always be made in the uncertainty budget for departure from the strictly circularly-symmetrical geometry assumed for calculating the standard capacitative correction [83]. A frequently encountered and much more serious problem introduced by the lack of symmetry of the mounting of the specimen in the air-line, however, is *the launching and resonance of higher order modes*.

The transmission mode employed in this coaxial method is the TEM mode, but unwanted higher-order modes also have to be taken into account because any departure from circular symmetry in the mounting of the specimen in the line will launch these modes. They can then resonate in the specimen and cause large errors. Figure 40 relates the frequency of the lowest-frequency higher-order mode to the specimen length and permittivity for 7-mm coaxial line. If the specimen length and its permittivity are known approximately, a maximum measurement frequency to avoid these effects can be estimated from the graph. Measurements near-to and above these frequencies cannot be guaranteed to be correct. For this reason, the shortest possible specimen, consistent with obtaining the desired measurement resolution ought to be used. Measurement of low loss may require longer specimens, but such measurements will be achieved at the expense of lowering the upper frequency limit for accurate measurement. The paper by Vanzura *et al.* [164] illustrates just how dominant the resonances of higher-order modes can be at higher frequencies.

One might think that measurements can be performed at frequencies *above* the lowest resonance, provided that one avoids frequencies close to resonances, where obviously erroneous results will arise. But this is not necessarily so - any modes that are launched are *still present* and are propagating in the system even at frequencies where they are not resonant. They typically affect the measurement of, say, ε' by making it appear to *rise* with frequency (see e.g. [164]). This is called the *ε'-Enhancement Effect.* As we know from Section 2.3, for a homogeneous non-composite material, ε' should always *fall* with frequency in the RF & MW range – the apparent rise in ε' is therefore is a *measurement artefact* of the method. If one is aware of the effect one can estimate its magnitude, perhaps correct for it, and thereby extend the upper frequency range beyond one or two resonances, but measurement uncertainties must also be increased to take account of the effect.

With *high-loss specimens*, such resonances cannot be seen and the ε'-enhancement effect is usually reduced because of the rapid absorption of the higher-order modes in the specimen. However, the effect *may* still be present though not detectable because the permittivity, ε', of high-loss materials falls relatively quickly with frequency, and so the effect may be masked. Errors and uncertainties caused by the enhancement effect are therefore very difficult to quantify. The best advice, as given above, is to use specimens that are as thin as possible, consistent with achieving one's achieving the desired measurement resolution. This will raise the resonance frequencies, hopefully above the band of interest. Another option is to use thicker specimens to improve resolution at low frequencies and thinner specimens for higher frequencies.

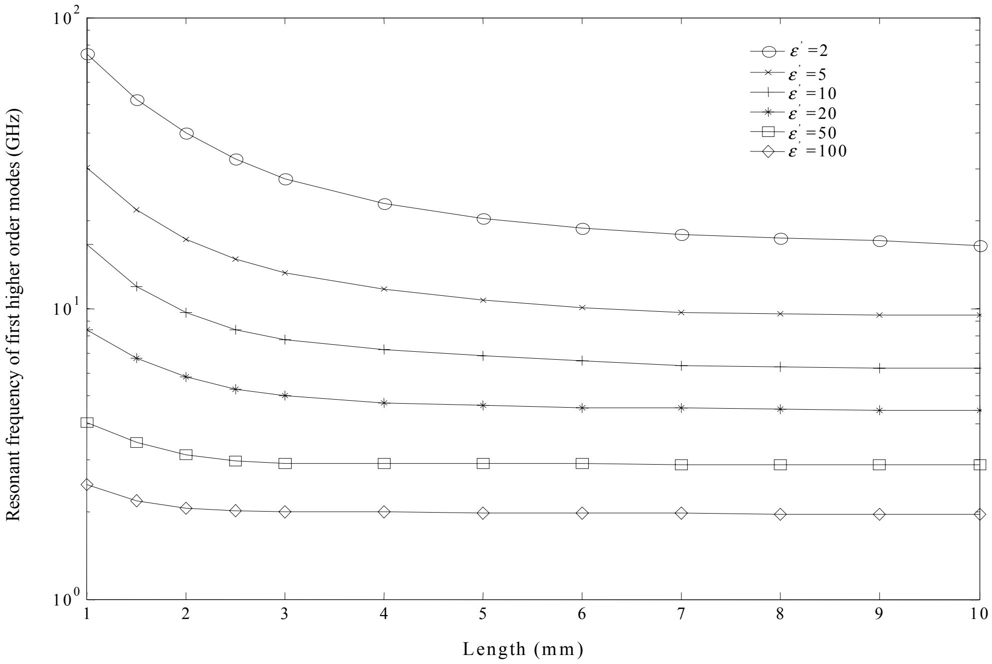


Figure 40. Onset of higher order modes in 7-mm coaxial transmission line measurements as a function of the specimen length.

<u>Liquid Measurements.</u> Clearly, to prevent evaporation and contamination, the liquid should be contained in a cell with a suitable window at both ends (but just the input end in a reflection measurement), see Figure 38 and [79]. Clearly, also, windows should be made from a material that will not absorb or dissolve in the liquids to be measured! If the range of permittivities to be measured is known, it is recommended that the cell be modelled to optimise (i) its length, (ii) the window thickness and (iii) the window permittivity, so as to minimise measurement uncertainties. *Thin* windows are not necessarily the best choice and they are more fragile and likely to leak. Thick and even lossy windows can readily be taken into account in the measurement equations, because the cell can readily be modelled as a series of cascaded two-ports. For all other considerations please refer to Section 5.3.3.

<u>Reducing Uncertainties by use of Time Domain Gating</u>. The ANA-based technique known as *Gating* [106] described in Section 7.1.19 can help one to reduce measurement uncertainties in transmission-line measurements as it enables one to distinguish signals from the specimen from those caused by mismatches elsewhere in the transmission-line system. It achieves this by *gating* or *windowing*, i.e. selecting out the wanted signals in the Time Domain and rejecting the rest. Most modern vector-ANA models allow for gating as a measurement option.

<u>Uncertainties: Transmission Line Measurements by Transmission vs. those by Reflection.</u> At microwave frequencies one often finds that measurements using only transmission coefficients are more accurate that those that can be obtained by measuring only reflection coefficients. This is borne out by uncertainty modelling of measurements in transmission lines. Given the wide range of S-parameters and specimen lengths measured by this technique, there may be some frequencies, however, where reflection measurements are more accurate than transmission measurements. This demonstrates how important it is to *<u>model</u> possible measurements* before performing them. One can thereby optimise one's transmission line measurements so as to minimise

uncertainties. As shown in the worked-examples in Section 9.7, uncertainties can readily be estimated in this way, provided one has some idea of what value ε^* is likely to have. By this means, one can optimise the size of the specimen to be used (if one has a choice in the matter) to minimise uncertainties and also decide whether to trust to transmission or reflection measurements (or both). Bear in mind that at low frequencies specimens some centimetres in length may be used, but only if (i) they can be machined accurately enough and (ii) they can be readily inserted and removed from the air-line without inflicting damage. As mentioned above this can more readily be done by stacking shorter specimens in series if they have flat, parallel faces.

A problem that reflection measurements in a matched-line system suffer from, but which transmission measurements do not, is the uncertainty of the position of the specimen in the line. As shown in the worked example in Section 9.7, this causes phases changes in S_{11} and S_{22}, which can give rise to measurement errors. However, if both S_{11} and S_{22} are measured and the average result taken, the errors should cancel out provided they are small.

Clearly the choice between transmission and reflection coefficients in measurements is only available for non-magnetic specimens. If the specimens are magnetic, both coefficients must be measured and one will have to put up with the uncertainties that result, though again, if one has some idea of what values ε^* and μ^* are likely to have, one can make use of pre-modelling to optimise the measurement.

At LF and at low RF (typically $f < 10$ MHz) most transmission-line methods cease to be effective for low- to medium-loss specimens because of the shortness of typical specimen lengths compared with the wavelength: Transmission measurements hardly ‘see’ the specimens because they are so comparatively thin, while reflection measurements do not ‘see’ them because reflections from both surfaces tend to interfere destructively, so making the specimen virtually ‘invisible’ for very long wavelengths. This is not true, though, for high-loss specimens where absorption may prevent a significant signal magnitude from reaching the back of the specimen, let alone from being reflected back to the front face again. For such specimens transmission measurements may deliver large errors if there is leakage through air-gaps around the specimen, while reflections from the mismatch of the *front surface only* may deliver accurate values for ε^*.

Reflection Measurements at the End of Transmission Lines There are a number of circumstances in which such reflection measurements are to be preferred. Here are a few examples:

(i) If no ANA is available, only a reflectometer, or a slotted line.

(ii) If measurements must be performed at an elevated temperature in a cell that demands such a geometry e.g. [169].

(iii) If measurements must similarly be performed at cryogenic temperatures in a Dewar.

(iv) If such measurements are cheaper or more cost-effective to perform and are quite accurate enough.

As mentioned above, the traditional transmission-line technique is the Roberts & Von Hippel method [162] in which the specimen is backed by a short-circuit, though this method has more often been applied in waveguide transmission-line (Section 7.1.11). Sources of uncertainty in this method include the effect of unwanted gaps between the specimen and short-circuit. In some cases it can prove to be difficult to realise an effective short-circuit (see [170] – but this is in a waveguide measurement). Even magnetic specimens may be measured in this way if they are placed in a number of positions, typically spaced $\lambda/4$ apart [1]. An alternative which should be considered is to back the specimen with an *open-circuit* instead of a short-circuit. This is done in the *discontinuous-inner* geometry for liquid measurements [79], discussed as an extension of the coaxial probe technique in Section 7.1.12.

Other Specimen Geometries in Coaxial Lines A number of other specimen geometries have been used for dielectric measurements using coaxial mounts or coaxial transmission lines. Some of these were reviewed in [3]. A number of these methods can be useful if only very small specimens are available. For example, a ‘puck’-shaped specimen with outer-diameter matched to that of the inner-conductor of the coaxial line can be used, either for a short distance *in place of* the inner-conductor in a transmission cell or else to terminate the inner-conductor against a short-circuit. One of the most important recent developments based on a coaxial-line cell is that of Pelster [171], who uses small capacitor electrodes in-line with the coaxial-line inner-conductor.

Other TEM Transmission Lines. Other TEM-mode transmission lines can be used for specific types of materials. Slab-line (parallel-plates) have been used for liquids [172], while large TEM transmission lines have been used for measuring RAM panels (Radiation Absorbing Material – see Section 5.1.12) [173, 174]. In one of these methods [173] a pyramidal reflectometric cell of square cross-section is used. The inner-conductor tapers out to the size of a single RAM block, while its outer-conductor tapers out to a linear dimension of three RAM blocks on a side. In this way near-normal incidence reflection measurement from eight RAM blocks surrounding the inner-conductor is possible. In another example of RAM measurement, [174] both transmission and reflection measurements were performed in a conventional TEM-cell with a flat-septum inner-conductor of the sort normally used for calibrating RF field-strength probes. In this case, the whole of the cross-section of the TEM line must be filled with the RAM. ANA calibration of the TEM-line itself can be achieved by using a metal sheet which has spring-finger contacts to both the inner-conductor and the septum as a sliding sort-circuit.

► *Summary: Coaxial Transmission Lines*

Advantages of the Coaxial Transmission Line Method. Relatively broad-band frequency coverage, often covering a range of 30:1 in frequency with a single specimen. Suitable for magnetic materials – one of the best techniques at microwave frequencies for high-loss and medium-loss specimens. Uncertainties for ε' better than ± 1% can be obtained for $\varepsilon' < 5$.

Disadvantages of the Coaxial Transmission Line Method. The cost of machining specimens accurately enough to produce accurate measurements. For solid specimens significant errors are caused by air-gaps: notably the dilution of the apparent permittivity and the launching of higher-order modes.

Coaxial Transmission Line Measurements: Typical Sources of Dominant Uncertainties:

With *solid* specimens:
- Air-gaps between the specimen and the *inner*-conductor.
- Air-gaps between the specimen and the *outer*-conductor.
- The launching of higher-order modes.
- With *magnetic* materials:
  - Uncertainty in the longitudinal position of the specimen in the cell.
  - Uncertainty in the thickness of the specimen.

With *liquid* specimens:
- Uncertainty in the length of the cell
- Uncertainty in the thickness of the windows that delineate the cell, and possibly uncertainty in the permittivity of the window material.
- The temperature of the liquid.

In *reflection* measurements - in addition to the above:
- Unwanted gaps between specimens and, e.g. short-circuits.
- Unwanted mismatches in the air-line between the measurement cell and measurement plane.
- Possible poor performance of short-circuits (especially if they are sliding-shorts).
- Uncertainty in the position of the specimen in the transmission line.

► *Do's and Don'ts for Transmission Line Measurements*

Do machine the inner and outer diameters of solid specimens as accurately as possible to fit into the air-line and onto the inner-conductor.

Do measure air-gaps (typically by using air-gauging), correct for them and also increase the estimated uncertainties to account for them.

Do, for broad-band measurements, use the thinnest specimens possible, consistent with obtaining the desired measurement resolution for permittivity and loss, so as to push higher-order mode resonances up as high as possible in frequency.

Do use specimens of more than one thickness to obtain best uncertainties over a broad band of frequencies.

Do consider whether it is most fruitful to perform transmission measurements, reflection measurements or both in your frequency range of interest. Model the measurement to see which will give the least uncertainty.

Do, if you have the option of choosing specimen thicknesses, model the measurement using the *measurement equations* so as to choose optimum specimen thickness to minimise uncertainties

Do use a known check-specimen or material to check your calibrations and the correct working of the technique.

Don't use the technique if you want ε' uncertainties to be significantly less than ± 1%.

Don't attempt to use the technique for measuring the loss of low-loss specimens (the permittivity, ε', of low-loss specimens may be measured however).

Don't underestimate the effect of air-gaps between specimens and inner- and outer-conductors.

Don't underestimate the magnitude of errors caused by the launching of higher-order modes.

7.1.11 Waveguide Transmission Line

Most of the considerations discussed for coaxial transmission lines in Section 7.1.10 apply to waveguide measurements, so Section 7.1.10 should be referred to for the basic principles, limitations and problems of the method. The main *advantage* of using a waveguide as opposed to a coaxial transmission line, for dielectric measurements is that one does not have to machine axial holes through the specimen to tight tolerances to allow it to be fitted onto a coaxial inner-conductor. The absence of the inner-conductor also makes waveguide cells more suitable for temperature control. The main *disadvantage* of waveguide transmission line measurement is that one is normally restricted in frequency coverage to a single waveguide band (less than an octave in frequency coverage). Furthermore, at frequencies up to 18 GHz one is required to use significantly larger specimens than those that are appropriate in a 7-mm coaxial transmission line. If this is not a problem, however, larger size may be seen as offering an advantage: the larger specimen and its convenient rectangular shape should allow more accurate measurements to be undertaken, perhaps down to uncertainties of ± 0.5% for ε' in optimum circumstances. A larger specimen size can also be used to *average* the properties of inhomogeneous materials that have a structure-length larger than the diameter of a typical coaxial line. For example, such a cell 72 mm × 34 mm in cross-section and 10.0-mm thick, has been used at 2.45 GHz at NPL in a variable-temperature system designed to measure processed foodstuffs including 'ready meals'.

As in the discussion of coaxial-line measurements in Section 7.1.10, the appropriate techniques and equations are included in the existing literature, which should be referred to for more details - notably the NIST Technical Note 1341 [83] and the ASTM Standard D 5568-01 [165]. Note however, in this case, that the correction for air-gaps around specimens *cannot* be derived from a simple capacitative model because the normal propagation mode in the waveguide is the TE_{10} mode, not a TEM mode. This is an important point and so is discussed in a separate section below.

As with coaxial transmission line, both transmission and reflection methods are possible and similar considerations apply here as those discussed in Section 7.1.10. Note that the Roberts & Von Hippel Technique was originally developed for waveguide measurements [162]. A reflection method may be particularly useful for high temperature measurements as there is less of a thermal shunt out of the measurement cell [169]. Again, care has to be taken when it is used with sliding short-circuits, especially when they are immersed in liquid specimens [170]. This is because short-circuit designs that are suitable for air dielectric may well fail to function well when immersed in liquid dielectrics in which the wavelengths are shorter.

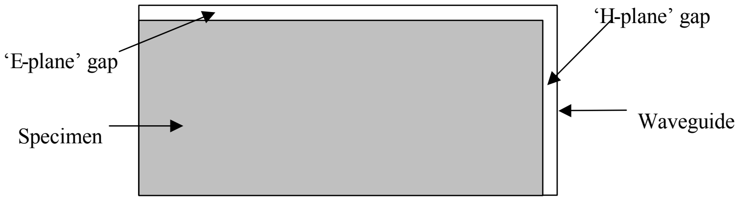


Figure 41. Air gaps around a specimen that is being measured in a waveguide cell. The Figure shows a cross-section of the waveguide with the specimen in it.

Corrections for Air-Gaps around Specimens in Waveguide Transmission Lines. Just as with coaxial line measurements, such corrections to the measurement equations are necessary if the air-gaps are not to dilute the apparent measured permittivity of the specimen. However, in this case, it is not possible to use a simple capacitative model to calculate the effect of the sort of air-gaps shown in Figure 41. A capacitative model is appropriate in the case of TEM propagation (as in coaxial lines) because the propagation parameters Z_0 and γ depend upon the capacitance per unit length in the line [21] and one can see directly how that parameter depends upon the permittivity of dielectric materials placed between the inner and outer conductors. The normal propagation mode in waveguide, however, is the TE_{10} mode, which has a more complex field geometry. Even if one wished to apply a correction model based upon capacitance, it is not clear which capacitance one should use. The situation is complicated by the fact that one can have both 'E-plane' gaps (parallel to the long face of the guide) and 'H-plane' gaps (parallel to the short face of the guide), as shown in Figure 41. When such air-gaps are present in a waveguide, one formally has a *heterogeneous* propagation medium. The propagation modes are formally *hybrid* modes and the modal analysis becomes somewhat complex. In fact, to the best of our knowledge the full modal analysis of the geometry shown in Figure 41 has never been completed for magnetic materials – though studies are in progress at the time of writing this guide. Suffice it to say that it has been empirically observed that, for 'small' gaps, i.e. less than 1% of the height of the guide, (i) H-plane gaps have virtually no effect on ε^* measurements because the E-field of the TE_{10} mode is very low in the gaps, (ii) the measured correction for E-plane gaps is smaller than that which is computed from a capacitative model based of the capacitance from top-to bottom of the waveguide. Generally speaking, if specimens are machined to give a good sliding fit into the waveguide (taking into account the rounding of the internal waveguide corners) the air-gap effects in waveguides up to 18 GHz will be smaller than those obtained in coaxial transmission line measurements over the same frequency range because the specimens will be larger in size and because the critical problem of the air-gap around the inner-conductor does not arise.

7.1.12 Coaxial Probes and Waveguide Dielectric Probes

Introduction. Coaxial probes, see Figure 42, are widely used for non-invasive dielectric measurements the world over because of their convenience. Their popularity has been ensured by the fact that they are available commercially with user-friendly software to relate the measured reflection coefficient to permittivity. NPL has published a number of papers and reports on such probes [175, 176, 177, 178, 179] and reference to them is recommended to understand the details of their metrology. These probes have been used widely elsewhere, see e.g. [180] and the references in [179]. For liquid measurements a comparison of their use with that of other types of cell is included in [79] and measurements on a range of reference liquids are described in [181].

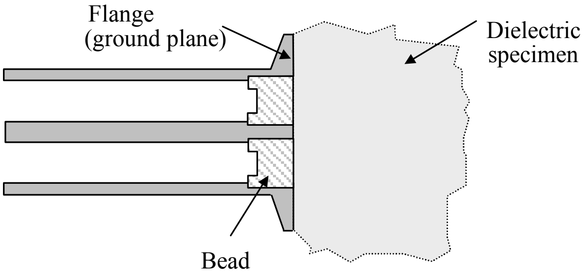


Figure 42. A coaxial probe fitted with a dielectric bead that is designed to match the probe to 50 Ω, see [175]. The probe is also fitted with a flange to make it calculable (see text).

The principle of operation of the conventional flat-faced probe is illustrated in Figure 43. A TEM travelling-wave propagates in the coaxial line up to its end where it launches fringing E.M.-fields from the open end of the probe into the dielectric specimen, which is placed up against its face. Their magnitude and geometry depends upon the complex permittivity, ε^*, of the dielectric and so the reflection coefficient of the TEM-wave from the end of the probe will depend upon the value of ε^*. One can relate the measured reflection coefficient, Γ, to ε^* by using (i) a modal analysis of the fields in the coaxial line and (ii) an analysis of the fields in the dielectric under test (d.u.t.) that treats the probe as an antenna. This relationship can be graphically illustrated by means of a *mapping* as in Figure 44. This approach is described in the above references. Simpler analyses have been used, especially those that use a simple capacitative model for the fringing-fields, but they all have their limitations [175] and at sufficiently high frequency it is necessary to treat the probe as an antenna because it actually *radiates* into the d.u.t. so that $|\Gamma| < 1.0$ even if the dielectric is lossless [79], see Figure 44. The power is radiated out through the specimen to 'infinity' - this is a feature that a simple capacitative model cannot reproduce.

Flat-faced coaxial probes, such as the one shown in Figures 42 and 43, represent just one member of a whole family of reflectometric and non-invasive probe designs that can be used for dielectric measurements. One other flat-faced probe option is the *open-ended waveguide probe* [182, 183, 184]. This type of probe has the capacity to measure anisotropic materials [185] - detail on this is given below. Coaxial probes are widely used for characterising lossy solids like biological tissues because of their ability to perform measurements by contacting just one face of the specimen, rather than having to machine the specimen to fit into a measurement cell. This makes them very convenient to use. They are also ideal for measuring lossy liquids and are widely used for SAR liquid characterisation [51, 52]. But with liquids one does not need to use a flat-faced probe and there are often advantages to be gained by using other geometries - see the '*Liquid Cells*' section below.

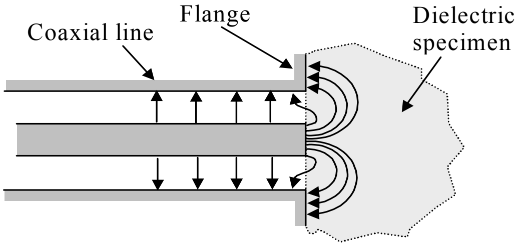


Figure 43. A coaxial probe, showing the fringing fields that emerge from its ends.

As with all measurement techniques, however, the coaxial probe method has its limitations. There are many types of measurement for which it is tempting to use a probe but for which it would be the wrong metrological choice. We will discuss these below as we look at what the scope of the method actually is.

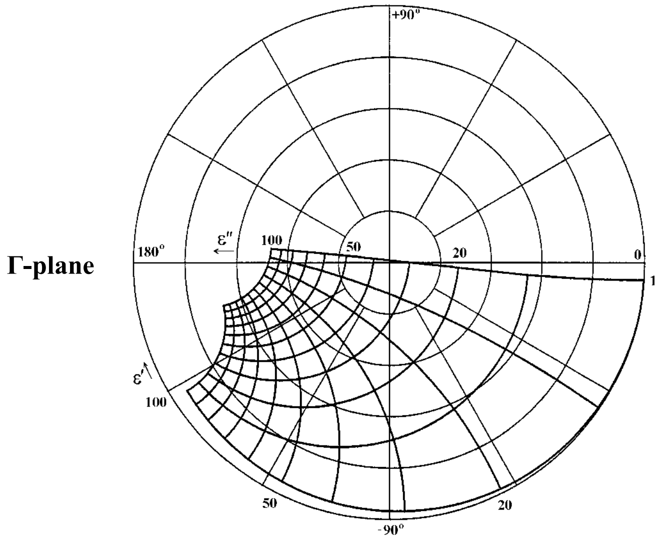


Figure 44. A mapping of ε^* onto the complex impedance plane, Γ, for an open-ended 50-Ω coaxial line with an outer conductor diameter of approx. 15 mm. The mapping is for 1 GHz. Details are given in [79]. The outer contour on the mapping is for $\varepsilon'' = 0$. Note that $|\Gamma| < 1.0$ even for this contour, which is for a *lossless* dielectric. This is because the probe acts as an antenna and radiates power away from itself, giving rise to radiation losses.

Scope of the Method. A single flat-faced probe can typically operate effectively over a frequency range of about 30:1 (e.g. 100 MHz - 3 GHz for a 15-mm probe) whilst retaining a reasonable uncertainty performance of the order of ± 3 % for ε' for suitable materials. The actual frequency range depends upon the diameter of the coaxial aperture of the probe and the permittivity of the d.u.t, because, in a very crude capacitative model, the fringing capacitance is roughly proportional to the probe outer-conductor diameter and to the permittivity of the d.u.t. The mapping in Figure 44 shows the relationship between ε^* and Γ at one frequency only, 1 GHz. For the probe in question in which the outer-conductor has a diameter of 15 mm, 1 GHz is, in fact, close to the frequency of optimum sensitivity for ε^*. At very low frequencies the sensitivity falls off because the ε^*-mapping bunches close to the 'open-circuit' point at the right hand side of the chart, while it bunches close to the 'short-circuit' point at the left hand side of the chart at very high frequencies. The resolution for ε^* becomes very small in both cases. Best sensitivity is obtained when the mapping fills a significant area in the lower (capacitative) half of the chart, as shown in Figure 44. For these reasons one requires a larger capacitance - and so a larger probe - for lower frequencies and a smaller probe for higher frequencies. For the same reason, a given probe can be used up to higher frequencies when measuring lower permittivities. Typically for ε′ in the range 10 - 80, 15-mm diameter probes can be used up to about 3 GHz (but may have to be limited to lower frequencies by resonances caused by finite flange size), 7-mm probes up to 6 GHz and correspondingly smaller probes up to 18 GHz.

In air dielectric an abruptly open-circuited coaxial line presents a significant mismatch to the travelling TEM waves in the line so at low frequencies |Γ| into air is about 1.0. As Figure 44 shows, however, the coaxial probe is better matched when measuring lossy dielectrics that have high real permittivity. This has the consequence that coaxial probes are usually *best used* for measuring higher-permittivity lossy materials (e.g. ε' & ε'' above 10). Rectangular waveguide probes, on the other hand, are naturally better matched into free-space and so are better optimised for lower permittivities [185], see the discussion on such probes below. With either type of probe, coaxial or waveguide, the sensitivity for ε'' is similar to that for ε' if $\varepsilon' \approx \varepsilon''$, as can be seen from the mapping. Probe measurement does not (normally) use resonance detection (but see [186]) and so the method is best suited for medium to high-loss materials. One can measure ε' for low-loss materials, of course, but one will not have the resolution to measure ε'' for such materials.

Limitations of the Technique. As stated above coaxial probe uncertainties of the order of ± 3 % or less for ε' and ε'' can be obtained for suitable materials and specimens. However, that is only in the *best* circumstances and with *large specimens*. In many cases specimens are small, and then problems can arise. The assumption is made, in the basic theory that is commonly used to relate ε^* to Γ, that there are no reflections of waves from the extremities of the specimen nor from permittivity steps or gradients within inhomogeneous specimens. In medium to low-loss specimens this is often *not* the case. For example, if we measure de-ionised water in a glass beaker at 1 GHz, we have a material with $\varepsilon' \approx 79$ and $\tan\delta \approx 0.1$ and so the whole body of water can act as a dielectric resonator, which can resonate with a Q-factor of ~ $1/\tan\delta$, i.e. ~ 10. Furthermore resonances can be caused by finite flange size [178]. Such resonances are easy to detect with coaxial probes and in a well-defined coaxial geometry they can be modelled very accurately, see [179]. The metrological problem arises in practice because most probes are used most of the time with mathematical analyses that assume that these resonances and reflections are *not* occurring and so if they *are* occurring, one will derive the wrong value of ε^* from the measured value of Γ. The errors can be very large, even over 100%.

The best advice is that - unless one is using the probes in a fully calculable geometry with an adequate mathematical model (i.e. unless one is explicitly taking into account such reflections and resonances in the liquid or resonances caused by flanges, as in [176], [178] or [179]), one should restrict the use of the probe method to lossy materials in which all of the power is absorbed before it is reflected from the extremities of the specimen back to the probe face. This explains the popularity of the probe method for measuring biological tissues and tissue equivalent materials (t.e.m.s), because they are all lossy. But even here there can be problems because reflections also occur at interfaces between materials of different permittivity in inhomogeneous (e.g. layered) specimens. This can occur, for example, in tissues like skin [179].

For similar reasons, coaxial probes are better suited to measuring *malleable* materials that accommodate themselves to the shape of the probe, but they are not ideally suited to measuring *hard* dielectric specimens because they invariably leave uneven air-gaps between the specimen face and the probe face. The conventional theory assumes that there are no such air-gaps, and as the probe is particularly sensitive to the permittivity of the material closest to its face, even a small gap can give rise to a large error of measurement [187]. Such gaps can be modelled [178, 179], however (see below), and the utility of the probe extended thereby. But if the conventional ‘large specimen’ theory is being used, the advice must be to measure only malleable lossy solids and lossy liquids if large measurement errors are to be avoided.

Fully-Calculable, Partially-Calculable, and Non-Calculable Probes. As we have seen, if one is working with a calculable probe geometry one can relate ε^* to the measured value of Γ by using, say, a modal-analysis or an antenna analysis of the E.M.-fields associated with the probe [177]. In practice one has to reproduce the modelled geometry used in one’s actual measurements and it is the failure to do so that leads to the errors mentioned above. The need for such calculability leads, for example, to the fitting of flanges to probe faces because the theory actually assumes that the coaxial open-circuit lies in an infinite conducting plane [175]. In these circumstances the use of probe flanges that are too small can give rise to significant measurement errors [178]. If *appropriate* modelling is carried out, however, we can say that we have a *fully-calculable* probe.

An alternative approach that greatly widens the range of probe designs and their range of application is to reduce one’s dependence on *full* calculability and to rely more upon *probe calibration by use of known reference materials* to supply the accuracy that the theory lacks. Thus, one may prefer to use a probe without a flange (see e.g., [180] - it is smaller and more convenient than a probe with a (supposedly infinite) flange. One can use the ‘with-flange’ theory with the flangeless probe, then measure a range of known reference liquids with it using the data thus obtained to make corrections in all subsequent measurements, see Section 7.1.13. This will automatically take account of the absence of the flange. Typically, the fringing capacitance will be 2 - 10 % lower than that with the flange present, so the correction effectively applied by the calibration is not large. This approach is widely used with some success. One may call it a *partially-calculable* approach. To some extent one can actually avoid making such corrections *explicitly*. ANAs are specifically designed to make small error corrections, so as long as one uses a reference liquid standard which has ε^* close to that of the d.u.t. one can effectively use the ANA's own internal calibration software to correct for the imperfections of the theory used.

But one may go further - one can regard the probe as a 'black box', but one that has the property of being reasonably stable and of presenting one with a *one-to-one* and *smoothly varying* relationship between Γ and ε^*. By measuring a suitably large number of reference liquids having known ε^* values, one can interpolate measured values of Γ to calculate ε^* for d.u.t.s with reasonable accuracy. Probes that are used in this way may be called *non-calculable probes* and they are discussed further in Section 7.1.13.

It will be noted that *partially-calculable* and *non-calculable* probes are highly dependent upon the use of reference liquids for their calibration and even a fully calculable probe normally requires at least one reference liquid for its calibration. This makes it important to know how to use such liquids properly, see Section 4.4.2.

A Note on the Design of Coaxial Probes This is a less onerous task for partially-calculable and non-calculable probes than for fully-calculable probes. However, all of them need to be leak-proof and the dielectric bead material must be chosen so that it does not react with the materials, particularly liquids, to be measured. Fully calculable probes may be designed to be matched throughout [175], as shown in Figure 42, but this is not absolutely necessary because the ANA corrects for any mismatches in the probe itself, though their presence does contribute to residual errors and reduces measurement sensitivity. For the same reason it is probably best to use bead materials with a relatively low permittivity ($\varepsilon' \sim 2$ - 4) because they present less of a mismatch. The fact that perfect matching is not *needed*, however, makes it possible to design large-diameter probes for low frequencies by simple taper or step arrangements within the probe to bring the coaxial diameter down to fit one of the standard connector sizes. Fully-calculable probes may need flanges as in Figures 42 and 43 (depending upon the theory used), and one also needs to know the permittivity of the dielectric bead in such probes.

Probe Calibration. Coaxial probes are normally connected to an ANA by means of a good quality flexible coaxial cable (see Section 6.1.7) for determination of Γ reflectometrically. Such measurements normally require that the ANA be calibrated with at least three different calibration standards. These calibrations are essential for the proper use of the probe and so must be performed correctly and the calibration should be checked after it is performed. Any error in a calibration measurement may produce an error of up to 4 times the magnitude in subsequent measurements! This is unlikely, however, if the reference liquid has similar dielectric properties to that of the d.u.t, in which case a calibration error will produce errors of *similar* magnitude in subsequent measurements to its own. With coaxial probes the calibration standards one normally uses are (1) an open-circuit into air, (2) a short-circuit and (3) a measurement of a known reference liquid. (Other schemes are possible, including the use of artefact standards [175], but they are often less convenient). Such calibrations are critical to successful measurements and, as experience unfortunately shows, they are often prone to error. Some guidance is therefore given here:

(1) The *open-circuit* measurement into air (i.e. a measurement upon an air-dielectric 'reference fluid') is not usually problematic as long as the end of the probe is clean!

(2) Experience shows that the *short-circuit calibration* is often the most difficult to achieve properly as it requires good electrical contact of the short-circuit onto both the inner and outer-conductors. Many schemes have been devised to achieve this. The one which has been found to be most perennially effective at NPL is to use a piece of clean, unwrinkled aluminium foil, pressed into place by hard pressure from the thumb or via a rubber backing of similar consistency. If the inner-conductor is recessed behind the face of the dielectric bead by even a fraction of 1 μm, experience shows that adequate contact is often very difficult to achieve. It is preferable to have the inner-conductor *very slightly proud* of the bead (ideally by less than 5 μm) as better contacts are thereby achieved. This should not cause a problem if one is measuring malleable materials and liquids. It is the contact to the inner-conductor that is crucial for a good calibration - the outer-conductor has a much larger surface area than the inner and so is less problematic. Statistical analysis at NPL (see below) shows that the smaller the probe the more difficult it is to make a good short-circuit contact, and so a 7-mm probe is often preferred, even where, say, a 3.5-mm probe would have given adequate resolution.

(3) The third calibration standard - the <u>*reference liquid*</u> should be handled carefully, following the guidance of Sections 4.4.2 and 5.3.3. Avoid contamination of the liquid, which will change its permittivity, and *measure its temperature to ± 0.2 °C* so that the correct value of ε^* can be used in the calibration. *De-ionised water* is often used as the calibration liquid, but, as pointed out above, it is not actually a very lossy liquid in at frequencies below 2 GHz), one should take into account the fact that calibration measurements with water may actually be 'seeing' unwanted reflections from the extremities of the vessel [178] in which it is held, thereby giving rise to calibration errors. It is probably better at these frequencies to use a more lossy calibration liquid. The usual practice at NPL is to use *ethanol*, though this is more expensive.

Having performed the calibration one should first *check it* by measuring *another* reference liquid, i.e. *not* the liquid used for calibration. To re-measure the same liquid will not tell us very much apart from whether or not we can perform repeatable measurements! If the reference liquid were contaminated, for example, one would again measure the supposedly 'correct' permittivity for it - but this would actually be the wrong permittivity and the whole calibration would be faulty! Only by measuring a second *independent* reference liquid can one get confidence that the calibration has been performed correctly. It is generally good practice, where possible, to perform the check with a reference liquid that has properties close to that of the d.u.t. - this can help one to estimate uncertainties in the measurements. However, it <u>*is*</u> good practice to re-measure the *short-circuit* standard because their major problem is often their gross lack of repeatability – i.e. one can all-too-easily fail to achieve a 'good' short-circuit. If the re-check of the short-circuit measures $\Gamma = -1.0 + 0.0j$ (± acceptable discrepancies) then the short-circuit calibration was most probably valid. Otherwise one may have to repeat the calibration.

In fact, if the post-calibration checks reveal a poor calibration, one has little choice if one is using an ordinary ANA calibration procedure, other than to run the whole calibration procedure all over again. This can be very time consuming, not to say tedious. It has been found at NPL that an alternative calibration scheme, namely *Least-Squares Calibration*, can overcome this problem. It has many other advantages as well. Normal ANA calibration is mathematically a process that delivers the value of three complex 'error-parameters' associated with the reflectometer in the ANA (see [10, 12, 105]) - these values are needed in order to perform the corrections necessary for accurate measurements. As there are three 'error parameters', calibration with just three standards should give one just sufficient information to calculate their value. This is what is done in a conventional ANA calibration. However, one can calibrate with *more* than three standards (e.g. 4, 5, 6 etc.) – i.e. with an *over-determined* set of standards – one can then infer the best values for the 'error parameters' on a *statistical* basis. Each of the standards in the calibration can be given a different weighting in the statistical analysis and this can prove useful (see (iii) below). For coaxial probe measurements there are at least three benefits of this calibration scheme:

(i) Clues to uncertainties in ε^* arising from errors in the calibration can be derived as a natural extension of the method.

(ii) More than one reference liquid can be used, e.g. one's normal reference liquid (say ethanol) and another one which has properties closer to that of the d.u.t.

(iii) If there are problems with any one of the calibration standards they will be shown up clearly by comparison with the other standards.

It is this last feature that allows one to save time in calibrations, whilst taking account of repeatability and contact problems with the short-circuit standard. One can measure the short-circuit, say, three times, one after the other and treat each measurement as a measurement on a separate standard. When the full calibration is complete one can compare the three short-circuit measurements. If one of them is in error it will show up against the other two. The least-squares algorithm is then run again with zero weighting allocated to the poor short-circuit measurement. Considerable time is saved because only the computer program is run again - not the full calibration.

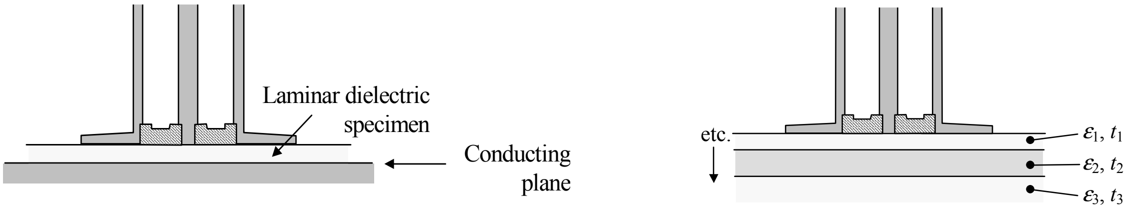
<u>*Coaxial Probe Measurements.*</u> Having achieved a good quality calibration, performing the measurements on the d.u.t. is usually a relatively simple process by comparison. Provided 'good practices' are followed, all should be well, but three points may be made:

(i) For the reasons given above, don't expect to get good measurements upon small or hard specimens, especially if they have low loss.

(ii) Make sure that you make a good full-face contact onto solid specimens and avoid air-gaps between the probe and d.u.t.

(iii) Remember that the coaxial cable that connects the probe to the ANA can produce spurious phase changes (and perhaps amplitude changes) when it is flexed. These will give rise to measurement errors. You can measure these changes in a separate experiment on the ANA and estimate the magnitude of error that they will introduce. Old cables can degrade very rapidly, so check them from time to time (see Section 6.1.7). High quality cables that have been designed specifically to reduce these effects are commercially available and it pays off metrologically to use them, though they can be quite expensive.

Measurements on Soft Specimens. Coaxial probes are well suited to measurements on malleable materials as the material can conform to the face of the probe by applying a little pressure. Measurements on *very soft* specimens (e.g. rubber or polymer foams) may cause a problem, though, because the probe will compress the material, and this will increase its permittivity during the measurement. One way to correct for this is to place the d.u.t. on a *scale-pan* and bring the probe down to bear vertically on the d.u.t.'s top surface, measuring the permittivity as a function of force applied. The permittivity obtained can then be extrapolated back to zero force (i.e. that of an uncompressed specimen).



(a) (b)

Figure 45. Use of a coaxial probe **(a)** for measuring a dielectric lamina backed by a metal conducting plane, **(b)** for measuring a multi-layered specimen. Each layer, *i*, has thickness t_i and permittivity ε_i

Other Calculable Coaxial Geometries. Most of the discussion above centred on the conventional use of coaxial probes with software that assumes *large* (formally infinite or semi-infinite) specimen sizes. A number of other coaxial geometries are calculable by means of modal or similar analyses and these can allow probes to be used accurately with smaller specimens, as shown in Figure 45:

(i) Laminar d.u.t.s can be measured with a conducting plate behind them [176].

(ii) Multilayer geometries are calculable and allow the effect of air-gaps to be estimated. More usefully they can be used to measure one dielectric *through* another, assuming the layer closest to the probe is thin [177, 178]. One example of this is measurement of the curing of an adhesive through a thin layer of PTFE film. The film is necessary to prevent the adhesive from adhering to the probe! It has also been pointed out also [188] that better accuracy can sometimes be obtained in probe measurements, especially if the specimen has a rough surface, by using '*Lift-Off*', i.e. a deliberate but small air-gap of known average thickness is left between the specimen and the probe, see also [179]

(iii) Finite-diameter specimens can be measured in cylindrical metal cells [178, 179], see Figure 46. They may be laminar, multilayered or liquids. Such cells can unintentionally act as resonators, however, so in general, smaller diameters are to be preferred because they resonate at higher frequencies: resonances in supposedly non-resonant measurements are normally to be avoided. There is no reason why the cell diameter should not be the same as (or even slightly smaller than) that of the outer-conductor of the probe (see Figure 46 (a) and the next sub-Section). One *good* reason for making a larger-diameter cell, is that a solid specimen which has been designed for measurement by another technique (one which requires a large-diameter specimens) can also be measured by a coaxial probe without cutting the specimen down in size – there are always metrological advantages in measuring by more than one technique, as explained in Section 4.4.3

All of these calculable geometries retain the flat probe face. With liquids this is unnecessary, as discussed next.

Calculable Coaxial Cells for Liquids By extending the outer-conductor of a coaxial probe and by orientating it to point upwards as in Figure 46 (b) one can make a very simple and effective, easily sealed, cell for measuring the dielectric properties of liquids [79]. The liquid can be poured in until its meniscus is sufficiently far away from the end of the inner-conductor that no change in reflection coefficient Γ is seen if more liquid is poured in. Mathematically, the modal analysis of such a cell is actually easier than that of the probe radiating into free space. If coaxial symmetry is maintained, the relation between ε^* and Γ is normally obtained from an analysis that includes only TEM and TM_{0n}-modes only. The viable frequency range of such a cell will depend upon the capacitance between the inner and outer-conductors through the liquid. If the end of the inner-conductor is flush with the dielectric bead, as in Figure 46 (a), the capacitance is quite small (of the order 0.08 pF in 7-mm line in air dielectric). In order to obtain more sensitivity at lower frequencies one can extend the inner-conductor into the liquid, as in Figure 46 (b) giving a much higher capacitance, typically ~ 1 pF cm^{-1} in air. These cells can give very accurate measurements of dielectric properties and they have been used in programmes of reference liquid characterisation at NPL [79, 37].

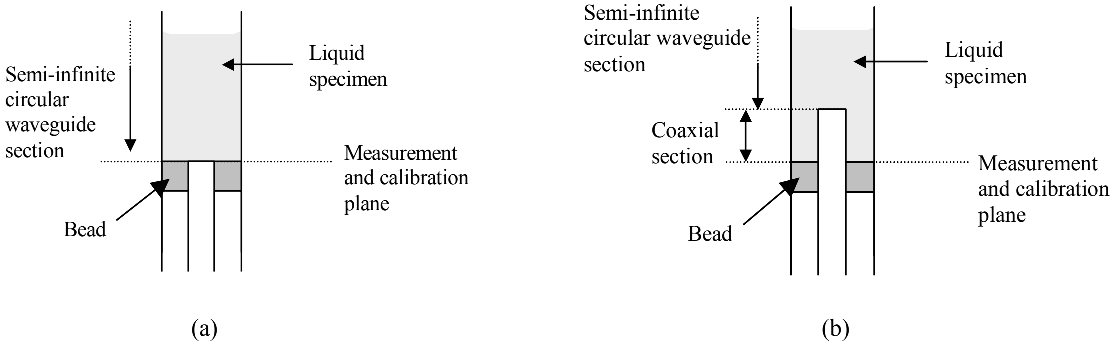


Figure 46 Liquid Cells. **(a)** A modification of the coaxial probe shown in Figure 43 to allow easy measurement of liquids. **(b)** *The Discontinuous Inner-Conductor Cell:* a further modification that can be used to increase the measured capacitance of the dielectric liquid. For a given liquid cell-(b) will be more sensitive at lower frequencies than cell-(a). The inner-conductor extension is of any appropriate length. Both cells are *calculable*

Resonant Coaxial Probes. One disadvantage of the conventional coaxial probe is that it normally has to be used in conjunction with an expensive reflectometer, such as an ANA. In a production or quality-control working environment or in reproducibility testing, a much cheaper option might be preferred. If the measurements can usefully be performed at one frequency only, one option is to turn the coaxial probe into a resonator: details are given in [186] and Ch. 11 in [22] (R J King *et al*). As in many resonant techniques, changes in the resonant frequency, Δf_r, and Q-factor, ΔQ, on contacting the d.u.t., can be used to calculate ε^*. The technique can be used with calculable coaxial probes and non-calculable probes.

Waveguide Probe Measurements. Open-ended waveguide probes [182, 183, 184] are used less often than coaxial probes, partly because, like all waveguide-based systems they are limited in frequency range and they may be physically quite large. However, the required probe size for a given frequency range can be reduced if the probe waveguide is itself filled with a 'loading' dielectric material. Reference [185] describes one such probe, based on a WG16 (normally 8.2 – 12.4 GHz) waveguide adaptor, which was loaded with glass dielectric ($\varepsilon' \approx 6$) to allow it to operate in the range 3.5 – 5 GHz. Note that *all* of the waveguide must be filled with the loading dielectric otherwise part of the guide will be below cut-off. This requires special treatment in the vicinity of the coaxial interface into the waveguide.

Waveguide probes offer two potential advantages [185] over coaxial probes for specific applications. One, as explained in the general introduction to open-ended probes above, is the fact that such probes are better matched for measuring lower permittivity than coaxial probes of similar size and at a similar frequency. The other, perhaps more important, advantage is that waveguide probes, being linearly polarised, can measure *anisotropy* in dielectrics (see Section 5.3.11). Anisotropy is measured by carrying out two measurements on the flat face of the specimen, with the specimen orientated in two orthogonal directions. The measurement equations used for this technique will almost certainly assume that the two orthogonal orientations correspond to the optic-axis in the specimen being *parallel to* and *orthogonal to* the E-field in the waveguide, respectively. A little experimentation may be required to identify the direction of the optic-axis of the specimen if it is not previously known. In studies on such a probe at NPL [185], a modal analysis was used to perform calculable measurements on *square* laminar anisotropic specimens that were placed in a square cell at the end of the probe in both of the required orthogonal orientations with respect to its face. The study showed that the numerical analysis must take full account of specimen anisotropy if the degree of anisotropy is to be computed correctly. One cannot just use the isotropic theory to interpret the measurements upon the anisotropic materials, measured parallel and perpendicular to the optic axis and expect to get the correct values for $\varepsilon'_{\parallel}$ and $\varepsilon'_{\perp}$

In all other respects waveguide probes are employed just as coaxial probes are and the same warnings apply: e.g. take care to avoid air-gaps between probe and specimen and to avoid errors caused by the departure of the measurement geometry from that assumed in the numerical models. Uncertainties available from waveguide probes are similar to those of coaxial probes – i.e. they amount to a few percent or possibly ± 1% *at best*, so there are other more accurate techniques available. Measurements of the *degree of anisotropy* can be performed much more accurately however, perhaps better than ± 0.1%. Given this fact, and the limited frequency range and the air-gap problem it is best to regard waveguide probes as a specialised tool either for measuring the anisotropy of suitable specimens or for measuring dielectric liquids with lower permittivities (e.g. $\varepsilon' < \sim 20$).

► *Summary: Coaxial and Waveguide Probes*

Advantages of Coaxial and Waveguide Probe Techniques. Quick and easy to use (though not necessarily to calibrate). Potentially portable: so there is a possibility of use in the field. A single probe can potentially be used over a frequency range of 30:1 with suitable specimens. Specimens need not be contained in cells - the method is therefore well-suited to non-destructive testing. Relatively cheap to use compared with many other methods.

Disadvantages of Coaxial and Waveguide Probe Techniques. Air-gaps between specimens and probes are difficult to avoid with hard specimens, giving rise to large errors of measurement. Calibration is prone to error and is sometimes difficult and time-consuming to get right. One must be aware of the limitations of the method regarding specimen size and homogeneity. With typical uncertainties of ± 3% for ε' in the best circumstances, the method is generally less accurate than a number of others described in this Guide.

Coaxial and Waveguide Probe Measurements: Typical Sources of Dominant Uncertainties:

With *solid* specimens:
  Air-gaps between the specimen and the end of the probe – especially with hard specimens.
  Departure of the measurement geometry from the geometry assumed in the numerical analysis:
    - errors caused by reflections from the extremities of small specimens.
    - resonances within the body of the dielectric specimen.
  Specimen inhomogeneity, if the numerical analysis employed does not take it into account.
  Poor calibration measurements – especially of the short-circuit standard.
  Reference calibration liquids failing to have the assumed complex permittivity - e.g. because they are at the wrong temperature or because they are contaminated.
  Phase changes arising from the flexing of the cable that connects the probe to the ANA.

With *liquid* specimens:
  As with solid specimens – apart from air-gaps, though watch out for errors caused by bubbles, especially bubbles adhering to the face of the probe.
  Open liquid surfaces are prone to contamination and evaporation, thereby cooling the liquid (see 5.3.3).

► *Do's and Don'ts for Coaxial and Waveguide Probe Measurements*

Do check that the specimen is suitable for measurement by the probe – it may not be suitable if it is *small*, *low-loss*, made of a *hard material* or if it is *inhomogeneous*.

Do, if small, hard, low-loss or inhomogeneous specimens *are* to be measured, carry out an uncertainty analysis to establish whether the probe technique can achieve the required uncertainty.

Do remember that good calibrations are important: check that calibration liquids are at the temperature assumed when calculating their permittivity and that they are pure. Check that adequate contacts are made to the probe in short-circuit calibration measurements.

Do measure a reference liquid as a check *after* each calibration to ensure that the correct complex permittivity is obtained - if not, the calibration may have to be repeated. The liquid should *not* be the one employed for the calibration.

Don't use the technique if you want ε' uncertainties to be less than a few percent.

Don't attempt to use the technique for measuring the loss of low-loss specimens (ε' of low-loss specimens may be measured however).

Don't underestimate the effect of errors introduced by inhomogeneities in the specimens, by reflections from the extremities of specimens and by resonances within specimens.

7.1.13 ‘Non-Calculable’ Dielectric Probes.

A whole range of reflectometric probe designs is possible, of which coaxial and waveguide probes are just two examples. For a given probe diameter a higher capacitance can be obtained, for instance, by using a capacitor made up of multiple coaxial rings on the probe face [189] or by interdigitating the ‘outer’ and ‘inner’ conductor electrodes on the probe face. As with the liquid cell shown in Figure 46 (b), the advantage of the higher capacitance is better sensitivity at lower frequencies or for lower values of ε' . In this case however this is achieved at the cost of the loss of calculability by conventional numerical analysis techniques. ‘Calculability’ here refers to one’s ability to relate the measured reflection coefficient, Γ, to the complex permittivity, ε^*, of the d.u.t. by numerical analysis (see the Glossary). This usually requires a high level of symmetry in the measurement geometry for analysis to be effective. Removal of such symmetry or, in fact, adding any degree of complexity to the geometry (as exhibited by interdigitated fingers) renders conventional numerical analysis too intractable to be useful – but see point (b) below.

Calibration by Use of Many Reference Liquids (MRL). As stated in Section 7.1.12, however, lack of calculability does not mean that such probes are unusable. As long as there is a *one-to-one* and *smoothly varying* relationship between Γ and ε^* one can calibrate the probe by measuring a suitable range of reference liquids that have known ε^* values (but see point (a) below). Mappings such as that shown in Figure 44 for a coaxial probe can then be set up. One can then interpolate measured values of Γ to calculate ε^* for the d.u.t.s. Probes that are used in this way are called *non-calculable probes* in this Guide.

Advantages of Non-Calculable Probes. Here are a few reasons why these probes may prove to be useful and why they should always be considered as possible alternatives to conventional coaxial or waveguide probes.

(i) Higher sensitivities may be obtained from higher capacitances as exemplified by the discontinuous inner-conductor cells for liquids, see Figure 46 (b) [79].

(ii) Better penetration into a material may be obtained by using *inductive*, rather than purely capacitative coupling, i.e. by coupling into the dielectric properties of the d.u.t. via the *eddy-currents* induced by magnetic field set up by a magnetic coil probe. Preece *et al.* [190, 191] achieved this by inverting the normal operation of an inductive hyperthermia applicator, i.e. they used it as a reflectometer.

(iii) In order to remove errors caused by air-gaps, *conformal probes*, i.e. probes with *curved faces* can be used. Preece *et al.* have also studied a flexible probe for the same reason.

Two points may be made that may help to improve the utility of such probes.

(a) Contrary to what was said above, the relationship between ε^* and Γ does not strictly have to be *one-to-one*, though it should be *few-to-one,* i.e. only a few values of ε' should be possible for a given measured value of Γ. There are a number of conventional dielectric measurement techniques in which there is ambiguity in ε' if one does not know how many wavelengths of E.M.-wave lie in the specimen (e.g. transmission line methods, Section 7.1.10 and, many resonance methods, e.g. those in Section 7.1.7., 7.1.17). This may not be a problem in such methods as long as one has a reasonable idea of the permittivity to be expected. Thus, if we know the range in which ε' lies, the ambiguity can generally be resolved. In most of the other methods in which such ambiguities occur, however, they can be *definitively* resolved by measuring specimens of the same material with different thickness. This may not be possible with probes.

(b) Since the advent of discretised E.M.-field computer-modelling, e.g. by FE, FI, FD and FDTD techniques, (see Section 6.9) *non-calculability* does not necessarily mean non-computability. By using such techniques, the relationship between ε^* and Γ may well be computed even for quite complex probe geometries. If such a facility is available, it is recommended that it should be used – at least to check on the MRL (*Many Reference Liquids*) calibration technique, see above. If resources allow, a complete *look-up table* relating ε^* to Γ may be constructed. A warning should be issued here that the computation itself should be checked in some way – e.g. by using reference liquids that are not part of the set used to calibrate the probe.

7.1.14 Free-Field Methods.

Introduction. Figure 47 shows a number of geometries employed for free-field measurements on dielectrics. The methods are best suited for materials which are *intended* for end-uses in the free-field, as they are likely to be the only materials available in large enough sizes to allow free-field methods to be effective. Typical materials are (i) *RAM* (Radiation Absorbing Materials – see Section 5.3.12) - high-loss materials used for absorbing free-field electromagnetic waves and (ii) *Radome materials* – typically low-loss materials used for protecting antennas from the elements (rain, wind, snow, etc.).

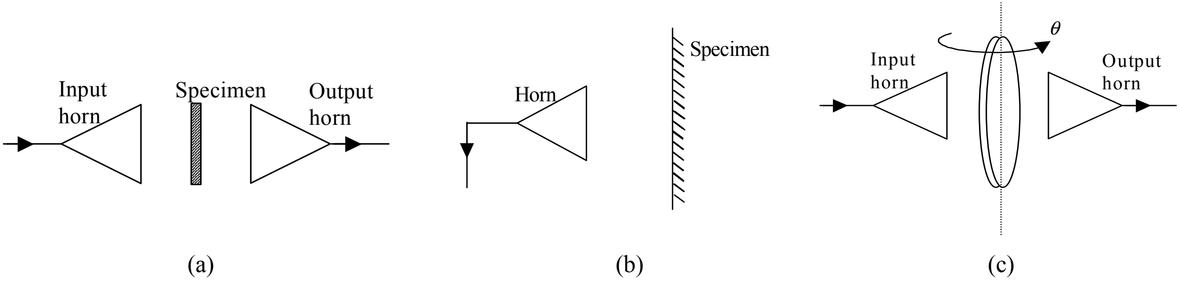


Figure 47. Free-Field methods: **(a)** normal transmission through a specimen between two horn antennas, **(b)** normal reflection from a specimen, **(c)** measurement of transmission as a function of the angle of incidence, see text.

Free-field methods may be categorised by three contrasting pairings of practical approaches to measurement:

(1) Transmission or Reflection? As with the *guided-wave* transmission line methods described in Sections 7.1.10 and 7.1.11, both reflection and transmission methods are possible. The discussion there may be followed to understand why transmission methods are often more accurate, but reflection methods are often better for high-loss materials and RAM, especially if only extrinsic parameters are required – see (2).

(2) Intrinsic or Extrinsic? *Intrinsic* measurements determine the intrinsic dielectric and magnetic properties of the d.u.t., i.e. ε^* and μ^*. *Extrinsic* measurements measure extrinsic parameters like *reflectivity* or *scattering* from materials and *transmission* through materials. End-users of free-field materials such as RAM and radome laminates are often more interested in the latter. Note, however, that intrinsic measurements are definitely required for electromagnetic design and optimisation purposes (as in the design of multilayer RAM see, e.g. [101]). For many free-field techniques, the actual measurement set-up needed to implement these two approaches may be identical, though extra mathematical treatments will be required to compute the intrinsic properties ε^* or μ^*. However, extrinsic measurements on a material ought ideally to be performed in a geometry that approximates to that dictated by the end use of the material, so one potentially has a wider range of measurement geometries at one's disposal for intrinsic measurements. For example, if the reflectivity of RAM at a 45° angle of incidence is required, then extrinsic measurements of reflectivity *should* be performed at this angle, whereas intrinsic measurements can be performed by any suitable method and the reflectivity at 45° incidence can subsequently be computed from Fresnel's equations [23, 10].

(3) Focussed (Quasi-Optical)-Beam or Unfocussed-Beam Methods? Free-fields are typically launched as *diverging* beams from antennas, this will happen in the unfocussed measurements measurement systems of Figure 47. In unfocussed beam methods no attempt is made to focus the beam with lenses or concave mirrors, so corrections for attenuation of the beam between transmitting and receiving antennas have to be incorporated into the method. Even if launched by a high-gain (e.g. a 25 dB-gain) antenna, the beam will typically extend beyond the aperture of the receiving antenna and may also spread significantly beyond the aperture of the specimen. Such *unfocussed* methods are therefore prone to errors caused by diffraction at specimen and antenna edges. They also require copious use of RAM around key system components to remove spurious signal reflections and their effects. The presence of diffraction inevitably limits the accuracy of such methods, compared with *guided-wave* and focussed-beam methods. In *focussed-beam* or *quasi-optical* methods, see Figure 48, lenses or concave mirrors are used to prevent divergence of the beam. An attempt may also be made to ensure that the beam is fully calculable.

Fully-calculable beams can be launched from *corrugated-horn antennas* [192, 193] as *Gaussian Beams* (GBs) [194, 2, 195] see also Figure 48. GBs are potentially fully calculable all along their length, they decay exponentially to insignificant amplitudes as one moves away from their axis of propagation and they can be focussed by concave mirrors or bloomed lenses [195]. Thus, diffraction problems may potentially be made negligible. The purest GBs used in RF & MW metrology are probably those set up in open-resonators (Section 7.1.17) because they are self-filtering. Methods based on travelling-wave focussed GBs, described below in this section, by contrast, are more difficult to control, though they should offer better uncertainties in measurements than unfocussed beams for the reasons given above.

One's practical ability to focus GBs improves as the frequency increases up through the millimetre-wave region and through the Terahertz region of the spectrum up to the infra-red. At sufficiently high frequencies *quasi-optical* methods essentially become *optical* methods. Practically useful Gaussian Beams typically have to be a few wavelengths in diameter at their narrowest point – their waist, see Figure 48 - otherwise they diverge too quickly to be considered properly as beams (i.e., in antenna parlance, they have low gain). At higher frequencies beams can be very narrow and exhibit low divergence – like a typical laser beam – and so significant problems of diffraction, especially diffraction by limited apertures, can be avoided.

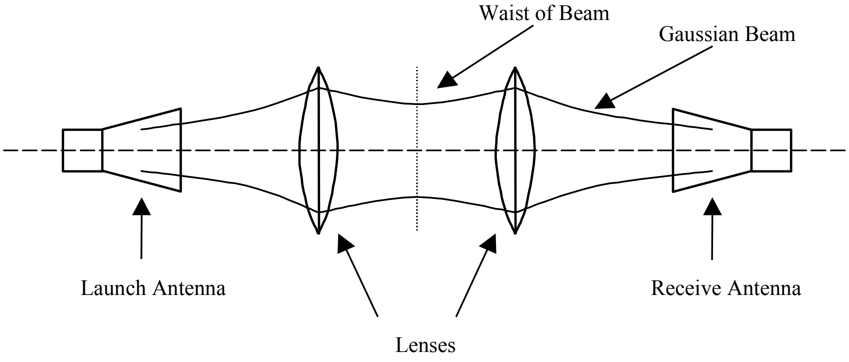


Figure 48. A focused free-field measurement system. The specimen is placed at the waist of the Beam. If corrugated-horn antennas are used, the beam could be a calculable Gaussian Beam.

Whether focussed or unfocussed, most conventional *free-field* travelling-wave methods share much in common with *guided* travelling-wave methods in that ε' is generally measured by phase change and dielectric loss is measured by the attenuation of the beam on insertion of the specimen. Likewise the use of open-resonators, which launch a *resonant* GB, see Section 7.1.17, shares much in common with resonant-cavity methods such as those used with the TE_{01}-mode cavity (Section 7.1.7) because ε' is generally measured by a change in length of the resonator or a frequency change, and the dielectric loss is determined from the Q-factor (see Section 6.1.9).

Time Domain Gating in Free-Field Measurements. The Method of *Time Domain Gating*, as described in Section 7.1.19, is very useful for improving the accuracy of free-field measurements (especially unfocussed measurements) as it allows the wanted signals from the d.u.t. to be separated from spurious reflections originating from elsewhere in the measurement system and from elsewhere in the laboratory.

Free-field Methods – A Short Survey. Examples of both unfocussed and focussed methods are given below. Full details are to be found in the quoted references. A method for characterising surface waves propagating on dielectric surfaces is also described briefly to demonstrate the versatility of free-field methods, this is based on *near-field* measurements with an *Optically Modulated Scatterer* (OMS).

(a) *The Arch Method: Unfocussed Measurements at Oblique and Near-Normal Incidence* [196, 197]. This is the 'traditional' method for measuring reflections from RAM as a function of angle. It suffers from diffraction and limited-aperture effects however, and so may not be effective for reflection coefficients less than about 40 dB – though this will depend upon how carefully the method is implemented. This technique is usually used to obtain the *extrinsic* parameters of RAM only (i.e. reflection coefficients only rather than ε^*), though the intrinsic properties can in principle be computed for plain-sided homogeneous RAM by using Fresnel's Equations [23] with a plane-wave approximation. *Time Domain Gating* (see above) can be particularly useful for reducing measurement errors in this technique.

(b) *Normal-Incidence Unfocussed Reflection and Transmission Methods.* A number of workers have used these methods for measuring large-area laminar specimens, see Figure 47 (a), (b) and [3] for a review. The measurements are readily performed by using matched waveguide horns attached via coaxial cables to an ANA, though, in the past, accurate measurement were performed with waveguide bridges [198] which may be considered as a cheaper option. Computation of transmission and reflection coefficients is relatively simple in a plane-wave approximation [199]. Such methods invariably become more accurate as the frequency increases towards the millimetre-wave bands because beams can be made narrower at higher frequencies, as discussed above. Clearly these methods can be converted into truly focussed-beam methods by using lensed-horns, which are commercially available. These implementations can be configured vertically to save space in laboratories.

(c) *Other Oblique-Incidence Unfocussed Reflection and Transmission Methods.* These use similar equipment to (b), however, a number of workers have introduced some degree of flexibility by rotating the specimen about an axis perpendicular to the direction of propagation of the beam, Figure 47(c). Thus, measurements can be performed at the Brewster angle of incidence [200] or else by measuring transmission coefficients as a function of the angle of incidence [201]. Either technique may well give better accuracy than a normal-incidence method (b), because the latter is more prone to problems from multiple reflections between the specimen and system components like lenses. Steps must be taken here, though, to absorb the off-axis beams that are reflected from the specimen in RAM so that they cannot reflect back and interfere with the measurement.

(d) *Normal Incidence Focussed Reflection and Transmission Methods.* These quasi-optical techniques have been made possible in recent decades by the advent of a full understanding of the theory of corrugated-horn antennas for launching travelling GB waves [192] and by the ability to manufacture such antennas. The corrugated horn can produce a 98% pure Gaussian Beam [193]. Complete measurement systems for laminar specimens are constructed either with mirrors [197] or lenses [202] for focussing. Systems are these days commercially available at frequencies from 1.5 GHz up to the sub-millimetre region of the spectrum. The antennas are generally connected via RF & MW coaxial cables or via waveguides directly to an ANA to measure transmission and reflection coefficients from the specimen. As in all GB measurements the specimen is best placed at its waist, where the beam is narrowest, so that the specimen can have a smaller diameter (see also Section 7.1.17 on open-resonators).

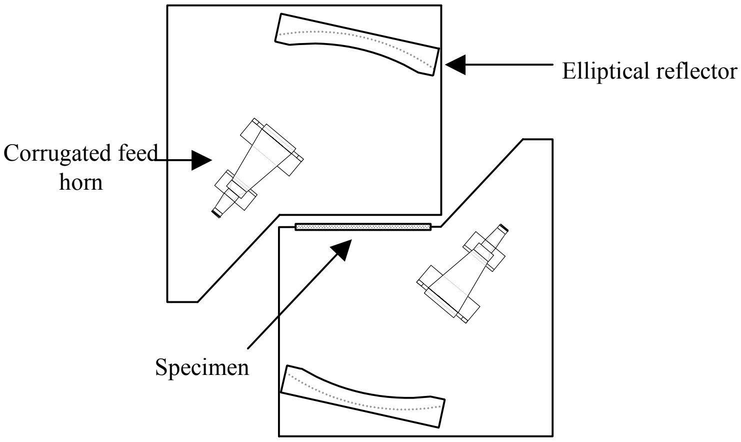


Figure 49. A quasi-optical focussed-beam technique for measuring the dielectric properties of a laminar specimen at normal incidence. Systems such as this have been constructed for frequencies as low as 1.5 GHz, but they also operate well up to sub-millimetre-wave frequencies.

A typical measurement system is shown in Figure 49. Concave ellipsoidal mirrors are preferred to lenses for focussing the beam because, even if bloomed [195], the latter suffer from multiple internal reflections, which effectively launch unwanted multiple GBs into the measurement system through internal and external reflections from their surfaces, thereby compromising the accuracy of the technique. As stated above, the GB is narrowest at its waist, where the specimen is placed and where the GB approximates to a plane wave. Measurements are invariably on laminar specimens that should have a minimum beam *diameter* of about five times that of the GB beam *radius* (as measured at its waist). The *measurement equations* (see the Glossary) that are generally used assume TEM plane-wave incidence and so are essentially the same as those used in ANA-based coaxial transmission line measurements (see Section 7.1.10) though, strictly, GBs exhibit an *anomalous dispersion* (see Section 7.1.17), i.e. their wavelength is not the same as that of a free-field plane-wave at the same frequency. This arguably ought to be taken into account when calculating ε' in the most accurate measurements. The size of the measurement system scales with wavelength, as does the requirement for high tolerances and quality of surface finish on the mirrors. Similar considerations apply to specimen flatness and positioning. One advantage is that air-gaps (such as those between the specimen and the cell shown in Figure 37) do not exist in this technique. However, much larger flat-sheet specimens are required. The ANA calibration method most suited to this technique is the TRL method [104]. Measurement uncertainties are higher in this technique than for the transmission-line method because of the use of plane-wave approximations and because of difficulties in specimen alignment and positioning. This method is particularly suited to measuring materials that have large-scale inhomogeneity, such as foams. Typically the beam diameter at the specimen surface approaches 300 mm at 8 GHz and so the measurement is effectively averaging the material properties over the area covered by the beam.

(e) *Measurement of Surface Waves above Dielectric Layers by an Optically Modulated Scatterer*. Surface waves [203] are evanescent E.M.-waves that 'adhere' to dielectric surfaces, exhibiting an exponential decay in amplitude as one moves away from the surface. Dielectric resonators exhibit this same phenomenon in all of their resonant modes but the phenomenon is most clearly seen in their Whispering-Gallery modes – see Section 7.1.15. In recent years, the study of surface waves has been revived [102, 103] and one device that is being used to study them extrinsically is the *Optically Modulated Scatterer* (OMS). A *Modulated Scatterer* [204] in general is a small dipole device (typically not more than 5 – 10 mm long) designed for measuring the magnitude and phase of free-field microwaves in a minimally invasive way. A portion of the microwave radiation incident upon such a dipole is scattered by it. By modulating the microwave impedance of the device, usually at a 1 – 100 kHz rate, the magnitude of the scattered microwave radiation is itself modulated and so it is possible to pick out this scattered wave from all other ambient radiation in the vicinity. The modulated scattered signal is received by a nearby antenna and synchronously detected. By scanning the scatterer through space on a regular grid or by using a regular array of modulated scatterers, one can map the E-field. *Optically* modulated scatterers have their impedance modulated by pulsed laser light incident upon an optical diode or transistor. This is usually guided by an optical fibre. Compared with other field-mapping techniques, e.g. the use of metal horn antennas for near-field-scanning, the OMS is minimally invasive because only the small dipole itself is made of metal. Such an OMS device has recently been shown to be effective for studying surface waves above low and high-loss Surface Wave Absorbing Material ('SWAM') surfaces in a specially constructed waveguide see Section 5.3.12 and References [102] and [103].

Uncertainties of Free-Field Techniques. It is difficult to be fully quantitative here because there are so many ways in which such measurements can be performed. It is safe to say, however, that unfocussed free-field methods, when used for intrinsic measurements of ε' will generally be less accurate than their guided-wave equivalents. Uncertainties may, however, lie in the range ± 1% to over ± 10 % for ε' and from ± 5% to over 20% for loss, even with high-loss materials. NB. Single-pass travelling-wave techniques will, as usual, be unsuitable for measuring low losses. There is no ultimate reason, however, why *focussed* methods cannot be as accurate as any guided-wave technique. This is evidenced, in fact, by the open-resonator (Section 7.1.17) - a quasi-optical resonator that offers one of the most accurate techniques discussed in this guide for measuring ε' and tan δ. This is probably only possible, however, if (i) focussing is carried out by concave mirrors rather than lenses (see above), and (ii) if the full theory of Gaussian Beams [194] is used rather than plane-wave and other approximations.

► ***Summary: Free-Field Methods***

Advantages of Free-Field Techniques.

Relatively easy mounting of specimens and easy access to specimens.
Ideally suited for materials that are ultimately to be used in the free-field.
Extrinsic measurements of reflection coefficient and transmission coefficients measurements can be performed in geometries close to those in which typical free-field materials (e.g. RAM or radome material) will ultimately be employed.

Disadvantages of Free-Field Techniques.

Unfocussed methods are generally less accurate than the equivalent guided-wave methods.
Specimens generally have to be large in cross section: typically more than 20 wavelengths across for unfocussed techniques and 6 wavelengths for focussed techniques.
Measurement systems can take up a large amount of laboratory space.

Free-field: Typical Sources of Dominant Uncertainties:

Diffraction from specimen edges and from antennas and other components.
Inadequate or oversimplified modelling of beams (e.g. use of plane-wave assumptions).
Mismounting of specimens (e.g. when the specimen is not situated exactly where the theory assumes that it is).

► ***Do's and Don'ts for Free-Field Measurements***

Do check that the specimen cross-section is adequate for the beam being used.

Do remove spurious beams and scattered radiation from the measurements system by copious use of carefully placed and carefully chosen RAM.

Do investigate to see if ANA-based *Time Domain Gating* (Section 7.1.19) can be used to improve your measurement uncertainties.

Don't forget that the ambient conditions in the measurement laboratory (e.g. air-flow, moist atmosphere) can affect specimens and the stability of your measurements.

7.1.15 Dielectric Resonators

Dielectric Resonators (DRs) are widely used in electronics and telecommunications applications as high-Q components for narrow-band filters. The theory of their resonances is well developed [127]. The resonators typically take the form of 'puck'-shaped cylinders of dielectric material. They can retain the E.M.-fields that are resonating inside them because they are totally-internally-reflected from the interior of the dielectric surfaces. A formal analysis of the fields [*ibid.*] reveals that there are also E.M. evanescent fields in the air (or other dielectric medium) that surrounds the resonator and that these fields decay exponentially in magnitude as one moves away from the resonator. It is the presence of these fields that allows one to couple RF & MW power into the DR, typically via coupling loops at the end of coaxial line feeds. However, these fields also interact with other objects in the vicinity of the resonator (e.g. its support or container) and if these nearby objects are lossy the resonance becomes loaded and the Q-factor falls – See Section 6.4. Figure 50 shows the coupling geometry for two resonator configurations commonly used in dielectric metrology. Typical resonator sizes range from tens of centimetres on a side in 900-MHz cell-phone base-station applications, down to a centimetre on a side or less at around 10 GHz. The resonant frequency depends, of course, not just on the size of the dielectric resonator but also on the permittivity of the material from which it is made. For most practical applications low-loss high-permittivity ceramic dielectrics are preferred ($\varepsilon' > 30$), Higher permittivity is better because smaller resonators can be used at any given frequency and because the fields of the resonator are contained more effectively within the dielectric, so the loading effect of lossy materials surrounding the resonator is less. One of the great challenges in the field of electroceramics in recent years has been the search for better DR materials with higher permittivities, especially, materials that also exhibit lower losses.

Dielectric Property Measurements with Dielectric Resonators. If used with suitable numerical modelling (FDTD or modal analyses are typical, see Section 6.9) DR measurements on 'puck'-shaped specimens offer one of the most accurate and sensitive methods available to us for measuring the permittivity and loss of low-loss dielectric materials. They have a major advantage over other resonant techniques for characterising low-loss materials: the attainable filling-factors (see Section 6.1.9 and Equation 6.3a) in this technique are normally close to 1 because most of the energy in the resonance is contained in the dielectric itself. There is therefore little dilution of sensitivity when measuring loss, as there is, for example, in typical TE_{01}-mode resonators or open-resonators in which the specimen only fills part of the resonator volume (see Sections 7.1.7 and 7.1.17, respectively).

The E.M. fields outside the DR offer both metrological advantages and disadvantages. As stated above, they allow one to couple power into and out of the resonance, but they also load the resonance through interactions with the DR's support structure and the metal cells into which it is placed. The major *disadvantage* here compared with the TE_{01}-mode or open-resonator methods is that a substitution technique (Section 4.4.1) cannot be directly applied to measure the surface resistance of the cell (Sections 6.1.9, 6.4.3) *at the measurement frequency*, because without the DR itself present, the cell generally cannot be made to resonate in the same frequency range. In general it will only resonate at a much higher frequency, so some other way must be used to correct for the unwanted loss. See below for methods that can overcome this problem.

Dielectric measurements with DRs are usually performed with the $TE_{01\delta}$-mode in which the E-field is circularly polarised, but higher order 'whispering-gallery modes' have also been used to obtain dielectric data at much higher frequencies (see below). When viewed on an ANA or spectrum analyser, one can see that DRs resonate in many different modes. One of the main preliminary steps that must be undertaken before measurement, therefore, is to identify the $TE_{01\delta}$ mode – methods for achieving this are also discussed below.

The diameter, *d*, to height, *h,* ratio *d/h* of a typical DR puck designed for practical applications is usually chosen to optimise the Q-factor for a given specimen volume. This optimum *d:h* ratio varies from about 0.4 for $\varepsilon' \approx 100$ to about 4 for $\varepsilon' \approx 2$. However, it should be pointed out that one is not restricted to this ideal ratio for measurements. *Any* shape will resonate, although for practical metrological applications one will normally be measuring a puck-shaped specimen since DR computer programs normally assume such a shape. This opens up metrological opportunities: specimens produced for measurement by other techniques (e.g. in TE_{01}-mode resonators or open-resonators) can also be resonated as DRs, even if they are flat laminar discs with $h << d$. This can have two advantages:

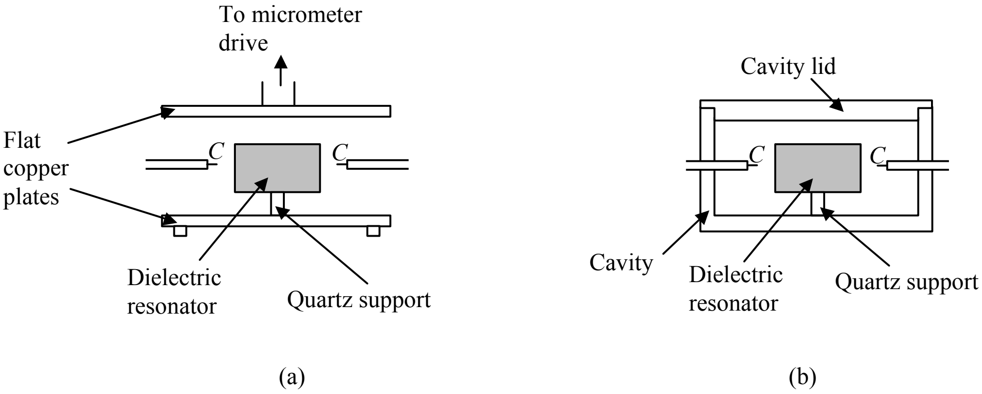
(i) Such a specimen will almost certainly resonate at a different frequency when used as a DR to that of the cell for which it was designed; so the one specimen can be measured at two or more frequencies.

(ii) Measurement comparisons are good practice (see Section 4.4.3). Even if the measurements are at different frequencies, the value of ε' obtained by resonating the specimen as a DR should agree with the value obtained by other methods, the reason being that if the specimen is low loss, ε' changes very little with frequency, see Section 2.5.

Many specimens used in practical applications are ring-shaped – with a hole on-axis through their centre. The mathematical theory of $TE_{01\delta}$-mode resonances in a ring-shaped specimen is a simple extension of that for a full cylinder, so this is also a viable DR shape for dielectric measurements. This enables one to measure specimens that are designed for the coaxial line method of Section 7.1.10 by the DR technique, though the measurement frequency of the latter will probably be higher than is practical for the coaxial line.

One important point must be noted here: fundamental $TE_{01\delta}$-mode DRs *must* be used in a container or cell, otherwise their Q-factors are loaded by *radiative losses*. The two types of cell most commonly employed in dielectric metrology are shown in Figure 50, they are designed to prevent this. In the *Courtney Holder* or *Hakki-Colemen Cell* of Figure 50 (a) the distance between the top and bottom plates must be less than $\lambda/2$ in air if this radiation loss is to be avoided. (NB. radiation loss is not so important for some higher order modes, like the whispering-gallery modes, see below). The *Cavity Cell* of Figure 50 (b) effectively prevents radiation escaping by completely enclosing the specimen around with metal walls. These walls should be well separated, ideally by at least one specimen diameter, from the specimen to minimise losses from currents flowing in them. For the same reason, the specimen is normally placed on a small post or tube made from low-loss dielectric (e.g. quartz) to displace it away from the base of the cavity. In either case the cells are often made from copper because its high conductivity reduces the metal losses in the walls. Further details on the use of these two types of measurement cells are given below.

Examples of the recent use of DRs for dielectric measurements can be found in the literature [205, 132] and they demonstrate the versatility of the technique.



(a) (b)

Figure 50. Dielectric Resonator Cells **(a)** The Hakki-Coleman Cell, otherwise known as the Courtney Holder and the Parallel Plate Cell, **(b)** The Cavity Cell. In both diagrams '*C*' marks the coupling loops.

<u>*Intrinsic and Extrinsic Measurements*</u> There may be two reasons why we may wish to perform DR dielectric measurements. Firstly we may wish to know the *intrinsic* dielectric properties of the DR specimen material:

(1) The real permittivity, ε', of the material of the resonator.
(2) The loss tangent, tan δ, of the material of the resonator.

Secondly we may ultimately wish to use the DR *as* a DR, e.g. as a filter in an electronic circuit. Then we will want to measure the following *extrinsic* parameters:

(3) The resonant frequency of the resonance
(4) The Q-factor of the resonance
(5) The temperature coefficient of resonant frequency (t.c.r.f.) of the DR.

In practice, similar measurement configurations can be used for either type of measurement, but note that t.c.r.f. is a very important parameter for practical applications. In fact, one usually wants the t.c.r.f. of a DR in its end-use application to be as close to zero as possible, so it is important to measure it accurately and this entails careful design of the measurement cell for variable temperature measurements. Note, also, that the t.c.r.f. cannot just be computed from the change of ε' with temperature – it also depends upon the expansion coefficient of the dielectric material.

Note that resonator size can be a problem: manufacturers of DRs for telecoms filter applications at 900 MHz often choose to test materials at higher frequencies (e.g. 4 GHz) because smaller DRs can be used.

International Standards IEC 61338, Sections 1-3 [60] provides a description of most aspects of the Courtney Holder method. It appears to have developed out of a measurement comparison [206] and is very comprehensive but it does not cover the cavity technique at all and so does not address the issue of choosing between the Courtney and cavity methods. Another standard IEC 60377-2, '*Part 2: Resonance Methods*' [59] does cover the cavity as well as the Courtney resonator but not in depth.

Use and Choice of Cells The type of cell shown in Figure 50 (a), is known variously as the *Courtney* [207], *Hakki-Coleman* [208] or *Parallel-Plate* resonator cell, see [127] Ch. 3.8, and has been in use at least since the 1960s for measurement of high-permittivity, low-loss dielectrics. It benefits from a very simple measurement configuration and easy access for introducing and removing specimens. For maximum flexibility and to help to identify resonance modes (see below) the plate spacing may be made adjustable, ideally under micrometer control. A high degree of flatness and parallelism of the resonator plates is very important in this technique if the Q-factor is not to be reduced. Flatness of the resonator plates should be within 5 μm for measurements of the intrinsic material parameters, ε' and $\tan\delta$. If necessary this can be achieved by 'diamond turning'. For the *extrinsic* resonator parameters (3) – (5) above, tolerances may sometimes be less restrictive, depending on application. The parallelism of resonator plates should be checked and optimised. One technique to achieve this [60] is to measure the parallel-plate resonances in the absence of a specimen (NB. this will be at a much higher frequency). The empty resonator should be connected to an ANA or spectrum analyser and changes in its resonance observed as one of the plates is tilted in and out of parallelism with the other plate. Assuming the plates to be perfectly flat, they should be parallel when the resonance peak is narrowest and has maximum amplitude, i.e. when the highest Q-factor is obtained, because in this case the radiation losses are minimised.

Cavity Cells clearly have an advantage over parallel-plate cells by being relatively easy to temperature control, say in an oven or by circulating water in the cavity walls. They are also suitable for cryogenic measurements [132]. However the *electrical isolation* of the specimen from the cell walls, especially if it rests in the centre of the cavity on a small spacer post, also ensures that the specimen is quite well *thermally isolated*. The thermal time constant of the DR specimen can therefore be quite long and time must be allowed in variable-temperature measurements for its temperature to settle.

In both the Courtney and the cavity cell of Figure 50 (b) a spacer, such as a narrow single-crystal quartz cylinder or tube, can be placed between the resonator base-plate and the lower specimen surface to reduce the effect of wall losses. Quartz is used because (i) it has very low loss, (ii) it has relatively low permittivity (~ 4) compared with most DR materials of interest. The spacer should be placed where the azimuthal electric field is minimum, i.e. at the centre of the cell. The measurement equations must take account of the presence of the air gap under the specimen, but the spacer itself can often be ignored in the modelling precisely because it lies at an E-field minimum. In all cases the cylindrical symmetry of the measurement geometry should be preserved.

The advantages and disadvantages of the two cells may be summarised as follows – the rationale for these judgements is explained elsewhere in this Section.

Parallel-Plate Cell Advantages:

(1) Capable of covering a larger range of specimen sizes if the metal-plate spacing can be changed.
(2) Mode identification is easier than in cavities if the metal-plate spacing can be changed.
(3) Easier access for specimens.

Parallel-Plate Cell Disadvantages:

(1) More difficult to temperature control.

Cavity Cell Advantages:

(1) More easily temperature controlled: usually the better cell for measuring t.c.r.f.
(2) Machining of the internal metal surfaces is not so critical as in the parallel-plate cell.
(3) For *extrinsic* measurements, the cavity geometry is similar to that employed in a simple filter, which may be the ultimate application of the DR material.

Cavity Cell Disadvantages:

(1) It can be difficult to identify modes.
(2) Access for inserting the specimen is more difficult.
(3) Speed of temperature control may be low because of the thermal isolation of the specimen.

Coupling probes. These may be made from semi-rigid cable, with the protruding inner-conductor formed into a loop, the end of which is soldered onto the outer-conductor. The loops should be orientated *parallel* to the end faces of the DR for detection of TE modes and to prevent excitation of TM modes. (These probes can temporarily be turned slightly, however, in preliminary measurements if one wishes to detect or identify TM modes, which one sometimes needs to do to ensure that one has identified the $TE_{01\delta}$ mode correctly, see next paragraph). Two approaches are commonly used for coupling into the resonator and measuring the Q-factor of the resonance. In *transmission* measurements (see Sections 6.4.2 and 6.4.5) it is recommended that the probes should not be placed too close to the DR because this will avoid distortion of the field configuration inside the specimen. A good compromise is to position the probes *symmetrically*, at a distance for which the resonant mode gives approximately 40 dB insertion loss on resonance. In this case the loading of the DR resonance by the coupling can usually be neglected for materials with $\tan\delta > 10^{-5}$. Losses from coupling can be made arbitrarily small, at the expense of worse signal-to-noise ratio, by using weaker coupling. The other approach that has seen wide use is a *reflection* method (see Section 6.4). It uses a large coupling loop that is adjusted to give *critical coupling*. A problem with this approach is that the coupling will certainly distort the E.M. fields in the cell in an unquantifiable way. It also requires more measurement adjustments to be made than the transmission approach. The transmission method is preferred at NPL because it has been used successfully for many years in conjunction with other resonators, as well as with DRs.

Mode identification. The Courtney parallel-plate cell has an advantage here over the cavity if the former has adjustable plate-spacing. If the $TE_{01\delta}$ mode is required, a rapid identification can be made by varying the specimen-to-plate spacing and selecting the lowest resonance for which the centre frequency *decreases* as the plate spacing is increased. For specimens with $d/h > 2$, this is typically the *second* resonance observed on the ANA. The HE_{111} mode [127] is typically the lowest resonance, and the $TE_{01\delta}$ the second. The HE_{111} is a doublet, although this cannot always be seen. It is much harder to identify the resonant modes in a fixed-geometry cavity but the task is made considerably easier when the permittivity of the specimen is known approximately in advance. One problem may be that one's numerical model of the cavity may not be able to predict the frequency of HE or TM modes because one will not normally be using them to characterise the DR material! One useful trick that can help here is to use a computer-modelling package that has an *Eigenvalue Solver* and which can plot E.M.-field patterns graphically, see Section 6.9. The eigenvalue-solver should be able to identify all of the resonant modes of the cell, and these can be compared in the frequency domain with those actually measured. The field patterns can be inspected visually on the computer screen using the modelling software in order to identify which of the resonant modes is the $TE_{01\delta}$ mode. This will only be helpful, however, if ε' is already known approximately. If it is not, it may be useful to perform a preliminary measurement in a Courtney cell to obtain an initial value for ε' even if one's 'definitive' measurements are to be performed in a cavity cell.

Correction for the metal losses in measurement cells. This is addressed to some extent in the international standards discussed above. An independent method to determine surface resistance of the metal in cells is to measure a very low-loss resonator material, e.g. single-crystal sapphire [205]. Alternatively, in the case of the cavity cell, one can resonate it *without* a specimen in it at a higher frequency and then estimate the metal loss at the measurement frequency by extrapolating the skin-depth down to the lower frequency (see e.g. [21] for the general behaviour of skin-depth with frequency). The relationship between skin-depth and metal loss is given in Section 6.4.3 in the discussion of Equation 6.3b. This method will probably not work with the parallel-plate cells because there could be significant radiative losses around the edge of the plates when they are resonated without specimens present. Note that the surface resistance is temperature-dependent and this has to be taken into account in variable-temperature measurements. The empty-cavity resonance method just described can be used to measure the variation in surface resistance, R_s, with temperature. Again, however, extrapolation to the frequency of measurement has to be carried out and one must make use of the assumption that the rate of change of R_s with temperature is the same at both frequencies.

Whispering-Gallery Modes. These are higher-frequency, higher-order resonance modes that store most of the power in their resonances close to the internal circumferential surface of the dielectric resonator. In a travelling-wave model of these modes, the wave travels internally around the circumference of the resonator, being continually totally-internally-reflected from the surface. If the wave returns to its starting point *in-phase* with itself, a resonance will occur. As ever with dielectric resonator modes, there are evanescent E.M.-fields in the air surrounding the resonator. If a puck-shaped DR resonates at, say, 1 GHz in the $TE_{01\delta}$-mode, its whispering-gallery modes of general interest may resonate, typically, in the range 8 – 20 GHz. Applications of these modes include the construction of high Q-factor resonators and filters, but they can also be used for dielectric measurements on the material of the puck [209]. This may be useful because the same puck can potentially be measured at a number of different resonant frequencies. The evanescent fields of whispering-gallery modes are typically much easier to contain than those of the $TE_{01\delta}$-mode and in some cases it may be possible to dispense with a purpose-built cell for these measurements. Whispering gallery modes have been employed for measurements both upon pure dielectrics [209] and upon ferrites [210].

► ***Summary: Dielectric Resonators***

Advantages of Dielectric Resonators

They offer one of the most sensitive techniques for measuring very low loss, including sub-microradian loss angles at cryogenic temperatures (but see Uncertainties, below).

They offer one of the most accurate methods for ε' at microwave frequencies - as long as specimens are well machined, uncertainties better than ± 0.5% may be possible.

Disadvantages of Dielectric Resonators

Suitable only for low-loss specimens, typically $\tan \delta < 0.003$, depending on one's ability to measure Q-factor.

Specimens must have a circular cross-section and should ideally be puck-shaped (though thinner laminas may be measurable as well).

Best suited to high permittivities, typically $\varepsilon' > 6$; lower permittivities can be measured, however, but with higher uncertainties.

Not a substitution method (Section 4.4.1) so the metal loss in the DR cell must be determined – this can be difficult to undertake accurately and can generate systematic error if the loading of the resonance by the metal loss is not estimated correctly.

Dielectric Resonators: Typical Sources of Dominant Uncertainties:

Uncertainties in the surface resistance of the metal plates or the cavity walls.

Uncertainties in the Q-factor measurement (due to noise, asymmetry and loading).

Surface roughness of specimens (causing the calculated real permittivity to be *over*valued).

Uncertainties in specimen dimensions.

7.1.16 The Resonator Perturbation Technique

High-Q cavity resonators are highly sensitive measurement devices and this makes it possible to measure very small dielectric specimens inside them. If a sufficiently small specimen is inserted into a resonator and if no other changes are made to the measurement geometry, the resonant frequency f_r and Q-factor, Q, of the resonator will both change by a small amount: let us say by Δf_r and ΔQ respectively. If both $\Delta f_r / f_r$ and $\Delta Q/Q$ are small (typically less than 5%) and the volume of the specimen is small compared with the volume of the resonator, computational techniques based upon a first-order perturbation theory may then be used to calculate the permittivity, ε', and the loss tangent, $\tan \delta$, of the specimen. Such a measurement technique is referred to as a *Resonator (or Cavity) Perturbation Technique* [115]. One example of this approach is the application of perturbation theory to measurements in TM_{010} or TM_{020}-mode cavities when they are used with narrow rod specimens as described in Section 7.1.6, see Figure 34. A number of advantages can accrue from using such a perturbation technique:

(i) The *measurement equations* (see Glossary) are much simpler. Insertion of *large* specimens into cavities can render a formal modal analysis of the fields inside them very difficult. In the days before the computer modelling of E.M. fields became readily possible it was important to develop measurement techniques that would render the mathematics tractable enough to deliver relatively simple measurement equations. This is somewhat less important nowadays, since full modal or discretised analysis (see Section 6.9) is possible using computer programs. Nevertheless, use of a perturbation method can still be more convenient because one does not have to commit time and effort to writing and validating complex computer programs to perform the analysis. An example of the simplicity of the measurement equations that can be derived is provided by Equations 7.4 (a) and 7.4 (b) in Section 7.1.6, which are for TM_{010} and TM_{020}–mode cavities.

(ii) Even *high-loss materials* may be measured, if the specimens are sufficiently small, and if $\Delta Q/Q$ is also small. In fact, perturbation methods are normally the only resonator methods commonly used for measuring high-loss materials.

(iii) By placing small specimens in a cavity at points where the direction of the E- and/or H-fields are well defined, the *anisotropy* of permittivity, ε^*, and of permeability, μ^*, can be measured. *Small* specimens have an advantage here. In *large* specimens the E- or H-field direction and field-strength will typically be different in different parts of the specimen (though there are some resonator modes where the E- or H-fields can be uniform over a larger volume, e.g. E-field on the axis of a TM_{010}–mode resonator). The advantage of using *small* specimens here is that the direction and field-strength of E.M. fields in a high-Q resonator *on resonance* is *locally* uniform and well-defined. For this reason the perturbation method is well suited to the measurement of microwave ferrimagnetic resonance and of the permeability of ferrites [31, 32, 211] under the influence of static H-fields. As pointed out in Section 5.3.5, small *spherical* specimens are used for these measurements, because only in such specimens is the internal static H-field uniform when the externally applied H-field is uniform [*ibid.*].

The perturbation method has been in use at least since the 1940s [150, 151], but Waldron later extended and popularised it in the 1960s. His book [115] still provides one of the best guides to its application. The important skill in designing such a cavity perturbation technique lies in placing the specimen into exactly the correct part of a cavity and in giving the specimen exactly the correct shape to make the field perturbation equations simple. The most commonly-used types of perturbation cavities are the TM_{010}–mode cavity (Section 7.1.6) and the cavities used for characterising ferrites, just discussed in (iii). There are a number of more recent applications described in the scientific literature, however, e.g. [137, 149, 212].

When deriving measurement equations, one has the option these days of choosing between a perturbation approach and a more comprehensive approach using computer-based numerical analysis. There are many *pros* and *cons*, some of which are discussed in Section 7.1.6 on TM_{010} and TM_{020}–mode resonators, but the most important issue to be decided is whether the anticipated size of the perturbation to be obtained will lie within the valid limits of the approximations used. Waldron [115] covers both this issue and that of the wider use of perturbation methods. From a practical point of view, the most important thing to do is to check the validity of perturbation measurements by measuring a reference specimen which has known dielectric properties (see Section 4.4.2).

► ***Do's and Don'ts for Perturbation Methods***

Do check that the approximations arising from the perturbation theory are valid for your specimens, if possible, by measuring a specimen of a reference dielectric material with already-known properties and with roughly the same size and dielectric properties as your specimens-under-test. This approach should deliver useful information that can be entered into the uncertainty budget for the measurements.

Don't assume just that the perturbation approximations are valid, check them!

7.1.17 Open-Resonators

Open-Resonators are microwave Fabry-Perot interferometers [213]. Such instruments provide one of the most accurate and important methods available to us for measuring low-loss dielectrics at millimetre-wave frequencies. Achievable uncertainties can be as low as ± 0.2% for ε' below 3 and better than ± 1% for $\varepsilon' \approx 50$. Uncertainties better than ± 10% for $\tan\delta$ are also possible for specimens with loss angle above 200 μradians, while resolutions for loss down to 20 μradians or less are possible if the unloaded Q-factor of the resonator is greater than 150,000. Typical unloaded Q-factors are 60,000 at 10 GHz rising to over 200,000 at frequencies above 100 GHz, though recently [117] much better performance figures have been achieved, see below. Two typical open-resonator configurations for dielectric measurement are shown in Figure 51. Typical sources of loss in an open-resonator are shown in Figure 21, Section 6.1.9.

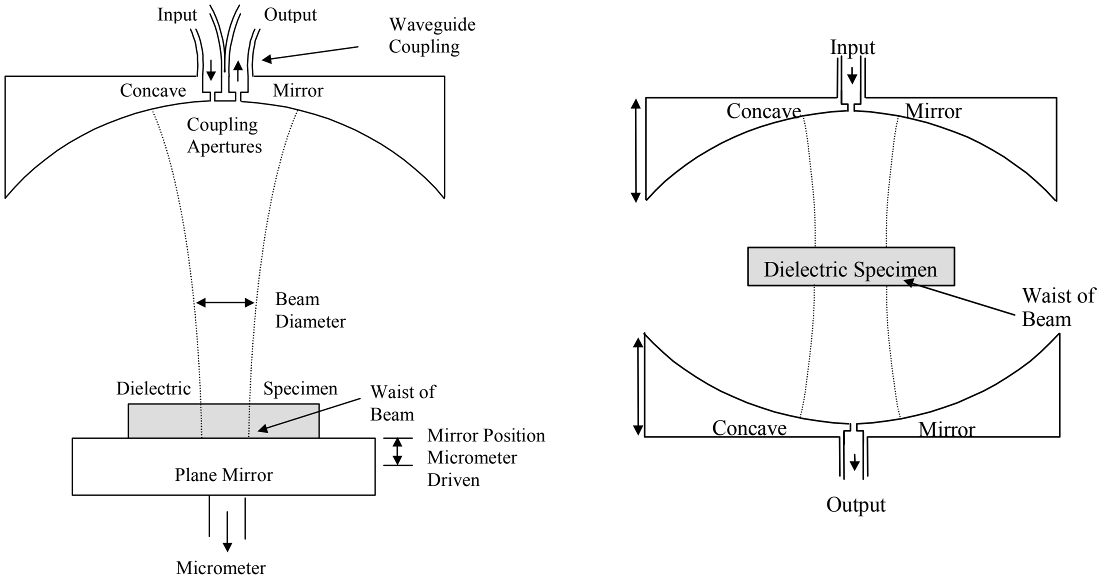
In terms of the classification of free-field methods given in Section 7.1.14, the open-resonator technique is a *focussed-beam* or *quasi-optical* technique. As a resonance technique it is naturally best suited for low losses, but it also possesses all of the advantages of free-field techniques, e.g. easy insertion and removal of the specimens. The resonant mode used in the open-resonator is the fundamental TEM_{00n} Gaussian Beam (GB) mode [194] and the resonance itself acts as a filter that ensures that this GB mode can be very pure.

The cross-sectional shape and size of specimens for open-resonators is not important *provided* they are large enough to encompass the whole cross-section of the Gaussian Beam at its waist. Required specimen diameters depend on frequency: the following are typical: at 10 GHz typically a 200-mm diameter is required; at 36 GHz a 50-mm diameter is required and at 72 GHz a 35-mm diameter is required. These values should only be taken as a guide, however. The best practice is to compute the *radius*, w_0, of the Gaussian Beam at its waist and then use a specimen *diameter* of at least $5 \times w_0$. A larger diameter is advised for very low-loss specimens as there could be small diffraction losses at the edge of the specimen even with a diameter of $5 \times w_0$. It can be seen that quite large diameters are required at lower microwave frequencies and this is one reason why closed cavities are normally preferred for measurements at such frequencies. For example, at 10 GHz, TE_{01}-mode cavities (Section 7.1.7) typically need specimens of only 60-mm diameter *or less*, whereas the open-resonator requires at least 200 mm. At millimetre-wave frequencies above 30 GHz, specimen diameter requirements for open-resonators are not such a problem and at these frequencies open-resonators have two advantages over typical resonators: (i) As stated above they allow easy access and removal of specimens, (ii) their resonance spectra are less dense than those of typical cavities of comparable size and so there is less likelihood of measurement errors being caused by mode-coincidences. A third advantage (at all frequencies) over most microwave cavities (e.g. TE_{01}, TM_{01n}, $TE_{01\delta}$) is that the resonant mode employed in these measurements is the fundamental TEM mode which is *linearly* polarised – thus enabling *specimen anisotropy* to be measured [100] – see below.

Practical Measurements The use of open-resonators for dielectric measurements has been described in great detail in a number of reviews, scientific papers and books: [214, 215, 216, 213], and the reader is referred to these sources for full details of methods and measurement equations. Reference [215] is, to our knowledge, the most comprehensive survey of microwave open-resonators compiled in recent years. We will limit ourselves here to practical advice, pointing out the pitfalls in such measurements. The rationale for the points made here is to be found in the references just cited above. Note that – as with most other resonator techniques discussed in this Guide - either the change-of-frequency [214] or change-of-length [216] (i.e. 'equivalent-thickness'), methods can be used for measuring ε'. Change-of-frequency is usually used for measurement of $\tan\delta$ – though change-of-resonator-length can be used for measuring Q-factor if the specimen is lossy, for example, if it reduces the loaded Q-factor significantly below 10,000 when it is inserted into the resonator.

(a) *Resonator Geometries.* Figure 51 shows two geometries that have been used for dielectric specimen measurements. If the configuration in Figure 51 (a) is used and the resonator axis is vertical, with the concave mirror above the plane mirror, one has the advantage that the specimen can easily be placed in the resonator directly onto the plane mirror. Complications can arise, however, if the specimen is warped (see (b) below). If coupling holes (see Sections 6.1.9 and 6.4.3) are used to couple power into and out of the resonator of Figure 51 (a) they should normally be in the concave mirror, as shown, rather than the plane mirror. The latter is at the waist of the beam where all of its power is focussed. For reasons given in Section 6.1.9, one should use as low a coupling coefficient as possible, consistent with achieving good detected signal-to-noise ratio. If the coupling holes were placed in the plane mirror, the proximity of the dielectric specimen (which rests on the plane mirror) will modify the coupling coefficient, thereby changing the loading of the resonator and so generating measurement errors in $\tan\delta$. (NB. Contrary to the principle just stated, resonators *have* been constructed with coupling holes in the plane mirror but this was done specifically to measure higher-loss specimens, and in this case the specimen is displaced from the mirror to prevent this spurious effect [217] – see also *'New Methods'* below.

(b) *Requirements for Specimens.* Specimen diameters have been discussed above. As in many other resonators, flat laminar specimens are used. Ideally, to achieve lower measurement uncertainties specimens should be an integral number of half-wavelengths thick (see Section 5.4.1) and just *one* half-wavelength thick if the permittivity is high (say, above 20) to avoid ambiguity over just how many wave-nodes lie in the specimen – this gives rise to ambiguity in measured ε'. Note that we are referring here to the half-wavelength in the medium of the dielectric specimen, which is reduced by a factor $\sqrt{\varepsilon'}$ compared with its value in air.



(a) (b)

Figure 51. Two open-resonator geometries that can be used for characterising dielectrics at millimetre-wave frequencies.

(c) *Warped Specimens.* Warped and dished specimens can cause significant measurement errors, particularly in the measurement configuration shown in Figure 51 (a). The warping of the specimen will raise its top surface away from the plane mirror. As the E-field close to the mirror is very low (it is at a node) any air-gaps there will have little effect on the resonance, so the net effect is to make the specimen appear to the E.M. fields to be *thicker* than it actually is. A measurement correction can therefore be made by taking *the height of the centre of the top of the specimen above the mirror* as its thickness. The *specimen centre* should be used because that is where the Gaussian Beam has its maximum amplitude and so it is where the presence of dielectric material will have the highest effect upon the resonance. If the specimen is *dished* (rather than arbitrarily warped), the same principle can be used to show that the specimen should be placed on the mirror *convex side downwards* so that it is *touching the mirror at its centre* and on the axis of the resonance, as the normal measurement equations assume.

(d) *Measurement of Anisotropy.* This is explained in the paper by Jones [100]. For uniaxial materials, any anisotropy of permittivity in the plane of the specimen will give rise to two separate resonances, $R_\perp$ and $R_{||}$. If the frequency is kept constant the system will have two different resonance lengths, $d_\perp$ and $d_{||}$, corresponding, respectively to the two different permittivities of the specimen $\varepsilon'_\perp$ and $\varepsilon'_{||}$, see Section 5.3.11. When the specimen is rotated about its centre, see Figure 52 (a), $R_\perp$ and $R_{||}$ will vary in amplitude in antiphase with each other, see Figure 52 (b). $R_{||}$ will reach its maximum when the E-field is parallel to the optic-axis (when the amplitude of $R_\perp$ will be zero) and $R_\perp$ will reach its maximum when the E-field is orthogonal to the optic-axis (when the amplitude of $R_{||}$ will be zero). When the E-field is at 45° to the optic-axis $R_\perp$ and $R_{||}$ will have equal amplitude. Therefore the optic-axis in an anisotropic dielectric can easily be identified by rotating the specimen about the axis of the resonator. Measurements of $d_\perp$ and $d_{||}$ will therefore deliver $\varepsilon'_\perp$ and $\varepsilon'_{||}$ as required. Similarly, if a frequency-difference (constant resonator length) method is being used to measure ε', two resonant frequencies $f_\perp$ and $f_{||}$ will readily be identified, and they will deliver $\varepsilon'_\perp$ and $\varepsilon'_{||}$, respectively. As $R_\perp$ and $R_{||}$ can be isolated from each other by rotating the specimen to the correct angles, the imaginary part of the two complex permittivities (i.e. their loss) can also be measured in the normal way (i.e. change of Q-factor). Degrees of anisotropy of 1% or lower can easily be measured by this method.

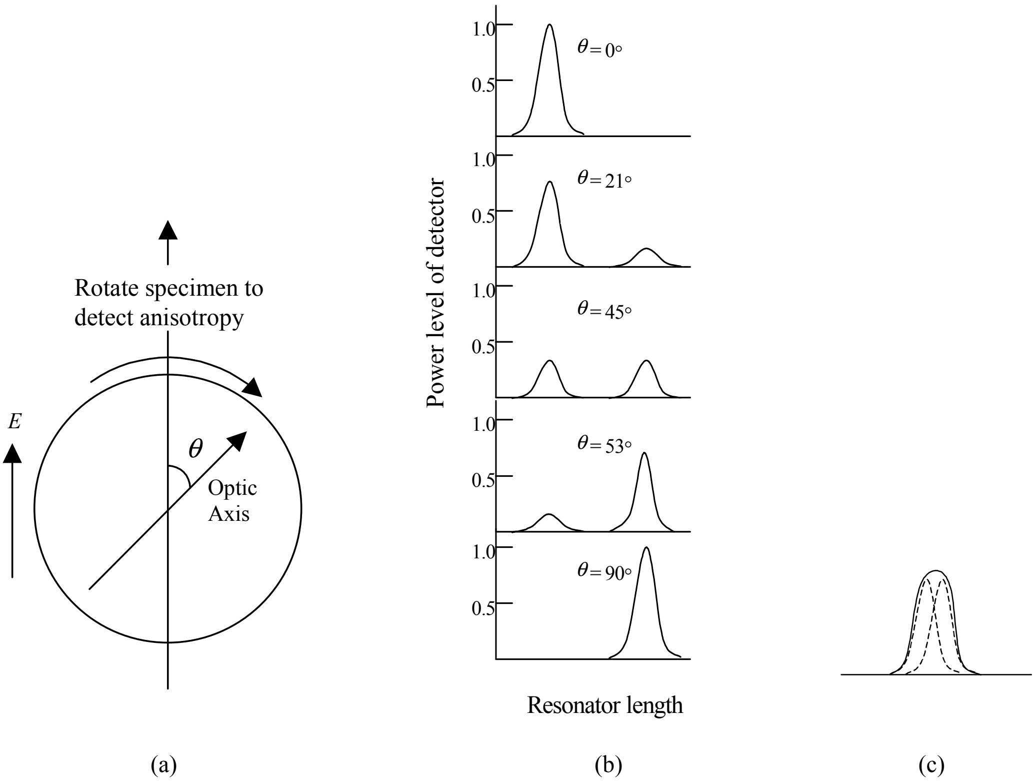


Figure 52. Measurement of uniaxial anisotropy in an open-resonator in a specimen in which the optic axis lies in the plane of the laminar specimen. **(a)** The specimen must be rotated about the resonator axis to detect anisotropy. **(b)** If the specimen *is* anisotropic two independent resonances can be detected whose amplitudes vary mutually out-of-phase as the specimen is rotated (after Jones [100]). **(c)** If the degree of anisotropy of the specimen is low, however, the two resonances can overlap and this can cause errors in dielectric-loss measurement. If the specimen happens to be oriented with its optic-axis at ~ 45° to the E-field, as shown in (a), it can appear that the resonance is wider than it actually is, as shown in (c). This will give an erroneously low estimate for the loaded Q-factor.

(e) *Measurement Problems caused by Low Degrees of Anisotropy* Paragraph (d) explained the procedure that can be used for measuring *significantly* anisotropic specimens (e.g. quartz crystal, sapphire, see Section 5.3.11). Measurement problems arise, however, if the degree of anisotropy is so low that resonances $R_\perp$ and $R_\parallel$ overlap, as in Figure 52 (c). Thus, if the difference in frequency, Δf_a, between $f_\perp$ and $f_\parallel$ is smaller than the resonance width Δf_r, where $Q = f_r/\Delta f_r$, (see Section 6.4.1) some difficulty may be experienced because the two orthogonal resonances overlap. But if specimen *loss* is to be measured this can cause a problem, for the following reason. If the specimen is sitting in the resonator in such a way that its optic-axis is at 45° to the E-field then both $R_\perp$ and $R_\parallel$ will be excited. If they overlap, it will appear that the resonance width is much broader than it actually is for $R_\perp$ or $R_\parallel$ taken in isolation, as shown in Figure 52 (c). An erroneously low value of Q-factor will therefore be measured from which spuriously high values of tan δ will be computed. Therefore one must check for anisotropy in *all* specimens measured in open-resonators by rotating them about their axis, typically by about 20° - 30° at a time. If the measured value of ε' appears to cycle as one rotates it about its axis, or if tan δ appears to cycle likewise, then it is a sign that the specimen exhibits a small degree of anisotropy. Where such anisotropy is detected the specimen must be rotated so that the E-field is either parallel to or perpendicular to the detected optic-axis, before it is measured in the normal way. It might be thought that this is a rare occurrence, but it is not. A number of materials exhibit low degrees of anisotropy, notably composites with a woven material structure in them and polymers with frozen-in stress – see Section 5.3.11. So if loss is to be measured, open-resonator measurements must always include a test for anisotropy, and this can unfortunately make them labour-intensive to use. Such tests are unnecessary when using cavities with circularly polarised fields, so that the additional time taken to carry out this test can certainly be accorded a disadvantage of the open-resonator method. However, if a class of material is known from prior experience *not* to exhibit this phenomenon, the check may be omitted and, as pointed out in Section 7.2, for *consistency-checking* on such materials, in the production environment, say, the open-resonator can potentially be used very quickly and cost-effectively.

(f) *Mode-coincidence and interference.* Though their modes are sparser than typical closed cavities of similar size, higher-order resonant TEM_{lmn} modes in open-resonators can overlap with the fundamental TEM_{00n}-mode that is used for measurements. Mode-coincidences can easily give rise to errors of measurement, most notably in loss measurements, generally producing spuriously high values of the apparent loss. It is recommended therefore that at least 2, but ideally 3 or more different axial resonances be investigated. The axial mode number *n* gives the number of antinodes present along the axis of the resonator, so modes corresponding to a number of different values of *n* should be used. Note that because of the *anomalous dispersion* in Gaussian Beam modes [214, 216, 215], if there happens to be a mode-coincidence with axial mode *n*, say, there is unlikely to be a coincidence (at least from the same higher order mode) for axial mode ($n + 1$). Inspection of measured values of tan δ using a number of values of *n* generally reveals which modes are giving spuriously high values and they can be eliminated. The need to make such checks again adds to the time required for carrying out open-resonator measurements (see (e)) though modal interference is a problem that is shared with other resonator techniques too. Again, as with (e) one can omit these additional checks when the *consistency* of properties of a number of similar specimens (i.e. same shape, same material) is being measured, provided a survey of modes was carried out initially to ensure that a suitable mode, free of interference, can be used.

(g) *Specimens made from Composite Materials.* As pointed out in Section 5.3.7 and in Section 2.3 paragraph 3 (iii) in the discussion on loss processes in composite materials, grains of high permittivity dielectric in such materials can readily scatter radiation out of the specimen if they are close to half a wavelength in scale. In open-resonators (as opposed to closed cavities) this effect scatters the radiation completely out of the resonator and effectively creates a significant source of loss, which is easily noticeable. As the frequency is increased this effect can have a very sudden onset. One should be aware of this when measuring composite materials in open-resonators. If such scattering *is* occurring, it can create significant errors and the open-resonator measurements should normally be abandoned – the detection of the presence of the effect itself probably demonstrates that the material has reached its useful upper frequency limit in any case! One can work out from the structure-lengths of the components in the composite, if they are know, and from their permittivities whether this effect is likely to happen. Note that in high permittivity ceramics ($\varepsilon' > 100$) the wavelength is significantly reduced below that in air. At millimetre-wave frequencies the wavelength in some ceramic components of composite materials can be smaller than a millimetre and so this effect could well be present if the structure-length of ceramic component lies in the range 0.1 – 1 mm.

(h) *Corrections to the Basic Theory*. Jones and Cook & Jones pointed out two types of corrections to the basic theory of open-resonator dielectric measurement, [218], that should be taken into account in the most accurate measurements.

Recent Developments. The microwave open-resonator technique came into its own for low-loss millimetre-wave measurements in the 1970s and it has been continuously developed since then. Work at NPL in the early 1990s addressed the measurement of medium loss specimens and investigated new techniques for measuring specimen loss [217]. More recently Heidinger and his associates in Germany [117] have implemented three major advances:

(i) Design and use of *variable-temperature* open-resonators – including cryogenic measurements,

(ii) At 140 GHz where the beam waist is only a few millimetres in diameter, they have placed large cross-section specimens (specifically CVD-diamond windows for particle accelerators approx. 200 mm in diameter) in mounts which allow them to be displaced *sideways* in the plane of the specimen, i.e. in the *x* and *y* directions, if the axis of the beam is in the *z* direction. This allows the loss of the specimens to be mapped across the face of specimen as the GB waist-diameter at this frequency is typically less than 10 mm.

(iii) By very careful attention to mirror machining and cleanliness and by careful machining of very small and thin coupling holes (see Section 6.1.9 and Figure 22) they have significantly reduced the contribution of coupling holes as a major source of loss in their open-resonators. Q-factors 2 or 3 times higher than those typically obtained before have been achieved, thereby increasing the sensitivity for low-loss measurement.

► ***Summary: Open-Resonators***

Advantages of Open Resonators

One of the most accurate methods available for ε' and tan δ of low-loss specimens, especially convenient for millimetre-wave frequencies.

Ability to measure specimen anisotropy.

At sufficiently high frequencies, the ability to measure specimen inhomogeneity across the face of a specimen.

Easy and convenient mounting of specimens.

Specimens do not have to have a specific diameter or cross-sectional shape, as long as they are large enough to encompass the Gaussian Beam.

Disadvantages of Open-Resonators

The method can be time-consuming to use because tests for anisotropy and mode-coincidences must be performed.

Large cross-section specimens have to be used at microwave frequencies but this is not normally a problem at millimetre-wave frequencies.

Open-resonators are less-easily temperature controlled than closed cavities.

Open Resonators: Typical Sources of Dominant Uncertainties:

Specimen warping, especially if specimens are not an integral number of half-wavelengths thick.

Mode-coincidences and interference.

For ε':

Ability to measure specimen thickness if the specimen is not an integral number of half-wavelengths thick.

For tan δ:

Uncertainties in the Q-factor.

Undetected low-level anisotropy.

► ***Do's and Don'ts for Open-Resonator Measurements***

Do carry out checks for specimen anisotropy and mode-coincidences to avoid the possibility of significant errors in loss measurement.

Don't use specimens that are too small in diameter to encompass the Gaussian Beam in the resonator.

Don't use specimens that are warped or with rough faces or without parallel sides if this can be avoided, as such features will significantly increase the uncertainties of measurement.

7.1.18 Dispersive Fourier Transform Spectroscopy (DFTS).

Dispersive Fourier Transform Spectroscopy (DFTS) [219, 18, 220, 221, 2] can be used to measure the complex refractive index (and hence the complex permittivity) of materials from approximately 100 GHz all the way up to infrared frequencies, though wide-aperture DFTS systems have also been constructed to work down to 30 GHz. DFTS is a free-field *quasi-optical* technique, see Section 7.1.14. The principle of operation is shown in Figure 53. The DFTS instrumentation essentially comprises a polarising Michelson interferometer, which allows a dielectric specimen to be placed in one arm. Interferograms are obtained by translating one of the mirrors with a computer-controlled motor drive, while recording the optical power at the output port, using a broad-band detector. The other mirror may be mounted on a transducer and vibrated sinusoidally by a fraction of a wavelength. The detected signal then contains a *wanted* modulated component, which can be detected on a Phase-Sensitive Detector (PSD), otherwise known as a lock-in amplifier, and so can be separated from the *unwanted* un-modulated noise. The interferometer is evacuated before use to avoid absorption lines from water molecules in moist air. Traditionally, mercury vapour lamps were used as sources as they produce a broad-band noise spectrum. These sources provide very low output power in the millimetre- and sub-millimetre-wave bands, so highly sensitive detectors are required. To improve the signal-to-noise ratio, PSDs and cryogenic detectors are often used. Suitable detectors include Golay cells and cryogenic (4K) indium antimonide hot-electron bolometers.

At 100 GHz 50-mm-diameter specimens are necessary, though smaller diameters can be used at higher frequencies. Low-loss specimens are usually measured in transmission by a double-pass method, while high-loss specimens can be measured in reflection. As this is a travelling-wave (double-pass) technique, sensitivity for low loss is limited but sensitivity does depend upon specimen sizes, frequencies, averaging of signals, etc.

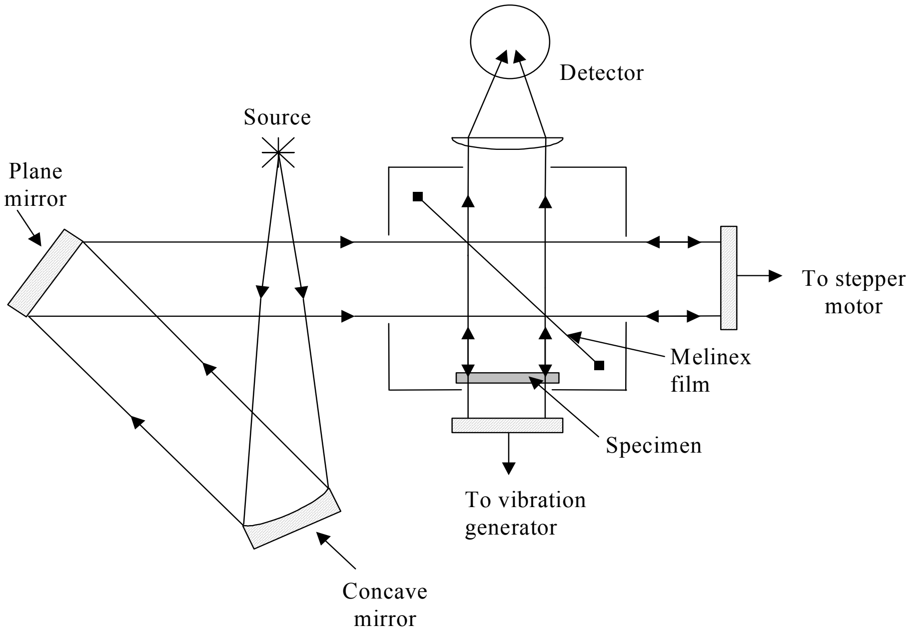


Figure 53. A typical Michelson Interferometer used for Dispersive Fourier Transform Spectroscopy (DFTS) at sub-millimetre-wave frequencies, typically above 100 GHz. The Melinex film is used as a partial reflector.

► ***Summary: Dispersive Fourier Transform Spectrometry (DFTS)***

Advantages of DFTS. Unlike microwave techniques this is a quasi-optical broad-band method that can cover many decades of frequency – from 100 GHz upwards – in one measurement session. The method can be very accurate for ε', with uncertainties better than ± 0.5% if $\varepsilon' < 5$.

Disadvantage of DFTS. This is quite a difficult and expensive method to use, requiring technical expertise to perform measurements properly. Resolutions for loss angle may be as high as 100 μrad, so very low losses cannot be measured accurately.

DFTS: Typical Sources of Dominant Uncertainties:

For ε':
- The uncertainty in specimen thickness.
- Diffraction from the edge of specimens and lenses.
- Aliasing effects in Fourier Transform computations.

For tan δ:
- As for ε' plus:
  - Low sensitivity of detection.

7.1.19 Time Domain Spectroscopy (TDS) and Other Time Domain (TD) Methods.

There are two main issues to be considered here – one is the use of TDS itself in dielectric measurements, the second is the use of *TD-based methods* in *Frequency Domain* (FD) measurement techniques. In the former case, TDS usually takes the form of *Time Domain Reflectometry* (TDR) when used for dielectric metrology.

Time Domain Reflectometry. TDR using pulse or step generators and sampling methods for detection, see Figure 54, was widely adopted in the second half of the 20th Century [222] for dielectric measurements and is still widely used [16, 223]. It is convenient to use for the purposes of scientific study, being a cost-effective broad-band method which requires only small specimens, making temperature-control relatively easy. Solid specimens typically fit into a 7-mm coaxial line. The main limitation of the method is its absolute accuracy, which is not generally as good as that of reflectometry based on a calibrated automatic network analyser (ANA). The main reason for the continued popularity of TD methods using pulse/step generators is that they are cheaper than fully-automated ANA measurements, and they are often more convenient to use. Note that Feldman *et al.*

[224] have published specific details of a method for correcting for the Electrode Polarisation Effect (see Section 5.3.3) in time domain spectroscopy on liquids.

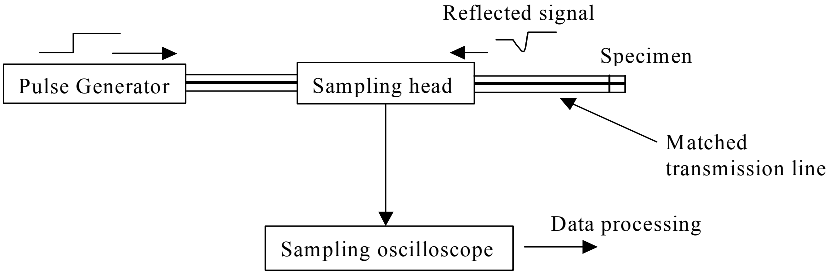


Figure 54. Block diagram of a typical Time Domain Reflectometer.

Time Domain Gating. TD methods once had at least one other advantage over FD methods – namely their ability to *separate out* in the received time signature the wanted reflections, i.e. those from the specimen, from those caused by spurious mismatches (e.g. from connectors) elsewhere in the system, see Figure 55. The unwanted reflections return to the sampling head at different times to those from the specimen and so can be omitted in subsequent analysis of the measurement results. This technique is referred to as '*gating'*. This one-time advantage of the TD method, has, however, in recent decades been appropriated by ANAs since the advent of *Synthesised Time Domain* techniques [106] and this technique is now very widely used. It is a very useful metrological technique – particularly in free-field measurements, Section 7.1.14. Care has to be taken, though, when selecting the time and period of the gating window, to ensure that wanted signals are in included in it, and not curtailed at their edges, while as many of the unwanted signals are excluded as is feasible.

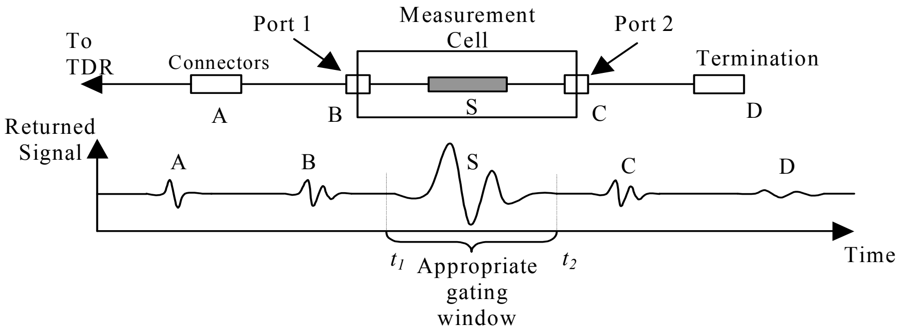


Figure 55. The Principle of *Gating*. Spurious reflections from connectors A, B, C and the termination D can be 'gated-out' by selecting only the wanted data, S, in the chosen time-window from t_1 to t_2 for subsequent analysis.

Synthesised Time Domain Techniques. [106] As explained in Section 2.1 the *Time* and *Frequency Domains* are simply two different, but mathematically equivalent, ways of looking at time-varying signals, including responses from dielectric specimens. In order to achieve a *broad-band* measurement, we can either sweep the sinusoidal frequency source, as is done in an ANA, or we can propagate a sharp pulse or step wave through our system, as is done in TDS: its Fourier Transform will contain all of the broad-band frequencies we need. In *Synthesised TDR* the data obtained from broad-band swept measurements on an ANA are Fourier Transformed to give the reflection coefficient from the measured system as a function of time. The *main reason* for doing this is to allow spurious reflections from the system to be gated out, as described above and in Figure 55. These days, many ANAs are supplied with software to perform precisely this synthesised gating function. This process of converting 'contaminated' data from the FD to the TD, using the TD to 'de-contaminate' it, then converting it back to the FD, is also sometimes referred to as '*de-embedding*' because the response data that we want from our specimen is *de-embedded* from the unwanted responses, which will otherwise cause measurement errors.

► ***Summary: TD Measurements based on Pulse/Step Generators.***

Advantages of TD Measurements based on Pulse/Step Generators. This is a broad-band and cost-effective method, well-suited for dielectric spectrometry in scientific studies. Gating techniques originating in TDS are invaluable, and they are commonly incorporated into FD ANA-based measurements.

Disadvantage of TD Measurements based on Pulse/Step Generators. Generally less accurate than ANA-based transmission-line techniques because signal-to-noise ratios are lower.

TD Measurements based on Pulse/Step Generators: Typical Sources of Dominant Uncertainties:

For ε' and tan δ:
  Signal-to-noise ratio.
  Jitter in the detection system.
  Otherwise similar to FD transmission-line techniques, see Sections 7.1.10 and 7.1.11.

7.2 Selecting the Best Measurement Techniques

Sections 7.1.1 – 7.1.19 above describe a wide range of dielectric measurement techniques and review the benefits and problems of each of them. This short Section attempts an overview of these techniques with the aim of helping the reader to make the best selection between them. The following paragraphs tackle points that may well help in the decision-making process. The various parts of Figure 56 should also help: they summarise most of the techniques discussed, showing the ranges of frequency and loss for which they are best suited. They are arranged mainly with clarity in mind, but measurements of similar type are kept together, where possible.

In writing this overview we should bear in mind that many readers will not have the luxury to choose freely between the techniques discussed above. It is very costly to set up new measurement systems and so the concern will more often be: '*How can I achieve my measurement aims with the equipment or techniques that I already have?*' This point and a number of related issues are also tackled below.

A Check List: In selecting an optimum measurement technique one should take account at least of the following parameters and issues:

The frequency range of interest.
The dielectric loss (high, medium or low) and expected permittivity range.
The type of material, e.g. hard, malleable or soft solids, volatile or viscous liquids.
Specimen machining imperfections and tolerances and their influence on uncertainty.
Specimen shape and size and their influence on uncertainty.
Specimen anisotropy and homogeneity and their influence on uncertainty.
Inhomogeneity and the presence of surface layers on specimens.
The possibility that the specimen may be made from a magnetic material.
The required uncertainties. What level of uncertainty can be achieved by available methods?
The specimen composition (e.g. does the specimen have a laminated structure).
The availability of suitable methods for machining and grinding specimens.
The presence of surface inclusions and pores, surface conditions in solid specimens.
Toxicity, contamination and evaporation of liquid specimens.
The cost of machining specimens and performing measurements: cost-effectiveness.
The time taken to perform the measurements – labour-intensiveness and the labour cost of measurements.

All of these issues have been discussed above in Sections 5, 6 and 7.1 above, to which reference should be made for detailed guidance. The last two issues of cost-effectiveness and labour-intensiveness are always important but they are especially so in the industrial production environment where two other factors come into play:

Which techniques are best suited to checking consistency, rather than providing absolute accuracy.
Which techniques can be de-skilled? How *proof* are the various techniques against inexperience?

Such issues relating to measurements in the production environment are discussed separately in Section 7.2.1.

Preliminary Measurements: If I do not know initially what the dielectric properties of my material are, how can I quickly and cheaply get an approximate ('ball-park') figure? If we have no idea what properties to expect from a material we need to perform a cost-effective preliminary measurement before we can choose the best technique for accurate measurements. In this case we should avoid methods that are time-consuming or that require expensive machining. An important point to remember is that preliminary measurements do not have to be performed at the ultimate frequency of interest: the permittivity of low-loss materials varies slowly with frequency (see Equation 2.21, Section 2.5 and Section 5.3.2). Also, if we find that our material has high loss at one frequency, we know thereby that it is likely to be quite lossy across the whole RF & MW band. If a high value of ε' is obtained in a preliminary measurement at a low frequency, we at least have an upper bound for ε' at higher frequencies (see Sections 2.3 and 2.5).

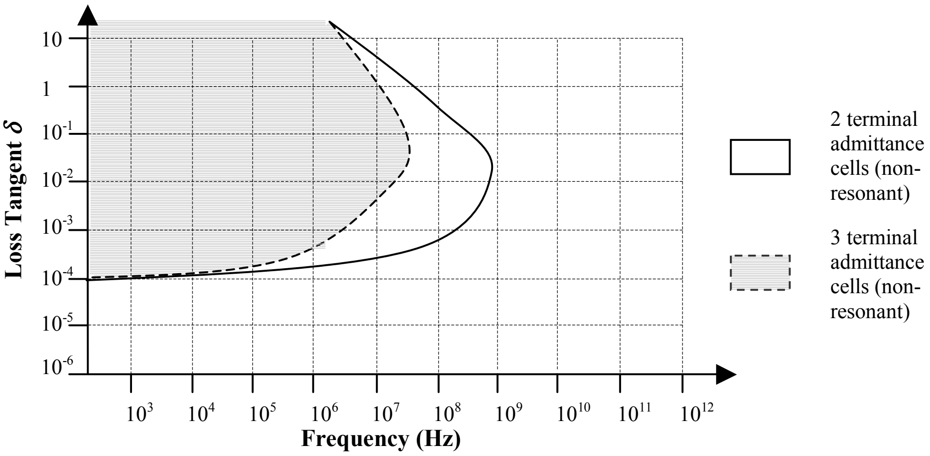


Figure 56 (a). The approximate frequency coverage and coverage for tan δ of non-resonant two-terminal and three-terminal measurement cells, see Sections 7.1.3 and 7.1.1, respectively.

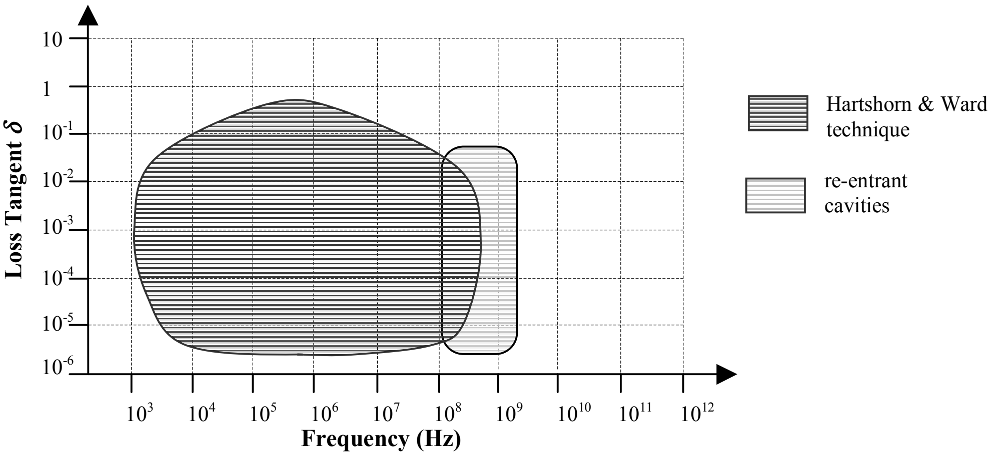


Figure 56 (b). The approximate frequency coverage and coverage for tan δ of resonant two-terminal (Hartshorn and Ward) and re-entrant cavities, see Sections 7.1.4 and 7.1.5, respectively.

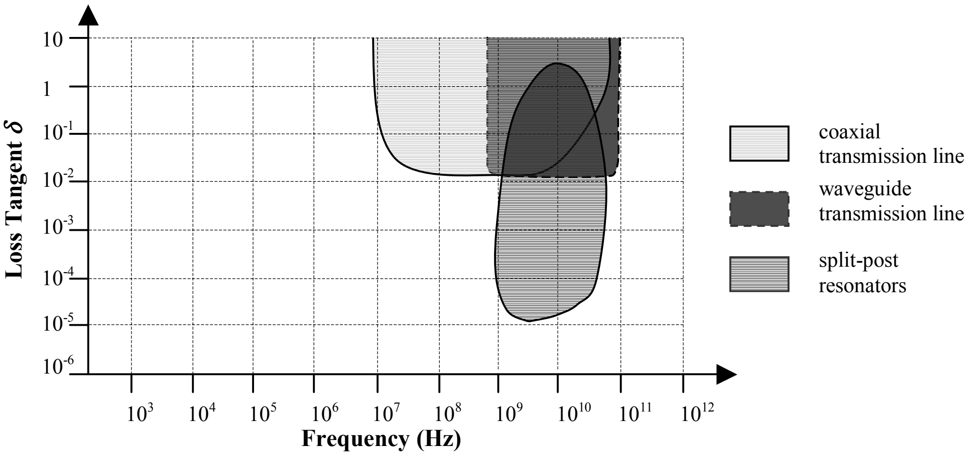


Figure 56 (c). The approximate frequency coverage and coverage for tan δ of the coaxial transmission line, waveguide transmission line and split-post resonator methods, see Sections 7.1.10, 7.1.11 and 7.1.8, respectively.

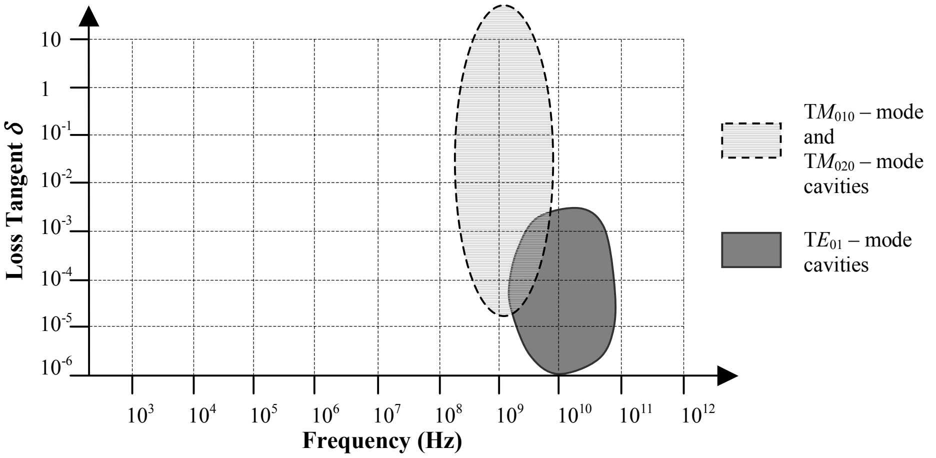


Figure 56 (d). The approximate frequency coverage and coverage for tan *δ* of the TM_{010}-mode, TM_{020}-mode and TE_{01}-mode cavity methods, see Sections 7.1.6, 7.1.7, respectively.

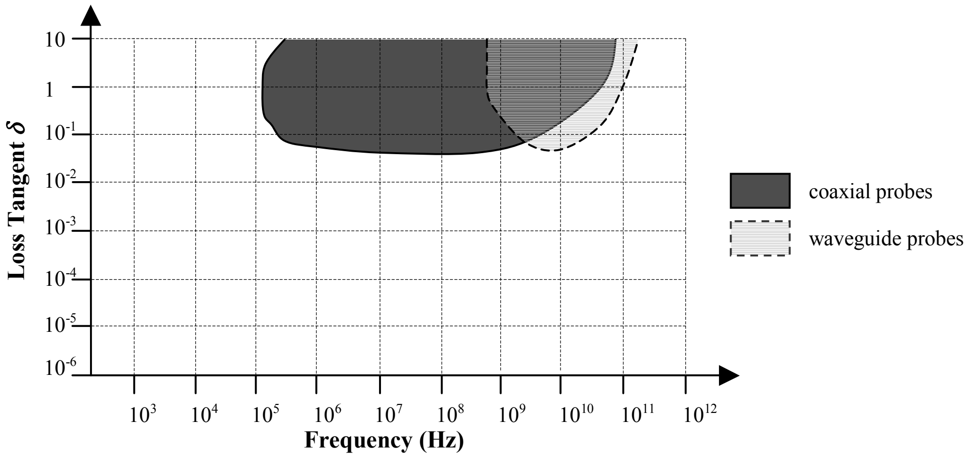


Figure 56 (e). The approximate frequency coverage and coverage for tan *δ* of the coaxial probe and waveguide probe methods, see Section 7.1.12.

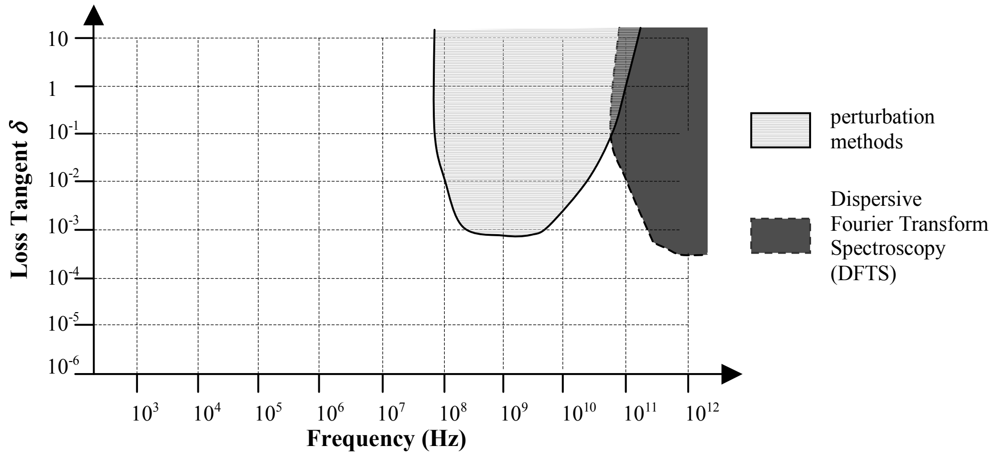


Figure 56 (f). The approximate frequency coverage and coverage for tan *δ* of the perturbation and DFTS methods, see Sections 7.1.16, and 7.1.18, respectively.

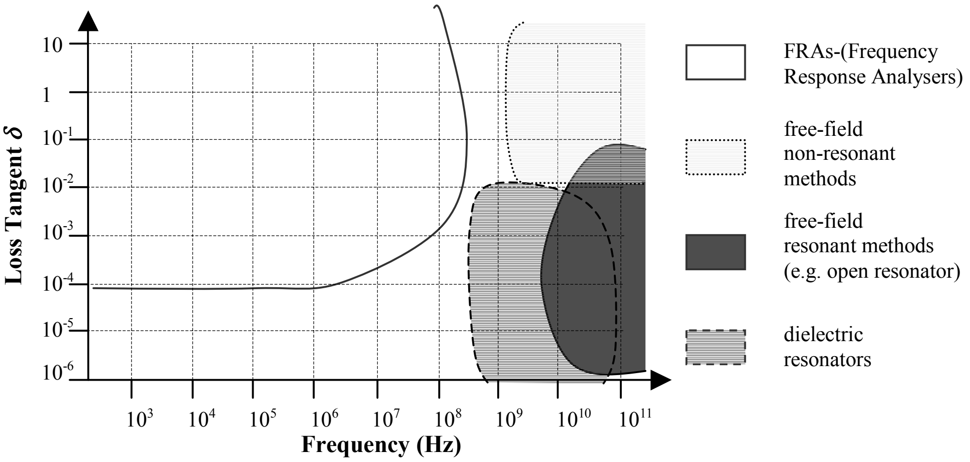


Figure 56 (g). The approximate frequency coverage and coverage for tan δ of admittance cell measurements in FRAs, free-field non-resonant methods, open resonators and dielectric resonators, see Sections 6.1.3 (& 7.1.3), 7.1.14, 7.1.17 and 7.1.15 respectively.

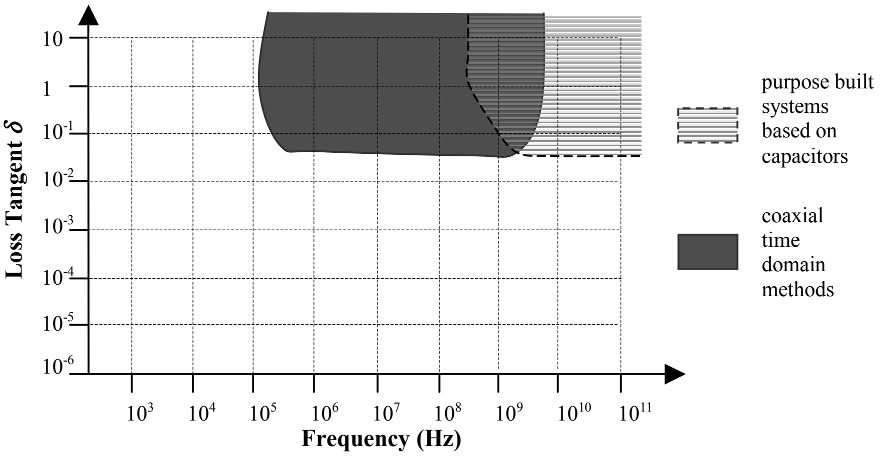


Figure 56 (h). The approximate frequency coverage and coverage for tan δ for time domain measurements – see Section 7.1.19.

If the specimen is available in laminar form, it can be measured quickly in an admittance cell (Sections 7.1.1, 7.1.3) or in resonance cells such as the Split-Post Dielectric Resonator (SPDR) (Section 7.1.8). 'Puck'-shaped specimens can be measured as dielectric resonators (Section 7.1.15). Inability to find a resonance in such measurements probably means that the specimen has high loss. If the specimen has one flat surface, or if it is a malleable solid or a liquid, a measurement with a coaxial probe (Section 7.1.12) may be able to provide the information that we need. Even in the case of *hard* specimens, for which the coaxial probe is not ideal because of the serious effect of air gaps between specimen and probe, such a measurement will give us a *lower* limit for ε' and should tell us if the material is of low or high loss. At higher frequencies, if a specimen in suitable laminar form is available, free-space (Section 7.1.14) and open-resonator methods (Section 7.1.17) can be employed and are relatively quick for checks because specimens do not have to be sealed into cells. Many of the checks recommended here take no more than half an hour to perform if the relevant equipment is available. Of course, none of these methods can be used if specimens of a suitable size and shape are not available. It is therefore unfortunately sometimes necessary to make up test specimens even for preliminary measurements, though machining tolerances can often be relaxed to save cost.

Is there always a single optimum technique? No. In many cases one cannot be sure, before embarking on measurements, which method will be the best. Ideally, more than one suitable technique should be used to check on systematic uncertainties (Section 4.4.3) and to discover for the longer term which technique should be preferred for a given class of material. In some cases one method may prove to be better for measuring loss and another for permittivity.

How can measurement techniques be modified to obtain results with the equipment that one actually has? As intimated above, we often find ourselves in a position where we have to use existing equipment and measurement methods to perform dielectric measurements, and (perhaps for cost reasons) the luxury of using an 'optimum' technique is not open to us. In these circumstances, the first questions to ask, as ever, are *'What are the best levels of uncertainty for ε' and tan δ that I can achieve for the material to be tested with the methods that I have available?'* and *'What levels of uncertainty do I need?'*. If the best uncertainties or resolutions that can be achieved do not match up to the need, one may have to admit, after all, that the measurements cannot be performed with existing equipment. However, *do* investigate very carefully what is actually 'needed'. Don't be afraid to ask your customer about the uncertainties *really* needed: sometimes little thought is put into measurement uncertainty specifications and there is often a tendency to ask for the best when one does not really need it. If the uncertainty specification can be relaxed, it may be that measurements can be performed on existing equipment after all.

If measurements *can* go ahead with existing equipment, we are still faced with an optimisation problem. It is at this point that uncertainty budget analysis of the sort discussed in Section 4.3 become very important. We need to optimise the *sensitivity coefficients* (Sections 4.3.2. and 4.3.3) in the measurement that relate the quantities we want (ε' and ε'') to the actual measured parameters (e.g. capacitances, conductances, frequencies, electrode displacements). There is a whole range of parameters that we can change to improve uncertainties here. They include specimen size, shape and finish, electrode separations, and choice of the best cell to use if we have a number of cells available. Signal-to-noise ratios in detectors can be improved by use of amplifiers or by signal averaging. Equipment stability can be improved by better temperature control. Very often, as is the case with admittance cells, the same equipment can be used with a number of techniques, so one can choose the best technique for the particular materials one has, e.g. the *equivalent-thickness* method may be better than the *capacitance method* for low permittivity, low-loss specimens (see Section 7.1.1). Don't regard yourself as being restricted to traditional techniques that you have always used. Do consult references given in this Guide to see if they can reveal any technique variations that could be useful to you. As ever, the steps you can take here will depend upon the time and funding available, but it will usually be the case that a little thought will help one to improve one's measurement uncertainties.

Destructive and Non-Destructive Methods. What *counts* as a destructive method depends upon the form in which our dielectric material is supplied.

Liquids can be readily sampled, but as pointed out in Section 5.3.3, once a specimen has passed through a measurement cell it may be contaminated in various ways and so it may not be advisable to re-use the specimen liquid for later applications. Whether this is so or not will depend on the ultimate end-use of the liquid and upon its desired loss: low-loss liquids are more prone to changes in tan δ through contamination.

Some solid materials, e.g. substrates, are supplied in laminar form in sizes that are ideal for measurement in admittance cells (Sections 7.1.1 - 4) and in split-post dielectric resonators (SPDRs) (Section 7.1.8). Dielectric rods, tubes and liquids can be measured non-destructively in TM_{010} and TM_{020}–mode cavities (Section 7.1.6). Even large dielectric sheets can readily be measured by free-field techniques (Section 7.1.14). Open-resonators (Section 7.1.17) have been designed with a cantilever structure to allow corners of large sheets to be measured. On the other hand, if our material is supplied with a size, shape, thickness and surface finish which is far from optimum for (or even un-measurable with) these techniques, then machining of suitable specimens becomes inevitable.

It is therefore difficult to classify any of the techniques discussed here as being definitively destructive or non-destructive. The best that one can say is that many of them have the *potential* for non-destructive testing (NDT) and measurement. Perhaps the best case can be made out for coaxial and waveguide probes (Section 7.1.12), which require only a flat surface on the specimen for measurement, though, even here, specimen size, flatness and hardness come into play.

General Advice: Look again at the *Do's and Don'ts* list in Section 5.1 and remember that in trying to choose between measurement options it is always a good idea to seek out experienced advice.

7.2.1 Measurements in the Production Environment

As pointed out above, the choice of an optimum technique for production control will generally be governed by different concerns than those which are of interest in laboratory measurements. They include:

Suitability for checking consistency, rather than providing absolute accuracy.
Levels of skill needed.
Long-term reliability of equipment.
Ease of measurement.
Ease of automation.
User-friendliness of computer readouts.
Costs and time taken for measurement.

However, a surprisingly large number of techniques discussed in this Guide can potentially be modified for production checking. Many measurements can be de-skilled if the major requirement is simply to check consistency from one specimen to another. Perhaps the methods that are least suitable are those in which adjustments have to be made, e.g. micrometer adjustments. Fixed-geometry cells are generally both easier and quicker to use. Cells which have to be disassembled for insertion and removal of specimens are not best suited for these applications: this can be time consuming and the repeatability of electrical contacts is often a source of uncertainty. However, sometimes the use of such cells is unavoidable. If so, steps to improve their user-friendliness can be taken. It may be possible to replace doors on cells with apertures, for example, or instead of using screws to assemble waveguide transmission cells (Section 7.1.11), clamps can be used, with fixed dowel-pins to ensure correct alignment.

In some cases physical specimen guides can be inserted into measurement cells to ensure correct positioning of specimens. Vacuum clamping of specimens to metal surfaces can be used to ensure good contact (where necessary) and to improve measurement reproducibility.

For checking of consistency, bear in mind that some techniques, e.g. two-terminal admittance cell measurements (Sections 7.1.3, 7.1.4), have far better *resolution* than *absolute accuracy* and this is very often all that is required, so that simpler and cheaper techniques can be used than those necessary in the calibration laboratory.

The general requirements for techniques that can perform well in production control and for consistency checking are rather similar to those discussed above in relation to preliminary check measurements. Fixed geometry cells with simple measurement readouts save time in both situations, for example. If specimens are available in laminar form, admittance cells (Sections 7.1.1, 7.1.3) are easily used, as are split-post dielectric resonator (SPDR) (Section 7.1.8). Dielectric resonator pucks (Section 7.1.15) can be measured quickly if the main requirement is consistency because the detection equipment (e.g. an ANA) can be permanently set to an optimum setting. As noted in Section 7.1.6, dielectric rods, tubes and liquids can be measured continuously - in real-time if necessary - and non-destructively in TM_{010} and TM_{020}–mode cavities [212]. Coaxial probes (Section 7.1.12) are easily used with lossy liquids and lossy malleable solids, but their calibration should be checked regularly by measuring a reference specimen. At higher frequencies laminar specimens can readily be checked by free-space methods (Section 7.1.14). Even open-resonators (Section 7.1.17) can be employed for consistency checking if the resonator length is set permanently to the optimum position, e.g. (by clamping the micrometer) - see the discussion on non-destructive measurements above).

We have also considered above in Section 7.1.9 one method that is particularly appropriate for testing of substrates and printed circuits in a production environment and in Section 5.3.8 we have looked at techniques that are used for dielectric measurements in the food processing industry. Moisture content measurement is explicitly addressed in [22], notably in Chapter 15 by F Völgyi, which is concerned with automatic process control, and in Chapter 11, where R J King *et al.* discuss the use of open reflection resonator sensors in industrial applications.

In production-testing it is best to use specially-written software with a user-friendly display to allow go/no-go checking of specimens. This should be used with fixed procedures and check lists – see Section 4.4.4.

It is *very important* when unskilled staff are performing measurements for them to undertake periodic checks with reference specimens (see Sections 6.2 and 4.4.2) to ensure equipment is working properly.

8. Presentation of the Results of Measurements.

Having performed suitable measurements, having checked them, having achieved confidence in the results and having derived at least approximate uncertainties for the results, we usually need to decide how we should present our results. This issue arises particularly when we are presenting them for publication. Inspection of the scientific literature demonstrates that both poor and good practices are followed here and one should realise that traditional methods of presentation are not always the best. So here are some thoughts on the presentation of results - the final stage in the measurement process.

The basic principle to be followed is to *present results in a way which will be maximally useful to their intended recipients*. Where results are being *published openly*, we cannot always sure who will be making use of them in future, so the general rule then is to *publish results in a way that will make them maximally useful to everybody*! The publishers of scientific journals sometimes do not make it easy to follow this dictum because of their preference for diagrams over tables of results – and *small* diagrams at that. It is true that 'a picture is worth a thousand words' and that graphical presentation of results generally promotes greater *scientific understanding* of experimental findings than any number of results tables, but if the *aim* of the measurement is to obtain *quantitative data* – as is often the case in electrical engineering - we need tables of results as well, because it is difficult to read data points accurately off small diagrams. If copious tabulated data has been assembled, the best compromise may be to preserve it in a separate report (perhaps 'in-house') or on an Internet page and to inform the readers of the published paper by means of references how they can access this data.

A special application of this principle is to the presentation of dielectric relaxation data covering a large range of frequencies. This applies, for example, to dielectric reference materials (Section 4.4.2, e.g [37]). Suppose that we in 'Lab A' have measured the complex permittivity, ε^*, of a material from 1 – 5 GHz but we know that another laboratory, 'Lab B', will be presenting results from 4 – 10 GHz in a year or two's time. At that point it may prove useful to combine the two sets of data to give the best representative data between 1 and 10 GHz. Now, it is usual practice to publish data on reference materials in a parameterised form, typically by using one of the relaxation models expressed in Equations 2.16 – 2.20: the Debye, Double (or multiple) Debye, Cole-Cole, Cole-Davidson or Havriliak-Negami, or else the Dissado-Hill [41] relaxation model. This has the advantage that only a few parameters like ε_s, f_r and ε_∞ (Equation 2.16) need be published at any one temperature to compute ε^* at any frequency. Now, indeed, this practice *does* follow the dictum of publishing results of maximum usefulness to the reader: an experimenter would find it more convenient to use an equation like 2.16 to compute the properties of a reference material than to have to resort to looking up results in a table. However, if both 'Lab A' and 'Lab B' present *only* their parameterised data to the world in their published papers, over the frequency ranges 1 – 5 GHz and 4 – 10 GHz respectively, there is *no formally mathematically correct method* for combining their data to give the overall best parameterisation between 1 and 10 GHz. As pointed out by Kaatze and Uhlendorf in their papers on the properties of deionised water [225, 226], this can only be done if the *original tabulated data* and *estimated uncertainties* are available from both laboratories. In programmes of measurement such as this, the general rule therefore is always to preserve the raw data, tabulated together with its measurement uncertainties. If the data may be useful to others in future, do make sure the scientific community knows how to access this data.

Given that graphical presentation of results is generally a good thing, we should also think carefully about the details of the graph we use. An example is the graphical presentation of the results of dielectric relaxation measurements. A Cole-Cole plot, Figure 8, may look interesting and it can be educational because it shows us just how ε' and ε'' are inter-related, but it does not show all of the data that we have because frequency is omitted in such a presentation (or else it has to be explicitly printed on the graph). The physics of dielectric relaxations is often better presented by plotting ε' and ε'' against log(f) or indeed by plotting log(ε') and log(ε'') against log(f) as in Figure 7 and [25, 28] (*passim*) where the important parameters of relaxation models can be discerned from the gradients of the curves.

These examples show how a little thought and a little extra-effort taken at the time results are recorded and presented can make them far more useful for others in the longer term – and this is certainly a good practice.

SECTION C: SUPPORTING MATERIAL

9. Worked Examples

9.1 The Dielectric Relaxation of Water – An Equivalent Circuit

Water exhibits a dielectric relaxation in the microwave region of the spectrum which is a close fit to that of a Debye Relaxation – see Section 2.3, Equation 2.16. It is instructive to draw up an equivalent circuit for water which behaves in the same way as the actual dielectric material. The Debye Relaxation parameters [225] at 25 ºC are

Static permittivity	$\varepsilon_s = 78.36$
High frequency permittivity limit	$\varepsilon_\infty = 5.2$
Relaxation time	$\tau_r = 8.27$ ps
... so relaxation frequency	$f_r = 19.24$ GHz, where $f_r = 1/2\pi\tau_r$.

Assume that we have a small capacitance cell with fixed electrodes and that it has a capacitance of 1 pF when it is filled with air dielectric. When we fill it with de-ionised water, however, the admittance of the cell as a function of frequency will be well represented over a large band of frequencies by the equivalent circuit in Figure 57. This circuit behaves in accordance with Equation 2.16 when the above numerical values for the above parameters are substituted into it. The circuit is thus an analogue of the Debye relaxation of water. Note that in units of picofarads $C_1 \equiv (\varepsilon_s - \varepsilon_\infty) = 73.16$, $C_2 \equiv \varepsilon_\infty = 5.2$, while $f_r = 1/2\pi RC_1$ Hz. The resulting dielectric response is shown in Figures 7 and 8, page 15.

In practice, *real* physical water diverges from this pure Debye behaviour at both low and high frequencies. Water is *always* partly ionised and this gives rise to electronic and ionic *conductivity* within it. At frequencies below 1 MHz this conductivity can be clearly seen in dielectric measurements as an apparent rise in the *phenomenological* value of ε'' (see Section 2.4), which eventually rises inversely with frequency as the frequency falls to 0 Hz. The apparent additional component of ε'' due to conductivity σ is $\sigma/2\pi f\varepsilon_0$, as explained in Section 2.4 and this *adds on* to the loss caused by the dielectric relaxation. Likewise at very high frequencies other loss processes take over from the Debye Relaxation. However, between approximately 1 MHz and 30 GHz, Figure 57 provides quite a good equivalent circuit for the electromagnetic properties of deionised water!

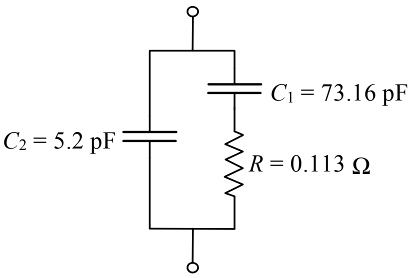


Figure 57. An Equivalent Circuit of the Debye Relaxation of Deionised Water. The circuit behaves electrically in the same way as a capacitance cell full of water. The cell would have a capacitance 1 pF if it contained only air dielectric instead.

Note that the Debye Relaxation which is used here is only an approximation (nevertheless one which is adequate for most metrological purposes). For more general information on the detailed physics of the relaxation and loss processes in water, please see the references in [70].

9.2 Checking the Mutual Consistency of Measured Values of ε' and ε''.

Two examples are given here which demonstrate the use of Lynch's formula, Equation 2.21 [45], as described in Section 2.5.

The first example is taken from a set of measurements in an admittance cell (Section 7.1.1). Measurements were made on a medium-loss polymer specimen with the following results:

Frequency	**Measured Real Permittivity** ε'	**Measured Loss Tangent** $\tan\delta$
10 kHz	2.85 ± 0.03	0.076 ± 0.08
300 kHz	2.48 ± 0.03	0.054 ± 0.05
10 MHz	2.25 ± 0.03	0.031 ± 0.03

Between 10 kHz and 10 MHz, 300 kHz is the 'centre' frequency on a logarithmic scale. Using Equation 2.21 with $m = 1.5, f_1 = 10$ kHz, $f_2 = 10$ MHz, we have $\log_{10}(f_2/f_1) = 3$. The appropriate value of ε' at 300 kHz is 2.48 and that of $\tan\delta$ is 0.054 and so we calculate that the fall in ε' from 10 kHz to 10 MHz should be:

$$\Delta\varepsilon' \approx m \ \tan\delta \ \varepsilon' \log_{10}(f_2/f_1) = 1.5 \times 0.054 \times 2.48 \times 3 = 0.60$$

In fact the actual measured change in ε' was also 0.60. This level of agreement must be regarded as somewhat fortuitous when we consider that $\tan\delta$ changes by a factor of 0.076/0.031 = 2.45 over our measured frequency range! As stated above, an agreement to within a factor of 1.5 may generally be regarded as acceptable.

For the check to be valid, the uncertainties in $\Delta\varepsilon'$ have to be small compared to the value of $\Delta\varepsilon'$ itself. In the above example, it would not be possible to perform the check if the uncertainty in $\Delta\varepsilon'$ exceeded, say, ± 0.3. In fact, the uncertainty in $\Delta\varepsilon'$ was certainly less than ± 0.05 (for a coverage factor of $k = 2$), so the check is a valid one. In the circumstances, had we measured $\Delta\varepsilon'$ to be much larger, e.g. over 1.0, or much smaller, e.g. less than 0.3, say, we could have had good reason to doubt the validity of our measurements. Likewise if, with $\Delta\varepsilon' = 0.60$ (as measured), we had measured $\tan\delta$ values as low as 0.02 or as large as 0.1 we should be equally suspicious of our results: they would not be consistent.

The formula clearly cannot be used if the value of $\tan\delta$ lies below the resolution of the measuring equipment but it may provide a useful means of checking just what the resolution is in those cases where it is not previously known. Here is another example of a measurement on a lower-loss polymer material:

Frequency	**Measured Real Permittivity** ε'	**Measured Loss Tangent** $\tan\delta$
10 kHz	3.085 ± 0.04	0.0074 ± 0.0008
300 kHz	3.025 ± 0.04	0.0097 ± 0.0010
10 MHz	2.965 ± 0.04	0.0075 ± 0.0008

This material exhibits a broad relaxation, which, in fact, reaches its loss peak near 300 kHz. With $m = 1.5$, $f_1 = 10$ kHz, $f_2 = 10$ MHz and $\log_{10}(f_2/f_1) = 3$ we have from Equation 2.21:

$$\Delta\varepsilon' \approx 1.5 \times 0.0097 \times 3.025 \times 3 = 0.132$$

As we can see, the actual value of $\Delta\varepsilon'$ is 0.120, so the agreement is well within Lynch's region of tolerance. One could argue that the 'mean' value of $\tan\delta$ between 10 kHz and 10 MHz is going to be somewhat less than 0.0097, which in fact is close to its peak value. Using a slightly lower value for $\tan\delta$ could give an even better agreement. On the other hand, it might be thought that as the uncertainty in ε' is ± 0.04, then the uncertainty in $\Delta\varepsilon'$ must be approx. ± 0.07, so the test should be regarded as only just sensitive enough for this material. However, this need not be the case: ± 0.04 was the *total* estimated uncertainty for each absolute ε' value. This *total* uncertainty takes account of systematic errors of measurement (like the effects of fringing-fields) which can give wrong *absolute* values for ε' but which *remain the same* at all measurement frequencies. As all of the data at all frequencies were obtained in one measurement run and without removing the specimen from the cell, the estimated uncertainty in $\Delta\varepsilon'$ is actually much smaller than the absolute uncertainty in ε'. In fact, in this case an uncertainty as low as ± 0.005 for the measured value of $\Delta\varepsilon'$ was estimated and we can again conclude that the above test is valid. It is not uncommon in dielectric measurements for uncertainties relating to *small changes* in ε' to be much smaller in magnitude than uncertainties in ε' itself. Measurement resolution (see Section 4.1) is commonly much better than *accuracy* in these measurements. This is a consequence of the fact that the same measurement instrument is being used throughout and that its systematic errors change only slowly with frequency.

9.3 A Dielectric Measurement in a Split-Post Dielectric Resonator.

This example gives details of a measurement upon a specimen in a 1.8 GHz Split-Post Dielectric Resonator (SPDR), see Section 7.1.8 and Figure 36, page 105. This is not so much a full worked example as an illustration of the simplicity of this type of dielectric measurement. However, it also illustrates one of the difficulties that arises in dielectric measurements when one comes to estimate measurement uncertainties.

In the laboratory the measured properties are determined by measuring just five parameters, the resonant frequencies with and without the specimen, f_i and f_o, the Q-factors with and without the specimen, Q_i and Q_o, and the mean thickness of the specimen, t_s.

As measured $f_i = 1807.65$ MHz
$f_o = 1820.39$ MHz
$Q_i = 2864.4$
$Q_o = 16855.4$
$t_s = 1.023 \pm 0.004$ mm

The results of the measurement were:

Permittivity (as measured in the SPDR): $\varepsilon' = 4.512 \pm 0.027 \pm$ systematic uncertainty
Loss tangent (as measured in the SPDR): $\tan\delta = 0.0161 \pm 0.0004 \pm$ systematic uncertainty

Not surprisingly, there are normally five dominant *practical* sources of uncertainty in this measurement technique, those associated with the measurement of t_s and those associated with the measurement of f_i and f_o, Q_i and Q_o. In SPDR measurements there is no simple explicit relationship through a *measurement equation* (see the Glossary) between these measured parameters and the permittivity and loss. This is because the required numerical relationships are encompassed in a *computer program,* based upon modal-analysis of the measurement geometry.

The uncertainties from the practical measurements in the laboratory are readily evaluated – the uncertainty budget (see Section 4.3.2) contains entries for the repeatability and reproducibility for measuring the frequencies and Q-factors and for possible temperature drifts and so on. Likewise, in this example, the specimen thickness, t_s, was measured using a bench micrometer at eight different points on the specimen face. The mean thickness and the standard deviation (s.d.) were calculated by conventional statistical techniques and the uncertainty budget associated with t_s is similar to that illustrated in the worked example in Section 4.3.2. The resulting uncertainties are the numerical values given above (at 95% C.L.).

However, the *total* uncertainty associated with these measurements must also include *systematic* contributions, to account, at least, for (i) approximations used in the model, (ii) departure of the measurement geometry from that assumed in the computer program model. For example, a warped specimen may not lie in the resonator in the position that is assumed in the model. These are much more intractable uncertainties to quantify, but in practice they can be estimated by measuring reference specimens and by carrying out measurement comparisons. Separate conclusions, based on experience may need to be drawn for ε' and $\tan\delta$. For this particular example the total uncertainty for ε' was not significantly increased by these systematic considerations, so one can quote the measurement result as being *'$\varepsilon' = 4.51 \pm 0.03$ (at 95% C.L.)'*. However, the additional systematic uncertainties for $\tan\delta$ are estimated at ± 3%, so one must quote the result *'$\tan\delta = 0.0161 \pm 0.0006$ (at 95% C.L.)'*, once the systematic uncertainties have been added in quadrature to those discussed above.

9.4 A Dielectric Measurement in an Admittance Cell

The *Equivalent-Thickness Technique* is described in a number of Sections of this Guide (notably Sections 4.3.2. 6.5 and 7.1.1), however Section 4.3.2, Figure 14, page 30, and Equation 4.1 are best referred to here in order to understand this example.

The equivalent-thickness technique was used to measure the complex permittivity of a polymer specimen at frequencies in the range 10 kHz – 10 MHz in a parallel-electrode three-terminal admittance cell with electrode radius 25.4 mm. Measurement of admittance was performed on an impedance analyser. The mean thickness of the specimen t_s was measured as 1.5517 mm. The micrometer reading with the specimen in the cell, M_i, was 2.980 mm and the micrometer zero-error, Z, measured by the method described in the next worked example, Section 9.5, was 1.334 mm. The air-gap above the specimen and top electrodes was thus:

$$t_a = (M_i - Z - t_s) = (2.980 - 1.334 - 1.552) = 0.094 \text{ mm}.$$

This is typical for the technique: approx 0.1 mm is large enough to ensure that the electrodes do not contact or compress the specimen, while at the same time, because t_a is less than 10% of the specimen thickness, the air dielectric portion of the total series capacitance formed by the air-gap and the specimen will not dilute the total effective conductivity sufficiently to reduce the sensitivity of the loss measurement significantly. As the permittivity of the specimen changes with frequency, Equation 4.1 shows that the reading of the micrometer, M_o, *without* the specimen between the electrodes *must* change with frequency, so it must be measured at each frequency. (Remember that M_o is the micrometer reading that gives the *same capacitance*, C_i, as that obtained when the specimen is between the electrodes and the micrometer reading is M_i).

The following results were obtained (NB: M_i = 2.980 mm):

Frequency	Measured capacitance C_i (pF) (with specimen in and $M = M_i$)	Measured conductance with specimen G_i (µS)	Measured conductance without specimen G_o (µS)	Micrometer reading without specimen M_o (mm)
10 kHz	53.852	0.0115	0.0003	1.9319
100 kHz	53.557	0.1491	0.0013	1.9383
1 MHz	53.224	1.44	0.0344	1.9458
10 MHz	53.663	37.8	25.9	1.9521

Note in Column 2 that the total capacitance, C_i, changes with frequency. Its fall between 10 kHz and 1 MHz is caused by the fall in the permittivity of the specimen with frequency, but the rise in capacitance at 10 MHz is an artefact caused by series inductance in the coaxial leads, see Section 2.2.1 Equation 2.8a. This illustrates how errors could occur if the 'capacitance method' were being used here instead of the equivalent-thickness method. Note also the rapid rise of the stray conductance in Column 4 between 1 and 10 MHz – this is typical of shunt losses in the leads to the cell and in the impedance analyser. The spurious increases in the apparent values of the capacitance and conductance at 10 MHz are both indicative of the fact that this particular measurement-cell/coaxial-lead/analyser combination has reached its frequency limit at 10 MHz. In order to obtain the conductivity of the specimen in the cell (i.e. the equivalent conductivity of the series specimen/air-gap capacitor) we must subtract G_o from G_i at each frequency.

The computation of the real permittivity ε' of the specimen is by means of Equation 4.1 (or 7.2) and yields the following results:

Frequency	Measured permittivity ε'	Calculated capacitance of the dielectric specimen C_d (pF)
10 kHz	3.081	35.634
100 kHz	3.043	35.187
1 MHz	2.999	34.677
10 MHz	2.962	34.260

The geometric capacitance, see Equation 7.3, of the dielectric specimen alone, C_d, (without its series air-gap) given in the last column of the table is computed directly from ε' and the specimen dimensions. From the thickness of the series air-gap we can also compute the capacitance of the air-gap, C_a, as 190.30 pF. Note that the cell used in these measurements also had a stray capacitance C_s (see Equation 7.3), which was calculated by the method given in the next worked example, Section 9.5, as $C_s = 23.86$ pF. Note also that the measured value of C_i in Column 2 of the first table above results from the *series* combination of C_d and C_a in *parallel* with the stray capacitance, C_s. The dielectric loss of the specimen is readily computed from, C_d, C_a and $(G_i - G_o)$. The results are given in the following table.

Frequency	**Measured Loss tangent** tan δ (radians)
10 kHz	0.0074
100 kHz	0.0094
1 MHz	0.0090
10 MHz	0.0077

The results are similar to those used in the second example of Section 9.2 (as this is a second specimen of the same material). The check on the consistency of the fall off, $\Delta\varepsilon'$, of ε' with frequency and the value of tan δ is fully explained in Section 2.5 and Section 9.2. In this case, the computed value for $\Delta\varepsilon'$ between 10 kHz and 10 MHz using Equation 2.21 is $\Delta\varepsilon' = 0.122$ while the measured value is 0.119 – this is good agreement for this particular check method and so it gives one confidence in the measured results.

9.5 Micrometer Zero-Error and Admittance-Cell Stray Capacitance.

This worked example illustrates two features of dielectric measurements in an admittance cell: (1) the capacitative measurement of *Micrometer Zero-Error* in a two-terminal cell as discussed in Section 6.1.5, and (2) the determination of *Stray Capacitance*, C_s, as discussed in Section 7.1.3, see Equation 7.3. This method is of use in all two-terminal or three-terminal parallel-electrode admittance cells, such as those shown in Figure 28, page 88, and discussed in Sections 7.1.1 – 7.1.3. It is *not* suitable for measuring the zero-errors of micrometers employed for the measurement of specimen thickness (see Section 6.3.1 for that). Neither is it suitable for measuring zero-errors in cells in which the electrodes are effectively short-circuited together, e.g. re-entrant cavities, Section 7.1.4. However, for a wide range of admittance-cell measurements it can provide much the best and most convenient method for measuring the zero-error. In such cells remember that the zero-error, Z, is the correction to the micrometer reading, M, which tells us what the actual *mean* gap, D, between the driven electrodes actually is, i.e. $D = M - Z$. The *mean* that we really *want* for our dielectric measurements is *the mean that is most appropriate for admittance measurements in the cell.* A capacitance determination of Z gives us just this mean – directly – and it is much more convenient to perform than taking a set of physical measurements, say, with a ball-gauge, and averaging them.

In this method the total capacitance, C, between the cell electrodes is measured as a function of M, and is also modelled by the following simple but approximate equation:

$$C = \frac{A}{M - Z} + C_s \qquad \dots (9.1)$$

where A is a constant for a given cell and C_s is the stray capacitance in the cell – see Equation 7.3, Section 7.1.3. C_s is just that part of the capacitance across the cell which is independent of M. C_s typically arises from the capacitance between the mountings of the two electrodes. In three-terminal measurements (Section 7.1.1) C_s should be very small compared with C. In two-terminal measurements (Section 7.1.3) C_s will not be small: in fact C_s may be as large as a typical value of the first term on the right side of Equation 9.1.

By comparing Equation 9.1 with Equation 7.3, the nature of the approximation being used here will readily be seen: *Equation 9.1 omits a term for the fringing capacitance, C_f.* This C_f-term is very important when it comes to actual dielectric measurements (as explained in 7.1.3) but for the present purpose it may be ignored for the following reason. In determining Z from capacitance the most sensitive capacitance measurements will occur when the actual gap between the electrodes, D, is very small. For instance, in the following worked example, the smallest value of D used is approx. 0.07 mm (whereas the smallest value used in actual dielectric measurements might be typically 0.3 mm). For such small gaps the fringing capacitance becomes almost negligible in magnitude compared with the geometric capacitance – see Section 7.1.3 for more detail. In fact the effect of fringing is not completely negligible but it can to a large extent be compensated for by allowing A to be a free parameter in the curve fitting that follows. This can be done because A is not a quantity that we need to know for our present purposes. In general the smaller the electrode gap, D, for which we measure C the better will be the approximation and in practice, for typical admittance cells, the approximation employed in Equation 9.1 results in a very small error for Z.

The measurement procedure is as follows. First clean the electrodes, then connect an impedance analyser, or equivalent, across the cell. Use just one convenient frequency, typically in the range 10 kHz – 1 MHz, and adjust the bridge to give a rapid capacitance readout. First, adjust the micrometer by hand and monitor the bridge reading by eye, following the value of C as M is reduced *very carefully and slowly.* Determine the approximate micrometer reading, M_T, at which the electrodes are almost touching: this will be clear because C will rise very rapidly as M falls at this point. You may find that the electrodes actually do touch (if so the bridge will refuse to give a sensible reading). This is ideally to be avoided, but the contact should not cause damage to the cell as long as M is reduced *very slowly*. Forceful and fast changes of M are to be avoided: if the electrodes are screwed together forcefully the cell and the electrodes can be damaged. Having determined the approximate contact point, back the micrometer off and readjust the bridge for accurate (probably slower) capacitance measurements. Record measurements of C for M values in the range approximately 0.25 – 0.05 mm greater than M_T. This range should be suitable for a typical capacitance cell with electrode diameters in the range 20 – 50 mm, but once you have practiced and begun to get a feeling as to how this method works, it is advisable to experiment to see if another range is better for your particular cell. Note that you should remove your hand from the cell and withdraw at least 0.5 m from it when making capacitance measurements: the presence of the hand will change the apparent cell capacitance.

The following table shows a set of such capacitance measurements on a two-terminal admittance cell of 50-mm electrode diameter in which the electrode contact point has been estimated as approximately $M_T \approx 1.4$ mm. The following nine capacitance measurements were subsequently obtained:

i	1	2	3	4	5	6	7	8	9
M_i mm	1.62	1.60	1.58	1.56	1.54	1.52	1.50	1.48	1.46
C_i pF	88.83	99.88	115.15	125.67	138.67	156.06	180.63	217.07	279.76

Now the free parameters in Equation 9.1, namely A, Z and C_s, should be fitted to this data. This can be done by hand or graphically, but such methods can be time-consuming. These days there are many user-friendly computer-based tools available that can facilitate this fitting exercise (e.g. *Matlab*, *Mathcad*, *Mathematica*). The fitting can be done as follows. Equation 9.1 should be used to define the capacitance function $C(M, A, Z, C_s)$, then the following expression should minimised:

$$B = \sum_{i=1}^{9} \left[\frac{C(M_i, A, Z, C_s) - C_i}{C_i} \right]^2 \qquad \ldots (9.2)$$

Where M_i and C_i are the measured values in the table. In this case the following optimum values are obtained:

$A = 17.297$ pF mm, $\quad Z = 1.391$ mm, $\quad C_s = 23.33$ pF

Z is the value we want for the zero-error. Note that in Equation 9.2 the denominator on the right-hand-side acts as a *weighting factor* which defines the importance of the individual measured data points. The result for Z is usually robust: by experimenting with a range of different plausible weighting schemes, e.g. by using a different denominators in Equation 9.2, and with some experience we can show that the uncertainty in Z introduced by uncertainty about which weighting scheme ought to be used will typically be ± 0.003 mm, or in some cases, less than this.

The large value of C_s compared with C is typical of a two-terminal cell. This can dilute the sensitivity of two-terminal measurements and is yet another reason why three-terminal cells are to be preferred whenever possible.

We don't actually need to know the value of A, but by reference to Equation 7.3, we can readily show that for 50-mm diameter circular electrodes in the absence of fringing-fields, we would expect $A = 17.38$ pF mm. The slightly different value obtained is accounted for by the fact that fringing-fields actually are present in the measurement cell, but have been omitted in Equation 9.1. The value of C_s obtained is approximate for rather similar reasons, but again one rarely needs to know it very accurately

9.6 Loaded and Unloaded Q-factor

Referring to Section 6.4 in general and Section 6.4.3 in particular, if the Q-factor of a resonator is measured *by transmission* with a calibrated instrument such as a network analyser, we see that Q_0, the unloaded Q-factor, may be obtained from Q_l, the loaded Q-Factor, by means of the simple correction formula given in Equation 6.12, page 70. This formula assumes that the resonator is weakly-coupled [124, 125]. Theoretically, the ANA must be calibrated in the measurement-plane of the coupling ports, however the usual approach is to calibrate in another plane and assume that the transmission-line sections couplings between the calibration planes and the resonator are lossless and free from mismatches. The correction formula is

$$Q_0 = \frac{Q_l}{1-d}$$

in which d is the Q-circle diameter (see Figure 25 (b), page 72). In scalar measurements this may be obtained from $d = \left|S_{21}\right|_{at\,peak}$ provided that the leakage in the system is negligible (see Section 6.4.2). The validity of the correction may be investigated by varying the coupling. If all is well Q_0 should not vary significantly. In many instances the assumption that the couplings are lossless does not hold, so much larger uncertainties must be attributed to Q_0 than to Q_l. In the absence of good quantitative information on these uncertainties, it pays to be conservative. It has been found by experiment that, for Q-factors in the range 10^3 to10^5, a simple and generally successful strategy is to take the uncertainty in Q_0 to be the difference between Q_0 and Q_l. (at coverage factor $k = 2$), subject to a minimum uncertainty of ± 1%. If the signal-to-noise ratio is poor, a repeatability contribution to the total uncertainty should also be taken into account.

Here is a simple worked example. Assume Q_l is measured as 15718, and $\left|S_{21}\right|_{at\,peak}$ = 0.011. Then Q_o = 15718/(1.0 - 0.011) = 15893. Using the above rule of thumb the uncertainty in Q_o at coverage factor $k = 2$ can be taken to be ± (15893 -15718) = ± 175, i.e. approximately ± 1%, to which other contributory uncertainties in the measurement of Q-factor should be added.

9.7 ANA-Based Transmission Line Measurements

This example illustrates the measurement of permittivity and loss in a coaxial transmission cell, see Section 7.1.10. For simplicity, the example assumes zero conductivity loss in the metalwork of the transmission line, though in practice this conductivity loss is sometimes significant, especially with lower-loss specimens.

Let us assume we are using a 50-Ω 7-mm diameter coaxial line, 20 mm in length, that the length of the specimen is 5.0 mm and that it is a non-magnetic specimen with complex permittivity $\varepsilon^* = 10.0 - 1.0j$. Assume also that the specimen is placed flush with the Port 1 end of the cell, see Figure 58 (a).

If the specimen completely fills the cross-section of the line (no gaps), it is easy to calculate [83] that at 1 GHz the cell containing the specimen should have the following S-parameters:

$$\begin{array}{llll}
S_{11} = & -0.2389 - j\,0.3279 & |S_{11}| = & 0.4057 \\
S_{22} = & -0.3861 - j\,0.1247 & |S_{22}| = & 0.4057 \\
S_{21} = S_{12} = & 0.5844 - j\,0.6457 & |S_{21}| = |S_{12}| = & 0.8709 \qquad \text{(1 GHz)}
\end{array}$$

Note that $S_{11} \neq S_{22}$ because of the asymmetrical mounting of the specimen in the cell. However $|S_{11}| = |S_{22}|$, showing that it is only the *phase* of these reflection coefficients which differs, as is to be expected in a lossless transmission cell. Note, however, that, by contrast, $S_{21} = S_{12}$, showing, in general that the position of the specimen in the cell does not affect the transmission coefficient. This has practical measurement advantages and is one of the reasons why computing ε^* *only* from S_{21} and S_{12} and not S_{11} or S_{22} often delivers better measurement uncertainties (*Warning:* this is not always so, especially at lower RF frequencies where $|S_{21}|$ and $|S_{12}| \rightarrow 1.0$, and transmission measurements become insensitive to ε^*). Note also that the quadrature sum of $|S_{11}|$ and $|S_{21}|$, i.e. $\sqrt{\left|S_{11}\right|^2 + \left|S_{21}\right|^2}$ = 0.9608 which is less than 1.0, demonstrating that the specimen is lossy (as we already knew).

In practice one would *measure* these S-parameters and compute ε^* by means of iteration, using, say, a Gauss-Newton algorithm, as discussed in Section 7.1.10, or else one could use another explicit method, e.g. [168] and so the computation discussed above would be reversed.

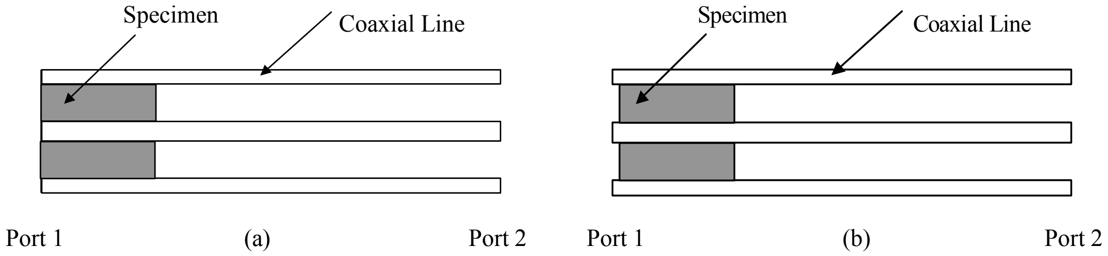


Figure 58. (a) A dielectric specimen placed at the end of a coaxial line for measurement by the transmission line method. In **(b)** the specimen has been placed erroneously in the wrong position in the line.

We can also use this example to look at the effect of measurement errors, as follows.

Measurement Error 1: The Effect of Air-Gaps Around Solid Specimens, see Figure 37, page 107. The specimen has to be machined to fit easily, without binding, into the coaxial cell, so, in practice, air gaps will be present between it and the outer- and inner-conductors, as shown in Figure 37. Unless a correction is made, the apparent measured value of ε^* will be diluted by the permittivity of the air in the gap. It is instructive to compute just how large this dilution effect can be, using the formulae in the report by Baker-Jarvis *et al.* [83]. Assume again that we are using a 50-Ω 7.00-mm diameter coaxial line, so that the internal diameter of its outer-conductor should 7.00 mm and the diameter of its inner-conductor should be 3.040 mm. Once again our specimen has $\varepsilon' = 10.0$ and $\varepsilon'' = 1.0$ and we place it in the same measurement cell discussed above with the specimen once more flush with the Port 1 end of the cell as in Figure 58 (a). However, this time we will carry out the measurements at 10 GHz (to make the errors larger). Without any air-gaps we obtain:

$S_{11} = -0.2244 - j\,0.1281$ $\qquad |S_{11}| = 0.2583$

$S_{22} = -0.2249 - j\,0.1271$ $\qquad |S_{22}| = 0.2583$

$S_{21} = S_{12} = 0.7252 - j\,0.1848$ $\qquad |S_{21}| = |S_{12}| = 0.7484$ $\qquad$ (10 GHz)

Now at 10 GHz …

> … if there is a 5-μm gap between the specimen and the *outer-conductor* only, i.e. if the specimen's outer diameter is 6.990 mm, and *if no correction is made for this gap*, the apparent measured permittivity will be $\varepsilon' = 9.85$ (1.5% error) and $\varepsilon'' = 0.968$ (3.2% error).
>
> … if there is a 5-μm gap between the specimen and the *inner-conductor* only, i.e. if the specimen's inner diameter is 3.050 mm, and *if no correction is made for this gap*, the apparent measured permittivity will be $\varepsilon' = 9.66$ (3.4% error) and $\varepsilon'' = 0.929$ (7.1% error).

Notice that the effect of the gap around the inner-conductor is much greater, because its diameter is so much smaller than that of the outer-conductor, see also Figure 39, page 108. Notice also that in both cases the error of measurement is quite large. This illustrates how important it is to make corrections for annular air-gaps in this technique.

Measurement Error 2: Misplacement of the Specimen in the Measurement Cell. As pointed out above, the phase of S_{11} depends upon the positioning of the specimen in the measurement cell. Let us say that the specimen is misplaced by 0.1 mm – i.e. that it is not flush with the end of the measurement cell, as shown in Figure 58 (b). Then at 10 GHz we will measure a different value for S_{11}, in this case we obtain $S_{11} = -0.2295 - j0.1186$: compare this with the figures given above. If we do not know that the specimen is in the wrong position in the cell, i.e. if we assume that the specimen *is* flush with the end of the cell, and we compute ε^* from this S_{11}, we will compute $\varepsilon' = 9.61$ (3.9% error) and $\varepsilon'' = 1.066$ (6.6% error). This error arises *only* if we compute ε^* from *reflection* measurements. As shown above, S_{21} is independent of the actual position of the specimen, so mis-positioning of the specimen will not cause a measurement error if we rely solely on *transmission* measurements. This demonstrates one way in which reflection measurements are more prone to error than transmission measurements. Note, though, that this particular error can be compensated for if we measure both S_{11} and S_{22} and take the average result. Note also that this discussion applies only to *homogeneous, non-magnetic* specimen materials – the conclusions drawn here do not necessarily apply to *heterogeneous* or *magnetic* specimens.

10. Glossary

10.1 Introductory Notes.

This Glossary provides an explanation of the important technical terms used in the Guide. Please note the following important points:

Preferred and Non-Preferred Terminology. A number of terms that have long been in use for the description and characterisation of dielectric properties are no longer recommended internationally, either because they give an *inaccurate description* of the parameters they name (e.g. 'dielectric constant' is not constant) or because they may be *ambiguous* (e.g. 'dissipation factor'). It is helpful to understand how these terms are commonly used, however, so we have included some of these terms in this glossary but have clearly indicated those which are to be preferred and those which are not.

Terms Unique to this Guide. Some *non-conventional* terms, such as '*Measurement Equation'*, have been adopted purely for convenience within the Guide and have been used throughout with a unique (non-ambiguous) meaning. They are explained below, but their non-conventional status is noted because their use without explanation outside the context of this Guide may cause confusion.

Abbreviations. Recognised terminological abbreviations are used throughout this Guide for convenience. For example 'permittivity' is used to stand for 'the real part of the relative complex permittivity', 'loss factor' stands for 'the imaginary part of the relative complex permittivity'. The Glossary should help to clarify such usage.

Conventional Symbols. While there is some degree of uniformity in the symbolic conventions used for quantities encountered in dielectric studies, conventions are far from universal. The symbol for the relative permittivity itself, for example, may be ε, ε^*, ε', ε_r, E, E', k or κ; depending on the publisher and originator of the text! The consistent set of symbols used in this Guide corresponds to just one of the common conventions. The Glossary explains correspondences with other common conventions, where appropriate.

General Metrological Terms. Most terms relating to *uncertainty*, *errors*, *calibration*, *traceability,* are explained in greater detail in Section 4.1.

10.2 Glossary

3-dB points. The two points on a resonance curve, on either side of the resonance peak, at which the amplitude of the response is 3 dB below that at the peak of the resonance – i.e. the points at which the amplitude is $1/\sqrt{2}$ of that at the peak, otherwise known as the 'half power points'. The 3-dB points can either be defined in terms of the frequency of excitation of the resonator or else in terms of the 'length' of the resonator, if it can be adjusted by a micrometer. This 'length' may be the physical length if the resonator is a cavity or it may be some other key linear parameter such as the distance between electrodes in an admittance cell. See Section 6.4.

ANA. An Automatic Network Analyser, see Section 6.1.1 – the work-horse of the modern microwave laboratory. The ANA is an automated instrument for measuring S-parameters (Section 2.2.2). There are two common types of ANA in use – *Vector Network Analysers* (VNAs), which measure both the *amplitude* and *phase* of the S-parameters, and *Scalar Network Analysers* which measure only their amplitude. Vector ANAs are more useful in dielectric measurements and provide greater opportunities for assessing and reducing uncertainties. *Note that in this Guide the term 'ANA' is used to signify a Vector ANA, unless otherwise specified.*

Anisotropic Material. See Section 5.3.11. In the context of dielectrics, this signifies a material that exhibits at least two different permittivities at any point inside it. In a *uniaxial* anisotropic material, for example, the two permittivities are $\varepsilon^*_\perp$ and $\varepsilon^*_\parallel$, which apply, respectively, when the E-field is *perpendicular to* and *parallel to* the *optic-axis* in the material. These two permittivities can readily be distinguished in measurements, see for example Figure 52, page 136 in Section 7.1.17. More general forms of anisotropy exist, and complex permittivity should strictly be seen as an intrinsic *tensor* property of a medium [1, 4]. The concept of isotropy (and anisotropy) should not be confused with the homogeneity (*q.v.*) of a medium. A material like a pure crystal may be *both homogeneous and anisotropic*.

Calculable Geometries & Calculability in Mathematical Models. A measurement geometry may be said to be analytically *calculable* if a finite set of *measurement equations* (*q.v.*) can be used to derive the values of the quantities to be determined (e.g. ε' and $\tan\delta$) from the parameters actually measured (e.g. capacitances, inductances, frequency and length changes, Q-factor changes, etc.). The derivation should be performed with sufficient precision to allow one to obtain one's required uncertainty from the measurement. Traditionally, before the advent of powerful computers, *measurement geometries* (*q.v.*) were kept as simple as possible and contained a high degree of symmetry (e.g. cylindrical symmetry) so as to ensure that the mathematics used in deriving the measurement equations was tractable and, if possible, that it would deliver *explicit formulae* for the quantities to be determined. With the aid of computers a much wider range of measurement geometries (including, for example, that of the coaxial probe – Section 7.1.12) has become calculable through the use of modelling schemes such as modal analysis, boundary-value analysis, etc. The requirement for *explicit* formulae is also reduced if computers are employed because the quantities to be determined can be obtained by *iteration* of formulae in which their values are *implicit* (see Section 7.1.10). Calculable geometries are to be contrasted to *non-calculable* complex measurement geometries with low degrees of symmetry that can only be modelled by *discretised* E.M.-field techniques (*q.v.*), see also Section 6.9. (The ongoing march of progress in computer modelling may yet prompt a re-assessment of this distinction, however). Metrologically, the importance of having a calculable geometry (as opposed to a non-calculable measurement geometry that has to be calibrated, see, e.g., Section 7.1.13) is that it delivers more confidence in the measurements performed. The derivation of the measurement equations is in the public domain. They can be tested both analytically and by comparing computations with measurements on reference materials (*q.v.*) and so they can be *validated* (Section 6.9) and hence be accepted as part of a traceability chain.

Cavity. A *resonator cell* (*q.v.*), usually made from low-loss metal, that is a total enclosure for the specimen. In dielectric measurements the specimen may fill just part of, or all of, the cavity. Some cavities may have small apertures for insertion and removal of specimens, but others may have to be disassembled for this purpose.

Cell. In dielectric measurements, the cell is the enclosure, usually made from a low-loss metal, that holds the dielectric specimen during its measurement. The cell is designed to set up an E.M. field pattern in the specimen that will facilitate its accurate measurement. The cell may also be used to control the physical conditions (temperature, humidity, pressure etc.) under which the specimen is being characterised. Some cells are *resonator cells* (*q.v.*) and some resonator cells are called *cavities* (*q.v.*).

Change-of-Frequency Method in a resonator or cavity. In measurements of ε', this is a method in which ε' is computed from the change of resonant frequency, f_r, obtained when the specimen is inserted into the resonator. In a $\tan\delta$ measurement it either refers to a method in which the Q-factor is computed from the difference in frequency between the two 3-dB points (*q.v.*) or else one in which it is computed automatically by curve-fitting the shape of the resonance curve as a function of frequency - see Sections 6.1.9 and 6.4.

Change-of-Length Method in a resonator or cavity. In measurements of ε', this is a method in which the length of the resonator, or of some key linear parameter in the resonator such as the distance between electrodes, is changed in such a way that it resonates at the same frequency both with and without the specimen in it. In a $\tan\delta$ measurement it either refers to a method in which the Q-factor is computed from the difference in length between the two 3-dB points (*q.v.*) or else one in which it is computed automatically by curve-fitting the shape of the resonance curve as a function of the length – see Sections 6.1.9 and 6.4.

C.L. = *Confidence Level* (*q.v.*) in uncertainty statements.

Complex Permittivity, ε^*. As commonly used and as used in this Guide, the term '*Complex Permittivity*' is equivalent to 'complex relative permittivity'. It has real and imaginary parts and is defined in such a way as to make the numerical value of the imaginary part (the loss factor) always positive for passive lossy dielectric materials: thus $\varepsilon^* = \varepsilon' - j\,\varepsilon''$, where $j = \sqrt{-1}$. Note the minus sign, which is explained in Section 2.1. From a practical point of view and at sufficiently low frequencies in a lumped impedance approximation (see Section 2.2.1) ε^* can be defined by the ratio of the admittance, Y, between two electrodes with and without the dielectric present (see Sections 5.3.3 and 7.1.1):

$$\varepsilon^* = \frac{(Y \text{ when the space around the electrodes is completely filled with the dielectric})}{(Y \text{ when the space around the electrodes is completely filled by a vacuum})}$$

This forms the basis for many of the RF techniques for ε^* described in this guide, but it is usually only practicable to fill *all* of the space around the electrodes when the dielectric is a liquid (or a vapour or a gas). For measurements on solid dielectrics, methods have to be modified to correct for E-fields which do not pass through the dielectric under test (see Sections 7.1.1 – 7.1.3) *(Preferred Term).*

Conductivity, σ. A formal definition in physics is: ‘a factor such that the conduction-current density is equal to the electric field intensity in the material multiplied by the conductivity’. The term therefore formally describes electrical properties of *free* charge carriers (ions, electrons, holes). However in dielectric metrology the term is often used *phenomenologically* to cover *all* electrical loss effects *including* dielectric loss: see Section 2.4. *(Preferred Term).*

Confidence Level (C.L.) in uncertainty statements. A quantification of the degree of confidence that one has in one’s stated uncertainty of measurement, see Section 4.2.2 for details.

COSHH - *Control of Substances Hazardous to Health* regulations are specified by the UK Government’s Health and Safety Executive (HSE), see Reference [82].

Coverage Factor in uncertainty statements – see *Confidence level* (C.L.) and Section 4.2.2 for details.

Customer. The person, institution or organisation that has commissioned measurements from you. (You may, of course act as your own customer). This is used as a term of convenience in this Guide and is *not* a conventional usage. (*Informal term*).

Degree of Anisotropy. Uniaxially anisotropic dielectric materials exhibit two permittivities, $\varepsilon^*_\perp$ and $\varepsilon^*_\parallel$, when the E-field is, respectively, perpendicular to or parallel to the *optic-axis* in the material. In this Guide we have used the term *‘degree of anisotropy’* to quantify the magnitude of the difference between $\varepsilon'_\perp$ and $\varepsilon'_\parallel$. It is defined as $|\, \varepsilon'_\parallel - \varepsilon'_\perp \,|/\varepsilon'_\parallel$, usually expressed as a percentage. This is not a general usage, however. (*Informal term*).

Dielectric Constant. The term ‘dielectric constant’ is widely used to mean ‘real permittivity’ or ‘the real part of the relative complex permittivity’. But this term is also used by some authors for the low frequency permittivity or ‘*static permittivity*’ (*q.v.*) in contradistinction to ‘*permittivity*’ taken to be a function of frequency, which is used to describe RF & MW real permittivities. Though still in wide use, ‘dielectric constant’ is not a preferred term, partly because of this ambiguity, but also because permittivity is *not* constant - it changes with frequency, pressure and temperature, relative humidity, etc! (*NOT a preferred term – use ‘Permittivity’ instead).*

Dielectric Loss. An informal term for the power removed from the electromagnetic field by a dielectric process, the power usually being converted into heat. Also used loosely to refer to ε'', tan δ, δ, σ, etc. See the more general term *Loss*. *(Informal term).*

Dielectric Material. In common usage this is actually quite an ill-defined term. Formally, a complex permittivity parameter, ε^*, can be defined to characterise virtually *any* body of material (solid, liquid, gas, plasma, etc.) which exists in a sufficient quantity to be modelled on a macroscopic scale. So, in a sense, *all* materials can be dielectrics if they exist in macroscopic quantities. However, in the practical RF & MW world, the term tends to be used to describe *non-metals* and *non-superconductors* - both metals and superconductors being taken to be very good conductors. *Semiconductors* are sometimes also differentiated from dielectrics, because they are typically used *actively* in electronics, whereas dielectric components are ideally meant to respond *passively*. At LF the term ‘dielectric’ is sometimes taken to mean ‘insulator’ but many dielectrics are actually very poor insulators. In this Guide we take ‘dielectric material’ to signify *any* material for which ε^* can be meaningfully defined. Note that dielectric materials may also be *magnetic materials* (*q.v.*).

Discretised Modelling – of a computer modelling technique implies a method in which the continuous physical space in which the electromagnetic fields and currents are being modelled is represented by a regular *discrete* grid of points – this is usually three-dimensional but many problems can be reduced to two-dimension problems, thus saving computing time if suitable symmetry exists in the geometry. Typical methods are FD, FDTD, FE, FI and TLM (*q.v.*).

Dissipation Factor. ‘Dissipation factor’ is usually equivalent to ‘loss tangent’. This is a widely used term for describing dielectric properties but it is not a preferred term because it can have a very different meaning in other disciplines. *(NOT a preferred term).*

Double-Pass Technique. In *travelling-wave methods* (*q.v.*) a double-pass technique is a *reflectometric technique* (i.e. one in which reflection coefficient is measured), as opposed to a *transmission technique* (which is often referred to as a *single-pass technique* (*q.v.*)). ‘*Double-pass*’ refers to the fact that the wave passes through the specimen twice, once on its outward path before reflecting from a mismatch at the back of the specimen (e.g. a short or open-circuit) and once more on its return path. See also *multi-pass technique*.

D.U.T. or d.u.t. – in this Guide a convenient abbreviation for *dielectric under test* – this is not a conventional usage, however.

E-field. A common abbreviation for electric field.

Eigenvalue Solver. A software module in an electromagnetic-field modelling package which is able to identify the resonant frequencies, and other information about, the resonances of a resonant structure. This tool is invaluable for helping one to identify modes in resonators. See Section 6.9 and *discretised modelling*.

Electrode Polarisation Effect. A conduction effect that affects 'free' charge carriers in liquids, typically at LF. When an electric field is applied between electrodes across a conducting liquid, some of the charge carriers, typically ions, find it impossible to move freely into the electrode – which the electric field is encouraging them to do. They therefore accumulate there and produce a series impedance, see Figure 16, Section 5.3.3, page 43, which is very difficult to quantify. The effect can produce a significant error in LF liquid dielectric measurements. Various techniques are used to avoid the effect, see Section 5.3.3 (see also Section 7.1.19 for time domain measurements). It falls away rapidly as the frequency rises through the RF range.

E.M. or **e.m.** = electromagnetic.

Extrinsic Measurement or Property. *Extrinsic* measurements quantify *extrinsic* properties of specimens, such as (i) reflectivity, (ii) diffuse scattering (iii) transmission through specimens. *Intrinsic measurements* (*q.v.*), by contrast, determine the *intrinsic* properties of a material, e.g. ε^* and μ^*.

FD, FDTD, FE and FI Modelling Techniques: respectively *Finite Difference*, *Finite Difference Time Domain, Finite Element*, and *Finite Integration* methods for modelling electromagnetic fields and electric currents by means of *discretised* (*q.v.*) computer algorithms. See Section 6.9. See also '*TLM'*. Many of these methods are particularly suitable for modelling dielectrics in measurement cells and are useful for helping to estimate errors of measurement in them.

FD is also an abbreviation for *Frequency Domain* (*q.v.*) as opposed to *Time Domain* (TD) (*q.v.*).

Free-Field Methods, in contrast to *Guided-Wave Methods*, are methods in which the E.M. waves travel in free-space, usually without any form of lateral guidance. The waves are typically controlled and directed by lenses, concave mirrors, antennas, etc. See Sections 7.1.14, and 2.2.2 for an introduction.

Frequency Domain (FD). We say that we are working in the *Frequency Domain* if we are measuring parameters like ε' and ε'' as a function of *frequency*. Compare *Time Domain (TD)*. Most of the techniques described in this Guide are frequency-domain techniques.

Filling-Factor, F_f. In resonators and cavities this is the ratio between the electromagnetic energy stored in the specimen on resonance and the *total energy* stored in the resonator on resonance (*including* that in the specimen). See Section 6.1.9 and Equations 6.3a & 6.4 [116] pages 62 and 63. Filling-factors quantify how sensitive a resonator is to the presence of the dielectric specimen.

Gating. A measurement technique (more strictly a *post*-measurement data-treatment technique) that can be used to increase measurement accuracy in *travelling-wave methods* (*q.v.*) by distinguishing in the *time domain* (q.v.) between signals from the specimen and spurious signals originating from elsewhere in the measurement system. Gating can be used both in *time-domain* measurements and, since the advent of computer-controlled ANAs, in *frequency-domain* measurements too. See Section 7.1.19.

Geometric-Factor, G_s. In resonators and cavities this is a parameter with the dimensions of resistance (i.e. ohms) that relates the loss in the resonator caused by conduction in its metal walls to the surface resistance R_s of the metal. See Section 6.1.9 and Equations 6.3b & 6.5 [116].

Guided-Wave Methods, in contrast to the *Free-Field Methods,* are methods in which the E.M. waves travel through, and the specimen is contained in, transmission-lines. These may be coaxial line, waveguide, microstrip, co-planar line, etc. See 7.1.10 and 7.1.11, and 2.2.2 for an introduction.

Heterogeneous Material. See *Inhomogeneous Material.*

H-field. A common abbreviation for magnetic field.

High Frequency Permittivity Limit, ε_∞. Used in parameterised dielectric relaxation models, Section 2.3 (e.g. The Debye model, Equation 2.16). ε_∞ gives the lowest value which ε' reaches at very high frequencies *in the parameterised model*. It does *not* usually correspond to the actual physical permittivity at these frequencies because other loss processes that are not included in the model reduce ε' still further (ultimately to 1.0 at gamma ray frequencies!).

High-Loss. In this Guide, for convenience of description, this term is used typically to apply to materials with a loss tangent greater than 3×10^{-2}. This is *not* a conventionally recognised definition for high loss, however.

Homogeneous Material. A material whose macroscopic *intrinsic properties* (*q.v.*) are independent of their spatial position in the body of the material. This is to be compared with an *Inhomogeneous Material* (*q.v.*) which does not have this property. Compare also with *Anisotropic Material* (*q.v.*). NB. *Homogeneity* and *inhomogeneity* are opposite terms, *homogeneity* and *anisotropy* are not: a material may be *both homogeneous and anisotropic*.

Imaginary Permittivity, ε''. As commonly used and as used in this Guide, the term '*Imaginary Permittivity*' is equivalent to 'loss factor' or 'the imaginary part of the complex relative permittivity'. See also Section 2.1 *(Preferred Term).*

Inhomogeneous Material. A material whose macroscopic *intrinsic properties* (*q.v.*) are a function of spatial position in the body of the material. This is to be contrasted to a *Homogeneous Material* (*q.v.*). *Heterogeneous* materials are inhomogeneous materials.

Intrinsic Measurement or Property. An *intrinsic material property* of a body of material is one which is independent of its external geometry and (on a macroscopic scale) of its size. Complex permittivity, ε^*, and magnetic permeability, μ^* are macroscopic *intrinsic* material properties (note that they are defined, strictly, only on macroscopic scales). *Intrinsic Measurements* are those that are designed to determine the intrinsic electromagnetic properties of materials, e.g. ε^* and μ^*. They are to be contrasted with *Extrinsic Measurements* (*q.v.*), which determine *extrinsic parameters* like reflectivity or transmission coefficient.

LF – Low Frequency. In this Guide, for convenience of description, typically frequencies below 1 MHz. This is *not* a conventionally recognised definition for LF, however.

Loaded Q-factor. Q_l, see Q-factor. To be compared with *Unloaded Q-factor* (*q.v.*). In general usage, a Q-factor in a resonator that is *reduced in magnitude*, either (i) by the presence of the specimen in the resonator, or, (ii) by loss of power through the coupling ports of the resonator. There is often ambiguity in normal usage as to whether the 'loading' is by the *specimen* or by the *coupling* into the resonator – see Sections 6.4.3 and 6.4.4 – so do make sure that you understand the sense in which this term is being used. By extension, the Q-factor can also be said to be *loaded* by any other source of loss that it suffers from (e.g. loss in a post that is supporting the specimen). (*Ambiguous*).

Loss. An informal term for power that is removed from the electromagnetic field in a material or measurement system. It may be absorbed by dielectric specimens or by the metal walls in cells and converted into heat or it may escape by diffraction or through coupling holes, etc. See Figure 21, page 64. Any physical process, including dielectric relaxation which gives rise to loss is called a *loss process*. '*Loss'* is also used loosely to refer to parameters such as ε'', tan δ, δ, σ, etc. *(Informal term).*

Loss Angle, δ. 'Loss Angle' is the smallest positive angle, δ, measured in radians, of which the loss tangent ('tan δ') is the tangent (i.e. it is the arctangent of the loss tangent). The term is usually used for materials with medium to low loss, e.g. those having a loss tangent less than 0.03. For such materials the loss angle, expressed in radians, is invariably numerically equivalent to the loss tangent to within practical measurement uncertainty limits. The use of 'loss angle' instead of 'loss tangent' allows the loss to be expressed in terms of the convenient unit 'microradian', µrad, which has the value 1×10^{-6} radians ('milliradian', mrad, is also used for 1×10^{-3} radians). Verbal expression of such low losses is often more convenient in terms of loss angle then in terms of loss tangent because the latter is unitless and cannot be expressed in terms of radians. Thus, it is easier to say "*the loss angle is 60 microradians*" than to say "*the loss tangent is 60 times-10-to-the-minus-6*". The hybrid "*the loss tangent is 60 microradians*" is clearly an error! See Section 2.1. *(Preferred Term).*

Loss Factor, ε''. 'Loss factor' is equivalent to 'imaginary permittivity' or 'the imaginary part of the relative complex permittivity'. See also Section 2.1 *(Preferred Term).*

Loss Tangent, tan $\delta = \varepsilon''/\ \varepsilon'$. The loss tangent of a material is the ratio of its imaginary permittivity to its real permittivity. Loss tangent is often referred to as 'tan δ' where 'δ' is the loss angle. See also Section 2.1. *(Preferred Term).*

Lossy: an adjective used to describe any material that exhibits significant electromagnetic loss (whether dielectric or magnetic).

Low-Loss. In this Guide, for convenience of description, this term is used typically to apply to materials with a loss tangent below 3×10^{-4}. This is *not* a conventionally recognised definition for low loss, however.

Lumped Circuits and Lumped-Impedance Methods. At LF *lumped circuits* are those whose response to an electromagnetic signal can conveniently be modelled by a (small) finite number of *R*, *L*, and *C* (i.e. *resistance*, *inductance* and *capacitance*) components. *Lumped-Impedance Methods* for measuring dielectric properties, typically derive their *measurement equation* (q.v.) by modelling the dielectric specimen and cell as a lumped circuit (see, e.g. those in Figures 2, 9 and 57). In such methods dielectric properties are measured as an *impedance*, or more commonly as an *admittance* (e.g. on an impedance analyser or bridge). Lumped-impedance methods can also be used at RF, but as the frequency rises *wave methods* (*q.v.*) become more convenient and are less prone to error for dielectric measurements, see Sections 2.2.1 and 2.2.2.

Magnetic Material. In this Guide we refer to materials for which μ^* differs significantly from (1.0 - j0.0) at any frequency in one's frequency range of interest as *magnetic materials*. This is not a formal term, though widely used (*Informal term*).

Matching. A component connected to a transmission line is said to be *matched* if it produces zero reflection coefficient, Γ, (or, at least, a very low reflection coefficient: $\Gamma << 1$) when E.M. waves that are travelling through the line are incident upon it. Equivalently, if the characteristic impedance of the line is Z_0, the component will be *matched* if its own input-impedance is Z_0, see Section 2.2.2 and Equation 2.9, page 10.

Measurement Equation. In this Guide we have used the term *Measurement Equation* for the equation associated with any measurement system that relates the actual measured parameters (e.g. capacitances, frequencies or displacements) to the quantities to be determined – usually ε' or ε'' in dielectric measurements. A simple example is Equation 7.2. By partial differentiation of ε' or ε'' with respect to the measured parameters in the equation (as discussed in Sections 4.3.2 and 4.3.3) one can compute *sensitivity coefficients* (*q.v.*) which help to quantify how uncertainties in the measured parameters contribute to uncertainties in the complex permittivity. Inspection of the measurement equation is to be recommended to help one to optimise the sensitivity of measurements, for example by choosing specimens of the optimum thickness to minimise uncertainties in ε^*. In practice many of the more sophisticated measurement methods described in this Guide have to employ more than one measurement equation, or else the relationship between their measured parameters and ε^* has to be captured in a computer program. But the principle of the propagation of uncertainties from measured parameters to the complex permittivity remains the same. In more complex cases the partial differentiation is best done numerically, as described in Section 4.3.3. Note that the term '*measurement equation'* is used for convenience in this Guide and is *not* conventional.

Measurement Geometry. In any given measurement, two distinct *Measurement Geometries* may be distinguished. (1) The *physical geometry* of the specimen and the cell in which it is contained during the measurement. (2) The geometry of cell, specimen, specimen holder, E.M. field, etc. that is *assumed* when *modelling* a measurement method in order to derive its *measurement equations* (*q.v.*). This is the *Modelled Measurement Geometry*. Systematic errors arise in measurements if the salient features of the latter do not adequately model the former.

Measurement Plane. In S-parameter and ANA-based measurements the *measurement plane* is the point in the transmission line system (i.e. a defined position along the length of the transmission lines) to which all S-parameter measurements are referenced. This is also the zero phase-offset point in the system in the sense that a short-circuit measured in that plane would give a reflection coefficient of exactly –1. Measured in any other plane the short circuit would be measured to have a phase offset. It is often convenient to place the measurement plane at the mating plane of a pair of connectors, e.g. at the connection *port* (*q.v.*) into a measurement cell. This is not essential, however, and in some measurements it may be better to define the measurement plane to lie on the surface of, or in the centre of, the specimen. This is a conventional term in ANA metrology.

Measurement Port see *Port*.

Medium-Loss. In this Guide, for convenience of description, this term is used typically to apply to materials with a loss tangent lying between 3×10^{-4} and 3×10^{-2}. This is *not* a conventionally recognised definition for medium loss, however.

Metal Loss. Loss of electromagnetic energy into metalwork in a measurement cell. Usually this is conduction loss.

Metrologist. A person who has the task of performing accurate and traceable measurements.

Metrology is the science and craft of accurate and traceable measurement – see Section 4.1 for more details.

Microradian. *Microradian*, abbreviated μrad, is a unit of angle equivalent to 1×10^{-6} of a radian. It is often used to quantify low dielectric loss in terms of loss angle, δ. *(Preferred Term).*

Microwave. In this Guide the term *microwave* typically covers frequencies in the range 1 GHz to 150 GHz (overlapping with *millimetre wave* above 30 GHz). See also Section 2.2.1.

Millimetre-wave or **Millimetre Wave** – In this Guide, typically frequencies in the range 30 GHz to 300 GHz (overlapping with the microwave region above 30 GHz) i.e. frequencies at which the wavelength is typically quantified or quoted in millimetres.

Milliradian. *Milliradian*, abbreviated mrad, is a unit of angle equivalent to 1×10^{-3} of a radian. It is often used to quantify medium dielectric loss in terms of loss angle, δ. *(Preferred Term).*

Multiple-Pass or Multi-Pass Technique. An *E.M.-wave-based* resonance technique. It is so called because the wave is viewed as a multiply-reflected travelling wave in the resonator cell. It passes through the specimen many times, thereby increasing the sensitivity of the measurement for dielectric loss and permittivity. See also *Single-Pass* and *Double-Pass*.

MW is a convenient abbreviation for Microwave (*q.v.*). NB. It is used in this Guide only in the combination RF & MW. MW is *not* used for Megawatt at any point in this Guide so ambiguity is avoided! (*Informal usage*).

NMI = National Measurement Institute. NPL is the NMI for the UK.

Non-Magnetic Material. In this Guide we refer to materials for which *for all practical purposes* μ^* is effectively equal to $(1.0 - j0.0)$ at all of our frequencies of interest as 'non-magnetic materials'. Not a formal term, though widely used (*Informal term*).

Permittivity, ε' or ε_r. As commonly used and as used in this Guide, the term '*Permittivity*' when used alone is equivalent to 'real permittivity', which is equivalent to 'the real part of the complex relative permittivity'. *(Preferred Term).*

Permittivity of Free Space, $\varepsilon_0 = 8.8542\times10^{-12}$ F m^{-1} (farads per metre): the factor which converts *relative* permittivities (dimensionless quantities) into absolute permittivities – see Section 2.1. Also known as the *Electric Constant*.

Plane, in S-parameter and ANA measurements, see *Measurement Plane*.

Pole of a resonance, when viewed on a polar S-parameter chart, see Figure 25, page 73. The point on the resonance-circle trace corresponding to frequencies *very far away* from resonance, either *above* or *below* the resonance frequency. This point is usually diametrically opposite to the resonance *peak* point on the resonance-circle trace.

Port. In *wave measurements* (*q.v.*) and in S-parameter theory (see Section 2.2.2) a *Port* is a point at which travelling-wave signals enter and leave a component, device or (in measurements) a cell. In travelling-wave transmission-line measurements (see, e.g. Sections 7.1.10 and 7.1.11) the ports are typically fitted with coaxial connectors or waveguide flanges. In resonators the ports are typically coupling holes or loops. In free-field measurements (see Section 7.1.14) they are typically antennas. Devices, components and cells are commonly referred to in terms of the number of significant ports they possess, e.g. a reflection cell is a *one-port* cell, and transmission cell is a *two-port* cell, etc.

Q-factor, *Q*. Formally, in the context of dielectric measurements, Q-factor relates the *power stored* in the electromagnetic field of a resonator or a cavity (or indeed any well-defined enclosure) to the *power lost*, via all loss processes at the frequency of concern.

$$Q = 2\pi \times \frac{\text{Energy stored in the resonance}}{\text{Energy lost per cycle}}$$

See Equation 6.3, Section 6.1.9 for further details. Only if the power is stored in a single resonant mode in such a structure (see Section 6.4.1), does the well-known relationship hold between the Q-factor and the resonance width of the Lorentzian 'spectral line' which characterises the resonance - but this relationship is *not* the formal definition of Q-factor. If the *filling-factor* (*q.v.*) of a specimen in a cavity or of a dielectric resonator in its holder is (for all practical purposes) equal to 1.0 then the 'Q-factor' of the specimen may be taken to be numerically equal the reciprocal of the loss tangent of its material. Indeed 'Q-factor' is commonly the term used for the reciprocal of tan δ when dealing with dielectric resonators, but its use in this sense is not to be preferred because there is ambiguity between this expression of an *intrinsic* property of a material (i.e. its tan δ) and the *extrinsic* Q-factor of a circuit or cavity in which it has been measured. By contrast, the practice of associating a Q-factor with each loss process, L_1, L_2, etc., in a resonator, such that $1/Q_{total} = 1/Q_{L1} + 1/Q_{L2}$ … etc., (see Sections 6.1.9 and 6.4.3) is not problematic and is often quite convenient.

Real Permittivity, ε' (sometimes ε_r). As commonly used and as used in this Guide, the term '*Real Permittivity'*, in some cases shortened to just '*Permittivity*', is equivalent to 'the real part of the complex relative permittivity'. *(Preferred Term).*

Relative Permittivity, ε_r. Strictly speaking this is the same as the *complex permittivity*, ε^*, as defined above, but it is often taken to be just its real part, i.e. ε' or ε_r. The relative permittivity of a material should be multiplied by ε_0, the *Permittivity of Free Space* (see entry above and Section 2.1), to give the absolute permittivity in F m^{-1}. In this Guide we use ε' for relative real permittivity and ε^* for complex relative permittivity to prevent the confusion that can arise by the use of the symbol ε_r. (ε_r' and ε_r'' are alternative symbols for ε' and ε'' which are sometimes used in other publications and they too are unambiguous). *(Preferred Term).*

Relaxation Frequency, f_r. Used in parameterised dielectric relaxation models, Section 2.3, (e.g. The Debye Model, Equation 2.16). f_r (Hz) is the parameter that characterises the rate of the relaxation. In many theoretical treatments the *relaxation time*, τ_r, is used instead of f_r. They are related by the equation $f_r = 1/2\pi\tau_r$.

Residual Impedance or Admittance. In an LF measurement system or in a lumped equivalent-circuit model of that system, this is any *unwanted* but *small* impedance (e.g. a resistance, *R*, capacitance, *C,* or inductance, *L*) in the system that can significantly affect, or may potentially affect, the accuracy of the measurement if not taken into account in the *measurement equation* (*q.v.*). See Section 2.2.1.

Resonator (Cell). In dielectric metrology a measurement *cell* (*q.v.*) in which the E.M. fields are designed to resonate so as to increase the sensitivity of the cell for dielectric permittivity and loss measurement (usually the latter). Some resonator cells are called *cavities*. (*q.v.*)

RF – Radiofrequency – In this Guide, typically frequencies in the range 1 MHz to 1 GHz. This is not a uniquely recognised definition for RF, however.

RF & MW = RF and Microwave.

Samples and Specimens, for convenience in this Guide we use the term *sample* to refer to any macroscopic piece of a dielectric material that has been supplied by a *customer* (*q.v.*) for measurement. *Specimens* are, by contrast, the carefully prepared pieces of the material that are actually measured in measurement cells. A number of *specimens*, perhaps of different shapes for use in different measurement cells, may therefore be taken or machined from a single *sample*. This usage is not widely recognised but it does make a useful practical distinction and so can be recommended.

SAR is the *Specific Absorption Rate* of power, measured in watts per kilogram, of electromagnetic radiation into a dielectric medium – most of this power is converted to heat.

Sensitivity Coefficient. The factor that relates a small change in the quantities of ultimate interest in a measurement (e.g. ε' and ε'' for dielectric measurements) to actual measured parameters, e.g. capacitances or electrode displacements, see Sections 4.3.2 and 4.3.3. Sensitivity coefficients are important for computing measurement uncertainties and for optimising measurements. See also *Measurement Equation*.

Single-Pass Technique In *travelling-wave methods* (*q.v.*) a single-pass technique is a *transmission technique* (i.e. one in which transmission coefficients are measured), as opposed to a *reflection technique* (often referred to as a *double-pass technique* (*q.v.*)). '*Single-pass*' refers to the fact that the wave passes through the specimen only once in travelling from the input port to the output port of the measurement cell. See also *multi-pass technique*.

Specimens – see *Samples and Specimens*.

Standing-Wave Method. In contrast to *travelling-wave method (q.v.)*: a resonance method in which the E.M. waves set up a pattern of nodes and antinodes in a resonance measurement cell. See *wave methods*

Static Permittivity, ε_s. As used in parameterised dielectric relaxation models, Section 2.3 (e.g. The Debye Model, Equation 2.16). ε_s gives the highest value that ε' reaches at very low, e.g. sub-Hertz, frequencies *in the parameterised model*. It does *not* usually correspond to the actual permittivity at these frequencies because other loss processes and non-linearities, which are not included in the model, can increase ε' still further.

Structure-Length, *s*, in composite and structured materials, the *structure-length, s,* corresponds to a typical linear dimension of the component particles or structures that make up the material. See Sections 2.3 and 5.3.7. Knowledge of *s* is important because composite dielectrics can spuriously scatter E.M. radiation if the radiation wavelength in any one of its components (particles or structures) is close to *s* for that component. Note that this term is used for convenience in this Guide and is *not* conventional.

tan $\boldsymbol{\delta}$. This is the *symbol* for the loss tangent and a commonly used *term* for the loss tangent. Its use can be accepted because it is not ambiguous. 'tan δ' is the tangent of the loss angle 'δ'. *(Informal term: 'loss tangent' is more formally correct).*

TD, TDR, TDS. *TD* is an abbreviation for *Time Domain* (*q.v.*) as opposed to *Frequency Domain* (FD) (*q.v.*). *TDR* is *Time Domain Reflectometry* and *TDS* is *Time Domain Spectroscopy* or *Spectrometry*. See Section 7.1.19.

Time Domain. We say that we are working in the *Time Domain* if we are measuring parameters such as ε' and ε'' as a function of *Time*. Compare *Frequency Domain (FD)*. Time Domain techniques are discussed in Section 7.1.19 of this Guide. See also Section 2.5.

TLM *Transmission Line Modelling* is a method for modelling electromagnetic fields and electric currents by means of a *discretised* (*q.v.*) computer algorithm. See Section 6.9. See also *FD*, *FDTD*, *FE* and. *FI*.

Travelling-Wave Method. In contrast to *standing-wave method* (*q.v.*). In such a method the signal *propagates* through the measurement system and is measured after transmission through the specimen or reflection from it. See Sections 2.2.2, 7.1.10 and 7.1.11 for guided-wave travelling-wave methods and 7.1.14 for free-field travelling-wave methods – see also *wave methods.*

UKAS – The *United Kingdom Accreditation Service* – this is the organisation which accredits calibration laboratories in the UK. http://www.ukas.com/

Unloaded Q-factor, Q_0. The Q-factor that a resonator would possess if it were not loaded by the presence of the specimen or by the power loss through its coupling ports. See *Q-factor* and *Loaded Q-factor*.

VNA A *Vector Network Analyser* – see ANA.

Wall Loss see *Metal Loss.*

Wave Methods These are measurement methods, usually employed at higher RF and at microwave frequencies, in which the measurement signal is treated as an E.M. wave. Typically they are methods in which the measurement cell is comparable in size to the wavelength of the E.M. radiation. Both travelling-wave and standing-wave (resonant) methods are used in dielectric metrology. The term is often used in contrast to *lumped-circuit* or impedance measurement methods. See Sections 2.2.2 and 2.2.3.

11. Further Information

Regrettably it has not been possible to cover all aspects of RF & MW dielectric metrology in this one guide, but fortunately further information is available elsewhere. The following sources are well worth pursuing for useful metrological advice.

(1) *Published Papers in the Scientific Literature.* It has to be admitted that from a practical metrological point of view they are often far less helpful than one might hope because it is a scientific convention that one skates over many of the details of measurements in such publications - especially difficulties and mistakes! However, they are invaluable for providing the formal *measurement equations* associated with measurement methods and also the design of measurement cells, etc. Keeping up with this literature is a 'must' if one is to stay abreast with the technology!

(2) *Text Books and Earlier Guides to Dielectric Measurement.* Some of these (e.g. [1] and [5]) have become classics. While the electrical measurement equipment they describe may be dated, their treatment of dielectric theory, measurement principles and the design of cells remains valid, very informative and educational.

(3) *Manufacturers' Literature: Catalogues, Manuals and Application Notes.* Of course these are essential when one is actually using the manufacturers' equipment, but a number of manufacturers also provide *Application Notes* (e.g. [26]) and other documents that are well worth reading in their own right. They often provide a valuable insight into the measurements that they describe and provide treatments in much greater depth than can be found in most other publications.

(4) *Other NMIs* (National Measurement Institutes) have similar responsibilities in their own countries to those that NPL has in the UK. A number of NMIs produce detailed *Technical Notes* and *Reports* on measurement techniques which are of great practical value. Notable among NMIs which produce Technical Notes on dielectric measurements is NIST in the USA, see e.g. [83]. Details can often be found on NMI web-sites, e.g. [227] for NIST.

(5) *Measurement Clubs and Experts' Societies.* There are a number of *ad hoc* clubs and societies that currently exist in the UK whose *main purpose* is to advise members on measurement. The most relevant for the topic area of this Guide is the NPL *Electromagnetic Materials Measurements and Applications Club* (*EMMA-Club*) [76]. The NPL *ANAMET Club* (for network analyser users) [228] and the *Piezo-Club* (piezo-electric measurements run jointly with QinetiQ) [229] may also be relevant to readers of this Guide. Many of these clubs publish *Technical Notes*, e.g. [43, 70, 112], and run measurement comparisons to help us to obtain confidence in our work.

(6) *Ad hoc Societies, Clubs and Associations* that cater for the needs of a particular sector of RF & MW industry are also well worth investigating too. Their main focus may not be measurement as such, but it is a topic that regularly comes to the surface in their deliberations. In the UK, two such organizations that are relevant here are the *ARMMS RF & Microwave Society* (covering general microwave topics) [230] and the *Ampere Association* (covering '*Electroheat*', i.e. dielectric heating and microwave processing) [231]. A number of learned societies also run active dielectrics groups, notably in the UK the *Institute of Physics* [232], and groups that cover dielectric-related topics, notably the *Institution of Electrical Engineers* [233].

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