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The cover design depicts the microwave planar near-field scanner being developed at NPL.

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Improving measurement confidence and achieving traceability in dielectric measurements through the use of reference materials.

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1. Introduction

The UK National Measurement System was established in recognition of the positive benefits to British Industry of traceability to national standards. Materials measurement at microwave frequencies is an area in which these advantages have not been able to come fully into play because of the lack of agreed traceability routes to these standards. The major reason for this lack is that many disparate techniques are employed for dielectric measurements. It could be difficult to establish common traceability schemes for all of these methods and counterproductive to attempt to standardise on just some of them. Seen as a microwave measurement problem, materials measurement presents the metrologist with a larger number of parameters to control than, say, measurement of attenuation or reflection coefficient. Microwave components are usually fitted with standard connectors which allow the measurement to be referred to a well-defined plane. Dielectric specimens, by contrast, because of the diversity of materials applications, can come in many forms: sheet, block, tube, liquid, etc. and at microwave frequencies no standard interface to them exists. Nevertheless, a recent survey within the industry shows that there is a perceived need for traceability checking in dielectric measurements. Provision of reference materials is the preferred method for doing this because it can be applied with any technique. The value of such procedures has been recognised internationally by the setting up of a BIPM International Intercomparison of polar dielectric reference liquids, for which NPL is the pilot laboratory.

This paper will look at five aspects of the task of providing reference materials for RF and Microwave dielectric measurements. Much of the paper is concerned with what still remains to be done in this area, but recent work at NPL to meet some of the requirements is also summarised.

2. Use of reference materials

Reference materials may be used in two ways in dielectrics measurements: (1) to check, audit, or provide traceability for a measurement system which is already considered to be calibrated; (2) to calibrate the measurement system. The second is potentially the more exacting requirement because errors in the assumed properties of a reference standard can result in much greater measurement errors. An example of (1) might be scattering coefficient measurements upon a plug of dielectric in a transmission-line (waveguide or coaxial line) which is otherwise filled with air. Provided the specimen has plane faces, such measurements are, in principle, well understood and can easily be performed using an automatic network analyser (ANA). Such measurements apparently have direct traceability via impedance standards, but a typical source of error is the launching of higher order modes in the dielectric specimen. Measurement of a reference material with *known* properties similar to that of the specimen is the most convenient check to ensure that this is not happening and it can be made part of an audit procedure to demonstrate traceability.

Use of reference materials as standards in their own right for measurement-system calibration, (2), is common in the case of coaxial dielectric sensors [6]. A 'known' dielectric, usually a liquid, acts as one of the calibration standards (one of three in ANA reflection measurements). A typical error which may arise here is that if its 'known' permittivity, ϵ^*_{ref}, at any given frequency is in error by, say 3%, a subsequent measurement upon a very different dielectric may be in error by up to four times this amount: errors can effectively be magnified by the calibration process. The difficulty is compounded by the temperature dependence of dielectric properties, which is substantial for

many liquids. It was in recognition of this second use of reference materials that a program of reference liquid measurements was embarked upon at NPL [7,8].

3. Choice of materials

Both solid and liquid reference materials are used. Where measurement systems can accept liquids, their measurement is often more accurate than that of solids. Reference liquids must be used with care to ensure that they do not become contaminated, as is all too easily done with popular reference liquids like ethanol and methanol, because they absorb atmospheric moisture. It is not recommended in general [1] that liquids be used as standards of *low-loss* ($\tan\delta < 0.001$) because it has been found that the loss is not reproduceable from one batch of liquid to another. Low loss liquids like cyclohexane are, perhaps, best regarded as standards of the *real* part of their permittivity, ϵ', only. Stable solid materials provide better standards for such low losses. *High* loss liquids are commonly used for checking or calibrating measurements on aqueous dielectrics (eg. biological tissues, foodstuffs) and have the advantage that suitable reproduceability is available from liquid grades which can be purchased directly from the manufacturers [7].

Solid dielectrics are even more variable in their properties from one batch to another and so their recommended mode of use as reference materials is different from that of liquids. A complete batch of stable material should be obtained and samples should be cut from it such that a number of laboratories can perform an intercomparison. If agreement can be obtained, then one of the laboratories should stockpile the material (checking the stability of its properties from time to time) so as to make samples of it available to users. This procedure was proposed in an EC *Bureau Communautaire de Reference* (BCR) exercise over fifteen years ago [1], but has never been put fully into operation.

Choices of materials for reference purposes must be based upon a wide range of criteria, the most important of which are stability, homogeneity, isotropy, availability and cost. Depending on the eventual application, further criteria can play an important role: linearity, thermal expansion softening point, moisture absorption and hardness have been singled out in a recent comprehensive study of dielectric reference materials [5]. Materials which have actually been used as standards include polyethylene, quartz crystal, alumina, rexolite, ferroflow (a magnetic material). Other materials which are being proposed include fused silica, sapphire crystal, barium titanate and titanium oxide, covering low to high permittivities. Many commonly used materials are unsuitable. PTFE for example has a lattice phase transition near room temperature and so is mechanically unstable. Polymers like polyethylene degrade if exposed to sunlight, and can be non-calculably anisotopic if stress is 'frozen' in when they are moulded. The dielectric loss of ceramics such as alumina can increase significantly if they are touched because of the effects of metallic impurities from sweat. It is clear that any comprehensive programme of reference material usage must include consideration of correct handling and storage conditions.

All of the above considerations apply to measurements at near-ambient temperatures. There is considerable interest at present in ceramic measurements at elevated temperatures (eg. up to 600°C). In the absence of agreed standards, improved confidence in such measurements can probably best be achieved through programmes of intercomparisons between laboratories.

4. Presentation and maintenance of data on reference materials

A persisting problem with dielectric reference materials is the lack of any formal agreement upon their actual dielectric properties. A researcher who wishes to use, say, methanol as a reference will turn to the scientific literature. But a thorough literature search will reveal discrepancies of $\pm 3\%$ in even the low frequency permittivity. Tabulated data from reputable laboratories [3, 10-14] are not always easily accessible and in some cases dates date back to measurements performed over 40 years ago [13] which can possibly be improved upon by more up-to-date techniques.

Furthermore, reliable data are not available for all of the materials one might wish to use. The usual practice of providing succinct summaries of measurements in the scientific literature fails to fulfil the requirement of the serious user of reference materials. Parametric fits of measured data to dielectric relaxation models, such as the Debye or Cole-Cole relaxations, produced by different laboratories are not strictly commensurable if they are obtained over different frequency ranges [7, 8]. Finally, any attempt to use the stated uncertainties of different workers to choose between their published figures are again undermined by the succinctness of uncertainty statements in published papers. They are further undermined by the fact that discrepancies in published permittivities produced by different workers are sometimes found to exceed their combined stated uncertainties.

A way around all of these problems has been proposed [11]. Workers who wish their data to be used as part of a database of reproduceable reference materials should publish the individual measured complex permittivities at each frequency and temperature, together with realistic uncertainties, rather than a parameterised fit to a dielectric relaxation model. This comprehensive measurement data can then be combined with similar data from other workers in the correct statistical manner. A laboratory which is maintaining the reference material database should be able to publish suitable models from time to time. Numerical models are more convenient to use than tables of data, they but should be updated whenever significant new data becomes available. This procedure is to be used in the forthcoming BIPM intercomparison.

5. Measurement Intercomparisons

If any evidence were required of the need for some form of traceability in dielectric measurements, one need only consult the results of dielectric measurement intercomparisons. Whilst agreement upon the real part of the permittivity, ϵ', is often good enough to provide confidence, agreement upon loss in low-loss intercomparisons can be very poor. Discrepancies of factors of 8 in $\tan\delta$ have been reported [2]. Such intercomparisons are the best way of showing where improvements are required, and with certain techniques, are often the only way of providing confidence in measurements. Whilst ANA-based scattering parameter measurements arguably have direct traceability to impedance standards, this is not the case with much purpose-built instrumentation. For example, many resonance techniques are not clearly traceable and so can benefit from intercomparisons.

In establishing a database of information upon reference materials, intercomparisons between laboratories using substantially different measurement techniques are the best method for bringing to light, and hopefully reducing, systematic errors. Where agreement can be achieved, such an intercomparison at an international level is also the best way of ensuring acceptance of, and confidence in, the database.

6. Measurements upon Reference Materials

The need for the provision of data upon lossy reference liquids (eg. n-alcohols), was recognised some years ago in the NPL dielectrics measurements programme, chiefly because of their common use in calibrating and checking coaxial sensor measurements [4,7,8]. A limited exercise of high-loss polar-liquid measurements was performed up to 3 GHz, but there is a need for further work to cover higher frequencies and a wider range of temperatures. Some of the requirements can be addressed by the on-going programme of work at NPL, particularly the following through of the BIPM intercomparison. Considerable experience was gained in the earlier BCR reference liquid intercomparison [1], during which dependable values for the complex permittivity of deionised water were obtained [10,11]. Specimens of solid reference materials were also prepared at that time (1978) which are still in storage, and from which valuable information can be obtained on the stability of the materials concerned.

7. Conclusion

Considerable advantages can accrue to the microwave materials industry from the use of RF and microwave dielectric reference materials, but their use must be properly co-ordinated on both a national and international scale to gain maximum benefits.

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