

Low loss dielectric measurements in the frequency range 1 to 70 MHz by using a Vector Network Analyser.

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Abstract. The resonance of a coil inductor and a parallel-electrode capacitor can be used to determine the permittivity and loss angle of sheets of material at RF frequencies. By using high-stability instrumentation to measure resonant frequency and Q-factor, the loss angle of very low-loss materials can be determined with a resolution of below 10 microradians. In the 1970s much work went into developing Q-meter-based systems, based on series resonance, which were typically used for measuring the loss angle of polymers such as polyethylene. These systems were also used for measuring ceramics used as RF windows in prototype fusion reactors. This paper describes how one of these systems was updated to replace the now obsolete Q-meter with a Vector Network Analyser (VNA), using parallel resonance. A second aspect to the work is calculation of the fringing (edge) capacitance between the electrodes, which are unguarded, by using MIT FastCap software. This enables measurements obtained by the equivalent-thickness method to be corrected for the effects of fringing capacitance. Measurements using an unmodified Q-meter system, a VNA-based system, and two other techniques are compared for polyethylene, fused silica, macor and alumina specimens. Good agreement is obtained. The measurements are presented with comprehensive evaluations of uncertainty.

Keywords: Dielectric permittivity, dielectric loss, measurement, uncertainty, RF windows, Hartshorn and Ward method, fringing capacitance, edge capacitance, FastCap.

1 Introduction

There is a long history of development of radio frequency (RF) techniques for measurement of the dielectric loss of very low loss materials (loss angle $\lesssim 100 \mu\text{rad}$) [1, 2, 3, 4]. Historically, the laboratories of polyethylene manufacturers and national post offices needed such data for evaluation of long-distance-cable communication systems [4, 5]. Applications include measurement of the loss angle of materials used as RF windows in accelerators and fusion reactors [6, 7, 8, 9, 10, 11, 12, 13]. This is an important parameter as temperature rises from absorbed energy at the the extreme power levels used (which can exceed 1 MW) can cause such windows to fracture. Suitable materials for RF windows include very low-loss ceramics and synthetic diamond. Ceramics that have very low loss are also used in narrowband filters [14] and as substrates for electronic devices [15]. Measurements on low-loss materials at GHz frequencies are typically performed in cavities, which are the subject of numerous publications [16, 17, 18]. At lower frequencies (especially below 300 MHz), the large size of cavities is a practical limitation and alternative methods are needed.

Pioneering work to develop a method for measuring permittivity and loss angle of laminar specimens at MHz frequencies, based on the resonance of an LC circuit, was performed by Hartshorn and Ward (H&W) at NPL in the 1930s [19, 20]. The H&W method [21] is in many respects similar to admittance-cell techniques [22] that use LCR meters or impedance bridges, but by the use of resonance the resolution for loss angle is improved [1]. In broad terms the resolution is approximately $100 \mu\text{rad}$ with an admittance cell, but $< 10 \mu\text{rad}$ with a modern H&W system. In the 1970s and 1980s, ERA Technology Ltd produced H&W systems [1, 2, 13] based on analogue Q-meters. These have a frequency range of 1 MHz to 70 MHz. Kakimoto and his co-workers [23] extended the method to 200 MHz. Specimens are typically discs or rectangles that are larger than electrodes, but developments described by Heidinger [13] enable disc- and ring-shaped specimens that have smaller diameter than electrodes to be measured. This can improve measurement uncertainty significantly for materials that have $\epsilon' \gtrsim 10$. Since the 1990s, there have been few publications on similar methods for measuring permittivity and loss angle by using resonant LC circuits.

In the H&W method specimens are measured in a ‘dielectric test-set’: a capacitance cell that has disc-shaped electrodes and a high-precision micrometer to set the gap between them. A second micrometer (the ‘side micrometer’) allows adjustment of a coaxial capacitor. The dielectric test-set [1, 2] is engineered for high mechanical and thermal stability. It was originally integrated with a Hewlett-Packard 4342A Q-meter [24] and a coil (one of a set) that is chosen for the required resonant frequency. In the work described in this paper, an ERA system was updated by replacing the now-obsolete Q-meter by a modern Vector Network Analyser (VNA). The Q-factor and resonant frequency are determined from measured S-parameters [25]. A change from series to parallel resonance has been made as this eliminates the need for the

low-impedance drive circuit (semiconductor amplifiers and step-down transformer) that is built into the Q-meter. The effects of stray capacitances and lead inductances associated with the LC circuit will differ for the two configurations. These include self-capacitance of the coil which, in the parallel configuration, shunts the dielectric test-set. Comparisons of dielectric measurements give important insight into how the resolution and precision of measurements is affected by these changes.

Microwave cavity resonant systems typically have Q-factors of several thousand or more (depending on the design of the cavity and the frequency). A high Q-factor is often seen as necessary for measurement of the loss of low loss materials. This is not always the case; the LC resonators described in this paper typically have Q-factors in the range 200 to 400 at MHz frequencies. By using measurement systems that have high stability and low noise, very small changes (of order 0.01) in Q-factor can be detected. The loss of very low loss materials can therefore be measured, even using analogue Q-meters.

Section 2 of this paper describes the ERA system and the changes needed to allow operation with a VNA instead of an analogue Q-meter. In Section 3 the principles of measurement of relative permittivity (ϵ') and loss angle (δ) by the equivalent-thickness method [18, 26] are discussed. Section 4 presents measurements with the new system at MHz frequencies. Section 5 is a comparison of measurements on materials that have δ in the approximate range 1 μ rad to 3 mrad that are made with an updated (VNA-based) instrument, an unmodified (Q-meter) instrument, and by three other techniques. Conclusions are presented in Section 6.

1.1 Loss angle definitions

The dielectric loss of materials is normally represented by the loss tangent, $\tan\delta$. For low loss materials it is convenient to write $\delta \approx \tan\delta$. The loss angle, δ , is often expressed in milliradians (mrad) or microradians (μ rad). Low loss typically indicates $\delta < 3$ mrad, and very low loss typically indicates $\delta < 100$ μ rad. For materials that are used in microwave resonators and filters, it is common to refer to Q_f , which is given by $Q_f = 1/\delta$. For further discussion of these topics see reference [18].

2 Hartshorn and Ward Systems

2.1 Dielectric test-set

The dielectric test-set (Figure 1) is of the two terminal type [18] as it uses unguarded electrodes (gold-flashed cylinders 50 $\varnothing \times 10$ mm length). It is designed for measuring rectangular specimens (70 $\times$ 54 mm) with typical thickness 2 mm. A precision large-barrel micrometer (resolution 0.0002 mm) is used to set the main capacitor C_1 . The RF

electrode is held in position by insulators (not shown) that are made from fused silica, which is chosen because of its low expansion coefficient. The coaxial capacitor C_2 is adjustable over a small range (approximately ± 1.2 pF) by means of the side micrometer, which is normally set in its centre position. The capacitance changes almost linearly with translation (limited by the bore uniformity). Electrical connections are made via a pair of robust coaxial connectors (General Radio GR874).

2.2 Test-set calibration by using an LCR meter

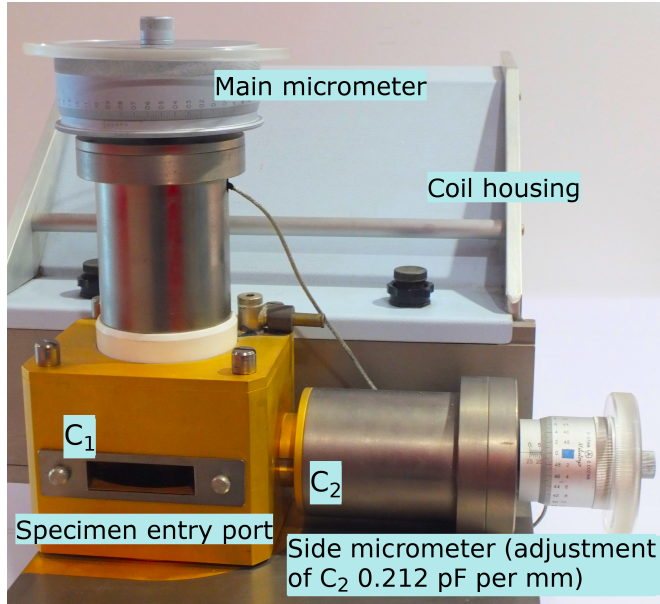
Measurement of loss angle requires the coaxial capacitor, C_2 , to be calibrated (Section 3.2). This can be achieved by detaching the test-set from the baseplate and connecting it to an LCR meter (Hewlett-Packard 4285A). The LCR meter is connected via a four-terminal-pair using four cables [18, 27] and an adaptor plate with GR874 connector. Readings were shown to be accurate to within 0.03 pF by using calibrated four-terminal reference capacitors (10 pF and 100 pF). The calibration factor (σ) of the side micrometer was determined as $\sigma = 0.212(2)$ pF/mm, uncertainty evaluated for coverage factor $k=2$. The LCR meter was used for additional tests and calibration measurements, which will be described in Sections 3.3.2 and 4.1. All of these measurements were made at 1 MHz.

2.3 Coils

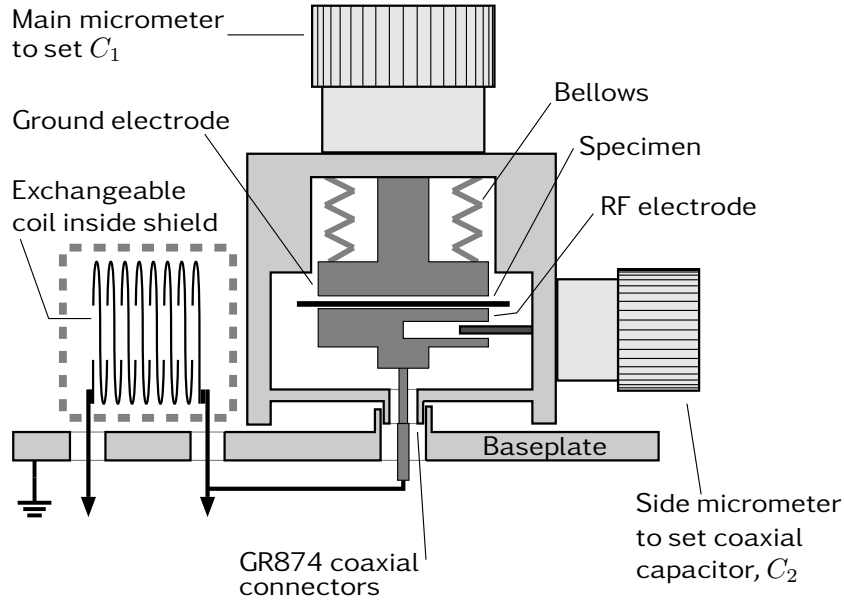
Exchangeable coils (Table 1 and Figure 2) allow the resonant frequency to be chosen in the nominal range 1 MHz to 70 MHz. The actual resonant frequency at which measurements are made varies ($\pm 20\%$) with the specimen permittivity, thickness, and the setting of the air gap between the specimen and the ground electrode. The 6 MHz and 33 MHz coils are encased in expanded polystyrene to improve thermal stability. The 6 MHz, 33 MHz and 70 MHz coils were fashioned from flattened copper pipe.

Table 1: Coils

Nominal frequency	Q	Comment
1 MHz	240	Hewlett-Packard Q-meter coil, type 16479A
6 MHz	440	Multi-turn copper loop
33 MHz	370	Copper loop
70 MHz	160	Copper loop



(a) Photograph



(b) Schematic

Figure 1: Mechanical parts of the Hartshorn and Ward system developed by ERA Technology Ltd [1, 2]. The electrode assembly, consisting of capacitors C_1 and C_2 , is referred to as the dielectric test-set. The main micrometer, which moves the ground electrode of capacitor C_1 , has resolution 0.0002 mm. A coaxial capacitor, C_2 , can be adjusted by means of the side micrometer. This has a resolution of 0.001 mm.

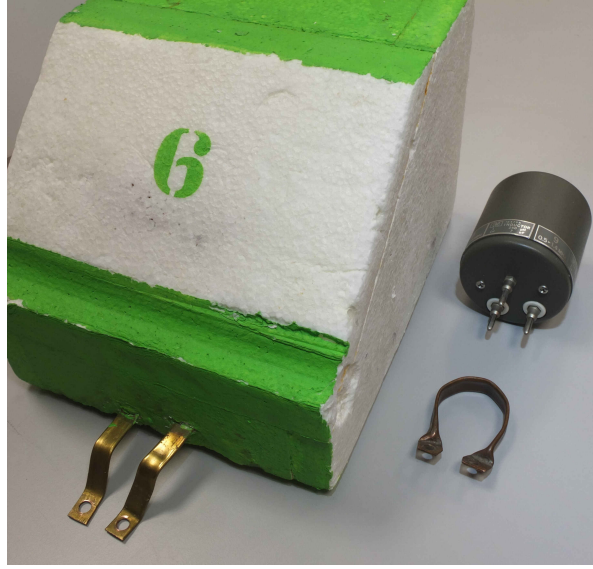


Figure 2: Exchangeable coils: Hewlett Packard 1 MHz coil (top right), 6 MHz coil (encased in expanded polystyrene), and 70 MHz single-turn coil.

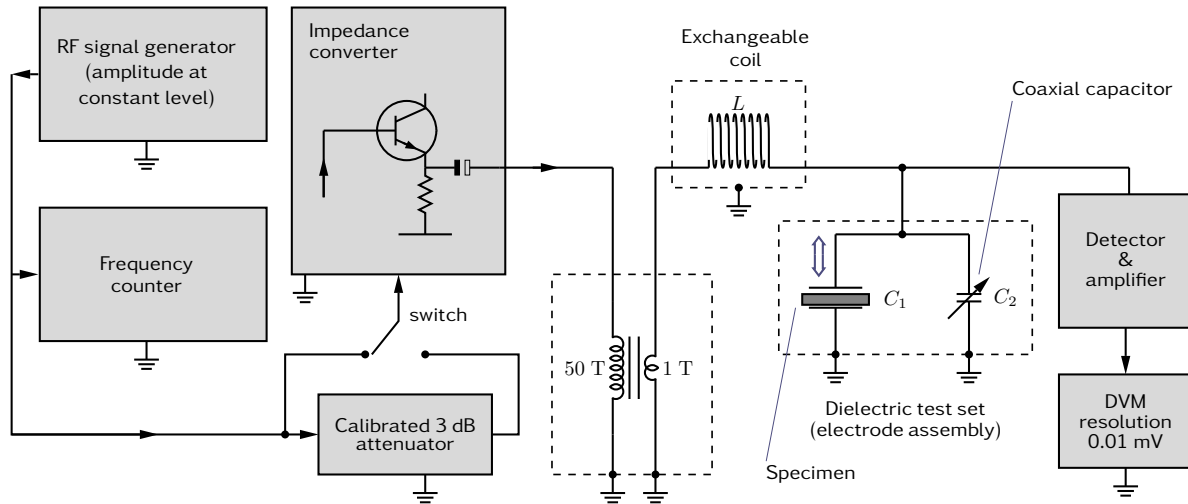


Figure 3: Simplified schematic of the series resonant system based on a modified Hewlett-Packard 4342A Q-meter.

2.4 Series-resonant H&W system based on an analogue Q-meter

The series resonant H&W system (Figure 3) uses the RF signal source and detection system of a Hewlett-Packard 4342A Q-meter. The method is summarised here; see Hill [2] and Heidinger [13] for more detailed descriptions. Readings of source frequency and detector output voltage are made with modern digital instruments. To minimise loading of the resonant circuit, the Q-meter uses an impedance converter and a step-down transformer to obtain a low source impedance ($30\text{ m}\Omega$). Measurements are made

by the equivalent-thickness method (Section 3.1), using the DVM to measure the indicated Q-factor. By using an attenuator, it was shown that the linearity of detector and amplifier was sufficient to allow accurate measurement of the small changes in indicated Q-factor that occur when a specimen is inserted or removed. Measurement of the 3 dB points of the resonance obtained without a specimen is also required to establish a calibration for Q-factor measurement. These are found by re-tuning using the calibrated capacitor, C_2 , while the signal frequency is constant [2]. A calibrated switched attenuator has been added to enable them to be made at the same power level [18, 25]; enabling traceable measurement without the need to calibrate the detection system.

2.5 Parallel resonant H&W system based on a VNA

One of the available H&W systems was modified to use a VNA instead of a Q-meter. This section explains the required design changes and how the VNA is used; the calculation and measurement procedure is described elsewhere (Section 3). The step-down transformer (Figure 1) was not easily separable from the Q-meter, and is also a likely cause of uncertainty. It was therefore decided to use a transformerless design. The omission of the transformer made it necessary to use a parallel resonance configuration (Figure 4) on account of the comparatively-high impedance of the VNA ports ($50\ \Omega$). The facility to change the resonant frequency by replacing the coil was retained. For the 1 MHz coil, coupling was via fixed-value 3.9 pF capacitors. For the other coils, coupling was via adjustable capacitors with fixed and moving plates that are isolated from ground. These are adjusted for each coil as the required coupling level varies. Connections to the VNA were made via coaxial cables. The parallel resonant system contains no sources of heat, which improves its thermal stability.

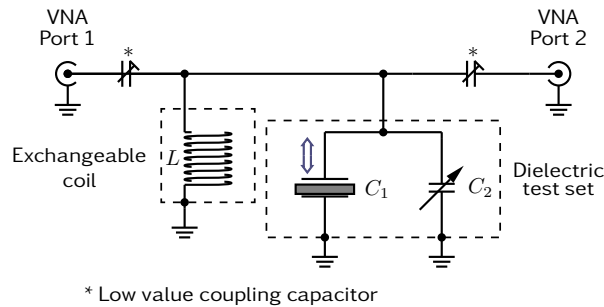


Figure 4: Schematic of the parallel resonant system.

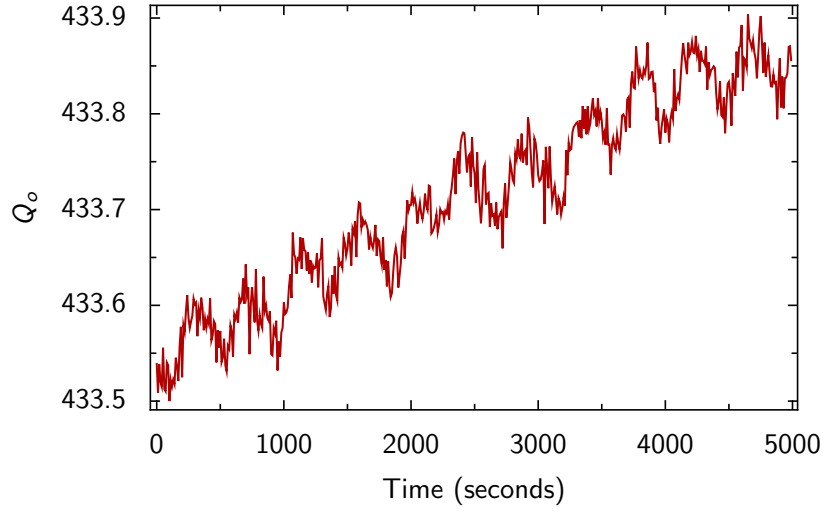
2.5.1 Q-factor measurement by using the VNA Q-factor and resonant frequency are determined by fitting a model to swept-frequency measurements of the S_{21} Q-circle in the complex plane [25]. The model gives a very accurate description of the observed resonances. Leakage (i.e. any signal that bypasses the resonator) is accounted for by a fitted complex term. The coupling capacitors are adjusted to give weak coupling (transmission loss in the range 30 dB to 40 dB). Provided that the coupling factors at the two ports are similar, unloaded Q-factors can be determined by following simple procedures [25]. Comparison of $|S_{11}|$ and $|S_{22}|$ traces on the VNA screen allows the coupling factors to be adjusted until they are approximately equal. The VNA is used uncalibrated, but a normalisation factor based on a measurement of the $|S_{21}|$ of a ‘thru’ connection is used in the calculation of unloaded Q-factor. The VNA (Hewlett-Packard 8753E) was used with the following settings: source power 3 dBm, IF bandwidth 30 Hz, 101 points in frequency sweep, averaging factor 4. The repeatability of Q-factor measurements is found to be optimum at the 3 dBm power setting for this VNA [25]. Measured Q-factors (Figure 5) are subject to random noise and temperature drift. These determine the minimum resolvable loss angle. The Q-factor is very sensitive to small changes in temperature, so cyclical variations that are associated with the laboratory temperature-control are observed. The standard deviation of measurements of unloaded Q-factor is 0.04 for the 6 MHz and 33 MHz coils. For the 1 MHz and 70 MHz coils, the standard deviation of measurements of unloaded Q-factor is 0.02. Non-repeatability and drift of the measured resonant frequency are not significant.

3 Theory

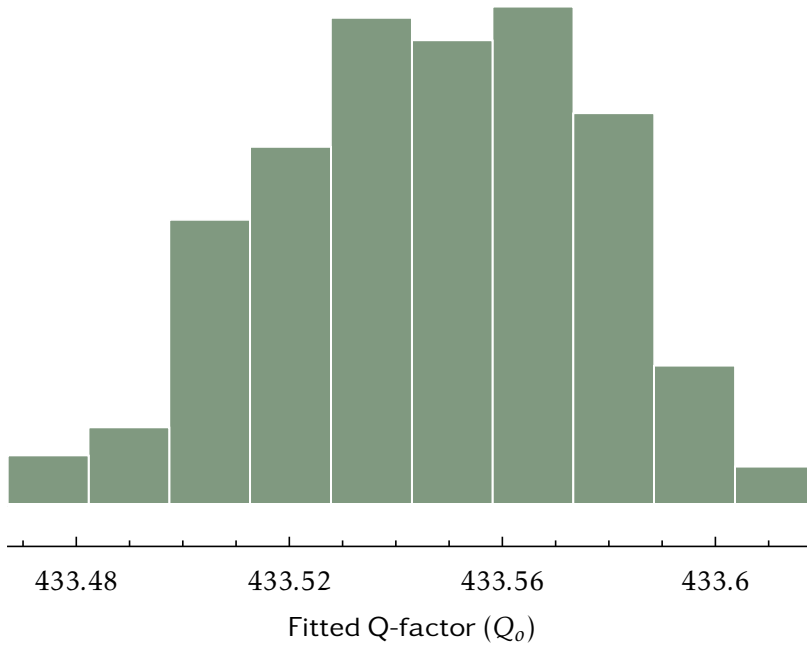
3.1 The equivalent-thickness method for determination of permittivity and loss angle

The equivalent-thickness method can be used for determining the relative permittivity and the loss angle of planar specimens from measurements of the admittance of a parallel electrode cell. Lynch, in his original paper on this technique [26], used an impedance bridge for making the measurements. The measurement procedure [18] is as follows: The capacitance is measured with the specimen *in situ* with a small air gap (t_a) above the specimen – see Figure 6. Then the specimen is removed and the separation between the electrodes is reduced (by turning the micrometer) to restore the measured capacitance. Leaving a small gap above specimens reduces error caused by mechanical imperfections such as micrometer backlash, and lack of flatness of the electrodes and specimen. If only geometric capacitance is considered (i.e. if fringing capacitance at the edge of the electrodes is neglected), the permittivity ϵ'_{app} is obtained from the micrometer readings (M_{in} and M_{out} respectively) from eqn. (1):

$$\epsilon'_{app} = \frac{t_s}{M_{out} - M_{in} + t_s} \quad (1)$$



(a) Drift



(b) Histogram (linear drift subtracted)

Figure 5: Q-factor repeatability measurements (6 MHz coil).

in which t_s is the specimen thickness. It is convenient to refer to the quantity given by this simple formula as the *apparent* permittivity, ϵ'_{app} , because it is not corrected for the effects of fringing capacitance. The equivalent-thickness method allows ϵ'_{app} to be determined when the electrodes are in a circuit that includes a parallel capacitor (or stray capacitance) that has unknown but constant value.

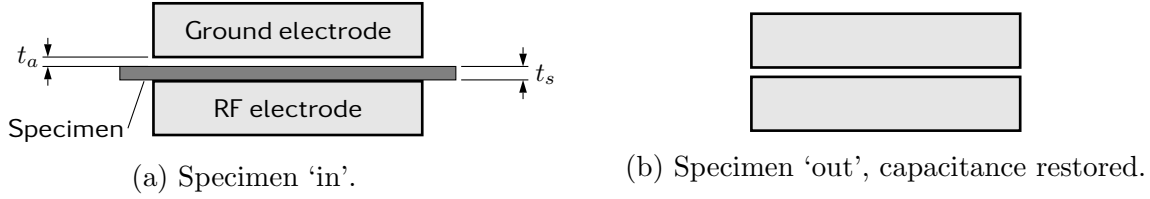


Figure 6: The equivalent-thickness method.

For measurement by the H&W technique the same procedure is used, except that resonant frequency is measured (and restored) instead of indicated capacitance. The dielectric test-set shown in Figure 1 is typical in that the RF electrode is fixed in position (only the ground electrode moves), so the stray capacitance changes negligibly between the 'in' and 'out' measurements.

The change in the measured dissipation factor [28] that occurs when the specimen is removed provides a direct measurement of the loss angle when t_a is zero. When $t_a > 0$ a correction factor for 'dilution' of the loss is needed [26]. For the isolated electrodes of Figure 6, equation 2 is obtained:

$$\delta = (D_{in} - D_{out}) \times \frac{M_{out} - \Delta}{M_{out} - M_{in} + t_s} \quad (2)$$

where D_{in} and D_{out} are the dissipation factors, and Δ is the zero error of the micrometer (defined as the reading at which the electrodes nominally touch).

For the H&W experiment, $D_{in} = 1/Q_{in}$ and $D_{out} = 1/Q_{out}$ where Q_{in} and Q_{out} are the unloaded Q-factors with and without the specimen. It is not necessary to establish the frequency dependence of losses in the system (e.g. in the coil) as the two measurements are performed at the same frequency.

3.2 Modifications to the calculation of loss angle to account for shunt capacitance

In the H&W experiment, the loss in the circuit is reduced because C_1 is shunted by parallel capacitance that enables a portion of the resonant current to bypass the specimen. For series resonance, only C_2 and some small stray capacitance (e.g. in the GR874 connectors) shunt C_1 . For parallel resonance, C_1 is also shunted by the self-capacitance of the coil that is used. Modifying equation 2 to include another 'dilution' factor (K) allows a correction to be made:

$$\delta = (D_{in} - D_{out}) \times K \times \frac{M_{out} - \Delta}{M_{out} - M_{in} + t_s} \quad (3)$$

where

$$K = \frac{C_{tot}}{C_1^{in}} = \frac{C_{tot}}{C_1^{out}} \quad (4)$$

in which C_{tot} is the total capacitance ($C_1 +$ shunt capacitance). The ‘in’ and ‘out’ measurements of C_1 are identified with superscripts. The test-set is designed to ensure that the shunt capacitance does not vary significantly with the electrode separation.

For the updated (parallel resonant) system, to account for ‘dilution’ requires K to be determined by an additional procedure. This uses the coaxial capacitor to make incremental adjustments of the total capacitance by a small quantity, C_{inc} . Measurements of resonant frequency enable C_{tot} to be calculated by using the resonance equation

$$\omega^2 = \frac{1}{L \times (C_{tot} + C_{inc})}. \quad (5)$$

In practice, readings at several positions of the side micrometer are made, allowing an averaged value for the C_{tot} to be obtained. The calibration factor of the coaxial capacitor σ (pF/mm) is determined by using the LCR meter (Section 2.2). The calculation of K also requires C_1^{out} , which is given by,

$$C_1^{out} = \frac{A\epsilon_o}{g} + C_f, \quad (6)$$

where A is the geometric area of the electrodes, the inter-electrode gap $g = M_{out} - \Delta$ and C_f is the fringing capacitance for the ‘out’ measurement. Previously-reported corrections for ‘dilution’ of the loss for series resonant systems [2, 13] omitted the C_f term as a means of calculating it was not available at that time. A polynomial expression presented in this paper (equation 7) now enables C_f to be included. This amendment is applied to all of the H&W measurements of δ that are presented in this paper. The measurements of δ for HDPE and alumina are reduced by 12 % and 6 % respectively.

3.3 Fringing capacitance corrections

For the unguarded electrodes of the dielectric test-set (capacitor C_1 in Figure 1), fringing capacitance (C_f) has significant effect on measurements by the equivalent thickness method. C_f depends on the gap between the electrodes, and for ‘in’ measurements, on the permittivity and thickness of specimens. For the ‘out’ measurement (air spacing) C_f is 5 % of the total inter-electrode capacitance for a 0.3 mm gap, increasing to 12 % for a 1 mm gap. Calculations of C_f with computer software (Section 3.3.1) can be used to improve the precision of measurements. Different approaches are required for measurement of ϵ' and δ :

For measurement of ϵ' . Variations in C_f between ‘in’ and ‘out’ measurements are used to determine a correction (Section 3.3.3) to the apparent permittivity given by equation 1. The effect of the micrometer zero-error, Δ , on this correction is small.

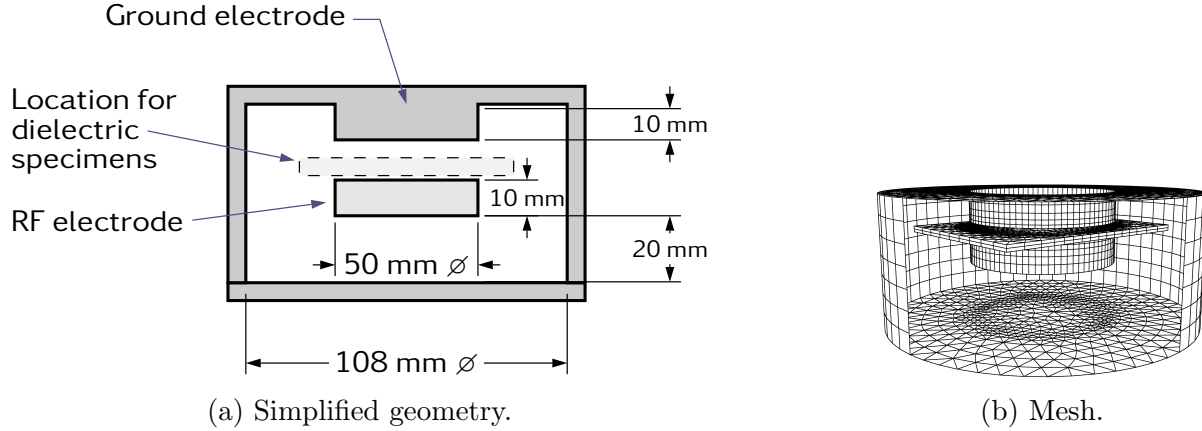
For measurement of δ . The value of C_f for the ‘out’ measurement is used in the calculation of the ‘dilution’ caused by shunt capacitance (Section 3.2) and in determinations of Δ by electrical methods (Section 4.1). The specimen is assumed to be large enough to ensure that fringing fields at its perimeter are negligible. Fringing fields diminish rapidly as a function of distance from the electrode edges, so using specimens that are 54 mm width (for 50 $\varnothing$ mm electrodes) is sufficient to ensure that this is the case.

Hartshorn and Ward [19] used the Kirchoff formula [29] to estimate the C_f of air-spaced unshielded electrodes. Estimates of C_f with and without a dielectric specimen were obtained by Lynch [30]. Appendix B of the report [31] presents comparisons of results from various sources for unshielded disc electrodes, and enclosed geometries such as coaxial capacitors. These help to establish the accuracy of calculations of C_f with MIT *FastCap* software [32] that are used in this paper.

3.3.1 Calculation of C_f by using FastCap To enable solution with *FastCap*, the shielded geometry of the test-set is simplified (Figure 7). *FastCap*, which uses the Boundary-Element Method [32], requires the surfaces of dielectric boundaries and conductors to be meshed. This was achieved by using *netgen* [33]. C_f is obtained by subtracting the geometric capacitance (calculated between parallel regions assuming a uniform charge distribution) from the total capacitance calculated by using *FastCap*. Residual fringing-capacitance between the outer shield and the underside of the RF electrode is neglected. A number of computational experiments were carried out to optimise the mesh for best accuracy. The calculated C_f for the ‘out’ measurement (which has no boundaries between dielectrics) is generally more accurate than that for the ‘in’ measurement. The report [31] describes these calculations in more detail.

Figure 8 shows *FastCap* calculations of C_f as a function of the gap g between the electrodes for the ‘out’ measurement. For convenience, they are fitted to a polynomial,

$$\begin{aligned}
 C_f \times g &= a_0 + a_1 \times g + a_2 \times g^2 + a_3 \times g^3 \\
 &\quad + a_4 \times g^4 + a_5 \times g^5 + a_6 \times g^6 + a_7 \times g^7 \\
 C_f &\equiv C_f(g).
 \end{aligned} \tag{7}$$

Figure 7: *FastCap* model for the Hartshorn and Ward test-set.

3.3.2 Measurement of C_f by using the LCR meter The *FastCap* calculations of fringing capacitance for air-spaced electrodes were compared to measurements on the dielectric test-set made with the LCR meter. Indicated capacitance C_{LCR} is related to the gap between the electrodes by

$$C_{\text{LCR}} = \frac{A\epsilon_o}{g} + C_f^{\text{ex}} + C_p$$

$$C_f^{\text{ex}} \equiv C_f^{\text{ex}}(g) \quad (8)$$

where C_p is the sum of C_2 and the stray capacitance, and C_f^{ex} is the value of the fringing capacitance to be determined by the experiment. C_f^{ex} and C_p cannot be determined independently from measurements with the LCR meter, but as C_p is constant the variation of C_f^{ex} with g can be established. The value for C_p was chosen so that C_f^{ex} matches the *FastCap* value (C_f) at a convenient point. Good agreement between C_f and the variation in C_f^{ex} is obtained (Figure 8). For $g \lesssim 0.4$ mm, C_f^{ex} is not very reproducible because it is extremely sensitive to the micrometer setting. It is evident, however, that C_f^{ex} exceeds C_f for $g \lesssim 0.2$ mm. The most likely reasons for this are that the electrodes are not exactly parallel, and that the accuracy of *FastCap* calculations for the smallest gaps is limited by the mesh size.

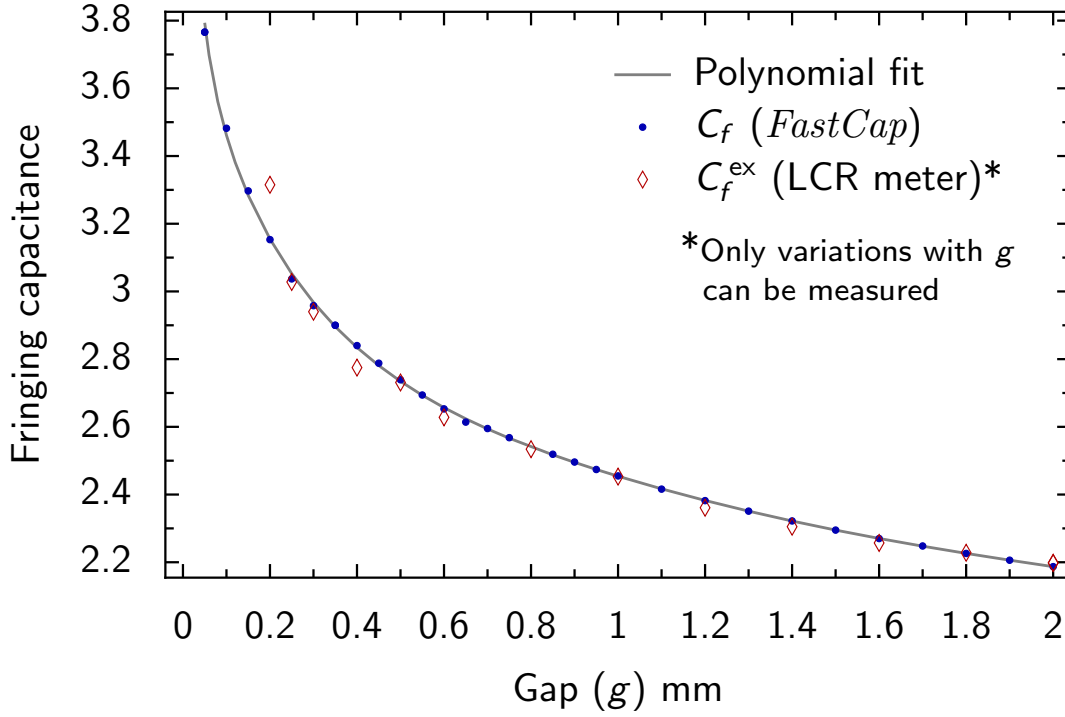


Figure 8: Dependence of fringing capacitance on the gap between air-spaced electrodes for the geometry shown in Figure 7a.

3.3.3 Permittivity corrections A sheet of dielectric material placed between and contacting the electrodes causes the geometric capacitance to increase in proportion to ϵ' , but *FastCap* models show that C_f increases by a greater factor. For measurements by the equivalent-thickness method, a lower value of M_{out} is therefore required to match the resonant frequency of the ‘in’ measurement, and the measured ϵ' is increased. Calculated corrections [31] as a function of the air gap t_a (defined in Figure 6a) are shown in Figure 9 for a specimen that has dimensions $70 \times 54 \times 2$ mm. It is observed that the correction increases with both ϵ' and t_a . Using a small value for t_a is clearly advantageous for high-permittivity specimens; measurements are normally made with $0.1 \text{ mm} < t_a < 0.2 \text{ mm}$.

Several candidate correction formulas were fitted to the data shown in Figure 9. It was found that a corrected value, ϵ' , can be calculated from the apparent (measured) value ϵ'_{app} with sufficient accuracy from

$$\begin{aligned}
 \epsilon'_f &= 0.014 t_a (\epsilon'_{app})^2 + 0.016 \epsilon'_{app} - 0.02 \\
 \epsilon' &= \epsilon'_{app} - \epsilon'_f \\
 &\text{for } t_a \lesssim 0.2 \text{ mm and } t_s \approx 2 \text{ mm.}
 \end{aligned} \tag{9}$$

Similar equations can be obtained for specimens that have other thicknesses.

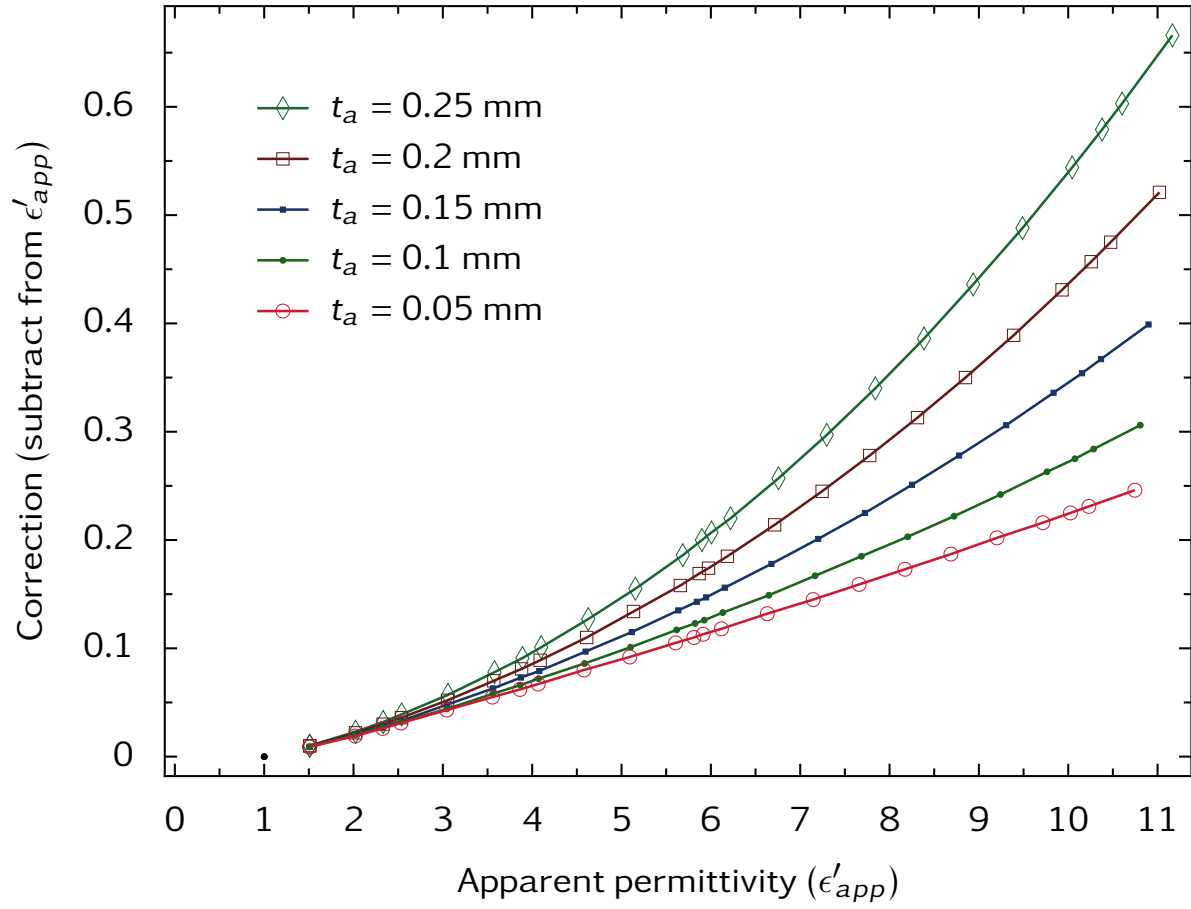


Figure 9: Permittivity corrections for measurements by the equivalent-thickness method for a rectangular specimen $70 \times 54 \times 2$ mm. Calculated for the geometry shown in Figure 7.

4 Measurements on specimens with the updated H&W system

4.1 Determination of Δ

Measurement of loss angle requires Δ (the zero-error of the main micrometer) to be measured – see equation 3. It can be found by three methods:

Mechanical measurement: By using a gauge block inserted through the entry port (Figure 1a). A sheet of fused silica with flat and parallel faces was used for this purpose. The flatness and parallelism of the electrodes was estimated from micrometer readings at which contact between electrodes and the gauge block was obtained.

Capacitance measurement: By measuring capacitance as a function of gap by using the LCR meter. The calculation takes account of the variation of fringing capacitance with gap (equation 7). Δ and stray capacitance for the LCR-meter measurement

are obtained by fitting. The uncertainty in Δ was evaluated from estimates of the the uncertainty associated with capacitance and micrometer data. These are respectively 0.6 % and 0.0004 mm (quoted as expanded uncertainties at coverage factor $k=2$).

Capacitance interchange: By interchanging capacitance between the main capacitor, C_1 , and the calibrated coaxial capacitor, C_2 , while maintaining resonant frequency. Δ is obtained from the micrometer readings by solving a quadratic equation. The calculation takes account of the variation of fringing capacitance with gap (equation 7).

Table 2 shows measurements of effective capacitance by the three methods. These are averaged to obtain an estimate for Δ for the effect of lack of parallelism of electrodes. The uncertainty of the averaged Δ is chosen conservatively as the models used take no account of electrode parallelism.

Table 2: Determinations of Δ by the three methods.

Method	Δ (mm)	
Gauge block	-0.023	(0.012)
Capacitance measurement	-0.022	(0.004)
Capacitance interchange	-0.016	(0.003)
Average	-0.02	(0.02)

Bracketed values are the expanded measurement uncertainty at coverage factor $k=2$

4.2 An experimental test of the FastCap model: Measurements of ϵ'_{app} as a function of t_a

Measurements on a high permittivity specimen (alumina) were made with the parallel resonant H&W system to test the accuracy of the *FastCap* model for the effects of fringing capacitance (Section 3.3). A Split-Post Dielectric Resonator (SPDR) [34] was used to measure the permittivity of the specimen at 2450 MHz ($\epsilon'=9.68(3)$, uncertainty at coverage factor $k=2$). As this material has very low loss, the permittivity at the lower frequencies used by the H&W experiment will differ negligibly [18] from this value.

Figure 10 shows measured and predicted values of the apparent permittivity of the alumina specimen as a function of the air gap, t_a . Rises in both sets of values occur as t_a is increased because fringing capacitance is not accounted for. The apparent permittivity values are consistently higher than the actual permittivity for all values of t_a including zero (by extrapolation). The uncertainty corridor for modelled results is obtained by

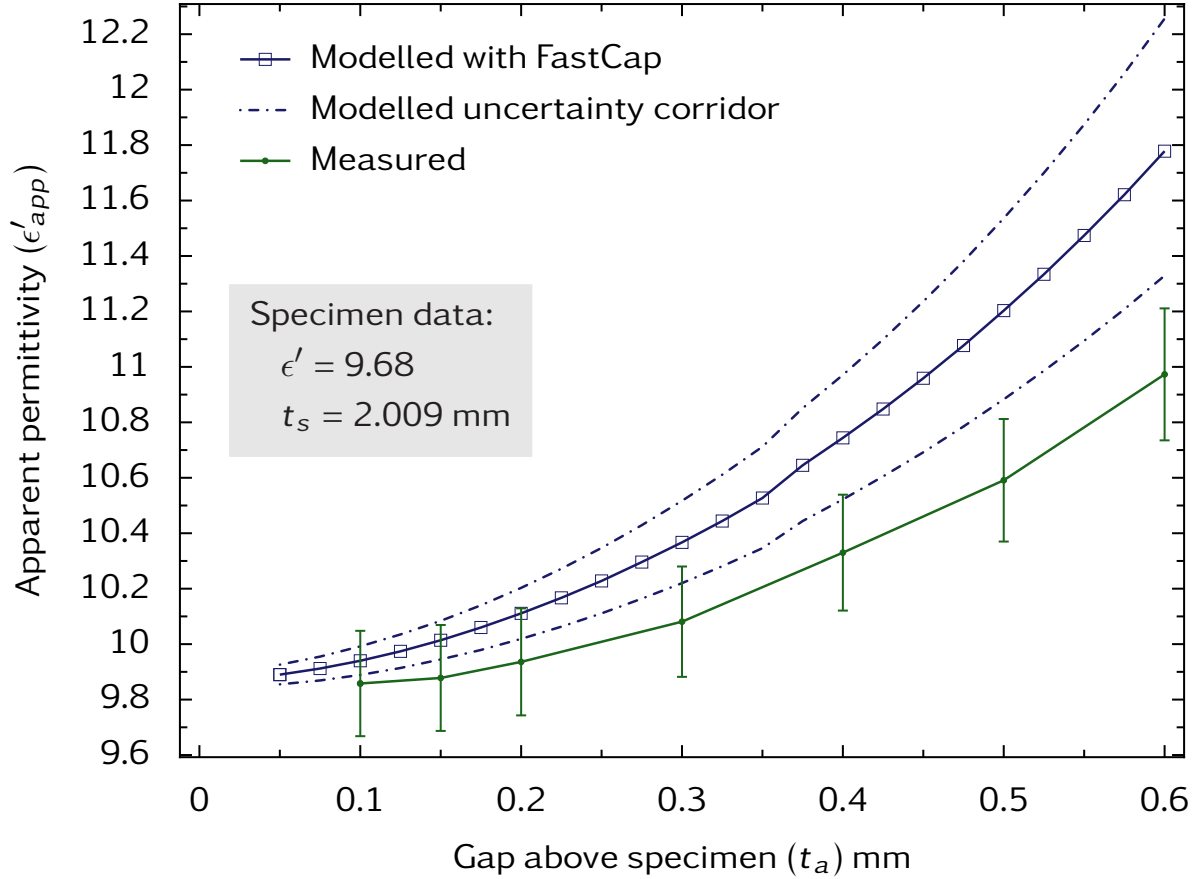


Figure 10: A comparison between measurements of ϵ'_{app} by the equivalent-thickness method as a function of t_a , and predictions obtained by using a *FastCap* model. The measurements were made on an alumina specimen with the updated H&W system. The uncertainty corridor of the modelled results is obtained by using a representative uncertainty for *FastCap* calculations of capacitance. Measurement uncertainties are shown at coverage factor $k=2$.

using a representative uncertainty for *FastCap* calculations of capacitance [31]. When $t_a < 0.2$ mm (normally used for measurement) good agreement between modelled and measured ϵ' is obtained. For $t_a = 0.15$ mm the predicted ϵ' exceeds the actual permittivity by approximately 0.3 (this can also be seen in Figure 9). The measured and predicted values for $t_a > 0.2$ mm diverge significantly. This can be attributed to the absence of several features of the actual experiment from the simplified geometry, Figure 7. These include the entry port for specimens, the moveable ground-electrode and bellows, and the fused-silica supports for the RF electrode. The separation of the plotted curves allows the uncertainty of corrections for fringing capacitance to be estimated.

4.3 Measurements of ϵ' and δ of HDPE as a function of t_a

Measurements of ϵ' and δ for a specimen of High Density Polyethylene (HDPE) were made at several values of t_a . The data have been corrected for the effects of fringing capacitance, as described in Section 3.3. The updated H&W system with the 6 MHz coil was used for these measurements. The thickness of the specimen was 2.008 mm. The discrepancies between the measurements (Table 3) are smaller than the estimated uncertainties. Additional measurements on this specimen are given in Table 5.

Table 3: HDPE measurements as function of the air gap t_a (defined in Figure 6a) between the specimen and the ground electrode.

t_a (mm)	ϵ'	δ (μ rad)
0.05	2.36 (0.01)	72 (4)
0.1	2.36 (0.01)	73 (4)
0.15	2.36 (0.01)	72 (4)
0.2	2.35 (0.01)	72 (4)
0.25	2.35 (0.01)	73 (5)

Bracketed values are the expanded measurement uncertainty at coverage factor $k=2$

4.4 Uncertainty evaluation

Table 4 shows the uncertainty contributions for a measurement on HDPE made with the updated H&W system. The uncertainty of measured Q-factors must be separated into its Type A and Type B components. The Type A component can be evaluated from observations of noise and drift (see Figure 5), but the Type B component is more difficult to evaluate (see the discussion in [25]). Correlation of the Type B uncertainty components of Q_{in} and Q_{out} has to be accounted for to obtain realistic evaluations of uncertainty of the loss angle. The covariance is not known but, as $Q_{in} \approx Q_{out}$, the corresponding uncertainty component of the loss angle can be evaluated as

$$u(\delta)_q = u_B \times K \times \frac{M_{out} - \Delta}{M_{out} - M_{in} + t_s} \quad (10)$$

where

$$u_B = |(1/(Q_{in} + q) - 1/(Q_{out} + q) - (1/Q_{in} - 1/Q_{out}))| \quad (11)$$

in which the q is the type B component of the standard uncertainty of Q_{out} . For the well-shaped resonances of the H&W system q is evaluated as $0.5\% \times Q_{out} + 1$. Evaluations

of uncertainty of the loss angle of very low loss materials that are presented in Section 5 show that realistic evaluations of $u(\delta)_q$ are obtained on this basis.

The fringing capacitance correction, ϵ'_f , is not exact, as Figure 10 shows. A standard uncertainty contribution 20 % of ϵ'_f is assumed for $t_a \lesssim 0.2$ mm. Equation 9 was used to obtain ϵ'_f for the measured specimens, which were all approximately 2-mm thick.

Table 4: Standard uncertainty contributions for a measurement on HPDE at 6 MHz. Normal distributions are assumed.

Measurand	Value	Std. unc.	Type	$u(\epsilon')$	$u(\delta)$ in μrad
Q_{out}	461.10	0.04	A	N/A	1.0
Q_{in}	457.68	0.04	A	N/A	1.0
$\ddagger Q$		3.0	B	N/A	1.2
t_s in mm	2.008	0.004	A	0.007	0.5
M_{in} in mm	2.1571	0.0002	A	0.0006	0.02
M_{out} in mm	0.9890	0.0002	A	0.0006	0.01
Δ in mm	-0.02	0.01	B	N/A	1.7
σ in pF/mm	0.212	0.001	B	N/A	0.3
ϵ'_f	$\dagger 0.03$	$0.2 \times \text{value}$	B	0.006	N/A

$\ddagger$ Common to Q_{out} and Q_{in} . Calculated by using equation 10 for $q=3.0$

$\dagger$ Fringing capacitance correction calculated by using equation 9 for $t_a=0.15$ mm.

5 Measurement comparisons

Four specimens were chosen for measurement: HDPE (ICI Rigidex 2000), fused silica (Heraeus Spectrosil 2000), Macor (Corning), and alumina. The measurements of relative permittivity (ϵ') and loss angle (δ) are shown in Tables 5 to 8. By measuring the same actual specimens by all four methods, the effects of variability between batches (which could apply to all of these materials) are avoided. The faces of specimens were machined precisely to ensure that they were flat and parallel. The specimens were handled only with gloves, as finger grease causes a measurable increase in the loss of very low loss materials. The temperature of the laboratory is maintained at 21°C. The Relative Humidity (RH) has limited regulation and is normally in the range 30 % to 45 %.

SPDR [34] and a guarded-electrode admittance-cell [18, 22] methods were used to obtain reference data for each specimen. The fused silica specimen was also measured at 36 GHz by using an open resonator [35].

The measurements with the admittance cell (Hewlett-Packard 16451B) were made by the equivalent-thickness method (Section 3.1). To improve accuracy, the micrometer supplied with the HP16451B has been replaced by one that has a higher specification. The use of a guarded-electrode practically eliminates the effect of fringing capacitance on measurements provided that they are made with a comparatively-large gap above the specimen ($t_a \geq 0.6$ mm for the HP16451B). The required measurements of capacitance and dissipation were obtained by using an LCR meter (Hewlett-Packard 4285A). A four-terminal-pair [18, 27] configuration is used to ensure that impedances and stray capacitance associated with cables have negligible effect on measurements.

Table 5: HDPE (ICI Rigidex 2000), thickness 2.008 (0.008) mm.

Method	Frequency (MHz)	ϵ'		δ (μ rad)	
SPDR	2450	2.359	(0.009)	160	(80)
HP16451B admittance cell	6	2.36	(0.02)	100	(65)
Unmodified H&W system and Q-meter	6	2.35	(0.02)	79	(6)
Updated H&W system and VNA	1	2.36	(0.01)	75	(5)
	7	2.36	(0.01)	73	(4)
	34	2.36	(0.01)	77	(5)
	84	2.36	(0.01)	82	(8)

The H&W measurements used the four coils listed in Table 1. The actual resonant frequencies are shown.

Bracketed values are the expanded measurement uncertainty at coverage factor $k=2$

Table 6: Fused silica (Heraeus Spectrosil 2000), thickness 2.097 (0.004) mm.

Method	Frequency (MHz)	ϵ'		δ (μ rad)	
Open resonator	36000	3.82	(0.01)	250	(40)
SPDR	2450	3.81	(0.01)	<60	(60)
HP16451B admittance cell	6	3.83	(0.03)	<60	(60)
Unmodified H&W system and Q-meter	6	3.79	(0.02)	<4	(4)
Updated H&W system and VNA	1	3.81	(0.02)	<2	(2)
	6	3.81	(0.02)	<2	(2)
	32	3.81	(0.02)	<2	(2)
	77	3.81	(0.02)	<6	(6)

Table 7: Macor (Corning), thickness 2.008 (0.002) mm.

Method	Frequency (MHz)	ϵ'		δ (μ rad)	
SPDR	2450	5.72	(0.02)	5270	(90)
HP16451B admittance cell	6	5.82	(0.05)	2630	(110)
Unmodified H&W system and Q-meter	6	5.74	(0.06)	2610	(220)
Updated H&W system and VNA	1	5.82	(0.06)	2470	(230)
	6	5.79	(0.04)	2520	(230)
	30	5.78	(0.04)	2740	(250)
	71	5.78	(0.04)	3030	(300)

Table 8: Alumina, thickness 2.009 (0.004) mm.

Method	Frequency (MHz)	ϵ'		δ (μ rad)	
SPDR	2450	9.68	(0.03)	<50	(50)
HP16451B admittance cell	6	9.7	(0.2)	<100	(100)
Unmodified H&W system and Q-meter	6	9.4	(0.1)	18	(6)
Updated H&W system and VNA	1	9.6	(0.2)	17	(3)
	6	9.5	(0.2)	14	(2)
	27	9.6	(0.2)	15	(3)
	64	9.6	(0.2)	13	(7)

Measurements were made by the equivalent-thickness method in unmodified (Q-meter) and updated (VNA) H&W systems. The data have been corrected for the effects of fringing capacitance, as described in Section 3.3. A small air gap ($t_a=0.15$ mm) was used to minimise the size of permittivity corrections and therefore ensure the best accuracy. The same dielectric test-set (shown in Figure 1a) was used for both sets of measurements. These measurements were made in the four possible orientations for a rectangular specimen and averaged. The observed variations in ϵ' and δ are small compared to the overall measurement uncertainty.

Measurements with both H&W systems and the admittance cell were made at 6 MHz to allow results to be compared directly at this frequency. Additional measurements with the updated H&W system were made with the 1 MHz, 33 MHz and 70 MHz coils.

Significant outliers in measured Q-factors (and therefore in loss angle) for the updated H&W system occasionally occur. Where it was considered to be justifiable, such outliers were discarded. The updated system offers greater stability than the unmodified system and, in consequence, uncertainties of measured δ are smaller.

5.1 Interpretation of ϵ' results in Tables 5 to 8

The ϵ' measurements by H&W and admittance-cell techniques are very consistent, with the exception that measurements made with the unmodified H&W system are marginally lower than those obtained with the other techniques. The cause of this discrepancy, which is statistically significant for alumina, is unknown.

Rushton [36] reports that the series inductance of the bellows attached to the ground electrode (Figure 1) changes as it is extended. Measurements by the H&W method could in consequence show an apparent increase in ϵ' with frequency. This is not observed in the measured frequency range, even for alumina, the specimen with the highest ϵ' .

Comparison of ϵ' measurements at very different frequencies requires consideration of the material loss. The Lynch formula [18] can be used to make an estimate of how ϵ' changes as a function of frequency when δ is known. For the low-loss materials (HDPE, fused silica and alumina) ϵ' should change negligibly in the measured frequency ranges (up to 36 GHz in the case of fused silica). For macor, a medium-loss material, ϵ' is forecast to be approximately 0.1 greater at 6 MHz than it is for the SPDR measurement at 2450 MHz. This is confirmed by the measurements shown in Table 7.

5.2 Interpretation of δ results in Tables 5 to 8

Measurements reported by Baker-Jarvis *et al* [37] show that, in the microwave frequency range, δ for fused silica rises approximately in proportion to frequency. Whether proportionality is maintained down to MHz frequencies for Spectrosil 2000 is not established, but Mollá and Ibarra [38] note that for some grades of Heraeus fused silica, δ is close to 3 μ rad in the range 1 kHz to 100 MHz. On the assumption that δ is proportional to frequency, the open resonator measurement at 36 GHz (Table 6) can be used to calculate δ at MHz frequencies by extrapolation. The loss angle of fused silica is found to be extremely low ($\delta < 1 \mu$ rad) at frequencies ≤ 70 MHz. The H&W data in Table 6 is consistent with this prediction. Moreover, the evaluated uncertainties provide sufficient coverage. This is an important finding, as it provides evidence that the dissipation in the metal walls of the dielectric test-set is not changed significantly when the main micrometer is adjusted to restore the resonant frequency (Section 3.1).

The HDPE is from a reference stock held at NPL that has been measured by various techniques. The increase in δ with frequency (Table 5) is consistent with previous observations. Measurements of δ for alumina, a very low loss material, were in good agreement (Table 8), although below the resolution of admittance cell and SPDR methods.

Macor δ data obtained by several techniques is presented by Pells *et al* [39]. Two of these are based on resonant LC circuits: Another H&W system from ERA Technology Ltd that is based on a Q-meter (as described in Section 2), and a system designed by Kakimoto *et al* [40] that uses a guard ring to reduce fringing-capacitance. The δ results given in the present paper (Table 7) are lower than those given by Pells (reproduced in Table 9). Measurements with Pells' ERA system are expected to be increased by approximately 10 %, because the fringing capacitance term in equation 6 was almost certainly omitted as no value was available at the time. The presence of more significant outliers at ≥ 47 MHz suggest that the Q-meter becomes less accurate at the upper frequencies; perhaps a consequence of using a coupling transformer. Non-reproducibility of δ between macor batches has not been evaluated, and could also be a factor in the observed differences. Pells finds that δ has a U-shaped dependence on frequency, with a minimum at a few MHz. The range of the results given in Table 7 is not broad enough to make this clear.

Table 9: Macor δ data from Pells *et al.* [39]. The coverage factor of uncertainties is not specified.

Method	Frequency (MHz)	δ (μrad)	
ERA system	1	3150	(270)
similar to	10	2800	(110)
that shown in	30	3310	(120)
Figures 1	47	3900	(240)
and 3.	54	4220	(450)
Coil/capacitor	1	2900	(200)
resonator by	3	2700	(200)
using half-power	10	3000	(200)
gap variation	30	2900	(200)
method [40].	100	3300	(200)

6 Conclusion

The Hartshorn and Ward method, based on an LC resonator, can be used to measure the permittivity and loss angle of very low loss materials. The technique requires measurements of Q-factor with and without specimens to be made at the same resonant frequency by tuning the resonator. This avoids error in measured loss angle caused by the frequency-dependence of the conductivity of metal conductors. An H&W system that was originally based on an analogue Q-meter has been updated to use a Vector Network Analyser to improve the accuracy and ease of measurements of Q-factor and resonant frequency. The series-resonant circuit of the original system was changed to a parallel-resonant circuit to enable transformerless coupling. Eliminating the transformer is advantageous, as it is a source of uncertainty that is not easily evaluated. The total capacitance is determined by the settings of the two capacitors of the dielectric test-set, and by stray capacitance (which in the parallel configuration includes self-capacitance of the coil). An experimental procedure is used to determine the total capacitance as it is required for calculation of the loss angle. Stray capacitance increases the smallest resolvable loss angle, and should therefore be minimised as far as possible.

Fringing capacitance between the cylindrical electrodes of the H&W systems increases the overall capacitance and has a significant effect upon measurements of permittivity. The fringing capacitance was calculated by using MIT *FastCap* software to enable it to be corrected for. These corrections are based on an idealised geometry and are not exact, but by comparison with experimental data, uncertainty associated with them has been evaluated. The computational techniques used can be applied to other measurement systems that use unguarded electrodes.

Uncertainties associated with dimensional and Q-factor data, and fringing capacitance corrections, have been evaluated to enable traceable measurement of ϵ' and δ . Measurements obtained with unmodified and updated H&W systems, an admittance cell, a SPDR, and an open resonator have been compared. Consistent measurements and evaluations of uncertainty are obtained. It was demonstrated that unmodified and updated H&W systems both enable loss angle to be determined with a resolution of better than 10 μ rad in the range 1 MHz to 70 MHz. The resolution achieved is, for both of these instruments, governed by the ability to measure very small Q-factor changes. This is determined by random noise and thermal instability.

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