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Dielectric measurement: error analysis and assessment of uncertainty

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Abstract

The advantages and limitations of using partial differential analysis to assess the methodological uncertainty associated with the measurement of the dielectric properties of a material are discussed and an alternative pragmatic approach is proposed. It relies on repeat measurements of the dielectric properties of reference liquids and an analysis to estimate random and systematic uncertainties. Examples of measurement uncertainty are provided for well-defined monomolecular materials and for less homogeneous materials at microwave frequencies. All examples relate to measurement with an open-ended coaxial probe but the methodology is not specific to this technique. Examination of the components of uncertainty in the dielectric properties of biological tissue shows that, where the system is free of methodological bias, random fluctuations originating from sampling and natural inhomogeneity dominate the uncertainty budget. In such cases, the mean value of the measured parameter and the standard error of the mean can be taken as a good measure of the true value and its associated uncertainty.

1. Introduction

As with most physical quantities, the measurement of the dielectric properties of tissues is inherently simple in concept but relatively complex and varied in implementation. The choice of experimental procedure depends mostly, but not exclusively, on the frequency and temperature of interest, the nature of the sample and the purpose of the measurement. There are broadly two main approaches to the determination of the measurement uncertainty often described as error analysis and assessment, respectively. Error analysis informs on the effect on the measurement uncertainty of various elements of the experimental design including the theoretical derivation of the dielectric properties from the measured parameters. It can lead to the determination of theoretical bounds on the experimentally determined dielectric properties

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provided that the tolerance of the main contributors to the uncertainty and the limitation of the procedure are known. In practice, error analysis is often used to study the interdependence of the various parameters and the limits of applicability of the procedure in question. The assessment of the measurement uncertainty is often carried out using comparative measures that provide means for the evaluation of random and systemic errors and apply generally to experimental data irrespective of the specific methodologies used in their derivation.

In this paper we describe our approach to the determination of the measurement uncertainties illustrating our arguments with examples of measurements made with open-ended coaxial probes and vector network analysers (VNA).

The use of open-ended coaxial probes for dielectric measurement has been practised for nearly three decades; it remains the method of choice for the characterization of biological materials. The literature on the subject is quite extensive; by way of example we mention some references to aspects of the theoretical implementation, whereby an expression (model) is obtained relating the admittance of the probe–sample interface to the dielectric properties of the sample (Marcuvitz, 1951, Galejs 1969, Hodgetts 1989, Nevels *et al* 1985, Jenkins *et al* 1992), some early application to the characterization of biological materials (Burdette *et al* 1980, Stuchly and Stuchly 1980, Gabriel *et al* 1986) and applications to the measurement of lossy liquids (Wei and Sridhar 1991, Peyman and Gabriel 2006). Open-ended probes are fairly broadband, the optimal probe size is related to the frequency range and could adequately cover two frequency decades. They were used with instrumentation operating in the time domain (Gabriel *et al* 1986, 1987) but, today, are most commonly used with VNAs running essentially in the frequency domain but offering some time domain capabilities. VNAs have residual errors minimized using known impedance standards and appropriate error models. When used for dielectric measurements, the calibration is an integral part of the procedure and impacts on the limitations and uncertainty of the technique. A one-port calibration using three standards is required prior to using open-ended probes.

One way of achieving this is to use engineering calibration standards such as open and short circuits and a broadband load prior to connecting the probe. On connecting the probe, the phase is adjusted to account for its electrical length. This could be followed by transforming the response into the time domain to view and gate out the reflection of the probe connector before transferring back to the frequency domain. Authors who use this approach proceed to normalize the measured admittance using the response of standard materials. For example, Misra *et al* (1990) used a linear fractional transformation relating measured reflection coefficients and calculated admittances. Stuchly *et al* (1994), Anderson *et al* (1994) and Popovic and Okoniewski (2002) normalize their system by deriving the probe-related parameters in the admittance model from the response to standard materials. The parameters are calculated once and used for subsequent measurements. This renders the accuracy of the procedure particularly sensitive to the dimensional tolerance and geometric imperfections of the probe (Popovic and Okoniewski 2002).

The most commonly used calibration procedure is to introduce the standards at the open end of the probe. More often than not, the responses used for calibration are those of the probe in air, short circuited and when immersed in a standard liquid, a procedure that concurrently calibrates and normalizes the response.

There are numerous publications dealing with error analysis for this procedure, of which Anderson *et al* (1994) and Wei and Sridhar (1991) derived expressions relating the error in the dielectric properties resulting from errors in the measured parameters. Baker Jarvis *et al* (1994) analysed calibration and measurement errors and investigated the effect of bad contact between the probe and the sample. Many authors, including Nyshadham *et al* (1992) and Hagl *et al* (2003) have assessed the effect of the uncertainty in reference liquids as they apply to their methodologies.

Our approach to the assessment of uncertainty is pragmatic, relying on repeat measurements of standards to estimate random and systematic errors. We provide examples of measurement uncertainty for well-defined monomolecular materials and for the less homogeneous biological materials at microwave frequencies. Although all the examples relate measurement with an open-ended coaxial probe, the methodology is not specific to this technique.

2. Error analysis: a numerical approach

The general expression for the complex admittance is

$$Y = G + \mathrm{j}B, \tag{1}$$

where the components G and B are the conductance and susceptance terms respectively and j is $\sqrt{-1}$. In the case of a coaxial probe in contact with a semi-infinite, isotropic, homogeneous sample, assuming TEM propagation, the conductance and susceptance terms can be written as series expansions that are functions of the complex permittivity $\varepsilon = \varepsilon' - \mathrm{j}\varepsilon''$, the frequency and the probe dimensions and impedance (Misra *et al* 1990, Wei and Sridhar 1991, Gabriel *et al* 1994).

The complex derivatives $\mathrm{d}G/\mathrm{d}\varepsilon$ and $\mathrm{d}B/\mathrm{d}\varepsilon$ can then be calculated and combined to provide

$$\frac{\mathrm{d}Y}{\mathrm{d}\varepsilon} = \frac{\mathrm{d}G}{\mathrm{d}\varepsilon} + j\frac{\mathrm{d}B}{\mathrm{d}\varepsilon} \tag{2}$$

as the derivatives of complex functions can be expressed in terms of the derivatives of their simpler, component functions.

It is useful to obtain the derivatives with respect to the reflection coefficient or S_{11} scattering parameter, the parameter obtained from fundamental quantities in network analysers. This can be done through the relationship $S_{11} = (1 - YZ_0)/(1 + YZ_0)$ where Z_0 is the characteristic impedance of the probe

$$\frac{\mathrm{d}\varepsilon}{\mathrm{d}S_{11}} = \frac{\mathrm{d}Y}{\mathrm{d}S_{11}}\bigg/\frac{\mathrm{d}Y}{\mathrm{d}\varepsilon}. \tag{3}$$

The relative error in ε can be calculated if the residual instrumental errors in the magnitude $\Delta|S_{11}|$ and phase $\Delta\theta$ of S_{11} can be estimated. The partial derivative with respect to either the magnitude or the phase is obtained from

$$\frac{\delta\varepsilon}{\delta|S_{11}|} = \frac{\mathrm{d}\varepsilon}{\mathrm{d}S_{11}}\frac{S_{11}}{|S_{11}|} \qquad \frac{\delta\varepsilon}{\delta\theta} = \frac{\mathrm{d}\varepsilon}{\mathrm{d}S_{11}}\mathrm{j}S_{11} \tag{4}$$

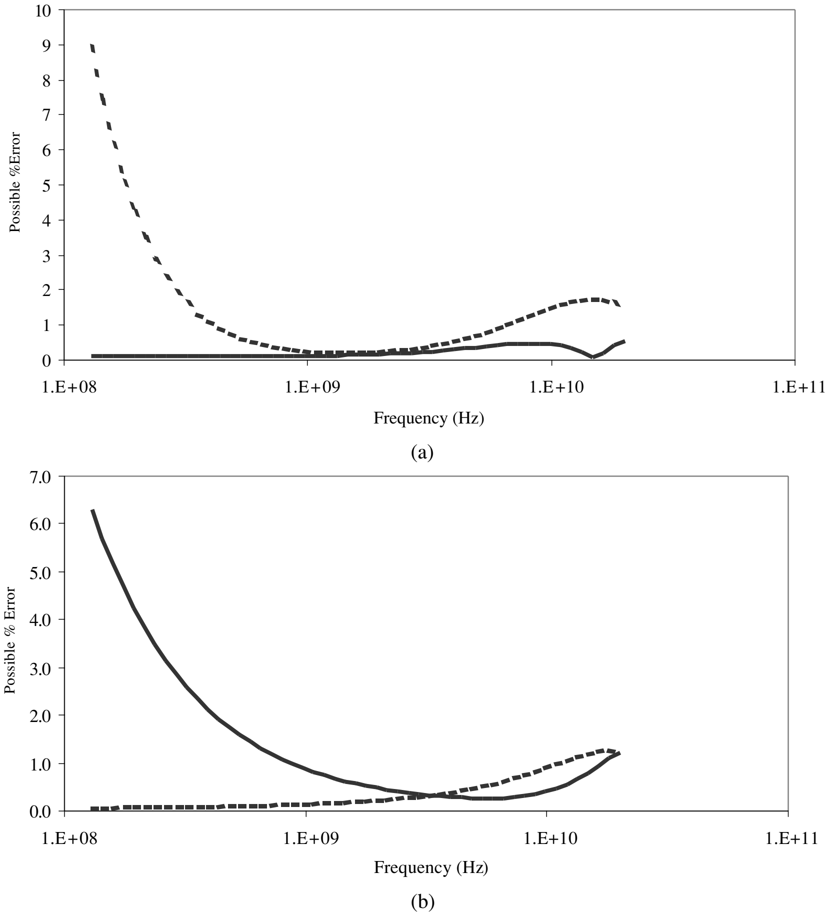
furthermore

$$\frac{\delta\varepsilon}{\delta|S_{11}|} = \frac{\delta\varepsilon'}{\delta|S_{11}|} - \mathrm{j}\frac{\delta\varepsilon''}{\delta|S_{11}|} \qquad \frac{\delta\varepsilon}{\delta\theta} = \frac{\delta\varepsilon'}{\delta\theta} - \mathrm{j}\frac{\delta\varepsilon''}{\delta\theta}. \tag{5}$$

From (4) above, $|S_{11}|\delta\varepsilon/\delta|S_{11}| = -\mathrm{j}\delta\varepsilon/\delta\theta$—in accordance with the Cauchy Riemann condition—consequently, there are in fact only two independent terms.

One can calculate the uncertainty in either ε' or ε'' due to any residual uncertainty in the reflection coefficient from

$$\frac{\Delta\varepsilon}{\varepsilon} = \frac{1}{\varepsilon}\left[\frac{\delta\varepsilon}{\delta|S_{11}|}\Delta|S_{11}| + \frac{\delta\varepsilon}{\delta\theta}\Delta\theta\right]. \tag{6}$$



(a)

(b)

Figure 1. Percentage error in ε' (solid line) and ε'' (dashed line) for (a) water and (b) a 2 M NaCl solution at 20 °C using a 2.98 mm diameter, PTFE filled probe, assuming a 0.1% uncertainty in the magnitude and phase of the reflection coefficient at all frequencies. Note that the ionic conductivity of 2 M NaCl solution is 13.3 S m^{-1}, much higher than naturally occurring electrolytes; by comparison, the conductivity of seawater is of the order of 3 S m^{-1} and that of physiological saline (equivalent to a 0.15 M solution) is 1.4 S m^{-1} at the same temperature.

There are endless permutations of probe dimensions, frequencies and material properties that one can input to provide informative snapshots of the measurement uncertainty under specific conditions. It is practically impossible to make a unique statement on optimal measurement conditions but these are useful tools to test the suitability of the situation at hand. To illustrate the usefulness and limitation of this approach we calculated the percentage error in ε' and ε'' for water and 2 M NaCl solution, at 20 °C and a 2.98 mm diameter probe, assuming a 0.1% uncertainty in the magnitude and phase of the reflection coefficient (figure 1(a) and (b)). In figure 1(a), at frequencies below 300 MHz, the uncertainty in ε'', in percentage terms, is relatively high due to a combination of related factors: (i) the measurement is more sensitive to ε' than ε'' and (ii) the numerical value of ε'' is very low. By contrast, for 2 M NaCl the situation is reversed, the uncertainty in ε', below 300 MHz, is relatively high, that in ε'' is remarkably small, under these conditions, the measurement is now more sensitive to ε'' than it is to ε' and the value of ε'' is very high. From this we learn that the technique and measurement conditions are not best suited to the characterization of very low loss factors and that we should expect high uncertainty in the characterization of the permittivity in the presence of very high conductive loss factors.

In the above analysis, the calculated uncertainty in the dielectric properties depends, ultimately, on the assumed uncertainty in the measured quantity, which is difficult to estimate objectively under any given set of experimental conditions. Our estimate of 0.1% (used in calculating the data for figures 1(a) and (b)) is of the same order of magnitude as the residual error, estimated from re-measuring the short circuit after calibration, this being the most challenging standard to reproduce. Otherwise, it is an arbitrary figure that serves well for the purpose of illustration.

3. Assessment of uncertainty: an experimental approach

To get a more realistic assessment of the uncertainty we propose to use repeat measurement on reference materials as previously reported (Gabriel *et al* 1994). The repeatability of the measurement gives the extent of random errors while deviation of the mean value from that known for the standard is a measure of systematic errors; both contribute to the total uncertainty in the measurement of the dielectric properties. The effectiveness of this approach depends on the identification of suitable reference materials to use as standards.

3.1. Reference materials

We define reference materials as those with well-characterized dielectric properties. We opted to use high purity (Analar grade) monomolecular liquids to ensure integrity and homogeneity of the material. Our experimental data will be presented as relative permittivity ε' and conductivity $\sigma = \omega\varepsilon_0\varepsilon''$ where ω is the angular frequency and ε_0 the permittivity of free space. The following materials were considered as possible reference materials.

3.1.1. Water. Water is probably the best available reference material; its dielectric properties are among the most studied and reported in the literature. A comprehensive list of references and a historical overview of the subject can be found in Ellison *et al* (1996). There are other notable reviews by Kaatze (1989) and Liebe *et al* (1991). The consensus is that data from 0–100 GHz exhibit a near perfect Debye dispersion with fairly well-defined parameters. Table 1 gives the Debye parameters for water at 20 °C from three review studies. The Debye expression models the frequency dependence of the complex permittivity

$$\varepsilon(\omega) = \varepsilon_\infty + \frac{(\varepsilon_0 - \varepsilon_\infty)}{1 + \mathrm{j}\omega\tau}. \tag{7}$$

The limiting values of the permittivity ε_s and ε_∞ (at $\omega\tau \ll 1$ and $\omega\tau \gg 1$, respectively) are known as the static and infinite permittivities and τ is the relaxation time. Evident from table 1 is that the static permittivity and the relaxation time are fairly well defined, less so the infinite permittivity. Fortunately, this parameter has little impact on the dielectric data in the low gigaHertz range because its value is only a small percentage of the permittivity in that frequency range. This relatively large uncertainty highlights the fact that even this most studied, pure substance is not a perfect reference liquid and that much remains to be done in the characterization of the dielectric properties of water at teraHertz frequencies. In our laboratory de-ionized water is used as a calibration standard, for this reason it cannot be used to assess residual systematic errors in the measurement system.

Table 1. Debye parameters for de-ionized water at 20 °C.

Reference	ε_s	ε_∞	τ (ps)
Kaatze (1989) (3.1–40 GHz)	80.2 ± 0.2	5.6 ± 0.2	9.36 ± 0.05
Kaatze (1989) (3.1–40 GHz)[a]	80.2	5.2	9.47
Liebe *et al* (1991) (0–100 GHz)	80.1	5.3	9.35
Büchner *et al* (1999b)	80.2[b]	6.0[c]	9.42 ± 0.10[c]

[a] Calculated from empirical equations (dependence of dielectric parameters upon temperature).
[b] Taken from Ellison *et al* (1996) from a comprehensive survey of literature data.
[c] Parameters of the main dispersion obtained by fitting two Debye processes to the experimental data from Barthel *et al* (1995), Ellison *et al* (1996) (0.1 GHz–150 THz) and Ronne *et al* (1997) (0.1–2 THz).

Table 2. Model parameters for methanol at 20 °C .

Reference	Model	ε_s	ε_∞	τ (ps)	α
This study (50 MHz–20 GHz)	Debye	33.9 ± 0.1	4.7 ± 0.1	53.2 ± 0.8	–
This study (50 MHz–5 GHz)	Debye	34.0 ± 0.08	5.68	55.01 ± 1.06	–
NPL (2001) (30 MHz–5 GHz)	Debye	33.64 ± 0.06	5.68 ± 0.18	56.6 ± 1.05	–
Jordan *et al* (1978) (10 MHz–70 GHz)	Cole–Cole	34.8 ± 0.5	4.5 ± 0.7	56 ± 2.0	0.044 ± 0.027

3.1.2. Methanol. Methanol is one of the reference liquids measured by the UK National Physics Laboratories (NPL) between 1989 and 1991 and included in their 2001 database (NPL EMMA-club dielectrics database 2001). The measurements were carried out at temperatures from 20 °C to 30 °C and comprised measurement of the static permittivity and measurements in the microwave region between 100 MHz and 3 GHz.

More recently, NPL extended their measurement to 5 GHz and reported data over an extended temperature range (NPL, Report CETM 33 2001). Their technique and the traceability of their static permittivity measurement were reported later in the context of a comparative study with previously published data (Gregory and Clarke 2005). In the case of methanol, their experimental data (up to 5 GHz) were fitted to a Debye model, the parameters of which are given in table 2. Our own data were obtained by curve fitting to the Debye model measured permittivity and conductivity data in the frequency range 50 MHz–20 GHz. Jordan *et al* (1978) fitted their data (15 data points between 10 MHz and70 GHz) to a Cole–Cole model:

$$\hat{\varepsilon} = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{1 - (\mathrm{j}\omega\tau)^{1-\alpha}}; \qquad (8)$$

this is a modified version of the Debye expression where α is an empirical distribution parameter indicating the presence of a symmetrical distribution of relaxation times, evident in the extended spectrum covered by Jordan's data. Earlier work (Grant 1957, Saxton *et al* 1962) found a relaxation time of 53 ps at 20 °C and some evidence of a second process at higher frequencies. The agreement between our static permittivity value and that of NPL is well within the 95% confidence interval and differs by about 2% from Jordan's data. With data up to 5 GHz only, the NPL model returns an effective value for ε_∞ that does not represent the true high frequency limit. Altogether, methanol is not sufficiently well defined to be used as a reference liquid outside the bounds of NPL's data.

Table 3. Model parameters for ethanediol at 20 °C.

Reference	Model	ε_s	ε_∞	τ (ps)	β
This study (130 MHz–20 GHz)	Cole–Davidson	41.5 ± 0.1	3.8 ± 0.1	157.18 ± 4.04	0.82 ± 0.01
Levin and Podlovchenko (1970) (3–17.6 GHz)	Cole–Davidson	41.4	3.7	164	0.8
NPL (2001) (30 MHz–5 GHz)	Debye-Γ	41.9 ± 0.1	7.03 ± 0.15	147.23 ± 2.45	–
NPL 2006 (30 MHz–5 GHz)	Cole–Davidson	41.9 ± 0.1	5.02 ± 0.31	161.41 ± 6.88	0.88 ± 0.02

Table 4. Model parameters for dimethyl sulphoxide (DMS) at 25 °C.

Reference	Model	ε_s	ε_∞	τ (ps)	β
This study (130 MHz–20 GHz)	Cole–Davidson	47.2 ± 0.1	3.9 ± 0.7	22.82 ± 1.26	0.85 ± 0.03
Kaatze *et al* (1989) (1 MHz–40 GHz)	Cole–Davidson	47.0 ± 0.6	3.9 ± 1.0	21.1 ± 0.2	0.88 ± 0.01
NPL (2001) (30 MHz–5 GHz)	Debye	46.5 ± 0.1	6.6 ± 1.2	19.18 ± 1.24	–

3.1.3. Ethanediol. Our measurement on ethanediol best fits a version of the Debye equation known as Cole–Davidson:

$$\hat{\varepsilon} = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{(1 - \mathrm{j}\omega\tau)^\beta}, \tag{9}$$

where β is a distribution parameter defining a non-symmetrical spread of relaxation times (table 3). Cole–Davidson parameters for the dielectric spectrum of ethanediol are also provided by Levin and Podlovchenko (1970), Demau *et al* (1972) and NPL (2006). Jordan *et al* (1978) were able to identify two separate Debye dispersions in the dielectric spectrum of ethanediol based on experimental data extending to 70 GHz. Table 3 shows that there is an excellent agreement in ε_s from all sources. Where the same model is used, there is reasonable agreement for τ, β and ε_∞.

3.1.4. Dimethyl sulphoxide (DMS). As with ethanediol, our data for DMS were best fitted to a Cole–Davidson model (9), as did more extensive data (to 40 GHz) by Kaatze *et al* (1989). NPL's data (to 5 GHz) were represented by a Debye model, providing a value for ε_s in good agreement with Kaatze's data and ours. Table 4 summarizes the comparative analysis.

3.1.5. Formamide. Formamide has a static permittivity of about 111 at 20 °C, its permittivity is higher than that of water at frequencies below a few gigaHertz. It is used in our laboratory to test the instrumentation after calibration but prior to measurement. Our measurements show that the dielectric data for formamide are well represented by a Debye model and that it has a non-zero static conductivity. To model our experimental data we used the following expression:

$$\hat{\varepsilon} = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{1 - \mathrm{j}\omega\tau} - \frac{\mathrm{j}\sigma_s}{\omega\varepsilon_0}, \tag{10}$$

where σ_s is the static conductivity. Formamide does not feature in the NPL (NPL, Report CETM 33 2001) study. Jordan *et al* (1978) based their analysis on data above 1 GHz and therefore did not characterize σ_s as its effects become apparent at lower frequencies. To compare our data to Jordan's we have to subtract the contribution of σ_s from our measured

Table 5. Model parameters for formamide 20 °C.

Reference	Model	ε_s	ε_∞	τ(ps)	σ (S m^{-1})	β
This study (130 MHz–20 GHz)	Debye	111.6 ± 0.2	5.4 ± 0.2	40.1 ± 0.4	0.028 ± 0.003	–
Jordan *et al* (1978) (1–70 GHz)	Debye	111.8 ± 0.9	6.9 ± 1.0	42.0 ± 1.0	–	–
Barthel *et al* (2002) (0.2–89 GHz)	Cole– Davidson	110.5	5.3	46.8	–	0.91

Table 6. Model parameters for 0.1 M NaCl at 20 °C.

Reference	Model	ε_s	ε_∞	τ (ps)	σ (S m^{-1})	α
This study[a] (50 MHz–20 GHz)	Cole–Cole	78.2 ± 0.2	5.0	9.08 ± 0.2	0.96 ± 0.0	0.006 ± 0.008
Stogryn (1971) (9.3, 24.2 and 48.4 GHz)	Debye	78.1	4.9	9.25	0.92	
Nörtemann *et al* (1997)[b] (20 MHz–40 GHz)	Debye	79.7 ± 1.1	5.6 ± 1.0	9.17 ± 0.4	–	–
Büchner *et al* (1999a)[c] (0.2–20 GHz)	Cole–Cole	79.1	5.6	9.38	0.96	0.017

[a] The value of α is such that the model reverts to Debye.
[b] Interpolated between the data from 0.05 M and 0.17 M concentrations.
[c] Interpolated between the data from 0.09 M and 0.13 M concentrations.

data or else limit the comparison to frequencies above 1 GHz. Table 5 gives the Debye parameters from our and Jordan's data.

In a more recent study Barthel *et al* (2002) used waveguide interferometry and time domain reflectometry to obtain the dielectric properties of formamide in the frequency range of 0.2–89 GHz. The authors fitted their experimental data to a Cole–Davidson model (table 5).

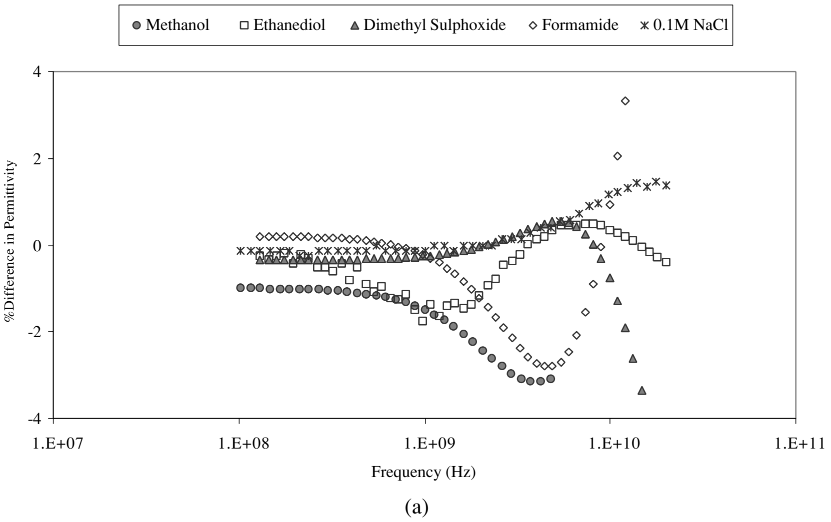
3.1.6. NaCl solutions.

Aqueous NaCl solutions are easy to prepare using de-ionized water and Analar grade, anhydrous NaCl. Their dielectric properties are described by

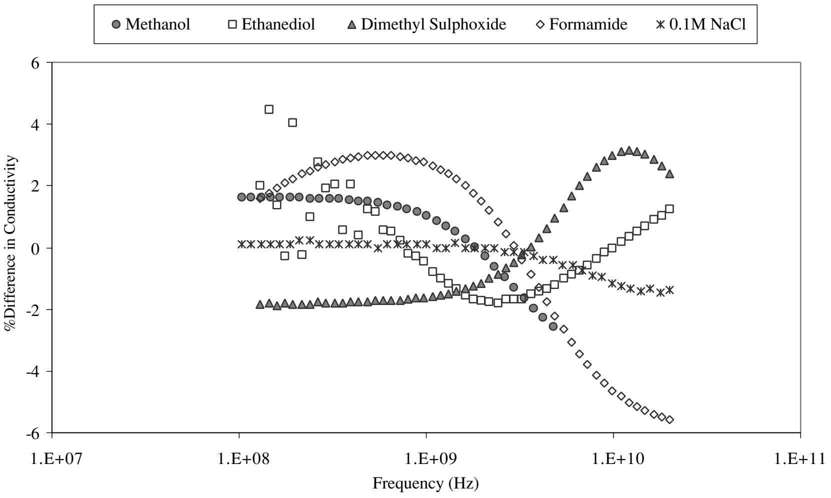
$$\hat{\varepsilon} = \varepsilon_\infty + \frac{(\varepsilon_s - \varepsilon_\infty)}{1 - (\mathrm{j}\omega\tau)^{1-\alpha}} - \frac{\mathrm{j}\sigma_i}{\omega\varepsilon_0} \qquad (11)$$

which is a Cole–Cole expression and static conductivity term originating from ionic drift. At very low concentrations (<0.2 M), the value of the distribution parameter α is very small, difficult to characterize and can be neglected for all practical purposes. Stogryn (1971) published models for the concentration and temperature dependence of ε_s and τ. Stogryn's models were based on limited experimental data, in the gigaHertz range, by Lane and Saxon (1952) and Grant (1957), α was assumed zero throughout. Stogryn's model for the conductivity of NaCl solutions does not strictly apply to concentrations below those equivalent to the salinity of seawater (about 0.7 M) as it was derived using high concentration data (Chambers *et al* 1956, Chambers 1958). More recently, Nörtemann *et al* (1997), on the basis of their own experimental data in the frequency range 20 MHz–40 GHz, reported values for ε_s and τ for concentrations in the range 0–0.6 M at 20 °C, their measured conductivity data are not explicitly reported. With experimental data in the frequency range of 200 MHz–20 GHz, Büchner *et al* (1999a) reported ε_s, τ and α values having obtained σ_i from a physical chemistry database (ELDAR). Table 6 gives the dielectric parameters of 0.1 M NaCl solution at 20 °C obtained from our measurements and from the literature.

Examination of tables 2–6 shows no evidence of systematic error in the data obtained from our measurement procedure as implemented. The observed differences are within the bounds of the stated uncertainty or can be explained by the limitation of the analysis, for example,



(a)



(b)

Figure 2. Percentage difference in (a) permittivity and (b) conductivity as a function of frequency. Methanol data are compared to NPL (2001), Ethanediol data are compared to Levin and Podlovchenko (1970), dimethyl sulphoxide data are compared to Kaatze *et al* (1989), formamide data are compared to Jordan *et al* (1978) and 0.1 M NaCl data are compared to Büchner *et al* (1999a).

the model used to describe the frequency dependence, the extent of the frequency range of the data set fitted to the model and the assumptions made about ε_∞. The spread in dielectric parameters is a measure of the uncertainty in the characterization of the reference materials and is therefore inherent in any measurement procedure. Figure 2 shows how differences in the model parameters (tables 2–6) translate into percentage differences in permittivity and conductivity as a function of frequency.

Of the materials considered, the 0.1 M NaCl solution was deemed an appropriate reference to use to assess the uncertainty in measuring the dielectric properties of biological materials. This is because (i) we can draw on recent publications (Büchner *et al* 1999) to assert its

Table 7. (a) and (b) Example of compilation of uncertainty elements and calculation of standard uncertainty for measured (a) permittivity (ε') and (b) conductivity (σ) of 0.1 M NaCl in the frequency range >300 MHz–10 GHz. The nomenclature and methodology were taken from NIST (1994) and ISO (1995). The calculation was made at each measurement frequency and averaged across the frequency range. The elements of uncertainty were obtained experimentally in terms of permittivity and conductivity; hence the sensitivity coefficient is unity throughout.

	Uncertainty component (%) a	Probability distribution	Divisor b	Sensitivity coefficient c_i	Standard uncertainty (%) $u_i = \left(\frac{a}{b}\right) c_i$
		(a)			
Repeatability (SDM)	0.18	N	1	1	0.18
Deviation from reference	0.26	R	$\sqrt{3}$	1	0.15
Drift	0.07	R	$\sqrt{3}$	1	0.04
Cable movement	0.0	U	$\sqrt{2}$	1	0.0
Combined standard uncertainty (u)		$u = \sqrt{\sum u_i^2}$			0.24
Expanded uncertainty ($k = 2$)		$= ku$			0.48
		(b)			
Repeatability (SDM) (type A)	0.61	N	1	1	0.61
Deviation from reference (type B)	1.26	R	$\sqrt{3}$	1	0.73
Drift	0.24	R	$\sqrt{3}$	1	0.14
Cable movement	0.0	U	$\sqrt{2}$	1	0.0
Combined standard uncertainty		$u = \sqrt{\sum u_i^2}$			0.96
Expanded uncertainty ($k = 2$)		$= ku$			1.92

dielectric behaviour and, (ii) at the limit of low concentrations, the model and parameters are underpinned by our knowledge of the dielectric properties of water. Moreover, the conductivity of a 0.1 M NaCl solution is of the same order of magnitude as that of biological tissues. Its measurement uncertainty must first be characterized before it can be used in the assessment of biological materials.

3.2. Calculation of the uncertainty in the measurement of 0.1 M NaCl solution

The uncertainty budget for the measurement of the 0.1 M NaCl solution was drawn in accordance with established guidelines (NIST 1994 and ISO 1995). Tables 7(a) and (b) show the calculation of the uncertainty for the frequency range 300 MHz–10 GHz. To achieve this, four main sources of uncertainty were identified, quantified and expressed in terms that can be added to provide the total combined uncertainty. (1) Random errors were obtained from repeat measurements, carried out under strict temperature control, over many sessions (having re-calibrated the VNA in between sessions), at different time intervals within a session; the mean and percentage standard deviation of the mean (SDM) were calculated for the permittivity and conductivity at each measurement frequency and averaged over the frequency range. The measure of repeatability entered in the table is two times the SDM. (2) The percentage deviation between measured and literature data (Büchner *et al* 1999) was taken as a measure of the uncertainty originating from the instrumentation and measurement methodology. (3) Drift of the calibration with time is another error factor that was considered and quantified experimentally. (4) Movement of the test cable is a possible source of error that was avoided in this case by moving the sample to the probe rather than the other way round.

Table 8. Summary of elements of uncertainty in the measured permittivity (ε') and conductivity (σ) of 0.1 M NaCl across the spectrum. All the terms are expressed as percentage of the relevant parameter.

Frequency region (MHz)	Repeatability (SDM)		Deviation from reference		Drift		Total combined uncertainty	
	ε'	σ (S m^{-1})	ε'	σ (S m^{-1})	ε'	σ (S m^{-1})	ε'	σ (S m^{-1})
50–300 MHz	0.7	0.3	0.1	0.8	0.1	0.1	0.7	0.6
>300 MHz–10 GHz	0.2	0.6	0.3	1.3	0.1	0.2	0.2	1.0
>10–20 GHz	0.5	2.1	1.4	1.0	0.2	0.2	0.9	2.1

Random errors follow a normal (N) distribution, by contrast accuracy (deviation from reference) and drift errors are uniformly distributed forming a rectangular distribution (R). Cable movements, when present, introduce errors better described by a U-shaped distribution. The probability distribution determines the factor by which the uncertainty value must be divided to be equivalent to one standard deviation.

The uncertainty components must be expressed in terms of the dielectric parameter under consideration or else converted to it using a sensitivity coefficient. In accordance with the nomenclature in the guidelines, the uncertainty value is termed a, the divisor b the sensitivity coefficient c_i (table 7). Each uncertainty component in permittivity or conductivity is $u_i = (a/b)c_i$. The total combined uncertainty is the square root of the sum of the squares (RSS) of the individual components. The expanded uncertainty is the total uncertainty multiplied by a coverage factor (k) to give an uncertainty value to a required confidence level. In this case, $k = 2$ gives a confidence level of approximately 95%.

The same procedure was used to calculate the uncertainty in the ranges 50–300 MHz and 10–20 GHz (table 8).

4. Uncertainty in the measurement of biological materials

Table 9 gives the components and total combined uncertainty for some tissues obtained from a recently completed study on the dielectric properties of porcine tissue, the measurements were carried out *in vivo*. The measurement procedure was as for the NaCl solution except that in this case, the test cable and probe were moved to reach out to the tissue sampled. Random variations were obtained from the SDM of repeat measurements on a large number of specimens (the number is deemed sufficiently large when the data follow a normal distribution). The uncertainties associated with the instrumentation (deviation from reference and drift) calculated from the comparative analysis of the 0.1 M NaCl reference apply, in addition to that originating from cable movement. Errors from other sources such as temperature variations should be avoided (as in this case) or else estimated and added to the uncertainty budget.

Examination of table 9 shows that for most tissue types the SDM is by far the largest uncertainty element; in most cases it is not significantly different from the total combined uncertainty. However it was essential to scrutinize the measurement technique and to quantify the methodological uncertainty before concluding that, for biological tissue, the uncertainty originates mostly from the sampling procedure and the natural inhomogeneity of the sample. Consequently, in this case, the SDM would be a good measure of the uncertainty in the dielectric properties of biological materials. This statistical uncertainty can be improved by ensuring the integrity of the sample, maintaining consistency in the sampling technique and increasing the number of samples measured.

Table 9. Total combined uncertainty for the permittivity (ε') and conductivity (σ) of porcine tissues measured *in vivo*.

Frequency region	Tissue	Repeatability (SDM)		Deviation from reference[a]		Drift[a]		Cable movement		Total combined uncertainty	
		ε'	σ	ε'	σ	ε'	σ	ε'	σ	ε'	σ
50–300 MHz	Grey matter	1.8	4.5	0.1	0.8	0.1	0.1	0.4	0.3	1.8	4.5
	White matter	5.7	7.8							5.7	7.8
	Cornea	0.8	1.4							0.8	1.5
	Long bone	4.2	8.4							4.2	8.4
	Cartilage	1.7	7.3							1.8	7.3
	Liver	1.5	2.7							1.5	2.8
	Fat	7.0	10.6							7.1	10.6
>300 MHz–10 GHz	Grey matter	2.1	3.0	0.3	1.3	0.1	0.2	0.2	0.4	2.1	3.1
	White matter	4.5	6.3							4.5	6.3
	Cornea	0.9	1.0							0.9	1.3
	Long bone	4.4	5.0							4.4	5.1
	Cartilage	2.0	4.4							2.0	4.5
	Liver	1.2	1.7							1.2	1.9
	Fat	6.7	9.0							6.7	9.0
>10–20 GHz	Grey matter	2.8	5.6	1.4	1.0	0.2	0.2	1.0	2.4	3.0	5.9
	White matter	4.4	7.9							4.5	8.1
	Cornea	1.4	2.2							1.8	2.8
	Long bone	5.5	5.3							5.6	5.6
	Cartilage	2.9	4.2							3.1	4.5
	Liver	1.4	2.9							1.8	3.4
	Fat	6.1	8.5							6.2	8.6

[a] From table 8. All terms are expressed as percentage of the relevant parameter.

5. Conclusions

Numerical and experimental procedures were used to calculate the uncertainty in the measurement of the dielectric properties; the examples used to illustrate the arguments relate to the use of open-ended coaxial probes and VNAs; the technique most commonly used for the measurement of biological tissues.

The numerical approach requires an assumption to be made about the uncertainty in the measurement of the reflection coefficient, which is difficult to estimate objectively. However, this type of analysis serves two purposes: it demonstrates the complexity of the dependence of the calculated uncertainty on multiple parameters, and it can be used to set theoretical bounds on the uncertainty under a given set of experimental conditions.

The experimental approach involves data analysis to characterize the precision and accuracy of the measurement. The method relies on the availability of well-characterized materials to use as reference standards. For this reason and prior to its implementation, the dielectric properties of five pure liquids and those of an aqueous ionic solution (0.1 M NaCl) were critically reviewed and the uncertainty inherent in their characterization was highlighted. The ionic solution was deemed an appropriate reference for biological materials; it was measured and the data analysed to provide an uncertainty budget comprising random and instrumental components from which the total measurement uncertainty was calculated.

To determine the uncertainty in the measurement of biological tissue, we obtain the random variations from repeat measurements and assume that the instrumental and methodological uncertainties calculated for the NaCl solution apply. In practice, for most tissue types the random uncertainty is by far the largest element in the uncertainty budget.

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