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Towards Validating a Coaxial Transmission Cell for Dielectric Measurements on Liquids

Bayan Tallawi[1, 2], Floriane Sparma[2], Eric Georgin[1], and Pierre Sabouroux[2, *]

Abstract—Moisture measurement in industrial applications, both in liquid and solid materials, presents a significant challenge. In the field of biofuels, this becomes even more critical. Among the various approaches developed for this purpose, indirect electromagnetic techniques have emerged as a valuable tool for accurately estimating moisture content. These techniques utilize the complex dielectric permittivity ε as an intermediary parameter, which is influenced by the water content in the material. As a first step toward this purpose, a $1''5/8$ two-port coaxial transmission cell, developed at *LNE-CETIAT*, was studied to make dielectric measurements on liquids. Characterization and validation steps were requested to demonstrate the accuracy of this cell. For this purpose, an intra-laboratory comparison has been performed first at *LNE-CETIAT* using the $1''5/8$ cell and *EpsiMu*® coaxial cell — a fully validated reference tool. Then, an inter-laboratory comparison with the *Fresnel Institute* has been performed using a coaxial probe and another *EpsiMu*® cell. The measurements were carried out under identical ambient conditions, using liquid reference materials. In this work, the performance of the developed cell in the frequency band [0,1–1,1] GHz has been validated, as well as the accuracy of the three electromagnetic techniques used. The results of the experiments confirm the effectiveness of the $1''5/8$ cell developed at *LNE-CETIAT* for measuring the dielectric properties of liquids.

1. INTRODUCTION

Accurate determination of water content is essential for ensuring process or product quality and efficiency in many industries. During the last couple of years, *LNE-CETIAT* has been involved in different projects related to moisture measurement and how metrology may underpin industry such as: *EURAMET Project 1400* — Moisture metrology development, *JRP SIB64 METefnet* — Metrology for moisture in materials, and more recently the development of calibration services for enabling SI traceability to industry, funded by French Metrology, and *JRP 19ENG09 BiofMET* — New metrological methods for biofuel material analysis [1]. This last project is namely funded by the European Metrology Program for Innovation and Research *EMPIR* co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation program.

From a metrological point of view, electromagnetic techniques are considered as secondary methods, for measuring moisture, since they need to be calibrated to primary method. These techniques are however useful to measure the moisture content of products in several sectors (food industry, drying processes industry, pharmaceutical industry, petrochemical industry...) [2–4]. Permittivity measurements can be used in a variety of applications beyond water content determination. In biomedical applications, for example, permittivity measurements can be in the development of medical imaging techniques such as magnetic resonance imaging (MRI) and microwave imaging [5–7]. These

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* Corresponding author: Pierre Sabouroux (pierre.sabouroux@fresnel.fr).
[1] CETIAT, 25 Avenues of the Arts - BP 52042, Villeurbanne 69603, France. [2] Aix Marseille Univ, CNRS, Centrale Marseille, Institut Fresnel, Marseille, France.

techniques rely on differences in permittivity between different types of tissue to generate images of the body.

The subject of interest for these secondary techniques is the identification of the material under test's properties, when being set in electromagnetic fields of radio and microwave frequencies, such as reflection, attenuation, shift of resonant frequency, and shift of quality factor [8–10]. They are increasingly in demand in the industry considering that the measurement or characterization techniques using microwaves are nondestructive and allow very fast quantitative estimation of moisture content. In addition, they can be classified according to their ability to be used in contact or without contact [11, 12]. This is typically one of the most important criteria in selecting a method for industrial usage.

When an electromagnetic wave encounters a material, it can be transmitted, reflected, or absorbed. The extent to which each of these occurs depends on several factors such as the frequency of the wave, , dielectric constant, magnetic permeability of the material, and the angle of incidence of the wave. Furthermore, the properties of the material itself can affect how it interacts with the electromagnetic wave. For example, the presence of moisture or other impurities in the material can affect its dielectric properties, which can in turn affect the behavior of the wave. The Maxwell's equations describe these interactions mathematically [13]. Following those equations, we can characterize a material by three intrinsic quantities: ε, the complex dielectric permittivity, μ, the complex magnetic permeability, and σ, the conductivity of the material.

In this work, we are interested in studying the dielectric permittivity ε of the material as a function of frequency. It can be expressed as a complex quantity, with both real and imaginary parts. The real part of the permittivity is known as the "dielectric constant" or "static permittivity", and the imaginary part is known as the "loss factor" or "dielectric loss". The complex permittivity can be represented using the following equation:

$$\varepsilon = \varepsilon_0 \varepsilon_r = \varepsilon_0(\varepsilon' - j\varepsilon'') \tag{1}$$

where ε_0 is the dielectric permittivity of vacuum ($\varepsilon_0 = 8,85418782 \times 10^{-12}\,\mathrm{F{\cdot}m^{-1}}$); ε' and ε'' are respectively the real and imaginary parts of the dielectric permittivity. The dielectric constant (ε') represents the ability of a material to store electric charge in an electric field, while the dielectric loss (ε'') represents the energy lost in the material due to the conversion of electrical energy into other forms of energy (such as heat) during the polarization process.

Waveguides are frequently used as propagation lines to characterize dielectric and/or magnetic materials. There are three types of waveguides: circular, rectangular, and coaxial ones. The principle of this technique is based on the use of a waveguide to transmit an electromagnetic wave through a material. Indeed, the propagation of the electromagnetic wave in this type of device is guided, and therefore, the transmitted signal is very stable and can be accurately measured. The propagation velocity of this electromagnetic wave through the material under test, calculated according to the Maxwell's equations, is directly related to its dielectric constant and magnetic permeability.

In this study, a coaxial cell developed at *LNE-CETIAT* for measuring complex dielectric permittivity was investigated. Experimental studies were conducted to characterize and determine the frequency limits of operation of the cell. This cell was validated by comparing the results obtained with those obtained with the *EpsiMu*® coaxial cell and the *SPEAG DAK 3,5* coaxial probe on liquid reference materials with permittivity values known from the literature.

2. MATERIALS AND EXPERIMENTAL METHODS

The type of coaxial cell used in this work is based on the normal plane wave transmission method. This device offers high precision and sensitivity to small variations in material characteristics. There are two methods of measurement using a coaxial line: the reflection method and transmission/reflection method. In this paper, only one measurement procedure has been used and improved.

The principle of measurement with a coaxial line using the transmission/refection method consists in emitting electromagnetic waves through a coaxial cable toward a sample under test. A portion of the waves is reflected at the interface between the sample and the coaxial line, while the rest of the waves are transmitted through the sample and detected by a receiver. The reflected and transmitted waves are then measured by a Vector Network Analyzer (VNA), which provides the amplitude and phase of the waves. From these measurements, the complex reflection coefficient and transmission coefficient of the sample can be calculated [14]. This method allows for the simultaneous measurement of both permittivity and permeability of the sample, which is not possible with the reflection method. However, it requires careful calibration and measurement of the coaxial line, as well as proper handling and preparation of the sample to ensure accurate results. By using the *Anritsu MS2038C* VNA, connected to the two ports of the coaxial cell, the scattering parameters S_{ij} of the material under test can be measured.

Furthermore, when electromagnetic waves propagate along the coaxial cable, an attenuation and a phase rotation occur, due to the inherent properties of the transmission line. In order to compensate these propagation effects, from the output of the VNA to the face of the sample under test, a de-embedding operation is applied. The process involves moving the reference planes so that the electromagnetic properties of the tested material can be measured separately, without interference from the properties of the transmission line.

Indeed, the VNA is first calibrated up to the calibration reference planes 1 and 2, which mathematically and physically represent the terminations of the cables used for the measurements. The information collected is primarily the total matrix of the coaxial cell $[S_{\text{cell}}]$, from which the sample matrix $[S_{\text{sample}}]$ located at the calibration reference planes $1'$ and $2'$ must be extracted, which physically represents the position of the sample in the measurement device (Figure 1) [15].

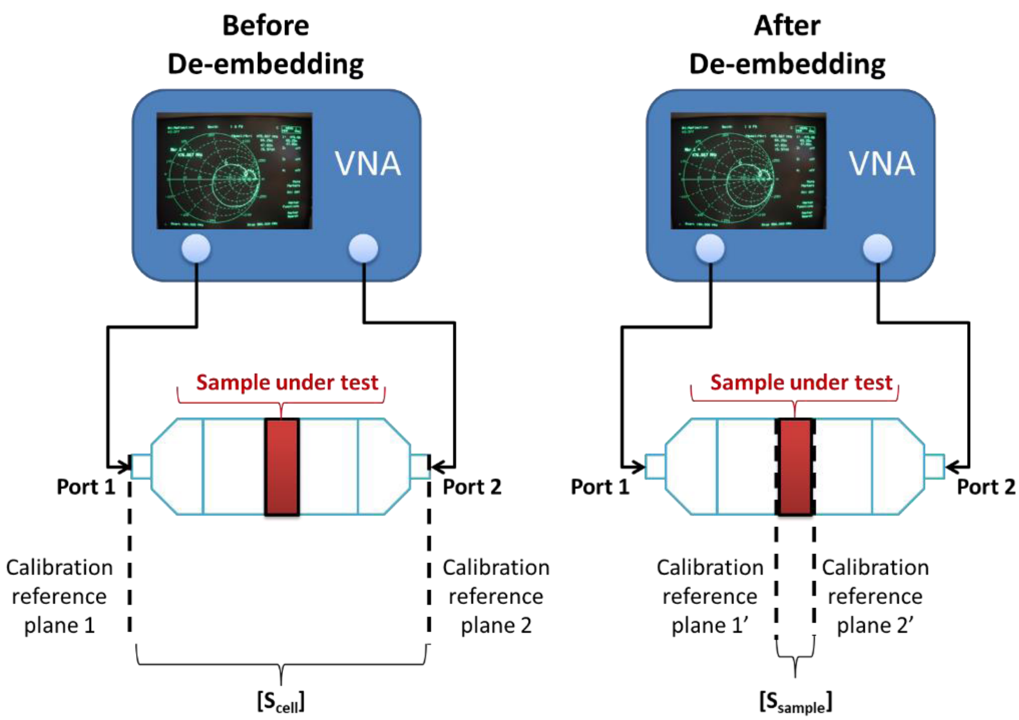


Figure 1. Measuring principle with the coaxial cell: Transfer of calibration plans (de-embedding operation).

The $[S_{\text{sample}}]$ parameters are related to the reflection coefficient Γ, and the transmission coefficient T is as follows [12]:

$$\Gamma = \frac{1+S_{11}^2-S_{21}^2}{2S_{11}} \pm \sqrt{\left(\frac{1+S_{11}^2-S_{21}^2}{2S_{11}}\right)^2-1} \tag{2}$$

$$T = \frac{S_{11}+S_{21}-\Gamma}{1-(S_{11}+S_{21})\Gamma} \tag{3}$$

Finally, according to *Nicolson and Ross* [16], these parameters are related to the complex dielectric permittivity and to the complex magnetic permeability of the tested sample by the following specific formulas:

$$\varepsilon_r = j\frac{c}{2\pi f L}\left(\frac{1+\Gamma}{1-\Gamma}\right)\ln\left(\frac{1}{T}\right) \tag{4}$$

$$\mu_r = j\frac{c}{2\pi L}\left(\frac{1-\Gamma}{1+\Gamma}\right)\ln\left(\frac{1}{T}\right) \tag{5}$$

with c being the speed of light in vacuum (which is equal to 299792,458 km/s) and L the length of the tested sample. We consider the classic hypothesis that the speed of propagation of an electromagnetic wave in the spectral range around gigahertz is equal to the speed c.

At *LNE-CETIAT*, a software has been developed that uses the aforementioned equations to determine the complex dielectric permittivity and complex permeability of the material under test in quasi-real time.

2.1. $1''5/8$ LNE-CETIAT's Cell

A cylindrical coaxial cell (Figure 2) has been developed at *LNE-CETIAT* in collaboration with *Fresnel Institute* for dielectric measurements in solids and liquid samples [8], in four different configurations: Short circuit, Open circuit, Load, and Transmission. This coaxial cell is based on a $1''5/8$ standard line. The mechanical dimensions of the a $1''5/8$ standard [17] cell are:

- The internal radius $a = 8,41$ mm
- The external radius $b = 19,40$ mm.

The physical dimensions of the coaxial cell body have a tolerance of $+/-0.05$ mm.

The choice of using liquids as reference materials for measuring dielectric permittivity is primarily due to their well-defined and consistent properties. Liquids offer several advantages in terms of their stability, homogeneity, and ease of handling. Thus, in order to validate the coaxial cell, two sample-holders dedicated to liquids, have been developed, having the length, L, of 30 and 35 mm. They are

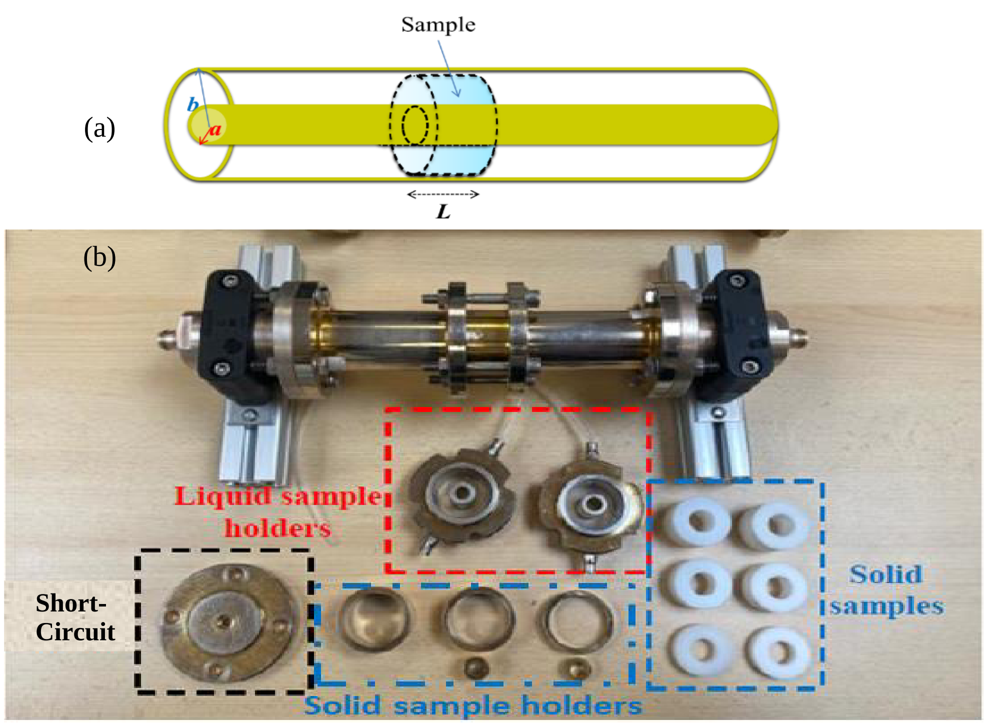


Figure 2. (a) Illustration of a coaxial cell structure. (b) Picture of the $1''5/8$ coaxial cell developed at LNE-CETIAT and some reference samples.

sealed by two quartz windows, glued on both sides. Standard N-type connectors are used to connect this cell to the VNA for measuring the S-parameters.

2.2. *EpsiMu*® Cell

With a frequency range from 1 MHz to 8 GHz, *EpsiMu*® — *PE13* (Figure 3) analyzes the electromagnetic characteristics of any type of material: liquids, gels, granules, and powders. The removable waterproof sample holder, available in various lengths (3, 6, and 12 mm), distinguishes the *EpsiMu*® cell from other similar devices. The measurement can be performed once the material is contained in the sample holder [18]. The connection of this cell to the VNA, allowing the measurement of S-parameters, is made using standard PC7 connectors.

Figure 3. The *EpsiMu*® coaxial cell and its accessories.

The dimensions of the PE13 sample holder with different lengths, 6 mm (PE13-6) or 12 mm (PE13-12), have an outer diameter of 13 mm and an inner diameter of 5,65 mm to maintain an impedance of 50 Ω in the sample holder. The *EpsiMu*® software for permittivity control allows users to monitor and control the accuracy of measurements in real-time. This cell has already been developed and validated; it is now a reference for dielectric measurements in solids and liquids. Comparing two techniques, from two different cells, increases confidence in the results obtained. In general, permittivity measurements published in various articles are very rare, if ever, compared with other techniques.

2.3. *SPEAG DAK 3,5* Open Ended Coaxial Probe

The open-ended coaxial probe used in this work is the *SPEAG DAK 3,5* (Figure 4) provided from *SPEAG* (*www.speag.swiss*). The principle of the *SPEAG DAK 3,5* open-ended coaxial probe is based on the transfer of electromagnetic energy between the probe and the liquid under test. When the probe is placed in contact with the material, the open end of the coaxial cable acts as a small antenna, which can couple electromagnetic energy to or from the device under test (DUT). This coupled energy can then be measured by connecting the other end of the coaxial cable to a VNA. The measuring principle is based on the measurement of the reflection at the end of a coaxial guide associated with a capacitive model [19]. The *SPEAG DAK 3,5* coaxial probe is designed to operate in a wide frequency range from 200 MHz to 3,5 GHz.

To accurately measure the permittivity of a material with the coaxial cell, it is important to choose a frequency band where the measurement is most sensitive to changes in the permittivity. Typically, this is a frequency range where the wavelength of the electrical signal is similar to the dimensions of the coaxial cell. In this range, small changes in the permittivity of the material will result in measurable changes in the transmission characteristics of the coaxial line.

The frequency is an extra variable that has been introduced in the accuracy test of a coaxial cell for dielectric measurements to study the behavior of the material under different electromagnetic fields. By introducing frequency in this study, we can study how the accuracy of the coaxial cell

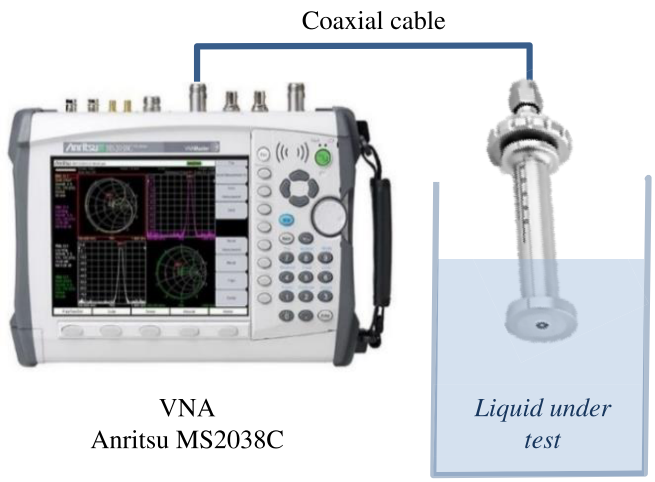


Figure 4. Schematic representation of the measurement with the coaxial probe. The *SPEAG DAK 3,5* open-ended coaxial probe.

changes at different frequencies. Analyzing the performance of the cell and the materials under test across different frequency ranges can yield valuable insights, enabling the identification of optimal and suboptimal operating frequencies for the cell.

Furthermore, the choice of frequency band can also affect the accuracy of the measurement. At high frequencies, the transmission characteristics of the coaxial line can be affected by other factors such as conductor losses and reflections, making the measurement more challenging. Therefore, it is important to choose a frequency band that balances sensitivity and accuracy to obtain accurate and reliable measurements of the complex permittivity of materials with the coaxial cell.

After considering all relevant factors, we have established the frequency range for conducting experiments with coaxial cells, as indicated in Table 1. The upper limit of this frequency range is determined by the highest measured permittivity value, observed at approximately 1 GHz in this study, with ethanol serving as the reference material. This calculation enables us to determine the maximum frequency suitable for conducting measurements on a given sample. Consequently, the frequency range selected for this study spans from 0.1 GHz to 1.1 GHz.

Table 1. List of influence parameters on the working frequency band. The maximum frequency with sample is calculated.

Technique/ Manufacture	**Inner radius (mm)**	**External radius (mm)**	**Flange diameter (mm)**	**Maximum Sample length (mm)**	**Maximum Frequency (GHz)**	**Frequency max with ethanol (GHz)**
EpsiMu cell/ Fresnel Institute	2.83	6.5	-	12	8	2.5
1″5/8 cell/ *LNE-CETIAT*	8.41	19.4	-	35	3.4	1.1
Probe (DAK 3,5)/ *Speag*	0.46	1.75	18	-	20	-

3. RESULTS AND DISCUSSION

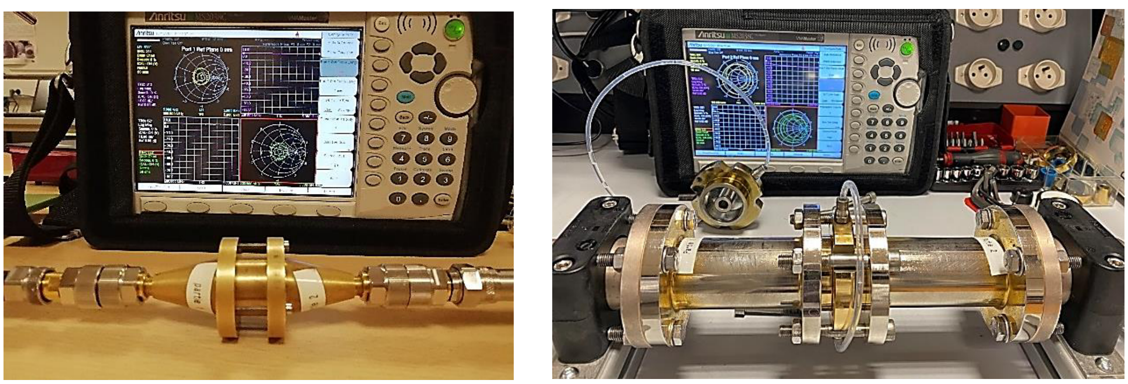
Most reference materials for dielectric permittivity are liquids because liquids are relatively easy to prepare and provide a homogeneous and stable medium for accurate measurements. This is particularly important for the calibration and validation of instruments used for measuring dielectric permittivity, such as coaxial probes or transmission cells. Liquids also have a wider range of dielectric properties than solids, which makes them more useful as calibration standards for instruments that need to operate over a range of dielectric permittivity values. Finally, liquids are less prone to effects from environmental factors such as temperature, humidity, and electromagnetic fields, which can affect the accuracy of the measurement.

In this work, pure alcohols (such as decanol, heptanol, and cyclohexanol) were considered as liquid reference materials for the validation of the 1″5/8 cell. Liquid reference materials, provided from *Sigma-Aldrich*, cover the permittivity range of 8 to 25. Measurements were performed between 0,1 GHz and 1,1 GHz with the same VNA, calibrated to cover the working frequency band. The VNA used in this work is an *Anritsu MS2038C*.

First, an intra-laboratory comparison has been carried out. The two systems used were the 1″5/8 cell (Figure 5), with the two lengths of sample holders: 30 mm and 35 mm, and the *EpsiMu*® cell, with two length of sample holders, 6 mm and 12 mm (Figure 6). Noting that all the sample holders were filled at the same time. The comparison of the results is presented in the graph below (Figures 6 to 11).

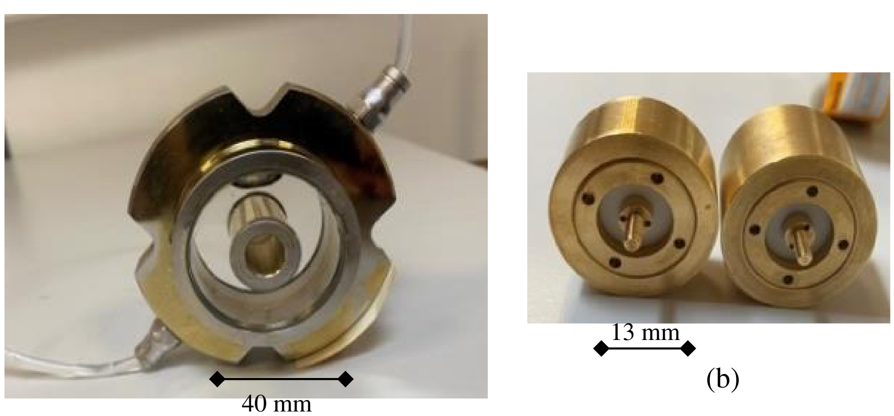
Second, an inter-laboratory comparison, between *LNE-CETIAT* and *Fresnel Institute (IF)*, has been carried out. Three devices have been used, the 1″5/8 cell, the *EpsiMu*® cell, and the *SPEAG DAK 3,5* coaxial probe. The comparison of the results is presented in Figures 12–13.

All the experimental results are in the end compared with the published values proposed by *NPL* (*National Physical Laboratory*) [21] and *NBS* (*National Bureau of Standards*) [22]. Therefore, the results reflect the raw measurements without any averaging or smoothing applied to the data.



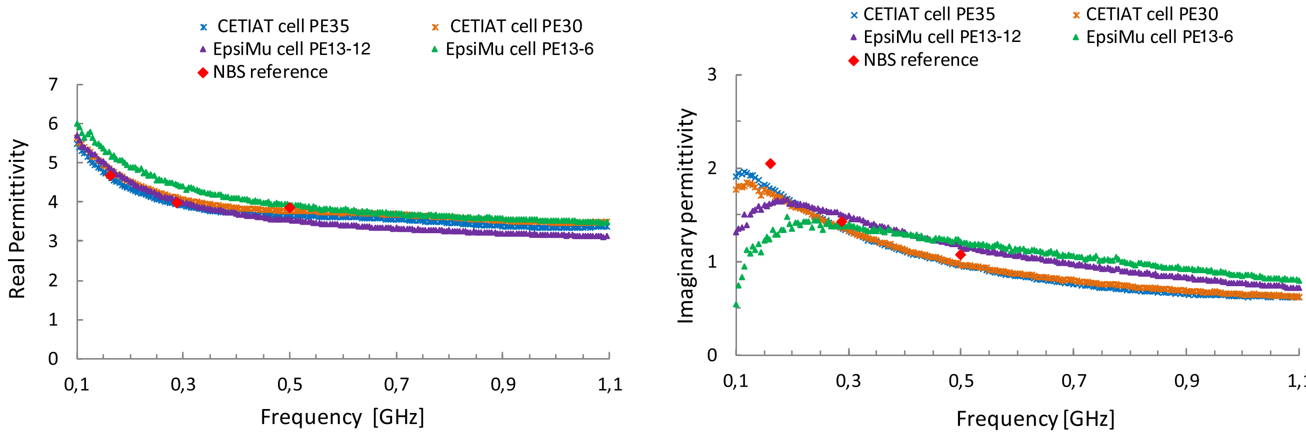
(a) (b)

Figure 5. Photos of the coaxial cell connected to the VNA (a) *EpsiMu*® cell, (b) 1″5/8 cell.



(a) (b)

Figure 6. Photos of the sample holder for liquid, (a) 1″5/8 cell, (b) *EpsiMu*®.



(a) (b)

Figure 7. Comparison between the two coaxial cells for decanol at 25°C. (a) Real part of the permittivity. (b) Imaginary part of the permittivity.

In addition, another intra-laboratory comparison was performed, using the 1″5/8 cell, with different decanol-water mixtures such as 100%, 95%, 90%, 85%, 80%, and 75% in concentration. The aim was to investigate the concentration sensitivity of the developed cell to the water content (Figure 15).

These operations enable also comparisons between different data processing programs as well as measurement methods.

3.1. Intra-Laboratory Comparison at LNE-CETIAT

This section presents the results from experiments carried out at *LNE-CETIAT* using the two coaxial cells with various sample holders for decanol, heptanol, and cyclohexanol.

3.1.1. 1-Decanol

For the measurements, a 99% pure solution of decanol (CAS 112-30-1) was used. For a better comparison with the reference *NBS* data, the measurements with this alcohol were performed at a temperature of 25°C. The figures below illustrate the measurement results.

For the real and imaginary parts of permittivity, the results using the two coaxial cells show a good agreement with the reference. The *EpsiMu*® values measured for the imaginary part, however, show a significant error of approximately 1.5 in the low frequencies. As the frequency increases, this inaccuracy gets quite small. Figure 8 provides a visual representation of the difference between the measured values and the reference values at different frequencies.

3.1.2. Heptanol-1-ol

A second alcohol, which was a 99% pure heptanol (CAS RN 111-70-6), has been used to continue this comparison.

The results indicate that the measured values of the real part of permittivity using the 1″5/8 cell are very close to the reference values over the entire frequency range, suggesting that the measurement method used is reliable and accurate for this parameter over the entire frequency range from 0,1 GHz to 1.1 GHz.

However, this is not the case with the *EpsiMu*® cell, for which the absolute error is significant in low frequencies below 200 MHz. This suggests that the measurement method may not be as accurate for this parameter at low frequencies.

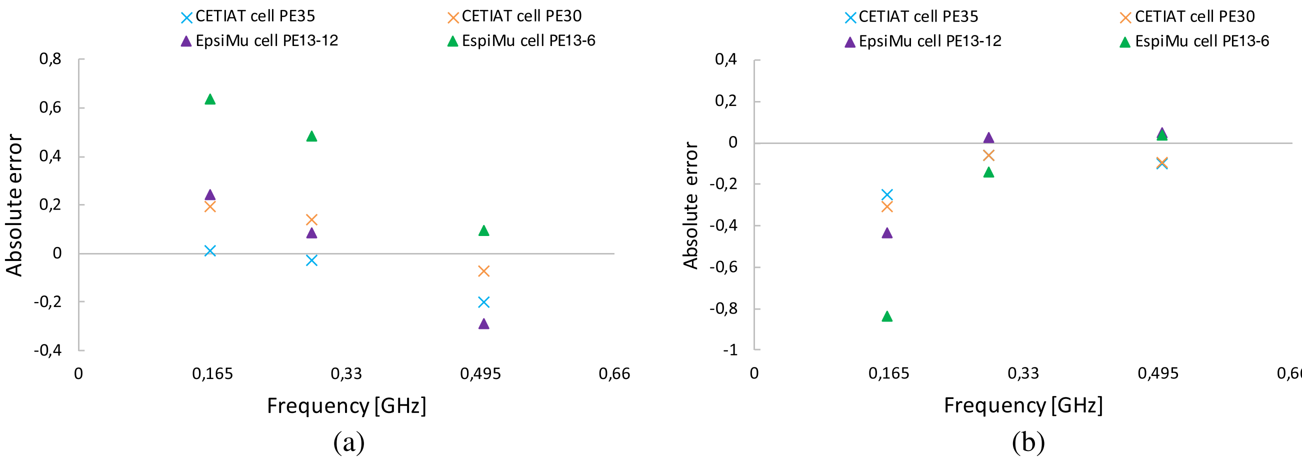


Figure 8. Measurement errors with respect to the reference value at 25°C.

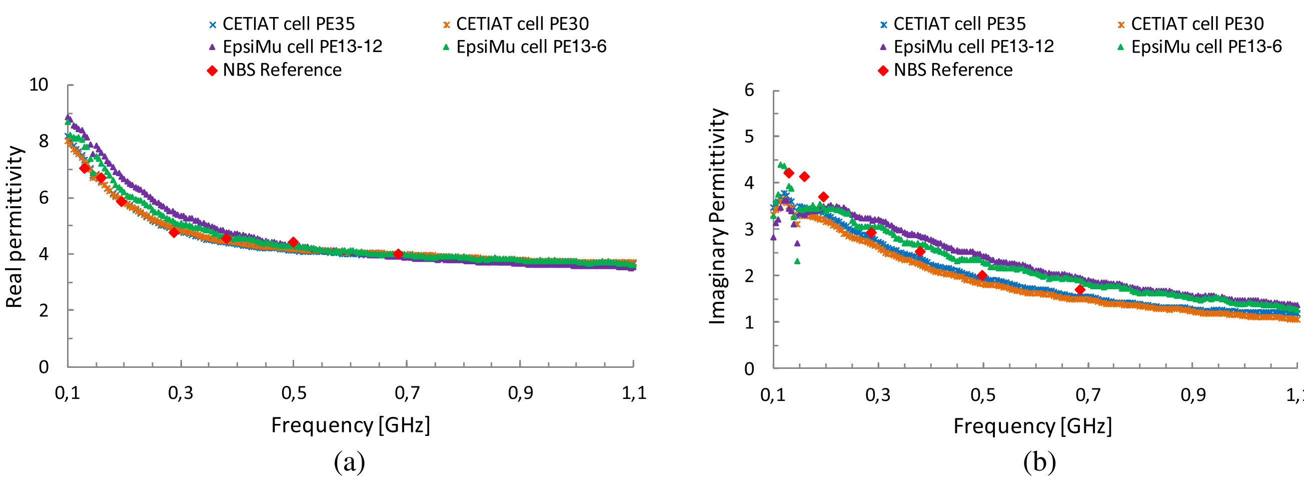


Figure 9. Comparison between the two coaxial cells for heptanol at 20°C. (a) Real part of the permittivity. (b) Imaginary part of the permittivity.

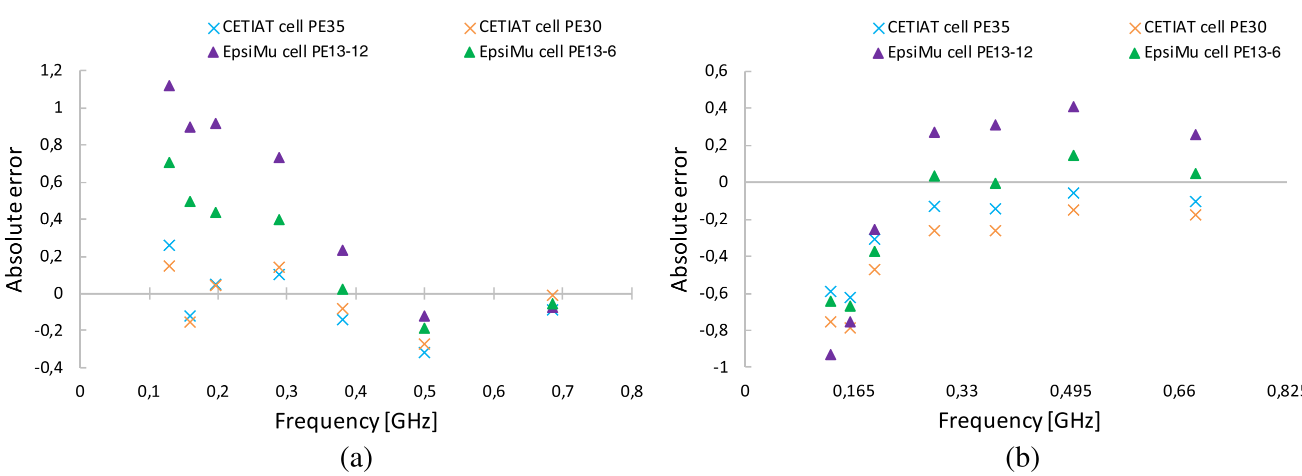


Figure 10. Measurement errors with respect to the reference value for heptanol at 20°C.

3.2. Cyclohexanol

Cyclohexanol (CAS number 108-93-0) was the third alcohol that we tested the measuring cell with. Figure 11 presents the results.

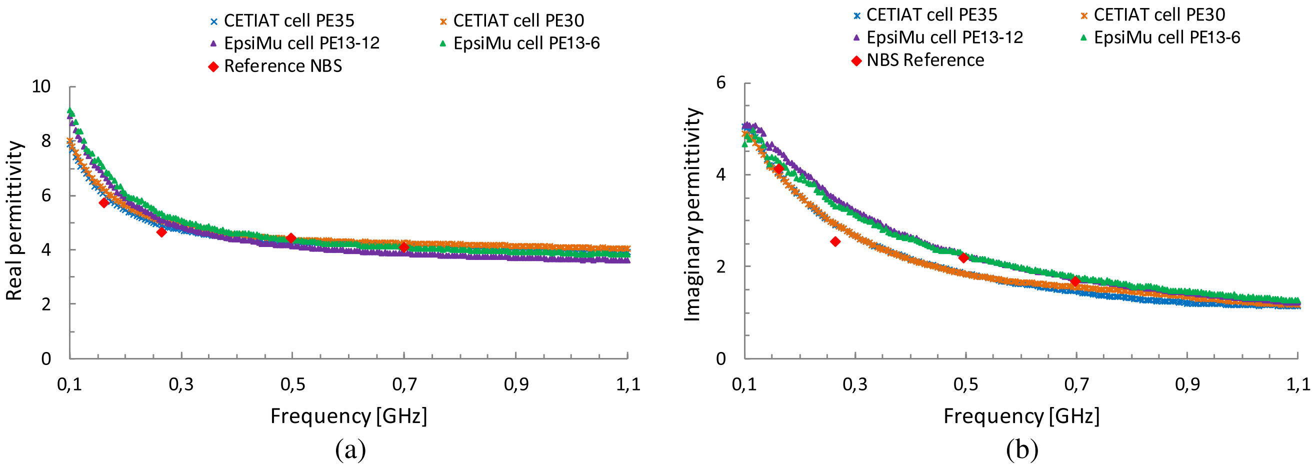


Figure 11. Comparison between the two coaxial cells for cyclohexanol at 20°C. (a) Real part of the permittivity. (b) Imaginary part of the permittivity.

Figure 12 indicates that the real and imaginary parts of permittivity measured using the 1″5/8 cell are remarkably close to the reference values over the entire range of frequencies. This suggests that the measurement method used is reliable and accurate for measuring the properties of cyclohexanol and that the substance may have relatively stable properties at this temperature and over this frequency range.

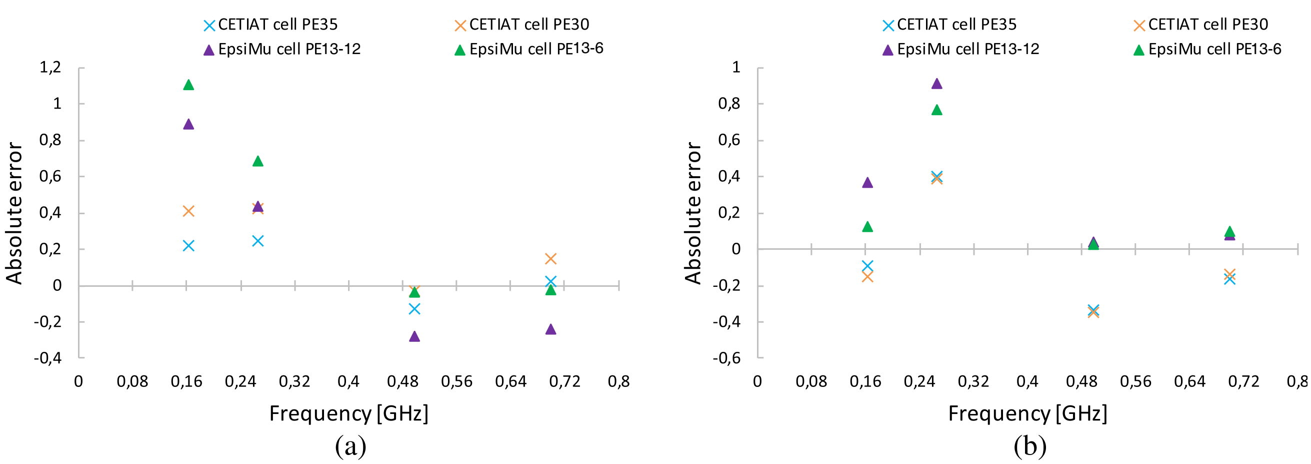
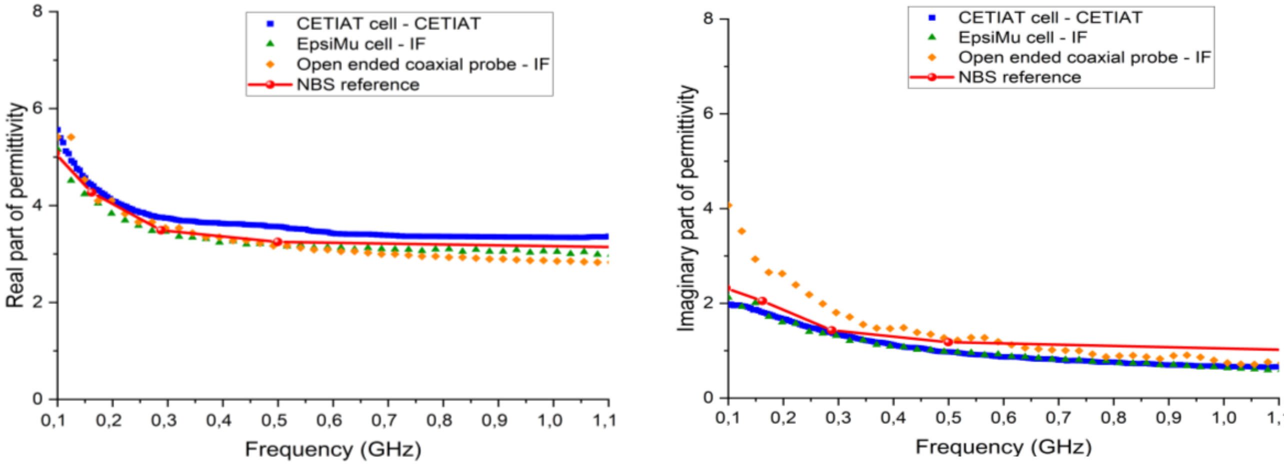


Figure 12. Measurement errors with respect to the reference value for cyclohexanol at 20°C.

Similarly, in the case of cyclohexanol, the experimental results obtained with the *EpsiMu*® cell remain significant at frequencies below 200 MHz.

However, across all the results, it is observed that while certain trends may be present, the differences between the results obtained with the two sample holders for the same cell are considered non-significant. These findings suggest that the choice of sample holder may not have a significant impact on the accuracy of measurements, at least within the scope of the experiments described in this article.



(a) (b)

Figure 13. Inter-laboratory comparison for dielectric parameter of decanol at 25°C. (a) Real part of permittivity. (b) Imaginary part of permittivity.

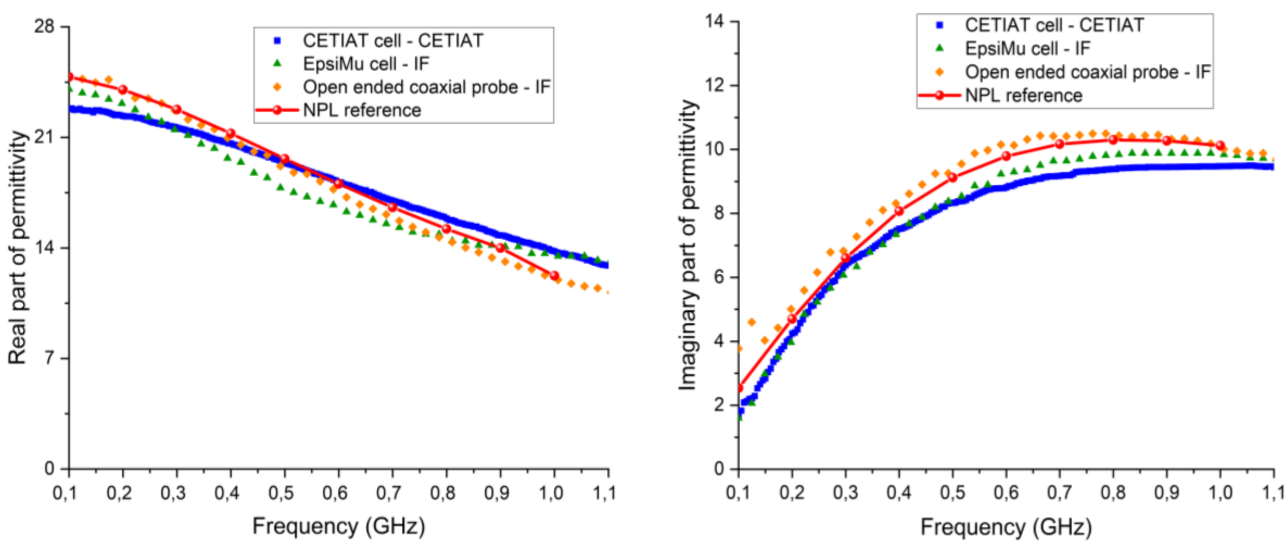
3.3. Inter-Laboratory Comparison

An inter-laboratory comparison was carried out between the measurements performed at *LNE-CETIAT* and those at the Fresnel Institute to ensure the accuracy and consistency of the techniques. In this section, we present the results of measuring decanol and ethanol using three different techniques for dielectric measurements: the 1″5/8 coaxial cell, *EpsiMu*®, and the open-ended coaxial probe *DAK 3,5*.

3.3.1. 1-Decanol

The results for decanol are presented in the figure below (Figure 14).

The real part of permittivity results, obtained using the three methods, are in good agreement



(a) (b)

Figure 14. Inter-laboratory comparison for dielectric parameter of ethanol. (a) Real part of permittivity. (b) Imaginary part of permittivity.

with the reference. The measured imaginary part of permittivity values with an open-ended coaxial probe below 300 MHz show a significant variation, which is expected as on the DAK probe 3.5 mm specifications the lower boundary is set for 200 MHz.

3.3.2. Ethanol

The measurement results of ethanol's complex permittivity at 20°C in the [100 MHz–1.1 GHz] band are presented in the curves in Figure 14 and compared to the *NPL*'s reference values.

The measurement results with ethanol, using the open-ended coaxial probe, show, for both parts of permittivity, that the measured values are very close to the *NPL* reference ones. Nevertheless, for the two coaxial cells, the relative error to the reference is less than 2 for the real part and less than 1 for the imaginary part, in the case of ethanol.

Both open ended coaxial probe and coaxial line techniques have different accuracies. The open ended coaxial probe technique is based on a calibration against a known material (such as short circuit, open circuit, water content, etc.), while the coaxial propagation line technique is independent of all reference values. Furthermore, the comparison of different techniques, in terms of both hardware and data processing, increases the confidence in each measurement method developed and implemented in both the laboratories.

3.4. Sensitivity of the Measuring System to Water

The sensitivity of the 1″5/8 coaxial cell to water content was investigated by testing various ethanol concentrations. Figure 15 illustrates the results of measuring the complex dielectric permittivity of these ethanol concentrations at 20°C within the frequency range of 100 MHz to 1.1 GHz.

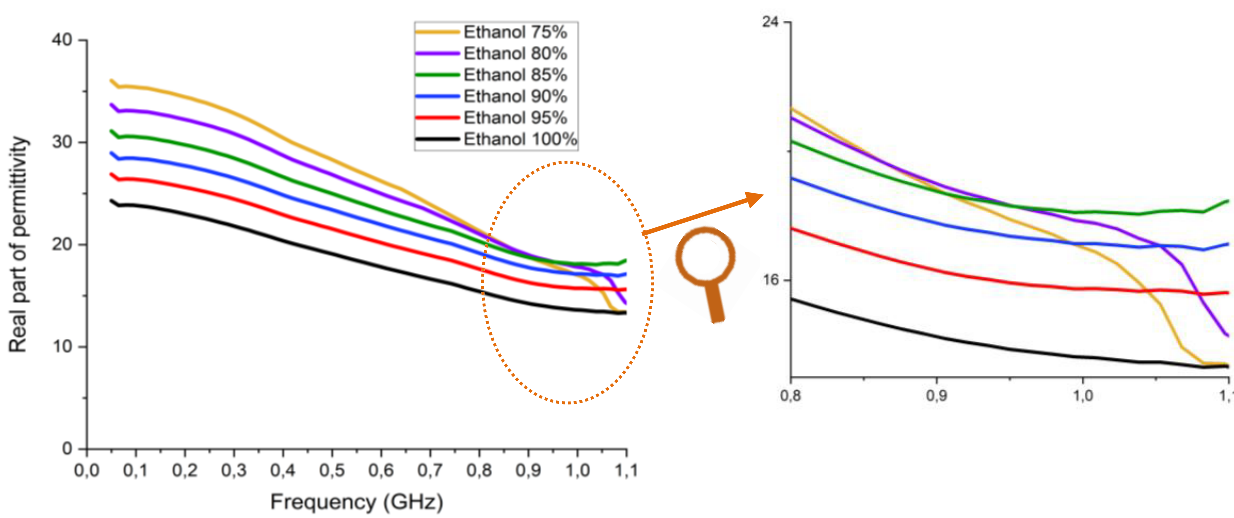


Figure 15. Study of the sensitivity of 1″5/8 LNE-CETIAT coaxial cell to water — results obtained with different concentrations of ethanol.

The measurement outcomes for different ethanol concentrations validate the correlation between water content and dielectric constant (real part of permittivity). These results also demonstrate that the frequency spectrum for this fluid type follows a consistent model. Furthermore, the measurement results for ethanol with varying water contents reveal an increase in the real part of permittivity as moisture content rises. This finding supports the selection of the specified frequency range outlined in Table 1. This choice aims to avoid a phase rotation phenomenon that occurs when the electrical length of the sample reaches half the wavelength, as indicated in Figure 15(b) [20].

The conducted experiments investigated the sensitivity of the 1″5/8 coaxial cell to water content in ethanol. The measured complex dielectric permittivity of different ethanol concentrations confirmed a clear relationship between water content and the real part of permittivity. The results also showed that the chosen frequency range was appropriate to prevent phase rotation issues. These findings contribute to the understanding of how moisture affects the dielectric properties of ethanol and highlight the reliability of the measurement method employed.

The work done here is crucial for improving the validation of the 1″5/8 cell for dielectric measurements in liquids. The 1″5/8 cell has the advantage of being able to measure samples with a significantly bigger volume than the *EpsiMu*® cell can but with a frequency range smaller (Table 1). On the contrary to the coaxial probe, which can only be used with liquids, the sample under test can be either solid or liquid when using the 1″5/8*LNE-CETIAT*'s cell. Moreover, this comparison gives confidence to reduce the typical error below 5% for these techniques.

4. CONCLUSION

This work was carried out in the framework of the *European BIOFMET* project, which focuses on the study of solid and liquid biofuels in industrial applications. This paper compares two approaches for propagation line measurement (two cells and two software) and an open coaxial probe technique. Although the uncertainty assessment is not yet complete, the coherence of the data from different methodologies has been shown. It demonstrates the validation of the techniques described in this paper. In addition, the sensitivity of the measurement to the water content of the cell developed at *LNE-CETIAT* has also been studied. Based on these results, these techniques will be used in the *BIOFMET* project to measure permittivity and moisture content in real samples of liquid biofuels.

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