A Comparative Analysis Between Customized and Commercial Systems for Complex Permittivity Measurements on Liquid Samples at Microwave Frequencies

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***Abstract*—In this paper, different customized systems for microwave permittivity measurements on liquid samples, based on reflectometric measurements, are presented and analyzed. Their performance is compared against the one deriving from the most widely adopted commercial measurement setup. The systems are designed with the aim of providing less expensive solutions without compromising measurement accuracy. The purpose of the first proposed solution is to replace the commercial measurement software exploiting a reformulation of the classical theory. Based on this alternative formulation, a "homemade" probe is built by properly modifying an N-type coaxial connector, thus providing a system requiring a lower quantity of liquid under test. Moreover, a different experimental approach which uses time-domain reflectometry (TDR) instrumentation is presented. Such solution is by far the least expensive, as it allows avoiding the use of costly instrumentation (such as a vector network analyzer). In order to metrologically characterize the proposed solutions, a series of repeated measurements is performed on a set of well-referenced liquids. After extracting the Cole–Cole parameters through each of the considered measurement methods, the resulting type A uncertainty is evaluated. Finally, comparison with literature data allows the estimation of measurement bias. The analysis evidences that custom solutions generally exhibit an accuracy comparable to the one of the commercial solution, with a slight degradation of performance for the TDR-based setup, which, however, compensates for this drawback with its appealing low cost.**

***Index Terms*—Open-ended coaxial probe, permittivity measurement, reflectometry, short-circuited coaxial probe, uncertainty evaluation.**

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I. Introduction

In THE last few years, there has been a growing interest toward the development of methodologies for noninvasive electromagnetic diagnostics based on dielectric spectroscopy, particularly operating in the microwave frequency bands. Such methodologies should allow the detection of the preservation status and quality of materials, possibly in a continuous way and directly on site, resorting to techniques and instrumentation with affordable costs, ease of fabrication, and widespread availability.

As a result, microwave dielectric spectroscopy has become an effective method for the qualitative characterization of liquids, which, differently from solids, exhibit an intricate short-range order only, due to the balance of disordering thermal effects on the one hand and structure promoting effects from intermolecular forces on the other hand.

Dielectric spectroscopy has been used for a number of quality control purposes; for example, measurements of the complex dielectric permittivity, $\varepsilon^*(f)$, have been used for quality and antiadulteration control of vegetable oils [1]–[3], for controlling the osmotic dehydration of fruit [4], for assisting the radio-frequency extraction of chemical compounds to be used in pharmaceuticals [5], for real-time control of levels and quality of liquids in industrial tanks [6], etc.

Particularly interesting applications of dielectric spectroscopy are related to monitoring and diagnosis in biology and medical applications [7]–[17]. The measurement of cell dielectric properties, apart from disclosing important information on cell physiology [7], may provide a screening tool for rapid detection and monitoring of cellular and molecular markers in living cells [8]–[12]. As an example, the process of monitoring the deterioration of hematocrit [13], as well as blood cell suspension used in transfusion processes [14], the identification of morphological cell changes for the early detection of anemia and leukemia [15], [16], and the accurate and fast screening of excised tissues from biopsy [17] are just some of the medicine-related applications based on dielectric spectroscopy, which are currently being used.

It is worth noting that, for biology- and medicine-related applications, the possibility of performing dielectric measurements on small quantities of sample is extremely advantageous both

because of the difficulties in having high volumes of biological suspension or tissues and for the need to limit the electrode polarization effect that influences the low-frequency dielectric response of the highly conductive biological suspensions.

Currently, the most popular and commercially available setup for microwave permittivity measurements in liquid samples is based on an open-ended coaxial probe, connected to a vector network analyzer (VNA), which has a tip immersed in the liquid [18], [19]. The frequency-dependent complex permittivity is derived from the measured reflection coefficients with the probe immersed in the liquid under test (LUT) and in a calibration liquid. This commercial setup is rather expensive because of the high cost of the dielectric probe, of the VNA, and of the associated management software.

Starting from these considerations, the aim of this paper is to present and metrologically characterize possible customized "alternatives" characterized by lower costs and by the possibility to operate with extremely small volumes of liquid sample. In particular, such alternatives are built aiming at quality control and diagnostic applications, and their low cost might pave the way toward their use as actual substitutes for the commercial setup in large-scale applications, possibly even on site.

The first proposed solution aims at replacing the commercial measurement software and is based on a reformulation of the classical lumped-element theory behind open-ended coaxial probes [19], which avoids the need for a preliminary probe fringing capacitance characterization, thus easily allowing the use of customized coaxial probes. The only drawback related to this approach is a slightly more onerous calibration procedure, involving two reference liquids instead of only one. On the other hand, the "modified" procedure is potentially more accurate, as it characterizes the probe at each calibration session, thus accounting for probe aging effects.

Based on this alternative formulation, a custom-made probe is built by properly modifying an N-type coaxial connector; in this way, the costly commercial probe is substituted with an inexpensive probe which, as a further advantage, also requires a lower quantity of LUT. This solution still requires VNA measurements.

In order to investigate another alternative solution requiring only low-cost instrumentation, a different experimental approach is finally presented. Such approach resorts to a portion of liquid-filled coaxial line in conjunction with a time-domain reflectometry (TDR) piece of equipment. This solution represents a truly low-cost and portable permittivity measurement system, which overcomes the need of using high-purity reference liquids for calibration and the need of a VNA.

In order to characterize the examined systems on the basis of a set of parameters, the measured permittivity spectrum is modeled through the well-known Cole–Cole formula [20]. The evaluation of Cole–Cole parameters is performed through a curve fitting of the experimental data with the Cole–Cole formula, exploiting a suitable minimization procedure. Indeed, it is important to point out that such parameters prove effective for describing the dielectric characteristics of most liquids in the microwave range and are related to the liquid quality and status, thus making them a possible choice for quality control and diagnostic applications.

The proposed measurement solutions are described in Section II, while in Section III, a series of experimental tests carried out on a set of well-referenced liquids using both the customized systems and the commercial setup is reported. These tests allow comparing the performance of the customized solutions with that of the widely used commercial kit. Particular focus is given to the assessment of the typical uncertainty associated with the different measurement methods, and some considerations are made on the advantages of the proposed solutions with respect to the widely used commercial kit. Finally, in Section IV, some conclusions are drawn on the validity, the applicability, and the possibility of improvement of the two customized systems.

II. Permittivity Measurement Techniques and Systems

In this section, the adopted permittivity measurement solutions are described. First, the main theories behind the open-ended coaxial probe system are briefly reviewed and analyzed. Then, the proposed customized solutions are discussed, and their advantages and drawbacks are highlighted.

A. Open-Ended Coaxial Probe Theory

The commercial system for complex permittivity measurements, widely adopted by many researchers and used in this paper as a reference for the comparative analysis, is the Agilent 85070E dielectric probe kit (DPK) [18]. Such system employs an open-ended coaxial probe with its tip immersed in the LUT [19], [21], [22]. The probe is connected to a VNA, which measures the frequency-dependent reflection coefficient at the coaxial probe connector and converts it to a complex permittivity spectrum through the measurement software provided with the DPK. The simplest schematic representation of the measurement system is the lumped-element one, shown in Fig. 1 (radiation effects are not considered as they are usually negligible in the useful frequency range). The target of the measurement is the reflection coefficient $\Gamma_{LUT}(f)$ at the probe tip immersed in the LUT (calibration plane). Starting from such reflection coefficient, the complex relative permittivity of the LUT ($\varepsilon^*_{LUT}(f)$) is readily derived as [23]

$$\varepsilon^*_{LUT}(f) = \frac{1-\Gamma_{LUT}(f)}{j\omega Z_0 C_0 \left[1+\Gamma_{LUT}(f)\right]} - \frac{C_f}{C_0} \tag{1}$$

where ω is the angular frequency, Z_0 is the characteristic impedance of the coaxial probe (usually equal to 50 Ω), and C_f and C_0 are the internal and external fringing capacitances of the probe, respectively.

Several more complex and accurate models of the open-ended coaxial probe have been developed. Some of them rely on a full electromagnetic study of the probe–medium interface. However, in order to keep the problem computationally affordable, many simplifying assumptions are often made, and the final model is empirically corrected, on the basis of a series of measurements on known samples, to better match the real behavior. This kind of approach is most commonly

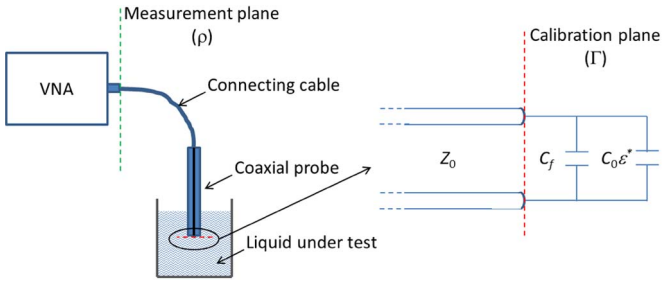

Fig. 1. Lumped-element scheme of the open-ended coaxial probe measurement setup.

followed for flanged probes [24]. For the case of flangeless probes, instead, empirical nonelectromagnetic models have been proposed. These models, once again, need a series of measurements on well-known samples in order to be fine tuned to the specific probe [25], [26].

The DPK software is based on the aforementioned electromagnetic/empirical models and allows a measurement procedure that is fully automated and directly yields the complex permittivity values as a function of frequency. To achieve this result, the DPK software takes into account a suitable model of the probe, which is directly characterized by the manufacturer. This prevents the use of the software with custom probes. The actual DPK measurement procedure includes a preliminary calibration step, which has to be carried out before each measurement session. Fig. 1 clearly shows, in fact, that the "actually measured" reflection coefficient $(\rho_{LUT}(f))$ refers to the one at the VNA connector (measurement plane). Therefore, it is necessary to de-embed the effect of the coaxial probe length (plus additional systematic errors) through a standard vector error-correction (calibration) procedure. The traditional short–open–load (SOL) one-port calibration procedure [27] cannot be directly applied at the probe tip. To circumvent this problem, the three standard SOL loads are replaced by three measurements performed with the tip short-circuited (via a suitable shorting block provided with the DPK), open in air, and immersed in a calibration liquid with well-characterized permittivity spectrum (usually distilled water), respectively [23].

The DPK includes different coaxial probes, having different dimensions and operating bandwidths. In particular, for the experiments reported herein, the so-called "slim-form probe" has been used (500 MHz–50 GHz frequency range), connected to an Agilent PNA-L N5230A VNA, whose operating range is limited to 6 GHz. The overall useful measurement bandwidth (whose lower and higher limits are fixed by the probe frequency limit and by the VNA frequency range, respectively) goes from 500 MHz to 6 GHz. The minimum sample size requirements, according to the probe specifications, correspond to a sample volume of about 1 mL (the minimum 5 mm of sample is required all around the probe tip). However, it should be pointed out that, if a sample container with the minimum required volume is adopted, particular care has to be taken in order to ensure that the probe tip is immersed at the prescribed depth. Otherwise, measurements can be carried out more easily, and without possible perturbations due to the boundary effect, employing larger volumes of liquid sample.

Analysis of the commercial solution evidences some clear advantages, namely, the very large operating frequency range and the relatively simple calibration procedure, requiring only one calibration liquid. However, some distinct drawbacks are present. The solution is very expensive because of the costs of both the VNA and the DPK; for ease of use, rather large sample volumes (around 10–100 mL) are required; and finally, the probe is characterized only once at the factory, thus possibly impairing measurement accuracy due to probe aging effects.

On the basis of these considerations, some alternative solutions are proposed aimed at circumventing some of the aforementioned DPK drawbacks.

B. Modified Lumped-Element Formulation for the Open-Ended Coaxial Probe

The first proposed customized solution aims at replacing the commercial measurement software and is based on a reformulation of the classical lumped-element theory behind open-ended coaxial probes, which avoids the need for a preliminary probe fringing capacitance characterization. This allows the use of customized uncharacterized coaxial probes (thus allowing a complete replacement of the DPK, both of the software and of the hardware components), with a consequent reduction in costs. The basic idea is to shift the calibration plane between C_f and C_0. In this way, C_f is included in the error-correction parameters with its actual measured frequency behavior. If three calibration liquids (or, more conveniently, only two reference liquids plus air) with *a priori* known frequency-dependent complex permittivity $\epsilon_i^*(f)$ $(i = 1, 2, 3)$ are considered, the corresponding theoretical reflection coefficients at the calibration plane are [23]

$$\Gamma_i(f) = \frac{1 - j\omega Z_0 C_0 \epsilon_i^*(f)}{1 + j\omega Z_0 C_0 \epsilon_i^*(f)}. \tag{2}$$

Starting from the corresponding measured reflection coefficients $\rho_i(f)$, the scattering parameters of the error network can be derived as (3)–(5) shown at the bottom of the next page [23]. Then, after measuring the reflection coefficient $\rho_{LUT}(f)$ with the probe immersed in the LUT, the corrected reflection coefficient $\Gamma_{LUT}(f)$ at the calibration plane is obtained from [23]

$$\Gamma_{LUT}(f) = \frac{\rho_{LUT}(f) - S_{11}(f)}{S_{22}(f)\rho_{LUT}(f) + S_{12}S_{21}(f) - S_{11}(f)S_{22}(f)}. \tag{6}$$

Finally, the desired $\varepsilon_{LUT}^*(f)$ is obtained from the following relation, identical to (1) but with the removal of the C_f term:

$$\varepsilon_{LUT}^*(f) = \frac{1 - \Gamma_{LUT}(f)}{j\omega Z_0 C_0 \left[1 + \Gamma_{LUT}(f)\right]}. \tag{7}$$

Substituting (2)–(6) inside (7), after simplifications, yields (8) shown at the bottom of the next page. It is important to note that, in the final expression, the external fringing capacitance C_0 is cancelled since it appears both in calibration and in LUT measurements. This implies that such capacitance is intrinsically characterized, with its actual frequency behavior, at every calibration session, thus eliminating possible inaccuracies

caused by probe aging and allowing its accurate estimation as a function of frequency. It is believed that this represents a major advantage compared to the alternative approach of performing a self-characterization of the probe, which is usually done on the basis of measurements of the resonance frequency of the probe with the tip short-circuited, open in air, and immersed in a low-loss liquid sample of known permittivity [22]. The only drawback is a slightly more complex calibration procedure, involving two reference liquids instead of only one. The whole measurement procedure, including a library of temperature-dependent Cole–Cole parameters for a predefined set of possible calibration liquids, has been implemented in Matlab. As a side note, the result (8) obtained with the aforementioned theory is equivalent to the one derived in [28] and [29] using a slightly different approach.

With regard to (8), it is clear that some uncertainty on measured permittivity will arise from uncertainties in reflection coefficient measurements and in the permittivity of reference liquids. The first contribution will mainly have an effect on measurement repeatability and measurement drift, which can be kept under control through the careful selection of VNA intermediate frequency (IF) bandwidth and averaging and through frequent recalibration, respectively. Owing to the high accuracy of VNAs, however, the main contribution to uncertainty is expected to be the one related to the uncertainty in the permittivity of reference liquids. The first mandatory step is therefore the use of well-characterized calibration liquids, with high purity and freshly opened, together with a tight control of liquid temperature. However, it is interesting to evaluate the sensitivity coefficients of the combined uncertainty of ε^*_{LUT} with respect to the ϵ^*_i. The corresponding analysis, carried out in [29], reveals that accuracy can be improved by selecting three calibration liquids with largely different permittivities such that one of these liquids has a permittivity as close as possible to that of the LUT.

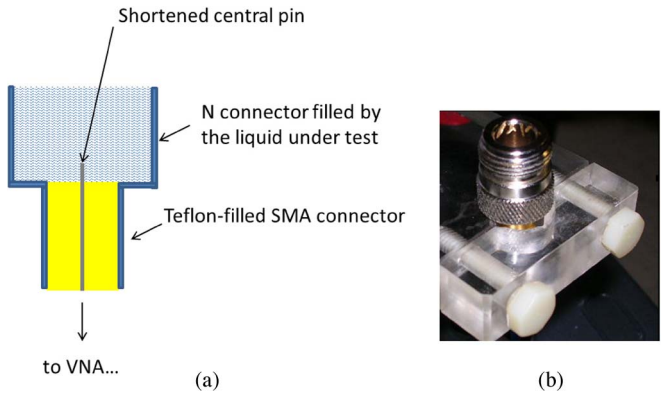

Fig. 2. (a) Scheme of the custom-made open-ended probe and (b) picture of the manufactured probe held in its fixture.

C. Customized Open-Ended Probe

Exploiting the measurement technique described in Section II-B, it is possible to use a customized coaxial probe, which might be easily fabricated starting from a length of semirigid coaxial cable (RG402, for example) properly connectorized at one end. However, one of the aims was to develop a probe that would easily allow the measurement of small quantities of liquid sample. To this end, a suitable probe has been built starting from a standard sub-miniature version A (SMA) to N-type female transition, with the central pin of the N connector properly shortened in order to have a length much smaller than the wavelength in the operating frequency range [30], [31]. In this way, the modified N connector acts as a sample holder with a capacity of mere 0.6 mL. The probe is schematically represented in Fig. 2(a), where it is evident that the SMA connector acts as an open-ended coaxial probe, while the modified N connector is the sample holder. A picture of the manufactured probe, held in a specifically developed fixture, is shown in Fig. 2(b).

$$S_{11}(f) = \frac{\Gamma_1(f)\Gamma_2(f)\rho_3(f)\,[\rho_1(f)-\rho_2(f)] + \Gamma_1(f)\Gamma_3(f)\rho_2(f)\,[\rho_3(f)-\rho_1(f)] + \Gamma_2(f)\Gamma_3(f)\rho_1(f)\,[\rho_2(f)-\rho_3(f)]}{\Gamma_1(f)\Gamma_2(f)\,[\rho_1(f)-\rho_2(f)] + \Gamma_1(f)\Gamma_3(f)\,[\rho_3(f)-\rho_1(f)] + \Gamma_2(f)\Gamma_3(f)\,[\rho_2(f)-\rho_3(f)]} \tag{3}$$

$$S_{22}(f) = \frac{\Gamma_1(f)\,[\rho_2(f)-S_{11}(f)] + \Gamma_2(f)\,[S_{11}(f)-\rho_1(f)]}{\Gamma_1(f)\Gamma_2(f)\,[\rho_2(f)-\rho_1(f)]} \tag{4}$$

$$S_{12}S_{21}(f) = \frac{[\rho_1(f)-S_{11}(f)]\,[1-S_{22}(f)\Gamma_1(f)]}{\Gamma_1(f)} \tag{5}$$

$$\varepsilon^*_{LUT}(f) = \frac{\varepsilon^*_1(f)\varepsilon^*_2(f)\,[\rho_2(f)-\rho_1(f)]\,[\rho_3(f)-\rho_{LUT}(f)] + \varepsilon^*_1(f)\varepsilon^*_3(f)\,[\rho_1(f)-\rho_3(f)]\,[\rho_2(f)-\rho_{LUT}(f)] + \varepsilon^*_2(f)\varepsilon^*_3(f)\,[\rho_3(f)-\rho_2(f)]\,[\rho_1(f)-\rho_{LUT}(f)]}{\varepsilon^*_1(f)\,[\rho_2(f)-\rho_3(f)]\,[\rho_1(f)-\rho_{LUT}(f)] + \varepsilon^*_2(f)\,[\rho_3(f)-\rho_1(f)]\,[\rho_2(f)-\rho_{LUT}(f)] + \varepsilon^*_3(f)\,[\rho_1(f)-\rho_2(f)]\,[\rho_3(f)-\rho_{LUT}(f)]} \tag{8}$$

The slightly protruding central pin, together with the surrounding N-connector metallic walls, results in a fairly large value of the fringing capacitance, which makes the probe ideal also for measurements at relatively low frequencies (excellent results have been obtained down to 100 MHz, compared to the 500-MHz slim-form probe limit). However, the particular configuration of the probe strongly limits its use at higher frequencies. The coaxial probe tip, in fact, does not terminate in an ideally infinite half space filled by the sample, as in the classical open-ended probe, but a length of sample-filled circular waveguide (the portion of the N connector with no pin) is present. Therefore, the electromagnetic field coming from the coaxial cable excites a series of modes in this circular waveguide. As long as such modes are under cutoff, they are damped along the waveguide length, preventing them from reaching the N-connector boundary and eventually radiating in free space. Therefore, the key points, in order to avoid consistent radiation effects, are that the frequency is low enough to ensure that the sample-filled circular waveguide is sufficiently below the fundamental mode cutoff, and that the sample holder (i.e., the upper N connector) is long enough to attenuate evanescent modes. The probe has been designed with such targets in mind (further details can be found in [30]). The theoretical cutoff frequency in air for the designed probe is about 20 GHz, but unfortunately, it scales as the square root of the LUT permittivity. Nonetheless, developing an alternative probe for higher frequencies starting from SMA or microcoax connectors is rather simple, at the expense of a decrease in fringing capacitance and a corresponding increase in the lower frequency operating limit. In practice, the customized probe should be tailored to the specific frequency range and LUT permittivity.

D. Liquid-Filled Coaxial Line

The solution depicted in Section II-B and C provides a low-cost alternative to the DPK with similar expected accuracy, but it does not remove the need of the VNA. Moreover, a couple of high-purity reference liquids must always be available for performing calibration. In order to overcome these two limitations and develop a truly low-cost and portable permittivity measurement system, a completely different solution is proposed. First of all, reflectometric measurements are performed with a TDR module, which is available in low-cost versions with adequate bandwidth. Second, the open-ended coaxial probe is replaced by a short-circuited liquid-filled section of coaxial line [19], [32]. The designed probe is a 66.4-mm-long portion of coaxial line, short-circuited at the distal end and equipped with a standard SMA connector [31], [33]. The probe is filled with the LUT and then connected to the TDR module (in particular, a Tektronix DSA8200 digital serial analyzer sampling oscilloscope, equipped with a Tektronix TDR 80E04 module, is used in the experiments presented in the following). A model of the probe is depicted in Fig. 3(a) while its picture is shown in Fig. 3(b). The Teflon inside the SMA connector that is mated to the probe acts as a seal, thus preventing the liquid from leaking outside. This "sealed design" makes it also quite easy to control the temperature of the LUT since it is sufficient to immerge the whole probe inside a thermostatic bath.

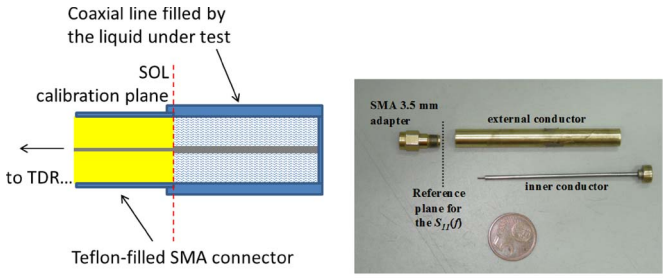

Fig. 3. (a) Scheme of the custom-made short-circuited probe and (b) picture of the manufactured probe.

The probe allows the direct estimation of the static permittivity (ε_s) on the basis of the measured time-domain reflection coefficient value Γ_s corresponding to the central section of the probe, before reflection from the end shorting plane. In particular, ε_s can be evaluated as

$$\epsilon_s = \left(\frac{Z_p}{Z_0}\right)^2 \left(\frac{1-\Gamma_s}{1+\Gamma_s}\right)^2 \tag{9}$$

where Z_p is the probe characteristic impedance in air and Z_0 is the characteristic impedance of the SMA connector (i.e., 50 Ω). From (9), it is evident that a preliminary characterization of the probe is necessary in order to estimate Z_p. Such characterization, however, only requires a simple TDR acquisition with the empty probe (i.e., filled with air).

A more complete picture of the permittivity spectrum of the DUT can be obtained converting the TDR-recorded measurements to the frequency domain by using a suitable procedure developed in Matlab, which also takes into account a standard SOL calibration step [33]. Indeed, the desired measurement plane exactly lies at the coaxial probe SMA connector, and therefore, SOL standards for a conventional one-port calibration are readily available. Starting from the $\Gamma(f)$ evaluated in the previous step, it is possible to derive the complex relative permittivity of the LUT solving the following implicit equation [32]:

$$\Gamma(f) = \frac{Z_p \tanh(\gamma L) - Z_0\sqrt{\varepsilon^*(f)}}{Z_p \tanh(\gamma L) + Z_0\sqrt{\varepsilon^*(f)}} \tag{10}$$

where $\gamma = j\sqrt{\varepsilon^*(f)}\omega/c_0$, with c_0 representing the speed of light in vacuum, and L is the probe length.

As a side note, it is worth mentioning that a more general formulation of (10) is usually adopted, accounting for the possibility to have an additional air region between the liquid-filled portion and the end shorting plane [32]. This more complex probe design can substantially decrease the uncertainty contribution due to the reflection coefficient measurements, requiring, however, accurate knowledge of the additional air region length. In particular, in [32], it is shown that the sensitivity coefficient of the measured permittivity with respect to $\Gamma(f)$ can be minimized by choosing the air region length equal to one quarter of the wavelength. This result, however, holds only for sample lengths much smaller than the wavelength inside

the medium and obviously can only be achieved at a particular frequency. For the case here presented, where the probe length is about 7 cm and the considered frequency range lies between 500 MHz and 3 GHz, the wavelength inside the tested liquids (approximately ranging between 1 and 30 cm) is comparable with the sample length. Under these conditions, the sensitivity coefficient values remain sufficiently small also when the air region is not present [32].

Unfortunately, (10) has no closed-form analytical solution and must therefore be solved numerically. Here, in particular, a minimization procedure based on the simplex method [34] developed in Matlab has been used. The described procedure does not need any calibration with reference liquids apart from the standard SOL procedure. However, it is well known that the short-circuited probe approach has severe limitations in its accuracy, particularly concerning the imaginary part of permittivity, because of losses in the coaxial probe walls [35]. Appropriate corrections can be applied, but as long as the final aim is the estimation of Cole–Cole parameters, the frequency position of the peak in the imaginary part is a reliable indicator of the relaxation frequency f_r, even with no corrections for probe losses. As concerns the operating bandwidth, the short-circuited liquid-filled coaxial probe has a low-frequency limit related to the errors in the evaluated $\Gamma(f)$ at low frequencies due to the truncation of the TDR waveform. However, this is not a real problem since the TDR waveform itself gives enough information for the evaluation of ε_s. The high-frequency limit, instead, similarly to the modified N-connector probe, is linked to the excitation of higher order modes in the coaxial line, which alter the probe model. For the particular probe here described, the theoretical cutoff frequency in air is about 20 GHz, but again, it scales as the square root of the LUT permittivity. It must also be stressed that a further limitation in frequency arises from the parasitic effects present at the probe head due to inevitable mismatches between the TDR module SMA connector and the probe connector.

It is worth mentioning that the same probe has been previously used within a different theoretical formulation, which, however, required a preliminary characterization procedure carried out through measurements on well-referenced liquids to estimate the probe head parasitic effects [31], [33]. However, this more complex procedure does not yield significant improvements in the final accuracy, as evidenced by a previous metrological characterization [31].

III. Metrological Evaluation on Reference Liquids

In order to test and compare the performances of the different measurement solutions considered, a set of four liquids has been used, namely, chloroform, ethanol, methanol, and tap water. Considering that the frequency band common to all the three systems ranges from 500 MHz to 2 GHz, the reference liquids have been selected according to the following criteria. The first one (chloroform) has been chosen so as to exhibit an almost flat frequency spectrum. The second (ethanol) has been chosen so as to present a relaxation within the measurement bandwidth. The third (methanol) has been chosen so as to present a relaxation at the boundary of the measurement bandwidth. The fourth one (tap water) finally has been chosen so as to present an almost flat frequency spectrum but with a low value of static conductivity. Apart from the aforementioned considerations, all tested liquids have well-documented temperature-dependent Cole–Cole parameters. Moreover, the four liquids cover a large range of permittivity values, roughly ranging from 4 to 80. For each liquid, a series of ten repeated measurements has been performed, and the measurement uncertainty on each estimated Cole–Cole parameter has been evaluated, through a type A (experimental) approach [36]. To estimate the measurement bias, the mean value of each Cole–Cole parameter has been compared with well-referenced data, thus taking into account also the systematic effects, overlooked by the type A uncertainty evaluation. The reference Cole–Cole parameters have been taken from available literature data [37]–[40], using linear interpolation when data were not available at the required temperature.

The next two sections report the results obtained with the solutions employing the open-ended coaxial probe approach and the short-circuited liquid-filled coaxial line one.

A. Experimental Results for the Open-Ended Coaxial Probes

A first series of repeated experimental tests on the set of liquids has been performed with the aim of comparing the modified approach discussed in Section II-B with the classical DPK approach. To this end, measurements have been carried out with the slim-form probe, evaluating the complex permittivity spectrum both by means of the DPK software and using the customized algorithm developed in Matlab. The calibration liquid used for DPK measurements is distilled water, while for the customized approach, the two chosen calibration liquids have been acetone and distilled water. Such liquids, along with air, cover a large permittivity range, and the respective permittivities are sufficiently different to ensure an optimal accuracy (see discussion at the end of Section II-B). As concerns the measured liquids, their temperature was monitored throughout the experiments using a thermistor, which was inserted in the beaker containing the LUT immediately after each measurement had been performed. Recorded temperatures are (21.3 ± 0.3) °C for chloroform, (21.9 ± 0.7) °C for ethanol and methanol, and (20.4 ± 0.2) °C for water. In order to avoid any uncertainty due to boundary effects and to ease probe positioning, a large quantity of liquid (200 mL) was used. Before each measurement, the probe was carefully cleaned employing acetone and then dried. Additionally, measurements with the probe in air were performed before starting a measurement session with each new liquid, in order to ensure that the probe was properly cleaned. The VNA was set with an IF bandwidth of 1 kHz and an averaging factor of 10.

As an example, Fig. 4 compares the complex permittivity for ethanol (real and imaginary parts) obtained using the DPK software and the customized algorithm (data refer to the first measurement of the series). The reference theoretical behavior [37] is also reported. The curves clearly show an excellent agreement with reference data and exhibit similar behavior for the DPK and for the customized approaches.

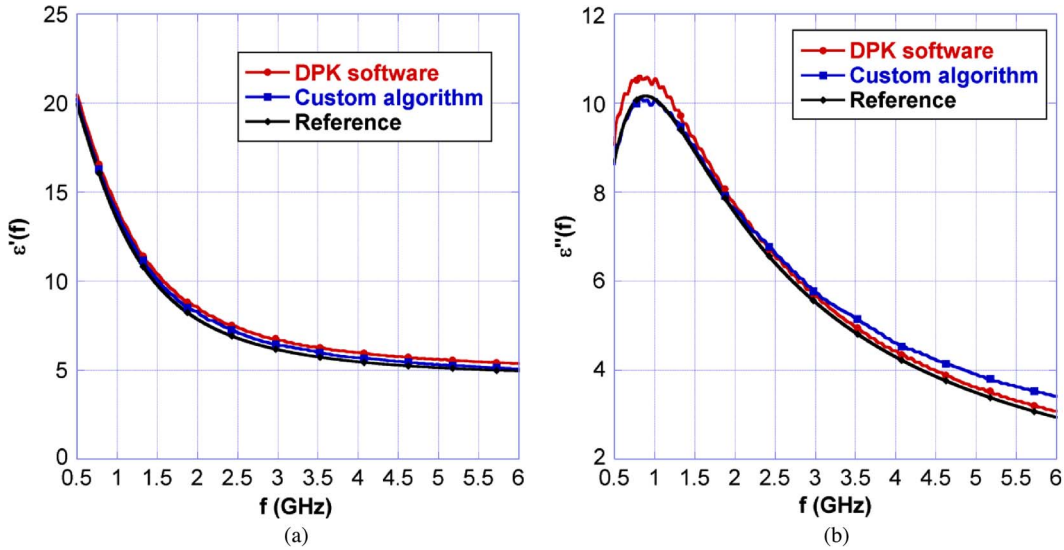

Fig. 4. (a) Real and (b) imaginary parts of the permittivity of ethanol sample: Measurements with the slim-form probe.

TABLE I
COLE–COLE PARAMETERS FOR THE TEST LIQUIDS ESTIMATED FROM SLIM-FORM COAXIAL PROBE MEASUREMENTS WITH DPK SOFTWARE

	ε_s			f_r (GHz)			ε_∞		
	Mean	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**
Chloroform	4.85	0.75%	1.2%	–	–	–	–	–	–
Ethanol	26.4	0.72%	6.1%	0.856	1.4%	3.1%	4.90	0.26%	8.4%
Methanol	33.9	0.39%	2.0%	2.97	0.79%	0.61%	5.99	1.0%	6.6%
Water	80.0	0.42%	0.29%	–	–	–	–	–	–

TABLE II
COLE–COLE PARAMETERS FOR THE TEST LIQUIDS ESTIMATED FROM SLIM-FORM COAXIAL PROBE MEASUREMENTS WITH CUSTOMIZED ALGORITHM

	ε_s			f_r (GHz)			ε_∞		
	Mean	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**
Chloroform	4.83	0.75%	0.59%	–	–	–	–	–	–
Ethanol	25.4	0.10%	2.0%	0.898	0.25%	1.8%	4.38	0.38%	3.1%
Methanol	33.0	0.08%	0.90%	2.99	0.15%	1.5%	5.33	0.13%	5.2%
Water	79.9	0.42%	0.35%	–	–	–	–	–	–

Tables I and II summarize the results obtained on the four liquids in terms of estimated Cole–Cole parameters. Uncertainty values refer to expanded uncertainties, with a coverage factor equal to 2, derived from the type A standard uncertainty of the average of ten repetitions. Biases are computed with reference to literature data. Data in Tables I and II show that the performance of the customized algorithm is comparable with the DPK software, with no loss in accuracy. In particular, measurement repeatability is generally better for the customized algorithm, but this is mainly due to the fact that such algorithm operates on the measured reflection coefficients referring to the average of ten frequency scans, while the averaging feature is not available within the DPK software. Biases for the two methods are very similar, with an overall better accuracy for the customized algorithm. It is worth emphasizing that the adopted slim-form probe had been used for a long time, and therefore, it is likely that its performance degraded (compared to the performance of a new probe). Indeed, a previous series of tests on a slightly different set of liquids, performed employing a brand new probe, revealed an improved accuracy with measurement bias within 5% [31]. However, this evidences that the customized algorithm, owing to the more robust calibration procedure, is able to fully characterize the real behavior of the probe fringing capacitance. As a side note, it must be pointed out that the relaxation frequency and high-frequency permittivity for tap water have not been reported in Tables I and II because they fell largely outside the operating frequency range and, therefore, the minimization procedure for such parameters was too strongly dependent upon the initial estimates.

After validating the performance of the customized permittivity measurement algorithm, a second series of repeated experimental tests on the set of liquids has been performed with the aim of assessing the performance of the "homemade" open-ended coaxial probe based on the modified N-type connector

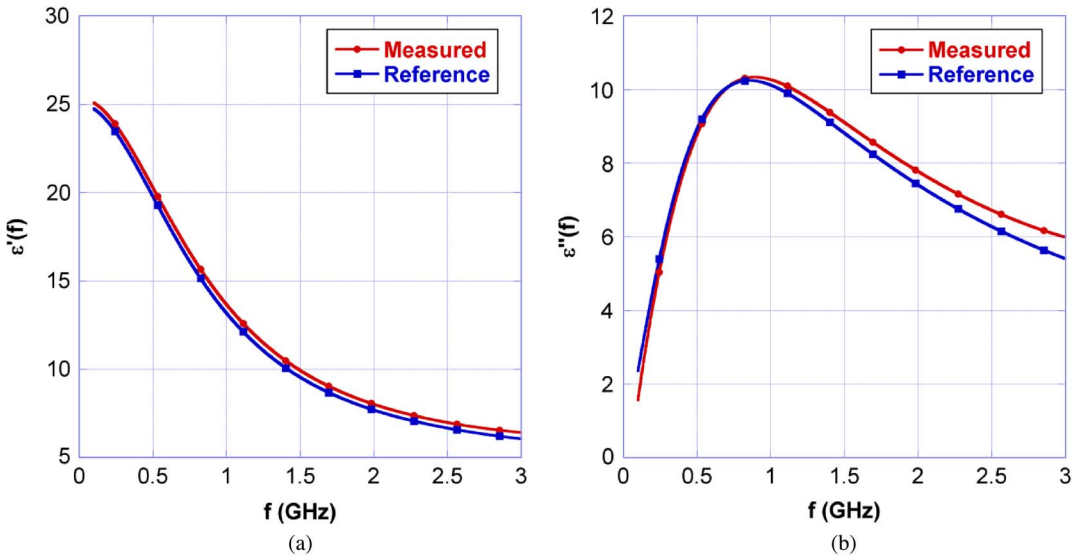

Fig. 5. (a) Real and (b) imaginary parts of the permittivity of ethanol sample: Measurements with the "homemade" open-ended coaxial probe.

TABLE III
COLE–COLE PARAMETERS FOR THE TEST LIQUIDS ESTIMATED FROM "HOMEMADE" OPEN-ENDED COAXIAL PROBE MEASUREMENTS WITH CUSTOMIZED ALGORITHM

	ε_s			f_τ (GHz)			ε_∞		
	Mean	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**	**Mean**	**Unc.**	**Bias**
Chloroform	4.98	0.89%	3.7%	–	–	–	–	–	–
Ethanol	26.2	1.5%	4.2%	0.908	0.56%	9.6%	4.54	0.64%	0.22%
Methanol	35.2	1.1%	4.1%	2.74	0.27%	0.55%	5.70	5.0%	0.75%
Water	82.6	0.40%	4.0%	–	–	–	–	–	–

described in Section II-C. The two calibration liquids used have been once again acetone and distilled water. It must be evidenced that, due to the permittivity-dependent frequency limit of the probe, the use of water as a calibration liquid limits the upper operating frequency to about 2–3 GHz. Similarly to the previous case, the temperature was monitored throughout the experiments using a miniature thermocouple, which was immersed in the liquid inside the probe immediately after each measurement was performed. Recorded temperatures are (21 ± 1) °C for chloroform, (20 ± 1) °C for ethanol, (19 ± 1) °C for methanol, and (21.5 ± 0.5) °C for water. In accordance with the probe container capacitance, 0.6 mL of liquid volume was employed. Before each measurement, the probe was carefully cleaned employing acetone and then dried through high-pressure helium jet. Additionally, measurements with the probe in air were performed before starting a measurement session with each new liquid, in order to ensure that the probe was properly cleaned. The VNA settings were the same as that of the previous measurement session with the slim-form probe.

As an example, Fig. 5 compares the complex permittivity for ethanol (real and imaginary parts) obtained using the "homemade" probe with the corresponding reference theoretical behavior [37]. The curves clearly show a good agreement with reference data, revealing performances similar to that of the slim-form probe, but with a different operating bandwidth. A slight loss of accuracy is visible around 3 GHz, particularly on the imaginary part, as a result of approaching the upper frequency limit for validity of calibration data. Indeed, distilled water, used as a calibration liquid, limits the usable frequency range to about 2–3 GHz, as already mentioned.

Table III summarizes results obtained on the four liquids in terms of estimated Cole–Cole parameters. Uncertainty and bias values are computed with the same approach as that of Tables I and II. Data in Table III show that the performance of the customized probe is comparable with the slim-form probe, with no significant loss in accuracy. In particular, measurement repeatability is generally within 2% with the exception of the relaxation frequency for methanol, whose larger standard deviation must be attributed to the upper frequency limit of the probe, which does not allow a reliable estimation of such parameter. Biases are generally within 5%, with the only exception of the relaxation frequency for ethanol.

From this whole analysis, it can be concluded that the customized approach, at the expense of a more onerous calibration procedure, yields a final accuracy comparable with that of the commercial software, with a potential improvement in case of poorly characterized probes. Also, the "homemade" coaxial probe shows a good accuracy level, with the great advantage of allowing the use of a small quantity of liquid in an easy and controllable way, with the only drawback of a limited useful frequency range. Under this aspect, however, it is worth noting that the modified N-type connector probe is able to yield reliable permittivity measurements down to 100 MHz, as compared to the 500-MHz slim-form probe limit. To better

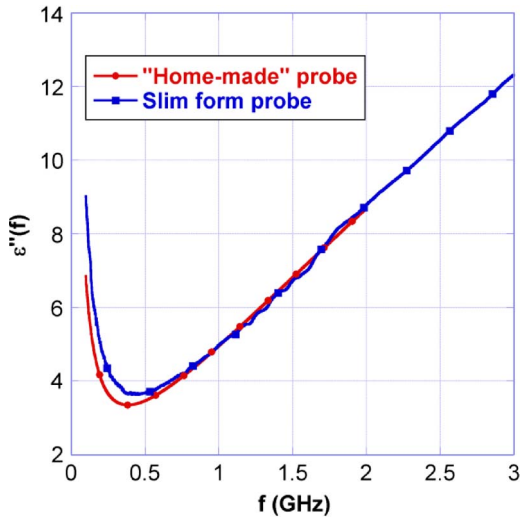

Fig. 6. Imaginary part of the permittivity of two tap water samples: Measurements with the "homemade" (sample #1) and slim-form (sample #2) open-ended coaxial probes.

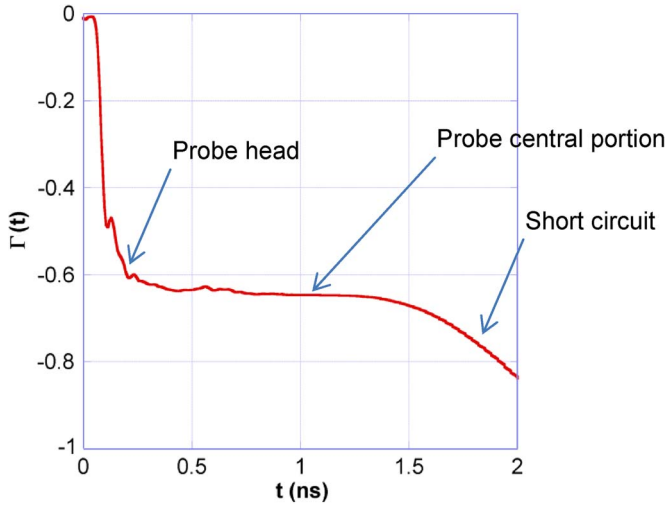

Fig. 7. Portion of TDR waveform acquired on ethanol sample with the short-circuited liquid-filled coaxial probe.

evidence the possible impact of this, Fig. 6 compares the imaginary part of tap water permittivity measured with the slim-form and "homemade" probes. Even though water was not at exactly the same temperature in both measurements and the two tap water samples exhibited different static conductivity values, the two curves can be compared to evidence the higher noise level present on slim-form probe measurements in the lower frequency region, due to the extremely low fringing capacitance value. Clearly, the performance of the slim-form probe was impaired by use near or even below its lower stated operating frequency. The problem might be easily circumvented by using another commercial probe, like the high-temperature one, characterized by a lower frequency limit. This would be done, however, at the expense of a significant increase in the required sample volume, due to the much larger dimensions of the probe tip.

B. Experimental Results for the Short-Circuited Liquid-Filled Probe

After the series of experimental tests with open-ended coaxial probes and the VNA, a last series of tests on the set of liquids has been performed employing the short-circuited liquid-filled coaxial probe in conjunction with TDR measurements, as described in Section II-D. Since previous tests with the TDR equipment, even though performed employing an alternative theoretical formulation, revealed excellent measurement repeatability [31], a single measurement was performed on each liquid, in order to simply assess measurement bias via comparison with literature data. The temperature of the measured liquids was monitored by controlling the external probe temperature, allowing a sufficient time for thermal stabilization. Recorded temperatures are 25.0 °C for chloroform, 25.1 °C for ethanol, 25.4 °C for methanol, and 24.9 °C for water. In accordance with probe capacitance, approximately 1 mL of liquid volume was employed. The TDR equipment was set to acquire the average of 2048 waveforms in order to limit the effect of noise.

First of all, the time-domain approach applying (9) has been employed to evaluate the static permittivity for all liquids. Preliminary to measurements on liquids, a TDR acquisition with the probe in air was performed in order to retrieve the Z_p value, which was found to be 52.0 Ω. This value is slightly different from the design target of 50 Ω due to mechanical tolerances in the fabrication process. As an example, Fig. 7 shows a portion of the TDR waveform acquired with the probe filled with ethanol, evidencing the reflection coefficient corresponding to the central portion of the probe, from which a static permittivity of 22.9 can be derived. Table IV summarizes the results obtained on the four liquids in terms of estimated static permittivity. Data in Table IV show that, with reference to the static permittivity, the performance of the short-circuited probe system with TDR instrumentation is slightly worse than that of the open-ended probes with VNA measurements, with biases as high as 6%. However, this slight loss of accuracy, which happens particularly for lossy liquids, is still acceptable and largely compensated for by the low cost of this solution. As a side note, it is important to emphasize the fact that the static permittivity might also be estimated from the real part of the permittivity spectrum obtained applying (10). This kind of analysis yields final results comparable to those reported in Table IV, with the exception of ethanol for which, due to the stated low-frequency limitations arising from TDR waveform truncation, the flat initial portion of the permittivity is not detectable.

Finally, with regard to the remaining Cole–Cole parameters, unfortunately, the limited operating range of the probe, resulting from parasitic effects at the probe head, only allowed the estimation of the relaxation frequency for ethanol. On the basis of the position of the peak in the imaginary part of permittivity, estimated using (10), a relaxation frequency of 0.979 GHz has been retrieved (see Fig. 8). The obtained result, summarized in Table IV, shows good agreement with literature data [37].

From this whole analysis, it can be concluded that the TDR approach with the short-circuited liquid-filled coaxial probe yields an acceptable final accuracy at an extremely low cost, making it an ideal candidate for large-scale *in situ*

TABLE IV
COLE–COLE PARAMETERS FOR THE TEST LIQUIDS ESTIMATED FROM SHORT-CIRCUITED LIQUID-FILLED COAXIAL PROBE MEASUREMENTS WITH THE TDR TECHNIQUE

	ε_s		f_r (GHz)		ε_∞	
	Meas.	**Bias**	**Meas.**	**Bias**	**Meas.**	**Bias**
Chloroform	4.77	1.1%	–	–	–	–
Ethanol	22.9	6.1%	0.979	1.3%	–	–
Methanol	31.1	4.6%	–	–	–	–
Water	74.1	5.7%	–	–	–	–

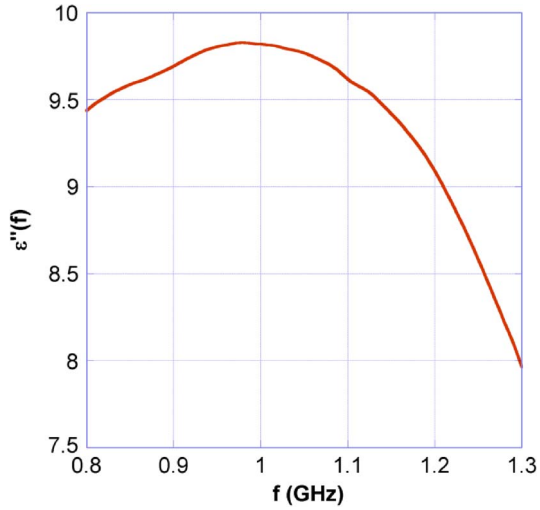

Fig. 8. Imaginary part of the permittivity of ethanol sample: Measurements with the short-circuited liquid-filled coaxial probe and TDR approach.

applications, as long as a 6% uncertainty is an acceptable budget. It must also be stressed, however, that, in its current version, the manufactured probe has severe limitations in its operating frequency range, but revised versions are currently under design and manufacture.

IV. DISCUSSION AND CONCLUSIONS

Some customized systems for microwave permittivity measurements on liquid samples, based on a reflectometric approach, have been presented, and their performances analyzed and compared against the one deriving from the most widely adopted commercial measurement setup. The customized systems are designed to provide less expensive solutions without compromising measurement accuracy.

The first proposed solution aims at replacing the commercial measurement software exploiting a reformulation of the classical lumped-element theory, which allows the use of "homemade" coaxial probes. Application of the formulation to measurements performed with a standard commercial probe revealed that the alternative approach yields very accurate results, sometimes even better than the commercial software, owing to its implicit characterization of the actual probe fringing capacitance frequency behavior. The only drawback is the slightly more onerous calibration procedure, requiring the use of two reference liquids instead of only one. Based on this alternative and effective formulation, a customized probe has been built by properly modifying an N-type coaxial connector, thus replacing the commercial probe with an alternative low-cost solution. The results of the comparative analysis carried out showed a good repeatability of permittivity measurements, and the measurement bias was generally within 5%. The overall performance is comparable with the commercial solution, with the great advantage of ease of measurement with fairly small amounts of liquid sample (less than 1 mL). The lower frequency limit is extended (as compared to commercial probes requiring approximately the same sample volume), at the expense of a narrower operating frequency range, which might be easily enlarged employing a set of probes of increasingly small diameter; indeed, the limited use for frequencies higher than 2 GHz for the presented modified N-type coaxial connector can be easily overcome by a proper tailoring of the probe to the specific frequency range and LUT permittivity.

Finally, a different experimental approach which uses a TDR piece of equipment in conjunction with a short-circuited probe has been explored. This solution is by far the cheapest one and appears to be affected by an overall uncertainty only slightly greater than that of the classical solutions making use of a VNA (measurement bias generally within 6%). However, a consistent limitation in terms of operating bandwidth is currently present, particularly with reference to the higher frequency range. This has to be mainly attributed to parasitic effects arising from the probe geometry and manufacturing process, which should be appropriately removed. Previous attempts at modeling such parasitic effects with lumped elements [31] did not yield significant improvements in accuracy. A better solution might be the introduction in the measurement procedure of a full calibration, similarly to the open-ended probe approach, but this would compromise the portability of the system requiring the presence of some reference liquids for calibration. A possible alternative, which is currently under development, is a redesign of the probe aiming at a minimization of discontinuities introduced at the probe head.

In conclusion, this paper offers a comparison of the accuracy and performance of different custom solutions, with different complexities and costs, for microwave permittivity measurements on liquid samples. This comparative metrological analysis provides novel and useful data, proving that customized systems for complex permittivity measurements on liquid samples at microwave frequencies can be flexible and less expensive solutions. As such, they can be considered as valid competitive substitutes to the widespread high-cost commercial

setup to be used in quality control, monitoring, and diagnostic applications. In particular, the first alternative approach appears to be the best answer if one wants to perform accurate measurements on low volumes of liquids, in the framework of an equipped laboratory where the calibration liquids are available and easy to maintain, supposing that a proper set of probes is realized to extend the system validity to the required frequency range. Conversely, the innovative TDR approach which has been proposed, owing to its extremely reduced costs, is confirmed as the ideal solution in all the practical conditions where inexpensive on-site measurements are required, once that a slight increase in measurement accuracy and overall performance is attained.

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Dr. Liberti has been a member of the Scientific Council of the European Bioelectromagnetic Association since 2008 and the Scientific Secretary since 2011.