TITLE: Data acquisition with Split - Post Dielectric Resonators

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SHORT DESCRIPTION: This document's intention is to provide a guideline to help in the process of acquiring reliable and reproducible data with a split - post dielectric resonator (SPDR). Areas covered include preparation and handling of measurement setups and material samples. Field distributions in SPDR are shown, in order to facilitate the overall understanding of its operation and the nature of the measured data, but extensive advice on data analysis is beyond the document's scope. Appropriate literature is referenced but also errors are pointed out in the existing IEC norm.

INSTRUMENT SPECIFICITY: This guideline is targeting to be instrument unspecific, subject however to two constraints:

1. At the time of writing, the only SPDRs commercially available on the open market are those from QWED. While interested users may construct their own SPDRs based on the open literature referenced hereinafter, it is not guaranteed that such devices provide the same simplicity of operation, in particular, that they come equipped with their device-tailored calibration software and require no further calibration by the user.
2. SPDR needs a microwave signal generator with a function of measuring transmission (S21) characteristics between the SPDRs two ports and extracting the resonant frequency and 3dB bandwidth. In principle, those functionalities can be provided by any VNA or a simpler customised device. However, the operation of QWED SPDRs is most straightforward when used jointly with KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite or QWED Microwave Frequency Q-Meter.

ABBREVIATIONS / TERMINOLOGY:

SPDR - Split - Post Dielectric Resonator

VNA - Vector Network Analyser:

Q-Meter - Microwave Frequency Q-Meter from QWED

S21 - Scattering parameter - transmission

MUT / SUT – material under test / sample under test

MW / RF - Microwave / Radio-frequency

ESD - Electrostatic discharge

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Introduction: principles of the SPDR method

This document attempts to provide a generic guideline for use of split - post dielectric resonators (SPDR). The intention is to provide basic information needed to perform reliable, repeatable and safe measurements. The text gives notes on resonator handling and maintenance, performing measurements, and common sources of errors. Special attention is paid to the different ways of operating the only commercially available SPDRs from QWED within the three types of measurements setups: a non-specific VNA, KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite or QWED Microwave Frequency Q-Meter.

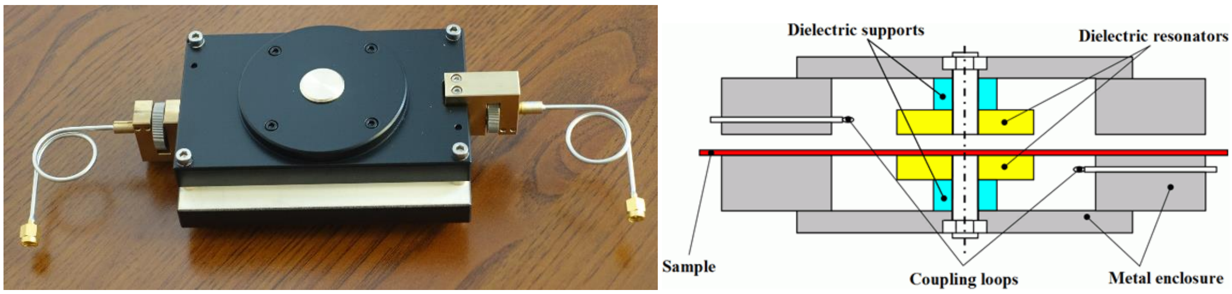

Fig. 1: Example SPDR and its internal construction after [1][2][3].

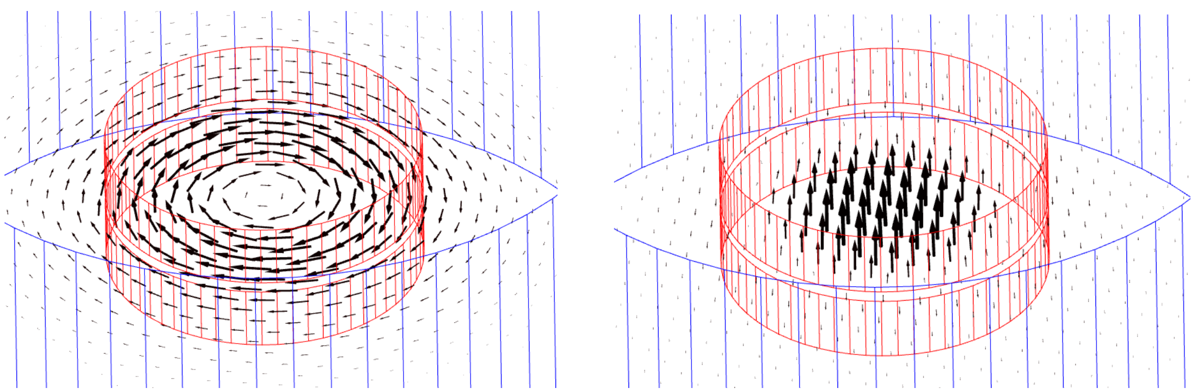

Fig. 2: Electric (left) and magnetic (right) field distribution in SPDR at the resonant frequency; the ceramic posts are here marked in red, the field lines in black, and the metal enclosure in blue.

SPDR is a rigid structure of well-defined, known dimensions and materials. Looking from the outside (Fig. 1 left) SPDR appears as a metal enclosure with a slot for easy insertion and support of a laminar sample. However, it should be stressed that the actual “resonator” is a pair of dielectric cylindrical posts made of high-permittivity low-loss ceramics (yellow in the diagram of Fig. 1 right, red in Fig. 2). At the operating frequency, a resonant mode if formed, with electromagnetic fields mostly confined in and between those ceramic posts, as shown in Fig. 2. Losses in the metal enclosure are thereby minimal, moreover, since the magnetic field has only a vertical component at the side wall of the enclosure, the currents in the side wall are only circumferential, and there is no radiation through the slot. All electromagnetic energy injected through the coupling loops in contained within in the SPDR “head” (inside the enclosure); an estimated 95% of energy is confined in and between the ceramic posts. *Note that the mode used in SPDR measurements is axisymmetrical, contrary to Figures B.2 and B.3 of norm [2], which are incorrect.*

The resonant mode has a particular resonant frequency depending on resonator's dimensions and materials but also, to some extent, on the electrical properties of the sample under test (SUT). Thus, each resonator is designed for a particular nominal frequency and the actual measurement is taken at a frequency close to the nominal one (Fig. 3). The IEC norm [2] refers to frequencies between 1.1 GHz and 20 GHz, the commercially available resonators from QWED cover several discrete nominal frequencies between 1.1 GHz and 15 GHz. SPDRs for other frequencies can be designed following the theory of ref. [1], however, at lower frequencies SPDRs become big and bulky, while at higher frequencies manufacturing tolerances and losses in the applied materials deteriorate the measurement accuracy.

The SPDR method requires making two consecutive measurements: one of the empty SPDR and one with the SPDR loaded with the sample. The principle of the method resides in extracting the real part of the SUT permittivity from the change of the resonant frequency, and the imaginary part - from the measured change of the Q-factor (with some corrections of the Q-factor due to the field pattern change in the presence of SUT). The details of the SPDR method are presented in publication [1] and summarised in the IEC norm [2].

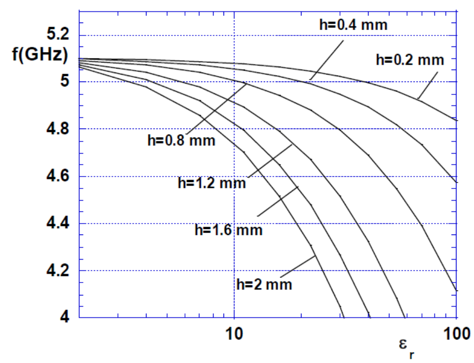

Fig. 3: Resonant frequency (f) of an example (nominally) 5 GHz SPDR loaded with a SUT of height h and relative permittivity ε_r.

Handling and safety of resonators

SPDRs are passive structures, and the measurement mode is well shielded from the environment (Fig. 2), so in normal use no electromagnetic hazards arise. However, if a resonator is erroneously driven by a signal significantly outside of its nominal operating frequency band, undesired modes may be excited which may be subject to leakage. Therefore, when SPDR is connected to and driven by a VNA (or an alternative active device), output power of that active device should not be increased about its standard value and frequency band should not be wider than expected for 3dB bandwidth measurement of the SPDR.

SPDRs provide accurate measurements because they are precisely designed and mounted, and each unit is individually calibrated upon manufacturing to adjust for the manufacturing tolerances. Therefore, each SPDR should be handled with care and kept clean. The coupling loops within the resonator (Fig. 1) are specifically delicate. QWED SPDRs are therefore terminated with semi-rigid cables and to prevent those cables from damage, additional flexible coaxial cables connecting them to the high-precision cables of the VNA are recommended.

Because of individual calibration at origin, each SPDR comes with its own (unit-specific) version of the software. A backup copy of the software should be kept by the user in a safe place and clearly linked to a particular SPDR unit.

SPDR must not be kept or used outside its operating temperature range dictated by the applied materials and cables. QWED declares the range from -270 deg C to 110 deg C for its standard SPDRs but customised higher-temperature units have also been used.

A practical observation stemming from QWED's 20 years of experience in producing and marketing its SPDRs is, that damages in normal use are rare; most reported damages concerned the semi-rigid cables; in only one case a resonator was damaged during shipment (or rather custom clearance) and a loss of the SPDR's own software happened.

Sample types, preparation and safety

SPDRs are originally intended for the measurements of the **complex permittivity of laminar dielectric materials**. Other documented applications include thin ferroelectric films deposited on low loss dielectric substrates as well as sheet resistance and conductivity of various conducting materials such as commercial resistive layers, thin conductive polymer films or high resistivity semiconductors. As per Fig. 2, electric properties of SUT are measured in the tangential electric field (and hence are in-plane permittivity and the dielectric loss tangent).

The sample under test (SUT) should be flat and thin enough to fit into the SPDR's slot. SUT's lateral dimensions must be big enough to cover the SPDR "head" (somewhat smaller SUTs are measurable but with reduced accuracy) while the width must be small enough to fit through the slot (which is a mechanical constrained that can be alleviated by a customised SPDR construction). There are no formal requirements on the minimum thickness of the SUT, also SUT rigidness and surface polishing are not absolutely necessary, thanks to the fact that *air slots between the SUT and the fixture do not affect the measurements* (which is quite a unique feature of the SPDR measurement method, resulting from the use of the SPDR tangential electric field, Fig. 2). However, for very thin SUTs the extraction of permittivity becomes ill-conditioned (see Fig. 3), and non-uniformity (Δh) of the SUT height (h) transfers into inaccuracy of its measurement. According to the IEC norm [2], to which QWED SPDRs comply, the errors of measuring SUT relative permittivity and loss tangent are within:

$$\Delta\varepsilon/\varepsilon=\pm(0.0015 + \Delta h/h)$$
$$\Delta\tan\delta=\pm 2*10^{-5} \text{ or } \pm 0.03*\tan\delta \text{ whichever is higher}$$

Note that the IEC norm [2] includes Table 1 "Speciment dimensions" and Table 2 "SPDR test fixture's parameter" for several nominal frequencies. Those tables should be interpreted with caution, taking into account our earlier remarks. The size of SPDR "head" and gap (Table 2 of [2]) may be somewhat different, depending e.g. on the ceramics used for the posts (undefined in [2]). On the other hand, maximum SUT dimensions (Table 1 of [2]) are not absolute electromagnetic constraints, but can be increased with a particular mechanical construction. QWED standard

SPDRs (as per table below) comply with the IEC norm [2] for "head" and gap sizes, generally allow for slightly wider samples, and pose no fundamental restrictions of SUT length. A recent design within the MMAMA project which additionally allows for surface scanning with an SPDR is discussed in the final section of this SOP.

Nominal frequency [GHz]	Min size of SUT [mm] (diameter D or square D x D)	Max thickness of SUT [mm]	Max width of SUT (standard version)
1.1	120	6.00	150
1.9	70	4.00	100
2.5	55	3.10	100
5.1	30	1.95	90
10	22	0.95	90
15	14	0.60	40

Table 1: Main specifications for QWED commercial SPDRs.

The operator inserts a SUT into the SPDR slot. This exposes the operator to the material under test as well as the material to the operator. Necesary care must be taken to avoid personal injury in case of harmful substances and to avoid damaging SUTs if they are soft or fragile. The use of gloves, masks, or tweezers is often recommended and in any case the instructions from the material manufacturer must be adhered to. Note that it is also possible to manouver delicate SUTs placed on a low loss dielectic foil such as polyethhylene or PTFE - such thin supporting sheets do not noticeably affect on the measurement, moreover, their effect can be eliminated by taking the SPDR loaded with the supporting sheet (instead of the empty SPDR) as reference. If, despite all caution, a sticky SUT leaves traces on an SPDR, the SPDR must be carefully cleaned.

In application to copper clad laminates, all cladding must be removed by etching, then a SUT must be cleaned and dried. Any SUT should be conditioned at the SPDR operating temperature prior to testing. SUT thickness must be measured with a micrometer of 0.001 mm resolution (or better). For each SPDR unit, at least one verification SUT should be maintained, preferably marked by an engraving pen or other suitable method.

Measurement setups, equipment and operator safety

SPDR measurements can be performed at room temperature or in environmental test chamber [2]. In all tests the ambient test temperature must not exceed beyond the operating temperature range of a particular SPDR declared by its manufacturer. The IEC norm [2] further strictly declares that the temperature variation should not exceed 1 deg C during test. In practice this means that the measurement of the SPDR loaded with SUT must quickly follow the measurement of the empty SPDR, and test environment (temperature, humidity) should be recorded together with the test results.

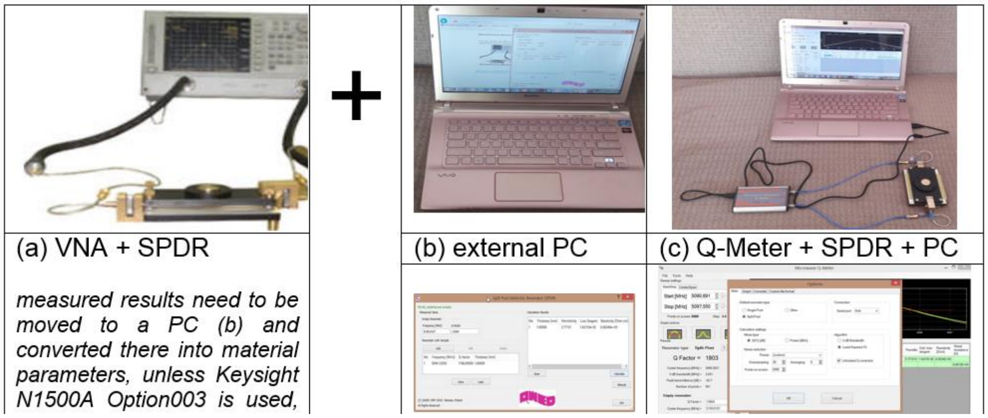

Fig. 4: Measurement setups bulit with SPDR: classical with SPDR connected to a VNA (a) which except for selected KEYSIGHT VNAs needs data tranfer to a PC (b); compact consisting of SPDR, Q-Meter and PC running a dedicated Q-Meter software.

SPDR + VNA measurement setup

A classical measurement setup consists of an SPDR and a VNA (Fig. 4a). VNA is sensitive to static electricity, special care should be taken to avoid damage from ESD. Conducting wrist straps should be used to prevent high voltages from accumulating on workers bodies, also anti-static mats or conductive flooring materials are desirable. No highly-charging materials should be in the vicinity. The inner conductors of any RF cables, connectors or probes should never be touched, or come in contact with electrostatically charged surfaces.

The frequency range of the VNA must naturally cover the operating frequencies of the SPDRs to be applied; and the dynamic range recommended by the IEC norm [2] is more than 60dB. Allow at least 30 min for the VNA to warm up and stabilise.

In case of KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite, if this is your first work with a particular SPDR, make sure this SPDRs specific software has been installed. In case of other VNAs, be prepared to copy the measured data and transfer it to a separate PC, with the SPDR specific software installed on it.

Connect the SPDR to the VNA and follow the VNA instructions. Start with the empty SPDR. Enable the option of S21 magnitude measurement. Set the centre frequency at the nominal frequency of the SPDR. Read the actual resonant frequency (peak of S21) and 3dB bandwidth. Check the minimum values of S11 and S22, if different by more than the second decimal place, use SPDR nuts to change the positions of the coupling loops and repeat S21 measurements. Collect the resonant frequency and Q-factor (or 3dB bandwidth) of the empty resonator. Repeat the same steps for the SPDR loaded with SUT.

In case of KEYSIGHT VNAs including Option 003 in their N1500A Material Measurement Suite, the material parameters will come up on the VNA screen. In case of other VNAs, move the raw measured results to the PC. In the latter case, maintain special care to clearly annotate those results.

SPDR + Q-Meter measurement setup

A fully-fledged VNA provides significantly more functionalities than needed for the SPDR measurements. The microwave engineer having access to a VNA and knowing how to operate it will typically choose the VNA setup. However, SPDRs have also found applications in various institutions, including food industries and material science, where neither microwave equipment nor engineers are available. For those users, low-cost easy-to-use devices substituting the VNA in SPDR measurements have been developed.

An example of such devices is Microwave Frequency Q-Meter from QWED, available for several frequency ranges compatible with the operating frequencies of QWED SPDRs. Essentially, Q-Meter is a computer controlled microwave oscillator system for quick and automatic SPDR measurements. A dedicated, user friendly and highly configurable application allows controlling the measurement process and enables easy management of the measurement results. A portable setup consisting of SPDR, Q-Meter and a laptop is shown in Fig. 4c. Its operating instructions will not be quoted here, as they are device- and application-specific. It is worth noting however, that the application itself guides the user through the measurement process.

2D SPDR scanning setup

In the MMAMA project, SPDR measurements have been extended to 2D surface imaging, following the concept illustrated in Fig. 5.

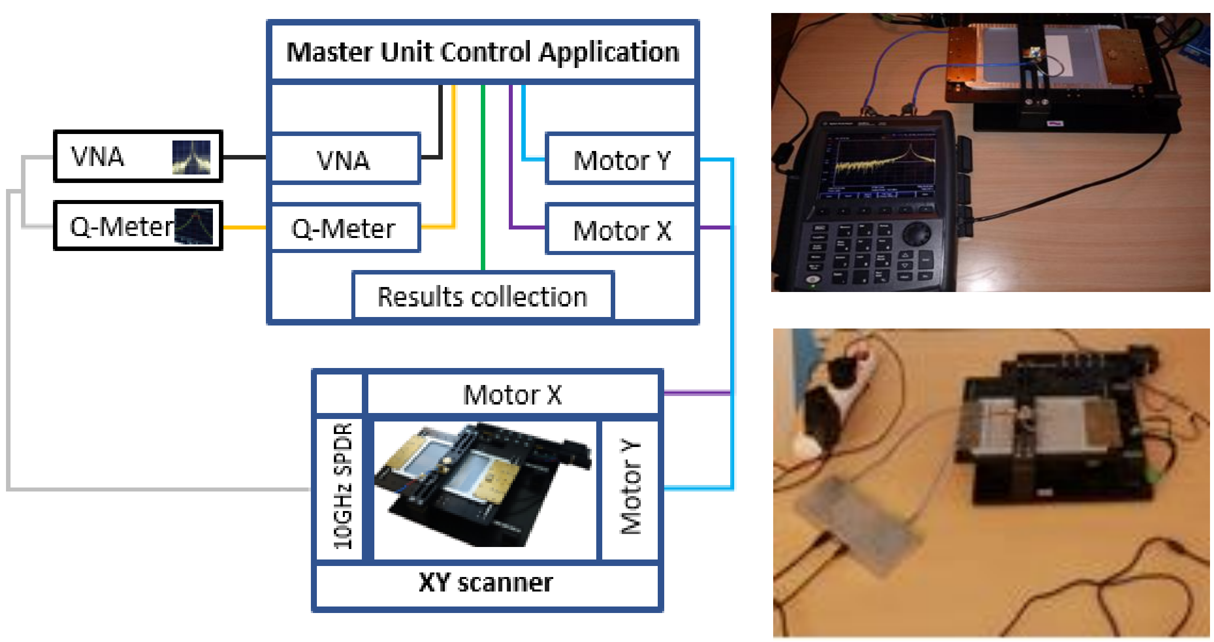

Fig. 5: Block diagramme of the 2D SPDR scanning setup (left) and validated connections of the scanner to KEYSIGHT FieldFox (upper right) and QWED Q-Meter (lower right).

To this end, a 10 GHz SPDR from QWED has been built into a 2D scanner. The scanner has been designed with the use of two linear stages driven by two commercial motors from Standa and Nanotec, respectively. Several modifications of the resonator have been made to adjust to the mechanical construction, e.g., angular connection of the feeds and calibration with a teflon sheet needed for sample positioning. As shown in Fig. 5, connections have implemented and tested to two portable network analysers: KEYSIGHT FieldFox and and a new 10 GHz Q-Meter developed by QWED in the same project. The whole setup is driven by Master Unit Control (MUC) application running on laptop. The main specifications for the 2D SPDR scanner are summarised in Table 2.

Measurement frequency [GHz]	10.188
Max thickness of SUT[mm]	0.8
Max recommended thickness of SUT [mm]	0.6
Max sample size [mm]	110 x 150
Max scanning area [mm]	57 x 115
Max recommended scanning area [mm]	50 x 90
Motor 1 (Standa) resolution [mm]	0.0025
Motor 2 (Nanotec) resolution [mm]	0.005
Approx. time of one point measurement (MW measurement and movement)	3 - 4 sec (depending on spatial step)

Table 2: Main specifications for 2D SPDR scanner.

Examples of applications of the 2D SPDR scanner to samples of laminates and organic semiconductors are published in ref. [4][5]. It is concluded that by incorporating the resonator into the scanner the following advantages extensions of the SPDR technology are achieved:

- Samples of size larger than permitted by the standard SPDR construction (cf. Tab. 1) can be characterised.
- Automatic scanning images surface inhomogeneities, which may be programmed in the material fabrication process or caused by artifacts.
- The 2D scans obtained in a controlled manner can be further post-processed through image processing techniques to obtain higher resolution maps of complex permittivity. As a result, the effective spatial resolution of SPDR measurements is enhanced below the size of the resonator head and samples smaller-than-head can be measured.

Operation of the 2D scanner requires more caution than that of stand-alone resonators and two main issues will be pointed out:

- The flatness and stiffness of SUTs are now important not only for the accuracy of the extracted material parameters, but also from the viewpoint of smooth SUT movement within the SPDR head, to avoid mechanical blocking. Therefore, maximum recommended SUT thickness is reduced with respect to stand-alone 10 GHz SPDR (see Table 1) to 0.6 mm (Table 2).
- When a scan of thousands of spatial point is made, which takes several hours, thermal variations of the SPDR parameters begins to influence the extracted material parameters. It is therefore recommended to minimise the measurement time by collecting only the raw

transmission curves; curve smoothing, extraction of the resonant frequencies and Q-factors, and then their conversion to the material parameters, is performed at the post-processing stage.

- It is also recommended to measure the empty resonator both at the start and at the end of the scanning process. A shift in the SPDR resonant frequency and Q-factor will be a measure of the additional material imaging error (absent in the classical use of SPDRs, where the reference and sample measurements are performed instantaneously one after the other). Studies on temporal and thermal stability of the SPDRs are currently under way in order to formulate further recommendation for temperature monitoring and multiple temperature-depended scanner calibrations.

Forthcoming revisions and variants of this SOP

This SOP summarises the principles of SPDR measurements based on the original literature [1] and the pre-existing IEC norm [2]. It also incorporates critical observations from QWED engineers made during the 20 years of their own experience in manufacturing and using SPDRs as well as assisting other SPDR users in the market segments supported so far. In particular, the industrial implementation of QWED SPDRs at MMAMA partners has brought new observations and led to the development of the large surface SPDR scanner, presented in the final section. On-going studies are concerned with long-term stability of SPDR measurements that must be considered when imaging large material surfaces in the course of many hours; the results will be discussed in forthcoming revisions of this SOP.

While the split-post configuration was originally selected for dielectric resonator measurements in the MMAMA project, current activities indicate that the single-post configuration may be more relevant to some of the considered materials, e.g. highly conductive composites. When experience is gained, a separate SOP on single-post dielectric-resonator measurements will be prepared.

References

[1] J. Krupka, A. P. Gregory, O. C. Rochard, R. N. Clarke, B. Riddle, and J. Baker-Jarvis, “Uncertainty of complex permittivity measurements by split-post dielectric resonator technique”, *J. Eur. Ceramic Soc.*, vol. 21, pp. 2673-2676, 2001.
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