LABORATOIRE NATIONAL DE MÉTROLOGIE ET D'ESSAIS LNE

Report on the activities in Electricity and Magnetism within the LNE between 2019 and 2021

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This report gives a brief outline of the main research and development activities in the field of Electricity and Magnetism since March 2019 at the Laboratoire National de Métrologie et d'Essais (LNE).

1 Kibble balance

The Kibble balance experiment developed at LNE in collaboration with the CNAM and the LNE-SYRTE has obtained a value of the Planck constant with a relative standard uncertainty of 5.7 parts in 10^8 in 2017. From this result, obtained in air at atmospheric pressure, LNE has decided to continue the developments on the KB to realise the kilogram with an aimed uncertainty of a few parts in 10^8. Improvements of different parts of the set-up are ongoing, mainly to improve the Type A uncertainty of the experiment, and to be able to work under vacuum (and thus improving Type B uncertainty).

Significant mechanical modifications around the concrete slab, which supports the apparatus, but also of the bearing structure of the apparatus ("V-shapped" feet to pillar feet), allow to increase the immunity of the experiment to nearby activity but also lead to a net improvement of the noise rejection in dynamic phase. Mechanical stability of the structure, of the coil and its suspension, but also of optics in vacuum, was confirmed with an uncertainty of some tens of microradians. Dynamic phases has been carried out in vacuum, as well as static phases: kilogram determinations will be the next step.

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2 Capacitance metrology

Thompson-Lampard calculable capacitance standard

The two major contributions to the uncertainty budget of the new Thompson-Lampard calculable capacitance standard at LNE are improved significantly which will enable to achieve a target uncertainty of one part in 10^8.

The first contribution is related to the alignment of parallelism of the five electrodes better than 0.28 µrad which translates to a maximum allowed tilt of about 100 nm of an electrode bar with a length of 350 mm. At this scale, it is crucial to implement a precise adjusting mechanical system, to control stability of the environment and most importantly to have a reliable measurement of parallelism of electrodes. The new measurement procedure of the parallelism of the electrodes was developed using ten sub-micrometer resolution capacitance-displacement sensors mounted on a common ring support. By choosing a reference electrode, one can measure rapidly and reliably parallelism of the four other electrodes in a single run since trajectory errors, which are common for all sensors taking measurements simultaneously, can be easily eliminated. The mentioned procedure allows us to align electrodes from outside the cavity formed by the five electrodes. The final validation of the parallelism of electrodes from inside, *i.e.* the validation of the cylindricity of the cavity is carried out with a capacitive ring mounted on the mobile guard.

The second contribution is related to the fine control of the lateral positioning of the mobile guard inside the cavity at any altitude with an accuracy better than 100 nm. It is realized using two piezoelectric actuators located at 90° from each other that place the mobile guard laterally without perturbing aligned electrodes from previous step. Then the position of the mobile guard with respect to the five electrode bars inside the cavity is measured using the capacitive ring on the guard bar. Using this precise positioning system of the guard at different altitude, the cylindricity of the cavity has been characterized. As a result, a total performance better than 80 nm was achieved combining both positioning of the guard and tilt of the electrodes.

Finally, the different parts of the calculable capacitance are now completed. The interferometer is implemented in the standard and the external electrical screens are installed. The first electrical measurements have begun at atmospheric pressure.

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3 QHE metrology

In order to achieve routine operation of the quantum Hall resistance standard (QHRS) in graphene devices under simplified experimental conditions, namely lower magnetic field ($B \leq 5$ T), higher temperature ($T \geq$ 4.2 K) and higher measurement current ($I \geq 100$ μA), LNE is currently working on improving the reliability of the technology of quantum Hall devices made from graphene grown by chemical vapor deposition on silicon carbide (G/SiC). This work follows on from the results obtained at LNE in 2015 on the demonstration of the QHRS in a graphene device under relaxed conditions with state-of-the-art accuracy (1.10^{-9}) (Nature Nanotechnology 10, 965 (2015)) and concerns the same technology. The present work includes notably the control of the charge carrier density, for which different solutions are currently investigated. This work is carried out in collaboration with C2N at CNRS/Université Paris-Saclay and with CRHEA at CNRS. In the same perspective of operating the graphene QHRS under simplified experimental conditions, for the wider dissemination of SI electrical units and the SI defining constants h, Planck's constant, and e, elementary charge, LNE is currently installing a cryomagnetic system, without liquid helium consumption, based on a pulse-tube cryo-cooler and a closed cycle helium 4 variable temperature insert. This system has already shown a base temperature below T = 1.3 K, as well as a sufficiently high cooling power and good electrical noise performances under magnetic field (up to 14 T).

Since 2019, LNE is involved in the EURAMET/EMPIR project 18SIB07 GIQS "Graphene impedance quantum standard" that aims to develop a quantum standard for impedance using the AC quantum Hall effect (QHE) in graphene and appropriate impedance bridges. Prior to QH measurements in the AC regime (in the kHz frequency range), LNE has already performed DC characterizations of a 400 µm-wide QH device in G/SiC, grown and processed at PTB. The sample was mounted on a double-shielded TO-8 holder developed at CMI, which is compatible for both DC and AC precision measurements. It travelled in a dedicated chamber provided by KRISS, which allows good control of the surrounding atmosphere and relative stability of the electronic properties (carrier density and mobility). The sample showed a hole-doping with a carrier density of $4.3 \cdot 10^{-10}$ cm^{-2} for a mobility of about 7000 cm^{2}V^{-1}s^{-1} at T = 1.5 K. The contact resistances were lower than 1 Ω. High-precision measurements showed that the Hall resistance R_{xy} in this sample is quantized to the $h/(2e^2)$, to within a few parts in 10^9 at B = 4 T, T = 4.2 K, and I = 50 μA, consistently with a former characterization of the device at PTB. These results showed that hole-like charge carriers are not detrimental to the Hall quantization.

Within the EURAMET/EMPIR project 17FUN04 SEQUOIA "Single-electron quantum optics for quantum-enhanced measurements", LNE is exploring the QHE in high-mobility graphene samples encapsulated by hexagonal boron nitride (hBN). The aim is to use the breakdown of the QHE for highly sensitive charge sensing. For comparison, LNE firstly characterized a Hall bar (100 µm-wide) made of G/SiC. From the current and temperature dependences of the longitudinal resistance R_{xx}, it has been shown that the breakdown mechanism was the variable range hopping (VRH). The detection of a small current I_{ac} was tested in the breakdown regime: I_{ac} was superposed to the main dc current I_{dc}, while the resulting ac longitudinal voltage is used for detection of I_{ac}. The current to voltage gain increases with I_{dc}

but the simultaneous increase of the $1/f$ noise with I_{dc} jeopardizes the sensitivity at frequencies lower than 10 kHz. The QHE was investigated in a second Hall bar (4 µm-wide) made of graphene encapsulated by hBN, showing an unexpectedly low mobility (2000 $cm^2V^{-1}s^{-1}$). Despite values of R_{xx} as low as 100 µΩ (up to 5 µA), R_{xy} was not quantized, with relative discrepancies of several 10^{-7}, possibly explained by the 100 Ω edge contacts resistance. The dissipation is here also explained by the VRH, with localization length values, ξ_{loc}, three times larger than in G/SiC, for similar mobility. This tends to confirm the specific short-range disorder in G/SiC. More recently, LNE started the characterization of a 4 µm-wide Hall bar showing large mobility, of about 70 000 $cm^2V^{-1}s^{-1}$ and 100 000 $cm^2V^{-1}s^{-1}$ at T = 1.5 K, for electron and hole doping, respectively. This low disorder allows to observe Hall plateaus at B = 1 T (*e.g.* plateaus at Landau level filling factor $\nu = \pm 2, \pm 6, \pm 10, \pm 14$) as well as the lifting of both spin and valley degeneracies at B = 18 T ($\nu = \pm 1, \pm 2, \pm 3,...$), and even the manifestation of the fractional state at $\nu = \pm 4/3$ at T = 0.3 K. R_{xy} was not quantized despite measurement of low values of R_{xx}. Further studies are ongoing to understand the absence of quantization before exploring the breakdown and performing small current detection.

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4 Quantum ampere

LNE has developed a practical quantum current standard realizing the new ampere definition based on the elementary charge (J. Brun-Picard et al, PRX 6, 041051 (2016)). It is founded on the application of Ohm's law to the quantum voltage and resistance standards that are combined using a cryogenic current comparator. The quantum standard is able to generate 10^{-8}-accurate currents in the range from 1 µA up to 10 mA. Our goal is to achieve relative uncertainties lower than 10^{-8} and extend the current range. A first step toward that goal has been taken by increasing the reliability of the experiment. We have identified and estimated the noise sources of the PQCG with the support of an electrical model based on magnetically coupled RLC circuits. The better understanding of the system has helped us implementing a more efficient cryogenic damping circuit with reduced noise, an optimized SQUID feedback circuit and an adapted grounding scheme. Calibrations of commercial precision ammeters with a relative measurement uncertainty of a few parts in 10^7 (only limited by their stability) have been reported. Furthermore, the improved PQCG was used to feed an Ultrastable Low-Noise Current Amplifier (ULCA) with currents in the 50-µA range. In this comparison, the two current values agree to -3.7 parts in 10^7 with a combined standard uncertainty of 3.1 parts in 10^7 (k = 1). Hence, there is no significant discrepancy between the realizations of the ampere from PTB and LNE laboratories within the expanded uncertainty of 6.2 parts in 10^7 (k = 2). Our results represent the state-of-the-art of currents in the range ± 50 µA. However, there is room for improvements for future comparisons with the goal to reach 1 part in 10^7 or less. We are currently working on a new version of the PQCG, including a new external current source and a new cryogenic current comparator, which is implemented in a lab dedicated to electrical current traceability.

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5 Electrical nanometrology

The general objective of this recent activity is the development of metrological instrumentation (reference calibration samples, probes, and electronics) and calibration methods for nanoscale measurement of electrical quantities using electrical scanning probe microscope (eSPM). One of the challenges is to reduce the total standard uncertainty down to a few percents in relative value under optimal conditions. This will allow one to determine with the same level of uncertainty the electrical properties (permittivity, doping concentration, mobility …) of materials and devices at the nanoscale.

The traceability to the SI of reference samples commonly used for the calibration and quantification of admittance measurements at GHz frequencies in scanning microwave microscopy (SMM) and similar techniques is not established. In these last two years, we have investigated most possible error sources that affect the uncertainty of capacitance measurements performed on commercially available reference calibration samples. These error sources come from the reference structure itself (dimensional properties, depletion capacitance), the instrumentation (stray capacitances) and the environmental conditions (relative humidity). Very recently, we have established a comprehensive uncertainty budget leading to a combined uncertainty of 3 % in relative value (k = 1) for capacitances ranging from 0.2 fF to 10 fF. This uncertainty level can be achieved even with the use of unshielded probes. Our work offers improvements on the classical calibration methods known in SMM and suggests possible new designs of reference standards for traceable measurements of capacitance and dielectric constant. Part of this work has been developed in the frame of the project ADVENT (Grant Number: 16ENG06) which has received funding from the EMPIR programme co-financed by the Participating States and from the European Union's Horizon 2020 research and innovation programme.

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6 Power and Energy

One of the research activities in this area focuses on the quantification and the reproduction of the harmonic emissions between 2 kHz and 150 kHz in smart grids. In the H2020-MEAN4SG project ("Metrology Excellence Academic Network for Smart Grids"), the attention was paid on the disturbances that may exist on electrical networks due to new generation sources (photovoltaic, wind) as well as to the multiplication of power electronic converters. Aiming at obtaining a representation of supraharmonic emissions occurring in a residential smart grid, the adopted approach was based on a metrological characterized measurement system and the design of experiments (multi-factor design to maximize the obtained information with a minimum number of measurements but relevant configurations). Both the method and the measurement system were used on Concept Grid, a French platform designed to study the new smart grid equipment.

New normative measurement techniques are the core of a new research project, JRP 18NRM05 SupraEMI ("Grid Measurements of 2-150 KHz harmonics to support normative emission limits for mass-market electrical goods"). LNE contributes to this project with measurements of harmonic emissions between 2 kHz and 150 kHz using CISPR 16 and IEC 61000-4-7 methods.

The need to periodically verify the Energy Measurement Systems installed on board trains appears recently with the publication of the European Directive n° 2008/57/EC. The tests and the requirements to be satisfied are indicated in the current standard EN 50463-2:2018. The work performed in the JRP 16ENG04 MyRailS ("Metrology for smart energy management in electric railway systems") leads to the development of fictive power sources and reference measurement systems. LNE completed and validated its calibration setup for the Energy Measurement Systems. The capabilities are (1) to generate fictive electrical power consisting of current signal (sinusoidal or 90° phase-fired waveform) up to 500 A rms value and up to 5 kHz harmonic content, respectively of high voltage potential (25 kV, 50 Hz) and (2) to measure the active power in a traceable with a relative expanded (k = 2) uncertainty of 0.1 % in sinusoidal conditions, respectively of 0.5 % in distorted conditions.

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7 High Voltage/High Current metrology

High voltage impulse measurements

The new reference system developed for lightning impulse voltage measurements has been validated according to the results of the Supplementary comparison EURAMET.EM-S42 Comparison of lightning impulse reference measuring systems. Because of LNE excellent results at 500 nanoseconds chopped impulse, this system has been studied for the measurements of fast transient with rise time as fast as 200 nanoseconds and for voltage up to 400 kV. The obtainable uncertainties of measurement are 1,6 % for the peak voltage and about 8 % for the rise time.

LNE High voltage team continues working in impulse measurements technics with the development of a calculable voltage divider for the traceable measurements of high voltage nanosecond impulses. The divider will be capable of measuring voltage amplitudes up to 0.5 MV and rise time from one to few nanoseconds with a target uncertainty of 3 % for the amplitude and 1 ns for the temporal parameters. Two measuring systems are under development in LNE, the main element of the first one is the use of coaxial capacitive voltage divider and the main element of the second one is the use of coaxial resistive voltage divider. Each system will have a characteristic impedance of 50 Ω in both high voltage arm and low voltage arm. The electromagnetic simulations show that the bandwidth of the system is higher than 1 GHz for voltage up to 500 kV. The mechanical design is on progress.

LNE has tested a new approach to generate reference impulses in order to calibrate digitizers used for impulse measurements for voltage up to 1 kV. The approach chosen by LNE is based on the use of high voltage and high-speed amplifiers. When they are connected to a high speed Digital to Analogue Converter, it is possible to generate any wave shape. Four amplifiers with different characteristics, three commercial and one LNE-made, have been tested and studied. The results show that this method could reach high metrological performances at least equivalent to traditional calibrators, which usually need a separate electrical bloc for any additional wave shape. The advantage of the use of high voltage amplifier is the flexibility to generate, in one bloc, any wave shapes with any rise time higher than 1 microsecond for voltage up to 900 V peak to peak.

High voltage capacitance measurements

In the recent years, manufacturers of compressed gas capacitors have put big efforts into improving the electrode coaxiality which is the most influent parameter on the voltage dependence. Some of them claim a voltage dependence below ppm level but the measurement of voltage dependence is not an easy task. Several technics could be used but all need complexes procedures, reference standards with known voltage dependence and high precision capacitance bridges. It is usually impossible to measure voltage dependence below ppm level. LNE have reviewed the kinetic method (derived from Latzel work in the 80s and 90s of the last century at PTB) which is more suitable to detect a possible quasi zero voltage dependence. At the first instance, the capacitor is charged by high DC voltage and a mechanical impulse is applied perpendicularly to the top of the capacitor toward its centre. The low voltage electrode oscillates and the time dependence curve of the alternating current flowing the capacitor is measured by mean of a storage oscilloscope across its input impedance. From the analysis of the current curves for each impact position, it is possible to calculate the initial eccentricity and the voltage dependence. The first results with a compressed gas capacitor with known internal dimensions have shown an excellent initial eccentricity of (0.020 ± 0.010) mm and also an excellent voltage dependence of $(0.028 \pm 0.030) \cdot 10^{-6}$ in relative value.

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8 AC resistance and current measurements up to 1 MHz

Traceability of AC resistances up to 1 MHz

Since 2019, LNE launched an internal project on the calibration of AC resistances in low-frequency range from 10 mΩ to 1 MΩ. The project has two scientific and technical objectives: develop an automated measurement method for calibrating AC resistances with a maximum uncertainty at 20 kHz of 50 μΩ/Ω and 50 μrad on AC-DC difference and impedance phase respectively. The second objective is to extend the frequency bandwidth up to 1 MHz (currently limited to 20 kHz). The complementary method is based on the use of a vector network analyser (VNA) with targeted uncertainties of at most 5 mΩ/Ω and 5 mrad respectively on AC-DC difference and impedance phase up to 1 MHz.

In 2020, a new calibration bench (Wheatstone bridge) for AC resistances up to 20 kHz was developed. The Wheatstone bridge is based on a dual AC voltage source and PXI modules. The dual AC voltage source supplies the Wheatstone bridge and the PXI modules that are used as a voltage injection system. A 10 MHz synchronization solution with an external signal has been used for the two instruments. The calibration bench has been validated for the 3-terminal resistances. High impedance stages are under development to adapt the bench for the 5-terminal resistances.

The second method to measure AC resistances between 20 kHz and 1 MHz and based on the use of a VNA is being validated. This method is conditioned by the characterization of RF adapters for N-type to UHF connectors and the possibility to model the resistances frequency behaviour by polynomial regressions (up to a few MHz). In 2020, the design of two analytically calculable RF adapters for N-type to UHF connectors has been launched, the mechanical assembly and the characterization of these adapters is in progress.

Design and modelling of a shunt for current measurements at 10 A and up to 1 MHz

The LNE has investigated the possibility to extend the calibration capabilities of high current sensors up to 10 A and 1 MHz and thus to improve the traceability of AC current measurements.

Firstly, LNE has developed a completely calculable (electromagnetic and thermal responses) current shunt standard for 50 mA based on theoretical basis and innovated design: at 1 MHz the phase shift and transposition deviation are -0.01 mrad and 15 μΩ/Ω respectively. In 2020, the objective was to realize a current shunt of 0.08 Ω for a current of 10 A. An optimization study on the electrical resistivity and the temperature coefficient of the resistive disks as a function of the thickness of the deposited layer has been launched. The validation of the current shunt of 10 A is in progress.

Secondly, LNE has developed a traceable calibration method to measure shunts up to 10 MHz. The measurement method, based on the use of a vector network analyser (VNA), allows the AC-DC difference and impedance phase of a shunt to be measured simultaneously with relative uncertainties less than $1 \cdot 10^{-3}$ at 1 MHz.

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9 RF and MW metrology

Specific absorption rate (SAR) and vector electric field measurement & EMPIR project Vector SAR

The aim of this project is to establish the traceability of specific absorption rate (SAR) measurement when using vector E-field probes. For this purpose, the complex permittivity of reference liquids and tissue equivalent liquids was extracted from S-parameter measurements in coaxial cells of different lengths using the Line-Line extraction method and a vector field probe calibration system was developed to operate in several telecommunication frequency bands between 400 MHz and 6 GHz, using reference waveguides for the corresponding frequency bands.

An automatic positioning system was also developed, allowing the E-field probe under calibration to be positioned accurately in the different reference waveguides, and to perform scans in a flat phantom for the calibration of SAR reference antennas as defined in the IEC-IEEE 62209-1528 and IEC 62209-3 international standards.

A physical and analytical model of the calibration system was devised to establish the traceability of electric field and SAR measurements when using electro-optic vector E-field probes, by determining the vector correction factor of the probe (ratio between electro-optic converter voltage output and electric field measured by the probe). This is done by measuring the *S* parameters of the section between the reference waveguide input and the probe tip and second, of the whole measurement chain from the reference waveguide input to the electro-optical converter output. The correction factor can then be determined by matching the electric field and voltage quantities to the probe tip input and to the electro-optic converter output, respectively.

Next, the traceability can be transferred to a reference antenna radiating into a flat phantom by using the calibrated vector E-field probe for scanning the electric field in the phantom.

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Nontraditional impedance S parameter measurement: balanced impedance, extreme impedance

The aim of this project is to implement *S* parameter measurements traceability for nontraditional impedance domains, focusing on balanced impedance and extreme impedance.

The work on balanced impedance was completed with the development of on-wafer mixed mode *S* parameter metrology as reported in the previous report, and an analysis of uncertainty propagation through the Multimode TRL calibration algorithm applied to ground-signal-ground-signal-ground (GSGSG) coplanar waveguide structures (CPW). An interesting result was the nonlinear uncertainty propagation of uncertainties using such an algorithm.

The second part, which deals with extreme impedance has then started with a preliminary work on the development of coaxial high impedance calibration standards to be used for the calibration of an interferometer based vector network analyser (VNA) as a feasibility demonstration, using the interferometric system available at LNE. The heart of the work will now be oriented towards the development of nanoscale impedance standards and the associated calibration method in the framework of a PhD work to be started soon.

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EMPIR project TEMMT (Traceable measurements at millimetre-wave and terahertz frequencies)

The aim of this European EMPIR project is to establish traceability of S parameters, RF power and complex permittivity of dielectric materials, at millimetre and terahertz frequencies. LNE is involved in the four technical objectives of this project.

First, coaxial airline diameters measurement capability has been extended to the 1.35 mm connector and LNE has participated in an interlaboratory comparison where both the inner diameter and the outer diameter of the outer and inner conductors, respectively, were measured using an air gauge system and an laser interferometer, respectively. A model has also been developed using 3D electromagnetic simulation, which allows parametrization of the shape of the 1.35 mm connector as an input to uncertainty evaluation of the interconnection.

Second, LNE has worked on the design of an on-wafer VNA calibration kit operating between 110 GHz and 1100 GHz, optimized to provide eight identical chips on the same wafer, to allow all project partners involved to conduct an interlaboratory comparison and uncertainty evaluation.

For the third objective, LNE has developed a twin type microcalorimeter in the waveguide band between 110 GHz and 170 GHz with the objective to calibrate new generation IEC WR07 power sensors, using bolometric and thermoelectric power measurement principles and to participate in an interlaboratory comparison.

Finally, LNE has participated in interlaboratory comparison on material electromagnetic measurement using terahertz spectrometers and vector network analysers. LNE has set up their time domain THz spectrometer for this comparison.

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EMPIR Project ADVENT (Metrology for advanced energy-saving technology in next-generation electronics applications)

The overall objective of this project is to achieve traceable and accurate measurements of the power consumed by ultra-low power and high frequency energy efficient electronic materials, devices and systems in order to support their development in both industrial and research sectors. LNE coordinates the project and is mainly involved in the first and third technical objective.

In the first objective, LNE is involved in the improvement of impedance measurement traceability at nanoscale and in the characterisation of advanced materials such as piezo and ferroelectric materials. LNE has designed capacitance standards (capacitance values from attofarad to femtofarad) and carried out their characterization by evaluating impact of different parameters (dimensions, probe positioning, humidity…). The uncertainties on capacitance measurements using capacitance standards fabricated are in the order of 10 %. To improve the reliability of dielectric constants of promising ferroelectric and piezoelectric materials, an SMM measurement campaign was carried out using two different piezoelectric materials after deposition of gold pads on their top surfaces. A strong variation in dielectric constant was observed between NMIs (LNE an METAS) indicating that measurement procedures for piezoelectric measurements need further investigation and improvement.

In the third objective, LNE has developed an on chip power standard to improve the on-chip power measurement traceability. The on-chip power standard is based on thermoelectric effect and has been fabricated on GaAs substrate. The standard has been completely characterized in terms of matching, linearity, sensitivity and scattering-parameters (S_{11} is below -15 dB up to 50 GHz). The power standard has been used in a power comparison to establish the traceability of the BI-CMOS power sensor developed by the University of LILLE.

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High frequency power calibration: traceability and extension towards terahertz frequencies

In 2018, LNE has participated to the international CCEM.RF-K27.W Key Comparison. The comparison is still in progress. LNE has already calibrated two power transfer standards from 50 GHz to 65 GHz.

In 2020, LNE has started a new national research project to improve and extend traceability of power measurement up to 170 GHz. In the framework of this new project, LNE is developing power transfer standards as well as primary power standards (microcalorimeters): coaxial transfer standard in 1.85 mm connector and waveguide transfer standards (R620, R900, and R1400). In the first year of the project, LNE has studied the impact of RF frequency parameters (transition between coaxial connector and planar transmission line, metallic and dielectric losses, and radiation losses) on the matching of the 1.85 mm coaxial sensor through electromagnetic simulations.

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