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CCEM/15-Report-LNE

Report on the activities in Electricity and Magnetism within the LNE between 2013 and 2015
29th CCEM Meeting, March 12-13 2015

1. Introduction

As usual, this progress report highlights the main results obtained by the Laboratoire National de Métrologie et d'Essais (LNE) in the field of Electricity and Magnetism over the last two years.
This field used to be is shared by two Divisions at LNE : "Advanced Metrology Research" dealing with fundamental electrical metrology and "Electrical Metrology" in charge of the electrical standards in the subfields of "DC & Low Frequency" and "microwave & RF" and including a calibration centre.
However there was an organizational change in July 2014 leading to the merge of both divisions into a global one, under the same management, divided into three Departments :

- Fundamental Electrical Metrology (including, among others, Thompson Lampard calculable capacitance, quantum electrical metrology, watt balance)
- Low Frequencies Metrology
- High Frequencies Metrology

The scopes of the previous teams were maintained except that the nanometrology activity moved to another LNE Division (Energy & Photonics).

In addition to the following highlights, it is worth pointing out that LNE further developed the "Club nanoMétrologie" founded in 2011, aiming at bringing together all private and public stakeholders involved in the measurement at nanoscale (industrial companies, academic entities, laboratories, governmental agencies…). This growing network of experts and users (310 members today - more than 30% from industry) will be able to prioritize the needs of industry at national level in the field of nanometrology.

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2. Watt balance

After separate developments of the different elements with continuous characterizations and improvements, the LNE watt balance has been assembled. Measurements of the Planck constant *h* were performed in air during the summer of 2014.

The value 6.626 068 8(20) $\times 10^{-34}$ Js has been extracted from these data. It differs in relative terms by -0.05×10^{-7} from the h_{90} value and by -1.1×10^{-7} from that of the 2010 CODATA adjustment of h. The relative standard uncertainty associated with this measurement 3.1×10^{-7} is thus larger than these differences. Currently, the major contributions to the uncertainty arise from voltage measurements, velocity measurements and suspension alignments. These contributions are not yet a limitation of our experiment and it will certainly be possible to reduce them. Part of this work was funded by the European Metrology Research Program (EMRP) in the framework of the KNOW joint research project.

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3. Thompson-Lampard calculable capacitance & AimQute

The operations of grinding and polishing the set of electrodes are still on-going. A first electrode is finished, with a cylindricity defect lower than 100 nm (50 nm on 80 % of its surface). Its surface's roughness is of the order of 5 nm. The final polishing process has been improved by alternating local polishing of the bumps and global polishing with protecting the gaps. The complete set of five electrodes will be available during 2015.
The adjustment process of the cavity of the standard has begun. The sub-micrometric-adjustment systems have been tested in-situ. These systems allow to position the electrodes with a resolution of 30 nm. They are within the initial objective. Their operating range is only 60 µm, which demands the initial positioning of the electrodes to be at least at 60 µm from the final position. In the current version of the standard, it is not possible to achieve the first positioning of the electrodes with this level of accuracy. A supplementary system of adjustment is under development which will allow pre-positioning the electrodes within 40 µm from the final position.
The integrated measuring machine present in the standard has been interfaced, it is operational and used during the process of the adjustment of the cavity. All the necessary mechanical equipments needed for the implementation of the movable guard have been realized. They will be implemented and tested after the next disassembly of the standard.
The LNE has participated in the EURAMET EM-S31 comparison, supplementary measurements of frequency dependence of 1.29 kΩ resistors have been performed and a second round of measurement of 10 and 100 pF capacitors, including NMIA allowing traceability to a Thompson Lampard capacitor, is in progress.
A new standard transformer has been realized and calibrated. The first results showed that the 10:1 ratio correction has been decreased from $9\cdot10^{-7}$ to $3\cdot10^{-7}$ at 1592 Hz compared to the previous ones. The voltage variation of the ratio stays within $2\cdot10^{-9}$ from 10 V to 200 V. The global uncertainty of the 10:1 ratio correction is lower than $1\cdot10^{-9}$. The transformer calibration bridge has been modified, the Wagner network has been removed, the voltage reference point is now defined with the guarding transformer and the UHF connectors has been replaced by MUSA ones. These modifications have allowed to decrease the measurement time and improved the reproducibility of the measurement by a factor of ten.
A new Haddad type coaxial calculable resistor of 1.2906 kΩ close to its nominal value ($5\cdot10^{-5}$) has been fabricated with the BIPM support. It will allow to determine with a lower uncertainty the frequency dependence of the 12.9 kΩ resistors used for the R_K determination at 1233 Hz.
Concerning the AIMQuTE project, the objective is to extend the range of traceable capacitance measurements to very small capacitance values (10 aF for the smallest value) so as to be able to calibrate measuring systems connected on devices such as probers or functionalized atomic force microscopes used in micro and nano-electronics industry.
The first stage consisted of designing and constructing the capacitance standards with low values from 10 aF to 100 fF. Two kinds of architectures have been analysed namely, Zickner's air capacitor and lithographed capacitor. Nine Zickner's capacitors with values of

10 aF, 100 aF, 1 fF, 10 fF and 100 fF and a lithographed capacitor of 10 aF have been developed.
The characterization measurements carried out on the first 10 aF prototypes between 800 Hz and 20 kHz have shown a good agreement with the expected value obtained from modeling. These measurements have been carried out with a two-terminal pair capacitance bridge used in -1:1 ratio configuration comparing successively two capacitances of 10 pF and later, one 10 pF in parallel with 10 aF to 10 pF. The difference between these two measurements gives the 10 aF value. A relative uncertainty of 0,3 % has been estimated at 1 kHz. The study of a programmable capacitance including a coaxial and low noise multiplexer and lithographed capacitors from 10 aF to 10 fF is in progress.
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4. QHE metrology

In the field of resistance traceability, LNE investigated the quantum Hall effect (QHE) in Hall bar devices made of graphene grown by several scalable production methods. In polycrystalline graphene, grown by chemical vapor deposition (CVD) on copper and transferred on SiO_2/Si substrate, LNE showed that the Hall quantization is strongly altered by the presence of line defects such as grain boundaries and wrinkles. This directs towards the investigation of the QHE in single CVD graphene crystals.

The accuracy of the Hall resistance quantization was demonstrated with relative uncertainties lower than one part in 10^9 in large Hall bars (100 µm x 420 µm) made of graphene grown by CVD on silicon carbide, in experimental conditions unattainable to GaAs and any usual semiconductors: magnetic fields as low as at 3.5 T, or temperatures as high as 5.1 K, or measurement currents as high as 300 µA. This makes a new breakthrough in the resistance metrology field. These relaxed conditions of operation indeed open the way to the development of an affordable and user-friendly helium-free quantum resistance standard, able to be widely disseminated towards National Metrology Institutes and calibration services. By measuring the agreement of the quantized Hall resistance in such a grapheme device and in a conventional GaAs device within an uncertainty of 8.2×10^{-11}, LNE gave a new convincing proof of the QHE universality.

The new resistance comparison bridge was used to perform the highly-accurate calibrations of resistors in terms of the GaAs and Graphene-based quantum resistance standards. For a nominal voltage of 0.6 volts, the ratio 100 Ω /(R_K/2) can be measured with a relative type A uncertainty of two parts in 10^{10} after 1 hour measurement.

Beyond, LNE showed, in principle, how the quantum resistance standard can be combined with the Josephson voltage standard using a cryogenic current comparator to provide an accurate and practical quantum current standard linked to the electron charge.

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5. Quantum ampere based on SET

The work of the last two years was carried out in the frame work of the EMRP JRP Quantum ampere. In the context of the redefinition of SI based on fundamental constants, the ampere, will certainly be redefined from the electron charge and the second. For the realization of this

future definition, the metrologists work on the development of a quantum standard of current based on a single electron tunneling device and which would be able to generate a current of about 100 pA within an uncertainty of one part in 107. For the last two years, we have investigated electron pumps based on silicon nanowires and fabricate at CEA-LETI. The principle of pumping electrons in these devices is based on Coulomb blockade and single electron tunneling effects. The devices we have studied consist in silicon nanowires with two top gates in series which act as tunable barriers. By applying alternative signals with frequency f on both gate electrodes, a quantized current I=ef is generated. The dependence of the quantized current delivered by these hybrid metal/semiconductor electron pumps have been measured as a function of different parameters at temperature below 1 K. This characterization was done with a 20000 turns cryogenic current comparator (CCC) in internal feedback mode. Stability measurements over more than 10 hours have been demonstrated with a relative type A standard uncertainty of 4 parts in 106. To benefit from the high accuracy of the CCC, we are currently improving the traceability of the current measurement by comparing the electron pump current to the current generated by a Josephson array and a high value resistor with the CCC in internal feedback mode.
Beside, a Cryogenic Current Comparator (CCC) designed to be used as SET current amplifier with a maximum current ratio of 30 000 : 1 has been developed and fully characterized. The equivalent input current noise was found lower than 2 $fA/Hz^{1/2}$ over a frequency range 1 Hz to 350 Hz in both operating external and internal feedback modes. A model has been developed to characterize the CCC and allows one to determine analytically the electrical behavior of the CCC windings. and to estimate the AC current ratio error. The total error on 30 000:1 ratio is estimated less 10^{-8} at 1 Hz.
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6. Electrical nanometrology (Solcell, 3D stack)

LNE started in 2013 the development of a new research activity on the traceable measurement of electrical quantities at the nanoscale and based on scanning probe microscopy (SPM) techniques. This new activity is conducted in the framework of two European projects, SolCell proposed in the EMRP Energy call of 2013 and 3D Stack in the EMPIR Industry call of 2014. LNE is coordinating both JRPs which gather 14 and 8 European partners respectively.
SolCell is aiming at developing accurate and spatially resolved metrology to determine the complete material properties layer by layer of multi-junction solar cells (MJSC) based on III-V materials which are part of the third generation of photovoltaic cells with solar energy conversion efficiencies as high as 44 %. 3D Stack is addressing the measurement challenges related to the 3D integration of heterogeneous semiconductor technologies which uses copper Through Silicon Vias (TSV) to electrically connect a stack of chips-bonded semiconductor wafers and dies to produce 3D stacked integrated circuits (3D-SICs) with an optimum combination of cost, functionality, performance and power consumption.
For both projects, LNE is developing the instrumentation based on Scanning Microwave Microscopy and Resiscope, from the development of capacitance and doping profile staircases to the complete evaluation of the uncertainty measurement of SPM techniques.

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7. Smart Grids – A Metrology Framework For Phasor Measurement Unit (PMU)

LNE participate in ENG04 and ENG52 Joint Research Projects, in order to develop Metrology Grade Reference PMU with both static and dynamical performances.

In first JRP, PMU was design around the standard IEEE C37.118-2005 which only specifies static tests. However, this standard has been amended in 2011 and 2013 and includes demanding dynamic tests, so that the PMU needs to be upgraded in order to meet present industry requirements.

During the first JRP ENG04, LNE was involved in the conception and development of hardware architecture with National Instruments solutions and in the software implementation using Labview real-time environment. With the current second JRP ENG52, LNE is working on implementation of new suitable and performing algorithms inside PMU and on hardware evaluation/comparison of PMU algorithms using synthesized waveforms generated by PMU calibrator. This work includes the development of all the required test procedures and uncertainty budgets.

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8. Measurement traceability of impedance and S-parameters between 100 kHz and 100 MHz

This project (2012 - 2015) aims to improve the traceability of impedance and S-parameter measurements in the frequency bandwidth from few tens of kilohertz to one hundred megahertz. This work is mainly focused on the measurements performed with Vector network Analyzer (VNA), LCR-meter and Line Impedance Stabilization Network (LISN). During these last two years different devices and standards has been developed:

- 50 W load female and male in type N connector has been fully developed and its modelling established. These standards complete the traceability of S parameters of type N devices in the frequency bandwidth [9 kHz – 100 MHz]. LNE will submit at the end of 2015 new current measurement capabilities (CMCs) for male and female type N of one port and two port devices.
- Adapters for measuring input impedance of the ESH3-Z5 LISN have been developed. These adapters are fully calculable and their modelling is traceable to the International System of Units (SI) trough dimensions and permittivity measurements. Using these adapters improve the measurement accuracy and traceability of LISN. The correction applied to the measurement by using these adapters (up to 5° for the phase) is larger than uncertainties (2 ° for the phase) provided by test laboratories. For the first time, these adapters clearly demonstrate the great of importance of the connection used by EMC test laboratories.
- A Calibration kit in BNC connector has been developed by LNE in cooperation with an industrial partner. This calibration kit allows establishing the traceability of VNA from 9 kHz to 10 MHz and to measure four terminal pair (4TP) impedance standards in the whole complex plane. CMCs will be submitted in 2015 for calibrating 4TP impedances up to 10 MHz with uncertainties from a few.10^{-3} to a few 10^{-4} depending on the impedance value to be measured. LNE will be the first European National Metrology Institute (NMI) to propose CMCs for calibrating 4TP impedances up to 10 MHz.

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9. Improved EMC test methods in industrial environments

The JRP (2013 - 2016) aims to study and improve the existing methods used in the industry and adapt EMC test laboratory capabilities, to establish the uncertainty budget of these methods. The project also aims to develop new test methods in line with the needs of European manufacturers. So far LNE has developed adapters for measuring input impedance of ESH2-Z5 LISN, complementary work done in French metrology project. These adapters have been developed in cooperation with the Slovene NMI.

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10. High voltage measurements by sampling technique

The aim of the project is to develop a new method, based on the sampling technique, for calibration of High Voltage dividers : HV transformers, HV resistive dividers or HV capacitive dividers.

The voltage transformer manufacturers are constantly working to reduce the errors of the high voltage dividers. Recently, the voltage transformers with errors of less than 0,005 %, and the phase shift between the primary voltage (input voltage) and the secondary voltage (output voltage) is less than 50 µrad are manufactured for the frequency of 50 Hz. The calibration of these transformers requires methods with an uncertainty which is better than the voltage transformers errors.

To reach these uncertainties, LNE has developed a new method based on the sampling technique; the voltage transformer under calibration is compared to a high voltage standard; a voltage transformer standard, a resistive divider standard or a capacitive divider standard. The output voltages are sampled by two sampling voltmeters; the ratio error and the phase shift of the voltage divider under calibration are calculated with a discrete Fourier transformer algorithm.

The voltage is up to 300 kV at frequency of 50 Hz, up to 100 kV at 60 Hz-1000 Hz frequency range and up to 30 kV at 15 Hz-50 Hz frequency range.

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11. Non-conventional voltage and current sensors for future power grids (JRP-ENG 61 FutureGrid)

This JRP focuses on the possibilities of novel non-conventional sensors for current and voltage measurement e.g. optical Faraday effect based sensors, hybrid electrical/optical sensors and air core Rogowski coils for current measurement; and voltage dividers or optical Pockels effect based systems for voltage measurement. The project aims to extend the accuracy of these technologies, to find complementary solutions for the calibration of new non-conventional technologies including the conventional instrument transformers with digital or optical readouts.

This 3 years project started in June 2014. LNE is involved mainly in the characterisation of components of a high accuracy current sensor based on the Faraday effect. It participated to the selection of possible candidates for the sensing fibres for the FOCS. Currently, Electrical Metrology Department and Photonique Department are working togheter on the

characterisation of the optical fibers: the wavelength dispersion of the Verdet constant and it's temperature dependence (-35 °C – 85 °C).

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12. Electromagnetic field measurement for modulated and complex signals

The purpose of the JRP Ultrafast (finished in 2014) and the on-going JRP MORSE is to develop traceable measurement methods to support the increase of communication data exchange. Area covered by both JRP are the development of traceable methods for the characterization of photodiodes and photodiodes modulator, the realization of in-door channel measurement at high frequencies (65 GHz) and the calibration of the antennas used for these measurement. LNE has manufactured and integrated a spherical far-field range covering the frequency band from 18 GHz to 75 GHz using micro positioner with a resolution of few tenths of degrees and several microns for antenna pattern measurement and gain calibration. The current JRP MORSE will support further this development by enabling the traceable measurement of planar near-field and the calculation of the far-field gain with propagation of uncertainties. The final objective is to develop and validate an algorithm that will correct the intrinsic deviation on gain when using an imperfect illumination antenna, with application to the Compact Antenna Test Range (CATR) systems. The methodology is developed with the support of the University of Rennes 1 (UR1) for the access to a CATR facility and measurement inside it, and with the Technological University of Delft (TUD) for the development of the algorithm and of a reference antenna.
Other activities developed in the frame of the JRP MORSE include the measurement of antennas under a temperature constraint and the measurement of telecommunication signals using several systems to compare direct power measurement to power calculation from time domain measurements.

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13. Electromagnetic characterization of materials for industrial applications up to microwave frequencies (including JRP-IND02 Eminda)

The aim of this project is to develop both traceable electromagnetic materials metrology and techniques of electromagnetic characterization of materials at the submicron scale at microwave frequencies (up to 110 GHz). The S-parameters measurement on coplanar waveguide was required to determine the electromagnetic parameters of materials. In order to obtain these results and their uncertainties, we developed an algorithm on MATLAB allowing the extraction of the electromagnetic parameters from the measurement of S-parameters. This algorithm was validated with measurements performed on materials whose parameters are known, such as quartz and alumina. Once the algorithm was validated, we worked on the extraction of the electromagnetic parameters of ferroelectric thin film. Two ferroelectric materials were characterized, the Barium Strontium Titanate (BST) and Lead Zirconate Titanate (PZT). The idea consisted in depositing this ferroelectric thin film between the coplanar waveguide and the wafer (quartz or alumina). Thus, we extracted the electromagnetic parameters of ferroelectric materials and their uncertainties on the frequency range up to 110 GHz.

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14. Traceable reference materials for calibrating terahertz measurement systems (including JRP-NEW07 Terahertz)

This work was carried out in the framework of European project EMRP NEW07. Our task was to establish a list of traceable reference materials, with uncertainties given for both absorption coefficient and refractive index, for a broad range of frequencies from a few hundreds GHz to few THz. A range of values and frequencies required for the calibration of THz spectrometers commercially available was selected. The transmission measurements and data processing were performed by LNE, among other project partners, on a time-domain spectrometer. The established uncertainty budgets on the selected materials like alumina, quartz, silicoin and parylene were generally ranging from 1% to 7%. In all cases, the main contributors are thickness of the sample and repeatability and reproducibility of the measurement.

Human skin equivalent materials for exposure assessment were also selected and evaluated, based on published recipes. The samples and recipes were provided to the other project partners for traceable measurements.

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15. HF circuits

This work is carried out in the framework of European project EMRP SIB62, started in 2013. The work is mainly on the traceable characterization of Vector Network Analysers (VNA) and calibration standards for S parameters measurement in order to extend the European capability in terms of calibration with special focus on frequency extension (up to 1,1 THz in waveguide and up to 110 GHz in coaxial line), on mixed mode, extreme impedance and nonlinear measurements and on update of uncertainty evaluation procedures.

LNE worked on the full characterization of a VNA in coaxial line up to 50 GHz and in waveguide in the range 110 GHz to 170 GHz, according to the procedure established by the project partners 45 MHz to 50 GHz waveguide 110 GHz to 170 GHz. LNE worked also on the establishment of traceable measurements in the 1.85 mm coaxial line based on dimensional measurement of verification standards, the design of planar (microstrip and stripline) calibration standards for mixed mode S-parameters measurement and the establishment, in collaboration with MSL, of a procedure for the automatic propagation of uncertainties in the case of the TRL calibration algorithm and equivalent algorithms where the equation systems generally do not take all calibration standards characteristics into account.

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16. Microcalorimeter for calibrating thermoelectric sensors from DC up to 50 GHz

The purpose of this project is to solve the progressive unavailability of thermistor mounts in the market, which were playing the role of primary reference standards for RF power. In order to replace these devices, manufacturers such as Rohde & Schwarz and Keysight Technologies have developed coaxial thermoelectric sensors with frequency ranges from DC up to 110 GHz. The calibration of thermoelectric sensors with a microcalorimeter is made by measuring the sensor heating difference between a low frequency level and a high frequency

level for a same sensor output voltage. LNE is going to upgrade these microcalorimeters in order to operate with thermoelectric sensors. During year 2014, LNE sold to a third-party NMI a full system allowing the calibration of thermoelectric sensors from DC up to 50 GHz with 2.4 mm connector and LNE plans to sell other microcalorimeters operating with thermoelectric sensors. So, LNE acquired an important knowledge in high-frequency metrology.

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17. Development of a coil for characterization of magnetic field meters

The regulations for protection of people against electromagnetic field is based on recommendation 1999/519/EC of April 1999 and directive 2004/40/E of July 2004 that should be applied to the French law. Taking into account this context, the LNE aimed to increase its capacities in magnetic field. New range from 0 T to 5 mT with frequencies from 0 Hz to 10 kHz has be reached and accreditation extension was approved by COFRAC in 2014. The main tasks of this project were the definition, the acquisition and the characterization of a Helmholtz coil system. This system is based on two parallel and identical coils with single axis.

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