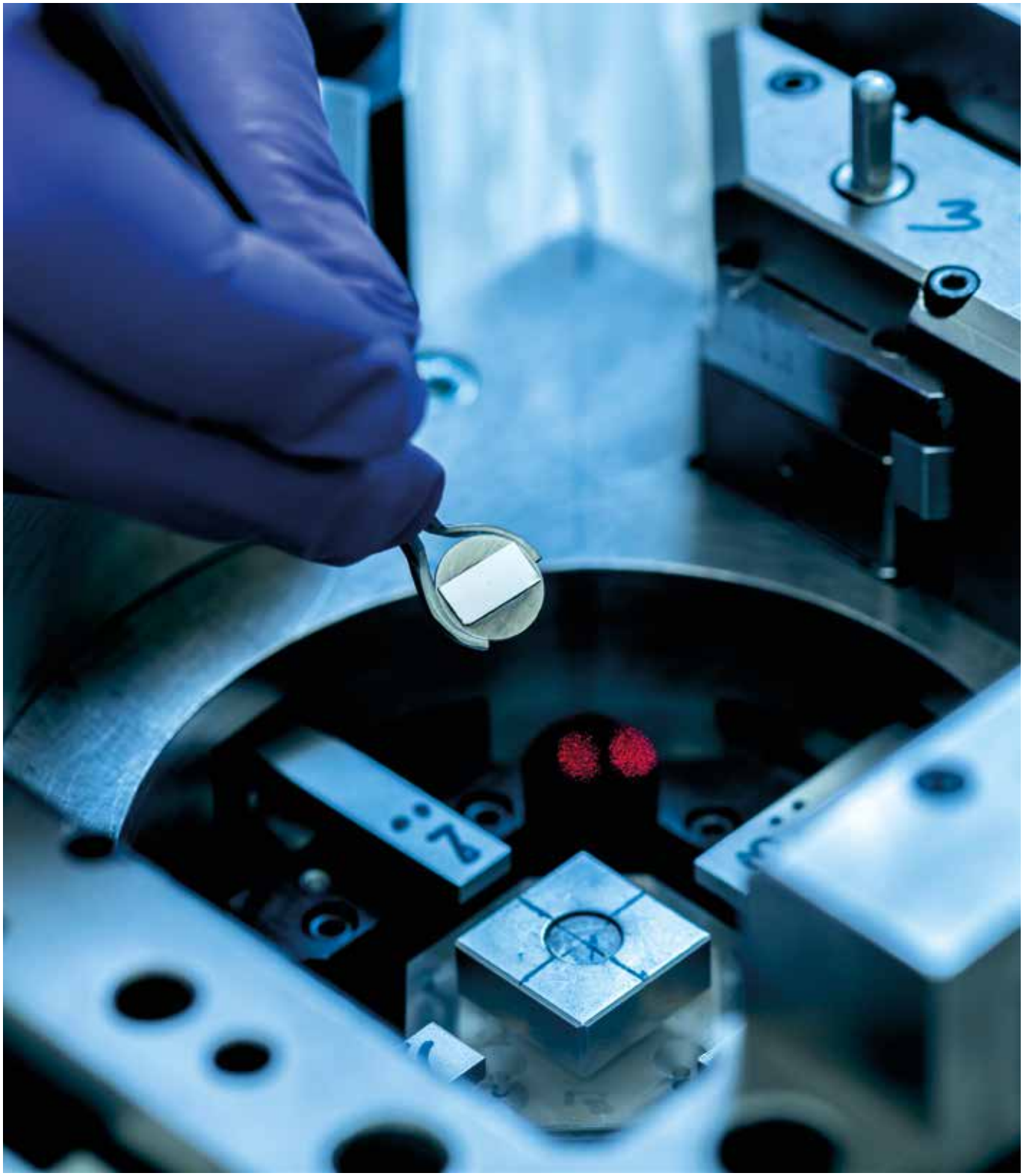


RESEARCH REPORT
2018

LABORATOIRE
NATIONAL
DE MÉTROLOGIE
ET D'ESSAIS

SUMMARY

COVER PICTURE:
METROLOGICAL ATOMIC FORCE MICROSCOPE.

FOREWORD

Thomas Grenon,
Managing Director

2018 was undoubtedly a historic year for metrology! The redefinition of the units of the International System (SI) enacted in November during the 26th General Conference on Weights and Measures (CGPM) marked a turning point in the world of measurement: the redefinition of all units of the International System on an immaterial basis, and particularly the kilogram, whose definition had not changed since 1889. This was a turning point to which the researchers of the LNE and the French National Metrology Network made a significant contribution through the work that they have done over many years. I am thinking in particular of our contribution to the redefinition of the kilogram using our watt balance, one of the three in the world and the only one in Europe, and our contribution to the new definition of the kelvin, with work that was able to determine the Boltzmann constant (*k*) at the best level worldwide. This historic event also marked an opportunity to highlight the major contribution of metrology to our society, to the safety of our fellow citizens and the competitiveness of our companies and to position our organisation as an essential player in research at the national and international levels.

Amongst this work, marking the 10th anniversary of our Research Prize, we recognised the contribution of two researchers in quantum electronic metrology who significantly contributed to the new definition of the ampere.

Reliability of medical analyses, storage of hydrogen, measurement of radioactivity, additive manufacturing, artificial intelligence, nanotechnologies. Once again this year, the diversity of the research topics covered by the LNE shows the wealth of projects carried out by our teams. Whether it is to protect citizens or support breakthrough innovations, the LNE is doing everything it can to advance our knowledge of metrology and ensure a safer and more innovative world. ■

LNE RESEARCH IN 2018:

25%
of the overall budget of the LNE

85
research projects

70
publications in peer-reviewed journals

18
doctoral students

200 doctors and engineers

A portfolio of **14 patents**

FRENCH NATIONAL METROLOGY NETWORK RESEARCH IN 2018:

148
research projects including 60 JRPs (*Joint Research Programs*)

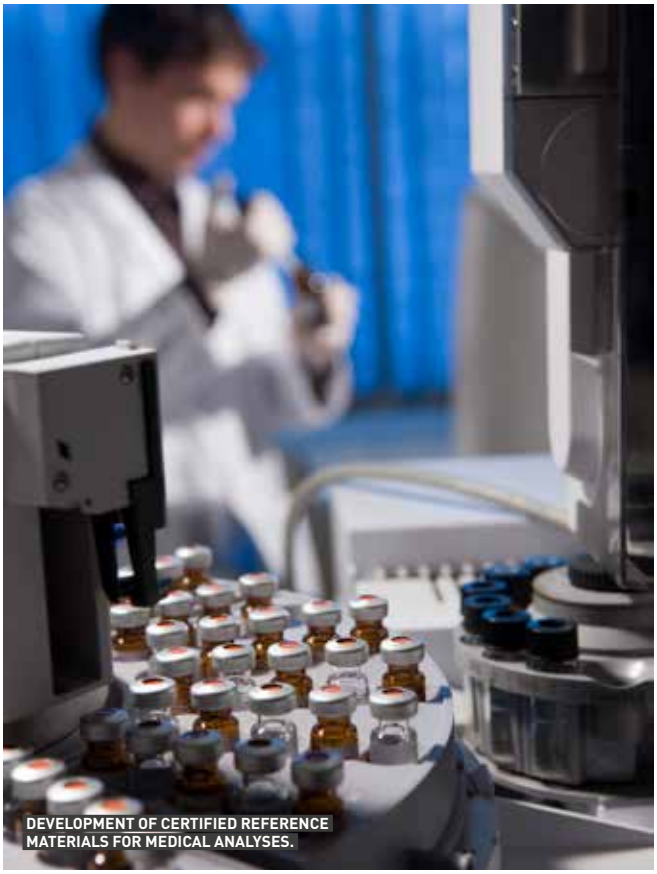
156
publications in peer-reviewed journals

250
communications

50
PhD thesis in progress

MEDICAL ANALYSES: SAMPLES TO IMPROVE RELIABILITY

The LNE has developed samples representative of those from patients for which the target values are certified to evaluate the reliability of several medical tests.



DEVELOPMENT OF CERTIFIED REFERENCE MATERIALS FOR MEDICAL ANALYSES.

The quality of a patient's course of treatment depends on that of the examinations they undergo. When it comes to medical analyses, however, the various methods used by the laboratories to dose the biological markers do not offer equal performance, which may lead to results that are contradictory and difficult to compare.

As a result, in 2016, the French National Agency for the Safety of Drugs and Health Products (ANSM) asked LNE to produce samples used for the mandatory national quality control, in which all medical biology laboratories are required to take part. This year, this control was based on certified reference materials as commutative for the first time.

In concrete terms, the laboratory researchers had two objectives.

> *« An outstanding showcase for the laboratory, our contribution to the national control of the ANSM is the culmination of 10 years of work. »*
>
> Vincent Delatour, biomedical-biomarkers expert.

First, certifying the target values of samples using reference methods validated internationally. *"Previously, we used the average of the results obtained by all participants as the target value, so significant risks of interpretation errors were possible,"* says Vincent Delatour, one of the researchers involved in the study. Secondly, we must guarantee what the specialists call the commutativity of samples, meaning that they satisfactorily mimic the behaviour of real samples.

In fact, for tests on such a scale, it is impossible to use the blood of a single patient, hence the use of mixtures. However, in 2016, the researchers at the LNE showed that according to the way in which these samples are prepared, they may have properties which move them away from real samples and can cause significant bias in dosage. The consequence was significant work by the researchers to select the best samples and guarantee their content in glucose (a marker of diabetes), in creatinine (a marker of renal insufficiency) and in cholesterol. *"An outstanding showcase for the laboratory, our contribution to the national control of the ANSM is the culmination of 10 years of work in developing methods for relating medical dosage to the international system of units,"* said the scientist. This work is now continuing for other biomarkers involved in the detection of neurodegenerative diseases, septicaemia and cardiovascular diseases. This will eventually produce reference materials for calibrating routine methods.

THE CARDIOVASCULAR RISK UNDER THE MICROSCOPE

The cardiovascular risk is determined from the dosage of different lipids, known as conventional markers. However, the real risk for the patient of developing cardiovascular diseases often depends less on the absolute concentration of these molecules (cholesterol, triglycerides, etc.) in the blood, than on the concentration of lipoproteins or biological nanoparticles which transport cholesterol in the blood. Hence the idea of the LNE's researchers to develop new reference methods arising from nanotechnologies that will be able to highlight them. *"The methods are ready and the standards are available,"* says Vincent Delatour. It is now up to the laboratories to use them! ■

BRACHYTHERAPY: TOWARDS NEW REFERENCES

Based on the introduction of an active source into the body of the patient, radiotherapy can deliver significant doses to the target volume, typically a tumour, while avoiding other tissues. Other than iridium 192 (^{192}Ir) sources, more and more treatment centres are now using cobalt 60 (^{60}Co) sources. At comparable dose distribution curves, this last element has a longer half-life, which simplifies logistics, especially those relative to the rules on radiological protection in terms of transport and renewal.

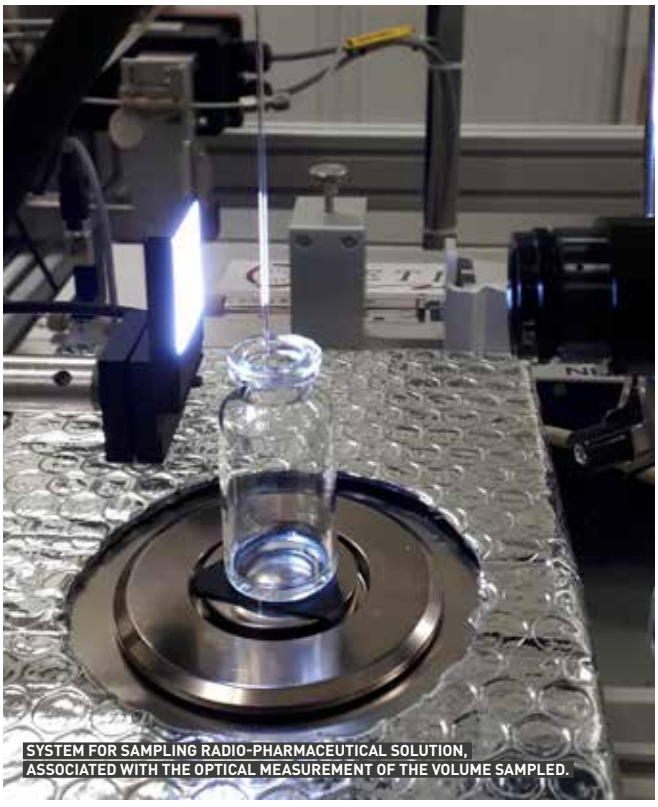
Against this background in 2018, the LNE-LNHB renewed its radiotherapy source projector, choosing a projector that can be alternatively loaded with ^{192}Ir and with ^{60}Co. In order to establish new references for the calibration of clinical radiotherapy sources, the laboratory started by calibrating a cavity ionisation chamber using its beams, which were metrologically characterised. Next, it used the same chamber to characterise the source of ^{192}Ir for its new projector. *"Indeed, last year, we established a new reference for ^{192}Ir,* explains Isabelle Aubineau-Lanièce, laboratory manager. *And we should soon have finished establishing the one relative to ^{60}Co."* A treatment centre will then be able to send its own medical dosimeter to LNE-LNHB for calibration, which can then be used for *in situ* calibration of medical sources. As a result, the laboratory is renewing its calibration offer for ^{192}Ir and providing a solution to the lack of a reference for ^{60}Co. ■

THE TRACEABILITY OF RADIO-PHARMACEUTICAL PRODUCTS WITH VERY SHORT HALF-LIVES IS NOW WITHIN REACH

Before it is injected, a radio-pharmaceutical product must have its activity measured using a calibrated activity meter. However, for products with very short half-lives – several minutes – there is no primary activity standard. Indeed, it is materially impossible to bring radioactive solutions from their place of production to the site where the primary measurements are taken with equipment that is then used for calibration. To overcome this shortcoming, the researchers at LNE-LNHB, in collaboration with their colleagues at LNE-CETIAT, have developed a means of primary measurement that can be transported to the radio element production site.

To do this, the specialists made use of a portable activity measurement device developed at LNE-LNHB. Moreover, in order to collect the certified nano-volumes of the products to be analysed, they proposed a system of sampling using a capillary and a means of optical measurement. As Philippe Cassette, the project manager, explains, "*in the laboratory, we use a precision balance to deduct several micrograms. But this equipment is impossible to move to the production sites.*" Another benefit concerning the handling of liquids at very high activity: everything is automated, enabling the operators to intervene remotely.

Validated in the laboratory, these two subsystems are ready to be integrated and the whole system will be tested during the year at the Frédéric Joliot hospital in Orsay. This establishes the first traceable chain for radio elements with very short half-lives. ■



SYSTEM FOR SAMPLING RADIO-PHARMACEUTICAL SOLUTION, ASSOCIATED WITH THE OPTICAL MEASUREMENT OF THE VOLUME SAMPLED.

THE METROLOGY OF NANO-AEROSOLS: TOWARDS A DEDICATED CHARACTERISATION

In order to offer tools to evaluate the health risks of nanoparticles, the LNE is developing reference methods for the measurement of size and particulate concentration in the aerosol phase.

Over the last 20 years, technological progress has highlighted the potential of nano-materials in numerous fields. Nevertheless, the production of a very large quantity of nano-objects, due to their small size, associated with their very strong chemical reactivity, raises the question of their risk for health and the environment. There is therefore a significant regulatory requirement, for which the development of harmonised reference methods validated for the physicochemical characterisation of nano-materials is necessary, particularly when they are in the form of aerosols.

Accordingly, last year, French metrologists continued their work finalising reference methods for the measurement of the size distribution of nano-objects in the aerosol phase. In particular, the specialists developed protocols for aerosol generation using a method known as electro-atomisation. The advantage is that, in contrast to conventional mechanical methods, it drastically limits the production of impurities which complicate the subsequent characterisation of the diameter of the nanoparticles. *"We have validated the measurement protocol associated with this aerosol generation technique, the whole of which offers a reference system with a very good signal-to-noise ratio,"* says François Gaie-Levrel, project manager.

At the same time, researchers at the LNE took part in an inter-laboratory comparison for the size characterisation of nanoparticles, carried out within the nanoMétrologie club. *"We worked using a mechanical method of aerosol generation common to the different laboratories involved in this comparison"*, explains the researcher. *"And we achieved very good overall coherence for the granulometric distribution measurements of nanoparticles of silicon dioxide, in particular."* A key benefit is a common evaluation protocol and a guide to best practice for these measurements, which are traceable using the International System.

> *"TiO_2 is used for its specific properties, such as its bactericidal, fungicidal or photocatalytic characteristics."*
> Dr François Gaie-Levrel, engineer in aerosol metrology.

Lastly, LNE began work with toxicologists. The objective was to evaluate the neurotoxic risks associated with the inhalation of a model pesticide with an additive consisting of nanoparticles of titanium dioxide. *"TiO_2 is used for its specific properties, such as its bactericidal, fungicidal or photocatalytic characteristics*, says François Gaie-Levrel. *"Nevertheless, we want to make sure that these benefits are not accompanied by other effects that are harmful for health."* Accordingly, as part of a doctoral thesis, the researchers developed an in vivo exposure bench. This can produce the model pesticide with a nano-additive in the form of an aerosol characterised metrologically in terms of granulometric distribution, morphology and total concentration by mass and by number. Following this, the installation was sent to partners for the toxicological testing phase, which is currently ongoing. This should establish any link between the physicochemical parameters of nano-materials and their risk for health. ■

MEASUREMENT OF THE GRANULOMETRIC DISTRIBUTION OF NANOPARTICLES BY DIFFERENTIAL ANALYSIS OF ELECTRIC MOBILITY COUPLED WITH AN ELECTROSPRAY.

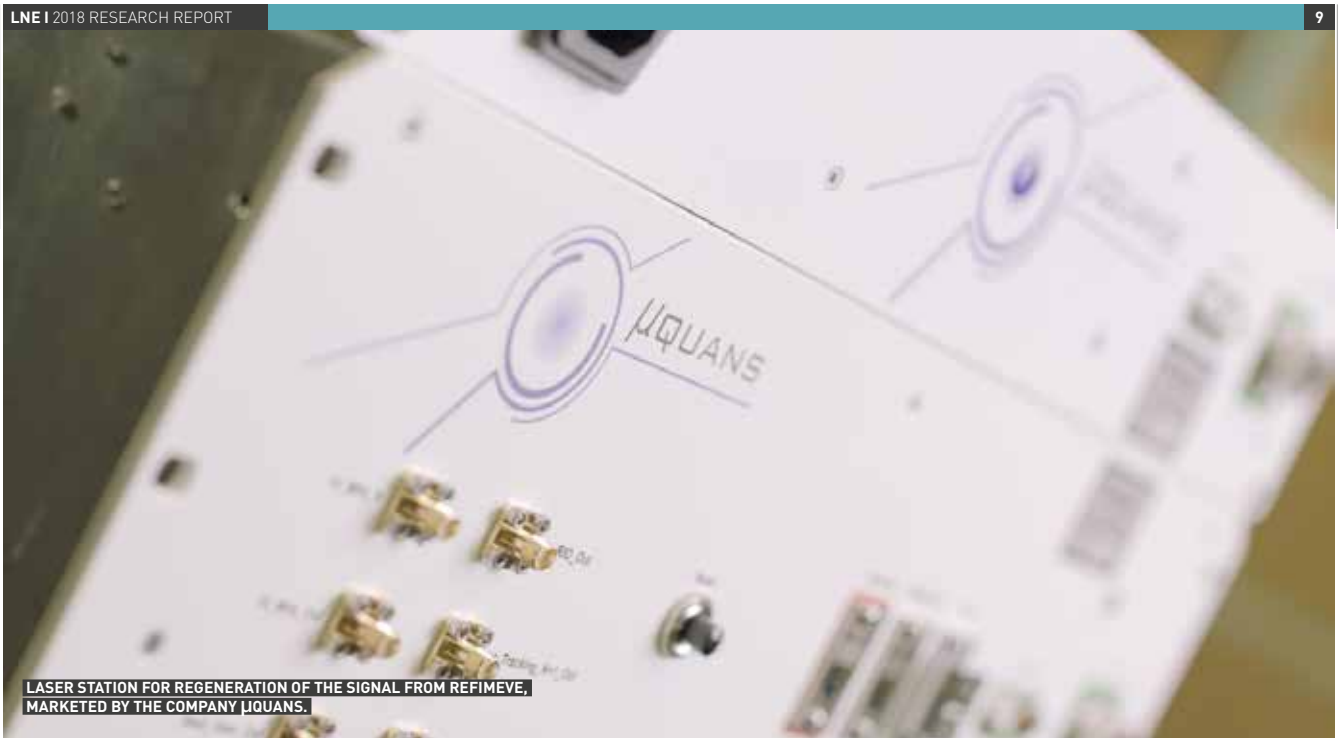
A EUROPEAN METROLOGY NETWORK FOR THE CLIMATE AND THE OCEAN

In the context of global warming, it is essential to have reliable metrology of quantities to monitor the development of climate changes. Furthermore, the climate is one of Europe's priorities. Accordingly, in 2018, EURAMET created a European Metrology Network (EMN) for the climate and oceans.
Its objective is to organise and coordinate the activities of the European metrological community, interacting with the various stakeholders (scientific communities, regulatory organisations, etc.), on the topics concerned.
In practice, the EMN, which is managed by the NPL (United Kingdom) covers three main areas: atmospheric, terrestrial and oceanic variables (for which LNE is responsible). *"We are going to start by making an appraisal of the requirements of our stakeholders and the skills available within the metrology laboratories, following which, we will prepare a research programme that eventually aims to establish reference standards, as well as guides to best practice in measurement and training media for users,"* explains Paola Fisicaro, the manager of the project for LNE.
The French laboratory is concentrating its efforts on the parameters associated with ocean acidification. In past years, a lot of work has been done to establish metrological references for measuring the pH of marine waters. *"At the same time, we are soon going to launch a similar programme for measuring total alkalinity,"* adds the chemist. This will eventually provide a global solution to a challenge that is itself global! ■

REFERENCE SOLUTIONS FOR pH MEASUREMENT OF SEAWATER CHARACTERISED BY THE LNE USING THE PRIMARY CELL (HARNED CELL).

METRODECOM II: STANDARDS FOR NUCLEAR DECOMMISSIONING

The decommissioning of a nuclear facility requires a prior inventory of its radioactivity. To do this, the CEA has developed the GAMPIX gamma camera, capable of mapping the "hot spots" of a site. As part of the MetroDecom II project, the specialists at LNE-LNHB made it operational using innovative calibration solutions.
The principle is simple: the camera's response is compared to radioactive sources with known activities. In practice, however, the metrologists had to cope with a dual objective for GAMPIX: first, to determine radioactivity levels correctly and secondly, to distinguish the numerous radionuclides that may be present on a nuclear site that is going to be decommissioned.
As project manager Valérie Lourenço explains, *"Usually, we provide radiation sources containing only a single radionuclide. These are obtained by depositing a drop of liquid that we dry before measuring between two solid layers."* Conversely, in this case, the metrologists had to develop sources composed of up to 192 drops of different radioisotopes, the whole having to form a homogeneous "material". An additional difficulty was that to ensure the calibration was as close as possible to the realities on the ground, it was decided to use a flexible, distorting substrate that adapts to different geometries.
Thanks to the standards offered by LNE-LNHB, a campaign of calibration and tests demonstrated the ability of GAMPIX to distinguish several radionuclides according to their emission spectrum. This will eventually enable optimal use by industrial partners during decommissioning operations. ■



LASER STATION FOR REGENERATION OF THE SIGNAL FROM REFIMEVE, MARKETED BY THE COMPANY µQUANS.

TIME-FREQUENCY REFERENCES: THE PRECISION OF FIBRE ACCESSIBLE TO ALL

The first coherent fibre link run by an industrialist for the dissemination of atomic time shows the maturity of the technology, to which LNE-SYRTE made a significant contribution.

In 2018, an industrialist – µquans – equipped and operated a coherent fibre link for delivering time references and frequencies between Paris and Lille for the first time. This was more than a symbolic feat and illustrates the validity of the Equipex REFIMEVE strategy. Since 2011, it has brought together user academic laboratories, the laser physics laboratory, LNE-SYRTE, the RENATER network and a consortium of industrialists, with the aim of making fibre the reference channel for the dissemination of atomic time.

Currently, time references are exchanged by satellite. Yet, at best, this provides a relative precision of 10^{-15} of a second, representing a level of uncertainty that is barely compatible with the precision of the best microwave clocks. And it will be totally insufficient when, as is likely in the medium term, the time unit will be redefined based on "optical" atomic clocks, which are accurate to 10^{-18}!

> *« This project illustrates the success of the transfer to industry of the technologies for the dissemination of atomic time by fibre."*
>
> Paul-Eric Pottie, research engineer at LNE-SYRTE.

Optical fibre is the solution to cope with the challenge: *"guided propagation, little loss and less noise; it's the ideal channel for exchanging time-frequency references,"* says Paul-Eric Pottie, of LNE-SYRTE. Hence the REFIMEVE project, which aims to develop and then industrialise, the necessary instrumentation (amplifiers, repeaters, etc.) for the distribution of frequency references on an existing fibre network.

The result: atomic time is currently delivered by RENATER, the national network for technology, teaching and research, to several fundamental research laboratories. For example, the Laser Physics Laboratory in Villetaneuse, for fundamental tests on parity violation, a fundamental symmetry, sought in molecules. Also, work with European partners has enabled comparisons of clocks by fibre networks at a level of precision never before achieved, notably between the French metrology laboratory and its counterparts, the German PTB and the English NPL.

The hurdle overcome by Muquans last year is therefore the logical completion of the process. *"It illustrates the success of the transfer to industry of technologies for the dissemination of atomic time by fibre, which are going to start to be disseminated beyond REFIMEVE,"* says the physicist.

With this in mind, LNE-SYRTE has been coordinating the European CLONETS project, involving 19 partners until 2019, for just over a year. The objective is to extend the REFIMEVE approach to Europe and beyond the academic sphere. *"After the success of REFIMEVE, the idea is to create synergies between metrologists, industrialists and operators of fibre networks across the continent,"* says Paul-Eric Pottie.

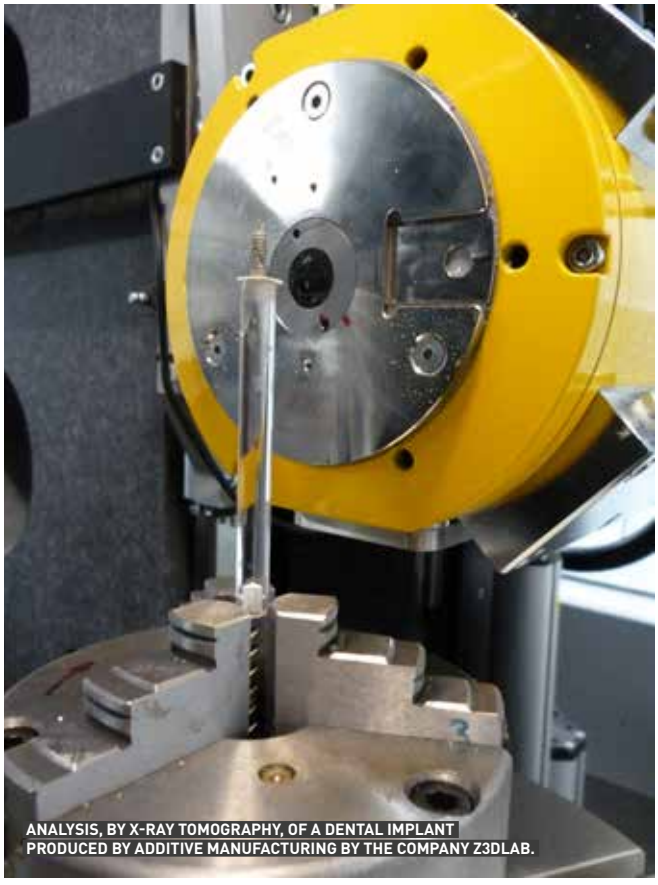
A key benefit is making it possible to disseminate, the time unit as broadly as possible, at maximum precision, to all potential users. *"There are numerous expectations, including comparisons between optical clocks for the redefinition of the second, tests of general relativity or geodesy and so on,"* says the metrologist. The coherent fibre link established between Paris and Lille is only the beginning! ■

LOW PRESSURES MEASURED "UNDER THE MICROSCOPE"

From electronics to pharmaceuticals and the nuclear sector, numerous industries require environments under controlled atmospheres. Hence the European project "pres2vac", which aims to improve the accuracy and traceability of pressure measurements in a range between 1 pascal and 10,000 pascals (the atmospheric pressure is around 1,000 hectopascals), to which the researchers of LNE-LCM have made their contribution.
For higher pressures, metrologists have measurement equipment directly connected to units of the International System. As a result, specialists in the laboratory take pressure measurements by balancing the force exerted by a gas on a piston-cylinder assembly with weights. *"But for 'low' pressures, the forces in play are too low, hence the use of a 'weigh cell,'"*, explains Pierre Otal, manager of the project for LNE-LCM. But due to difficulties in stabilising the pressure, the relative uncertainties are degraded.
To reduce them as far as possible, French scientists carried out an analytic and experimental study aiming to better control the sources of uncertainty in their equipment. At the same time, they produced a test bench playing the role of transfer standard, enabling comparisons between laboratories with different equivalent measuring equipment used by all partner laboratories throughout Europe. Lastly, they also took part in an inter-laboratory comparison campaign involving another type of standard based on "vacuum" measurements.
The key benefit is an improvement by a factor of nearly 10 in uncertainties at the level of the national references which, consequently, makes the entire metrological chain more reliable! ■

3D PRINTING: TARGETING METROLOGICAL PRECISION

The methods used in additive manufacturing, or 3D printing, are truly revolutionary. By allowing a part to be shaped by adding material layer by layer, they can make items of great complexity, with applications in aerospace and medicine, in particular.
To benefit from this, industrialists must nevertheless still learn to become proficient in all the subtleties. In particular, concerning the associated non-destructive testing methods, for checking the compliance of a part with its digital model.
With this in mind, LNE-LCM is participating in an ISO standards group, aiming to lay down best practices in the matter. Against this background, Anne-Françoise Baton was seconded to NIST (*National Institute of Standards and Technology*), in Gaithersburg in the USA, for the whole of 2018. Working in collaboration with researchers in the United States, her particular focus was on producing parts by additive synthesis containing various types of faults (layer and porosity faults and lack of melting). The objective is to test the appropriateness of various analysis techniques, including x-ray tomography, acoustic resonance and ultrasonic methods in highlighting these faults, which were, in this case, intentional.
As the scientist summarises, *"The results of these studies, as well as others, carried out by the various members of the group, will constitute the input data for a standardisation guide that will be published."* Eventually, the work of scientists at LNE-LCM also aims to attach all methods of non-destructive testing for additive manufacturing to a metrological chain that is traceable according to the International System. ■



ANALYSIS, BY X-RAY TOMOGRAPHY, OF A DENTAL IMPLANT PRODUCED BY ADDITIVE MANUFACTURING BY THE COMPANY Z3DLAB.

DIANNE SOFTWARE – CROPPING, IDENTIFICATION AND ANNOTATION FOR EVALUATION.

ARTIFICIAL INTELLIGENCE FOR WEED KILLING

May the best one win! This year marked the beginning of the ROSE Challenge, organised by LNE, Irstea, the French biodiversity agency (AFB) and the French national research agency (ANR). The objective is to evaluate the performance of automated solutions for weed killing proposed by four consortia, with the aim of reducing the use of phytopharmaceutical products.
In addition to its coordination role, LNE provides its expertise in the development of common standards for the evaluation of artificial intelligence (AI) systems. *"It is the first time that an AI challenge has taken place in the agricultural domain, in a test environment involving crops, i.e. in the living world,"* explains Virginie Barbosa, projects manager at LNE. *"The methodological issue therefore consists, for us, in finalising a controlled and reproducible evaluation protocol." And, the scientist adds, "The advantage of a challenge is less to determine a winner than to create collective competition via objective comparison in a common environment."*
Accordingly, the first few months of 2018 were devoted to establishing an evaluation plan. Following this, in June, the teams and organisers met to launch a "blank" or "dry-run" evaluation. Then, next May, the four robots should again prove themselves on controlled agricultural plots. They will then be ready for the "official" campaigns, the first of which will begin in October of this year. The results will be announced in 2021! ■

MOVING TOWARDS METROLOGY FOR ARTIFICIAL INTELLIGENCE

How can the performance or proper functioning of an artificial intelligence system be evaluated? Two ongoing projects, in which the LNE is taking part, aim to define standards for getting close to a metrology for AI (Artificial Intelligence), as recommended by Cédric Villani in his report, which aims to make LNE the competent authority in matters of AI metrology. Accordingly, Voxcrim, in collaboration with the technical and scientific centres of the French police and gendarmerie, aims to determine the criteria for evaluating voice comparison systems applied to forensics. The aim is to catch a suspect by comparing their voice recorded without their knowledge, for example during a telephone intercept and then when they are being questioned. *"It is about defining the conditions under which the comparison is carried out, such as optimal recording conditions to guarantee the reliability of the process and determining whether the result would be admissible in court,"* explains Agnès Delaborde, at LNE. The eventual objective is to define an accreditation standard for centres performing voice comparisons of this kind.
For its part, ALLIES aims to provide standards for evaluating the reliability over time of self-learning systems for translation or the identification of speakers. Certain metrics will therefore enable these intelligent systems to measure their own progress. Others will enable comparisons between different existing automatic learning algorithms and determine the most promising approaches. *"In 2018, we adapted the BEAT platform from our Swiss partner IDIAP, which enables the online evaluation of information processing systems, for ALLIES,"* adds Guillaume Avrin, at LNE. It will therefore be ready for the open evaluation campaign planned from this year! ■

FOOD: A RECYCLABLE "BARRIER" MATERIAL

Storing food requires packaging with a strong "barrier" effect against the gases in the environment, notably oxygen, which is a source of oxidation. Solutions involving multi-layer materials exist to do this. Due to their complex structure, however, they cause problems with recycling. As part of the NanoCoat project, researchers at the LNE[1] are offering a single-layer solution, which also provides a record barrier effect.

Specifically, the material is presented in the form of a 12-micrometre PET (Polyethylene Terephthalate) substrate with a coating, including one based on fish gelatine, nano-loaded with a vermiculite-type lamellar clay. As project manager Catherine Loriot explains, *"Incorporated in the coating, the nano-flakes of clay form a network between the mesh through which the gas slowly diffuses, providing a significant barrier effect."* Indeed, chemists showed that impermeability to helium was 100 to 1,000 times greater than that of a conventional barrier material such as PVCD (Polyvinylidene chloride). Likewise, impermeability to oxygen is increased by a factor of 350 compared to the case of non-coated PET, even though the sensitivity of the material to damp could still be improved.

Following these promising results, the specialists plan to continue their work in two directions. First, to bring their synthesis technique closer to industrial standards, and secondly, to analyse the details of the mechanical properties of their material and its behaviour when ageing. ■

[1] Project financed by the Actia and carried out in partnership with the Cnam (PIMM laboratory), the CTCPA, the University of Bourgogne (PAM laboratory) and the IPC in Clermont-Ferrand.

METROLOGICAL ATOMIC FORCE MICROSCOPE.

THE NANO-WORLD AT THE PRECISION OF THE... NANOMETRE

An innovative method for characterising the morphology of nanoparticles makes it possible to measure their size with unequalled precision, traceable in the International System of Units.

From paint to tyres, electronics, cosmetics, medicines and more – nano-materials are everywhere. Over the last 20 years, significant R&D effort has been put into techniques for synthesis and preparation. Nevertheless, difficulties remain for the fine characterisation of nanoparticles. Yet this shortcoming is weighing on the ability of industry to reproduce the properties of nano-materials observed in the laboratory, at the same time as it hinders their social acceptability. To tackle this, LNE-LCM is developing significant programmes in the metrology of nanoparticles. In 2018, it obtained remarkable results in the characterisation of their morphology.

Specifically, French metrologists proposed an innovative method to measure the size and evaluate the shape of nanoparticles. Their idea was to couple the two probes with the supplementary benefits of scanning electron microscopy and atomic force microscopy. As Nicolas Feltin explains, at the start of the project, *"The first can make bi-dimensional measures (typically length and width) of nanoparticles placed on a substrate. The second gives access to the third dimension by measuring their height."*

Once the concept had been established several developments were necessary so that this hybrid method could be implemented. The scientists therefore perfected a new substrate based on silicon and a method of depositing that was compatible with the two experimental techniques. In addition, working in collaboration with the company Pollen Metrology, they developed "co-location" software, that can make sure that a particle indexed on an electron microscopy photograph can also be unambiguously identified on another obtained by atomic force microscopy. Lastly, an algorithm for merging the measurement data from both probes, of different types, was created.

As a result, the laboratory specialists managed to take 3D measurements of model nanoparticles made of silica with a certainty of between 2 and 4 nanometres. As Nicolas Feltin explains, *"Currently, our method is the only one that has been the subject of an uncertainty budget."* In this regard, it is also the only one that can indicate whether or not a material comes within the "nano" category as defined in European regulations, meaning that at least 50% of the nanoparticles that compose it have a size less than 100 nanometres.

> *« Currently, our method is the only one that has been the subject of an uncertainty budget."*
>
> Nicolas Feltin, Scientific Manager of the LNE-NanoTech Institute.

Even better, 2018 saw the commissioning of the metrological atomic force microscope. Thanks to this primary resource, which is unique in France, it is now possible to guarantee the traceability of measurements made on nano-objects using the International System of Units. To do this, the researchers, in collaboration with the C2N laboratory of the CNRS, developed reference structures with patterns at the nano scale. Once the size of these has been measured using the metrological AFM, it is then possible to use these structures for calibrating other instruments. In this way, the metrologists of LNE-LCM ensured the traceability of their hybrid method. This firmly brings the nano-world into phase with the requirements of metrology. ■

REVISION OF THE INTERNATIONAL SYSTEM OF UNITS

Maguelonne Chambon,
Director of Scientific
and Technological Research

On 16 November, the member states of the BIPM adopted the revision of the International System of Units (SI), thus changing the definition of the kilogram, the ampere, the kelvin and the mole. Was this a historic vote?
Absolutely. It is the first time that four units had their definitions change simultaneously. Thus, in a system that is now highly coherent, all seven basic units are defined from fundamental physical constants. Moreover, through this revision, the BIPM abandoned the material artefact, a cylinder of platinum, which has defined the kilogram since 1889. A change of this magnitude has never occurred before!

As the Director of Scientific and Technological Research at the LNE, you were present at the vote. Could you describe what this event felt like?
It was both solemn, a little formal, and at the same time very friendly. It must not be forgotten that the General Conference on Weights and Measures, which takes place about every four years, is a diplomatic event that brings together 101 countries (59 member states and 42 associates) bound by the Metric Convention, the oldest international scientific treaty currently in force. This is not insignificant! Especially as, this year, about 400 people, diplomats and scientists, were present, twice as many as usual, including two winners of the Nobel Prize in Physics, Klaus von Klitzing and Bill Phillips. All of the resolutions adopted were quite obviously prepared long in advance, and are the fruit of at least a decade of scientific work; but at the moment of the vote, the euphoria and even a touch of agitation were palpable! It was very moving.

Especially as I imagine that the LNE has contributed significantly to this revolution?
Yes, the LNE, as well as the other French metrology laboratories in large organisations such as the CNAM or the Paris Observatory, directly took part in the redefinition of three of the four units included in the revision. We are amongst the three teams to have measured Planck's constant, *h* (from the so-called watt balance experiment), the constant on which the kilogram is now based, with the minimum of uncertainty. Concerning the measure of *k*, the Boltzmann constant, for the definition of the kelvin, our teams established a world record by taking the measurement with the lowest uncertainty ever achieved. Lastly, the LNE produced the first quantum standard of the ampere, which therefore expresses the new definition of the unit of current.
On the day of the vote, I said to my teams that we could therefore be proud of the work that was done. Not only at the LNE, but also in collaboration with all our colleagues throughout the world. Because, let us not forget that beyond individual contributions, metrology is a collective adventure, that of an entire community, which we all felt on 16 November.

> *"At the moment of the vote, the euphoria and even a touch of agitation were palpable! It was very moving."*

26TH GENERAL CONFERENCE ON WEIGHTS AND MEASURES IN VERSAILLES.

26TH GENERAL CONFERENCE ON WEIGHTS AND MEASURES IN VERSAILLES.

The revision of the SI was also a magnificent opportunity to communicate to the general public.

Indeed, in this regard, we are very happy with the success of the cycle of seven conferences, one per basic unit, which we organised at LNE, as well as the various events that we organised as part of the Science Festival. We were also an important contributor to the exhibition on the IS at the Musée des Arts et Métiers in Paris. This was also the venue for the evening event that followed the Conference, and we received very positive feedback on the exhibition, which I invite you to visit! Numerous delegations offered small organised activities or goodies for the occasion, including printed mugs, badges, games, films and so on... it was festive and very friendly.

Beyond the adoption of the revision to the SI, what will be the consequences?

First, the new definitions will not take effect until 20 May 2019, while various legal provisions are implemented. After that, it should all run smoothly, because everything has been done to ensure continuity between the old and the new systems. Even so, in laboratories and industries, various measurements will gradually be able to be done with greater precision. Also, greater precision probably means new applications, in the same way as the revision of the definition of the second, in 1983, enabled the development of positioning and navigation systems, such as GPS. What will emerge this time? Only the future will tell us! ■

CRYOGENIC CURRENT INDICATOR.

THE LNE RESEARCH PRIZE

10th LNE research prize: Wilfrid Poirier and Félicien Schopfer recognised for their work in electrical metrology.

This year, you received the LNE Research Prize. What does this award represent for you?
Félicien Schopfer: It is primarily an award for a team, as shown by the fact that we are both winners. And it is related to work which has admittedly enabled notable results over the last few years, but which has been consistent over two decades.
Wilfrid Poirier: It is the culmination of a process combining the will to push to the maximum the fundamental knowledge of the systems on which we are working, instrumental and metrological expertise and the desire to take innovative approaches.

You are specialists in the quantum Hall effect applied to electrical metrology. Can you explain what your work consists of?
Wilfrid Poirier: As electrical metrologists, our work consists of proposing, implementing and maintaining reference standards for electrical units. More specifically, Félicien and I are specialists in the quantum Hall effect through which it is possible to produce standards for resistance. Over the last few years, we also proposed the first quantum standard used for the new definition of the ampere, as adopted on 16 November 2018 during the General Conference on Weights and Measures.

What is the link between the quantum Hall effect and electrical metrology?
Félicien Schopfer: When we submit a conductor that is traversed by a current to a magnetic field perpendicular to this current, we observe a difference in electrical potential, or electrical voltage, between the edges of this conductor in the third direction. This is the classic Hall effect. In 1980, Klaus von Klitzing showed that at very low temperatures and under strong fields, a quantum version of this effect exists in certain materials: the relationship between the voltage and the current, which defines Hall resistance, varies by intermittent jumps when we increase the magnetic field, taking values that are sub-multiples of h/e^2, where h is Planck's constant and e is the elementary electrical charge. Because this phenomenon is robust, a priori independent of the material in which we observe it and directly related to fundamental constants, its implementation can define standards for resistance that themselves are highly reproducible.

From this, what was the main theme of your various research projects?
Wilfrid Poirier: Improving the implementation of electrical units, in particular the unit of resistance. Thus, my first notable achievement, to which Félicien has contributed since 2005, consisted of standards covering a range of resistance between 100 ohms and 1 mega ohm. To do this, we use an assembly involving putting into parallel and in series, via lithographic links, several hundred Hall bars made of gallium arsenide. This is currently the preferred material for producing the quantum Hall effect for metrological applications. But with a single component, we access a single value of Hall resistance which is really well quantified, hence the subsequent difficulty of guaranteeing the traceability of the unit of resistance over a wide range of values. Putting numerous components into series and in parallel resolves this problem.

Is it with this type of assembly that you then manage to perform record universality tests?
Félicien Schopfer: Indeed, by connecting four Hall bars, in order to perform the quantum equivalent of a Wheatstone bridge, we showed, in 2008, the reproducibility of the quantum Hall effect in different arrangements of gallium arsenide within the limit of a record relative uncertainty of 3×10^{-11}. More recently, we also extended these universality tests, showing that the quantum Hall effect implemented in gallium arsenide or graphene leads to the same level of quantification of Hall resistance, there again with a record uncertainty of 8.2×10^{-11}.

Did graphene also recently enable you to produce an innovative resistance standard?
Félicien Schopfer: Our group began a graphene activity in 2007, very shortly after its remarkable electronic transport properties were highlighted. In fact in this case, the Hall effect is less sensitive to temperature and the measurement current, and can be performed under a lower magnetic field than gallium arsenide. Thus, in 2015, after numerous developments, notably in collaboration with the laboratories of the CNRS, particularly C2N and CRHEA, we implemented a resistance standard in graphene, with an accuracy of 10^{-9} under highly favourable experimental conditions, demonstrating the potential of this material for simplified quantum electric metrology.

At the same time, did you also propose a system for implementing the new definition of the ampere, with unequalled accuracy?

Wilfrid Poirier: Exactly. With the help of our colleague Sophie Djordjevic, we implemented the first quantum circuit that can directly apply Ohm's law, linking current, voltage and resistance, on the quantum standards for voltage and resistance. To do this, we used the same solution as that implemented in our networks of Hall bars to overcome the interference resistance associated with electrical connections connecting the two standards, which caused errors on the reference quantum current. Thus, we produced a standard delivering a proportional current at the elementary charge, e, a constant on which the new definition of the ampere is based. Our standard is also accurate to 10^{-8}, compared with 10^{-6} with more conventional systems.

What are your projects for the future?

Félicien Schopfer: Concerning our graphene resistance standard, after having presented a demonstration in principle, we then need to stabilise it, with the objective of making this material the future reference in the field. We also plan to produce a version of our electrical current standard incorporating graphene, which will have the advantage of simplifying it. From a more general point of view, we are going to pursue the development of our electrical current standard, devoting a complete and independent experiment to it. The objective is to perpetuate it and gain a factor of 10 in its accuracy.

Wilfrid Poirier: We are also collaborating with the CEA, which wishes to take advantage of the quantum Hall effect to detect very low currents, possibly a single charge. In this context, we are providing support on the fine characterisation of the quantum Hall effect on their state-of-the-art graphene samples.

In the long term, we can envisage applications for this work in producing ultra-sensitive sensors. Also, this activity enables us to compare the benefits of the various technologies for the production and use of graphene for the quantum resistance standard, while incorporating know-how in terms of high-frequency rapid measurement. It is about breaking new ground for the future, according to a method that has been quite successful until now! ■

A LOOK BACK AT CPEM 2018

The CPEM the largest scientific and technological conference in the field of high-precision electromagnetic measurement. Took place from 9 to 13 July 2018. France had not hosted the event for more than 25 years. The conference was even more important insofar as it coincided with the key date in the history of the International System of Units: the adoption, in November 2018, by the CGPM, of the new definitions of the kilogram, the ampere, the mole and the kelvin.

Several sessions reviewed the forthcoming redefinitions of these measurement units and their practical use.

Opened by Thomas Grenon, the Managing Director of the LNE, this week of presentations brought together 529 participants from 47 different countries, and included presentations by two prestigious guests, the winners of the Nobel Prize in Physics, Serge Haroche and Klaus von Klitzing.

But as well as the revision of the IS, numerous other subjects were addressed during this week:

- Time-Frequency
- Quantum electric standards
- Optical and photonic metrology
- New sensors
- Current
- Electric voltage
- Direct current resistance
- Impedance
- Magnetism
- Power and energy
- Strong current and high-voltage radio frequency and microwave

The week also featured visits to laboratories, enabling participants to discover the LNE site at Trappes as well as that of the Paris Observatory, two laboratories that are heavily involved with the issues of electrical metrology. ■

THE FRENCH NATIONAL METROLOGY NETWORK

NATIONAL METROLOGY LABORATORIES

LNE-LCM/LNE-Cnam

The joint metrology laboratory brings together researchers from the LNE and the Conservatoire National des Arts et Métiers. The LNE-LCM/LNE-Cnam works in the fields of the metrology of lengths, optical radiation, temperature and thermal quantities, mass and related quantities (pressure, force, torque, acoustics, accelerometry and viscosity).

LNE

The LNE, which is in charge of areas such as electricity – magnetism, chemical metrology and mathematics and statistics, as well as the activities specific to the LCM.

LNE-LNHB/CEA

The Laboratoire National Henri Becquerel at the French Alternative Energies and Atomic Energy Commission, which is responsible for producing references in the field of ionising radiation, i.e., dosimetry and radioactivity.

LNE-SYRTE/OP

The Laboratoire des Systèmes de Référence Temps-Espace at the Paris Observatory, which is responsible for producing time and frequency standards.

LNE PARTNER LABORATORIES

LNE-CETIAT

The Centre Technique des Industries Aérauliques et Thermiques covers hygrometry, liquid-water flow metering and anemometry.

LNE-ENSAM

The Ecole Nationale Supérieure d'Arts et Métiers, Paris specialises in dynamic pressure.

LNE-IRSN

The Institut de Radioprotection et de Sûreté Nucléaire works in neutron dosimetry.

LNE-LADG

The Laboratoire Associé de Débitmétrie Gazeuse specialises in gaseous flow metering.

LNE-LTFB

The Laboratoire Temps Fréquences de Besançon is responsible for the transfer of time and frequency references, particularly for measurements of phase spectral density and stability of time and frequencies.

LNE-TRAPIL

The laboratory of the company Trapil is responsible for references in flow metering of liquid hydrocarbons.

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