



Liberté Égalité Fraternité



RESEARCH REPORT



or several years, we have been focused on our ambition of forging a powerful metrology sector in France with the ability to stay ahead of the innovation curve. This ambition is illustrated by our decision in 2021 to adopt our 2021-2025 Mid-Term Programme and reflected in the highly symbolic projects showcased in this report. In a fast changing world, the overriding aim is to break new ground and support our country in maintaining its competitive advantage, while moving the ecological transition agenda forward and preventing new health crises.

In terms of quantum technologies, the French metrology sector is reinforcing its position through various fundamental in maintaining its competitive advantage, while moving the ecological transition agenda forward and preventing new research projects, such as SEQUOIA for exploring the potential of graphene, and PhotOQuanT for developing a quantum We are also looking to help France carve a stronger foothold sensor to measure thermodynamic temperatures. Above all and preserve its influence in the European metrology arena. As and as part of the national strategy on quantum technologies, such, we can look back at the past year with a great deal of LNE is going to roll out a quantum metrology platform within satisfaction, since LNE has taken over chair duties for the the French National Metrology Network, which will mark the European Partnership on Metrology (EPM) Committee. The culmination of a revolution that was launched some 100 years EPM follows on from the EMPIR programme and will harness ago and will cement France's reputation as one of the leading the coordinated efforts within European metrology networks to nations for expertise in quantum technologies.





"WE ARE LOOKING TO HELP FRANCE CARVE A STRONGER FOOTHOLD AND PRESERVE ITS INFLUENCE IN THE EUROPEAN METROLOGY ARENA."

THOMAS GRENON CHIEF EXECUTIVE OFFICER

foster a self-sustaining integrated metrology system across the continent. We are also actively involved in a number of crucial topics within several of those networks, including Mathmet, Climate and Ocean Observation, Energy Gases, Traceability in Laboratory Medicine, Advanced Manufacturing and Quantum Technologies.

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EXECUTIVE SNAPSHOT

The exponential rise in new technologies and their ever growing inroads into everyday life, and the continued focus on sustainable development and public health policies at both a national and European level are driving the never-ending need for ever more reliable and indisputable measurements to make informed decisions at every level. Keeping pollution under control, leading the energy transition to drastically curb greenhouse gas emissions, tracking climate trends, health and biology. quantum technologies, the industry of the future, modelling and digitisation, artificial intelligence, food contact and new materials are just some of the research areas pioneered at LNE.

LNE is the driving force behind the French metrology system and coordinates the research activities of the French National Metrology Network (RNMF), which comprises 10 metrology laboratories. These laboratories have been officially accredited by European and international bodies for leading research in one or more specific fields. From implementing the International System of Units (SI) to developing test benches for calibrating against national standards, these laboratories are responsible for producing and improving the national metrological standards, taking part in the international benchmarking of national standards, and disseminating those standards to users.



≤ In August 2021, LNE and Paris-Saclay University, including AgroParisTech, CentraleSupélec, École Normale Supérieure Paris-Saclay and the Institut d'Optique Graduate School, signed a framework agreement to strengthen their collaborative efforts, especially in the fields of research, development and training. In terms of development and research, ties will be reinforced by sharing resources and pooling experimental facilities,

such as creating jointly supervised laboratories and engaging all staff in the process.

As for teaching, LNE will be involved in training programmes and supervising students (especially doctoral students) at Paris-Saclay University and the associated institutions.

RESEARCH AT LNE IN 2021

25 % of LNE's total budget

132 PhD graduates and engineers

> 24 PhD students

110 publications in peer-reviewed journals

> A portfolio of **18** patents

FRENCH NATIONAL METROLOGY NETWORK (RNMF)

LNE-LCM

The joint metrology laboratory (LCM) between LNE and the Conservatoire National des Arts et Métiers (CNAM) pursues metrology research in the fields of length and dimensional quantities, optical radiation, temperature and thermal quantities, mass and related quantities pressure, force, torque, acoustics, accelerometry and viscosity).

LNE

LNE is responsible for leading metrology activities in a number of fields, including electricity and magnetism, chemistry and biology, and mathematical and statistical tools, in addition to the activities within the LCM laboratory.

LNE-LNHB

The Laboratoire National Henri Becquerel of the CEA (French Alternative Energies and Atomic Energy Commission) in Saclay develops measurement standards in the field of ionising radiation, dosimetry and radioactivity measurements.

LNE-SYRTE

The Laboratoire des Systèmes de Référence Temps-Espace at the Observatoire de Paris is responsible for producing national time-frequency and gravimetric standards.

LNE-CETIAT

Aérauliques et Thermiques in Villeurbanne develops national standards in the fields of anemometry.

LNE-ENSAM

the École Nationale Supérieure d'Arts et Métiers de Paris is responsible for dynamic pressure standards.

LNE-IRSN

laboratory (LMDN) of the Institut de Radioprotection et de Sûreté Nucléaire in Cadarache produces national standards for neutron dosimetry.

LNE-LADG

The Laboratoire Associé de Débitmétrie Gazeuse within Cesame-Exadebit in Poitiers is responsible for developing national standards for high-pressure gas flow measurements.

RESEARCH AT THE RNMF IN 2021

Over **290** scientists

157 research projects including 74_{JRPs} (EURAMET Joint Research Projects)

160 publications in peer-reviewed journals

64 theses under preparation including **11** theses defended in 2021

The Centre Technique des Industries hygrometry, liquid-water flow metering and

The dynamic metrology laboratory within

The neutron metrology and dosimetry

LNE-LTFB

The Laboratoire Temps-Fréquence de Besancon within the University Burgundy-Franche-Comté is responsible for disseminating national time and frequency standards, especially for time interval measurements, frequency calibrations and phase noise in the field of RF.

LNE-TRAPIL

The Trapil laboratory in Genevilliers is responsible for producing national references for liquid hydrocarbon flow metering.

EPM PROGRAMME - YEAR 1: EUROPEAN METROLOGY ON THE THRESHOLD OF A NEW ERA -

THE NEW EUROPEAN PARTNERSHIP ON METROLOGY WAS LAUNCHED IN 2021. THIS LAST PROGRAMME BANKROLLED BY THE EUROPEAN COMMISSION LAYS THE FOUNDATIONS FOR A SELF-SUSTAINING INTEGRATED METROLOGY SYSTEM ACROSS THE CONTINENT.



European Partnership on Metrology (EPM) was officially ratified. As part of the European Commission's ninth Framework Programme for Research and Innovation (called Horizon Europe), the EPM follows in the wake of the EMPIR programme, whose final call for proposals was held in 2020. The various programmes spearheaded by the EC since 2008 have been instrumental in forging strong cooperative ties within the European metrology system. The EPM, the last programme of its kind, will usher in a new era of even greater maturity for the EU metrology system by fostering closer cooperation on metrology across the continent. The aim is to use the science

n 1 December 2021, the new | for addressing the major economic, | every programme and will underpin the environmental and social challenges of our time.

> Historically speaking, national metrology institutes have been responsible for developing primary measurement standards and transferring their performance to users. As explained by Maguelonne Chambon, LNE Director of Scientific and Technological Research and Chair of the EPM Committee (see interview): "Until about 15 years ago, metrology projects tended to be based on a given unit or quantity." However, ever since the "Metre Convention" was signed in 1875, there can be no doubt that metrology's development is intimately related to the growth of human activities in every field. "This correlation

EPM's philosophy, which is aiming to take a closer look at users' actual needs and draw on that intelligence to deliver a response to the social issues that often cut across several different sectors," she continues.

The bottom line is that the EPM will be endowed with a 600 M€ budget financed equally by the European Commission and the participating countries. "There are still some loose ends to tie up, but there's every likelihood that over 25 countries will ultimately take part," says Maguelonne Chambon. In addition, the EPM's ambition of fulfilling users' actual needs is clearly reflected in the decision to set up an advisory steering group featuring around 15 of measurement as a driving force | has become increasingly clear with | highly qualified people from outside the

be to advise the EPM Committee on the strategic directions for its calls for projects.

Basically, they will need to provide answers in a wide range of areas. On a fundamental level and in the wake of the revised definitions for the SI base units back in 2018, the next step will involve supporting changes within the next 10 years to the definition of what is the core unit of time, i.e. the second. As for the other topics, the EPM is gearing up to tackle all the economic and social challenges on Europe's doorstep. To help companies sharpen their competitive advantage, special focus will be given to guantum technologies, nanotechnologies, digital technologies, artificial intelligence and healthcare. This topic will also be addressed in the second major section of the EPM, which specifically targets the ecological transition issue and more generally the environment and energy.

The EPM had barely been launched than a first call for tenders was organised with a 25 M€ budget. Thirteen projects have gualified for funding. Nine of them relate to the energy transition and environmental monitoring, while the other four concern standardisation. Closer ties between the metrology and standardisation sectors are also an integral part of the EPM's roadmap. "For this first section on standardisation, we are funding a varied array of projects, from measuring luminescence levels on road surfaces to defining methods for certifying biomethane conformity, as well as developing wireless standards," explains Maguelonne Chambon.

metrology sector. The group's role will | The budget will rise to 43 M€ for the EPM's second year, where efforts will zero in on digital technologies, healthcare (diagnostics, therapy, quality of life, etc.) and integrated metrology: "In the long term, the aim is also to coordinate the process of developing Europe-wide metrological infrastructures, such as new calibration facilities and benches," says Maguelonne Chambon. This idea of coordinating metrology across Europe is one of the top priorities in the EPM, which will have to step up its interaction with users and customers in the future. As such, by the time that the EPM draws to a close in 2030, the entities leading and benefitting from the partnership must have set up the necessary structures for sustaining the synergies created by the various programmes that have come before. This is the very rationale that prompted EURAMET to launch the European Metrology Networks (EMNs) in 2019. The EMNs are responsible for analysing needs and implementing coordinated strategies between the different metrology stakeholders and users in terms of research, infrastructures, knowledge transfers and services. "Due to the very way in which they are structured, the EPM and the projects funded under the partnership, as well as the topics pursued, will work hand-inhand with the EMNs in paving the way for a self-sustaining integrated European metrology system". The new European Partnership on Metrology has barely been launched than it is already shaping the metrology system of tomorrow's

"THE BOTTOM LINE IS THAT THE EPM WILL BE ENDOWED WITH A 600 M€ **BUDGET FINANCED EQUALLY BY** THE EUROPEAN COMMISSION AND THE PARTICIPATING COUNTRIES."

world





Three questions for...

MAGUELONNE CHAMBON

LNE DIRECTOR OF SCIENTIFIC AND TECHNOLOGICAL RESEARCH AND CHAIR OF THE EPM COMMITTEE.

What does your appointment as Chair of the EPM Committee represent?

M.C.: This appointment bears testament to the many years of hard work and commitment that the researchers at LNE and the laboratories within the French National Metrology Network have demonstrated in the different European metrology programmes.

It's a major responsibility...

M.C.: Chairing the Committee will require a tremendous investment. But with more than 10 years' experience in taking part in the committees of the successive wave of European metrology programmes, and with a helping hand from LNE, I certainly won't be jumping in at the deep end! This appointment gives us a strategic vantage point, since the EPM Committee is responsible for managing the topics that are chosen for the calls for projects and then keeping a close eye on those topics over time

Does your appointment strengthen the French metrology system's influence in Europe?

M.C.: France is the third largest contributor to the metrology programmes funded by the European Commission, and our teams have shown that they are more than capable of leading key projects and tackling new topics. My appointment illustrates the excellence and vital role played by the French metrology system within Europe.

REALISATION OF THE FARAD: LNE'S NEW "LAMPARD" IS READY FOR ACTION!

LNE'S THOMPSON-LAMPARD CALCULABLE CAPACITANCE STANDARD WILL ALLOW THE FARAD TO BE REALISED WITH A RELATIVE UNCERTAINTY OF CLOSE TO 10⁻⁸. THE FIRST MEASUREMENTS CARRIED OUT LAST YEAR MARK THE END OF A SCIENTIFIC AND TECHNICAL ADVENTURE SPANNING MORE THAN 15 YEARS.



his is it, the LNE has finally got its "Lampard"! To be more precise, LNE now has a Thompson-Lampard calculable capacitance standard that can be used to realise the farad with a relative uncertainty in the region of 10⁻⁸. In doing so and following more than 15 years' development work, French metrologists have joined the elite circle of teams that are capable of achieving such a feat. Better still, LNE has become one of the few places in the world featuring the two solutions recommended by the CIPM for realising the farad (the other solution is based on traceability to the quantum Hall effect), thereby carving its reputation as a world leader in electrical metrology.

The new standard is based on an electrostatic theorem described in 1956 by Australian physicists A. Thompson and D. Lampard. The theorem states that a system of four perfectly parallel cylindrical electrodes of infinite length placed in a vacuum creates a set of crosscapacitors whose linear capacitance depends only on the permittivity of the vacuum. Determining the capacitance involves measuring the length, which thereby links the farad to the metre.

In practice, a calculable standard takes the form of a system of electrodes as defined by Thompson and Lampard, from which metrologists can calibrate a capacitor whose electrical capacitance needs to be determined. Specifically, the assembly is incorporated into an electrical circuit that can be used to compare a voltage ratio delivered by a standard transformer against a ratio involving the capacitances of the Lampard capacitor and the capacitor

"LNE HAS AN EXTENSIVE TRACK RECORD IN MAINTAINING ELECTRICAL UNITS AND HAS DEVELOPED SEVERAL VERSIONS OF CALCULABLE STANDARDS SINCE THE 1960s."

to be characterised. By creating | uncertainty of approximately 5×10⁻⁸ for two configurations of the Lampard capacitor - by moving a mobile electrode placed in the centre of the cavity formed by the other electrodes - and thereby producing two values of its capacitance, the unknown capacitance can be determined by measuring the displacement of the mobile electrode using laser interferometry. QED.

LNE has an extensive track record in maintaining electrical units and has developed several versions of calculable capacitance standards since the 1960s. Over the following decade, LNE developed a five-electrode standard. representing an original configuration that could be used to multiply the number of cross-capacitors passing through the inter-electrode cavity. In the early 2000s (the "Lampard" was operational at the

THE LAMPARD CALCULABLE STANDARD: AN ALL-OUT EXPERIMENT IN METROLOGY

The development of a Thompson-Lampard calculable standard is aimed at realising the electrical unit of capacitance - the farad. But to attain that goal, LNE's specialists had to leverage a wide array of skills encompassing every discipline. Therefore, to overcome the challenge of achieving a relative uncertainty of 10⁻⁸, they had to contend with extraordinary mechanical constraints in terms of polishing and adjusting the electrodes in their capacitor. In addition, they had to produce the world's most advanced electrical measuring instruments, especially a standard transformer, as well as masterminding an optical system to provide in situ traceability to the metre for measuring the displacement of the mobile electrode, dealing with the constraints associated with vacuum technologies, and developing software to automate the experiment while making sure that the devices (some of which are up to 40 years ago) were capable of communicating with each other, since the calculable standard is a long-term endeavour!

the capacitance measurement. But, as explained by Olivier Thévenot, who is in charge of developing the new standard: "We were experts in using that version and we knew we couldn't do any better with that version". But that wasn't good enough, bearing in mind that plans to revise the SI system were already in the pipeline.

The decision to revise the SI was ultimately given the green light in 2018 during the CGPM conference. Since then, the seven SI base units have been defined according to physical constants whose exact numerical values have previously been fixed.

As part of the work involved in preparing for the redefinition of the SI base units, LNE metrologists considered measuring time), the laboratory achieved a relative | the Von Klitzing constant in relation to

two constants in quantum mechanics, namely the elementary charge and the Planck constant, with a target uncertainty of 1×10⁻⁸. This measurement can be made from a calculable capacitance standard, provided that it realises the farad with the same relative uncertainty. "In 2005, the governing bodies of the French metrology system decided to embark on the adventure of building a new Lampard at LNE," remembers Olivier Thévenot.

Adventure is definitely the right word, or even an epic. Reducing the uncertainty by a factor of five represented an unparalleled and multidisciplinary challenge for LNE's metrologists (see box). In many respects, they needed to start from scratch, and the main component quickly proved to

be mechanical-related. Calculations actually showed that the target value could only be achieved if the geometry of the cylindrical electrodes was better than 100 nm. Similarly, the metrologists had to ensure that the electrodes would be parallel and positioned with the same level of accuracy, as well as making sure that their position would be maintained over time once the instrument had been adjusted. Those were just the mechanical aspects. Implementing the calculable standard also involved developing electrical metrology systems and optical instrumentation for measuring distances, all of which must function in a vacuum enclosure. "When we started the project, we had a good idea of the challenges standing in the way," says Olivier Thévenot, "and we had to change our game plan several times

when we came across problems that we hadn't necessarily anticipated.'

LNE | RESEARCH REPO

After more than 15 years' intense work (see timeline), researchers and technicians have masterminded two stainless steel polishing techniques and also pioneered an innovative instrument for measuring capacitance that allows the geometry of the electrodes to be characterised with the required precision. When it comes to adjusting the parallelism, they came up with a solution for independently moving both ends of each electrode with an uncertainty of a few tens of nanometres. As explained by Kamel Dougdag, Mechanical Projects Engineer at LNE: "This design keeps the electrodes parallel within the cavity created by the electrodes, even if they feature a slight conical defect." In addition, there is an embedded capacitive



FIVE KEY DATES IN THE CALCULABLE STANDARD

2005	2010	2014	2018
Decision to develop a new Thompson- Lampard standard at LNE	Start of production for the stainless steel electrodes	First assembly of the standard with dummy electrodes	Manufactur of the five electrodes the expecte specificatio

measurement system to check to the metre, they created a laser alignment and a clamping system that does not change the settings once they have been dialled in! Finally, to ensure the metrological structure's stability over time, it has been mechanically separated from the supporting structure, thereby isolating the standard from any external disturbances while measurements are taking place. As an extra touch, instrumentation researcher Almazbek Imanaliev, who joined the team in 2019, has set up a measurement method to check for correct alignment of the assembly from the inside, even when the system is operating in a vacuum.

Furthermore, in terms of the measurement instrumentation, the metrologists had to completely overhaul the electrical part of the infrastructure. In particular, they have designed a new standard transformer for delivering the voltage ratio required to relate the distance measurement to the value of the standard capacitance. "We raised the quality of all the coil windings, resized the assembly and added electromagnetic shields, all of which helped achieve a relative uncertainty of 10⁻⁹ for the transformer's contribution to the overall uncertainty level," advises Olivier Thévenot. Lastly, to ensure that the distance measurement is traceable Last year, the calculable standard was placed in a vacuum for the first time. "It was a tremendous moment to check that the capacitor's geometric settings, which had previously been made at atmospheric pressure, stayed the same under vacuum," says Olivier Thévenot. Almazbek Imanaliev sums up: "We now know that all the mechanical issues have been resolved. So we're ready for the first capacitance measurements involving the entire associated measurement system."

Once the Lampard is back in a vacuum, LNE will have a new and more direct way of realising the farad than relating the unit of electrical capacitance to the quantum Hall effect. The level of uncertainty achieved will establish the French metrology institute's place among the Australian, Chinese and American national metrology institutes, which are currently the only NMIs capable of realising the farad with a relative uncertainty of a few 10-8, using a Lampard-type standard. This will guarantee LNE's lasting place among the world leaders in electrical metrology.

E DEVELOPMENT OF THE THOMPSON-LAMPARD CALCULABLE STANDARD AT LNE IN FIGURES:

1×10⁻⁸: relative uncertainty in the practical application of the farad according to the SI definition.

100 nm: maximum size for any electrode straightness defects, achieved through mechanical polishing techniques - a tremendous achievement!

100 nm: level of precision for positioning the electrodes.

10⁻⁹: uncertainty for the electrical voltage ratio supplied by the standard transformer.

2021

ing to ed ons First operation under vacuum conditions and capacitance measurements

interferometer whose frequency is controlled by an iodine atomic line.



Three guestions for...

OLIVIER THÉVENOT DEVELOPMENT PROJECT LEADER FOR THE CALCULABLE CAPACITY STANDARD.

When you take a look back at the project, what comes to mind?

0.T.: What comes to mind is the sheer amount of perseverance that it took to see the project through to the end. Obviously we had our ups and downs. There were moments when our beliefs in what we were doing were unshakeable, but there were also times when we were plaqued by doubts, and we had to be incredibly single-minded to keep moving forward. In this respect, I'd like to express all my gratitude to the "Electricity-Magnetism" Scientific Committee and the other governing bodies within the French National Metrology Network (RNMF), who were always ready to encourage us and help us keep our eye on the ball when the going got tough.

What were the standout moments during the process?

0.T.: Producing the first electrode was clearly the most crucial step. When we received the stainless steel bar, it had defects of about 3 µm. Reaching 300 nm was fairly quick, but then it took us nearly a year to get that figure down to 100 nm. It may have been a huge hurdle, but it was a missioncritical milestone because we showed that we were capable of pulling the project off. Another highlight was completely assembling the standard. even though the final electrodes had not been finished. That's when we proved that we could position the electrodes to reach the required degree of uncertainty.

What are you going to do now?

0.T.: Assembling the standard and creating the vacuum conditions mark the end of an adventure. But now we're about to start another adventure as we begin taking the electrical measurements, and I'm sure there are plenty more surprises in store!

LNE RESEARCH **AWARD 2021** CHRISTOPHE BOBIN, JÉRÔME BOBIN AND ANNE DE VISMES OTT AWARDED FOR THEIR WORK IN IDENTIFYING AND QUANTIFYING RADIONUCLIDES.

You have received LNE's **Research Award for** developing a unique metrological approach for the spectral unmixing and identification of radionuclides. Can you tell us what the approach involves and how the project came about?

Christophe Bobin: As part of the ANR* NANTISTA project on developing portable systems for automatically detecting radionuclides back in 2015, I realised that there was a need for devices that non-experts could use to carry out reliable measurements, i.e. sufficiently robust, despite the low statistics, lowresolution detectors and limited on-board analytical capabilities. Literature showed that there was a lot of work in this area, but with little metrological content.

Anne de Vismes Ott: My work as a researcher at IRSN involves using gamma spectrometry to analyse samples and measure radioactivity levels in the environment. As such, the



are often extremely weak, and the challenge is all about reducing the level of background noise and lowering the decision thresholds and detection limits. However, it soon became apparent that the techniques we were using for spectral mixtures had reached their limitations and that we had to direct our attention towards the actual spectrum.

Jérôme Bobin: I'm actually something of a stranger to metrology and radioactivity measurements. I'm a researcher at CEA, and I specialise in signal processing at the IRFU.

Basically, I develop mathematical methods for data science and machine learning, which are mostly applied to astrophysics. As luck would have it. I met Anne through Christophe who was also working with IRSN as part of the NANTISTA project. As we were talking, we realised that it might be a good idea to work together in developing new algorithms for spectral unmixing applied to gamma spectroscopy. In practice, our collaboration was embodied in a doctoral thesis by Jiaxin Xu between 2017 and 2020, where I was the director, Anne was the supervisor and Christophe was a member of the oversight committee. That collaboration was extended through Rémi André's post-doctoral research.

What approach did you use?

Anne de Vismes Ott: Basically, the conventional approach to analysing spectra, which feature a mix of emissions from several radionuclides, involves identifying the characteristic peaks of a given element. In our case, however, the counting statistics for the spectra are so low that sometimes the characteristic peaks cannot be identified with the naked eye.

signals that we're looking to measure | Christophe Bobin: Based on that observation, we tried to take advantage of all the available information, including the data that were basically hidden beneath the intense level of experimental background noise, with the aim of delivering a quantitative analysis with a specific degree of uncertainty.

LNE | RESEARCH REPO

Jérôme Bobin: To achieve that aim, the important thing is to first model the data as effectively as possible, especially the associated statistical noise. It's a bit



technical, but the general idea is to model the random part of the data with Gaussian noise. This practical approximation provides sound results if good statistics are available.

However, in our particular case, this approximate method falls short. Therefore, we had to consider the true nature of the experimental noise, which happens to be Poisson noise and is harder to model.

Once the data had been properly modelled, we decided to go for an "inverse problem" strategy. This process



consists in building a mathematical estimator that creates a link between the data and the model's parameters. The parameters represent the activities of the different radionuclides in the spectra. The advantage with this approach is that it can also be used to identify the radioelements that contribute to the spectrum and their respective activities. Compared to more conventional approaches, it limits the false detection rate and allows the results to be associated with a level of uncertainty. Finally, we can apply it to different algorithms depending on the hardware that it has been implemented on.

What tangible results have you obtained with this new approach?

Anne de Vismes Ott: We worked on mixture spectra that had been obtained through numerical simulations, as well as on spectra obtained through measurements on aerosol samples taken via environmental monitoring activities. These spectra contain the emission signals of around 10 radioelements, and we tested our ability to extract the signature of caesium-137, whose activity in air is typically about 0.1 µBq per cubic metre of air, which represents a few millibecquerels to be measured in the sample. With a conventional analytical method, it takes about a week for the radon to decay to a level where caesium can be detected in an emission spectrum. Our approach has reduced this time to about four days. In addition, we've increased the rate of significant measurements from 80 % to 100 %.

measurements, we've demonstrated that our algorithms are capable of providing reliable results from measurements lasting only a few seconds. This could potentially be of great interest to manufacturers whose measuring devices need to give users the possibility of making guick decisions, such as border control or environmental monitoring agents. To be more precise, the strength of our algorithms lies in their ability to deal with the problem of false positives, and unlike conventional approaches, their rate is independent of the type of spectral mixture and the quality of the statistics.

How are you going to follow up on these developments?

Christophe Bobin: I'm working with a manufacturer to incorporate our algorithms into their handheld in situ detection devices. After tackling the problem of statistical variability in the spectra, we need to address the so-called issue of field variability, which is especially linked to the interaction of photons in the environment.

Anne de Vismes Ott: Since October 2020, I've been supervising Paul Malfrait's thesis, under Jérôme's supervision. His work is focusing on the simultaneous processing of multiple spectra to meet inline analysis needs. We'll also take a closer look at variability in the spectral signatures to be analysed according to



Christophe Bobin: In case of in situ | the type of detectors, the type of samples, and variability in the background noise and their influence on processing performance through spectral unmixing.

What does the LNE award mean to you?

Christophe Bobin: As a metrologist, this award represents an important endorsement of my work, my group's work and my laboratory (LNE-LNHB).

Anne de Vismes Ott: I'm a metrologist, but our metrological requirements at IRSN are obviously lower than LNE's requirements. So I'm very proud to see our work recognised by the metrology community. It proves that we're heading in the right direction with what is a highly interdisciplinary project!

Jérôme Bobin: This award pays tribute to three years' work on a project that clearly breaks new ground. There's often a tendency to think that data processing experts already have all the turnkey solutions. That's a misconception. In this case, we've demonstrated the value of working over the long term to design tools addressing a specific problem.

* The French National Research Agency (ANR) is a public administrative institution under the authority of the French Ministry of Higher Education, Research and Innovation, The agency funds project-based research carried out by public operators cooperating with each other or with private companies.

Since its inception in 2009, the LNE Research Award has paid tribute to researchers in a wide range of fields and areas, including nanotechnology, healthcare, the environment, telecommunications, energy, transport, information technology and materials. Over a period of more than 10 years, LNE has rewarded more than 15 researchers for their ability to combine science and pragmatism in leading research work with a strong impact on science, industry or society.

LNE-NANOTECH INSTITUTE: METROLOGY EXPERTS DRIVING THE NANOTECHNOLOGY REVOLUTION

THE EMERGENCE OF NANOTECHNOLOGY REPRESENTS A MAJOR TECHNOLOGICAL BREAKTHROUGH. TO SUPPORT NANOTECH'S DEVELOPMENT, THE LNE-NANOTECH INSTITUTE DELIVERS AN END-TO-END RANGE OF SOLUTIONS TO ALL PROFESSIONALS INVOLVED IN NANOSCALE MEASUREMENTS.



nanotechnology is gaining traction as an integral part of today's connected society and one of the keys for moving the ecological and energy transition soaring development, LNE created the LNE-Nanotech Institute back in 2017. This delocalised centre without walls

laboratory's nanotech infrastructures and expertise. It is development, whether performance assessments to support innovation, safety or regulations.

In the 2000s, LNE began pioneering reference instruments to bring metrological precision into the world of nanotechnology. As Institute Director Georges Favre points out: "One of the things that make nano-objects and nano-components so special is that they can only be characterised by using various complementary techniques and methods at the same time". This requirement has spawned a number of platforms at LNE over the years, such as CARMEN (physico-chemical properties), MONA (aerosol properties) and NAEL (electrical properties), which are supported by the various tools available in LNE's different teams. Therefore, researchers can establish a nano-object's profile, irrespective of its form (powder, dispersion, aerosol, etc.) and the complexity of the medium (manufactured product, biological medium, environmental matrix, soot, and so on). "When it came to meeting the complex needs of academia as well as economic and societal stakeholders, we naturally came up with the idea of creating a structure to share and coordinate LNE's solutions for characterising nanomaterials," adds Georges Favre. "That's what the LNE-Nanotech Institute is all about."

Materials, energy, medicine... These solutions naturally address the fundamental metrology problems associated with nanotechnology. By harnessing the laboratory's different resources beyond the realms of metrology's disciplines and/or methodologies, LNE-Nanotech is also capable of covering the entire chain, ranging from pure forward. To support nanotech's metrology through to standardisation, as well as disseminating standards and methods. "Whether it's for a manufacturer, a start-up, an institutional stakeholder or academia, LNE-Nanotech isn't just capable of controlling raw materials and serves as a hotspot for all the developing processes, but also assessing performance and risks, and lending support with defining standards, which are capable of addressing all the issues raised by nanotech's essential for building and implementing the regulatory framework," advises Georges Favre.



LNE-Nanotech is also playing a leading role in developing a community of professionals involved in nanoscale measurements. That explains why NanoMesureFrance was set up. This innovation hub is co-financed by the State and the Ile-de-France authorities, with LNE providing scientific oversight. "The aim is to create a platform where

manufacturers, start-ups, academic and private laboratories, instrument manufacturers and service providers can discuss their needs and skills in terms of characterising and assessing performance/exposure, while creating synergies to tackle all the challenges inherent in the soaring growth of nanomaterials," explains François-Xavier Ouf, R&D Coordinator of NanoMesureFrance at LNE. He adds: "NanoMesureFrance is responsible for identifying and prioritising the needs, while it's down to the LNE-Nanotech Institute to deliver tangible answers or solutions with its network of partners." These efforts should ultimately carve LNE's reputation as a leading force in the nanotechnology revolution.

ALEXANDRA DELVALLÉE

The LNE-Nanotech Institute is spearheading efforts to set up a common set of terms, definitions, references and best practices between teams from different backgrounds. This is essential for



nano-objects, which can only be characterised by using a combination of complementary techniques. A prime example is the Graal project, which is investigating the multidisciplinary metrological characterisation of graphene-based materials and products. The same samples are characterised using different local probe microscopy techniques. This raises a number of major constraints, especially when preparing samples, but they are easier to resolve when working within the same institute."

NOLWENN FLEURENCE

At the nanoscale, some of a material's properties may be different to those at the macroscopic scale. For example, they may depend on the dominant form of heat transfer within the material, which is influenced by the

structure and size of the material. Therefore, it often happens that the different properties need to be defined together to complete the process. That is why the LNE-Nanotech Institute is so beneficial. For example, it has become second nature for the different teams to offer global solutions when working on their research projects. A key example is the European NanoWires project, which is looking to develop techniques for characterising nanowire-based energy harvesters. The project's structural material characterisation, thermal metrology and electrical metrology teams all joined forces in responding to the call for projects. The Institute gives our partners a clearer insight into the complementary skills that we can tap into."

NICOLAS FELTIN

LNE-Nanotech encourages the idea of cross-functional work, not just between LNE's different departments, but also between the metrology and testing teams. This approach helps us get to know our colleagues



better and makes it easier to work together. Such close-knit ties are essential when working on the nanoscale, since processes are more complex. At the same time, the problems that exist at this scale really call for a joint effort from people specialising in different fields. As such, we can tackle a more complex range of issues and deliver an accurate response to our partners' needs more easily than if everyone were only focused on their own work. We've had chance to support several companies over the last few years by combining our skills in atomic force microscopy, scanning electron microscopy, chemical analysis and innovative aerosol techniques to identify the most relevant approach for fulfilling our partners' needs. We are the only national metrology institute that can offer end-to-end solutions when it comes to nanomaterials."

FRANÇOIS PIQUEMAL

The LNE-Nanotech Institute brings a formal edge to the contacts and interactions that already existed between the teams at LNE. It acts as a driving force when bidding on projects that require a cross-

functional response, especially in case of joint projects. LNE-Nanotech brings a certain amount of spontaneity when working together, even as early as identifying and searching for contracts or collaborative opportunities. For example, Georges Favre was contacted last year by a number of research centres in Grenoble and Paris for his input on a range of matters combining dimensional and electrical issues. It was only natural for him to tell me about it and for us to meet our contacts together. The Institute is also highly active in the standardisation and pre-standardisation scene. It gives us a positive incentive to ensure that our solutions incorporate this important aspect for both economic stakeholders and citizens.

FÉLICIEN SCHOPFER

For a material that boasts as many interesting physical and chemical properties as graphene, our partners need LNE-Nanotech's ability to coordinate resources. For example, we've been working

alongside Italian start-up Graphene-XT for several years. First of all, we carried out the structural characterisation of graphene flakes in the liquid phase, followed by thermal characterisation of graphene thin films. Now we're involved in the thermomechanical and chemical characterisation of lubricants. We should be able to draw on that experience in the future to lend our support to French start-ups operating in the same sector, such as Carbon Waters and BlackLeaf. Last year, public authorities and certain manufacturers also called on our expertise in aerosol metrology and microscopy to characterise the graphene nanoparticle content of FFP2 masks. The LNE-Nanotech Institute is also establishing a one-stop shop for fundamental metrology, characterisation, testing and the corresponding measurement services for customers, while addressing standardisation."

"IN THE 2000S, LNE BEGAN **PIONEERING REFERENCE INSTRUMENTS TO BRING** METROLOGICAL PRECISION INTO THE WORLD OF NANOTECHNOLOGY."







INDUSTRIAL COMPETITIVENESS

WITH ULTRA-HIGH-TEMPERATURE MEASUREMENTS, ARTIFICIAL INTELLIGENCE AND NON-DESTRUCTIVE TESTING, LNE AND THE RNMF ARE HELPING BUSINESSES SHARPEN THEIR COMPETITIVE EDGE BY GIVING THEM THE TOOLS TO CHARACTERISE AND EVALUATE INNOVATIVE **TECHNOLOGIES AND NEW INDUSTRIAL PROCESSES.**



MEASUREMENT OF THERMOPHYSICAL PROPERTIES: TRACEABILITY TO THE SI AT **ULTRA-HIGH TEMPERATURES**

AS PART OF THE EUROPEAN HI-TRACE PROJECT, LNE HAS IMPROVED THE PERFORMANCE OF ITS STANDARD MACHINES FOR MEASURING THERMAL DIFFUSIVITY AND SPECIFIC HEAT CAPACITY. THE MACHINES ARE NOW CAPABLE OF PERFORMING SI-TRACEABLE MEASUREMENTS UP TO 3 000 °C.

• everal manufacturers, especially in the aerospace and nuclear industries, need to know the thermophysical properties of materials at temperatures between 1 500 °C and 3 000 °C. However, until recently there was no way of guaranteeing SI-traceable measurements in this temperature range due to the lack of suitable metrological infrastructures. Setting up this very infrastructure was the challenge of the European Hi-TRACE project, which was completed in 2021 under LNE's coordination. Thanks to the project's accomplishments, traceable specific heat capacity, thermal diffusivity and spectral emissivity measurements can now be guaranteed up to 3 000 °C.

A NEW MACHINE FOR A WIDER TEMPERATURE RANGE

As Bruno Hay at LNE explains: "Before Hi-TRACE came along, no NMIs offered devices for measuring thermophysical properties operating in this temperature range. For example, even though our bench at LNE for measuring thermal diffusivity (relating to the velocity at which heat disperses through a material by conduction) was the most efficient in Europe, it was limited to a maximum temperature of 2 000 °C."

This bench specifically comprises an induction furnace in which a sample is thermally excited on one side through a brief laser pulse. The change in temperature on the opposite side is measured simultaneously as a function of time. The material's thermal diffusivity is then deduced by identifying with a theoretical model describing the heat transfer in the sample. To enable reliable measurements up to 3 000 °C, metrologists completely overhauled their installation: "We've changed the high-frequency electric current generator used for heating, dealt with the electromagnetic interference caused by the generator that is likely to affect measurements, modified the system to work in a neutral gas atmosphere to limit oxidation and contamination of the samples, and designed a system for the in situ calibration of the pyrometers used for measuring the sample's temperature," Bruno Hay advises.

Three questions for...

BRUNO HAY HEAD OF THE PHOTONICS-ENERGY DIVISION

Now that the Hi-TRACE project has drawn to a close, what position does LNE hold in thermophysical measurements at ultra-high temperatures?

B.H.: We're clearly strengthening our leadership by offering what is simply a world-unique measurement platform. Our platform is unrivalled for thermal diffusivity measurements, but it's also capable of performing specific heat capacity measurements, in addition to the benches that we've already got for emissivity measurements.

Aside from its purely metrological results, has the Hi-TRACE project had an impact on other aspects?

B.H.: Definitely, we've written a best practice guide for high-temperature thermal diffusivity measurements that will serve as a blueprint for the work of the Thermophysical Properties Working Group in the EURAMET Technical Committee in charge of Thermometry. In what was a rare occurrence for this type of project, Hi-TRACE gave us the opportunity of proposing a revision to the standard for measuring thermal diffusivity in ceramics.

Any there any other publications?

B.H.: In the wake of the Hi-TRACE project, 10 scientific articles were published in peer-reviewed journals. We've also developed five e-learning modules on thermal diffusivity measurements and on how to rigorously establish the associated uncertainty budget. These tutorials, the best practice guide and the experimental data obtained are freely available on the public Zenodo platform hosted by CERN. These publications clearly reinforce our position, especially among our partners in industry.



In addition, LNE's bench is capable of providing ultra-fine characterisation. Better still, it has also been fitted to measure specific heat capacity, which describes a material's capacity to store heat when its temperature rises. The new device is based on the so-called drop calorimetry principle. Once a sample is heated in a furnace, it drops rapidly into a calorimeter measuring the heat released during cooling. Once the measurement has been repeated for several initial temperatures, a model can be used to extract the quantity of interest

The German, Serbian and UK NMIs have also developed benches for measuring spectral emissivity, specific heat capacity and thermal diffusivity respectively. The UK's bench is adapted from a commercially available model. Subsequently, thermal diffusivity measurements at very high temperatures were compared among the different laboratories, including

KEY FIGURES

Empowered by its new standard machine, LNE can measure the thermal diffusivity of materials between room temperature and 3,000 °C. The extended uncertainty of these measurements is between 3 % and 5 %.

European NMIs and several manufacturers. The measurements were carried out on graphite, tungsten and molybdenum samples that had previously been prepared and characterised by LNE. The first observation is that "we have seen very good stability in the properties of these materials after several temperature cycles, which makes them ideal candidates for reference materials," says a delighted Bruno Hay. These facilities have also been used to characterise the thermophysical properties of metal alloys and composite materials at high temperatures.

THE ONLY FACILITY OF ITS KIND IN THE WORLD

In addition, all the thermal diffusivity measurements carried out by the different partners revealed a relative dispersion between 4 % and 9 %, "which is highly satisfactory", says Bruno Hay. To ensure the accuracy of their measurements, LNE's researchers conducted a rigorous assessment into the relative uncertainty. The outcome is that the relative uncertainty is less than 5 % between 23 °C and 3 000 °C. "This is simply the first time that such a result has been achieved for this type of measurement," says Bruno Hay, adding that, "when it comes to thermal diffusivity measurements, our bench is unrivalled around the world. It's also the only one with the ability to measure both a material's thermal diffusivity and specific heat capacity at such high temperatures." Even before the project had been wrapped up, LNE's new infrastructure had already been used by two companies in the space and metallurgical sectors to study the thermal behaviour of materials at very high temperatures.

LNE PURSUES ITS DEVELOPMENT STRATEGY FOR ELECTRICAL NANOMETROLOGY

A haracterising electronic microcomponents requires "We managed to achieve capacitance measurements with an the ability to measure their electrical properties uncertainty of 3 % when the sample to be characterised was Jon a nanometric scale. However, the metrological a reference structure, and less than 10 % when measuring infrastructure for such measurements still needs developing. the dielectric constant of ferroelectric materials, which are This is the aim of the MetroSMM project currently being especially used in nanoelectronics," says François Piquemal, led at LNE for high-frequency impedance measurements, Head of Nanometrology Activities at LNE. particularly for capacitance measurements, using a microwave local probe electric microscope (SMM).

One of the challenges involves the fine characterisation of the reference structures used to calibrate the SMM in terms of capacitance, as well as studying how the electromagnetic environment influences measurements. LNE's metrologists used calibration kits comprising microcapacitors developed by one of the project's industrial partners.

Electrical metrology researchers have also developed a so-called substitution method that allows an unknown sample to be characterised by comparing with measurements taken on the reference structure in a similar measurement environment.

LEIA 2: A PLATFORM FOR EVALUATING SMART ROBOTS

track, LNE was granted government funding in 2021 to Acreate the first generic platform for assessing artificial intelligence.

Basically, this artificial intelligence assessment laboratory (called LEIA 2), which has been slated for deployment in *testing.*" To be more precise, LEIA 2 will be especially suited to mid-sized robots and visual recognition systems, such as 2022, will take the form of a seven metre-high room with nine-metre sides. Inside, there will be a cylindrical screen smart cameras. for reproducing artificial environments to evaluate the At this particular stage, several letters of commitment have performance of the different types of smart robots. The already been confirmed with manufacturers and integrators of system will also feature physical load mechanisms, such as robotic systems. These platforms can also be used in research chassis dynamometers or conveyors. As explained by Anne projects aimed at supporting moves to standardise Al Kalouquine, the project manager responsible for setting up performance tests.



The same time, other complementary research activities are being pursued as part of the Éuropean ELENA (coordinated by LNE) and NanoWires projects. of photovoltaic nanowires used in energy harvesters.

s part of France's plan to get the economy back on the future laboratory at LNE: "LEIA 2 will be capable of performing intermediate tests between pure simulations and real physical tests. It will be part of an end-to-end offering that will ultimately include two other platforms, namely LEIA 1 for simulations and LEIA 3 for real-life

> "LEIA 2 WILL BE CAPABLE OF PERFORMING INTERMEDIATE **TESTS BETWEEN PURE** SIMULATIONS AND REAL PHYSICAL TESTS"

> > **ANNE KALOUGUINE, PROJECT MANAGER** FOR IMPLEMENTING LEIA2

LNEIR





NANOMETROLOGIA: AUTOMATED NANOMETROLOGY

o determine the size of nano-objects, metrologists rely on explained by Loïc Coquelin, a mathematics researcher at LNE: analysing the images produced by scanning electron microscopes (SEMs). LNE's experts are now able to carry out this process automatically using NanoMetrologIA, a web-based platform that was deployed at the laboratory last vear.

This infrastructure can be credited to the developments pioneered by the specialists from the Data Science and Uncertainty Department, who produced algorithms based on neural networks and deep learning models with the ability to automatically identify all the particles in an image, whether isolated or aggregated, and also reconstruct the contours of each particle from information that is often truncated. As

"From that moment on, the platform transparently provides users with a series of statistical information about the particles' size and aggregation level."

The experts initially developed their tools to identify titanium dioxide nanoparticles, which are widely used in many applications. The next step will focus on adapting these image analysis-based measurement tools to support a wide variety of samples and particles. On a theoretical level, efforts are already being made to guantify the uncertainty associated with those measurements. In the meantime, the world-unique NanoMetrologIA platform is reinforcing LNE's credentials as a trailblazer in the fast-growing nanoparticle metrology sector.

"THE PLATFORM TRANSPARENTLY PROVIDES **USERS WITH A SERIES OF** STATISTICAL INFORMATION ABOUT THE PARTICLES' SIZE AND AGGREGATION LEVEL."

LOÏC COQUELIN, RESEARCH ENGINEER, LNE



ECOLOGICAL TRANSITION

IN AN EFFORT TO TACKLE THE EFFECTS OF CLIMATE CHANGE CURRENTLY SWEEPING THE WORLD. LNE AND THE RNMF ARE HELPING DEVELOP ENVIRONMENTAL MONITORING METHODS AND SOLUTIONS TO REDUCE THE IMPACT OF HUMAN ACTIVITIES ON OUR PLANET.



ACIDITY MEASUREMENTS: LNE'S SOLUTIONS FOR COMPLEX ENVIRONMENTS

JUST BECAUSE MEASURING pH IS COMMON PRACTICE DOES NOT MAKE THE PROCESS ANY LESS COMPLEX. LNE IS DEVELOPING CHEMICAL METROLOGY INFRASTRUCTURES THAT ARE CAPABLE OF SUPPORTING ALL THE SOLUTIONS, NOT SIMPLY TO MEET INDUSTRY'S NEEDS, BUT ALSO KEEP TABS ON HOW RISING OCEAN TEMPERATURES ARE AFFECTING ACIDITY LEVELS.

hen it comes to measuring acidity, pH is one of the most widely measured properties in analytical laboratories and also one of the most important indicators. In environmental sciences, for example, ocean acidity is one of the parameters used to monitor the repercussions of global warming. However, measuring pH levels raises a number of challenges. In fact, the pH values measured in different media (organic solvents or solvent mixtures) are specific to each solvent and are consequently impossible to compare in practice. In addition, in case of aqueous solutions with a high salt content, there are no certified reference materials for pH to ensure that measurements are accurate and traceable to the International System of Units. Therefore, LNE has been directing its attention to this particular issue over the last 10 years, as evidenced by the progress achieved last year.

A UNIVERSAL AND ABSOLUTE **MEASURE OF ACIDITY**

2021 saw the completion of UnipHied, a European project coordinated by LNE and involving eight national metrology institutes, three university research teams and a manufacturer. The aim was to create a metrological structure to support the new universal pH concept, called absolute pH or pHabs. The pHabs concept was introduced in 2010 and is based on a measurement of acidity that does not refer to the proton activity in a given solvent, but instead relates to the activity of proton gas at a temperature of 298.15 K and a pressure of 1 bar. These conditions represent the universal standard.

"Our solution was to implement a differential method that involved using an ionic liquid to almost completely eliminate the liquid junction potentials that are the main source of uncertainty in pH measurements," explains Daniela Stoica, Project Coordinator at LNE. This method was used in the different partner laboratories and validated with various solvents (water, ethanol and water/ethanol mixture) representing several real-world applications. She adds: "Specifically, we developed a reference method as well as a simpler alternative method with a view to improving the uptake of this new pH concept as a routine measurement in laboratories for a wide variety of solvents.



Three questions for...

DANIELA STOICA

ELECTROCHEMISTRY R&D ENGINEER

How did LNE develop its expertise in pH metrology?

D.S.: Over the last 10 years, electrochemistry and especially pH-metry have been a major focus area for our department. We're playing our role as a steward of the country's primary standards in this particular area. LNE is maintaining its calibration and measurement capabilities by actively contributing to the work of the BIPM. We're demonstrating that our pH measurement results are internationally equivalent through our primary standards of measurement, especially the Harned cell, which is the national standard for the measurement of acidity. Furthermore, we're developing our skills and taking part in projects aimed at fulfilling the practical needs of communities using pH measurements.

Can you give us some examples?

D.S.: Examples include the European BIOFUELS and OCEAN projects, which focused on developing metrological standards for complex environments. The goal behind the BIOFUELS project was to satisfy manufacturers' biofuel needs, while the OCEAN project was aimed at monitoring ocean acidity levels in the wake of the changes affecting the environment. On a national level, we also took part in the ANR SApHIRE project for validating an innovative pH sensor.

Where does LNE stand in terms of pH measurements on a European level?

D.S.: LNE plays a vitally important role! LNE is leading the "Ocean Observation" topic within the European Metrology Network (EMN) implemented by EURAMET to harness the benefits of metrology for climate and ocean observation activities. Taking part in this network is in keeping with the "climate monitoring" research topic that has been identified as a strategic area for LNE.

METROLOGY FOR OCEANOGRAPHERS

At the same time, LNE researchers are drawing on their expertise and skills to ensure the quality of total pH or pH_{τ} measurement results. This parameter is determined from the total concentration of protons (free or associated with other ions) and is used by oceanographers to track changes in ocean acidity levels. Unlike usual pH, which is determined through potentiometric measurements, pH_{τ} involves the use of spectrophotometry and is actually considered by oceanographers to have lower uncertainties.

In 2021 on behalf of SOMLIT (the French Coastal Observation Network), LNE produced reference buffer solutions (TRIS-TRIS. HCl) that had been prepared in artificial seawater and characterised as a function of temperature with the laboratory's primary bench. The solutions were used for a comparison featuring 14 laboratories within the SOMLIT network. This collaboration was part of LNE's general programme to meet oceanographers' needs for data that can be compared in time and space (different geographical locations) and which are independent of the measurement technology used. This is also the aim behind a doctoral thesis started in 2021 that is being jointly supervised with the Marine Environment Chemistry Laboratory of the Mediterranean Institute of Oceanology in Marseille. As she explains: "On the one hand, the goal will be to provide oceanographers with the metrological concepts required to monitor trends in ocean acidity, whether using pH_{τ} or total alkalinity, and on the other, we'll be looking to learn from their experimental practices."

HARMONISING THE PROCEDURES FOR MEASURING pH_T

Finally, the European SApHTIES project coordinated by LNE was launched in 2021. The objective is to improve the ISO standard on the spectrophotometric determination of the pH_{τ} of seawater by introducing the fundamental metrological principles that it currently lacks. The first step will involve ascertaining information about the oceanographic community's practices using a questionnaire developed in partnership with IFREMER. This intel can then be used to identify and provide specific solutions for harmonising measurement practices. The various points that will be addressed during the project include extending the applicability of the spectrophotometric method to encompass the environmental conditions representative of nearshore and oceanic seawater (salinity and temperatures) and proposing essential tools for validating the spectrophotometric method, such as standard buffer solutions.

KEY FIGURE

zcientific articles co-authored by LNE were published in 2021 on all topics relating to acidity measurements in complex environments, demonstrating that LNE is clearly playing a frontline role in acidity measurements.

"THE OBJECTIVE IS TO IMPROVE THE ISO STANDARD ON THE **SPECTROPHOTOMETRIC** DETERMINATION OF THE pH_T OF SEAWATER BY INTRODUCING THE FUNDAMENTAL METROLOGICAL PRINCIPLES THAT IT CURRENTLY LACKS.'

DYNAMIC PRESSURE CALIBRATION: HIGH FREQUENCIES IN THE SPOTLIGHT

utomotive, aviation, power... the number of industries needing to calibrate their dynamic pressure and temperature sensors continues to grow. As part of the European DynPT project, which was completed in 2021, the LNE-ENSAM dynamic metrology laboratory helped set up new calibration services in the pressure ranges and dynamic systems of interest to manufacturers.

French metrologists have implemented two methods for calibrating reference sensors. The first method, known as the collective standard, involves performing a step-by-step calibration while exploring ever higher frequencies using different devices, especially shock tubes. "For pressures around 5 bar, we've managed to extend the frequency range to 30 kHz compared to the previous 10 kHz, while increasing the uncertainty by approximately 6 %," explains Christophe Sarraf at I NF-ENSAM





The second method, known as the chronometric method, is based on measurements carried out in static conditions using a model derived from the Rankine-Hugoniot relations. With this method, the corresponding dynamic quantities can be established. Even though it is subject to greater uncertainty, it has nevertheless allowed for calibrations up to 50 bar and should ultimately achieve 100 kHz up to 100 bar.

In addition to these two primary methods, the French metrology laboratory has also pioneered two other innovative methods for calibrating transfer sensors, which will give end users of the measurements access to dynamic pressure calibration. Furthermore, improving engine performance through expertise in dynamic pressure measurements can only have a significant impact on the environment.

WATER QUALITY: FROM CONTINUOUS MEASUREMENTS **TO METROLOGICAL PRECISION**

valuating water quality (tap water, river water or water from wastewater treatment plants) involves measuring measurement devices to meet users' growing needs (water several physico-chemical parameters, including the amount of micropollutants. According to traditional practice, these parameters are occasionally assessed using methods that require samples to be transported to the laboratory for analysis. Conversely, numerous sensors, in situ probes, inline analysers and portable devices now allow for continuous measurements of several parameters (temperature, pH, dissolved oxygen content, conductivity, turbidity, and chlorine, nitrate, chlorophyll and metal concentrations). But due to the lack of specialised metrological facilities, rolling out these measuring instruments is a slow business. To speed up the process, LNE has developed a special test bench for evaluating and calibrating all these innovative sensors.

Specifically, this development within the French National Metrology Network builds on the implementation of a standard agencies, manufacturers, etc.).

Project Manager Nathalie Guigues explains: "Our bench covers the whole range of applications and can be adapted to all types of sensors. Their performance is assessed by comparing the measurements made with those sensors on a recirculating fluid against the measurements made on samples of this fluid by primary or standard methods that are geared towards each physico-chemical parameter analysed."

After starting from a situation where no metrological data were available, LNE's chemists can now offer the possibility of continuous water quality measurements with uncertainties generally below 15 %. Several manufacturers, including Veolia, are already taking advantage of this innovation.



MULTI-PARAMETER PROBE FOR ASSESSING CHLOROPHYLL PIGMENTS, CIANOBACTERIA AND TURBIDITY.

"OUR BENCH COVERS THE WHOLE RANGE OF APPLICATIONS AND CAN **BE ADAPTED TO ALL TYPES OF SENSORS.**"

> **NATHALIE GUIGUES** PROJECT MANAGER, LNE





PUBLIC HEALTH AND SAFETY

WHETHER ASSESSING THE RISKS ASSOCIATED WITH A NEW SUBSTANCE, ENSURING FOOD SAFETY OR DEVELOPING NEW MEDICAL TREATMENTS, LNE AND THE RNMF ARE INVOLVED IN EVERY ASPECT OF PUBLIC SAFETY.



RENEWAL OF ACTIA SAFEMAT: RECYCLING AND REUSING FOOD GRADE MATERIALS

CREATED IN 2017, THE ACTIA SAFEMAT JOINT TECHNOLOGY UNIT HAS JUST SEEN ITS MANDATE EXTENDED BY ANOTHER FIVE YEARS. IT ALLOWS LNE AND THE SAYFOOD JOINT RESEARCH UNIT TO ADDRESS THE INDUSTRIAL ISSUES RELATING TO THE SAFETY OF FOOD CONTACT MATERIALS AND PACKAGING.

Bio-based materials, multi-layered packaging, nanostructured materials... whether responding to food preservation issues or tackling environmental challenges, the food packaging sector is clearly a hi-tech industry. As such, it is faced with the problem of assessing whether these materials are fit for food contact and capable of guaranteeing food safety. In response to this challenge, LNE and the SayFood joint research unit (between AgroParisTech and INRAe) teamed up by creating the ACTIA SafeMat joint technology unit in 2017. In light of the ever changing regulatory landscape, the prospect of phasing out single-use plastic packaging by 2040 and the development of recovery, recycling and reuse processes, ACTIA SafeMat has just been renewed for a five-year period and is ramping up its research projects.

Jean-Mario Julien at LNE explains: "Our aim in creating this joint technology unit was to strengthen the ties between our two entities and combine our strengths, especially SayFood's modelling skills and LNE's analytical expertise. In doing so, we've achieved the critical mass required to undertake largescale projects."

Basically, SafeMat specialises in studying the physico-chemical mechanisms controlling contamination, developing physicochemical methods to validate predictive approaches in real-life applications, and disseminating tools and data to industry and the relevant authorities.

From the outset, ACTIA SafeMat has been involved in the European MyPack programme by putting forward analytical solutions for characterising materials and has assisted the consortium's partners in assessing whether materials are suitable for food contact. The materials examined were derived from renewable resources and produced by manufacturing partners using seven innovative and sustainable technologies. "The idea was to help manufacturers modify the functional properties of their materials and align with the regulatory requirements applicable to food contact materials," explains Jean-Mario Julien at LNE.

Since 2020, ACTIA SafeMat has been coordinating a partnership research project called *"ABA Modelling"* involving CITEO and



Three questions for...

JEAN-MARIO JULIEN, R&D ENGINEER IN THE THERMOPHYSICAL PROPERTIES OF MATERIALS DEPARTMENT

What are your views on the transition from SafeMat to SafeMat 2?

J-M.J: It's a form of continuity, just like SafeMat. When SafeMat was created six years ago, it was a natural extension of the ties that had already been forged between LNE and the teams at INRAe and AgroParisTech in Massy. Having said that, SafeMat has really taught us how to work together, and we now form a laboratory. We may not all be working in the same room, but we're a tight-knit team. Now when we're looking at potential projects, it's become second nature to deliver a joint response.

How has the creation of SafeMat affected your image as perceived by your industrial and institutional partners? J-M.J: By bringing all of LNE's and SayFood's skills together under one roof, we've been able to address all the issues relating to food contact materials. SayFood is now recognised by industry, the European Commission and the FDA for its expertise in migration prediction and risk management tools.

What is SafeMat's philosophy for the next five years? J-M.J: When we were renewing our accreditation with ACTIA, our objectives were defined to reflect European regulations on reducing the impact of certain plastic products on the environment, as well as French regulations aimed at tackling waste and promoting the circular economy. We'll be taking part in projects that further the aims of those regulations.

LNE | RESEARCH RE



two other tech hubs (IPC and CTCPA), which aims to assess the risk of food contamination from three-layer ABA trays, where the B layer is made from recycled polyethylene terephthalate (rPET).

During the same year, ACTIA SafeMat also wrapped up the CosmetoPack project with the goal of developing a protocol for managing the chemical risks associated with interactions between cosmetics and their packaging.

In addition, it also coordinated the scientific part of the European Erasmus+ FitNESS project, which endeavoured to create a web-based training and information platform on food packaging for both students and employees in the companies involved. Empowered by the success of this initiative, Erasmus+ has now agreed to a second version of FitNESS. "Following the decision to renew the joint technology unit, we are going to focus on disseminating information to the public authorities," explains Phuong-Mai Nguyen.

ADDRESSING RECYCLING AND REUSE ISSUES

More generally, ACTIA SafeMat V2 will dovetail with the national strategy on promoting the circular economy by firmly embracing the challenges relating to recycling and reuse. "We're going to direct our efforts in three directions," explains

Jean-Mario Julien, "food contact compatibility of recycled materials, the ageing of recycled materials, and packaging/ product engineering.

SafeMat has been playing an active part in three ANR projects on these issues since last year. Researchers in the PolySafe project are assessing the biological effects of alternative materials to plastic. In particular, LNE's chemists are responsible for preparing biocompatible extracts from samples used in the catering industry and identifying the molecules present. Since they are likely to migrate, their impact on the human metabolism will then be assessed by the toxicology and biology teams. In addition, as part of the FoodSafeBioPack project, SafeMat's scientists are proposing multi-scale predictive models for the transfer of substances from printed paper/cardboard packaging to food. The objective is to develop a risk assessment tool that can be used by tech hubs and the authorities. Finally, the LNE researchers involved in the PackSafe project are going to develop new approaches that complement analytical and physico-chemical techniques by studying the potential toxicity of substances contained in packaging, in cases where each substance cannot be individually identified and quantified, with applications used to assess recycled materials. One thing is for sure: SafeMat, LNE and its partners are now key players in the field of food grade materials.

LNE-LNHB COMPLETES THE EXPERIMENTAL **DETERMINATION OF THE LOW AND MEDIUM-ENERGY X-RAY SPECTRA**

ranging between 10 keV and 300 keV, are distributed over four separate benches and used for calibration applications in the fields of medical imaging, radiotherapy and radiation protection. Over the last 10 years, the laboratory has spearheaded various projects to characterise the beams in detail. The last project has just been completed.

In the early 2010s, metrologists began building two benches geared towards the semiconductor detectors used to measure the energy emissions of the X-ray beams.

Until now, the highly specific energy spectra produced by X-ray generators could only be determined through deterministic and/or Monte Carlo calculations. Subsequently, researchers developed algorithms for correcting the measured spectra, while taking account of the biases associated with the measurements, including radiation-induced interference in the actual detectors, and they began characterising the beams at the same time. They finished characterising the beams between 2019 and 2021, which enabled the scientists at LNE-LNHB to determine the conversion coefficients for linking the lab-based measurements and the on-site measurements, and thereby calibrating the detectors for users in the radiation protection



KEY FIGURES

Since its inception, SafeMat has been involved in three ANR projects and two European projects: Erasmus+ and H2020. It has also hosted five PhD students and three PhD graduates. SafeMat's activities have culminated in the publication of 15 scientific articles and the dissemination of open source content as part of the European FitNESS project.

"SAFEMAT HAS REALLY TAUGHT US HOW TO WORK TOGETHER."

JEAN-MARIO JULIEN R&D ENGINEER. LNE

LNE-LNHB's 96 primary X-ray beams, with energy levels sector. These projects have had a considerable impact on this sector of activity by reducing measurement uncertainties by a factor of four on average and providing clearer insights into the energy of the weakest X-ray beams (less than 60 keV).

> 🖙 At the same time, the experts applied their enhanced method for characterising X-ray beams to the generators used for other applications, insofar as each beam has its specific attributes. "In line with the 2020, our work encompassed the X-rays used in interventional radiology for cardiology," explains Johann Plagnard, Overall Project Leader. This achievement heralds the prospect of calibrating to the highest level of measurement in all sectors concerned.

FUNDAMENTAL METROLOGY

DESIGNING AND IMPROVING NATIONAL MEASUREMENT STANDARDS, DISSEMINATING STANDARDS TO USERS WITH THE BEST UNCERTAINTIES, AND DRIVING INNOVATION AND PROGRESS IN FUNDAMENTAL METROLOGY SUPPORT DEVELOPMENTS IN ALL HUMAN ACTIVITIES.







COMPARING CLOCKS IN THE DIGITAL ERA



Time) and UTC (Coordinated Universal Time) is based on comparisons between remote atomic clocks using satellite links. As part of a project spearheaded by the French metrology system last year, metrologists at LNE-SYRTE equipped the laboratory's TWSTFT station (Two-Way Satellite Time and Frequency Transfer) with a software-defined radio (SDR) system, which paves the way for digitising the links between clocks and thereby reducing comparison uncertainty. The signals received by the station were previously processed entirely in analogue format. Now they are sampled during demodulation, before being processed at very high speed by a field programmable gate array (FPGA).

As an added bonus, clock signals from 20 stations can be compared simultaneously, as opposed to only two previously. In addition, the digitisation process brings a reduction in experimental noise, which lowers the instability of the satellite link to better than 10⁻¹⁶ in less than 10 days.

he process of establishing TAI (International Atomic Project Manager Joseph Achkar explains: "We've also developed a "travelling" SDR receiver, which has helped reduce the calibration uncertainty of the satellite link between LNE-SYRTE and the German National Metrology Institute (PTB) to a record value of 0.5 ns, compared to the previous 1 ns." This TWSDR link, which was inaugurated by the BIPM for calculating UTC, foreshadows an upgrade to the satellite network for achieving a level of precision that is compatible with the future definition of the second.

> **JOSEPH ACHKAR** was recently appointed Chair of the ITU (International Telecommunication Union) Working Party on Time Signals and Frequency Standard Emissions, and Chair of the EURAMET Technical Committee for Time and Frequency.

RADIONUCLIDES: DECAY UNDER THE MICROSCOPE



radionuclides, the branching fractions can be established, i.e. n-depth knowledge of radionuclide decay data is crucial to both nuclear medicine and radiation protection. In a the probability that decay will take place via a given pathway. bid to gain an even greater understanding of This information is vitally important when using the radionuclide decay, LNE-LNHB took part in the European measurement results obtained by other methods outside the MetroMMC project for developing and operating metallic metrology laboratories. magnetic calorimeters (MMCs). The project was completed As part of MetroMMC and other concurrent projects, French in 2021. researchers have examined the decay of about 10 radionuclides.

During radioactive decay, an MMC detects the energy of a particle through the temperature increase caused as it interacts with the absorber. Therefore, these detectors are sensitive to all types of ionising radiation. MMCs offer good energy resolution with a low trigger threshold.

As such, they are capable of measuring the total energy associated with radioactive decay, and that information can be harnessed to determine other associated data. For some

Matias Rodrigues, a researcher at LNE-LNHB, points out: "Until now, most of the branching fractions and other associated data could only be determined by calculation. Our measurements can be used to confirm or refine them, as well as validate the theoretical models used to determine the radionuclide parameters that cannot be measured." This development will help refine the entire metrological chain for measuring radioactive decay.

QUANTUM TECHNOLOGIES

LNE: A LEADING FORCE IN DEPLOYING THE NATIONAL QUANTUM STRATEGY

n 21 January 2021, French President Emmanuel Macron presented the National Strategy on Quantum Technologies (SNQ) with the aim of building an ecosystem to carve France's status as one of the leading nations for expertise in quantum technologies. LNE will play a key role in realising the plan's ambitions.

It has to be said that LNE is already highly active in the networks aimed at unifying and galvanising research and development efforts on quantum technologies. For example, LNE is a member of the Quantum Sciences and Technologies Centre at Paris-Saclay University (QUANTUM) and participates alongside other RNMF laboratories in SIRTEQ (the Francilien Network for Quantum Technologies) and its successor QuanTIP. In these areas, LNE has been fostering collaborative ties for many years with the teams at CEA and CNRS, especially the Centre for Nanosciences and Nanotechnologies (C2N).

In line with the SNQ strategy, LNE will be responsible for rolling out a quantum metrology platform within the RNMF. Félicien Schopfer from LNE explains: "The idea is to leverage all the progress achieved by quantum technologies to power the new generation of quantum standards, and conversely deliver solutions to meet the measurement needs for developing these technologies." Consequently, LNE has also been entrusted with coordinating the programme to develop measurement guidelines, standards and assessments for quantum technologies, which represents a critical part of the SNQ strategy for industry.



LNE: ACTIVELY ENGAGED IN EVERY EUROPEAN ASPECT OF QUANTUM TECHNOLOGIES



he soaring growth of quantum technologies across the continent partly depends on the metrology community's ability to analyse needs and implement coordinated strategies between the various stakeholders on a European scale. With this aim in mind, EURAMET set up the *European Metrology Network* (EMN) for quantum technologies in 2019, known as EMN-Q. For example, the EMN-Q produced the first version of its Strategic Research Agenda last year.

"Quantum technologies need metrology, and metrology has long been involved in their development," advises Sébastien Bize of LNE-SYRTE and Vice Chair of the Quantum Clocks & Atomic Sensors section within the EMN-Q, before adding, "metrology also acts as an interface with standardisation issues." This explains why the RNMF plays a central role within the EMN-Q, with two of its members sitting on the steering committee, especially since LNE-SYRTE has spent many years blazing a trail in time-frequency as well as inertial and gravimetric sensors. The RNMF's strong position can also be attributed to LNE's many achievements in developing





- quantum standards for electrical metrology and LNE-LCM/ Cnam's developments for characterising single-photon sensors.
- In 2021, LNE also joined the *Focus Group on Quantum Technology* (FGQT) set up by the CEN-CENELEC standardisation committee to help identify the specific standardisation needs for quantum technologies and ultimately support the deployment of these new technologies for industry.
- Lastly, LNE has been an associate member of the European Quantum Industry Consortium (QuIC) since last June. Alongside its industrial partners, LNE is actively involved in several standards working groups and helping develop industrial roadmaps.
- Most of these activities are carried out in alignment with *Quantum Flagship*, the large-scale European research and innovation initiative. This 10-year programme was launched in 2018 with a 1 billion of € funding plan from the European Commission, representing a groundbreaking strategic initiative in the field of quantum technologies!

QUANTUN TECHNOLOGI

TEMPERATURE MEASUREMENTS: QUANTUM PRECISION AT YOUR **FINGERTIPS**



GRAPHENE: A KEY MATERIAL FOR ELECTRICAL METROLOGY WITH PLENTY OF PROMISE FOR QUANTUM TECHNOLOGY



ncreasingly miniaturised electronic, photonic and optoelectronic components require the development of local temperature sensors offering sub-micrometre spatial resolution. Such was the challenge facing the European PhotOQuanT project coordinated by LNE-LCM/ Cnam, which ended in 2021. To realise the project's aims, French metrologists and their colleagues developed an optomechanical temperature sensor, which will ultimately serve as a quantum sensor for measuring thermodynamic temperatures.

Specifically, this sensor is designed as a micrometric optomechanical resonator whose temperature is determined from the thermal noise (quantum) that can be accessed by measuring the phase modulation of a probe laser created by the thermal vibrations combined with optical excitation of the resonator. "Working within the PhotOQuanT project, we had

to start from scratch, but we still managed to perform temperature measurements with our sensor, which accounts for 80 % of the objective," explains Stephan Briaudeau, a researcher and project manager at LNE-LCM/Cnam.

To reach the project's goals, physicists are now working on irrevocably linking the resonator's optomechanical excitation to the photons of the probe laser. This approach has a key benefit, insofar as quantum mechanics indicates that the resonator's temperature is then a direct function of the quantum correlation created between the phase and amplitude fluctuations of the probe laser. French experts will have an intrinsically thermodynamic thermometer and will consequently join ranks with their colleagues at the US Metrology Institute of Standards and Technology (NIST), who are currently the only metrologists to master temperature measurements in the guantum regime using an optomechanical sensor.

raphene is currently the preferred material when to which they are currently subjected. G making quantum standards of electrical resistance and exploring certain quantum technologies. As part of The measurements gave them a clearer understanding of the impact on the Hall dissipation and quantification properties, a European EMPIR project completed last year, which involved not only for electron mobility but also specific attributes of the three European metrology laboratories and two French samples (size, presence of an electrostatic graphite grid at a research laboratories (CEA and CNRS), LNE's researchers few tens of nanometres from the graphene, etc.). With the prospect of developing new quantum technologies, researchers were able to study graphene samples with very high electron mobility also took a closer look at the breakdown of the quantum Hall The global aim behind the SEQUOIA project was to explore the effect. They estimated the potential of this phenomenon for the rapid detection of electrons.

feasibility of ultra-fast, highly sensitive electromagnetic sensors based on single-electron interferometry. The French metrologists had the ideal opportunity to study the quantification of the Hall effect in two graphene samples encapsulated between two layers of boron nitride with the aim of producing guantum resistance standards functioning with a magnetic field of less intensity than the magnetic field



Wilfrid Poirier, Project Manager at LNE, explains: "In addition to the results obtained, this project represented a tremendous opportunity to broaden collaborative ties between the electrical metrology and quantum technology research communities. We also organised the project's closing meeting."

PHD THESES DEFENDED IN 2021

LNE

Huu-Hien Huynh

29 January 2021 "Development of a candidate reference method for quantifying procalcitonin in human serum" PSL Université (Paris Sciences & Lettres) / ESPCI Paris, Analytical Chemistry

Valentin De Carsalade Du Pont 16 April 2021

"Fractionation and characterisation of nanoparticles by a hydrodynamic method: modelling and application for consumer products" PSL Université (Paris Sciences & Lettres) / ESPCI Paris, Physics

Élodie Mirmont

29 April 2021 "Development of mass spectrometric guantification methods for steroid hormones and related compounds in environmental and biological matrices" University of Paris, Analytical Chemistry

Quentin Hamdaoui

28 June 2021 "Development of a controlled exposure device for analysing the neurotoxic inhalation effects of paraguat and TiO2 nanoobject aerosols: applications for neurodevelopmental and neurodegenerative conditions" University of Lyon / ENS Lyon, Molecular, Integrative and Cellular Biology (BMIC)

Nicolas Mézières

28 August 2021 "Contributions to fast and accurate antenna characterisation" University of Rennes 1, Electronics

Najoua Bouzakher Ghomrasni

30 September 2021 "Identification and metrological characterisation of nanoparticles in complex matrices" Paris-Saclay University, Chemistry

LNE-SYRTE

William Dubosclard

12 February 2021 "Enabling technologies for compact on-chip confined-atom interferometers" Sorbonne University (Paris), Physics

Héctor Alvarez Martinez 15 March 2021

"Characterisation of optical frequency comb-based measurements and spectral purity transfer for optical atomic clocks" Sorbonne University (Paris) and Universidad Carlos III (Madrid), Physics

Alexandre Bouvier

31 May 2021 "Studies on the medium/long-term stability of the double-modulation coherent population trapping clock" Sorbonne University (Paris), Physics

LNE-LCM/Cnam

Pascal Gambette

15 December 2021 "Towards a quantum standard for absolute pressure measurements" HESAM University / Cnam-Paris, Lasers, Nanosciences & Metrology

LNE-LNHB

Lorenzo Périssé

20 September 2021 "Modelling of reactor antineutrino spectra" Paris-Saclay University, Particle Physics

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