



Measurements for innovative technologies

A summary of the outputs and impacts from the EMRP joint research projects in the New Technologies theme.

The aim of this research theme is to develop the measurement methods and techniques needed to support innovation and promote the uptake of new enabling technologies into products and services that will underpin future economic growth. This research is focused on technologies for the nanoscale, for advanced material characterisation, and those for a safe and secure world.

Measurement matters

Measurement underpins virtually every aspect of our daily lives, helping to ensure quality and safety, supporting technological innovation, and keeping our economy competitive.

Supported by the European Commission, EURAMET's **European Metrology Research Programme (EMRP)** brings together National Measurement Institutes in 23 countries to pool scientific and financial resources to address key measurement challenges at a European level.

The programme is designed to ensure that measurement science meets the future needs of industry and wider society. Research is structured around six themes – Energy, Environment, Health and Industry – as well as the measurement needs of emerging technologies and the fundamentals of the SI measurement units that form the basis of Europe's measurement infrastructure.

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Introduction:

Europe is the birthplace of many innovations and has great potential to benefit from this capability, by better integrating of new technologies into products and services to sustain economic growth. The adoption of new technologies represents an important business opportunity in a market estimated to be worth at least €750 billion by 2021. To help achieve economic growth in this rapidly expanding area, the EU has identified 6 important Key Enabling Technologies for targeted research: micro and nanoelectronics, nanotechnology, industrial biotechnology, advanced materials, photonics, and advanced manufacturing technologies. Focusing on these areas will help deliver the EU's goal of a sustainable and high-employment economy based on a wide range of modernised traditional and emerging new industries.

Crucial for the introduction of advanced technology is the ability to reliably measure material and component properties and to robustly confirm performance. This in turn relies on measurements evolving to meet the requirements of technology advances. Europe is home to world leading metrology capabilities centred on its National Metrology Institutes (NMIs) and Designated Institutes (DIs), where knowledge and facilities across the measurement spectrum are able to underpin all aspects of industry and society.

Euramet promotes collaboration between national measurement institutes, and through its EMRP New Technology theme has supported 9 collaborative research projects. This has brought together 66 research groups from 17 NMIs and DIs with industry and academia to address some of the measurement challenges associated with rapidly advancing technologies in a range of cross-disciplinary fields:

- nanotechnology
- new materials
- security
- biotechnology
- mathematics and ICT for metrology

This report presents the key technical achievements of these research projects and highlights early examples of the impact generated. The projects are grouped into three sub-themes: **Technology on a small scale**, **Technology for advanced materials**, and **Technology for a safe and secure world**.

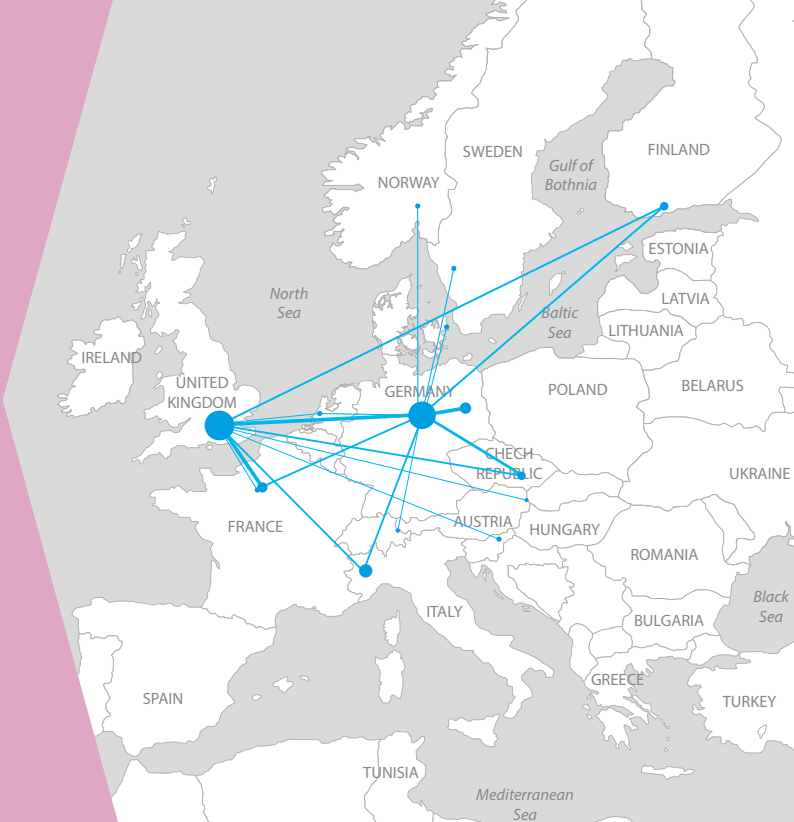


Highlights

Multidisciplinary measurement solutions for New Technologies

The Euramet EMRP theme has enabled the European metrology community to work collectively towards an improved measurement infrastructure for new technologies with the potential to underpin future economic growth. These projects have brought together metrology expertise in physics, chemistry, and biology to develop new analytical methods and statistical approaches that support greater uptake of novel materials or statistical approaches for new applications.

The European Commission and national governments invested €30 million in collaborative research projects, involving research groups in 17 European NMIs and Designated Institutes (DIs), 23 academic groups, and 9 businesses.



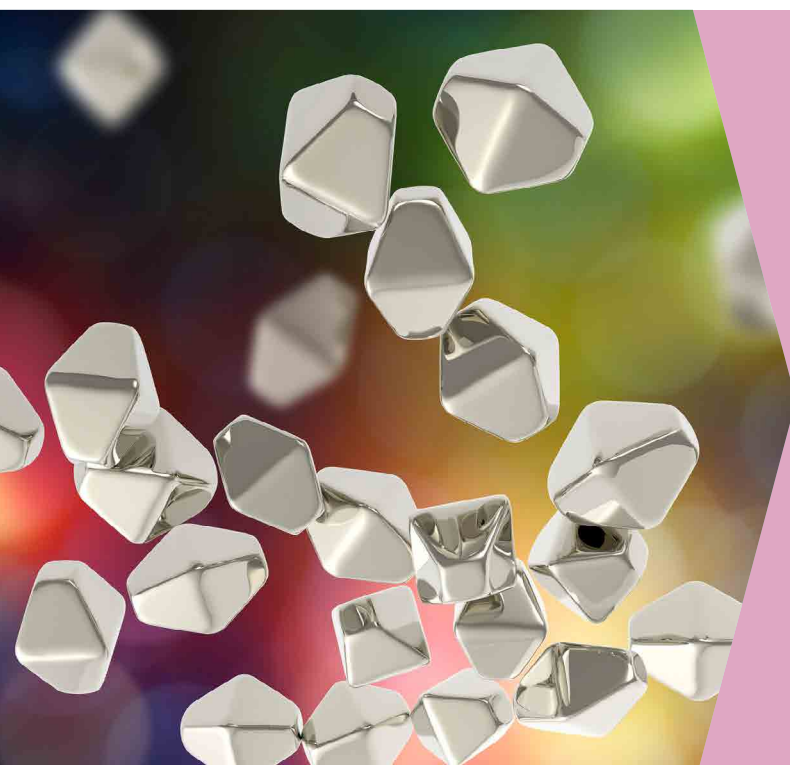
Map of participating NMI and DI

Innovation for next generation microelectronics

Reducing power consumption and increasing processing speed in next generation microelectronics, as silicon reaches its limit, relies on having confidence in the performance of new materials. EMRP research has developed robust techniques for nanoscale chemical analyses and also for nano-object mechanical testing – both essential for characterising microelectronic components and new materials.

For example, the accuracy of an advanced analysis tool – argon cluster sputtering – has been improved, making it suitable for determining individual chemical layer compositions in innovative multi-layer organic materials. A universal equation now permits the energy of argon cluster beams to be controlled and tailored to the material under test, so increasing analysis accuracy. This analysis is essential for confirming specific layers have been reliably produced during semi-conductor fabrication.

Further, the use of nano-wires as electrical conductors in next generation electronics requires confidence in their strength: For which, new mechanical testing techniques are required. EMRP research has successfully combined scanning electron microscopy with nanoscale sample preparation and strength testing for the first time. This new technique enables accurate selection and testing of specific material properties – an important first step towards innovation in electronics for future devices.

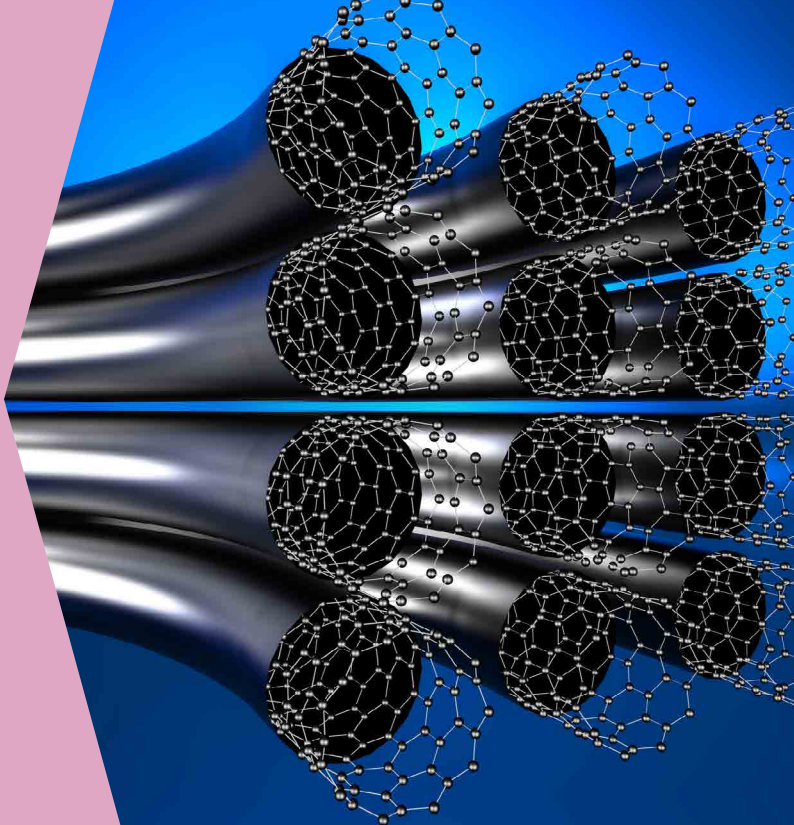


Evidence that nanoparticles are safe for use

Nanoparticles have great potential as future drug delivery methods due to the ease with which they can travel around inside our bodies. Understanding how they stick together in blood or travel across cell membranes once inside the body is key to determining their safety. EMRP research validated existing laboratory techniques using well characterised nanoparticles in biological media, as a first step towards a reliable measurement infrastructure for research into nanoparticle toxicity. Research highlighted the benefits of a two stage approach based on initial size segregation before analysis for accurately detecting the 'rare' particles in samples. This is important for risk assessments and toxicity studies.

Standardisation for nanoscale strength testing

International documentary standards form the basis for quality procedures that ensure measurements are reliable and reproducible no matter where or when they are made. As a result of EMRP research, atomic force microscopy (AFM) is now a recognised mechanical testing method under the governing ISO standard. For the first time, it is possible to reliably measure the mechanical properties of thin surface coatings and nanoscale features using a standardised approach similar to that used for bulk material samples. This enables the reliable comparison of data from different measurement scales. The revised ISO standard puts AFM strength testing on the same footing as testing for bulk material properties. Designers can now have greater confidence in nanoscale wire and pillar performance, essential information for including these features into new electronics.

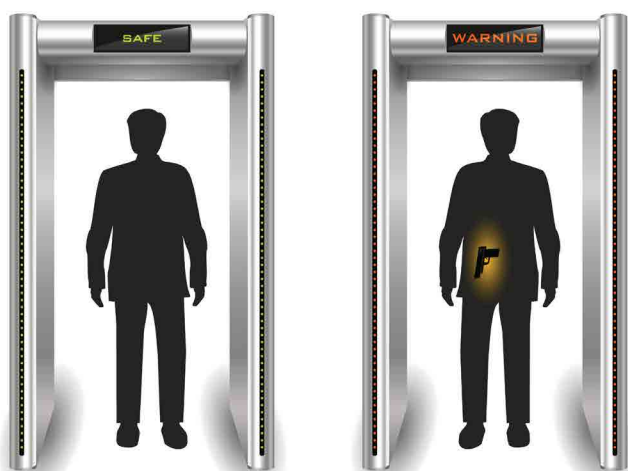


Innovation in programming validation and measurement software

Organisations need to be confident that complex computer programs are bug-free, and that those used for risk assessments based on new measurement uncertainty innovations are reliable. EMRP research has generated solutions for these programs using novel statistical approaches.

Applying the principles of instrument calibration to complex software, EMRP research is enabling computer programmers to be confident that new software routines are bug-free. The project developed an online system that provides standardised algorithms and ideal data sets to independently verify software performance. This has already provided users with software performance validation, and early adopters in a diverse range of applications including integrity safety testing and precision engineering.

In another project, new worked examples of how probability and existing data can be used to increase the certainty of measurements by reducing accuracy 'doubts' in a broad range of risk assessment applications have been generated. By introducing shortcuts in data processing and developing new statistical models, research has reduced reliance on multiple computer processors and calculation times without sacrificing accuracy. The project's statistical approaches have been successfully applied to diverse situations such as determining optimised re-test frequencies for new batches of energy smart meters and enabling the use of complex light scattering measurements in semiconductor quality control.



Evidence that terahertz security scanning is safe

Terahertz radiation is an ideal technology for quick and non-invasive security imaging, but confirming both operators and the travelling public are safe relies on accurately determining the effects it induces in the skin. EMRP research has developed and validated a new modelling approach for estimating terahertz radiation interactions with skin. It demonstrated that terahertz security scanning complies with the European "Physical Agents Directive" exposure limits for both instrument operators and the travelling public. This supports greater adoption of terahertz surveillance for public safety.

EMRP New Technology projects at a glance



Pooling expertise of

12 NMIs
and
5 DIs from
14 European countries



plus the NMI from **Brazil**



9 businesses



23 academic research groups



185

articles
in peer-reviewed
journals



training courses
delivered to over

1,800
people

296

presentations at
conferences



44

contributions
to

21

technical
committees and
working groups
of standards
organisations

Supported the
development of improved
instrumentation with
projected sales of

€4.5 M

21

contributions to draft standards
and published standards

Technology on a small scale

Measurement challenges

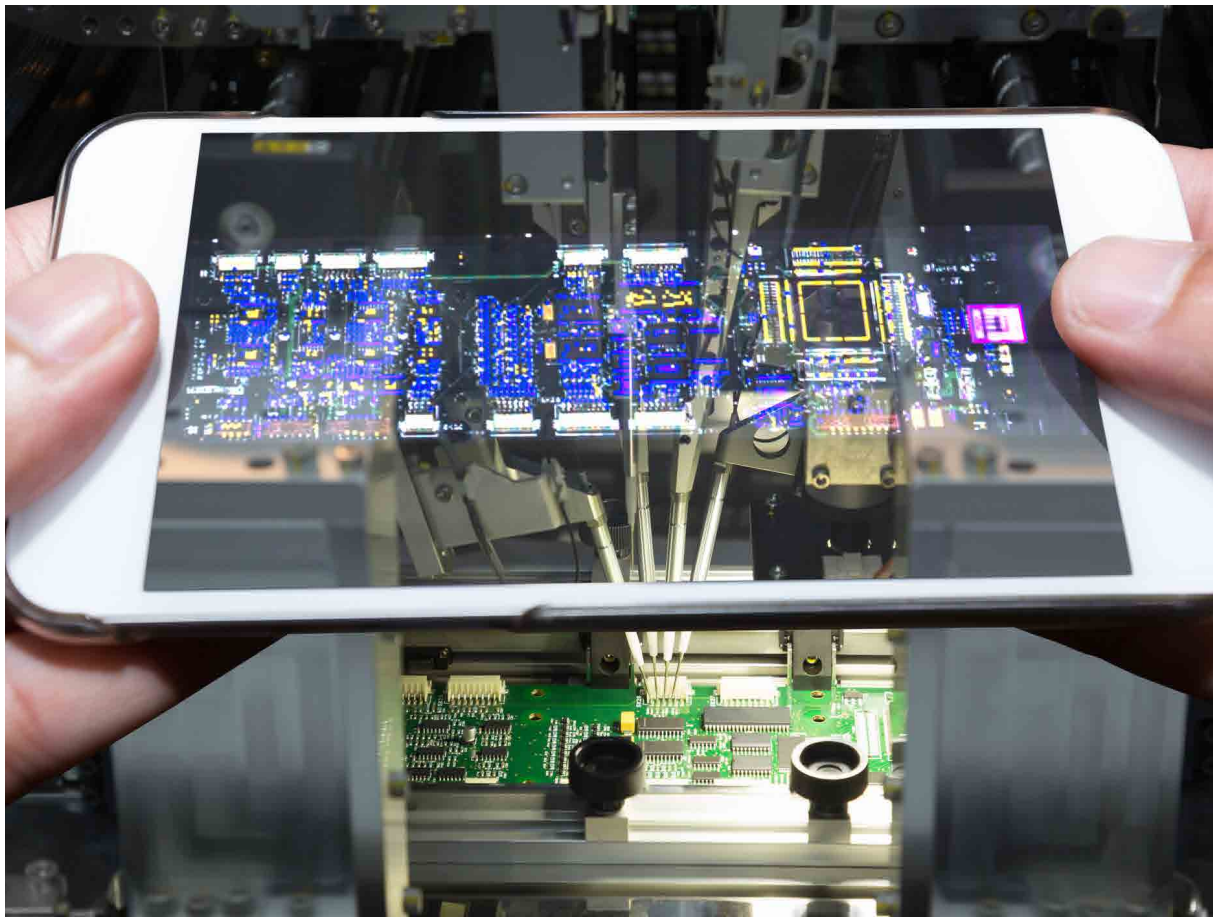
Innovations in high value industries, such as electronics, pharmaceuticals and bioscience, all hinge on our ability to characterise materials and products on an increasingly small scale. New developments like replacing traditional silicon devices with nanoelectronics based on organic semiconductors, or monitoring drug delivery to cells on a molecule-by-molecule basis, are now realities. For us to benefit from these rapid advances we need to ensure that their underlying physical and chemical properties can be accurately and reliably determined.

Current measurement methods cannot adequately characterise organic materials and new electronic architectures nor is any single technique able to provide all the information required. New advanced analysis methods for surface and near surface features are required to support innovation in the electronics industry.

In the pharmaceutical, healthcare, and biotechnology sectors Raman spectroscopy is routinely used as a non-destructive, contactless, fast method for identifying and mapping chemical distributions and nanoscale structures in 2D or 3D. However, the accuracy and reliability of this technique is limited by traceability limitations that constrain its use.

EMRP research has supported projects that address both of these problems:

- The development of new 2D and 3D electrical and chemical characterisation techniques for use with both inorganic and organic semiconductor materials.
- The development of an SI traceability chain for Raman spectroscopy to assist in the uptake of the technique into new applications, such as label-free quantitative imaging for processes occurring within living cells or environmental monitoring.



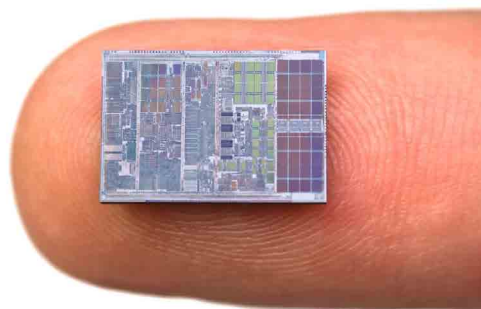
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Key technical achievements

Characterising nanolayered structures

A revolution is occurring in the world of micro and nano electronics in terms of miniaturisation, power consumption, and processing speed as new organic materials and multi-layer films are introduced. Novel inorganic semiconductor materials and new 3D structures are replacing traditional silicon devices and need new characterisation methods to facilitate their use in tomorrow's electronics.

The EMRP project **Traceable characterisation of nanostructured devices (TReND)** developed robust methods for characterising inorganic nanolayers and sub-surface features for semiconducting materials.



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The project:

- **Optimised and validated** non-destructive x-ray analysis methods for characterising and chemical depth-profiling of traditional inorganic nanolayers, such as silicon films. These new analysis techniques are important for achieving greater semiconductor miniaturisation.
- **Derived a 'universal equation'** relating argon cluster beam parameters to the ions it generates, important for 3D nanoscale chemical imaging of organic electronic materials using mass spectrometry. This was subsequently used in a different project to optimise the argon cluster ion beam for a novel 3D OrbiSIMS instrument.
- **Demonstrated high resolution 2D mapping** of local electrical and photo-electrical properties in organic semiconductor nanostructures using photoconductive atomic force microscopy.
- **Developed a visualisation tool** to extract 3D nanoelectrical information from 2D mapping data to enable future development of new direct 3D measurement methods.

In new electronic devices, miniaturisation is creating higher demand for a more detailed understanding of deposition processes and reaction mechanisms in manufacturing processes. To address this, the project optimised non-destructive x-ray analysis techniques for accurately determining changes in chemical composition with depth. Through a rigorous evaluation of grazing-incidence x-ray fluorescence it is now possible to verify calibration standards used for in-process nanofilm deposition quality control, where previously only reference materials were available.

The project's 'universal equation' has been applied to beam control in a revolutionary new instrument, the 3D OrbiSIMS developed in a collaboration between the pharmaceutical company GlaxoSmithKline, mass spectrometry company Thermo Fisher Scientific, the University of Nottingham and TReND project partners NPL and ION-TOF GmbH, an innovative instrument manufacturer for surface analysis mass spectrometers. Marketed as the 3D OrbiSIMS by ION-TOF, the instrument has the highest simultaneous spatial and mass resolving power achieved to date. This is important for accurately determining complex sub-surface organic nanolayer chemistry which has uses in both biomedicine and electronics. A follow-on EMPIR project **An International Standard for Reliable Chemical Depth Profiling of Organic Materials** is preparing TReND developments for incorporation into an ISO standard for organic depth profiling. Once issued the ISO standard will increase the reliability of novel organic layer analyses that are important for greater uptake of organic materials into new applications.

More information is available at	NEW01: Traceable characterisation of nanostructured devices (TReND) http://www.euramet.org/project-NEW01	
Contact	Ian Gilmore (NPL)	ian.gilmore@npl.co.uk

Reliable imaging at the nanoscale

Identifying cancer proteins from biopsies, or pollutant chemicals in air samples are just two uses of Raman spectroscopy. This technique measures the light scattered by samples to identify their constituent molecules. Improved calibration methods are needed to enable concentrations of molecules to be accurately determined in healthcare, nanotechnology and forensic science.

The EMRP project **Metrology for Raman Spectroscopy** developed measurement techniques and reference materials to provide a reliable calibration chain for Raman spectroscopy measurements. It also developed modelling methods and real-time 3D imaging techniques that now enable evaluation of the dynamic processes occurring in living cells.

The project:

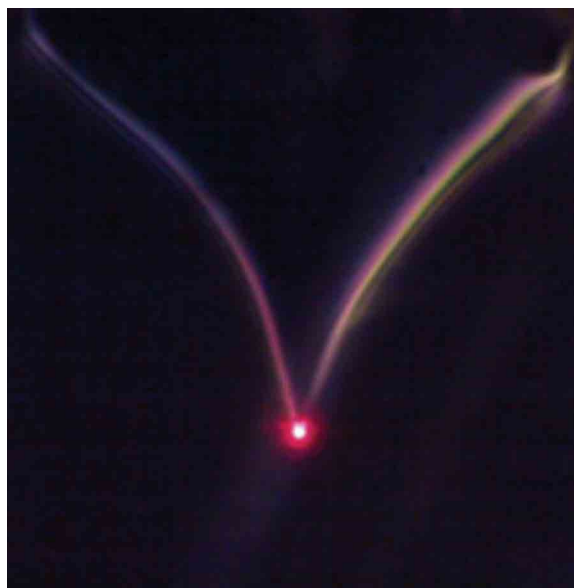
- **Developed a standardised Raman spectroscopy depth profiling procedure** based on reference materials, and used it to achieve the required sensitivity to detect very low target biomolecule concentrations in samples.
- **Developed a stimulated 3D Raman scattering microscope** and associated software capable of rapidly determining the concentration of molecules, such as in DNA, collagen, fat, and cytoplasm, in label-free bio samples from video images.
- **Established a method (using single-walled nanotubes) for measuring sub-nanometre dimensions of Raman tips.** This is important for precisely determining 2D distance measurements made in tip-enhanced Raman spectroscopy.

The methods developed in this project have made Raman spectroscopy a very attractive technique for label-free quantitative imaging of molecules in their native environments, and work has already begun to adapt Raman spectroscopy to new applications.

A video-rate Raman imaging microscope developed during this project allows real-time images to be produced of chemical and biological processes in molecules within living cells. For instance, this capability is being used to cost effectively investigate UV damage caused to surrogate skin samples during assessments of the stability of a sun cream formulation, and also in studies of drug uptake into living cells.

One of the greatest barriers to more widespread adoption of precise tip-enhanced Raman spectroscopy is the production of reliable gold coated tips for use. The project evaluated gold-plated tips and successfully implemented an improved production method that now produces quality tips in greater numbers than previously possible. Using tip-enhanced Raman spectroscopy surface structure resolution is now possible below 100nm, enabling generation of 3D information from sub-surface measurements.

Project developments will pave the way for Raman spectroscopy to be used in new research areas such as the identification of microbes in soil samples and the exploration of applying nanotechnology in biological systems.



More information is available at	NEW02: Metrology for Raman Spectroscopy (RAMAN) http://www.euramet.org/project-NEW02	
Contact	Ali Rae (NPL)	ali.rae@npl.co.uk

Technology for advanced materials

Measurement challenges

Both businesses and consumers are demanding increasing complexity, speed and performance from ever smaller electronic devices. To achieve this nanoscale wires, sheets, and tubes are being developed that are revolutionising how materials are used in new products and applications. Nanoelectro mechanical systems (NEMS) are a key disruptive technology that have potential for using graphene or novel piezoelectric materials in next generation computing and communications.

Introducing nanoscale components into new products relies on understanding the properties of the materials used in their construction at or below the nanoscale. Atomic interactions, negligible in bulk materials, now have far greater importance and need to be carefully considered. As the dimensions of devices shrink new precise measurement technologies and systematic approaches are needed to characterise nanomaterial properties to speed-up reliable product development and routes to market.

EMRP research has supported projects that address:

- The development of precise NEMS based nanoscale measurement methods for evaluating the physical properties of graphene as an initial step towards its uptake in applications such as security surveillance or ultra-sensitive bio sensing.
- The extension of SI traceability to existing Atomic Force Microscopy (AFM) methods and the development of new and improved new measurement methods for determining nano-object strength – important for ensuring the in-service performance of miniaturised components.
- The development of methods for characterising piezoelectric material properties at high temperatures to enable their use in challenging environments, such as power plant sensing.



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Key technical achievements

Characterising graphene for nanodevices

Devices are increasing in complexity, speed, and performance, while reducing in size. Nanoelectro mechanical systems (NEMS) are devices that integrate electrical and mechanical functionality at the nanoscale. These systems have the potential to provide solutions to a range of technological problems in electronics and computing, and also have potential for use in sensing applications. Graphene and piezoelectric materials have great promise for use in NEMS devices, but new preparation methods and measurement approaches are needed to determine material properties.

The EMRP project **Metrology with/for NEMS** investigated methods for characterising the electrical properties of NEMS devices and developed a new technique for manufacturing graphene to enable its use in new applications.

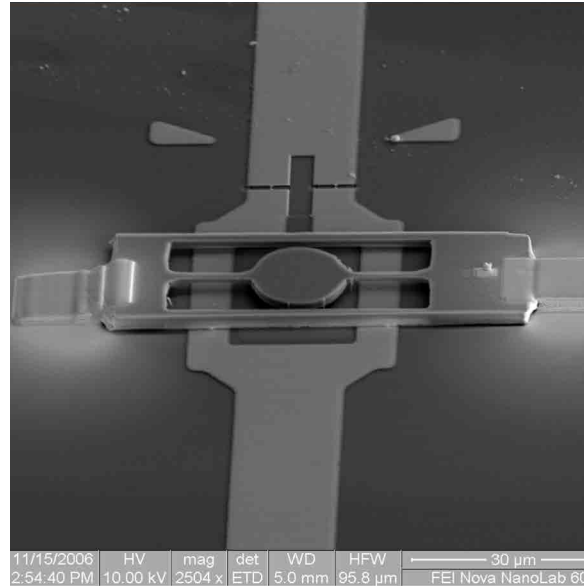
The project:

- **Developed techniques for growing and transferring graphene onto silicon support structures** to form combined silicon and graphene NEMS.
- **Established a non-contact method for the measurement of graphene resistance** and used it to demonstrate that silicon and graphene NEMS are suitable for low frequency security surveillance and telecommunication applications.
- **Developed near-field microwave systems** to simultaneously excite and detect oscillations from a mechanical NEMS resonator. These signals form the basis for new calibration methods and transfer standards for characterising periodically moving samples.
- **Developed a dynamic cooling technique** using near-field microwave methods and graphene NEMS resonators, reducing the effects of thermal noise on NEMS device performance during force and mass measurements.

This project developed sensors based on NEMS technologies to provide traceable nanoscale measurements for the characterisation of material properties and other novel sensors at the micro or nanoscale. Industrial users are interested in taking up the project's non-contact method for determining graphene sheet resistance. This method also has potential for extension to other 2D material electrical properties.

The enhanced sensor capabilities developed in the project **Metrology with/for NEMS** will further advance NEMS-based sensor technology into areas such as security surveillance and medical biosensors through the development of smaller and more sensitive sensors and detectors. In addition, NEMS sensors have potential for use in exciting new applications such as ultra-sensitive biodetectors able to distinguish between different types of cancerous cells by determining a tumour's mass. These developments have been made possible by the project's work on dynamic cooling to reduce the effects of thermal noise on NEMS.

The project's method for manufacturing graphene on a silicon substrate and electrical characterisation techniques for NEMS is an essential steps towards new types of nano-devices and novel future NEMS applications.

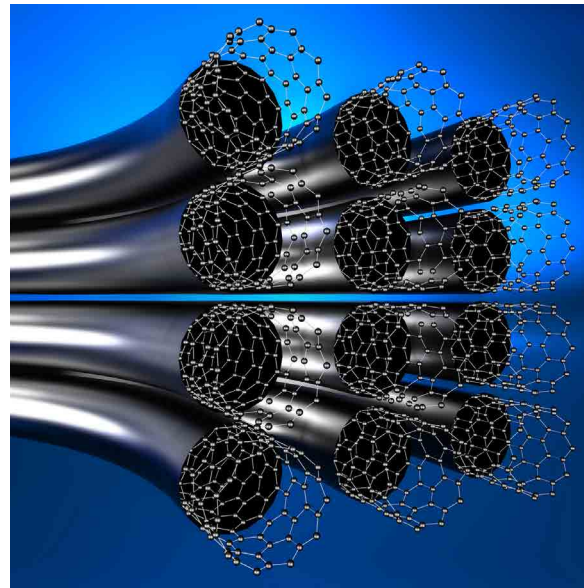


More information is available at	NEW08: Metrology with/for NEMS (MetNEMS) http://www.euramet.org/project-New08
Contact	Ling Hao (NPL) ling.hao@npl.co.uk

Determining nanoscale material properties

Nano-objects, such as wires, sheets, cubes and tubes, have the potential to revolutionise the performance of materials and structures. However, their small scale means their behaviour is fundamentally different to that of bulk materials. Material testing techniques based on making large indentations in material surfaces are commonplace for bulk materials but few nanoscale test methods are available. Atomic Force Microscopy (AFM) can produce indentations and has potential for nano-testing, but it lacks the reliability needed to produce comparable results. Increasing the number of different material property measurement methods and ensuring comparability of results is key to reliable use of nano-objects in new products.

The EMRP project **Traceable measurement of mechanical properties of nano-objects** developed reference materials and used these to characterise nanoscale material testing methods increasing result reliability and comparability.



The project:

- **Developed and characterised reference nanomaterials** consisting of arrays of pillars, cantilevers, and nanoparticles for evaluating the performance of nanomaterial test methods.
- **Improved a widely used method** for the calibration of AFM cantilevers used in nanomaterial testing and achieved far lower measurement uncertainties for this type of calibration than previously possible.
- **Developed a new method for determining the strength of nanowires based on scanning electron microscopy.** This now forms the basis of a new measurement service enabling nano-object preparation and strength assessment inside a single instrument.
- **Developed computer simulation models for nano-indentation experiments** and used these to predict how lattice dislocations and crystal orientation affects material deformation mechanisms.

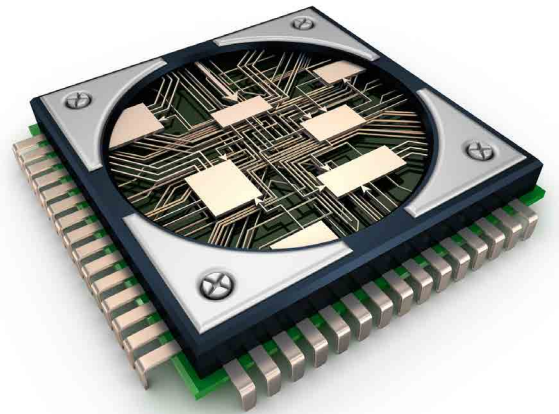
An understanding of the properties of new materials provides a catalyst for their introduction into innovative applications and has the potential to provide cost effective solutions to existing or emerging technological challenges. This EMRP project improved the rigour with which nanomaterial properties can be determined. Atomic Force Microscopy (AFM) measurements can now comply with the ISO standard on instrumented indentation testing enabling this type of measurement to be readily compared with other nano and microscale test methods.

Comparing the performance of reference and test AFM probes is now possible using an easy to use micro device developed in the project, whilst a new calibration capability for certifying AFM probe performance is contributing to greater material testing reliability. This project has increased the methods available for determining reliable nano-object material properties. These are required to support the integration of nano-objects into new products and technologies.

More information is available at	NEW05: Traceable measurement of mechanical properties of nano-objects (MechProNo) http://www.euramet.org/project-NEW05	
Contact	Uwe Brand (PTB)	uwe.brand@ptb.de

Measurements for functional materials

Both new functional materials, able to withstand high temperatures without losing performance, and electronic cooling technologies have the potential to increase efficiency or reduce energy consumption in a diverse range of applications. From power generation to electronic devices the use of materials able to convert mechanical movement into thermal or electrical energy and vice versa has the potential to extend functional capabilities. However, greater understanding of material properties and attributes is required before these novel materials can be reliably incorporated into new products.



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The EMRP project **Metrology of electrothermal coupling for new functional materials technology** developed the measurement capability required to improve thermal, mechanical, and electrical property characterisation for functional materials at high temperatures (up to 1000 °C) or in high electric fields.

The project:

- **Developed instruments for measuring thermoelastic coupling to 1000 °C**, so providing the devices needed to make reliable measurements of piezoelectric material performance.
- **Developed methods for measuring the electro-caloric effect in bulk (thick film) materials** in air and under vacuum, and used these to characterise films for potential use in removing surplus heat generated in microelectronic devices.
- **Upgraded National Metrology Institutes (NMIs) test facilities to enable the accurate measurement of thermophysical properties of thermoelastic or thermoelectric materials** and used these to develop validated models for heat flow in thin films with potential for use in future electronic devices.
- **Developed a piezoelectric reference material capable of performing reliably at temperatures up to 380 °C**. This enables the determination of measurement instrument uncertainties and has potential use in a wide range of industrial applications.
- **Validated models for nanoscale heat-flow in functional materials**. These provide product designers with information on how functional materials will perform under in-service conditions.

The performance of piezoelectric materials can now be characterised at higher temperatures than previously possible. This extends their potential sensing use to the temperatures encountered in power plant integrity monitoring. However, further research is required to increase measurement method reliability for the more extensive temperature range encountered in many sensing applications. This collaborative project has enabled European NMIs to gain an appreciation of the measurement challenges associated with heat transfer through thin films, and the implementation of electrical cooling.

More information is available at	NEW09: Metrology of electrothermal coupling for new functional materials technology (METCO) http://www.euramet.org/project-NEW09	
Contact	MSU Office, Euramet	msu@npl.co.uk

Technology for a safe and secure world

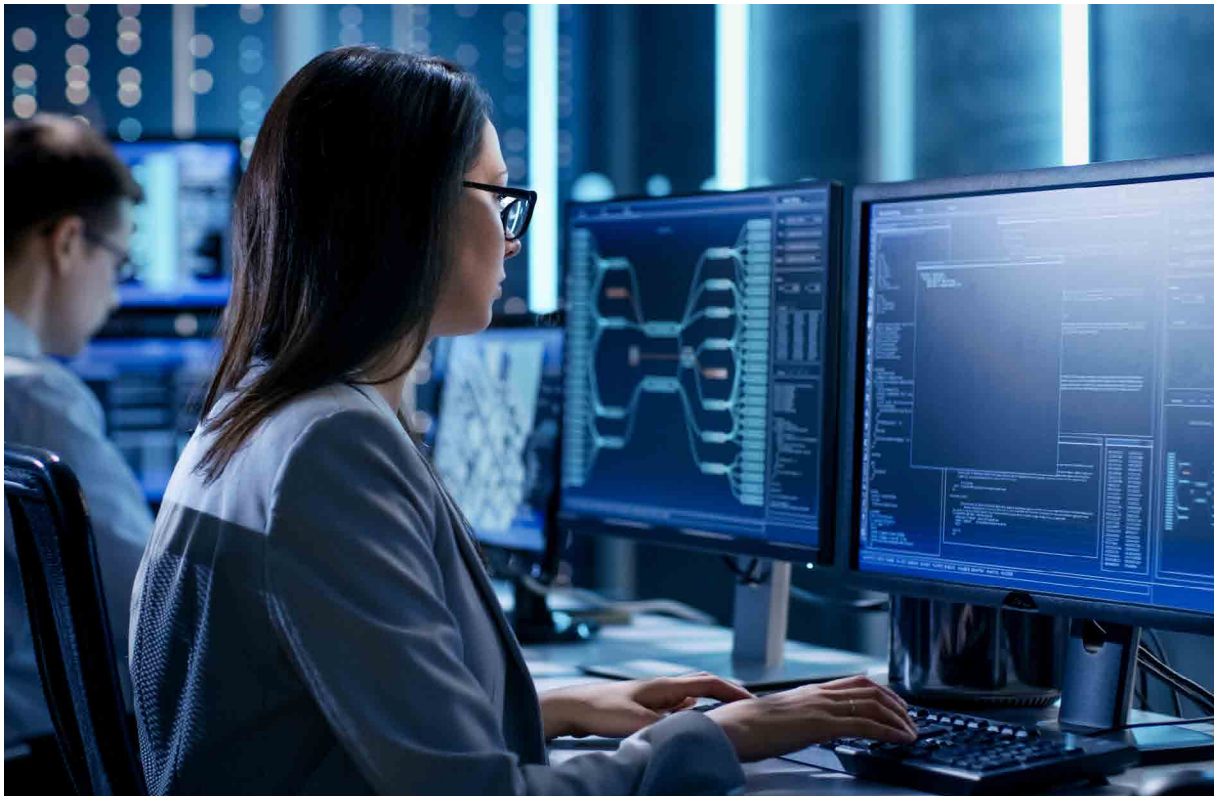
Measurement challenges

The introduction of new technologies and new ways to more reliably analyse data both have great potential to increase the safety and security of our world, but could also expose us to new sources of risk. Two recent innovations that benefit society are improvements to airport security scanning for illicit substances and increased accuracy for complex building safety risk assessments. However, innovations sometimes involve risks. For example, increased use of nanoparticles in cosmetic products and clothing may create new ways for nanoparticles to enter and travel inside the body.

Understanding how risks propagate, whether inside a building or inside our bodies, relies on accurate information based on a reliable measurement system. Relating measurement results to risks often involves the use of complex and extensive software, which in turn is reaching the point of being impossible to rigorously check manually. New strategies and approaches are needed to address these issues.

EMRP research has supported projects that address:

- The development of methods to characterise physical, chemical and optical properties of nanoparticles in biological environments – a prerequisite for reliable toxicological studies.
- The development of robust calibration methods and validated modelling approaches to show that terahertz radiation is safe for use in security surveillance.
- The development of new approaches for establishing confidence in complex measurement analyses and simulations used for risk assessments or conformity testing, demonstrated by appropriate case studies to facilitate user uptake.
- The development of a new approach to complex software validation using an internet based system to give both programmers and their customers confidence that software provides the correct results every time.



Key technical achievements

Nanoparticles in biological environments

Nanoparticles are used in products, from medical devices to cosmetics, in a global market worth €9.6 billion, but concerns are growing over their safety. Nanomaterials pose a potential risk to human health as their properties change during interactions with biological systems. Accurate measurement methods are needed to determine the size, concentration and toxicity of ingested or inhaled nanoparticles.

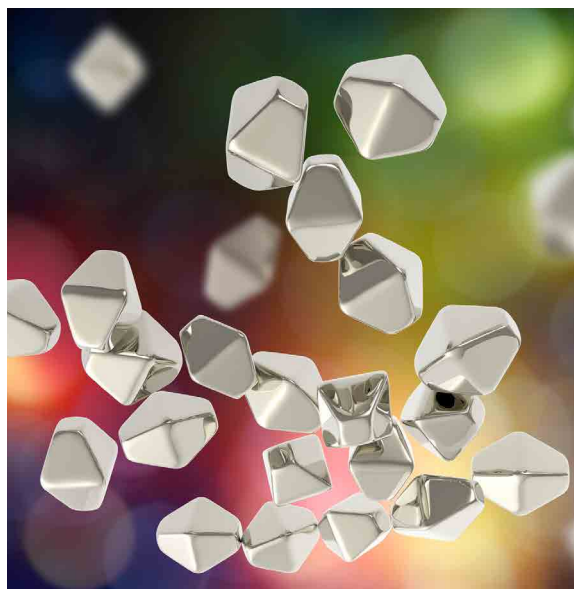
The EMRP project **Chemical and optical characterisation of nanomaterials in biological systems** developed methods to characterise the physical, chemical and optical properties of nanoparticles in biological environments – a prerequisite for reliable toxicological studies.

The project:

- **Compared the performance of a wide variety of physical and chemical methods for analysing nanoparticles** using characterised materials in both neutral solutions and biological serum.
- **Developed and validated a method for future chemical characterisation of reference nanomaterials** by combining existing methods for particle size separation and mass spectrometry.
- **Developed a validated spectroscopy procedure for determining the light from fluorescently labelled nanomaterials** in aqueous media and biological serum. This is important for determining nanomaterial characteristics such as the number of proteins adsorbed on to their surfaces in biological systems.
- **Developed a procedure for validating biotechnology laboratory processes based on the fluorescent labelling of antibodies.** This is important as fluorescent labelling is used to indicate the presence, and quantity of target molecules in a bio-sample.

The project addressed the need for accurate physical and chemical measurements in biological systems by validating routinely used laboratory techniques using well characterised nanoparticles in both buffer solution and biological serum. Testing highlighted the benefits of initial size segregation on particle sizing measurement accuracy. By adopting a two stage approach, it becomes easier to detect the 'rare' particles of interest in toxicity studies or disease diagnosis.

As a result of this project, important first steps have been made in understanding how molecular properties are affected by nanoparticle labelling. This lays the foundations for increasing the reliable use of nanoparticles in medicine and healthcare. The validated methods and protocols developed in the project will play a key role in ensuring the safe use of nanomaterials. Organisations developing nano-biotechnology and nanomedicine can now have increased confidence in the accuracy of measurements for toxicology studies and risk assessments. Research into the measurements needed to support the safe use of nanoparticles in new applications continues in the EMPIR project **Metrology for innovative nanoparticles**.



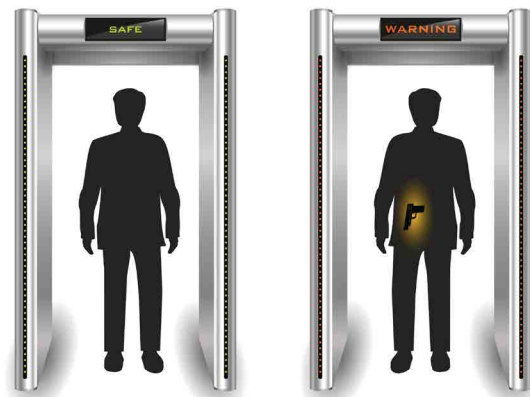
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More information is available at	NEW03: Chemical and optical characterisation of nanomaterials in biological systems (Nano ChOp) http://www.euramet.org/project-NEW03 Follow on project: Metrology for innovative nanoparticles 14IND12
Contact	Heidi Goenaga Infante (LGC) Heidi.Goenaga-Infante@LGCGroup.com

Microwave and terahertz security scanning

Terahertz radiation is an ideal technology for security scanning of the general public as it does not involve cell-damaging ionising particles. Scanning devices operating at these sub-millimetre wavelengths can quickly and non-invasively image through clothing, to identify concealed weapons, explosives, drugs and other contraband items. Assessing safety risks associated with terahertz radiation to both device operators and the scanned public requires accurate measurements of the devices power. Techniques are needed to calibrate important performance parameters, such as frequency, amplitude and power against SI definitions.

The EMRP project **Microwave and terahertz metrology for homeland security** developed robust calibration methods and a validated modelling approach that have shown that terahertz radiation is safe for use in security surveillance.



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The project:

- **Established improved methods for calibrating the three most common terahertz spectrometer types to SI units.**
- **Developed a range of complementary reference materials for reliably verifying and comparing microwave and terahertz spectrometer performance.**
- **Provided uncertainty analysis for the absorption and reflection coefficients measured in terahertz spectrometers,** enabling these to be assessed for the first time. This provides the necessary measurement accuracy to enable results from different spectrometers to be reliably compared.
- **Validated a modelling approach for the propagation of electromagnetic waves through skin layers,** therefore estimating the amount of heating in human skin from microwave and terahertz radiation.

This project has made fundamental contributions to improving the accuracy and reliability of microwave and terahertz calibrations with robust links to SI units. The project developed two independent SI traceability routes for terahertz radiation. One is based on the calibration of a new pyroelectric detector for use in transferring calibrations to industrial terahertz users. The other traceability route is based on a transportable table-top sized room-temperature radiometer that provides a direct calibration to the SI at an industrial location. These new capabilities will enable more effective design and testing of high-performance security devices, and will also ensure that the devices can be used safely.

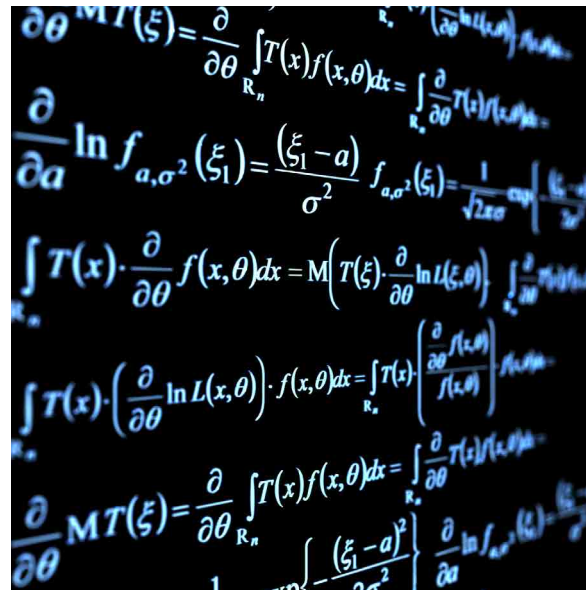
Compliance with the European “Physical Agents Directive” exposure limits for personnel working with high-frequency radiation can now be demonstrated using project developed techniques and models. These provide a way for manufacturers to prove that terahertz devices are safe for both operators and the public, and will assist in the introduction of new terahertz technologies.

More information is available at	NEW07: Microwave and terahertz metrology for homeland security (THz Security) http://www.euramet.org/project-NEW07	
Contact	Thomas Kleine-Ostmann (PTB)	Thomas.kleine-ostmann@ptb.de

New approaches to complex risk assessment measurements

Measurement uncertainties are important in many applications, from ensuring precisely engineered components fit together to confirming batches of sensors meet specifications. Accurately determining the uncertainty or 'doubt' for a measured value is key to having confidence that a product meets specifications or safety criteria. New mathematical methods and statistical techniques are required to reliably extend uncertainty determination into complex areas, such as risk assessments for safety or determining smart meter testing strategies to ensure reliability.

The EMRP project **Novel mathematical & statistical approaches to uncertainty evaluation** developed new approaches for determining uncertainties in situations where complex simulations are combined. The project also developed smart sampling techniques to reduce extensive computational times without compromising accuracy.



The project:

- **Developed statistical methods for using probability to model measurement results and the associated uncertainties.** These were applied to the highly accurate digital polymerase chain reaction technique used for counting the quantity of specific DNA sequences in biological samples.
- **Developed smart sampling methods and computer models to more rapidly determine a reliable result for computationally expensive calculations** based on careful selection of input parameters, and reducing the number of model iterations required.
- **Extended existing approaches on measurement uncertainty to conformity assessment and decision making** by linking the determination of calibration frequency to risk assessment.

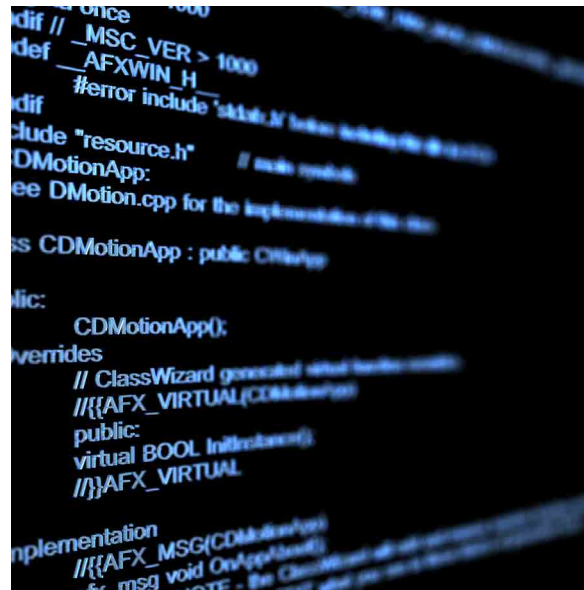
The project developed new approaches to uncertainty determination and increased the number of worked examples available, so that the methods can be applied in similar situations by scientists and engineers. The worked examples will be included in future revisions of the *Guide to the Expression of Uncertainty in Measurement* (GUM), and its supplements. The project's best practice guidance provides examples for some frequently encountered situations where models are used to determine measurement parameters. Fluid flow calibrations for installed flow meters and biochemistry immunoassays where very small concentrations of hormones or drugs require accurate detection are two examples that now have improved uncertainty determination methods. Other areas with new worked uncertainty examples based on probability include computationally expensive processes for conformity testing (important in large batch production processes, like the roll-out of smart energy meters across the EU) and risk assessments for fire engineering safety.

More information is available at	NEW04: Novel mathematical & statistical approaches to uncertainty evaluation (Uncertainty) http://www.euramet.org/project-NEW04
Contact	Markus Bär (PTB) markus.baer@ptb.de

Improving confidence in new software

Complex software and statistical modelling dominates all aspects of business and much of our daily lives. It is nearly impossible to confirm that complex computer programs running numerous statistical analyses, with multiple subroutines, are generating the correct answers. Small errors in the code can cause problems that are very hard to spot. New and improved independent methods are needed to give both software providers and their customers confidence that the supplied code computes the correct result every time.

The EMRP project **Traceability for computationally-intensive metrology** developed the *TraCIM SVS* – a new approach to software validation. This online system provides users with reference test data, and compares the results generated with those from an ‘ideal’ data set, leading to a software validation test report.



The project:

- **Developed a web-based searchable database containing standardised mathematical language to specific calculations** needed for coordinate measurement machine software.
- **Developed an assessment method for evaluating the performance of user software** based on ‘ideal’ reference input data and corresponding ‘ideal’ reference results.
- **Developed a method to combine multiple data set uncertainties to generate a single ‘figure of merit’ for the software under test.** This can be used as an estimation of the software uncertainty component during the calculation of overall combined measurement uncertainties.
- **Combined the components of the *TraCIM Software Verification System (TraCIM SVS)* into a web-based ICT system** that enables users to have software tested and leads to a validated independent software test report.

Confidence in the ongoing performance of quality assurance software and demonstrating that new software produces accurate results are important in many diverse applications, from safety assessment of structures such as fuel storage tanks to quality control assessments of intricately shaped 3D components vital for precision engineering. Results generated by complex software can contain errors that programmers find difficult to spot. These can include rounding approximations that are assumed to be insignificant, or iterative analysis processes that may stop too soon – leading to non-optimised convergence values.

The project’s *TraCIM SVS* has been used to verify applications where safety critical or very extensive data sets require complex modelling and multistage computer programs to generate results. A follow-on EMPIR Support for Impact project **Validation of software development and analysis tools** is extending the *TraCIM SVS* for use with the popular LabVIEW™ software tool. This will enable greater access to the project’s software validation tool and broaden its uptake by industries with complex data and software manipulation requirements.

More information is available at	NEW06: Traceability for computationally-intensive metrology (TraCIM) http://www.euramet.org/project-NEW06 Follow on EMPIR project: Validation of software development and analysis tools (15SIP06)	
Contact	Alistair Forbes (NPL)	alistair.forbes@npl.co.uk

Focus on impact

All EMRP projects engage widely with the user communities who can benefit from the research. The measurement capabilities and analysis approaches developed in the EMRP New Technology theme support improved characterisation of new nanoscale materials and components for future innovations in a broad range of industries. New approaches to statistical analysis and software evaluation are helping improve risk assessments and batch conformance testing whilst linking measurement results to the body's interaction with terahertz radiation or nanoparticles will increase safety assessments for new products.

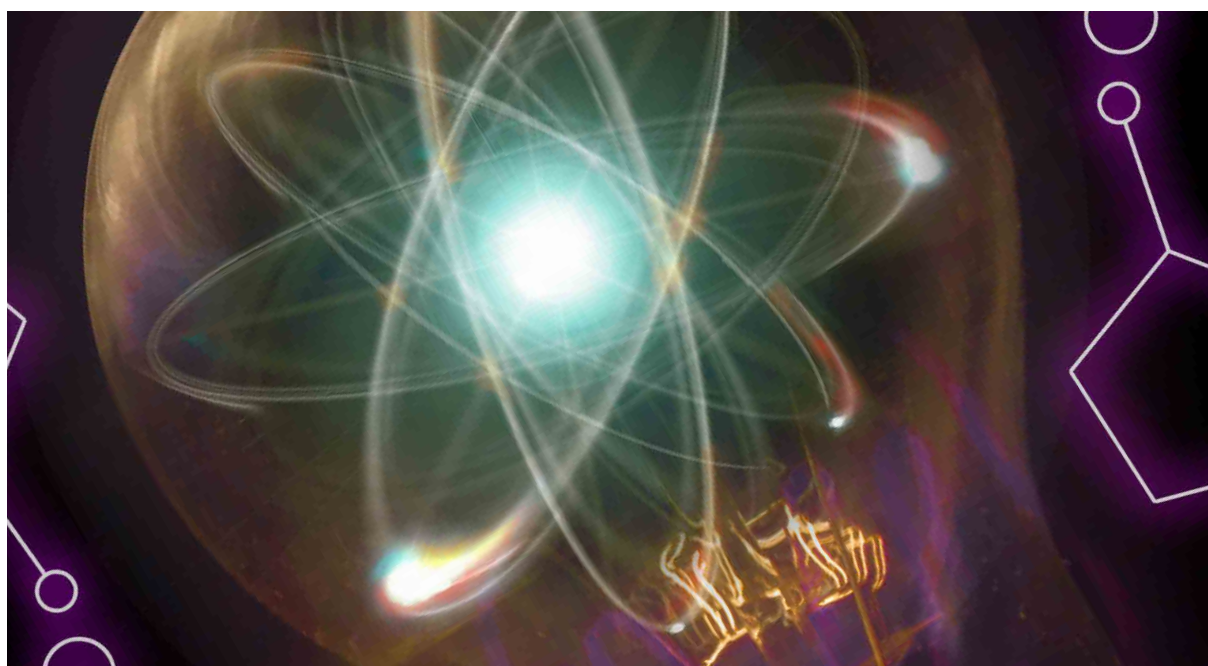
Innovation in 3D analysis

Global pharmaceutical company, GSK, is improving its understanding of how drugs are delivered to target cells, thanks to developments in the EMRP project **Traceable characterisation of nanostructured devices (TReND)**, and the application of these in an innovative mass spectrometer developed by the UK government funded *3D OrbiSIMS* project.

The EMRP *TReND* project developed a 'universal equation' that allows the sputtering yield, or ion production rate, to be calculated for different ion beams during argon cluster sputtering mass spectrometry. The *3D OrbiSIMS* project applied this to develop a powerful new mass spectrometry instrument for high-resolution imaging of organic materials. This instrument, marketed by ION-TOF GmbH, enables unprecedented high-spatial resolution imaging with the increased mass accuracy essential for confidence in molecule identification.

GSK has used a *3D OrbiSIM* instrument to identify where drugs go at the cellular level. This helps answer long-standing questions on whether drug concentrations are sufficient to deliver a therapeutic effect, or are building up in organs or cells causing toxicity. Spotting inconsistent drug delivery early may help explain reduced efficacies and reduce costly late-stage development failures.

The EMRP *TReND* project provides a good example of how measurement developments for one industrial sector, the rapidly evolving organic electronics industry, can have far reaching implications in a completely different industry.



Extending nano-accuracy

The chemical and optical resolution of layers and structures at the molecular level is important in pharmaceutical, microelectronics and semiconductor research. Raman spectroscopy's ability to resolve molecular sized features relies on using sharp probe tips with extremely smooth, thin gold coating. Producing these with the required quality is difficult. This problem has limited Raman spectroscopy reliability and reduced its practical application. The Natural and Medical Sciences Institute at the University of Tübingen, Germany, can now manufacture reproducible Raman spectroscopy tips in their clean room facility using a new process developed in the EMRP project **Metrology for Raman Spectroscopy**.

This new process, based on template stripping, has significantly increased the number of usable tips in each manufacturing batch, making commercial production possible. Raman spectroscopy can now be more widely used as a powerful molecular analysis technique as reproducible tip manufacturing enables improved accuracy in comparing biopsy results from different clinics.

Rapid mapping of cancer

In a recent clinical trial, King's College London demonstrated accurate cancer diagnosis by rapid analysis of Raman tissue images using improved software. Participation in the EMRP project **Metrology for Raman Spectroscopy** enabled King's to upgrade their analysis software to make Raman spectroscopy imaging viable as a routine diagnostic tool.

The software developed by King's College London uses cluster analysis to reduce the time it takes to analyse the millions of spectra generated in a single Raman image, from over 24 hours to just 30 mins. This iterative process generates rapid and more accurate results than previous methods which rely on experts to analyse data.

The speed and reliability of King's College London software was established during a clinical trial which compared methods for identifying cancerous tissue in patient samples. The results of this trial opened the way for Raman spectroscopy to be used for disease diagnosis.



Innovation in mechanical testing

Nanowires have the potential to form the current carrying connections in microsensors and microactuators but ensuring the wires are strong enough to perform throughout their in-service life relies on material testing. Researchers at Koç University, Turkey, were the first to benefit from an innovative measurement service based on nano-strength testing developed during the EMRP project **Traceable measurement of mechanical properties of nano-objects**.

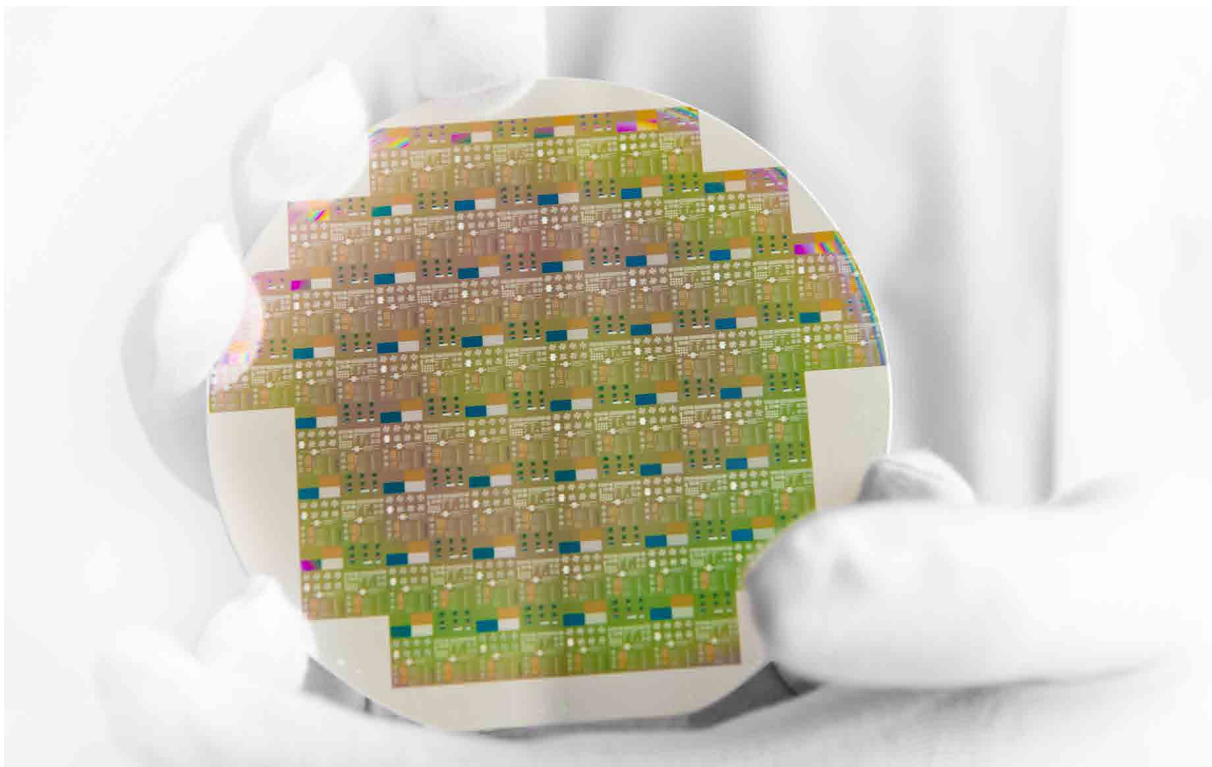
The service uses a new instrument combination to select material for analysis, prepare it, and then test them inside the vacuum chamber of a scanning electron microscope. A focused ion beam cuts the test piece from the selected area and a micro-manipulator positions it for mechanical testing. Loads are then applied, inducing distortion of the test piece to generate information on how hard or how much load the material can absorb before it is permanently deformed. Both are important material properties required by designers to ensure novel products will survive the rigours of use.

New material testing techniques such as this act as a catalyst for adopting novel materials into innovative applications and can also provide cost-effective solutions to existing or emerging technological challenges, so strengthening the competitiveness of European industries.

Standardisation for AFM material testing

Understanding the performance of nanowires, nanocoatings, and wafer surfaces used in the semiconductor industry relies on highly accurate and reliable mechanical strength and hardness measurements. The EMRP project **Traceable measurement of mechanical properties of nano-objects** made important contributions to the ISO 11775 and ISO 14577 standards for material testing by providing new meander type reference springs for calibrations of load applied through Atomic Force Microscopy (AFM) cantilevers. With these springs, available from the CiS GmbH, an improved AFM force uncertainty of 7% is achievable.

The ISO standards now allow nanomaterial testing to be performed using an atomic force microscope, provided loads are calibrated and tips meet shape requirements. With the increased robustness of AFM nanoscale material property measurements, results can now be compared with confidence – supporting the introduction of nano-objects and coatings in new applications.



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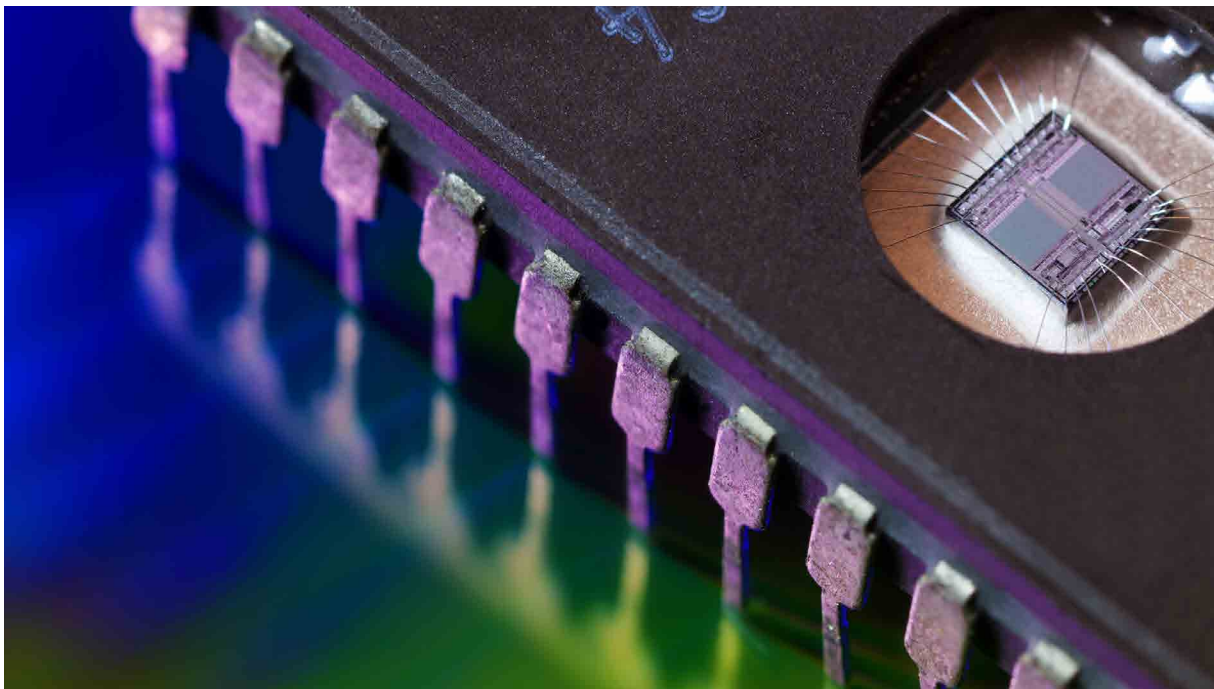
Nanomaterial properties

NanoWorld AG, a leading manufacturer of high quality AFM probe tips, can now guarantee the stiffness uncertainty of the cantilevers they supply, as a result of having the performance of their 'gold standard' cantilevers confirmed.

In nanomaterial strength testing, the AFM tip applies a known load to the test piece and this is determined by accurately measuring the tip's movement. The EMRP project **Traceable measurement of mechanical properties of nano-objects** increased the accuracy of AFM force calibrations by developing new calibration methods for AFM cantilever stiffness and by improving the well-known Thermal Vibration method in cooperation with NanoWorld.

NanoWorld now has improved calibration for all the AFM tips it produces enabling customers to confidently extend AFM use to nanomaterial strength and hardness testing for the first time. Over the last few years, the popularity of AFM based nanomechanical characterisation methods has increased and NanoWorld has noticed rising demands for their cantilever probes with well-known force constants. Therefore, they are planning to broaden their AFM product range by equipment investment and further research that will build on the results of this project.

Greatly improving the robustness of AFM measurements for determining nanomaterial properties will help smooth the introduction of these materials into exciting new applications, such as faster, smaller, flexible electronics.



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Faster measurements for chip production

JCMWave is a software provider developing fast programmes for analysing extensive scatterometry measurement data sets to enable this technique to be used for quality control in silicon chip and semiconductor manufacturing. New methods for smart data sampling and ways to combine existing knowledge based on previous measurements were developed in the EMRP project **Novel mathematical and statistical approaches to uncertainty evaluation**. These form the foundations for JCMWave's new software.

Scatterometry uses light scattered from features on a surface to accurately determine dimensions. However, measurements are time consuming due to the large amounts of data generated and necessary complexity of the computer programs performing the analyses. JCMWave is developing a new, faster suite of software programs to speed-up the calculation of results without compromising accuracy. Reducing the computational time for these measurement calculations will open the way for scatterometry to be used for process quality control. This is an important step as process measurements become more difficult as semiconductor size reduces.

Trusting complex software

Mitutoyo, a leading manufacturer of precision measuring equipment, has been one of the first to have its coordinate measuring machine (CMM) software independently verified using a new internet based system. The *TraCIM Software Verification System (TraCIM SVS)* developed by the EMRP project **Traceability for computationally-intensive metrology** enables software to receive a test report confirming performance.

The project developed a range of standardised data sets which can be used to test the validity of software used to process measurement data, just as standardised objects are used to verify that measurement tools are measuring accurately. The resulting *TraCIM SVS* evaluates and compares the results from the software being tested to ideal values, producing a test report on the degree of agreement.

Mitutoyo used the system to validate its CMM software, confirming it performed as advertised. The test report can now be used by Mitutoyo to demonstrate to customers that its software is fit for purpose. Such demonstrable independent validation provides much needed confidence for manufacturers that their products provide accurate results and match specification, an increasingly important issue as more complex products require ever greater precision and smaller tolerances.

Validating 3D software modelling

Technodigit, part of Hexagon AB, specialists in surface 3D measurement software, has checked the accuracy of their 3DReshaper software used in safety critical testing, such as the periodic integrity testing of fuel storage tanks at ports and airports. The software has been validated using a new process developed in the EMRP project **Traceability for computationally-intensive metrology**. The *TraCIM SVS* evaluates and compares the results from the software under test to idealised values, providing a performance certification process to confirm that simulation software runs properly and does not produce errors.

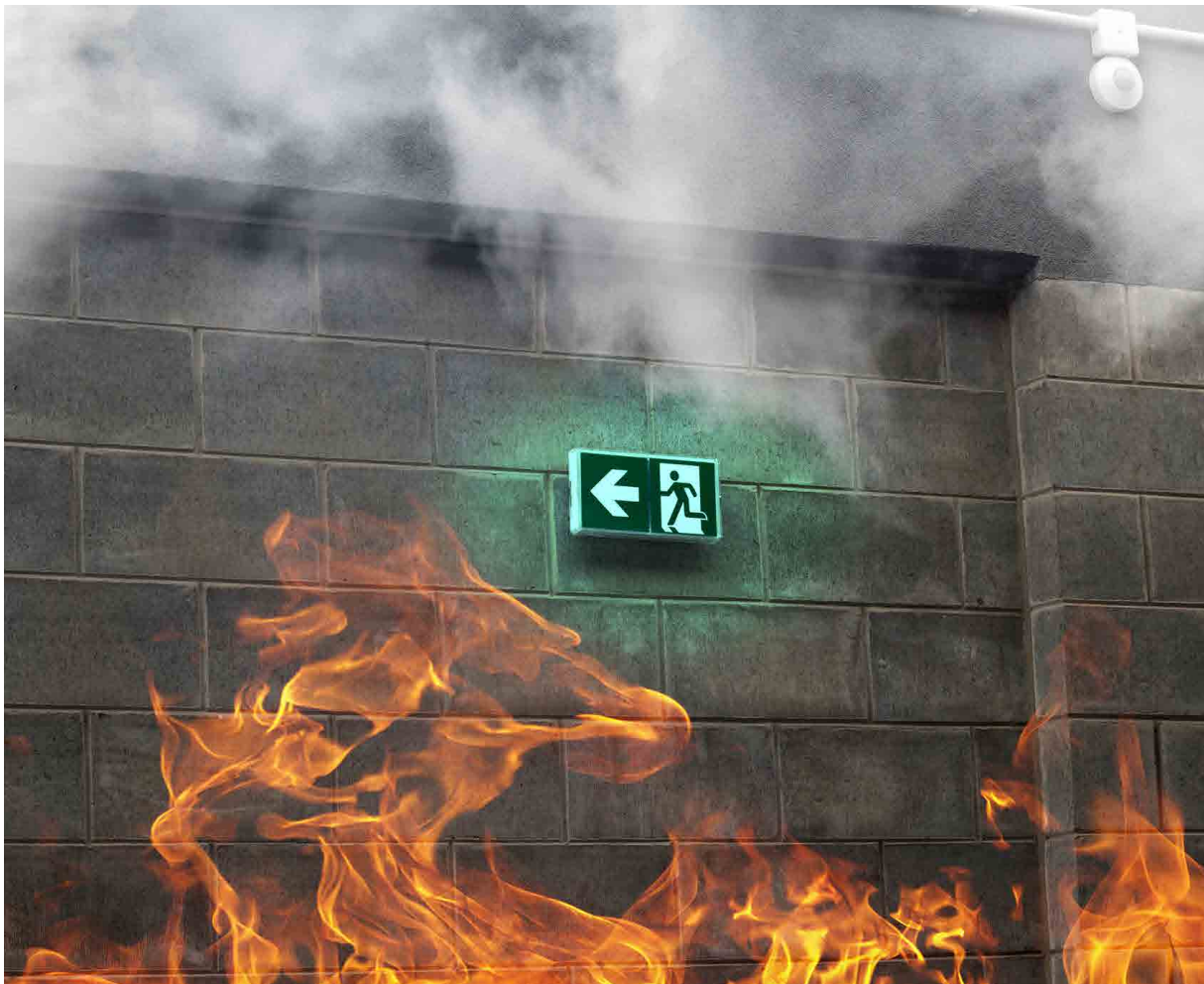
The project's *TraCIM SVS* was used to validate Technodigit's 3DReshaper software, which can automatically generate storage tank test reports in two hours with minimal human intervention, replacing the existing laborious two day process. Technodigit's customers can now have confidence in their results based on the *TraCIM SVS* validation of the 3DReshaper software. More data sets are currently being added to enable the system's extension to other measurement areas that use complex simulations.



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More efficient statistical tools for fire safety

Fire safety simulations help ensure public safety in shopping centres and theatres, and are an essential part of building safety regulations. These simulations use virtual sensors to monitor critical temperatures and contain hundreds of parameters on how fires start and spread. Individual simulations take over a month to perform, with multiple runs needed to assess a building's resistance to fire. The EMRP project **Novel mathematical and statistical approaches to uncertainty evaluation** constructed a database using data from different fire scenarios to improve the prediction of how fires evolve. This was then used to develop statistical methods that both reduce computational time and the processing capacity needed to generate a fire simulation result. Additional funding is enabling ongoing developments to further reduce simulation time by determining the most critical fire safety parameters, helping speed-up probability predictions of a particular outcome. Ultimately, this will reduce the time taken for multiple fire simulations to assess the conformity of buildings for fire safety. This will enable a more comprehensive fire certification process.



Accuracy for terahertz radiation

Greater confidence in terahertz (THz) frequency calibrations and the demonstration that terahertz is safe, is supporting the introduction of technologies for identifying hidden weapons so enhancing passenger safety. New calibration facilities and models demonstrating that terahertz radiation is safe for use were developed in the EMRP project **Microwave and terahertz metrology for homeland security**.

A technique routinely used for determining the power of solar radiation in climate studies has been adapted by the World Radiation Centre (Davos, Switzerland) for calibrating THz lasers at room temperature. The Davos team developed a table-top calibration chamber, by removing the need for bulky and expensive cryogenic cooling, that can perform independent THz laser calibrations at customer sites. Methods enabling robust calibrations of THz lasers and detectors are an essential first step in determining the safety of THz radiation to both security scanner operators and the travelling public, before its wider introduction for security purposes.

Safe airport scanners

SLT Sensor & Lasertechnik GmbH, who supply laser measurement products, developed a new type of terahertz radiation detector which will pave the way for adoption of terahertz technology to improve security and medical devices. SLT were one of the first to have a detector calibrated using the new terahertz calibration facility developed in the EMRP project, **Microwave and terahertz metrology for homeland security**. This facility enables manufacturers to have a terahertz laser's power accurately determined – an important requirement for demonstrating safety.

As a result of the project, SLT can now provide the first reliable method for transferring SI terahertz power calibrations to customers' instruments, enabling them to demonstrate that terahertz products meet specifications for laser power.

Terahertz lasers are finding innovative applications in more accurate body scanners at airports and non-destructive detection of material defects. The new SLT detector allows accurate measurements of lasers in the terahertz range and facilitates measurement comparability. As a result, reliable assessments of both operator and public safety to terahertz radiation exposure are now possible – giving manufacturers the confidence needed to develop new technologies based on terahertz radiation.



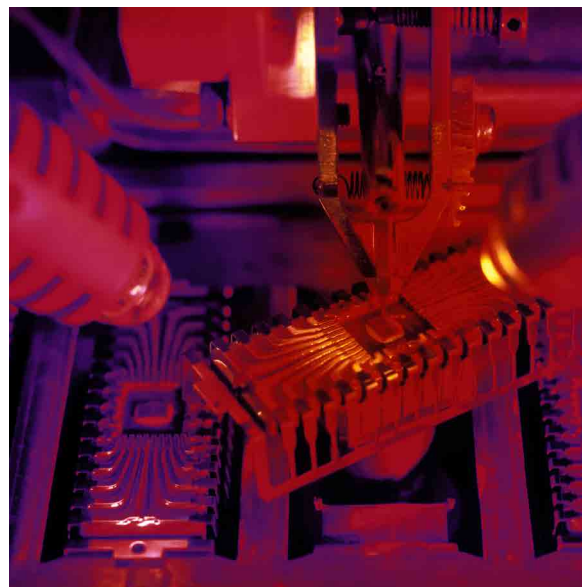
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Piezoelectric innovation

aixACCT, a leading manufacturer of instruments for measuring the electrical properties of materials, has improved the accuracy and operating temperature range of its aixPES Piezoelectric Evaluation system. New reference materials, developed by the EMRP project **Metrology of electrothermal coupling for new functional materials technology**, have enabled improvements which will drive innovation in piezoelectric materials research and increase their uptake into new products.

This project tested and characterised a new piezoelectric material, demonstrating its viability as a reference grade material suitable for validating the performance of material testing instruments at temperatures up to 380 °C. Using this material as well as project expertise, aixACCT was able to extend their aixPES system to higher temperatures than previously possible. Demonstrating instrument performance using the type of material it is made to test gives customers greater confidence in the test results used to compile material data sheets.

Accurate high temperature properties will lead to greater uptake of piezoelectric materials into advanced devices and sensors, bringing new and improved functionality to innovative new technologies.



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Installed sensing for power plants

IONIX, a university spinout commercialising innovative piezoelectric materials, has demonstrated the performance of its products at high temperatures and now can confidently produce material property data sheet needed to promote up take. Testing performed in the EMRP project **Metrology of electrothermal coupling for new functional materials technology** has made this possible, and is paving the way for IONIX's materials to be used in commercial sensors for integrity monitoring of power plant pipes.

Piezoelectric materials make ideal sensors. They produce electrical signals in response to sound or pressure waves, but traditional piezoelectric materials lose performance above 200 °C – a temperature frequently exceeded in power plant pipes.

IONIX has incorporated the characterised piezoelectric materials into a new line of ultrasonic sensors for use in extreme environments, based on their innovative HotSense technology. Independent verification of the properties of their piezoelectric material was vital for getting the sensors accepted for power plant monitoring – an industry where new technologies must demonstrate reliability. The HotSense prototypes are now undergoing extensive testing in order to obtain the necessary licence for power plant use. Once licensed, this new operational sensing capability should generate considerable power plant cost savings by enabling continual integrity monitoring and reducing the occurrence of unexpected component failures.



Smart meter reliability

Revision to Norwegian energy metering regulations are being considered based on reducing cost and risk to both consumers and suppliers from inaccurate smart meters. Statistical models using previous meter failure histories and incorporating cost and risk assessments have been developed in the EMRP project **Novel mathematical and statistical approaches to uncertainty evaluation** to improve electricity meter batch conformance testing.

The project looked at historical data from testing small numbers of electricity meters from large manufacturing batches, and developed models for predicting when installed meters were most likely to experience measurement problems. Costs of both replacing faulty meters after installation and the risks posed to both consumers and energy suppliers were incorporated into the models. This led to recommendations for a new meter inspection regime which would increase the likelihood of underperforming meters being spotted faster.

This is an example of how historic data can be used to generate accurate assessments of new products, and how risk assessment can be incorporated into statistical models to inform testing frequency for mass produced items. It illustrates how mathematical approaches can be combined and applied to assist in defining new and more rigorous testing regimes, and has potential applications in other areas where batch conformance testing including cost/benefit analyses are required.

Accuracy for installed flow meters

Optolution Messtechnik GmbH, a specialist in flow meter calibration, has upgraded its flow calibration system, allowing it to be used to calibrate flow in complex systems such as those present in District Heating Systems. Relating the calibration of a flow meter, made using ideal flow conditions, to its installed performance in the non-ideal flow produced by complex pipe systems is difficult. Using new software developed in the EMRP project **Novel mathematical and statistical approaches to uncertainty evaluation**, Optolution has now overcome this problem.

The project developed new statistical models to better estimate the effect of flow disturbance on meter measurements. Applying adapted versions of this model to its laser-based flow measurement system, Optolution can now determine the flow rate at any point across a pipe's bore. This can be related to the measurements made by the installed heating system's gauge, and so used as a calibration method.

Optolution is now applying for DAKKs accreditation, which will enable them to offer certified calibration services for installed flow meters, bringing improved accuracy to many fluid flow systems.



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Further information

More detailed information on outputs from the EMRP projects and the contact details for each project can be found at:

<https://www.euramet.org/emrp-health-si-newtech-2011>

Other EMRP and EMPIR projects can be found at:

<https://www.euramet.org/research-innovation/emrp/emrp-calls-and-projects/>

<https://www.euramet.org/research-innovation/research-empir/empir-calls-and-projects/>

Europe's National Measurement Institutes working together

The majority of European countries have a National Metrology Institute (NMI) that ensures national measurement standards are consistent and comparable to international standards. They also investigate new and improved ways to measure, in response to changing demands.

While traditional metrology stakeholders in manufacturing demand ever-increasing scope and greater accuracy, there is also a greater demand for accurate measurement in support of food safety, clinical medicine, and environmental quality, alongside a need to support emerging areas such as biotechnology and nanotechnology. This requires resources beyond the scope of most national metrology systems and therefore it makes sense for NMIs to significantly increase their level of collaboration. The European Association of National Metrology Institutes (EURAMET) is the body that coordinates collaborative activities in Europe.

EURAMET has implemented the European Metrology Research Programme (EMRP), a project programme organised by 23 NMIs and supported by the European Union, with a value of over €400 million. The EMRP facilitates the formation of joint research projects between different NMIs and other organisations, including businesses, industries, and universities.



EURAMET e.V.
Bundesallee 100
38116 Braunschweig
Germany

Phone: +49 531 592 1960
Fax: +49 531 592 1969
Email: secetariat@euramet.org

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European Metrology Research Programme
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The EMRP is jointly funded by the EMRP participating countries within EURAMET and the European Union

Publication date: August 2018