

The watt balance route towards a new definition of the kilogram

The need for the project

The kilogram is the last SI unit based on a material artefact, a Platinum-Iridium cylinder, kept at the Bureau International des Poids et Mesures (BIPM). However, the cylinder's mass is changing over time.

A promising way of supporting the redefinition of the kilogram is through the watt balance experiment, which links the kilogram to the Planck constant. Two European watt balance experiments are currently ongoing at the National Metrology Institutes in France and Switzerland (since 2002 and 1997 respectively).

This project will produce methods and devices to improve these watt balance experiments and support the development of future experiments.

Technical achievements

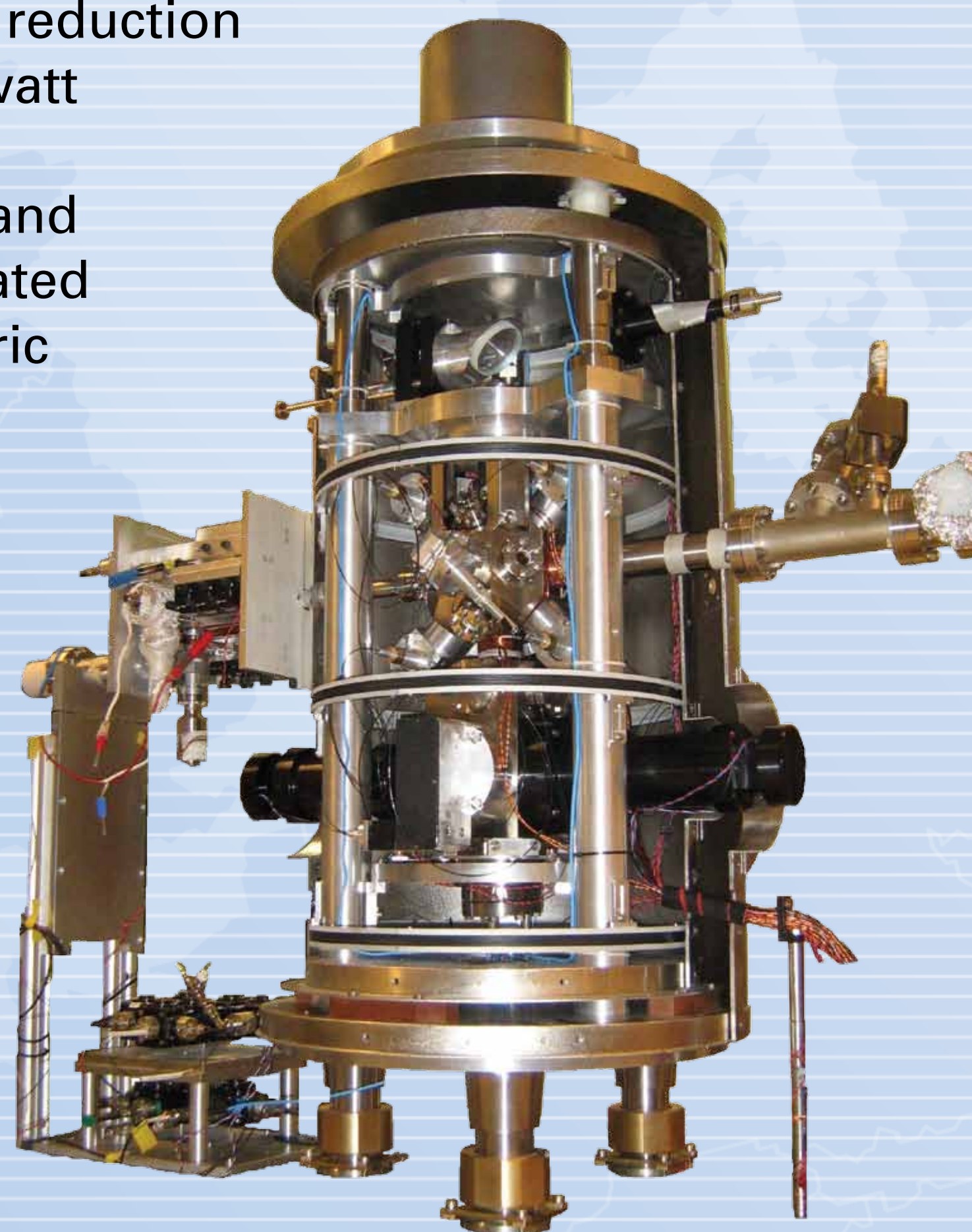
To reduce alignment uncertainty, the project developed mechanical tools to adjust the magnetic circuit induction field horizontality and the watt balance's coil displacement verticality. Improved position detectors based on propagation properties of laser beams and heterodyne interferometers together with specific collimation methods were also studied to characterise the watt balance's coil movements. The resulting misalignment uncertainty, modelled for the French watt balance damped suspension was less than 1×10^{-8} .

The project developed noise reduction based on the control of the watt balance's coil velocity; three heterodyne interferometers and sources intended to be operated in close loop with piezoelectric actuators were produced.

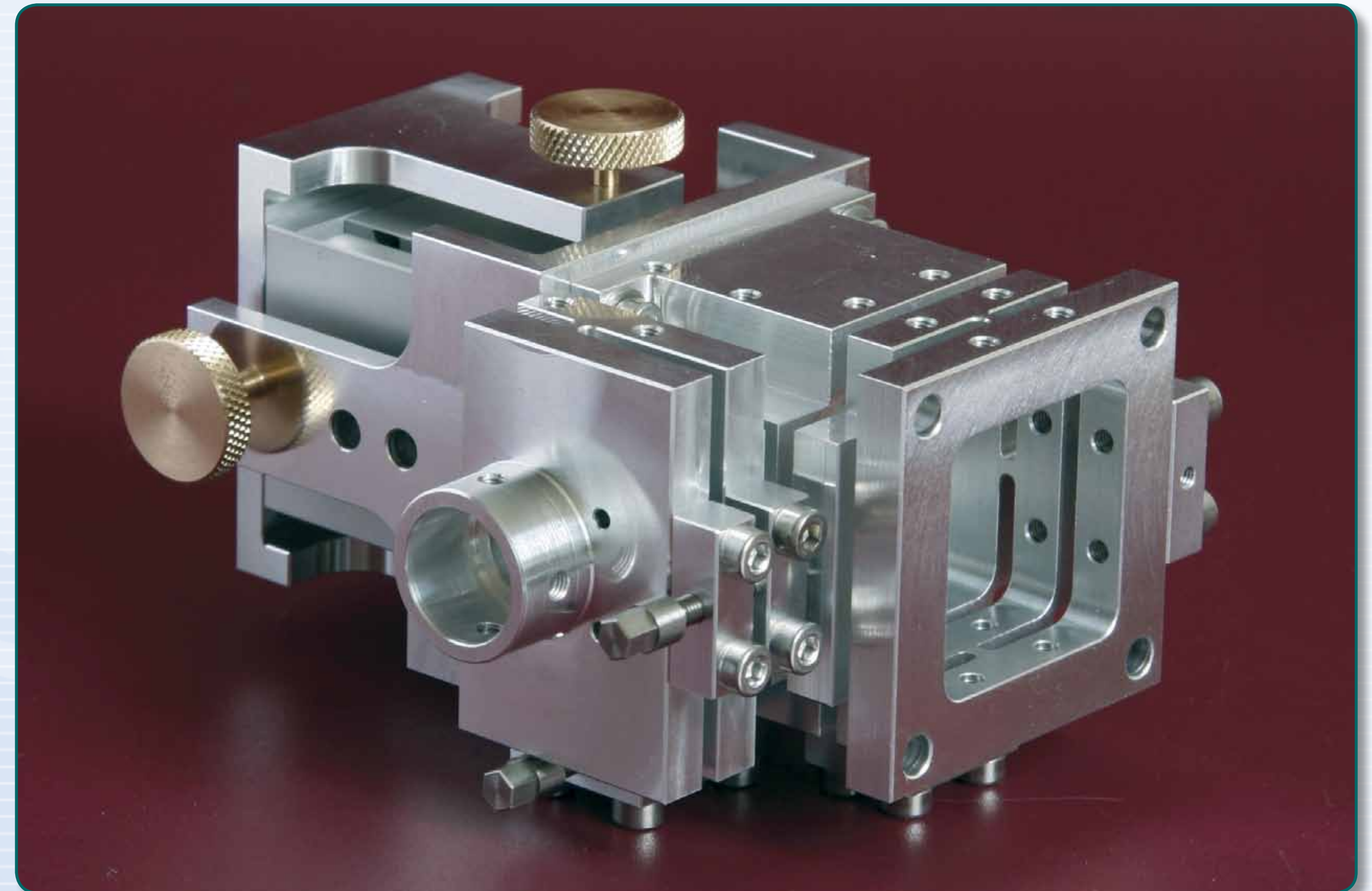
With the aim of improving the scattering of future values of the Planck constant, an *in situ* active compensation device was developed to reduce the disruptive external magnetic field effect on the effective watt balance magnetic field.

The acceleration of gravity can be measured with gravimeters based on different principles, such as atomic interferometer or falling corner-cube gravimeters. Three different gravimeters have been improved by the project and their uncertainty budget refined. To transfer the gravity acceleration value from the gravimeter to the standard mass of the watt balance, spatial variations were measured and modelled.

The watt balance in Switzerland determined a value of the Planck constant with a relative uncertainty of 2.9×10^{-7} .



Absolute cold atomic gravimeter with the magnetic shields partially removed.



Mechanical support of a position detector.

Improving watt balance experiments

Supported the two current watt balance experiments through enhanced synergy and the development and sharing of new tools, techniques and methods. The project will also support future watt balance experiments through its improvements in:

- the alignment of the watt balance
- the magnetic field behaviour of the magnet
- the noise level in the electric measurements
- the measurement of the gravimetric field

Some of the devices developed during the project can also be used by other scientific and industrial sectors, such as geophysics, inclinometry and position monitoring.

Supporting a redefinition of the kilogram

Enabled further developments in the two watt balance experiments by determining a value of the Planck constant with a relative uncertainty of 2.9×10^{-7} . This should lead to an even higher accuracy of values of the Planck constant and support the redefinition of the kilogram.

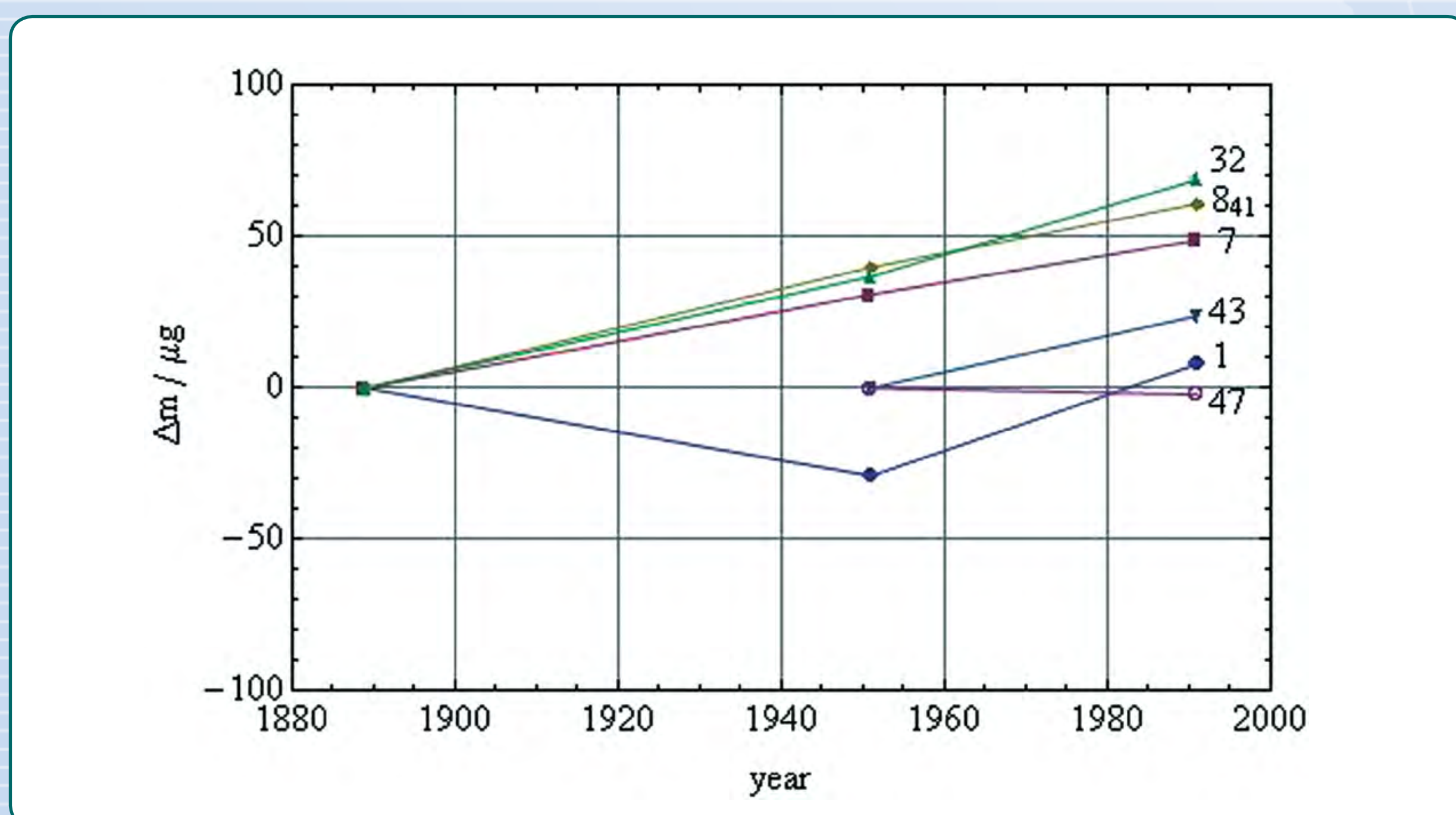
Avogadro and molar Planck constants for the redefinition of the kilogram

The need for the project

The kilogram is the only SI base unit to be defined by a material object - a Platinum-Iridium cylinder, kept at the Bureau International des Poids et Mesures (BIPM).

As a material object is vulnerable to its environment, the cylinder is changing as it ages and therefore a redefinition on the basis of a fundamental physical constant is needed.

The objective of this project was to link the mass unit, the kilogram, to the atomic mass unit via the Avogadro constant N_A , which specifies the number of atoms in one mole of substance. The project aims to demonstrate a direct kilogram realisation based on the mass of a silicon atom (isotope Si-28), expressed in terms of frequency and second. Determining the Avogadro constant in this way involves making measurements of the molar mass, volume, surface, density and lattice parameter.



Change in mass of the six official kg copies with respect to the mass of the international prototype.

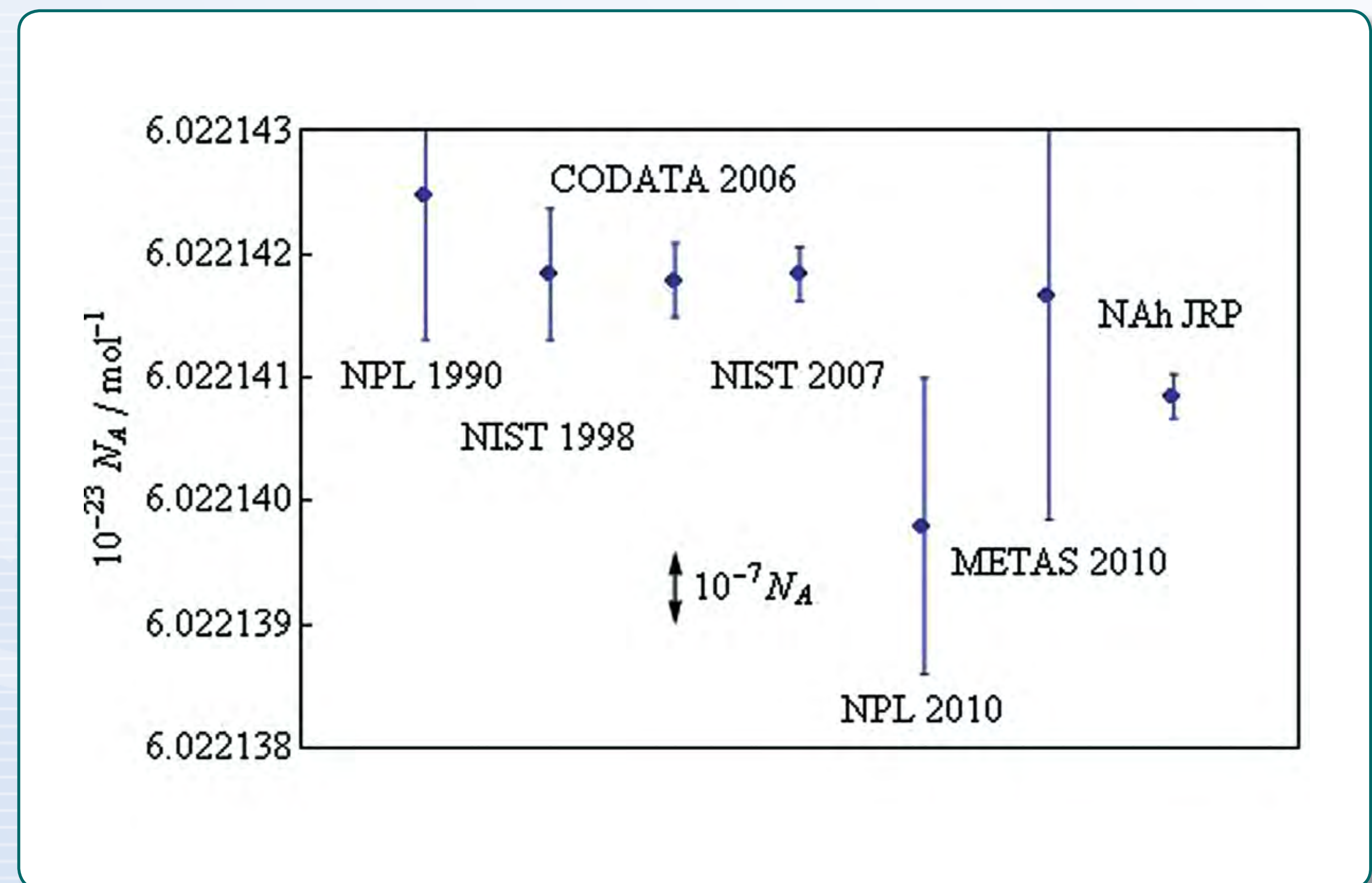
Technical achievements

Si-28 was purified to 99.99% (natural silicon contains around 92% Si-28) using centrifuges based at the Central Design Bureau for Machine Building in St Petersburg, Russia. This was then used to grow a crystal that was fashioned into two near-perfect spheres.

The enrichment relieved, but did not eliminate, the need to measure the isotopic composition of the crystal. This was done by means of mass spectrometry and by developing a novel measurement technique based on isotope dilution. Using laser interferometry and synchrotron radiation, each sphere's surface was mapped to measure its volume and to characterise its surface with atomic-scale accuracy. The crystal structure was imaged by x-ray interferometry to measure the atom spacing. The adequacy of crystal perfection and homogeneity to count the silicon atoms on the basis of their ordered arrangement in the crystal was also demonstrated.

By calculating the volume taken up by each atom, it was possible to work out how many atoms were in each sphere and, consequently, in a mole, $N_A = 6.02214082(18) \times 10^{23} \text{ mol}^{-1}$.

The present measurement uncertainty ($3 \times 10^{-8} N_A$) is 1.5 times higher than that targeted for a kilogram redefinition; it is limited by the performance of the measurement apparatus, not by crystal imperfection.



Comparison between the most accurate N_A values available. The bars give the standard uncertainty.

Redefining the SI units

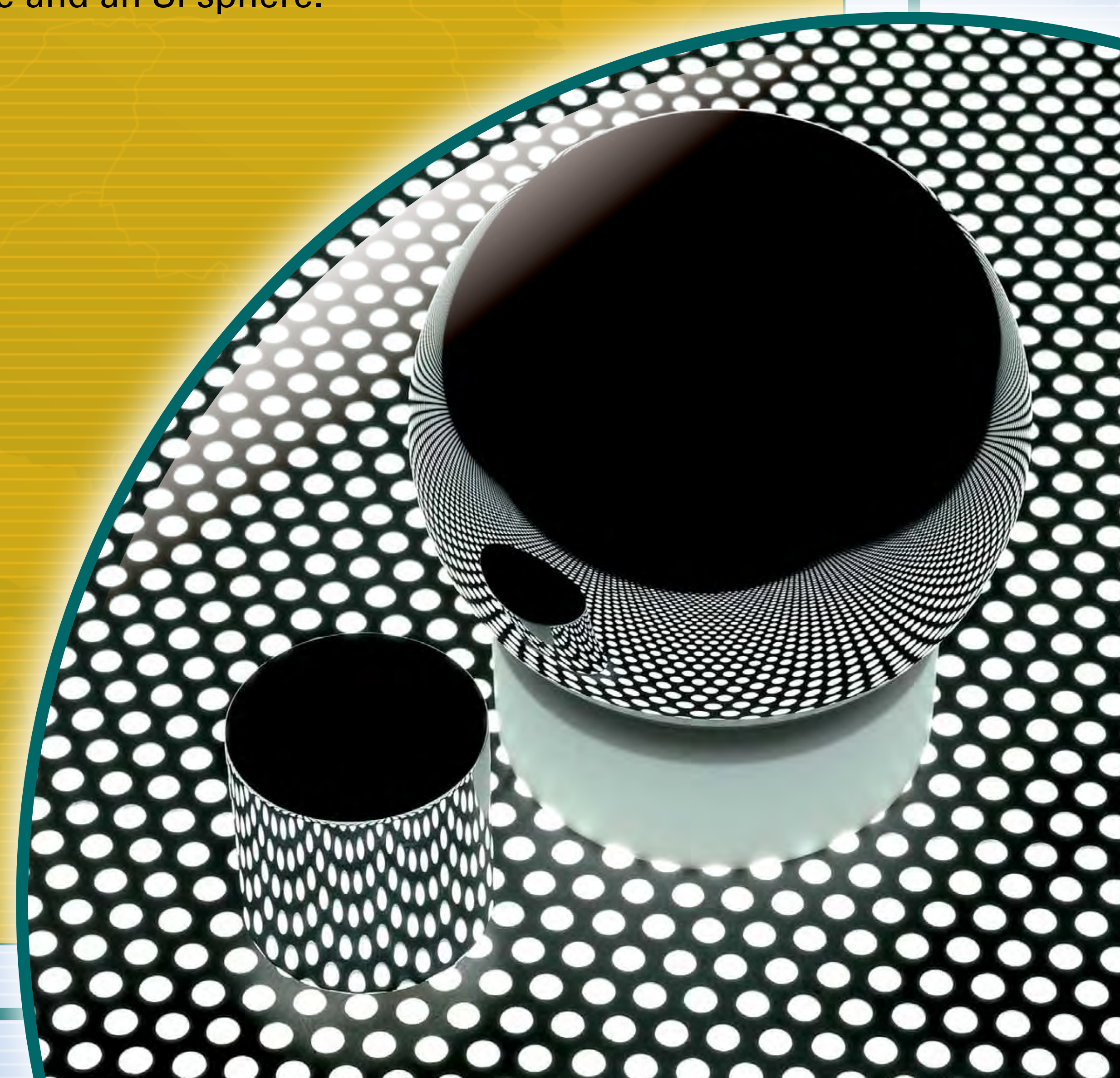
Project results will be exploited by the Committee on Data for Science and Technology and the International Committee for Weights and Measures to improve the quality and reliability of data for science and technology and to redefine the SI units on the basis of conventionally agreed values of fundamental physical constants.

The Si-28 spheres will be used to demonstrate the kilogram based on a fixed value of the Planck constant. The count of the atoms in the spheres will give the sphere mass.

Monitoring the international prototype

Enabled the monitoring of the stability of the international prototype kilogram, which is thought to drift by about 50 μg every 100 years. The prototype drift can be measured, or excluded, by mass comparisons between the prototype and an Si sphere.

A Pt-Ir kilogram reflected next to the Si-28 sphere.



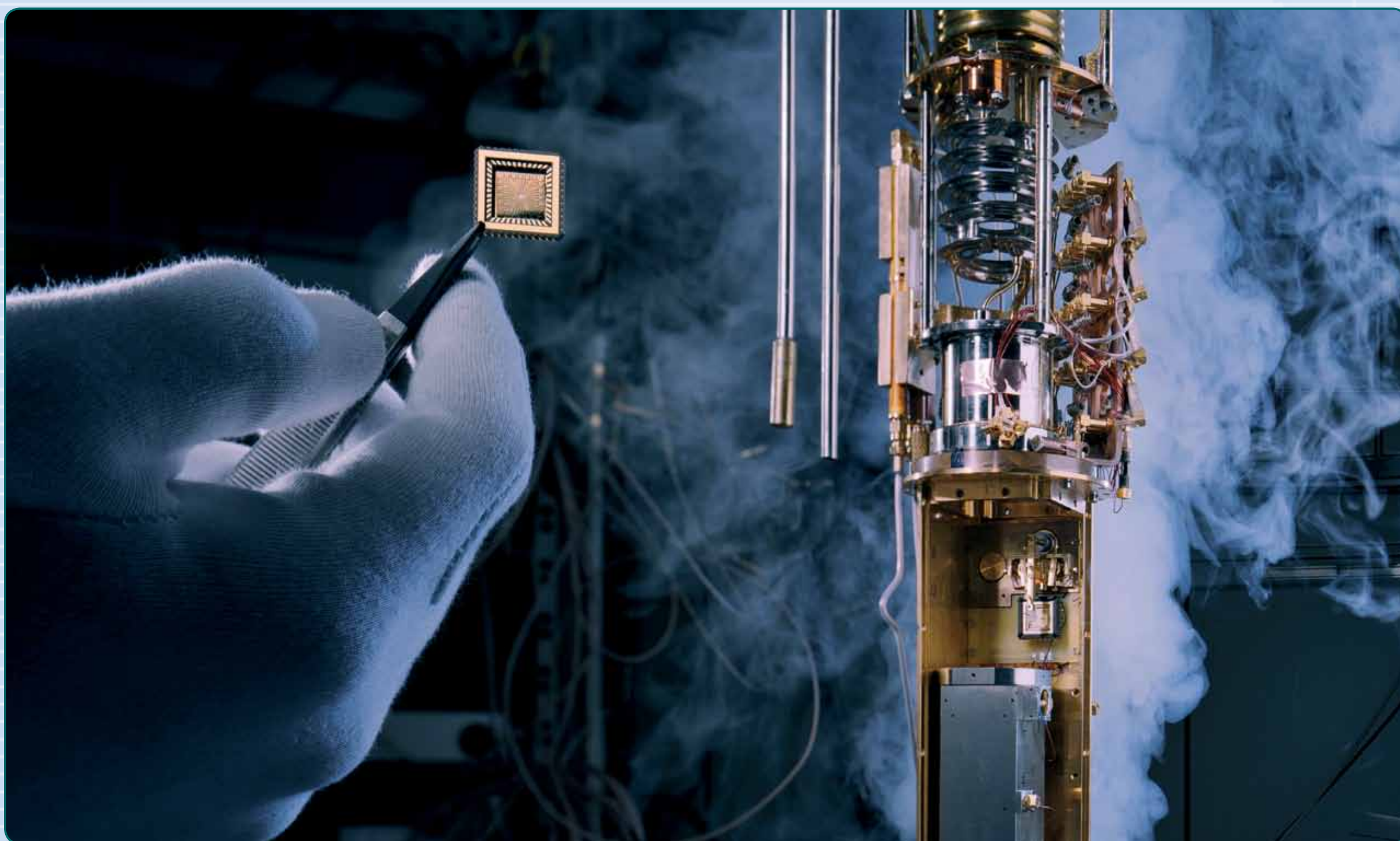
Foundations for a redefinition of the SI base unit ampere

The need for the project

In a proposed redefinition of the international system of units, the ampere and kilogram would be based on fixing values for e (elementary charge) and h (Planck constant). This would replace the 'conventional values' assigned to the Josephson constant (K_J) and the von-Klitzing constant (R_K) in the current system for defining the volt and ohm. In theory there are simple relationships between these four constants ($K_J = 2e/h$ and $R_K = h/e^2$) but these have not been experimentally verified with sufficient accuracy.

The work by metrologists to prove that these simple relationships can be relied upon to define the volt and ohm is an experiment commonly referred to as the Quantum Metrological Triangle (QMT). It requires devices, which generate a known current by counting single electrons.

This project aimed to close the QMT and improve the uncertainty associated with e and h values.



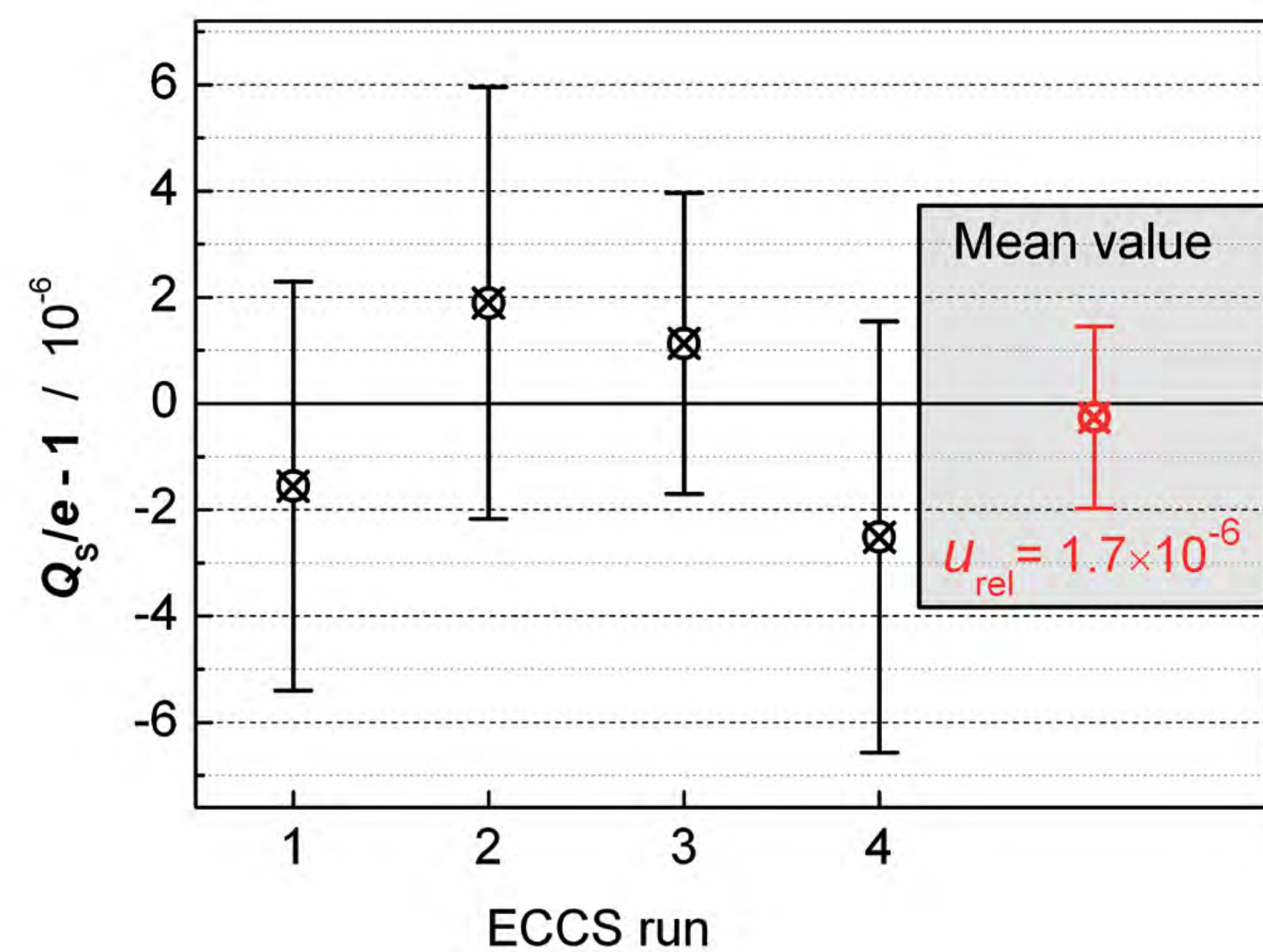
A chip carrier with a single electron pumping nano-circuit before being mounted into the milli-kelvin cryostat. The grey metal box in the lower part of the cryo-system houses the precision 1 pF capacitor which is charged with up to 63 million electrons.

Technical achievements

Two QMT setups were implemented, based on current and charge comparison. With the charge comparison QMT, a Single Electron Transport (SET) device was operated at an error rate sufficiently low to close the QMT. A precision linkage of the involved 1 pF capacitor to the von-Klitzing constant was performed. This QMT variant was closed with an uncertainty of 1.7 parts per million (ppm).

For the QMT based on current comparison, a cryogenic current comparator was developed which amplifies currents by a factor of 20,000. Using the traditional SET devices, evidence was collected that, even in seemingly perfect devices, peculiarities of the transport features may prevent reliable results. This finding has important consequences for the next generation QMT experiments employing high current devices.

Progress was made with high current SET devices; hybrid turnstiles featuring a combination of superconducting and normal conducting elements were made and operated in parallel to produce currents of 100 pA and above. The project also developed tunable semiconductor single electron pumps that allow similar or higher currents by higher frequency operation. Finally, an error detection concept was developed which will allow a precise determination of current even when the current generating device operates at error rates as large as 1 in 10^4 .



Results from the Electron Counting Capacitance Standard (ECCS) QMT experiment. In this first complete test run an uncertainty of 1.7 ppm was obtained after only 2 h of measurement time.

Supporting a redefinition of the SI units

The results of this project will improve the threshold uncertainty (7 parts in 10^7) associated with SI units based on h and e values, through the QMT experiment.

The unit of mass, and units derived from it, will also be affected, via the redefinition of the kilogram with the watt balance experiment.

The Committee on Data for Science and Technology has confirmed that it will consider the project's results in a new report.

New high current SET devices

Developed three high current SET devices; hybrid turnstiles, tunable semiconductor single electron pumps and an error detection concept to enable the precise determination of current. The high current SET devices are able to calibrate current meters with an uncertainty 20 times better than currently available.



Determination of the Boltzmann constant for the redefinition of the kelvin

The need for the project

Since the establishment of the SI units in 1960, extraordinary advances have been made in relating them to invariant quantities such as the fundamental constants of physics and the properties of atoms.

The present definition of the unit of thermodynamic temperature was given by the 10th General Conference on Weights and Measures in 1954, which selected the triple point of water as a fundamental fixed point, so defining the unit kelvin. However, the development of new primary methods of thermometry that are difficult to link directly to the triple point of water led the Consultative Committee for Thermometry to propose a redefinition of the kelvin using the Boltzmann constant (k).

This project aimed to determine the Boltzmann constant, via a number of experimental routes.

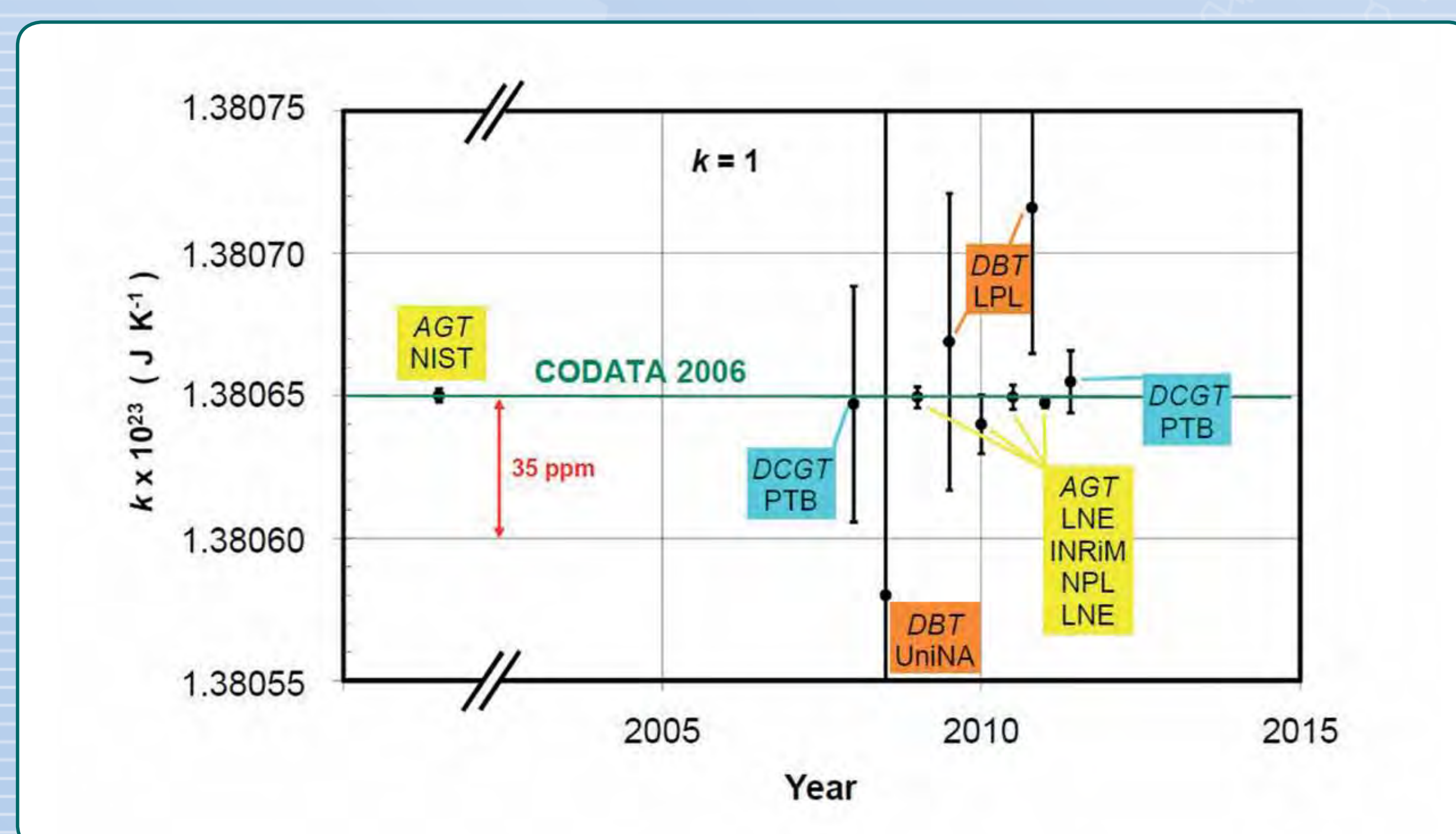
Technical achievements

In order to reduce the uncertainty in the determination of k , advances have been achieved in the fields of physical acoustics, thermodynamics, microwave measurements, thermometry, dimensional measurements, residual gas analysis, perturbation theory, pressure and capacity measurement, and laser-spectroscopy.

The project achieved determinations of k by three methods; acoustic gas thermometry, Doppler broadening thermometry and dielectric constant gas thermometry. All results were consistent with the currently accepted value of k . The standard uncertainty of the project's most recent result corresponds to a reduction in uncertainty by a factor of 1.5.



AGT Cell - Spherical resonator for acoustic gas thermometry (AGT).



Results for the Boltzmann constant k obtained by dielectric constant gas thermometry (DCGT), Doppler broadening thermometry (DBT) and AGT.

The results of the project, including the uncertainty budgets, have been provided to the interdisciplinary Committee on Data for Science and Technology (CODATA) for evaluation and qualification of the measurements. The outcome of this evaluation should set the standard uncertainty of k at 0.9 ppm, corresponding to a reduction in the uncertainty by a factor of 2.

Improving temperature measurement

Worked closely with the Consultative Committee for Thermometry and its task group on the Mise en Pratique for the Definition of the kelvin to help improve temperature measurement.

The kelvin can be calculated in two ways, by calibrating a working-standard thermometer against the readings of a primary thermometer and by using the International Temperature Scales. Although the International Temperature Scales do not rule out methods based on primary thermometry, the lack of their recognition leads to confusion. This project helped reduce this confusion by developing International Temperature Scales in the context of other methods.

Developing primary thermometry methods

Fostered the development of a number of primary thermometry methods (e.g. absolute spectral-band radiometry used in steel production) and developed special Johnson noise thermometers, benefiting manufacturers of low temperature refrigerators, superconducting magnets and ultra-sensitive sensors.

Supporting a redefinition of the kelvin

Reduced the uncertainty when measuring k by a factor of 1.5 and submitted results to CODATA for evaluation. It is anticipated that as a result of the project, the final uncertainty for k will be reduced to a temperature equivalent of $u(k=1) \approx 0.25 \text{ mK}$, by the time of the redefinition of the kelvin.

Joint Research Project (JRP) Short Name: Boltzmann constant • JRP-Coordinator: Joachim Fischer (PTB) • JRP-Partners: CEM (Spain), CNAM (France), DFM (Denmark), INRIM (Italy), JRC (EC), LNE (France), NPL (UK), PTB (Germany)

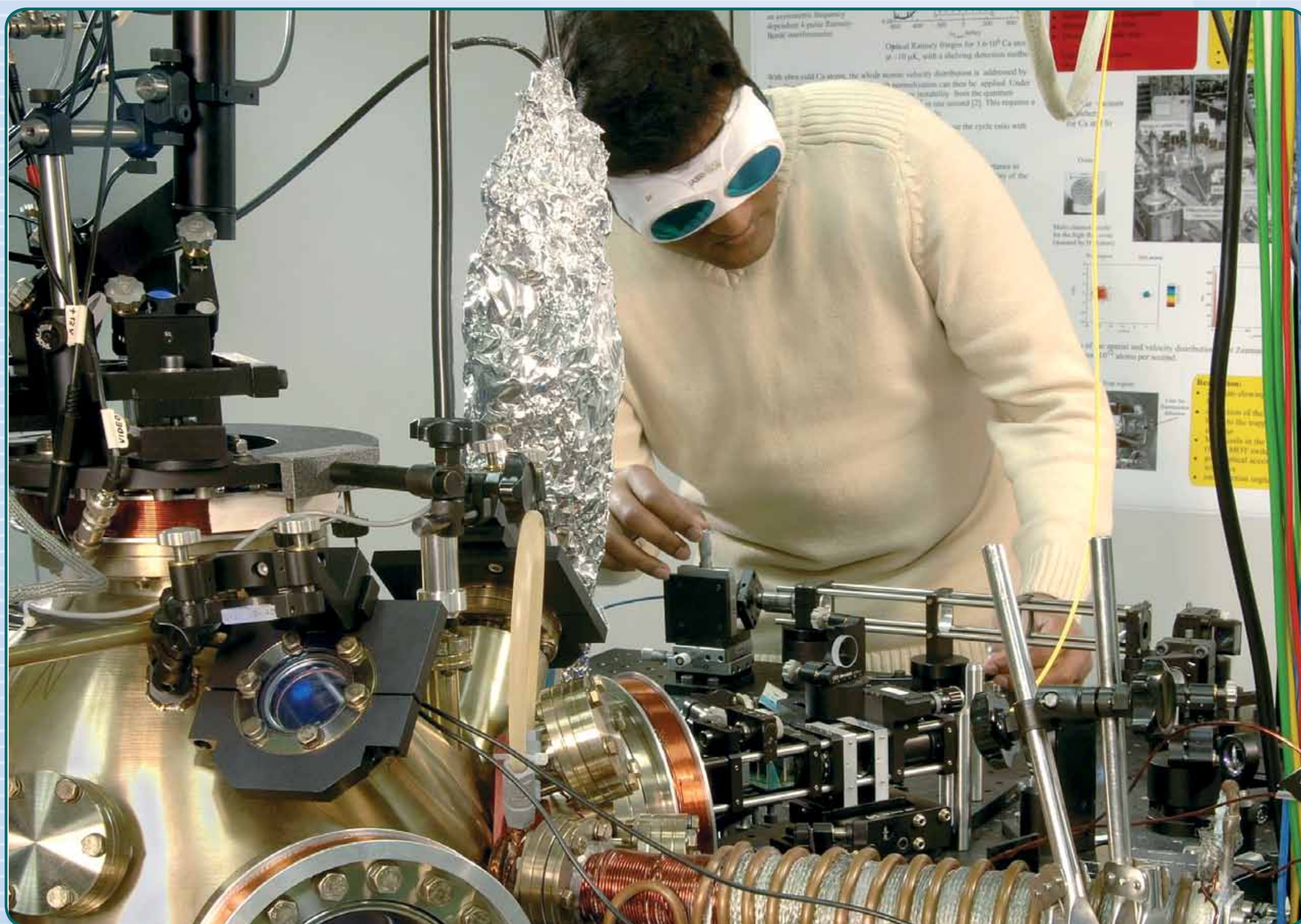
Optical clocks for a new definition of the second

The need for the project

Ultra high performance optical clocks now outperform the best microwave standards based on caesium (Cs) atoms, which are presently used to define the second. A new definition of the second is therefore needed so that it remains based on state-of-the-art technology.

The principle underlying the operation of these optical clocks is the use of an optical lattice to freeze the atomic motion. Atoms are strongly confined in a series of potential wells formed by the interference of lasers. By using a large number of atoms simultaneously, this type of clock has superior potential in terms of ultimate frequency stability - presumably better than 10^{-18} after one day of integration.

The aim of this project was a detailed investigation of a possible optical resonance for the new definition of the second: the 1S_0 - 3P_0 transition of atomic strontium (Sr).



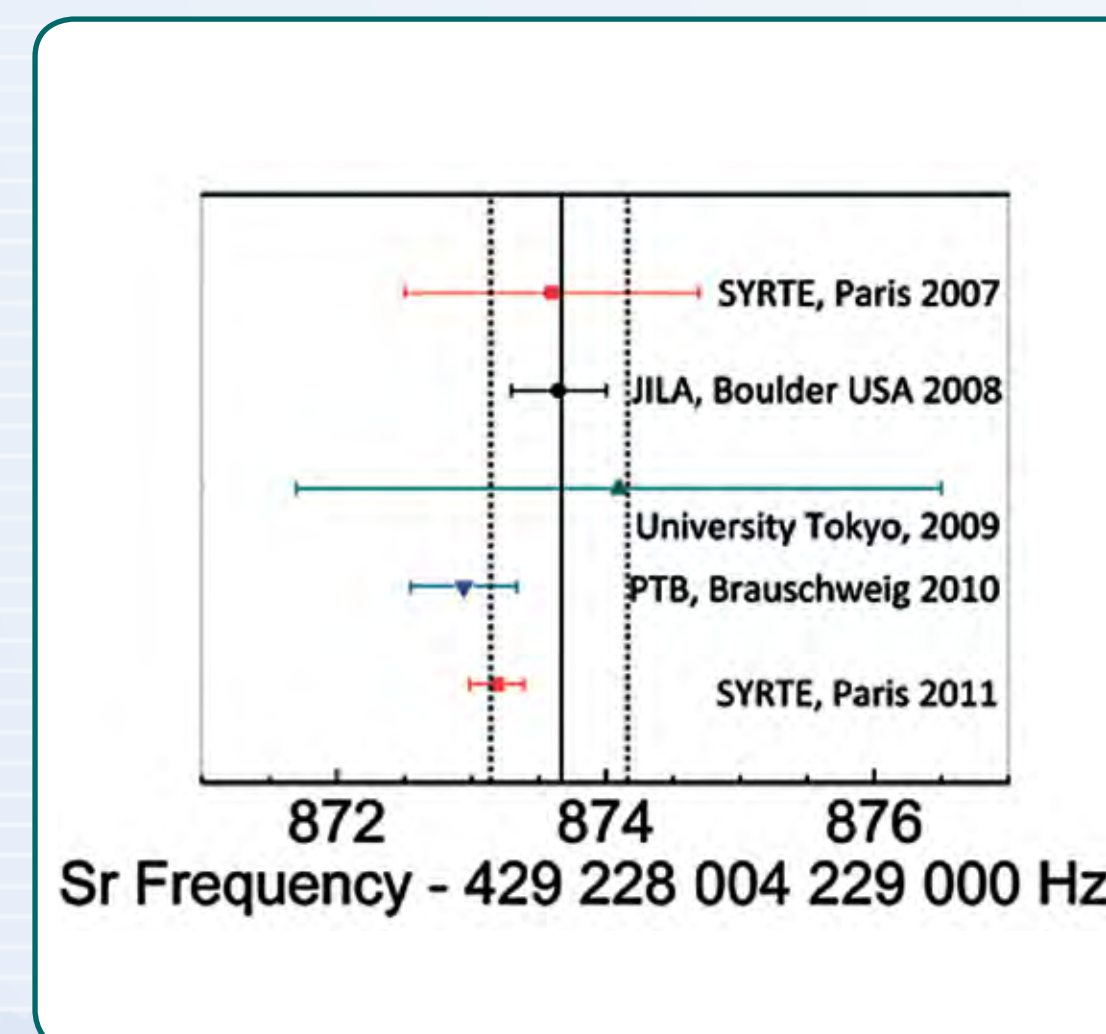
The vacuum system of PTB's strontium lattice clock.

Technical achievements

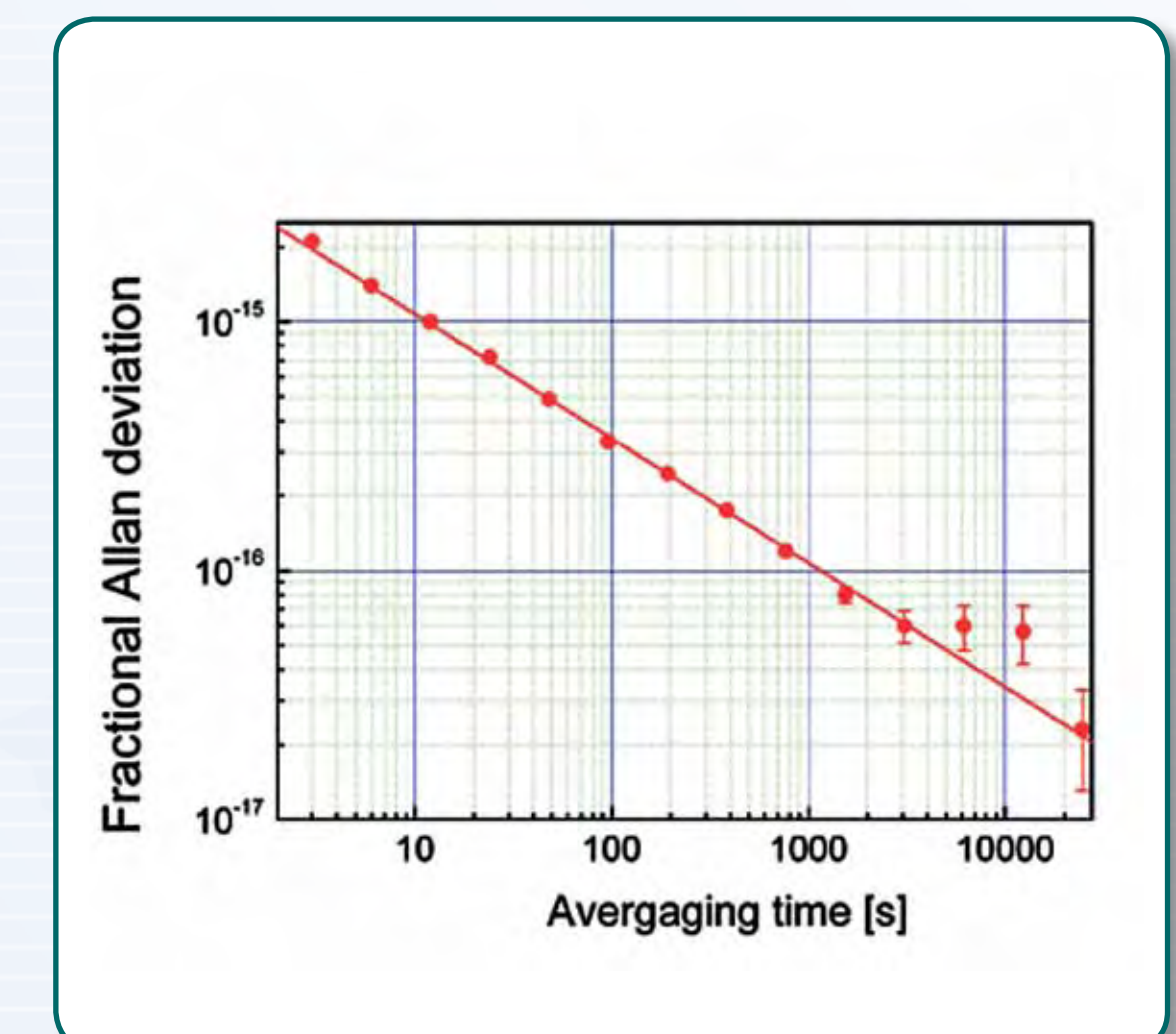
Both the accuracy and stability of optical clocks have been improved to a level significantly better than the best Cs fountain primary standards. By using optical resonances, as narrow as 3 Hz, corresponding to a quality factor exceeding 10^{14} , and performing the first high performance comparison of Sr lattice clocks, a fractional frequency stability as good as 5×10^{-17} was demonstrated after one hour of averaging time.

The most relevant frequency shifts were studied in detail both experimentally and theoretically. This included the residual effect of the lattice field, the effect of collisions between cold atoms, and the effect of the blackbody field radiated by the environment surrounding the atoms. This resulted in a fractional frequency accuracy of approximately 10^{-16} .

Comparisons with a large set of primary standards were performed, resulting in a measurement of the Sr clock frequency in SI units with unprecedented accuracy. Methods were developed for future improvements, such as the demonstration of a non-destructive detection method for the optimisation of the clock frequency stability, as well as technological developments for future transportable or space-bound clocks.



Frequencies of the ^{87}Sr clock transition measured by different laboratories. The vertical line gives the recommendation for ^{87}Sr as secondary representation of the second with its uncertainty (dashed lines). The two points PTB 2010 and SYRTE 2011 represent the best optical frequency measurement to date.



Allan deviation of the comparison between two independent Sr lattice clocks, demonstrating for the first time a mid-term stability in the low 10^{-17} range.

Advanced optical clocks

Developed three Sr clocks that can be used as a common reference to repeatedly measure the other clock transitions under investigation at National Metrology Institutes.

The project demonstrated the accuracy and stability of these optical clocks and increased knowledge of collisions between cold atoms, atomic motion in the quantum regime and atom-field interactions.

The clocks will also be at the heart of two future space missions: ACES/PHARAO and STE-QUEST.

Space project 1: ACES/PHARAO

Due to commence in 2014, over 18-36 months, it aims to demonstrate relative geodesy, by mapping the Earth's gravitational field using atomic clocks as sensors of the gravitational redshift. The clocks will be used as references for the evaluation of the gravitational field difference between different locations.

Space project 2: STE-QUEST

Building on the results of ACES/PHARAO, the STE-QUEST project will use a higher level of performance and a different satellite orbit. This mission is due to take place after 2020.

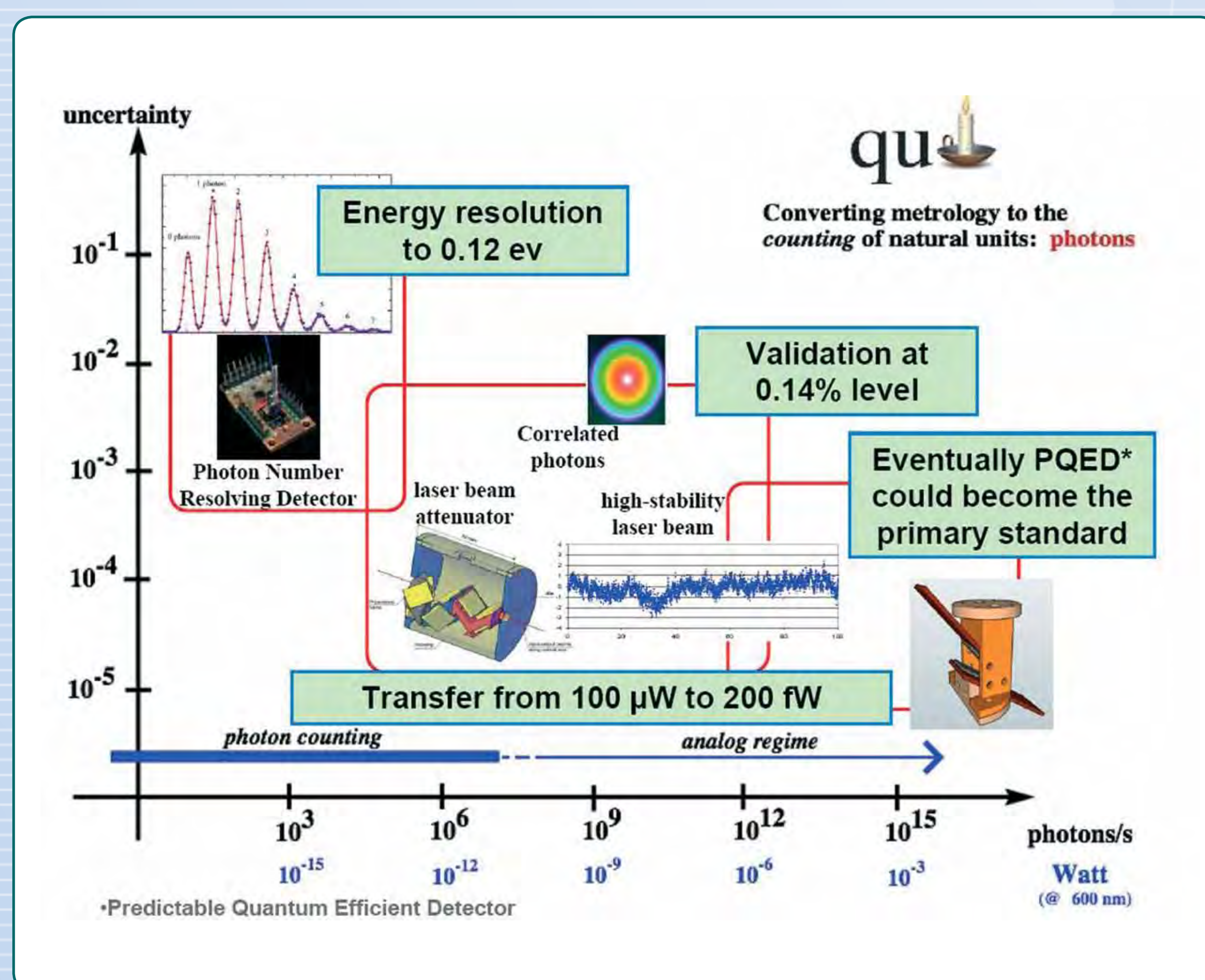
Candela: Towards quantum-based photon standards

The need for the project

The candela is the SI base unit of luminous intensity, however, its definition is not linked to the concepts of modern physics that underpin the development of quantum optical technologies.

A change in optical metrology is therefore required, in order to bridge the energy difference between emerging quantum technologies and classical radiometry and photometry, and connect the measurement of macroscopic quantities like optical power with the quantum world of photon number measurement.

This project aimed to develop standards for photon metrology from the signal level (10^{13} - 10^{14} photons/s) of existing radiometric standards (10-100 microwatts) down to single photons. It also aimed to address requirements for new SI traceable quantum based photon standards (in units of photons/second) and the challenge of expressing the candela in terms of photons per second.

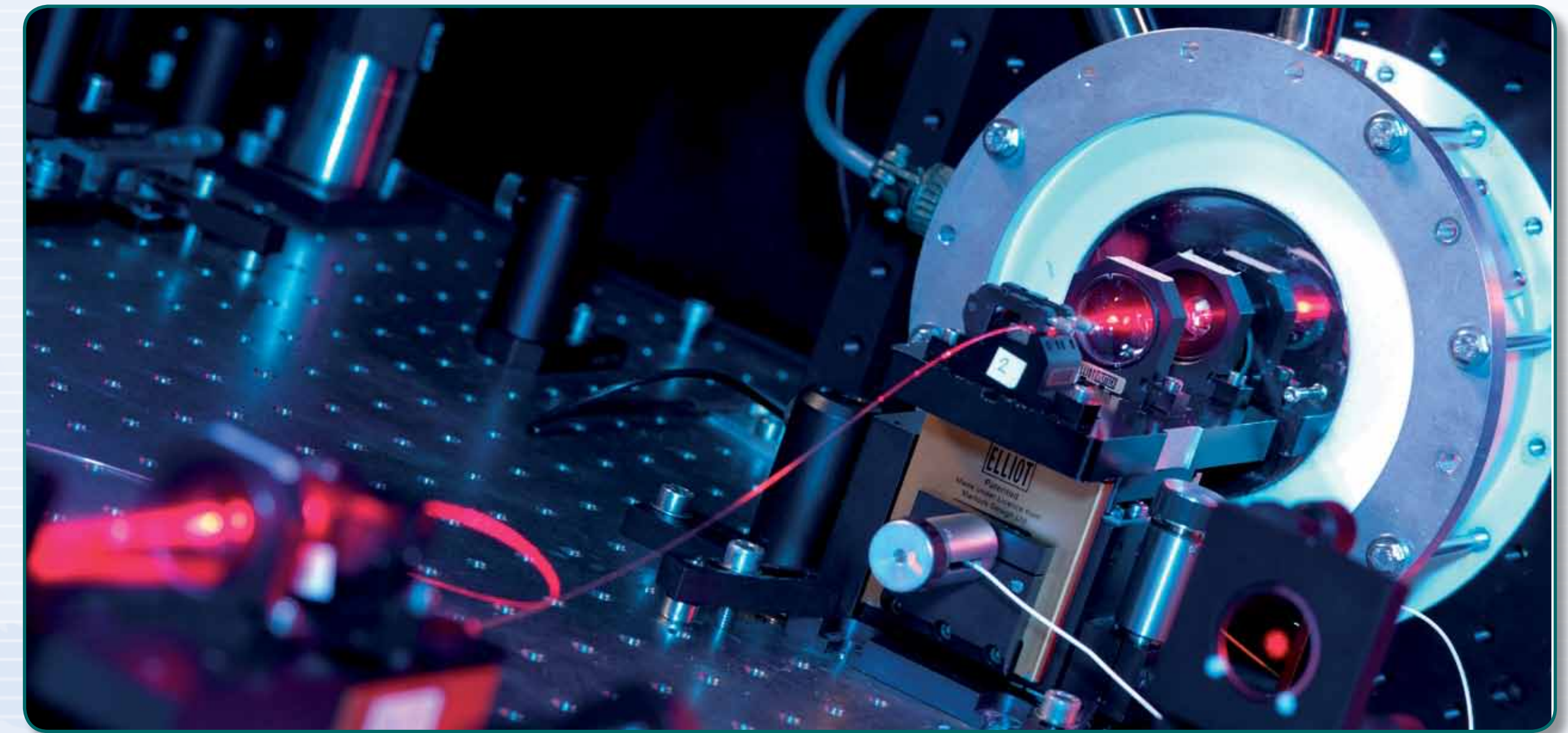


Schematic representation of the linkage of the few photon regime to the SI.

Technical achievements

The project developed silicon photodetectors with a quantum efficiency predictable with an uncertainty below 10 ppm over the whole visible range of wavelengths and suitable for the measurement of photopic fluxes. It also produced transition-edge detectors able to resolve up to 12 photons with an energy resolution better than 0.12 eV for the measurement of single photon fluxes. Techniques for scaling between low and high photon flux regimes, with uncertainties in the calibration chain of approximately 100 ppm were also developed.

These techniques were validated through the Planck constant; by the ratio between the photon flux and the radiant flux of a monochromatic beam at wavelengths 488 nm, 576 nm and 761 nm at 100 μ W and 761 nm at 1 pW.



Confocal microscope system developed to pump and collect single photons.

New primary standards

Developed new primary standards for the photometry community and a traceability chain based on the most widely used detectors - silicon photodiodes. These new standards are portable, cheaper and easier to use than current standards, making them more accessible to end-users (e.g. National Metrology Institutes and LED manufacturers).

Commercialisation of PQED devices

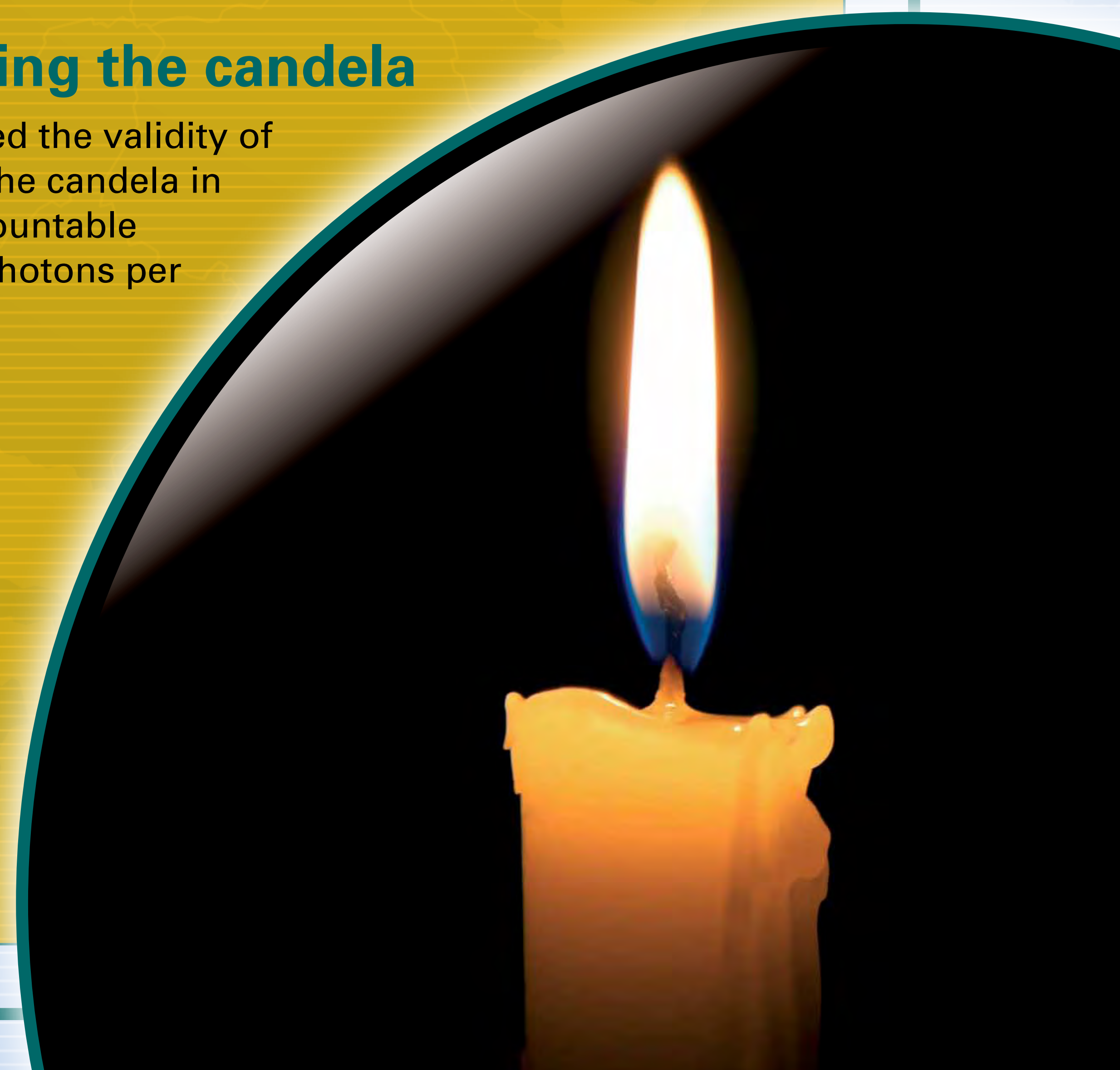
The project developed Predictable Quantum Efficient Detectors (PQED) which have been made commercially available by Fitecom Ltd, a Finnish service provider of measuring equipment.

Standards for photon counters

Provided validated and traceable standards for the manufacturers of photon counting devices. Specifically, the project carried out spatial uniformity measurements by characterising photon counters. Results showed that candidate channel photomultipliers and single photon counting modules showed poor uniformity results. Publication of this work led to Princeton Lightwave setting up their own spatial uniformity characterisation facility to improve the performance of their diodes.

Redefining the candela

Demonstrated the validity of expressing the candela in terms of a countable number of photons per second.



Breath analysis as a diagnostic tool for early disease detection

The need for the project

Early disease detection can literally mean the difference between life and death for diseases such as cancer, where the prospects of curing patients are significantly higher following early diagnosis. Breath analysis is a non-invasive method for monitoring the volatile organic compounds present in an individual's exhaled breath and is one of the clinical tests that can be used for early disease detection.

Recent advancements in laser absorption spectroscopy techniques have led to the development of small, calibration-free devices for performing breath analysis tests, with time-resolved measurements. However, despite these advancements, the accuracy and reliability of breath analysis measurements needs to be considerably improved.

This project addresses these measurement issues with the aim of producing reliable identification and quantification of selected compounds present in exhaled breath.

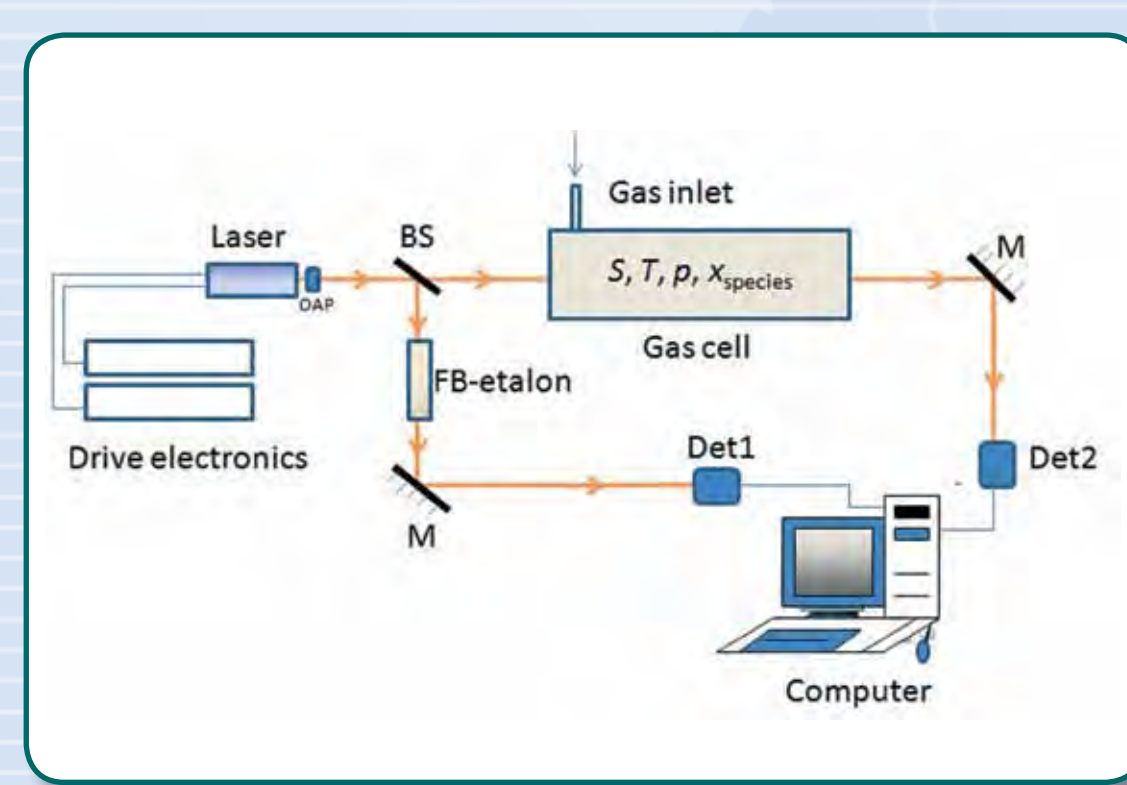
Technical achievements

The project has demonstrated that measurements of methane, carbon monoxide and carbon dioxide can be performed with an expanded uncertainty of 1-2% with detection limits down to amount-of-substance fractions of 1 nmol mol^{-1} or better. This level of uncertainty is negligible in comparison with the natural variations in amount of these components in human breath. For other targeted molecules, such as methanol, ethanol, acetone, formaldehyde and ammonia, the uncertainty ranges between 3-5%, because of adsorption effects in gas sampling systems.

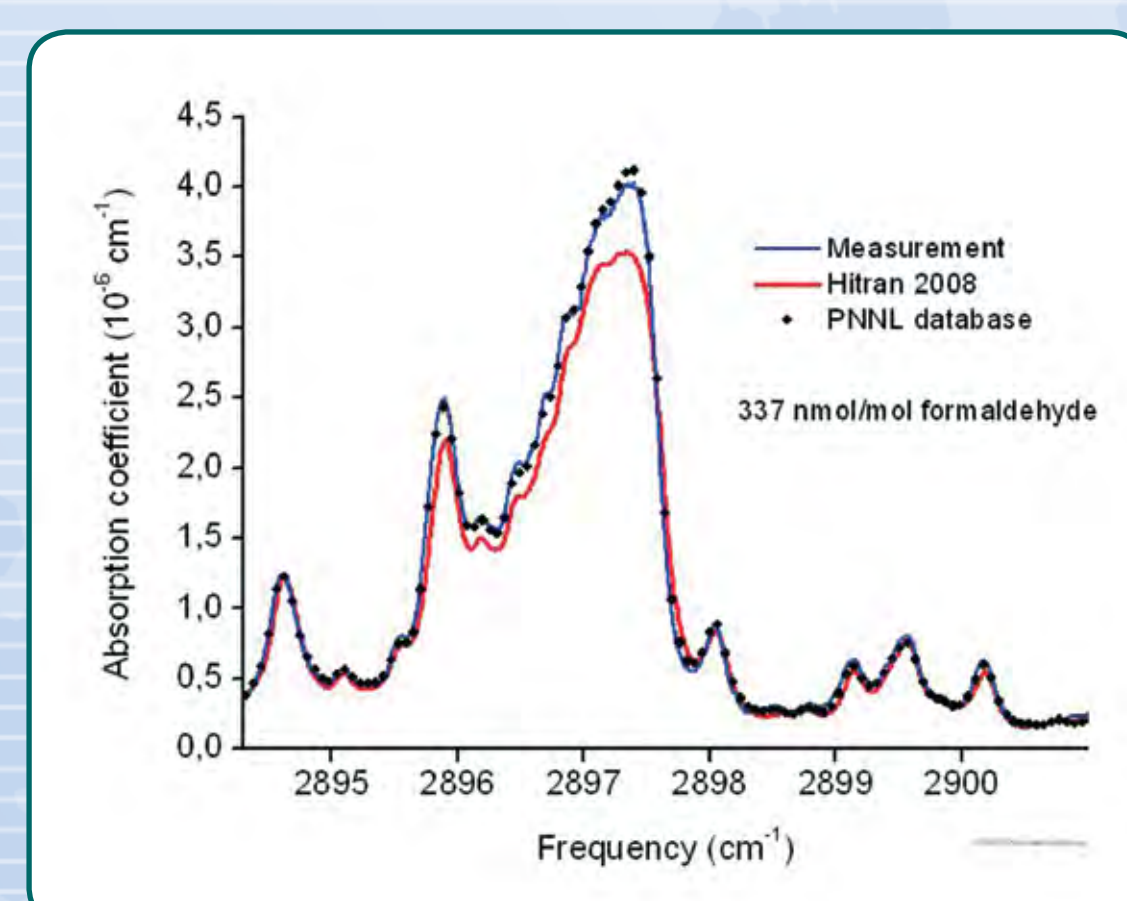
For the above listed molecules, reference data has been obtained and compared to spectroscopic databases, which are used as a basis to convert spectroscopic data into gas composition data. For ethane and formaldehyde it was found that the most widely used database HITRAN was incomplete and several tabulated line strengths had an error of more than 10%.

Methods and protocols were also developed for applying various spectroscopic techniques in breath analysis. One protocol linked laser-based spectrometric principles to metrological aspects of breath analysis and explained how equipment should be calibrated to obtain traceable measurements from spectroscopic reference data.

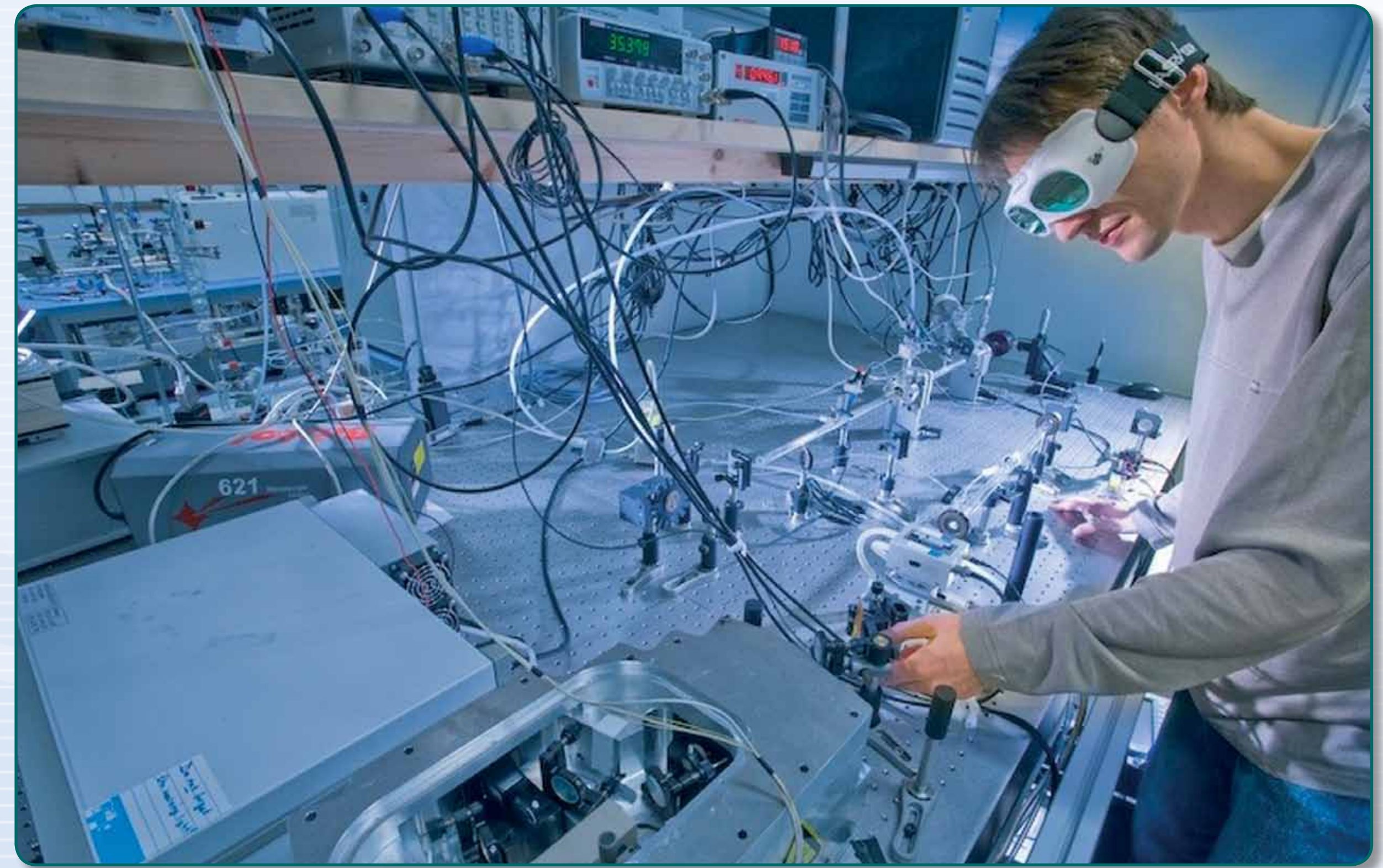
Experiments with moisture removal from gas mixtures have demonstrated the versatility of these techniques and enabled the quantification of errors introduced by sample preparation.



Tunable diode laser-based setup for breath analysis.



Comparison of the measured formaldehyde spectrum with the HITRAN and PNNL database. Formaldehyde could be of medical interest as some studies found elevated levels in the exhaled breath of patients with primary lung cancer.



Cavity ring down spectrometer operating in the mid-infrared wavelength region.

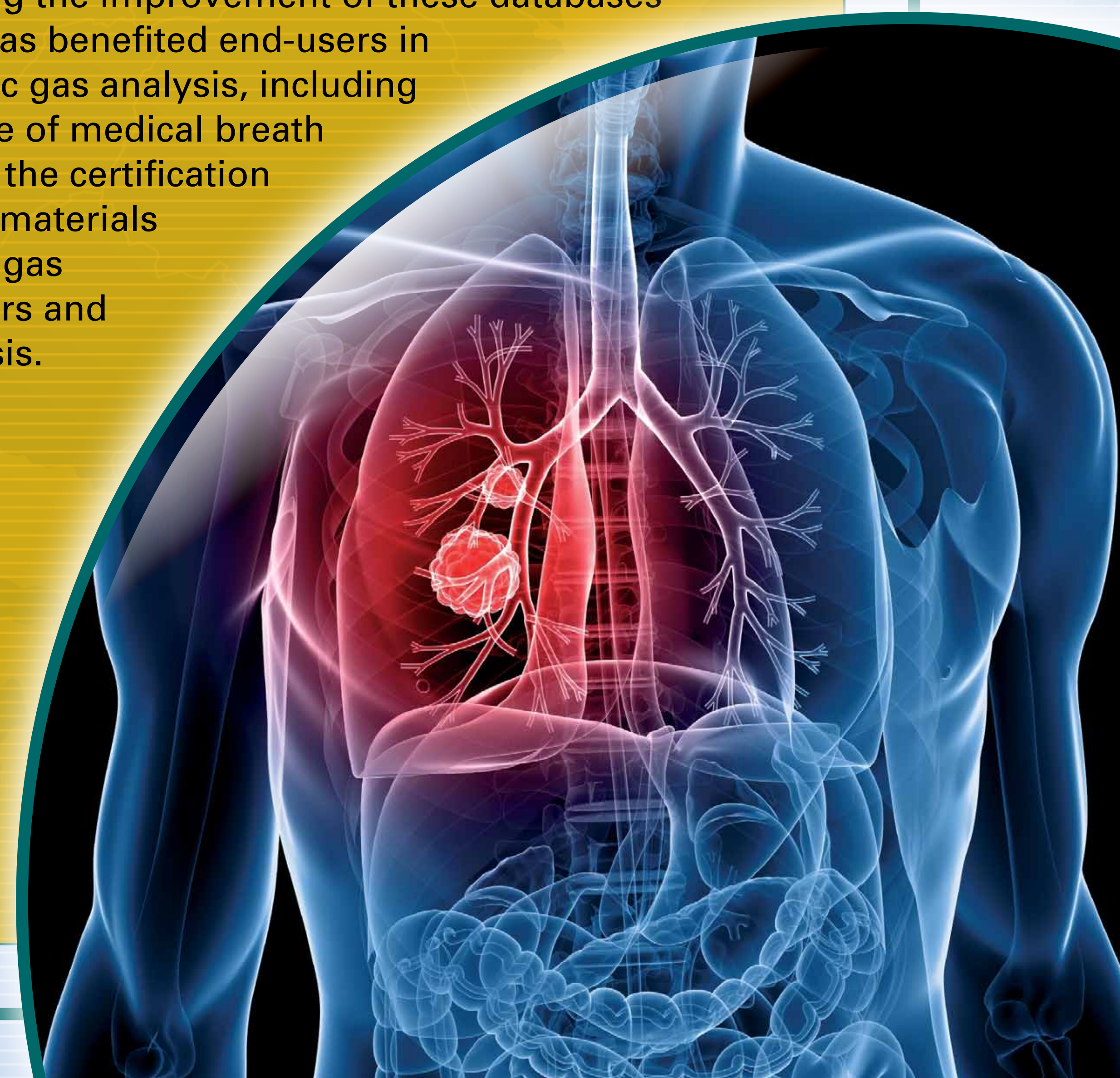
Supporting the medical community

Successfully demonstrated the potential application of spectroscopic techniques in the medical field. The techniques and methods developed have resulted in improved measurement quality for medical trials, leading to reduced repetition of measurements and an improved basis for combining the results from different trials.

Improving spectroscopic reference data

Virtually anyone who uses spectroscopic techniques uses databases such as HITRAN and PNNL in order to convert spectroscopic measurement data into gas compositions. The accuracy of the gas composition derived depends on the accuracy of the gas mixtures used for obtaining these reference data. The project showed that the HITRAN database was incomplete for ethane and formaldehyde and several line strengths had an error of more than 10%.

By supporting the improvement of these databases the project has benefited end-users in spectroscopic gas analysis, including those outside of medical breath analysis e.g. the certification of reference materials for specialty gas manufacturers and purity analysis.



Metrology on a cellular scale for regenerative medicine

The need for the project

With an increasingly ageing population there will be times when we will all require some form of healthcare support in order to manage disease and maintain our quality of life. However, the challenge for healthcare providers and governments is to manage these potentially spiralling health costs. Significant savings could be made through tissue regeneration; growing new cells to replace damaged or diseased tissue, thereby removing the need for long-term drug treatment and the possibility of adverse side effects.

This project aimed to support regenerative medicine by developing robust procedures for cell growth measurement and characterisation, and by defining cost effective metrics for assessing the consistency of products containing living cells.

Technical achievements

Support for regulation:

Techniques such as CARS (Coherent Anti-stokes Raman Spectroscopy) and MALDI (Matrix-Assisted Laser Desorption/Ionization Imaging) spectrometry have been developed. These can be used to characterise the behaviour of cells in regenerative medicine products.

Support for healthcare:

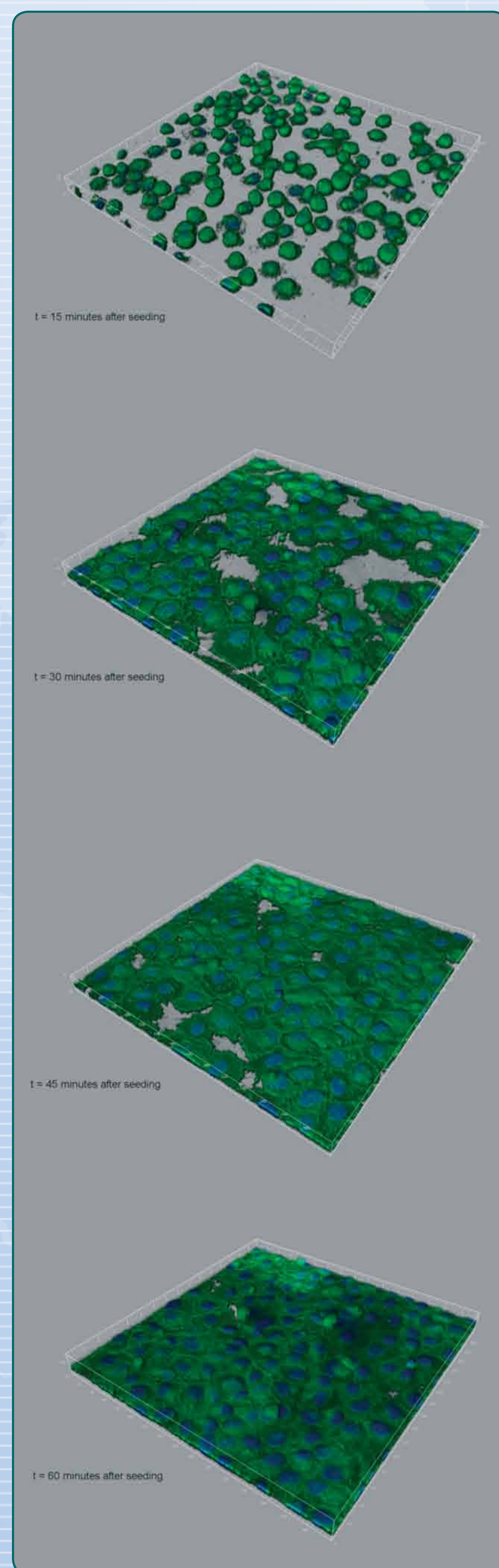
This project has developed specialised methods to understand the measurement uncertainty associated with diagnostic evaluation of cells in clinical samples.

Support for innovation:

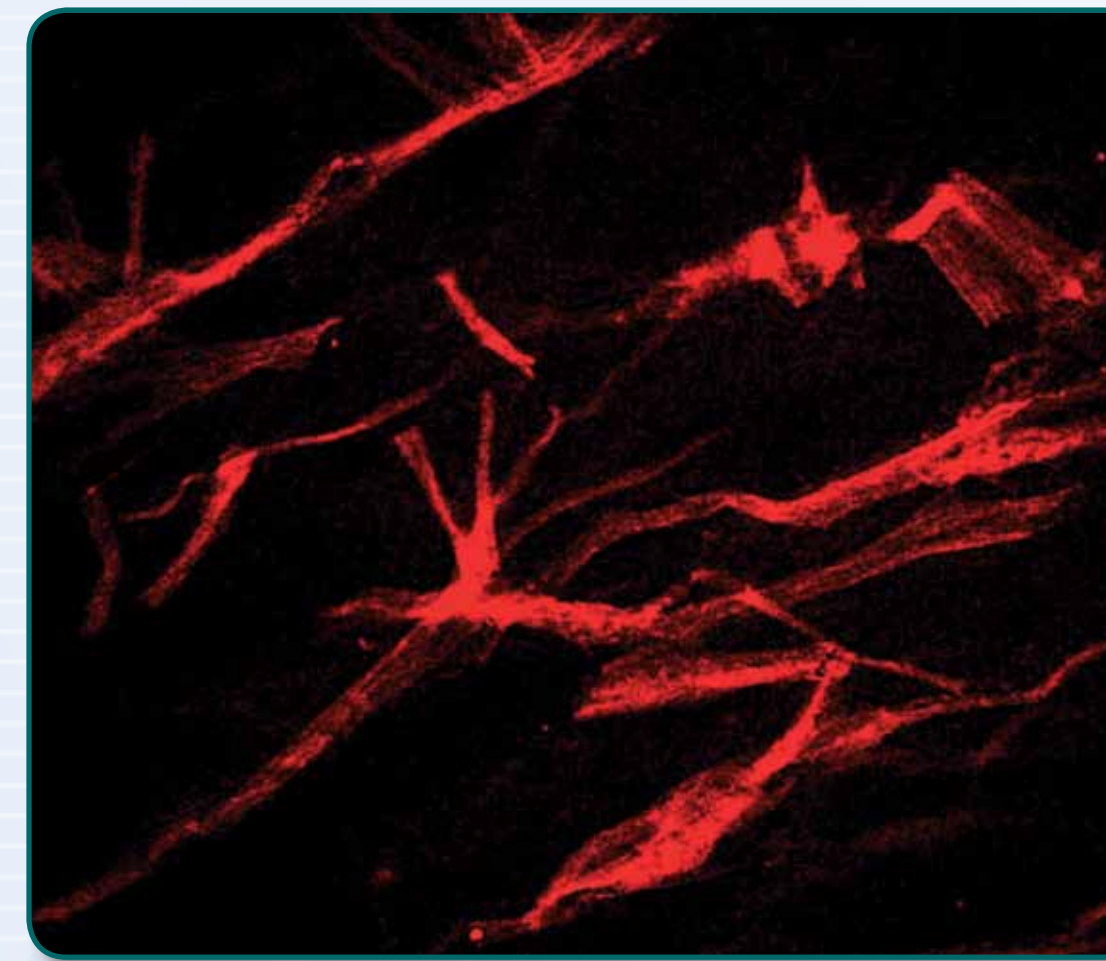
The measurement science developed in this project is being used to support work in EU specialised centres, such as through dissemination activities organised by the Fraunhofer Institute (e.g. The World Congress on Regenerative Medicine), and by direct links with technology innovation centres.

Support for the social acceptance of regenerative medicine products:

This project developed measurement techniques to ensure products are safe and well characterised when used in humans.



Quantifying the time dependent changes in cell shape that occur as they attach to surfaces can be exploited to enhance the biocompatibility of surfaces.



CARS imaging of fibroblasts grown in fibrin gel at day 7.



CARS imaging of fibroblasts grown in fibrin gel at day 21-note the collagen produced.

Meeting European legislation

Developed measurement technologies and methodologies that enable European companies to meet the requirements for cell characterisation, as defined in the Advanced Therapy Medicinal Product Directive 2009/120/EC. They have also input into standardisation bodies; ISO Technical Committee 150 - *Implants for surgery*, ASTM International Committee F04 - *Medical & Surgical Materials & Devices* and BSI Committee RGM/1 - *Regenerative Medicine*.

Centre of Excellence for regenerative medicine

Enabled project partners to act as a virtual 'Centre of Excellence' for regenerative medicine and the characterisation of cell containing products. The project has also provided stakeholders with access to cutting edge measurement technology such as Two-Photon Excitation Fluorescence Microscopy, CARS, MALDI and Desorption Electrospray Ionization (DESI) Mass-Spectrometry.

Support to the medical community

Supported medical companies in the development of new biomaterial surfaces that cells can easily adhere to, using traditional textile technologies.

Helped cell therapy companies to develop cell characterisation techniques that do not rely on animal testing.

Enabled spectroscopic techniques (developed for studying cell culture biomarker molecules) to be applied in the detection of counterfeit medicines.

Allowed companies using fluorescent biomarkers to obtain reliable results and to have more confidence in their analysis.

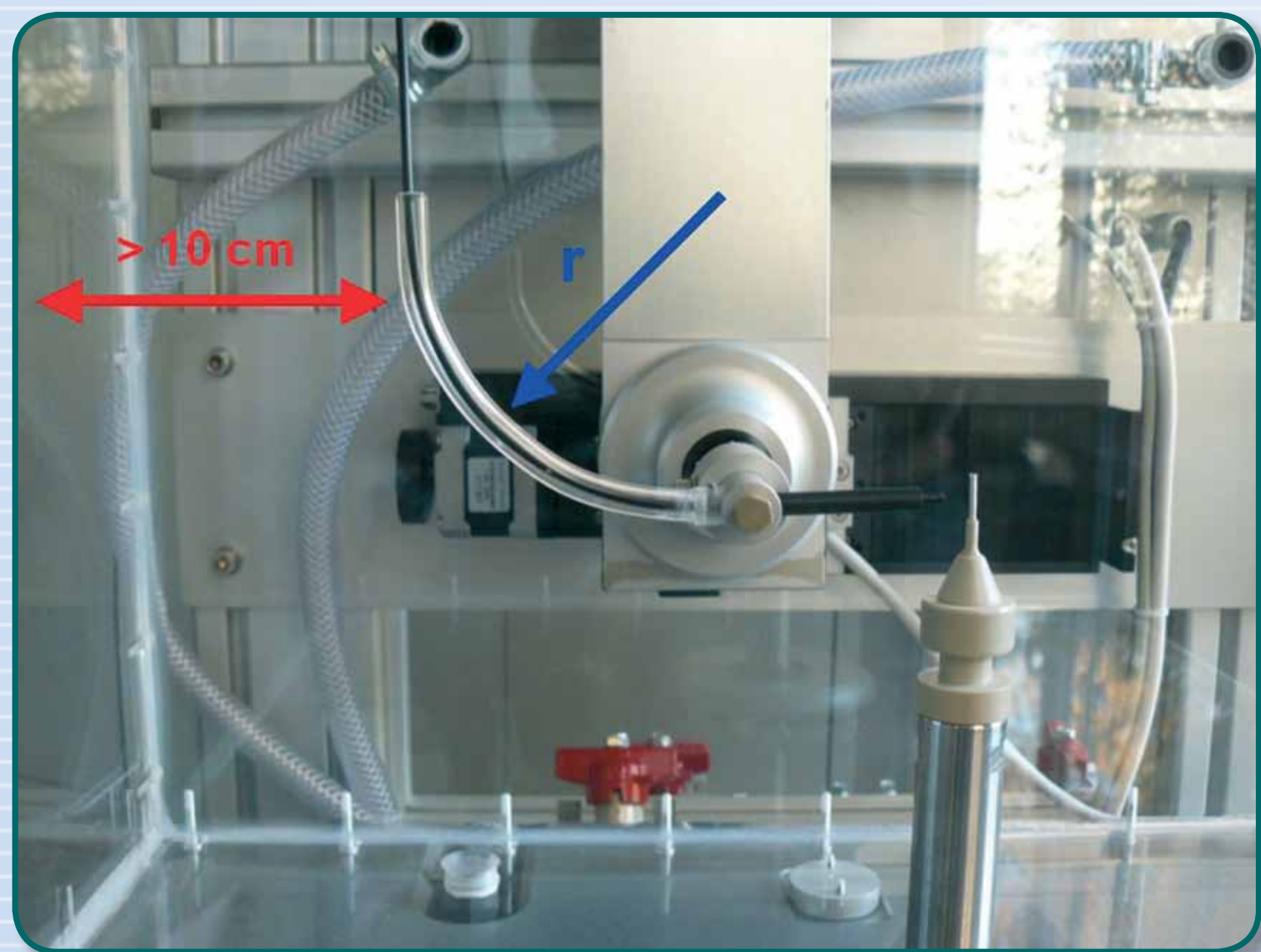
Increasing cancer treatment efficacy using 3D brachytherapy

The need for the project

Brachytherapy is a cancer radiotherapy approach where small, sealed radioactive sources are placed inside, or in close proximity to, the area requiring treatment. It is commonly used to treat cervical, prostate, breast, and skin cancer and can be used alone or in combination with other therapies.

In Europe, approximately 100,000 patients per year are treated using brachytherapy, but in order to optimise cancer treatment and satisfy the recommendations of the International Atomic Energy Agency (IAEA) TRS-398 2000, an improvement in the accuracy of brachytherapy dosimetry is required.

This project aimed to address this issue by establishing traceable measurements of brachytherapy radiation sources using absorbed dose to water (D_w) primary standards. These new D_w standards should simplify existing measurements and reduce dose uncertainty to below 5%, at the clinical level.



The PTB water phantom for LDR sources for operation with a small scintillation detector.

Technical achievements

A number of primary standards have been designed and built for measurements of D_w imparted by brachytherapy sources using low dose-rate (LDR) or high dose-rate (HDR) regimens. For LDR dosimetry, three standards were established by participating National Metrology Institutes, based on ionometric methods. For HDR dosimetry, two primary standards based on water calorimetry were developed along with two other HDR standards based on graphite calorimetry.

The project developed a calibration chain optimised to transfer the new reference quantity D_w to end-users. In particular, a measurement procedure was developed for selected models of well-type chambers. In order to select the models of well-type chambers a questionnaire was distributed to irradiation facilities using brachytherapy dosimetry at secondary standards laboratories and medical centres in Europe - 137 centres responded from 9 different countries.

To support the link between the current metrology (air kerma standards) and the new D_w standards, the dose rate constant (Λ) was re-evaluated for all brachytherapy sources used within the project.

The project also developed suitable portable methods to improve the verification of the 3D dose distributions of brachytherapy sources in water or in water equivalent phantoms.



Graphite calorimeter developed by NPL with Nucletron microSelection Classic afterloader. The treatment unit is connected to the calorimeter via a transfer tube.

New D_w reference standards

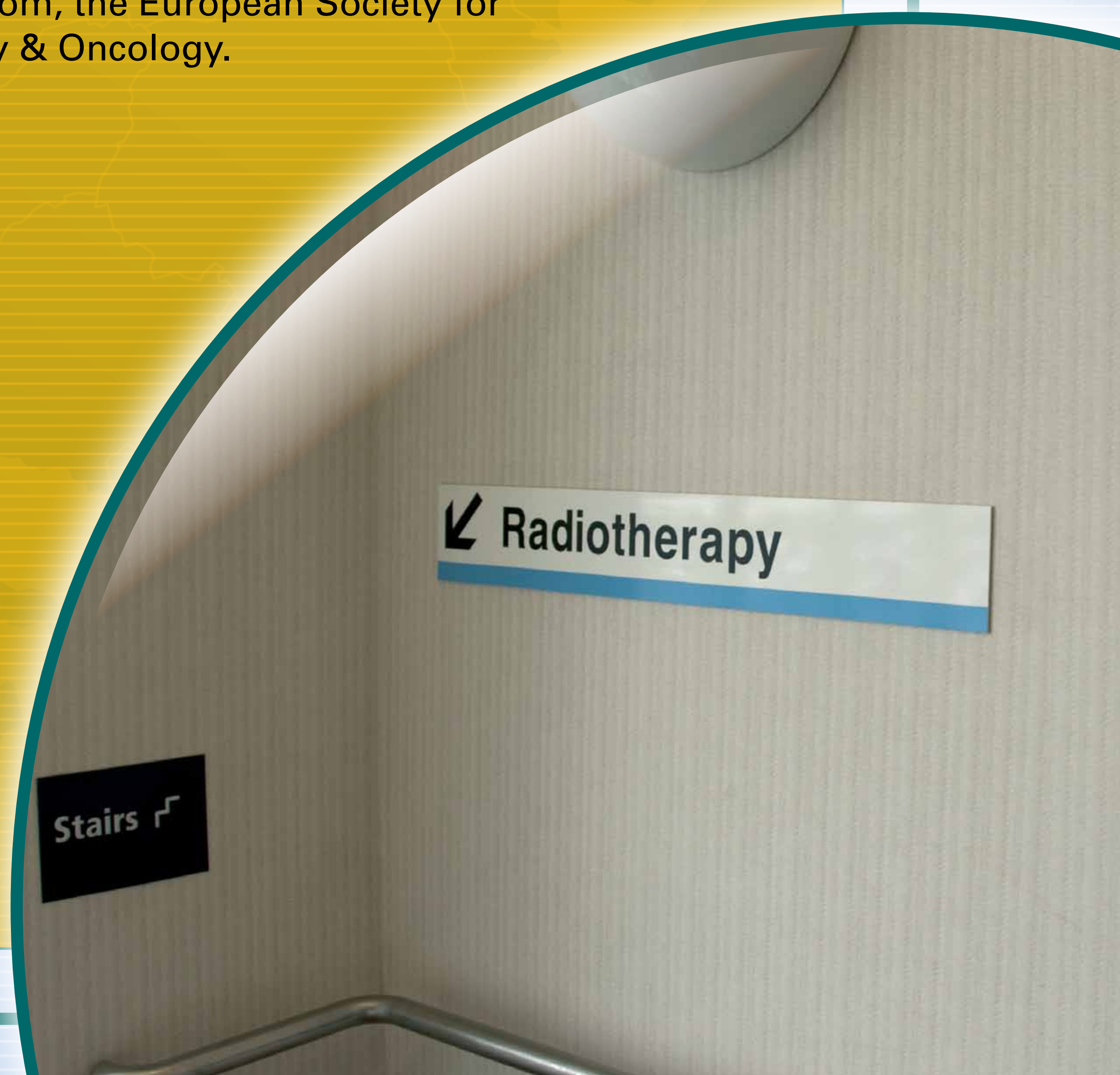
Developed seven new D_w primary standards for measurements of brachytherapy sources at low and high dose-rate regimens. These were then linked to existing air kerma standards by validating the use of the dose rate constant (Λ). The project also developed methods to verify the dose distribution of brachytherapy sources in water or water equivalent phantoms.

Input to standardisation

The project results have input into an AFNOR working group, which is a mirror of ISO TC 85/SC2 WG22 'Dosimetry and related protocols in medical applications of ionizing radiation' and have fed into the revision of DIN standard 6809-2 'Clinical dosimetry; Brachytherapy with sealed gamma sources'.

The results of the project have been presented to the IAEA, where the move from air kerma to D_w standards for dosimetry for brachytherapy was discussed with the support of the medical community.

The project also has the support of, and has benefited from input from, the European Society for Radiotherapy & Oncology.



External Beam Cancer Therapy

The need for the project

Approximately 1.3 million people will die due to cancer, in Europe, in 2011. Therefore, a way to improve patient treatment and increase survival is urgently needed.

This project aimed to improve dosimetry for high-intensity therapeutic ultrasound (HITU) and modern ionising beam radiotherapies. Therapies such as Intensity Modulated Radiation Therapy (IMRT) can be used to provide a more conformal dose distribution than conventional radiotherapy, thereby increasing the dose of radiotherapy to tumours, while sparing healthy tissue. However, to support the use of HITU further knowledge on the temperature of the 'dose' and its distribution during administration is required.

In addition, both HITU and IMRT techniques pose a challenge for dosimetry, due to the small size of the radiation field. The International Commission on Radiation Units states that the applied dose should have an uncertainty of less than 2.5%, but to achieve this, both the radiation and field size need to be precisely quantified.

Technical achievements

Dosimetry in small fields:

The measurand absorbed dose to water, (D_w) was measured using a graphite calorimeter and a water calorimeter with beam qualities 6, 10 and 12 MVX with field sizes of 10 cm x 10 cm down to 3 cm x 3 cm. The obtained uncertainties were as low as 0.3%.



Radiography of the core of the graphite calorimeter (LNHB) for the realisation of D_w in photon fields with a size down to 2 cm x 2 cm.

The calibration coefficients of ion chambers and the responses of alanine dosimeters determined in these fields showed no dependence on the field size within the limits of uncertainty. By Monte-Carlo simulations, the response of alanine was extrapolated for smaller fields (1 cm x 1 cm). No significant size dependence was found within the uncertainties. A diamond detector, developed in co-operation with the Roma Tre University, Italy, was used for measurements in the fields 1 cm x 1 cm, 6 and 10 MVX in comparison with alanine, and demonstrated limits of uncertainty of less than 1%.

Dosimetry for hadron therapy:

Measurements to demonstrate D_w in a 12C-beam with 280 MeV/u at GSI: The Centre for Heavy Ion Research in Darmstadt were performed successfully. The calibration coefficients of ion chambers were determined and were in agreement with the uncertainties given in the International Atomic Energy Agency (IAEA) report TRS 398.

HITU:

Better sensors for measuring spatial pressure distributions and total acoustic output power of HITU transducers were developed with the aim of determining the temperature applied with a higher accuracy. The first inter-laboratory comparison of HITU power measurement methods was successfully carried out.



Transportable water calorimeter (PTB) at the 12C beam line at GSI in Darmstadt, Germany.

Input to dosimetry standards

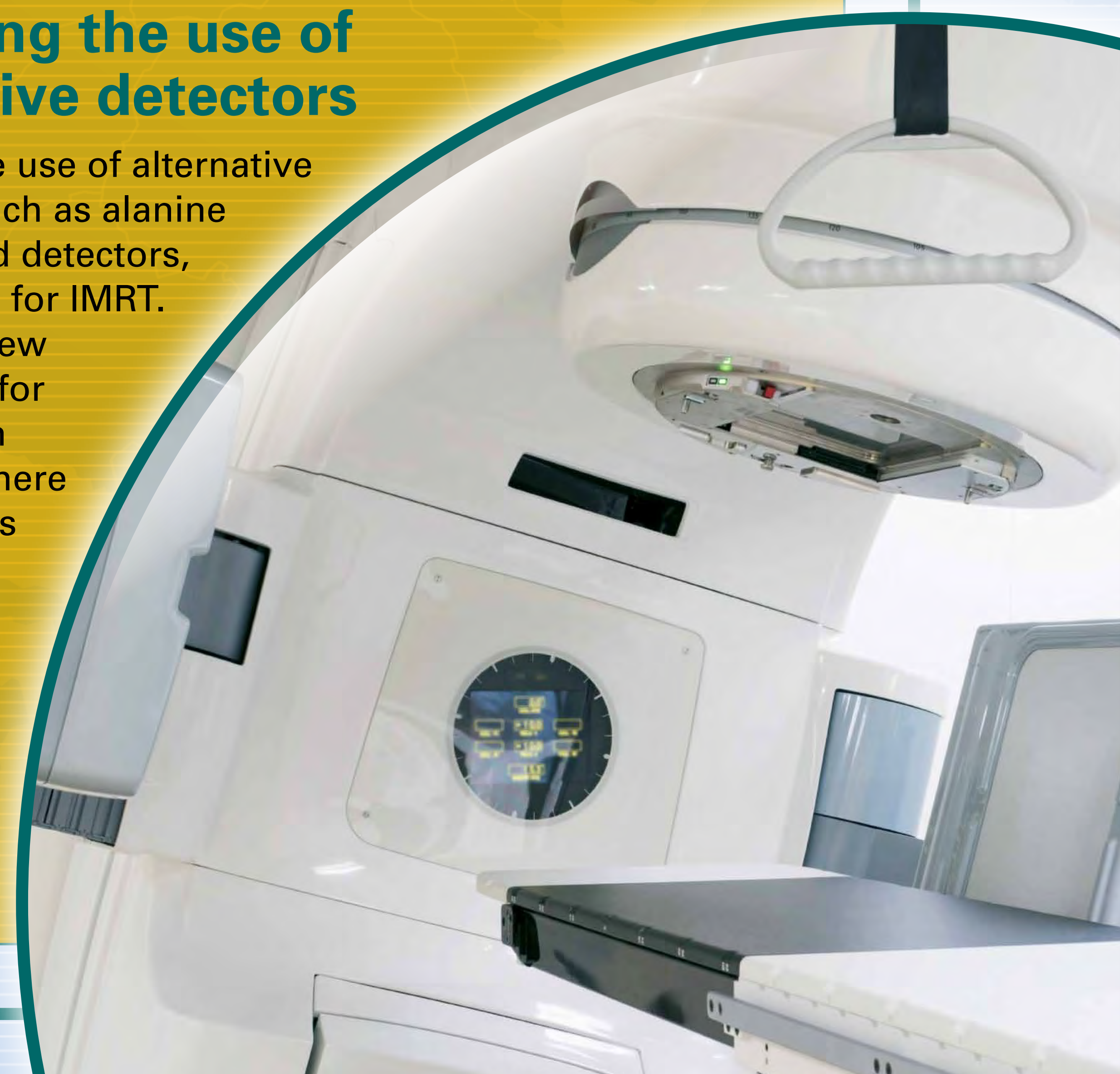
The project's beam quality correction factors (kQ factors) are being incorporated into an update of the German standard DIN 6800-2 'Procedures of dosimetry with probe type detectors for photon and electron radiation - Part 2'. The kQ factors will also be incorporated in a future version of the IAEA report TRS 398 'Absorbed Dose Determination in External Beam Radiotherapy: An International Code of Practice for Dosimetry based on Standards of Absorbed Dose to Water.'

Verifying dose delivery

The results of the project have supported two national dose verification studies for IMRT. In Belgium the quality assurance study BELdART will involve virtually all Belgian therapy centres. Whilst in the UK, a national audit was performed by the National Health Service and NPL, the UK's National Metrology Institute, between June 2009 and March 2010. The aim of both studies was to provide an independent check of IMRT delivery.

Validating the use of alternative detectors

Validated the use of alternative detectors, such as alanine and diamond detectors, as standards for IMRT. This offers new possibilities for calibration in situations where ion chambers cannot be used.



Traceable measurements for biospecies and ion activity in clinical chemistry

The need for the project

Traceable and comparable measurements in clinical chemistry are a mandatory requirement of EU legislation (In Vitro Diagnostic Medical Devices 'IVD' - Directive 98/79/EC). However, the measurement of biospecies in blood serum is currently restricted to the determination of their total amount, although it is well understood that a 'clinical' effect often depends on the identity and quantity of biospecies or ion activities rather than the total amount.

What is required is the ability to identify and quantify biospecies and measure ion activity in clinical samples. This is particularly important for calcium, one of the most frequently measured analytes in clinical chemistry, and heteroatom-containing drugs (e.g. sulphur and selenium metabolites), which are used to sensitise cancer cells to chemotherapy.

This project aimed to provide internationally accepted reference points to calibrate new and existing measurement systems in the medical diagnostic field.



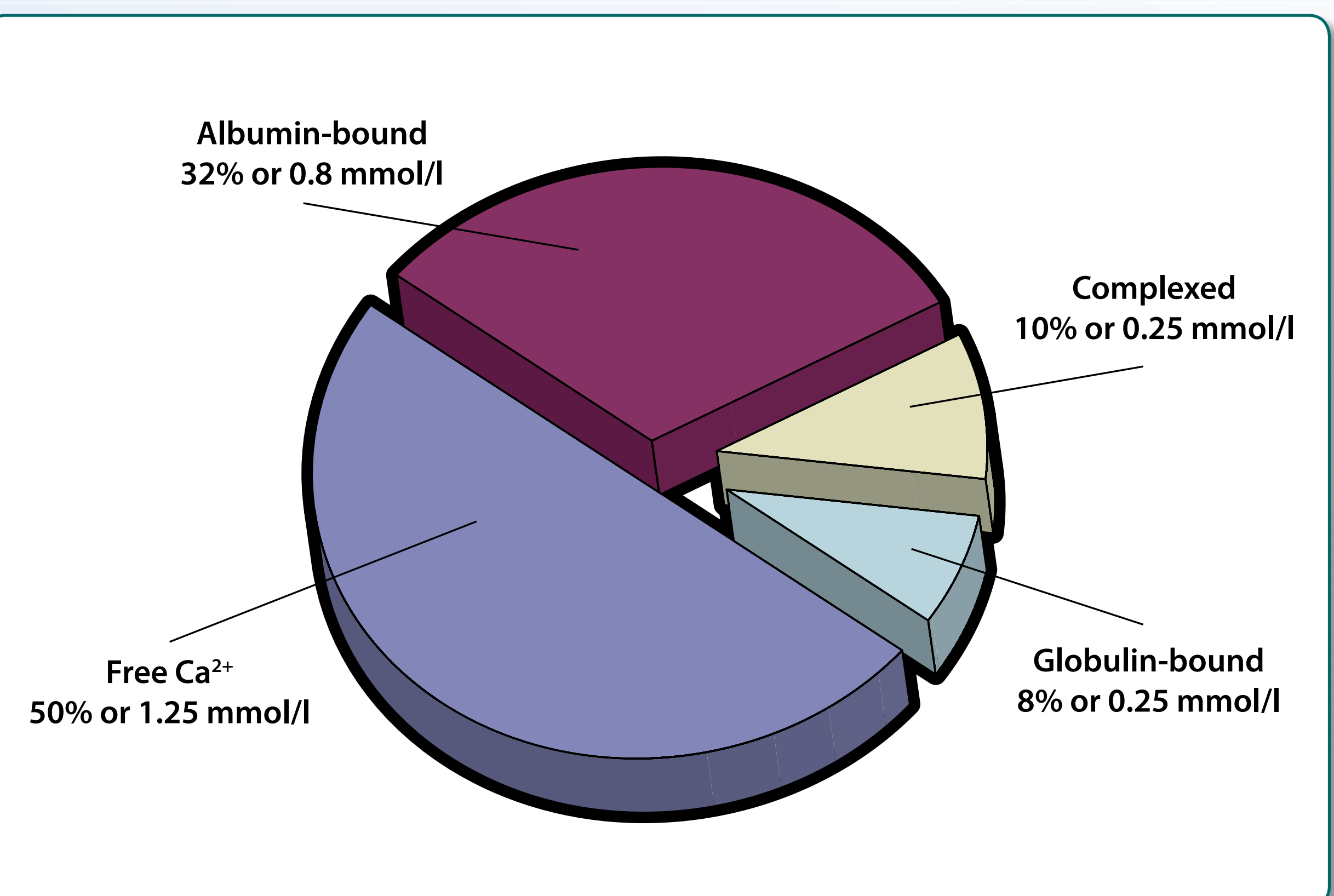
Photo of the closed loop measurement setup for conductivity measurements in purified water with controlled electrolytic contamination: the temperature control box can be seen on the right hand side, containing the conductivity measurement cell, the pump and the contamination vessel; the right hand side shows the purification system for highly purified water, the dosage unit for contamination and the ion chromatograph.

Technical achievements

Reference methodology for the accurate trace measurement and identification of toxic and essential heteroatom-containing species in human serum: selenomethionine (SeMet) and methyl-Se-cysteine were selected as target analytes. ⁷⁶Se-enriched SeMet was produced and characterised as a labelled spike, which was then used for species-specific isotope-dilution mass spectrometry method development. Enzymatic and acid hydrolysis procedures were also developed to extract and quantify SeMet from albumin. A largely accelerated extraction procedure was achieved by microwave-assisted extraction without compromising SeMet extraction efficiency.

A system for the SI traceable measurement of the ionic species activity in physiological matrices: the ions focused on were sodium, potassium, chlorine, magnesium and calcium. High purity materials were characterised on a primary level and subsequently used to prepare gravimetric mixtures as calibration standards. The unknown activities, the free biological active part of the compounds in these mixtures, were calculated based on established model calculations. Measurement systems as well as measurement methods including defined procedures for signal acquisitions and data evaluation, were developed in order to quantify the ion activity and measurement uncertainty.

An experimental set-up for low range conductivity of highly purified water at a primary level: the conductivity of highly purified water is an indispensable reference point for activity measurements and quality control parameters for pharmaceutical purposes.



Pie chart indicating different calcium species in serum. Only the ionised free calcium (Ca²⁺) is physiologically relevant.

Input to clinical trials

Developed methods that have been used in an ongoing European Clinical Trial *Selenium and Prostate Cancer: Clinical Trial on Availability to Prostate Tissue and Effects on Gene Expression*. The study is run by Wageningen University, the Netherlands, and is a double-blind, randomised, placebo-controlled intervention trial aimed at examining the effects of selenium supplementation. The trial examines the relationship between dietary selenium intake and changes in gene expression profiles that might be responsible for selenium-induced chemoprevention.

Intercomparison of SeMet measurements

Measurement methods for SeMet in human serum have been validated by an international intercomparison. The intercomparison (EURAMET TC-MC project 1165) included National Metrology Institutes and expert laboratories (e.g. NIMT Thailand, CCS Switzerland, DIMCI/DQUIM Brasil).

New measurement procedures

Submitted new measurement procedures for ion activity to the Joint Committee for Traceability in Laboratory Medicine (JCTLM).



Joint Research Project (JRP) Short Name: TRACEBIOACTIVITY • JRP-Coordinator: Bernd Güttler (PTB) • JRP-Partners: DFM (Denmark), EJPD (Switzerland), INRIM (Italy), LGC (UK), LNE (France), Metroser (Estonia), PTB (Germany), SMU (Slovakia), SP (Sweden), TUBITAK UME (Turkey), UT (Estonia)

Traceability of complex biomolecules and biomarkers in diagnostics

The need for the project

The International Federation of Clinical Chemists (IFCC) and the Joint Committee for Traceability in Laboratory Medicine (JCTLM) have highlighted the need to develop reference measurement systems to provide traceable values for complex biomolecules, such as disease state protein biomarkers. This should also enable in vitro diagnostic (IVD) and clinical measurement comparability and improve diagnostic efficiency and reliability.

However, few reference measurement procedures exist for protein biomarkers and biomolecular measurements and consequently standards can be affected by multiple parameters that need to be considered in the establishment of a traceability chain.

The critical measurement is not the total amount of a protein biomarker but the quantity of 'active/functional' component. A protein's structure, folding state and interactions with other proteins/ligands all define its activity and can influence the measurement result and its diagnostic 'clinical' value.

This project aimed to address these issues by developing measurement procedures for complex protein biomarkers in order to provide SI traceable results.

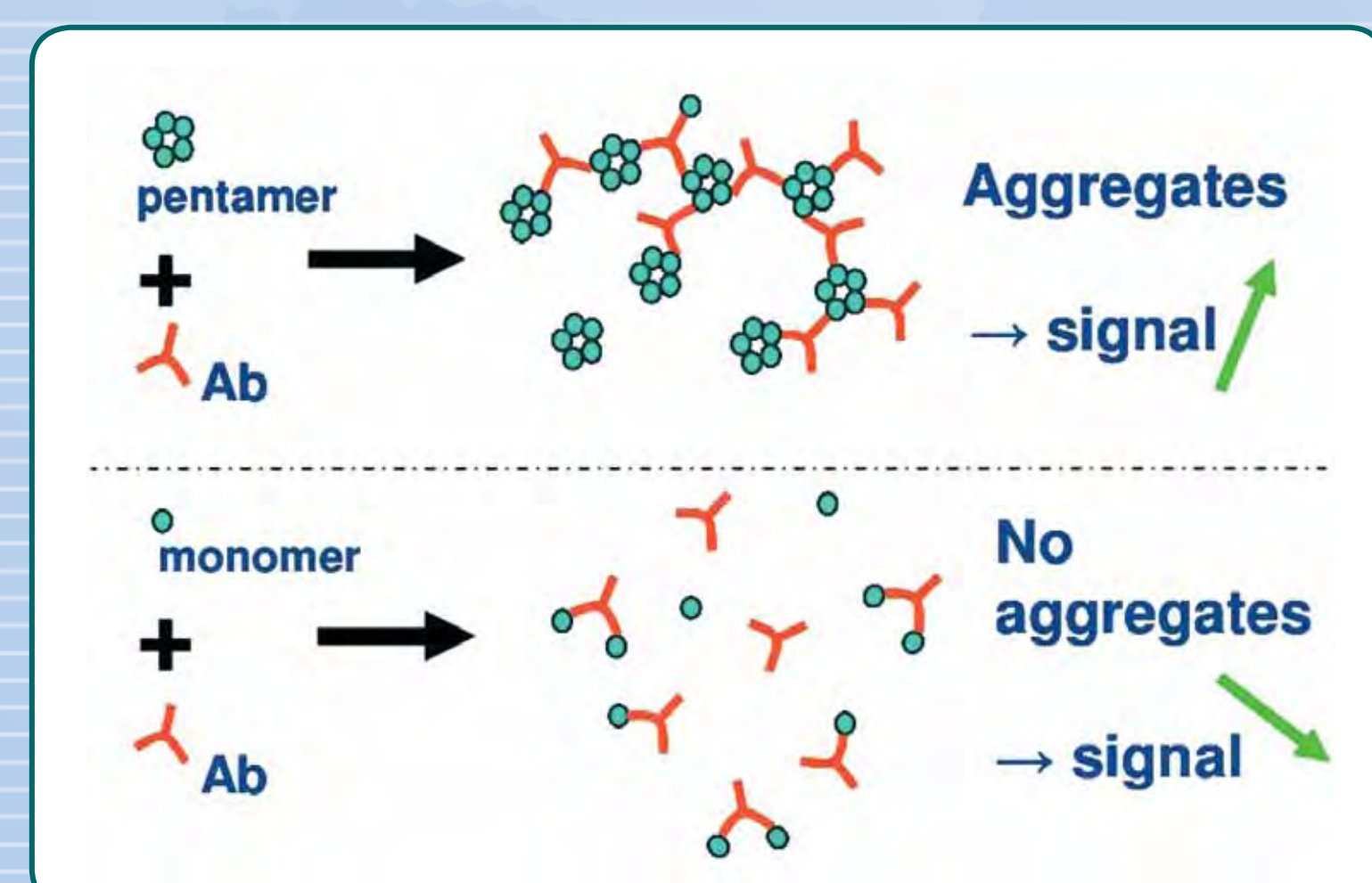
Technical achievements

The project's most significant achievement is the successful demonstration of the feasibility of developing reference measurements, that deliver SI traceable values and reference materials for two clinically important biomarkers; human growth hormone (hGH) and C-reactive protein (CRP).

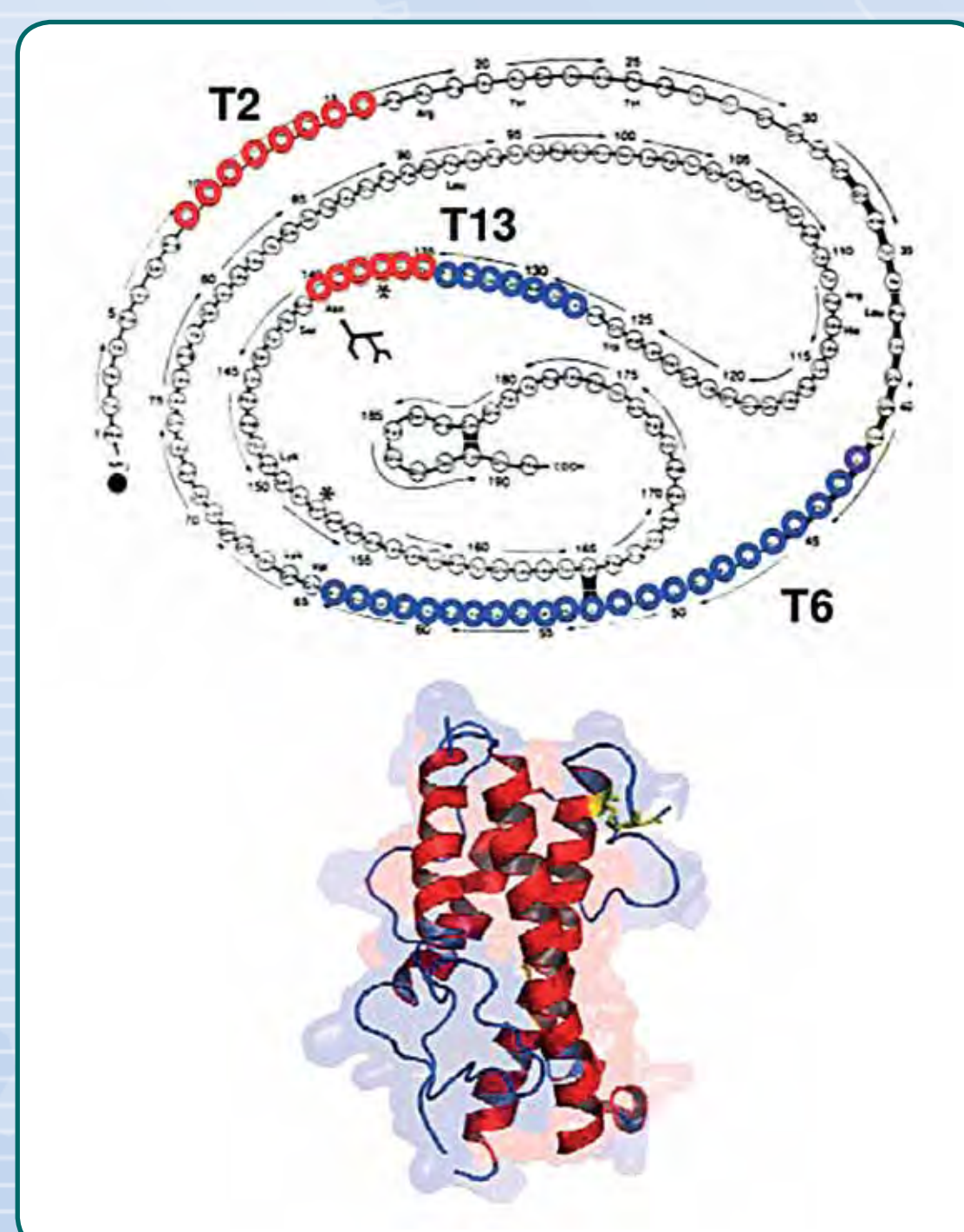
The project developed, validated and published strategies for the application of isotope dilution mass spectrometry (IDMS) methods for the quantification of hGH and CRP in purified materials and serum matrixes.

Development of robust and selective enzymatic digestion and cleanup protocols enabling assignment of SI traceable values for hGH in serum by IDMS using isotopically enriched peptides and proteins as internal standards.

Method development and optimisation for isoform profiling and quantification of oligomeric and aggregation state which has influenced immunoactivity in hGH and CRP reference preparations.



The impact of CRP oligomeric state on immunoassay response.



Amino acid sequence highlighting tryptic peptides used for quantification, and ribbon structure of Human Growth Hormone (22kDa form).

Evaluation of advanced mass spectrometry-based techniques for the elucidation of protein structure:

- Hydrogen Deuterium Exchange (HDX) in combination with proteolysis mass spectroscopy and Ion Mobility Mass Spectroscopy (IMS) were optimised to assess structural differences in the currently available reference standards for hGH
- the potential for HDX and IMS for quantification of protein folding states and protein ligand interaction was demonstrated
- the significant influence of sample preparation, heterogeneous isoforms and structures on clinical immunoassay response for both hGH and CRP was demonstrated

Input to standardisation & regulation

The World Health Organization (WHO) has expressed an interest in the project's multiparametric protein quantification providing SI traceable values for value assignment of relevant WHO International Standards for biologicals. Project results may be acknowledged in future revisions of ISO TC212 'Clinical laboratory testing and in vitro diagnostic test systems' documentary standards.

Supporting the clinical community

Engaged with key clinical and metrology stakeholders, including the IFCC, JCTLM and Consultative Committee for Amount of Substance (CCQM) BioAnalysis working group.

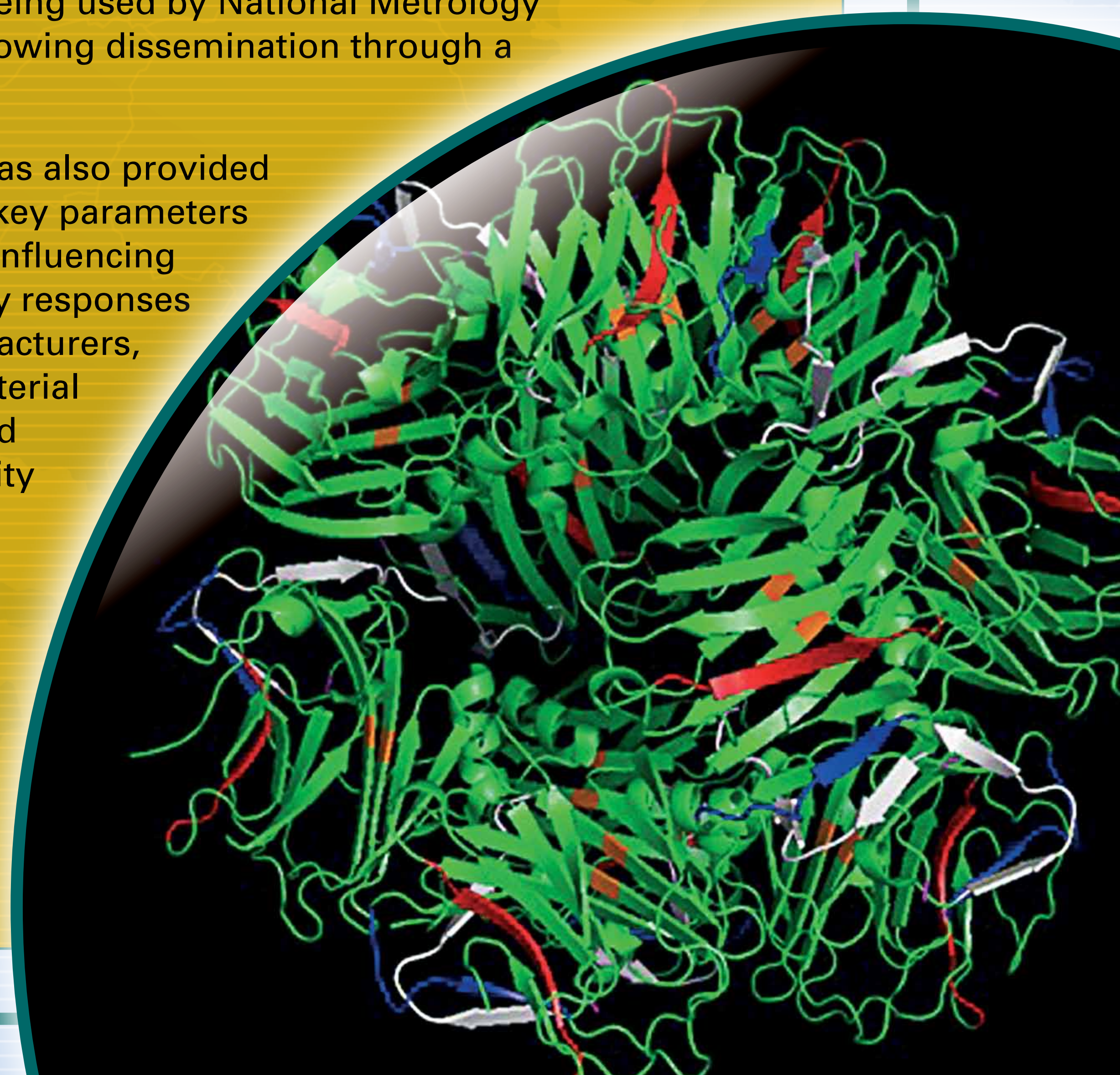
The project's JRP-Partners participated in the IFCC Scientific Division, IFCC hGH and plasma proteins working groups. The results of the project have input in to IFCC guidance, clinical test kits and the organisation of clinical commutability studies.

New reference methods

The IDMS method developed for protein quantification is currently being used by National Metrology Institutes following dissemination through a CCQM study.

The project has also provided guidance on key parameters identified as influencing immunoassay responses to IVD manufacturers, reference material producers and external quality assurance organisers.

Ribbon structure of pentameric form of C-reactive protein.



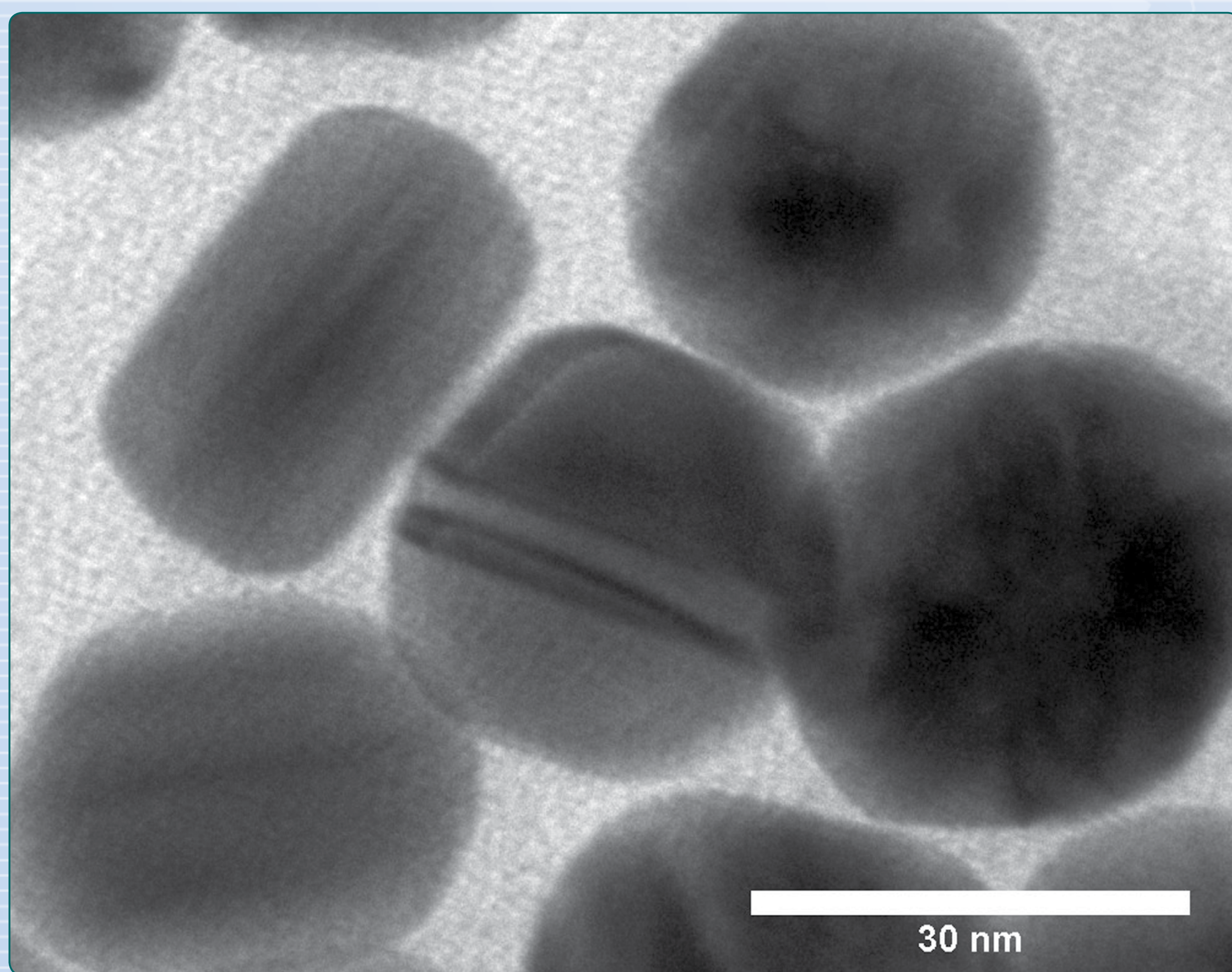
Traceable characterisation of nanoparticles

The need for the project

Nanoparticles have many applications, e.g. in suspensions in the ink industry, in drug delivery or as diagnostic agents for the pharmaceutical industry and in novel composite materials to improve properties such as strength and electrical conductivity.

However, at the start of this project, there were no formally recognised, traceable calibration standards and only one European National Metrology Institute could measure the size of spherical nanoparticles.

This project aimed to provide traceable calibration of nanoparticles smaller than 100 nm with better than 1 nm accuracy. It focused on nanoparticle sample preparation, traceable measurement of spherical nanoparticles and high aspect ratio nanoparticles.



TEM image of 30 nm gold nanoparticles. Note that the shape of the particles is not quite spherical.

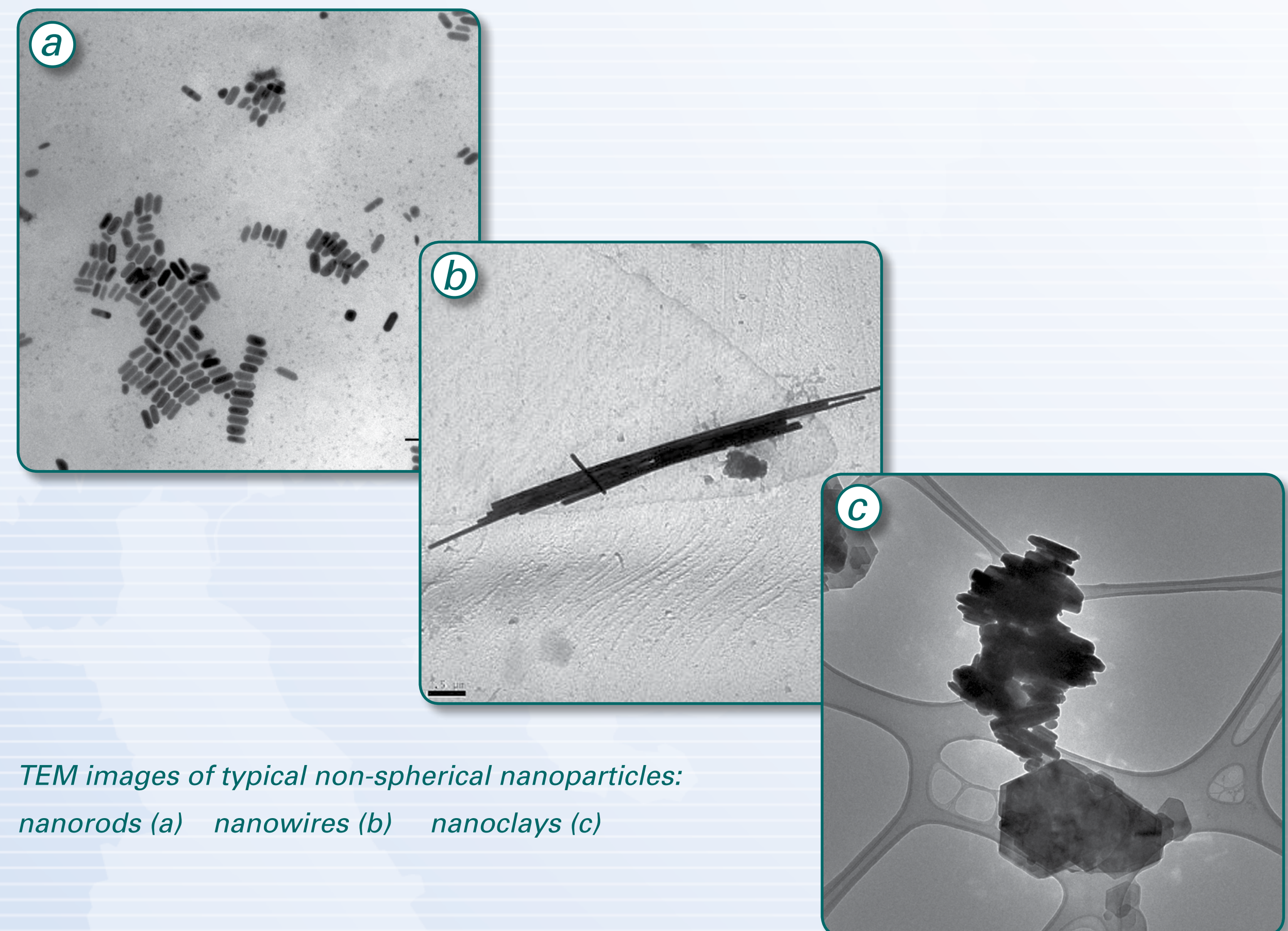
Technical achievements

This project established dimensional traceability for a range of nanoparticle measurement techniques:

- Scanning Probe Microscopy (SPM)
- Scanning Transmission Electron Microscopy (STEM)
- Transmission Electron Microscopy (TEM)
- Scanning Electron Microscopy (SEM)
- Dynamic Light Scattering (DLS)
- Small Angle X-ray Scattering (SAXS)

The project identified artefacts for these techniques and conducted inter-laboratory comparisons of particle size measurements. The results demonstrated an uncertainty of less than 1 nm.

A new prototype instrument for aerosol particle measurement was also produced and validated: SCAR (Single Charge Aerosol Reference), which produces singly charged particles in a wide particle size range.



TEM images of typical non-spherical nanoparticles: nanorods (a) nanowires (b) nanoclays (c)

Reference materials

Contributed to the first European Reference materials for Nanoparticles. To achieve this, four of the JRP-Partners participated in a validation exercise for nanoparticle measurement techniques (i.e. DLS, STEM, SPM, SEM, and SAXS).

Input to standards

The project results have fed into two international standard committees through JRP-Partner (CEM, NPL, INRIM – TC229 and MIKES – TC24) membership:

ISO TC229 Nanotechnologies

ISO TC24 Particle characterisation including sieving

Good Practice Guides

Prepared a Good Practice Guide on the accurate measurement of spherical nanoparticles, to enable end-users to set up and carry out measurement of nanoparticles with minimal uncertainty.

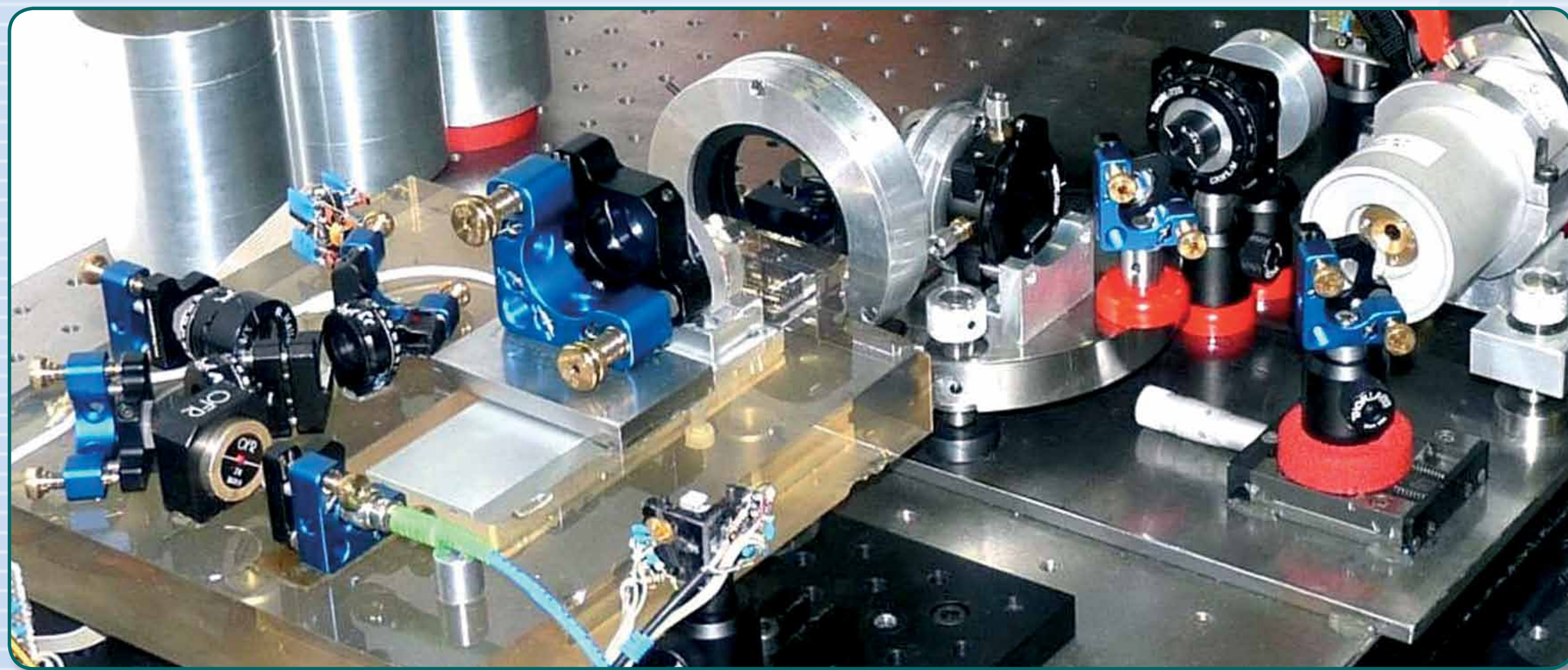
New traceability routes for nanometrology

The need for the project

As nanotechnologies and ICT (Information and Communication Technologies) play increasingly crucial roles in modern life, the demand for increased accuracy of dimensional measurements is rapidly growing.

One example is in semiconductor manufacturing, where laser interferometers are currently the essential measurement tool. The increased use of double-patterning techniques with a reproducibility of about 0.3 nm for mask metrology tools require more accurate techniques than are currently available.

This project aimed to achieve a 10 pm accuracy for displacement metrology by developing and refining next generation optical interferometers. The challenge was to reduce the uncertainty values by one order of magnitude with respect to the present state-of-the-art.

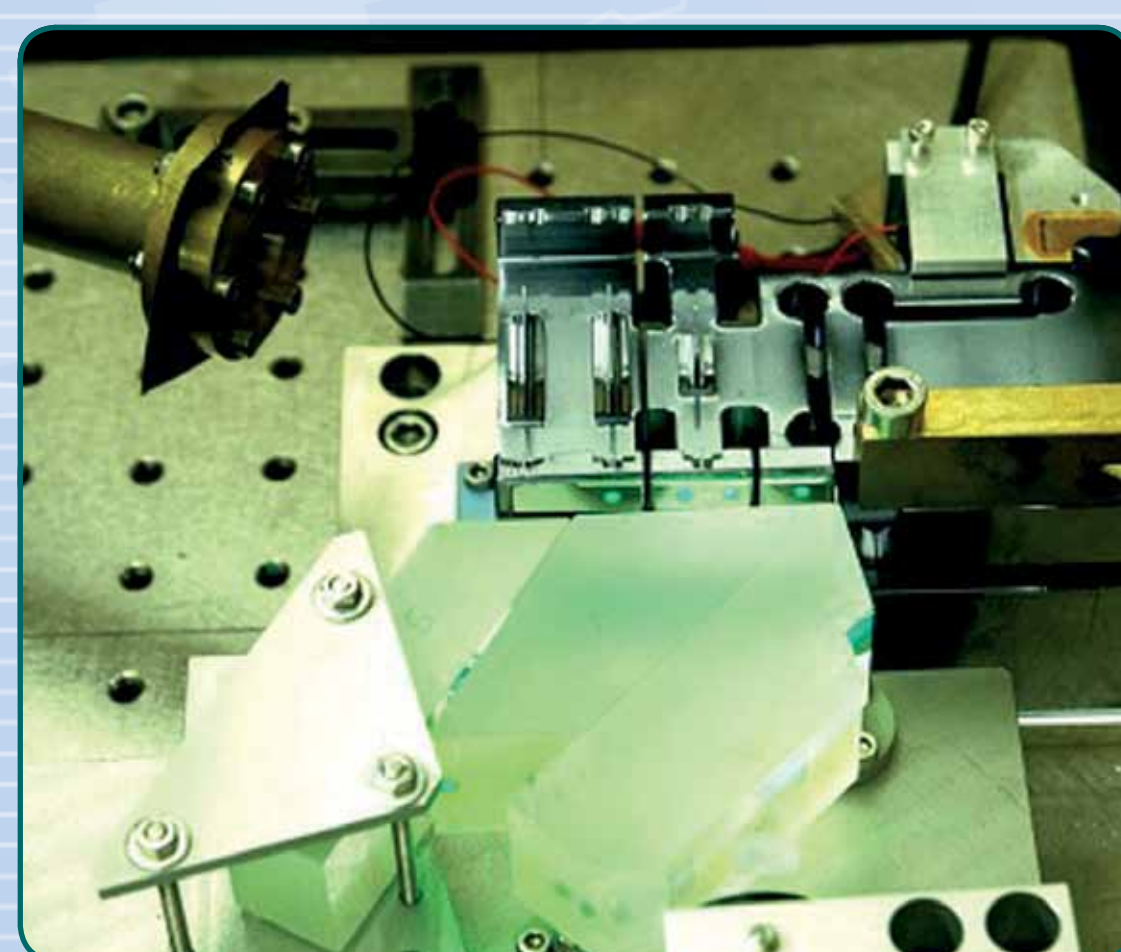


Optical set-up for the comparison between the EOM-based heterodyne interferometer (in the background) and the Transfer Standard based on multipass interferometer (in the foreground).

Technical achievements

The project produced six high-resolution interferometers, based on the different techniques developed in the project: including a cost-effective method that uses combined capacitive distance sensors for modelling and correction of interferometer non-linearity with 10 pm accuracy.

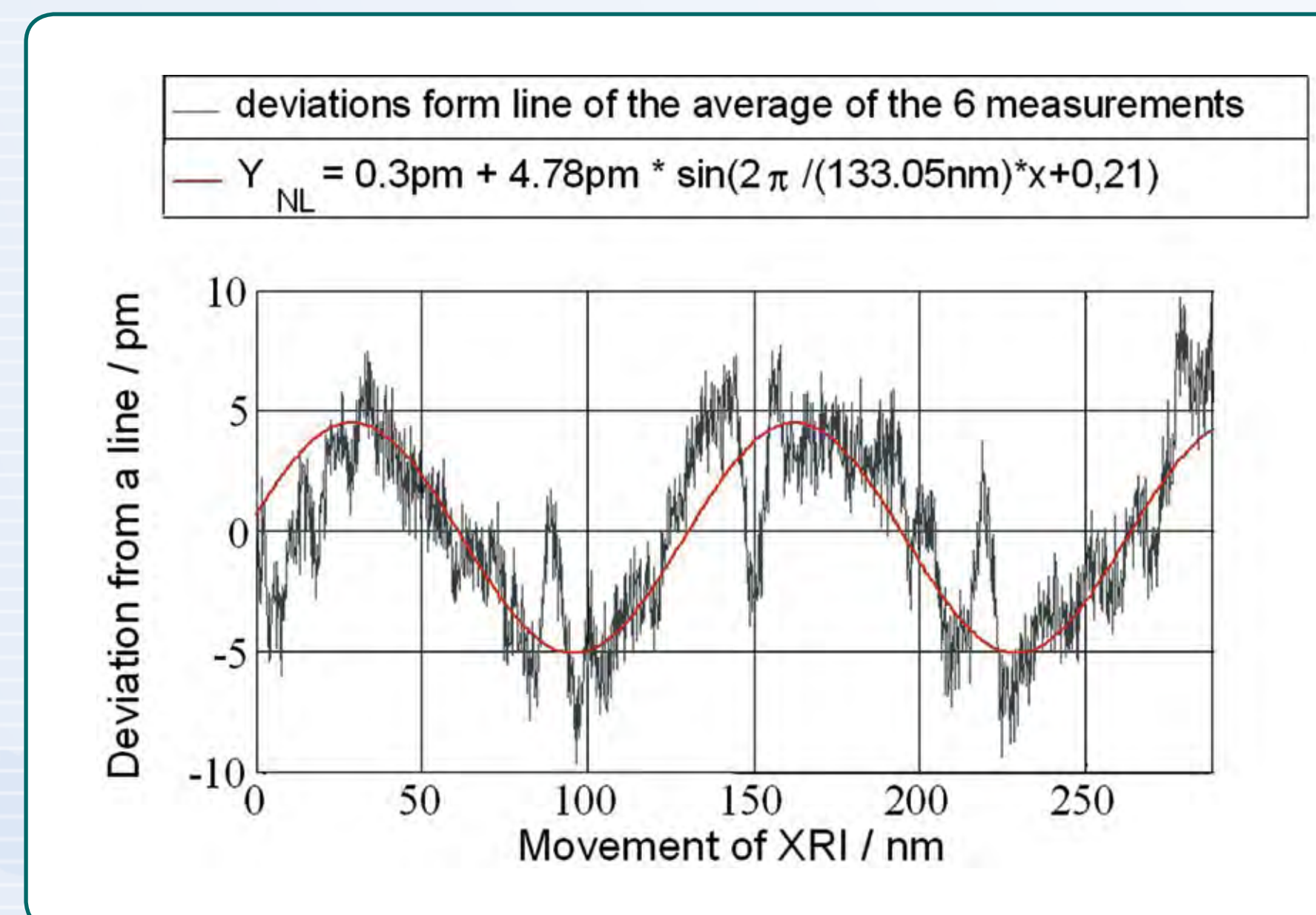
The performance of five of these optical interferometers has been verified using the NPL x-ray interferometry facility which was redesigned and refined. A fast phasemeter was developed, that will be used to upgrade PTB linescales and angle measurement facilities. Also a differential Fabry-Pérot interferometer, used for ultra-small angle measurements, was produced.



The separated beams interferometer (left) facing the x-ray interferometer (black silicon on the right) during the comparison.

Both the x-ray interferometer and the Fabry-Pérot interferometer have supported the results of the JRP NAH, where they will be used to accurately measure the lattice constant and determine the shape of the silicon-28 sphere, respectively.

Guidelines on the use of interferometers for traceability at the nanoscale, based on the experience gained within the project, will be available for researchers on the Nanotrace website.



Comparison between a separated beams interferometer and an x-ray interferometer. A maximum ± 5 pm error is observed.

New metrology tools

Produced a phasemeter and interferometer that can be used to improve the line scale comparator at PTB, Germany's National Metrology Institute, by reducing measurement uncertainty and short periodic errors to below 0.1 nm.

The phasemeter will also be used to improve the speed and accuracy of Atomic Force Microscopes and Scanning Electron Microscopes for the determination of line width, form and fluctuation, where the required accuracy is below 0.1 nm.

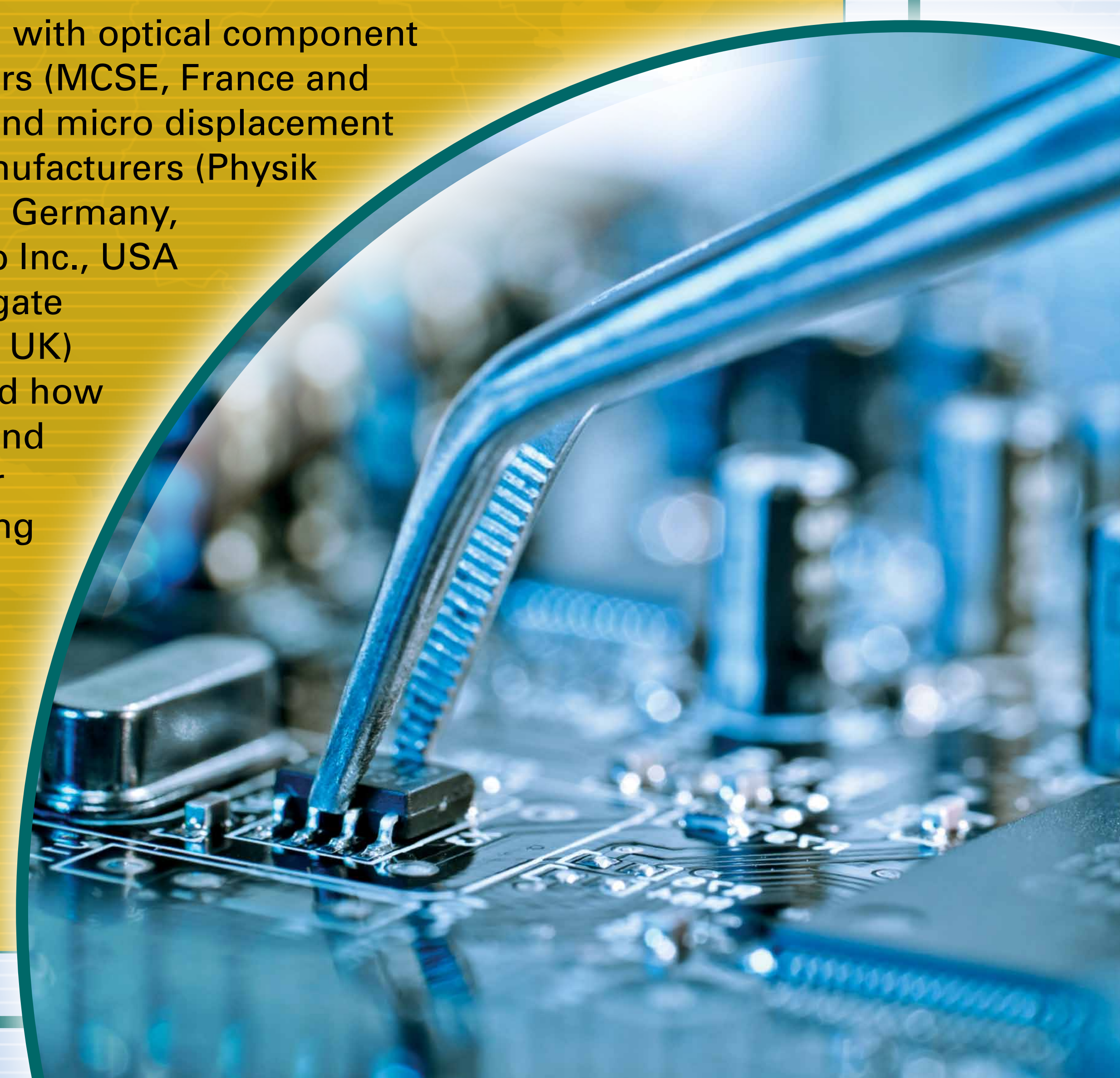
The x-ray interferometry facility will be available for measuring errors in optical interferometers and will be used to characterise instruments used in the semiconductor industry.

New transfer standard

Produced a new transfer standard (transportable actuator), which will be used for the validation of accurate displacement sensors such as interferometric, confocal and capacitive sensors, used in industrial measurements and for fundamental research.

Collaboration with manufacturers

Collaborated with optical component manufacturers (MCSE, France and SILO, Italy) and micro displacement actuator manufacturers (Physik Instrumente, Germany, Mad City Lab Inc., USA and Queensgate Instruments, UK) and identified how to improve and support their manufacturing processes.



Metrology for new industrial measurement technologies

The need for the project

Three main challenges in large-scale dimensional measurements prevent manufacturers from testing exactly what rolls off their assembly lines – e.g. wind turbine blades, machine tool parts, automobile and aircraft parts:

- a lack of calibrated measurement standards
- inadequate measuring techniques to implement task-specific measurement uncertainty of large parts directly on the shop floor
- a lack of internet infrastructure for online validation of Coordinate-Measuring Machine (CMM) evaluation software and other geometrical evaluation algorithms

This project aimed to improve the understanding of large scale dimensional measurements by developing new and traceable measurement technologies – different advanced measurement standards, sophisticated measuring techniques, and good practice guides on the use of indoor GPS, lasertracers and other mobile measuring technologies.



Measurement standards for traceability measurement and measuring system performance verification; from left: tetrahedron standard, large involute gear standard, freeform standard.

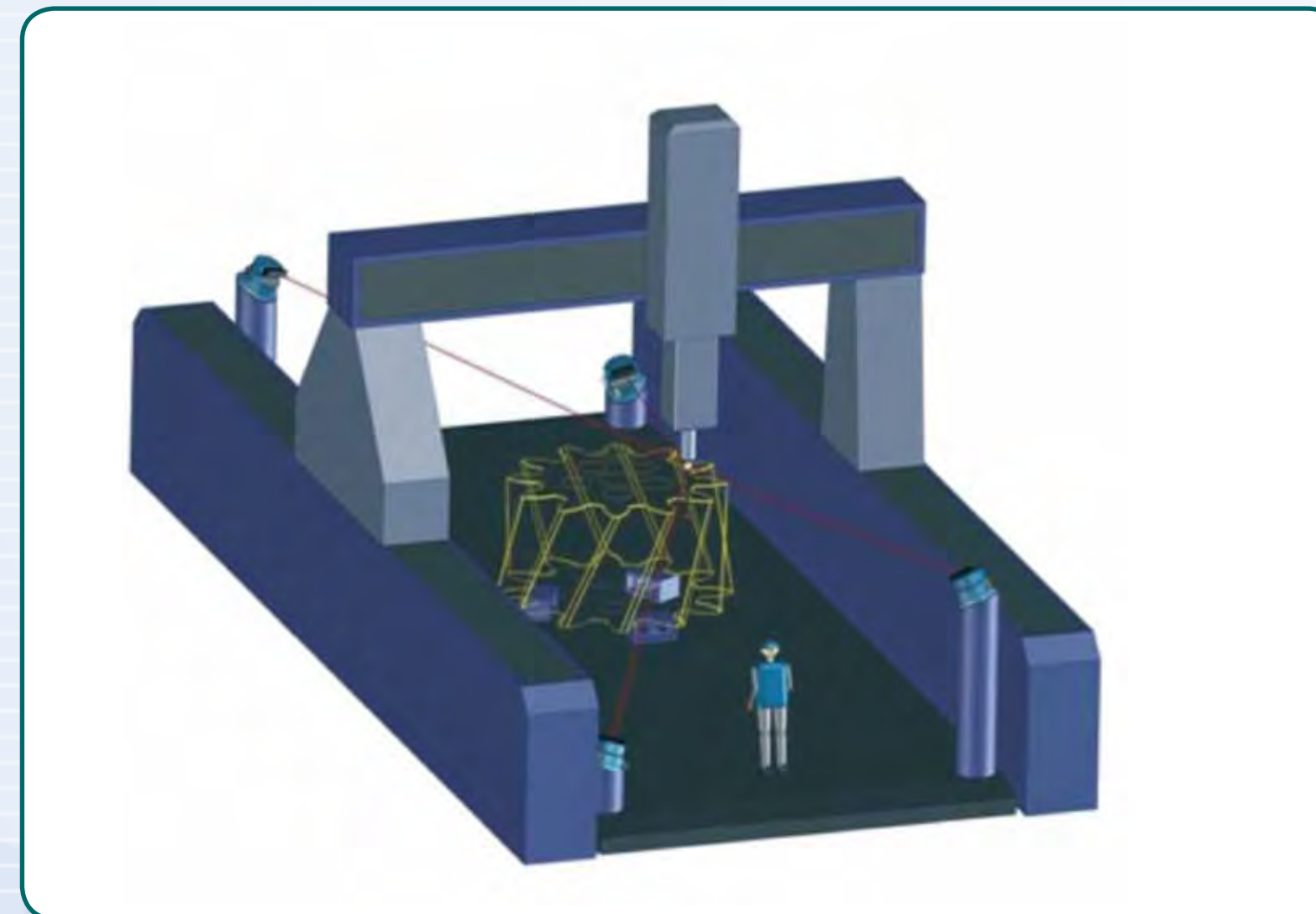
Technical achievements

Innovative and advanced standards designated for traceable measurements, calibration of parts, and performance assessments of measuring systems:

- a novel large involute gear standard with a diameter of 1 metre
- 3D freeform standards to validate optical-based CMMs and improve comparison measurements
- tetrahedron standards with ceramic optically cooperative targets for CMMs
- comparison measurements between optical and tactile based CMM measurements
- a waviness standard to assist the automobile industry

A new multi-lateration measuring system (M3D3) dedicated to the measurement of large objects has been developed and validated. This novel system can implement task specific uncertainty measurements and map CMM error, and can also be retrofitted into either a laboratory or industrial shop floor. The M3D3 is currently housed at PTB, Germany's National Metrology Institute, and will be used by manufacturers of large-scale parts, the automobile industry and other CMM users.

A new software validation technique has been developed; the Internet Aided Software Validation (IASV) infrastructure enables clients to communicate online for the purpose of CMM software and other algorithm validation.



Multi-lateration measuring system (M3D3) demonstrating measurement on an industrial shop floor.

Large gear and CMM calibration

Developed a large involute gear standard and M3D3 system to assist industries in assessing the performance of large-scale measurement machines, the implementation of task specific uncertainty measurement and the accurate measurements of complex parts.

Guidance for users

Produced comprehensive good practice guides and software for error detection and configuration of multi-sensor measurement systems that will be used by developers and users of multi-sensor networks, aircraft and automobile manufacturers, machine tool industries and calibration laboratories.

Interferometer and laser tracer calibration

Developed a new facility for the calibration of interferometers and laser tracers, used in the development of the new multi-lateration measuring system (M3D3). The facility will be used by calibration bodies and large object manufacturing industries, e.g. wind turbine, ship, aircraft and automobile industries.

Online validation of software

Launched the pilot Internet Aided Software Validation (IASV) infrastructure that is currently being tested by eight industrial partners: Carl Zeiss Industrielle Messtechnik GmbH, Klingelberg GmbH, Mahr OKM GmbH, Messtechnik Wetzlar GmbH, Mitutoyo Messgeräte GmbH CTL, Mahr OKM GmbH, FRESCO GmbH, PTB Berlin.



Absolute long distance measurement in air

The need for the project

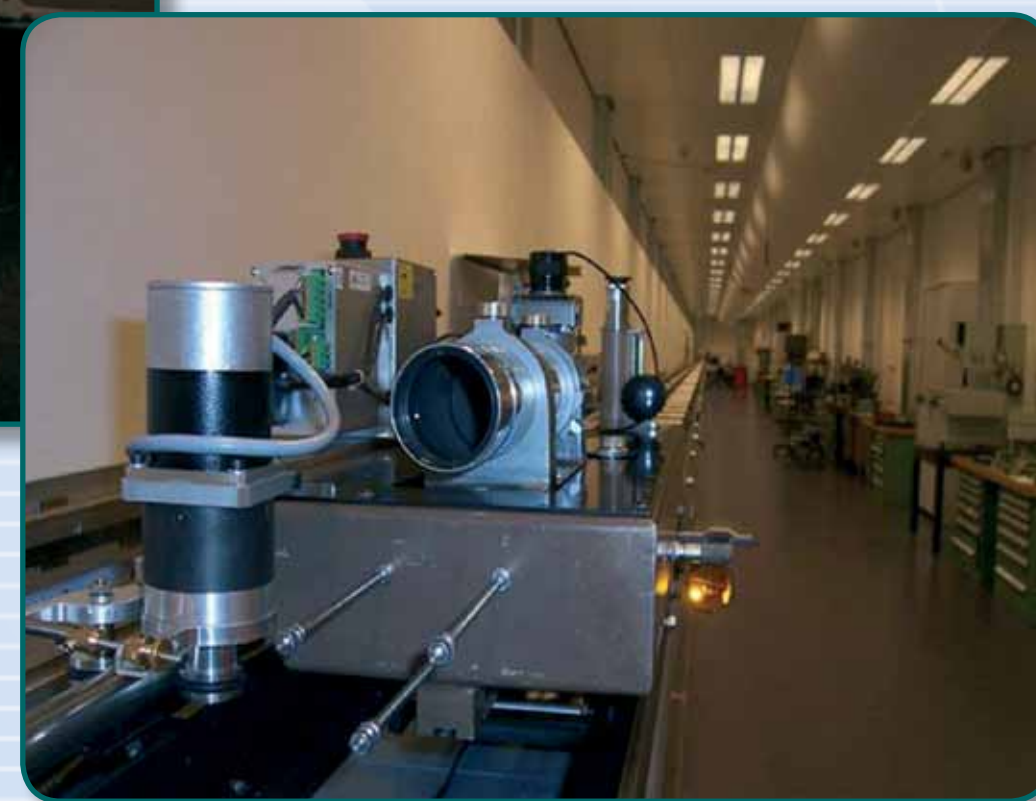
Large scale production, in particular in aerospace, global monitoring (geodesy) and waste management, all require the measurement of dimensions to levels of accuracy currently unachievable over long distances (over 10 m).

To date, the best instrument for long range distance measurement achieved an accuracy of approximately 5×10^{-7} when atmospheric parameters were sufficiently stable – but this instrument is no longer commercially available and required highly skilled operators.

This project aimed to improve the current state of the art in long range distance measurements in air to produce a relative accuracy of 10^{-7} . It also aimed to improve current techniques for the measurement of air refractive indices and develop and refine synthetic wavelength interferometers.



◀ PTB scientist preparing the prototype for baseline measurement.



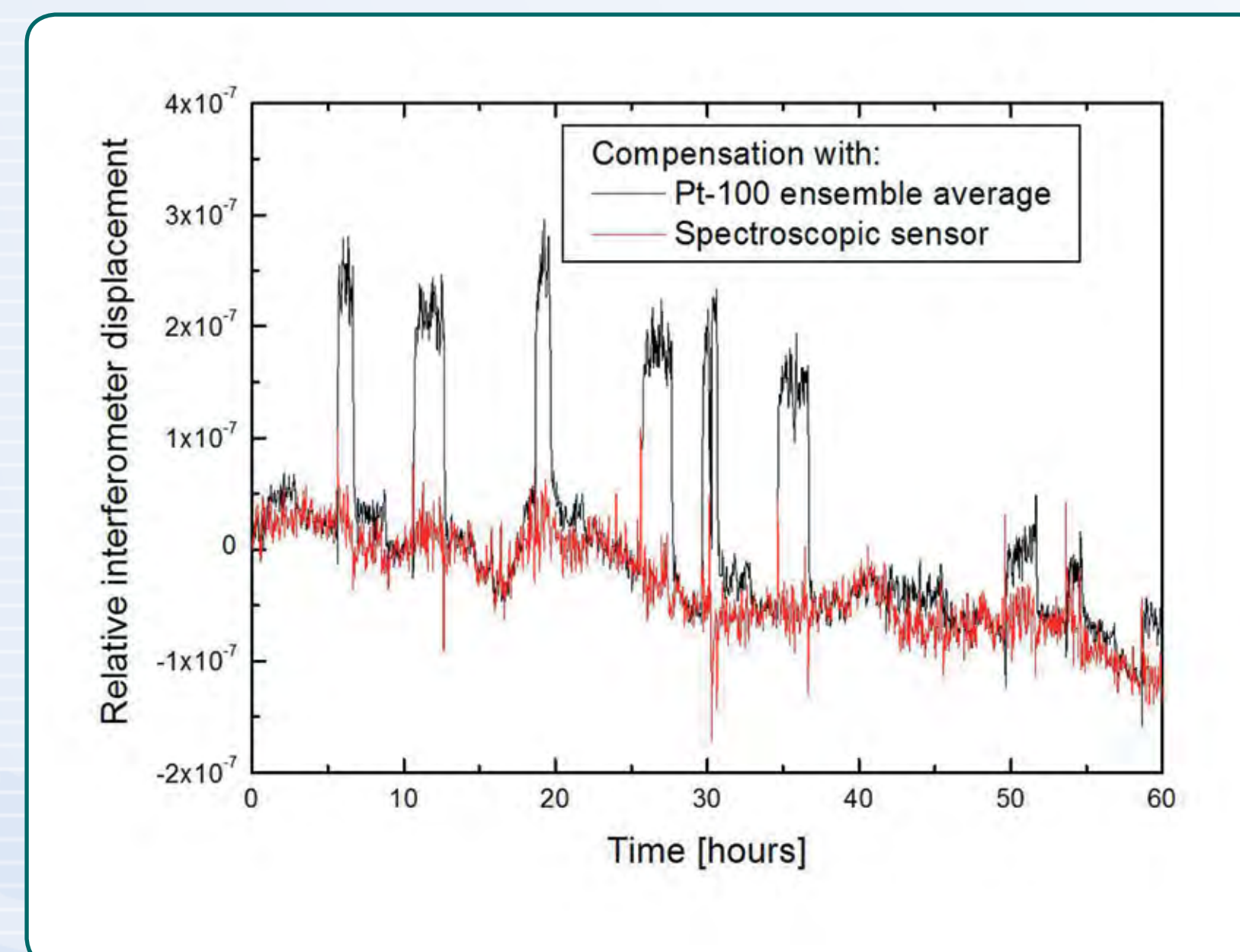
▶ VSL laser interferometer used for validation of pulse to pulse interferometry and femtosecond based telemeter.

Technical achievements

Spectroscopic sensors were built, enabling measurement of temperature and humidity along a given optical path. A resolution of 10 mK and an uncertainty better than 0.1 K was demonstrated for temperature measurement. An improvement of air index compensation compared to a classical thermometer was also demonstrated and relative humidity was measured with an uncertainty of less than 4%. The spectroscopic sensors were used successfully outdoors up to 72 m for humidity and up to 200 m for temperature.

Three new types of transportable telemeters were produced; two based on synthetic wavelength interferometry and one on time of flight techniques using femtosecond lasers. The best resolution obtained with synthetic wavelength interferometry systems was less than 1 mm with an uncertainty of less than 10 mm indoors for absolute measurement. Similar results were found with time of flight techniques up to 100 m.

Pulse to pulse interferometry was implemented for distance measurements up to 50 m (100 m propagation through air). A comparison with laser interferometry demonstrated measurement agreement within 2 mm for this distance (4×10^{-8} uncertainty). Dispersive interferometry methods were also compared and showed measurement agreement within 0.5 mm at 50 m (relative uncertainty $<10^{-8}$) compared to the counting laser interferometer.



Evidence of improvement of air index compensation. A comparison of distance measurements (30 m) with a classical thermometer and spectroscopic thermometer. The distance variations in black are due to uncompensated temperature variations that are compensated with the spectroscopic thermometer in red.

Improved long distance measurement

The project developed:

- a transportable spectroscopic thermometer
- transportable spectroscopic hygrometers
- a transportable distance metre with no air index compensation for indoor and outdoor application
- a transportable distance metre with air index compensation
- a distance metre based on femtosecond lasers for indoor application
- a distance meter based on femtosecond lasers for indoor and outdoor application

After further advancement to make them more compact and robust, these prototypes could support the metrological, geodesic and surveying communities.

Commercialisation

Initial contact has been made with measuring instrument manufacturers (e.g. LEICA, SIOS) and laser systems manufacturers (e.g. Toptica, Menlo systems).

The prototype spectroscopic thermometer has been used in the development of a commercial oxygen analyser by Gaset Technologies Inc.

Guidelines

Produced guidelines for the use of the new techniques and prototypes for measuring absolute distances, which will be available to all interested stakeholders and long distance measurement end-users.



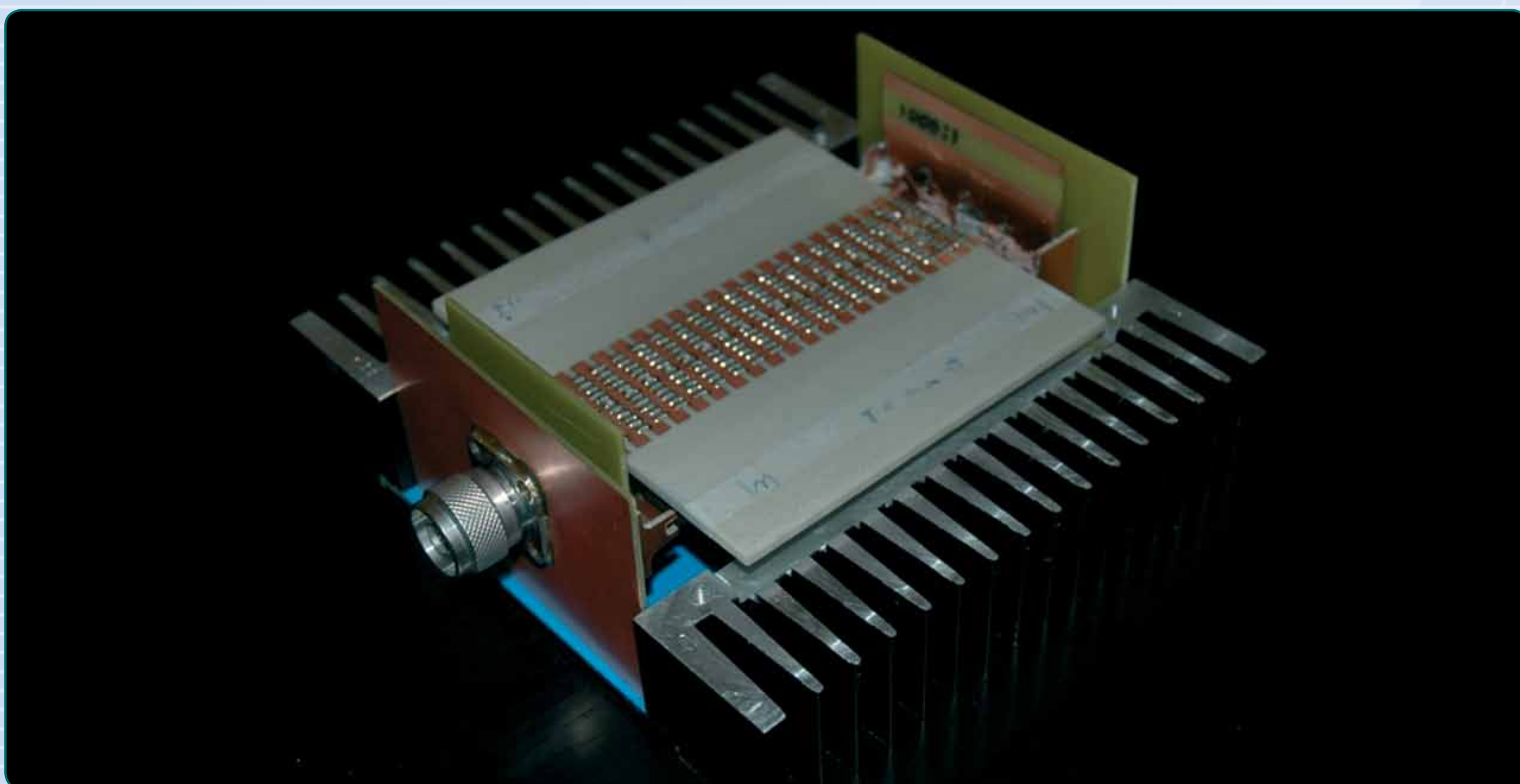
Next generation of power and energy measuring techniques

The need for the project

Europe faces potential energy shortages as fossil fuel supplies diminish and nuclear power facilities age, but there is also pressure to reduce greenhouse gas emissions and a commercial demand for an electricity supply of the highest quality - free from momentary voltage interruptions and interference.

These challenges are a catalyst for new technologies that require a new generation of power and energy measurements, made directly at generation and distribution sites and involving the accurate measurement and analysis of complex wave shapes.

This project aimed to develop new instruments, algorithms and methodologies to make these required measurements to support an EU regulatory framework that oversees the market for electrical goods and power generation.

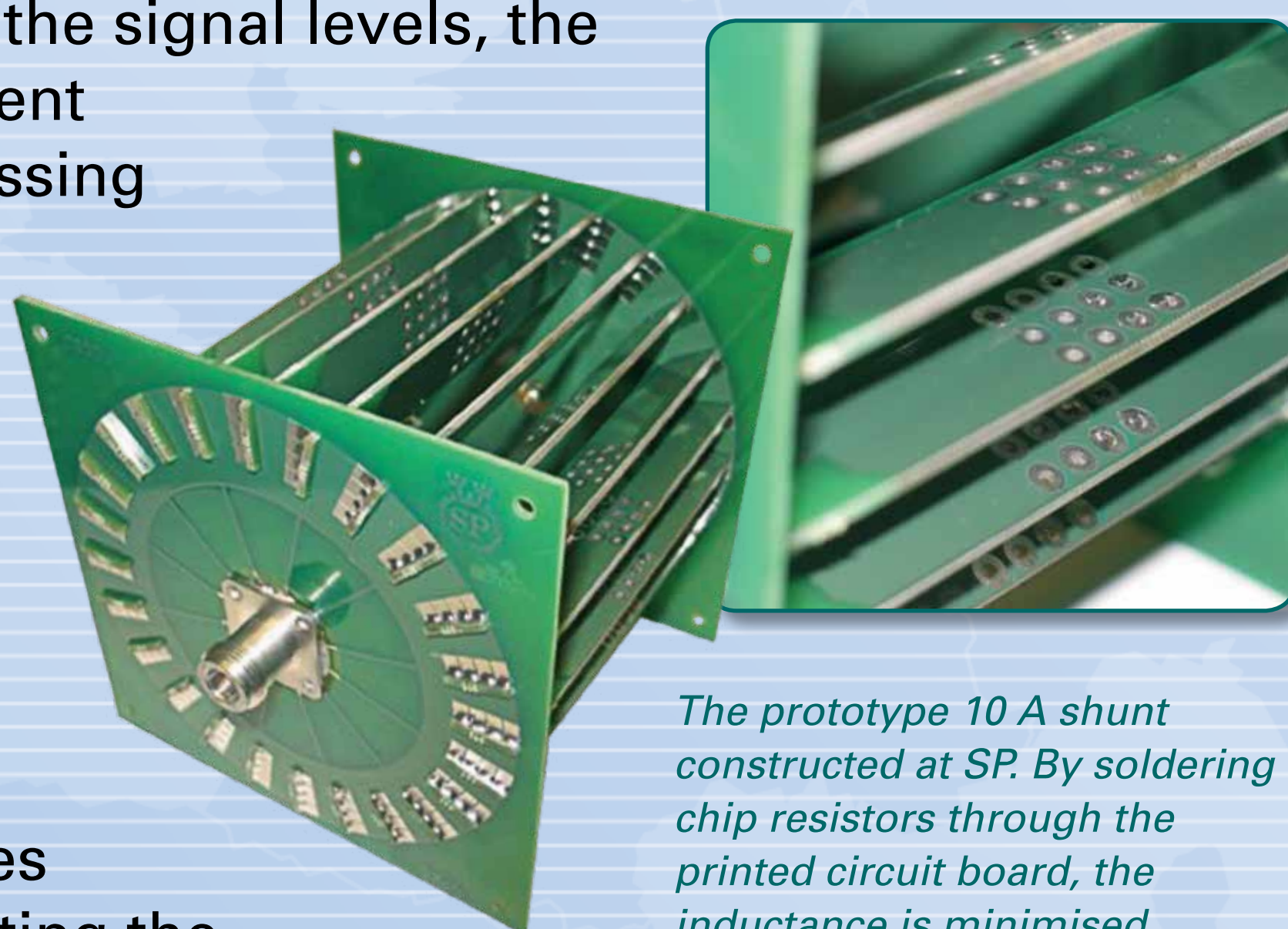


The JV Precision 1 kV Voltage Divider.

Technical achievements

The high current and high voltage levels that are prevalent at electricity distribution/generation sites were accurately transformed to lower measurable levels. This entailed the development and characterisation of lab-use and portable transducers to cover a wide range of currents and voltages up to 33 kV. These transducers can be connected to the electricity system without interrupting the supply.

Having transformed the signal levels, the electrical measurement and computer processing of the AC waveforms was made possible by digitising the signals using six channel analogue to digital converters that operated at high data output rates continuously converting the waveform with known fidelity.



The prototype 10 A shunt constructed at SP. By soldering chip resistors through the printed circuit board, the inductance is minimised. The chip resistors are newly developed with very low temperature coefficients.

The resulting data was processed using algorithms, producing the complex range of power quality metrics used by industry. The waveforms of interest are continuously changing as the electricity demand changes - this required the development of new waveform transforms to analyse these complex waveforms.



Precision High Current Shunts.

Regulation for electrical appliances

Reduced errors in conformance testing to help protect the multi billion euro electrical goods industry. This will also help to protect the electricity network from exposure to sub-standard equipment.

Calibration services

New services to calibrate instruments were developed using the technology, protocol methodology and guidelines developed in the project. The new services will be offered to end-users by project partners.

Validated power measurements

Made seven successful on-site tests in the UK, Finland, Italy and Sweden. The on-site tests included medium voltage networks (33 kV), high current measurements (kA), power loss measurements in network transformers and power quality at distribution substations.



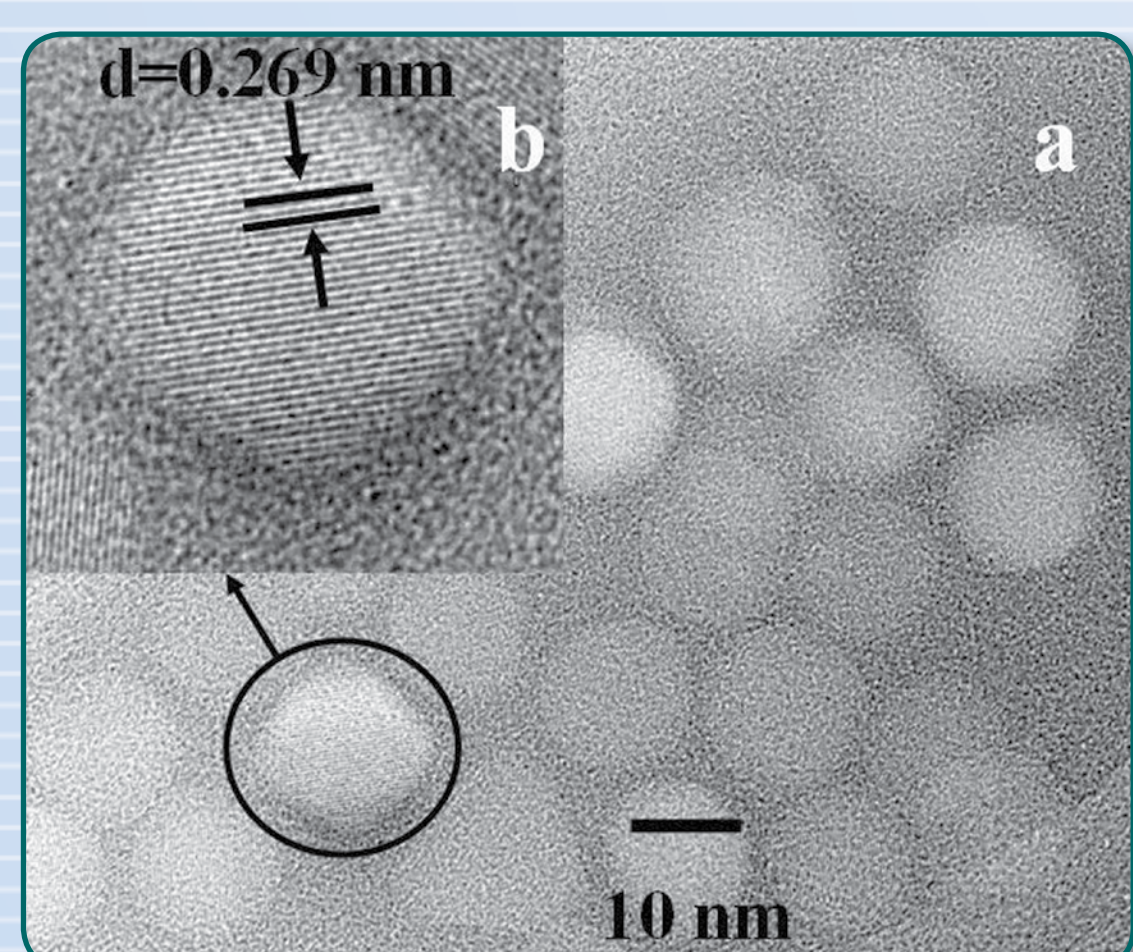
Joint Research Project (JRP) Short Name: Power & Energy • JRP-Coordinator: Paul Wright (NPL) • JRP-Partners: BEV (Austria), BRML (Romania), CEM (Spain), CMI (Czech Republic), EJPD (Switzerland), INRIM (Italy), JV (Norway), LNE (France), MIKES (Finland), NPL (UK), PTB (Germany), SIQ (Slovenia), SMU (Slovakia), SP (Sweden), Trescal (Denmark), VSL (Netherlands)

The need for the project

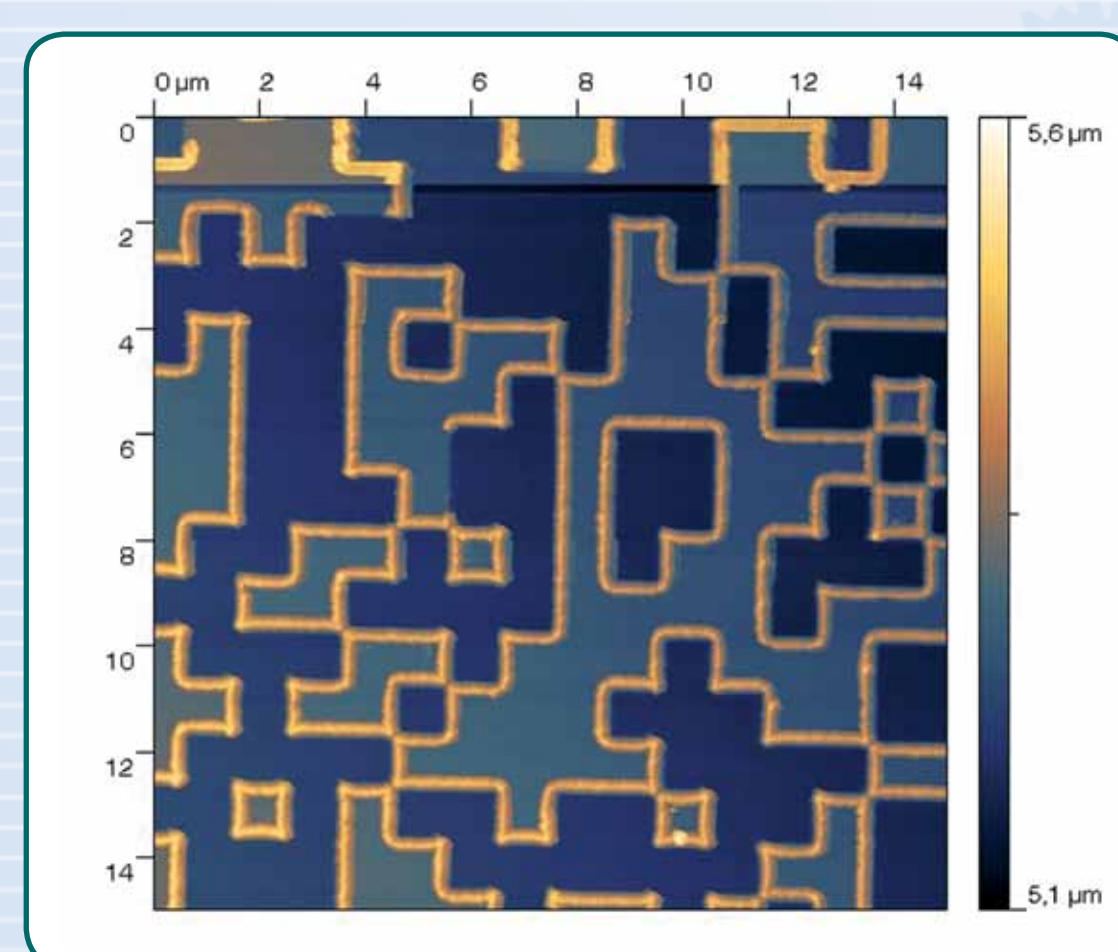
Developments in the fields of nanomagnetism and spintronics include a wide range of applications such as ultra strong magnets, spin polarized materials, ultra high density recording media (hard disks, flash memories/MRAM), spin transistors and DNA- and bio-sensors.

All of these developments urgently require measurement tools to reliably and traceably characterise magnetic nanomaterials.

The aim of this project was to establish a metrological basis for the field of nanomagnetism and to provide reference samples and measurement methods to industrial and academic end-users.



(a) High resolution TEM image of iron oxide nanoparticles (b) 12 nm maghemite (γ - Fe_2O_3) particle with visible atomic planes.



Magnetic Force Microscopy calibration sample.

Technical achievements

Reference Nanomaterials:

The project produced thin films of Permalloy integrated in coplanar waveguides for time and frequency domain dynamics measurements, Gallium Manganese Arsenide (GaMnAs) diluted magnetic semiconductor samples for precessional dynamics, size monodispersed nanoparticles for high resolution scanning probe microscopy and ultra sensitive magnetic moment detection and samples of hard magnetic materials with perpendicular anisotropy for high resolution scanning probe microscopy.

Time and frequency domain dynamics:

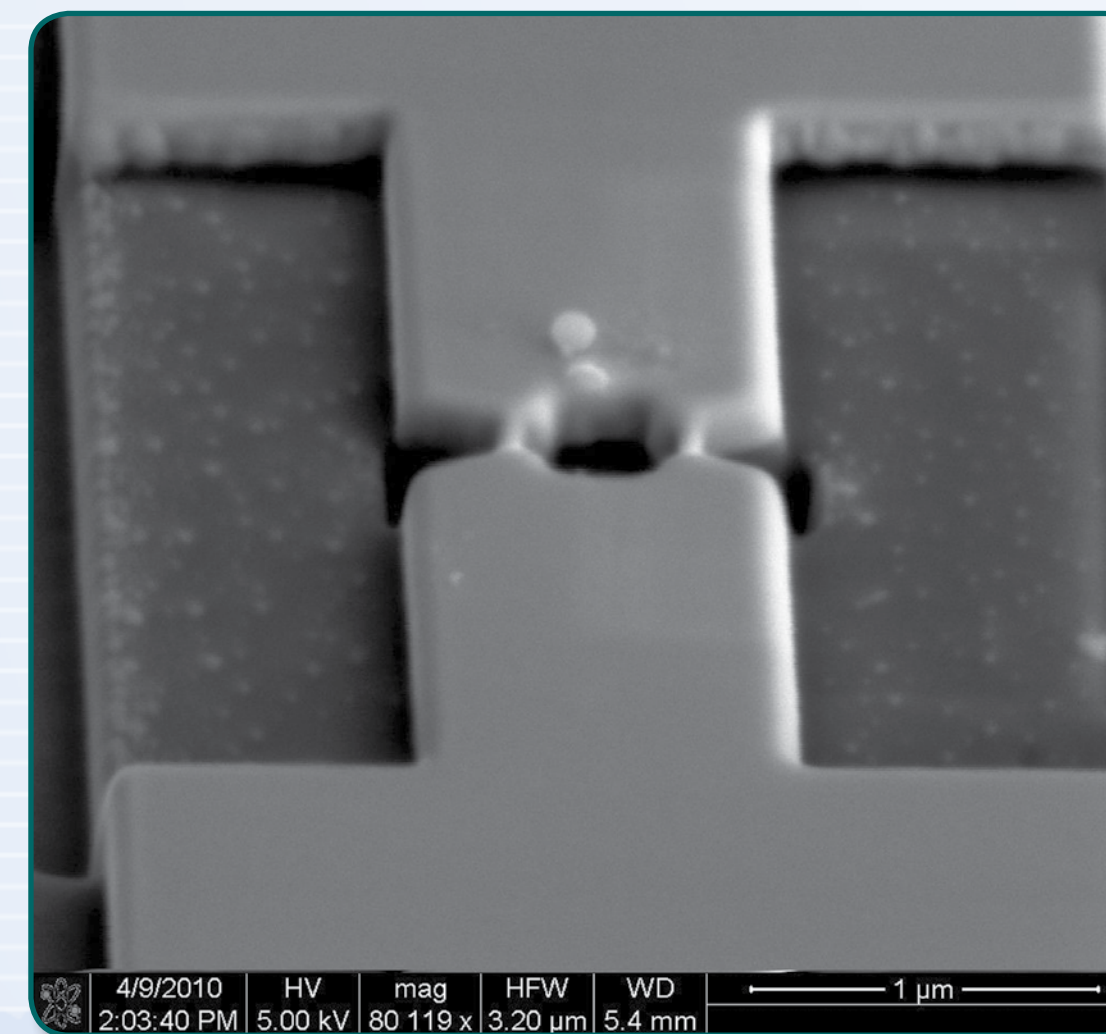
The project developed the inductive metrology of ferromagnetic resonance frequency (fFMR) and the Gilbert damping (α) of soft magnetic thin film have been established and validated and a set of calibrated soft magnetic reference samples is available for external inductive measurements of fFMR and α . Metrology for the Spin Torque precession of individual nanodevices in time and frequency domains has also been established.

High Resolution Scanning Probe Microscopy:

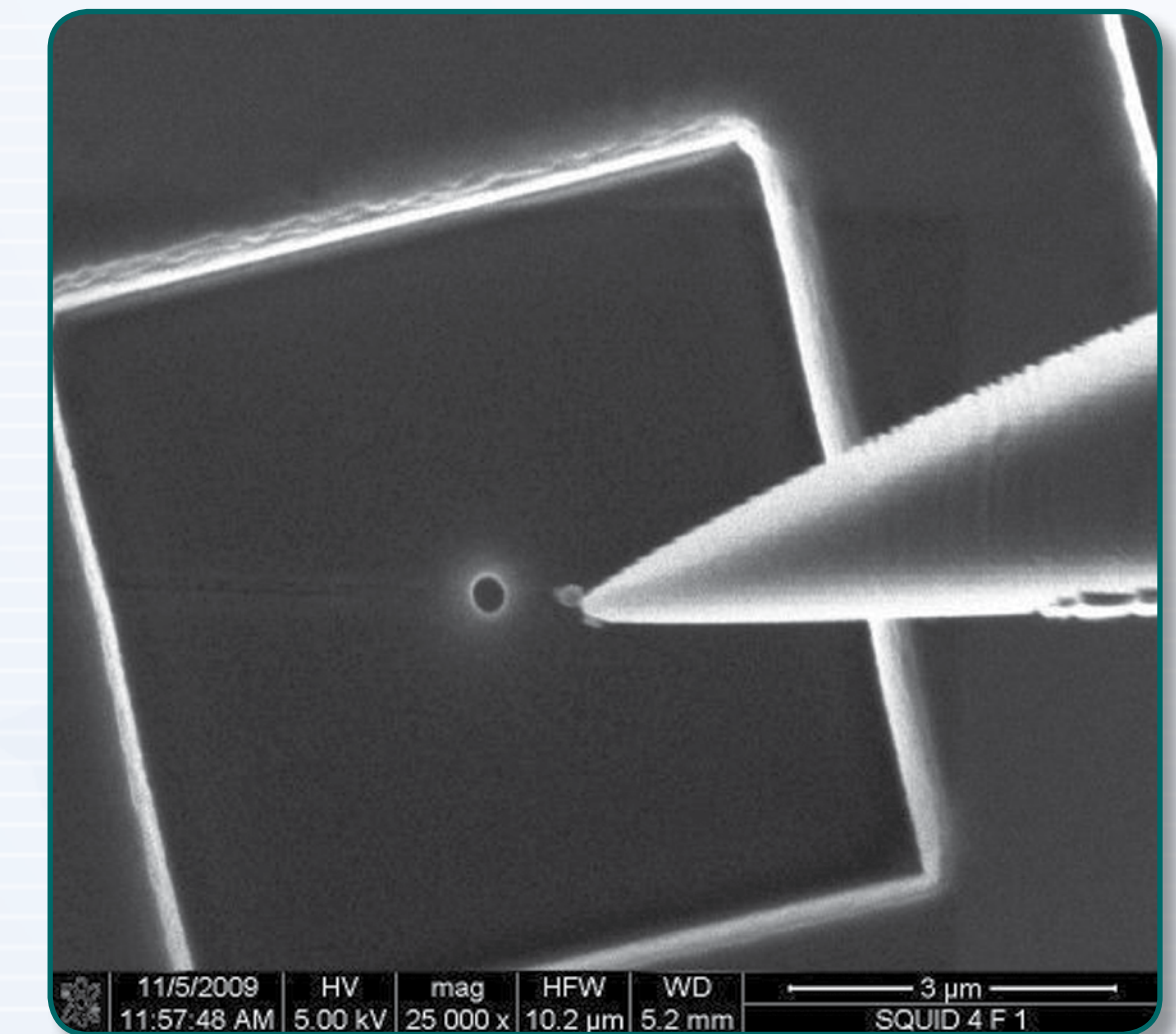
Quantitative Magnetic Force Microscopy with a resolution of less than 50 nm has been demonstrated using magnetic nanoparticles.

Ultra sensitive magnetic moment detection:

A prototype magnetic detector based on nano-SQUID magnetic moment sensitivity and a nanosized (<500 nm) metallic and semiconductor (i.e. two dimensional electron gas heterostructures) Hall sensors were developed.



Single particle detection with nano-SQUID: 120 nm $\text{SiO}_2/\text{Fe}_3\text{O}_4$ nanoparticles (104 mB) on nano-SQUID with the loop size ~ 300 nm.



Single particle detection with nano-Hall sensor: Manipulation of a 120 nm FePt nanobead.

New techniques and samples

New reference samples and techniques were developed and transferred to industrial and academic end-users, such as:

- time resolved damping techniques - used by Singulus Technologies AG and University of Bielefeld, Germany
- damping reference samples – used by Tohoku University, Japan
- nano-SQUID detection technique of a single nanoparticles – used by University of Tübingen, Germany
- Hall sensor detection techniques for single nanoparticles/nanowires – used by University of Duisburg, Germany and CSIC, Spain

Dissemination through end-users

A 'Nanomagnetism' group, involved in a wide range of scientific activities and their dissemination, was created with project collaborators, e.g:

reference nanomaterials: University of Duisburg, Trinity College Dublin, University College Cork, University of Vienna

sensor fabrication and nanomanipulation: Imperial College London, Cambridge University, Surrey University

preparation of hard magnetic thin films for hard magnetic reference samples: TU Chemnitz, Hitachi and IMEM Parma

ferromagnetic resonance and damping for microwave applications: Tohoku University, NIST Boulder, University of Colorado, Northeastern University Boston

tunneling Magnetic Junctions dynamics and point contacts for memory/sensor applications: Singulus AG, University of Bielefeld, NIST Boulder

high resolution magnetic microscopy/MFM calibration: TU Chemnitz, Hitachi GST, University of Parma, University of Göttingen, IFW Dresden, TU Braunschweig, Magnicon GmbH



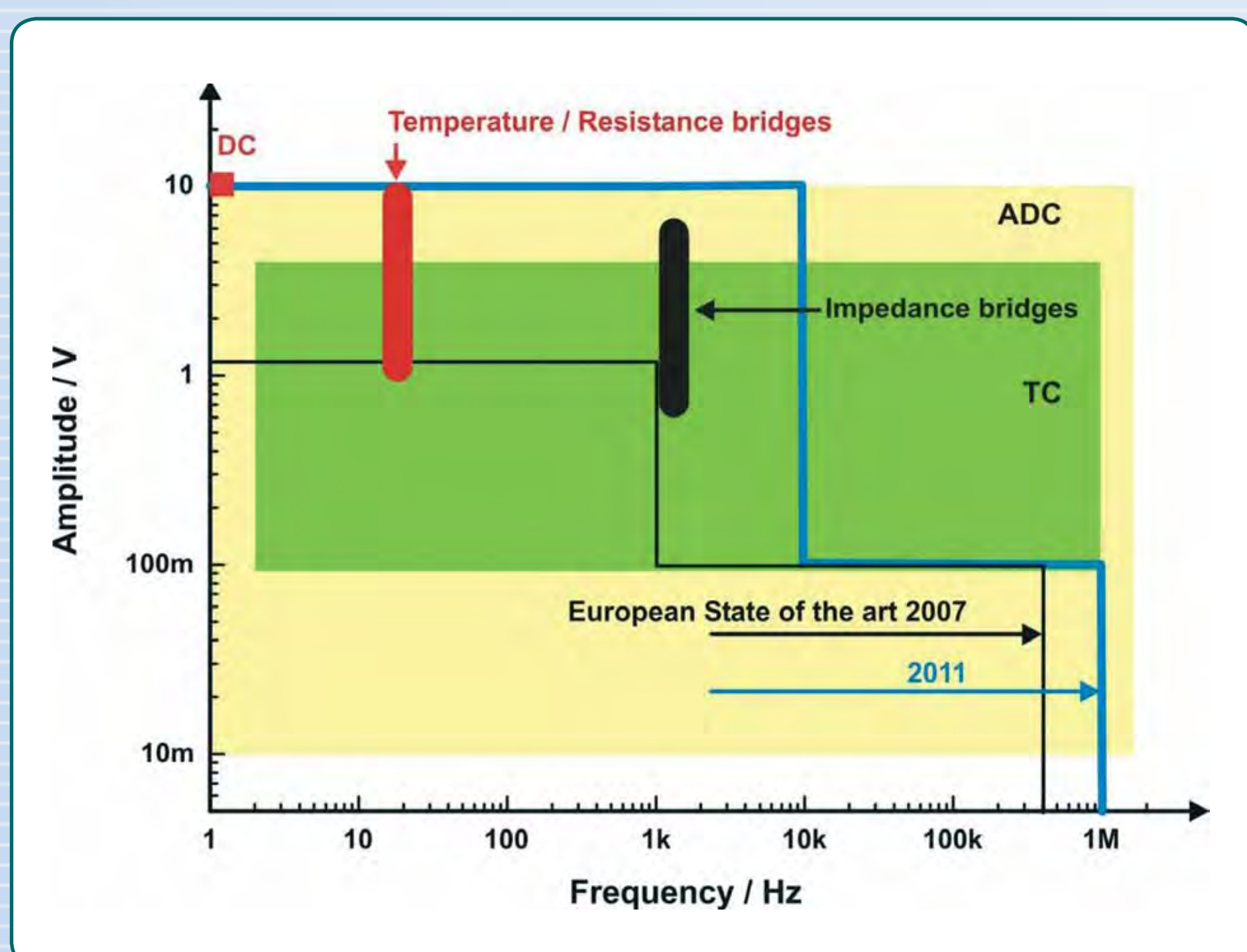
Next generation of quantum voltage systems for wide range applications

The need for the project

The use of microelectronic circuits in control systems in commercial devices is constantly increasing and is improving the performance and efficiency of mass-produced items.

However, the performance of a control system depends on the performance of electronic components (e.g. analogue to digital converters [ADCs] and digital to analogue converters [DACs]), and the performance of the components, in turn, depends on the performance of the electronic measurement equipment used to test them.

This project aimed to introduce quantum-based measurement systems into AC metrology, providing faster calibrations with lower uncertainties with the aim of supporting electronic measurement and test equipment used in research and development.



The voltage and frequency ranges addressed in this project. The coloured lines illustrate the most important frequency and voltage ranges of various AC applications.

Technical achievements

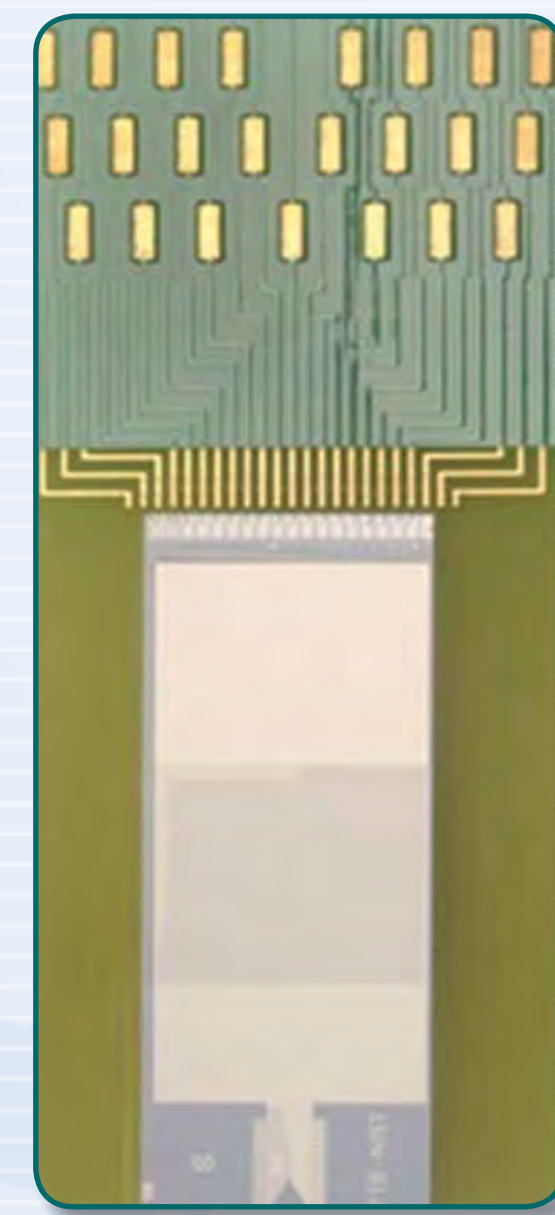
The project has extended the application area for Josephson based methods and produced much better Josephson Synthesizers (JoSys) and quantum-based voltage measurement systems.

A new technology based on a more robust barrier material, Nb_xSi_{1-x} , will be the new basis for Josephson array fabrication. The first wafers containing binary-divided 10-V Josephson series arrays, fabricated for 70-GHz operation and consisting of about 70,000 junctions, showed a good yield and wide constant-voltage steps with a width of around 1 mA.

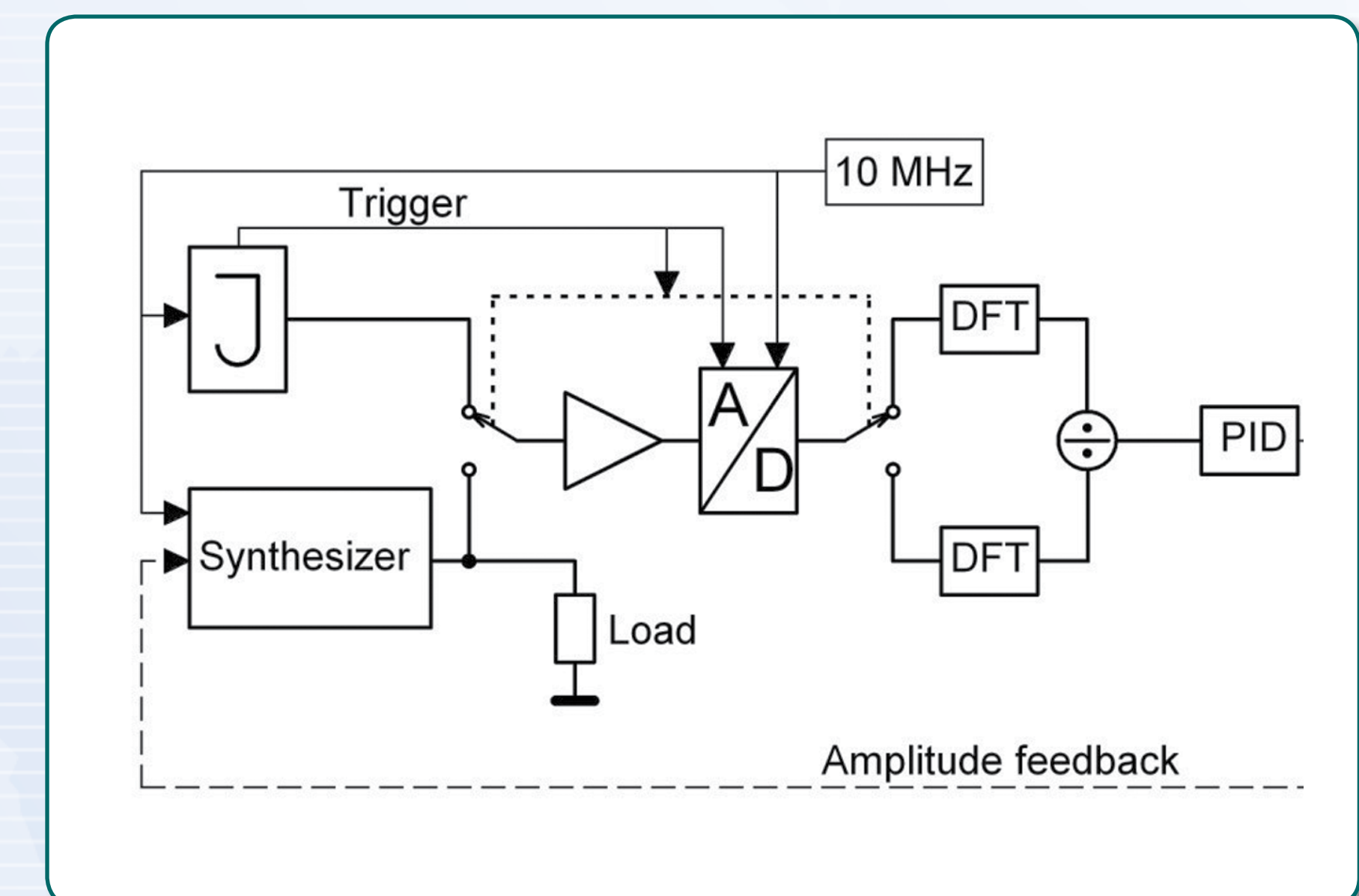
A Josephson voltage standard locked synthesizer (JoLoS) has been developed. The JoLoS with amplitude feedback can be used as a source to drive low impedance with an uncertainty below $1.5 \mu V/V$, ranging from 100 mV to 1 V and 10 Hz to 1 kHz.

Differential sampling methods achieve uncertainties below $0.1 \mu V/V$ for frequencies up to 100 Hz in thermal converter measurements.

A flexible Josephson two-terminal-pair bridge method has been developed and shows at 10 k Ω uncertainties of a few parts in 10^8 , comparable to those of conventional impedance bridges. The Josephson bridge can measure over a much wider frequency range, from 25 Hz up to 10 kHz and over a wider range of impedance ratios than conventional two-terminal-pair bridges.



10-V array based on Nb_xSi_{1-x} -SNS Josephson junctions.



Schematic diagram of the Josephson voltage standard locked synthesizer (JoLoS).

New generation quantum voltage systems

Established a new generation of quantum voltage systems for synthesizing and measuring waveforms - enabling numerous previously unavailable calibration methods based on an intrinsically stable quantum effect (i.e. the Josephson Effect).

Calibration of instruments

Established methods for the calibration of thermal converters, ADCs and DACs, inductive dividers, amplifiers, spectrum analysers, waveform generators, AC bridges and other instruments with a frequency dependent response.

Industrial testing

Carried out a successful on-site test in an industrial location, esz AG, Eichenau, Germany, which demonstrated the quality and robustness of quantum-based voltage standards. Two technology transfer projects in Germany have also started to make the new Nb_xSi_{1-x} fabrication process commercially available and two companies, Supracon and esz AG, have expressed an interest in this commercialisation.



Enabling ultimate metrological Quantum Hall Effect (QHE) devices

The need for the project

The Quantum Hall Effect (QHE) has been the official representation of the electrical resistance unit since 1990. It provides a quantised resistance which is dependent on the electron charge and Planck constant. It is extremely reproducible, with typical uncertainties as low as 10^{-9} and provides the ability to link the ohm (Ω) to fundamental physical constants.

This project aimed to deepen the understanding of QHE and develop the next generation of QHE based resistance standards with improved performances that match current industrial needs. This includes developing QHE standards, making them easier to use and implement, at reduced costs, and establishing a wide quantised resistance scale ideally from $100 \Omega - 1 \text{ M}\Omega$.

To do this, the project aimed to investigate new devices (Quantum Hall Arrays (QHARS)) and the use of novel materials, such as graphene in QHE devices.

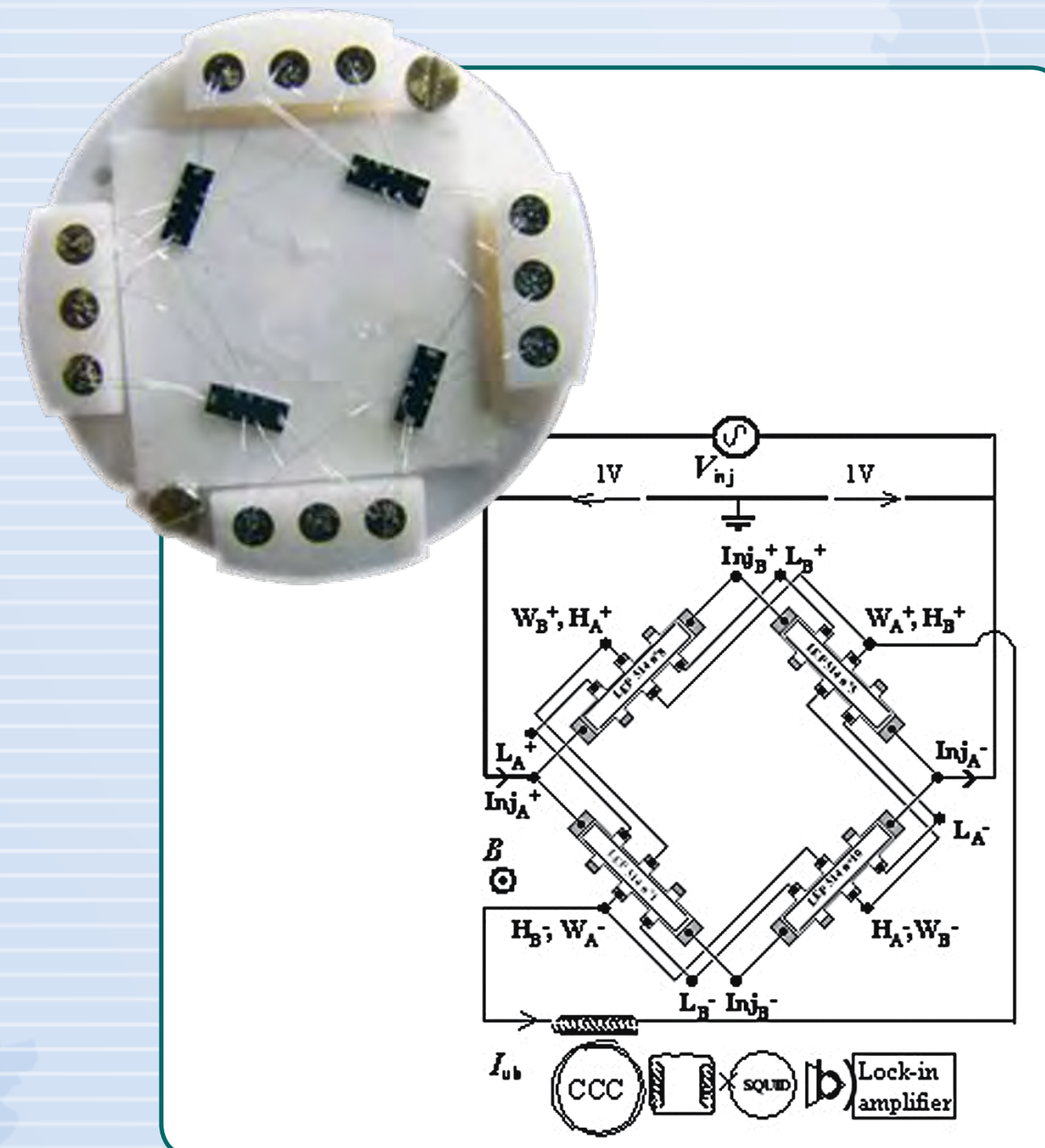
Technical achievements

The uncertainty of QHE in Gallium Arsenide (GaAs) (commonly used to fabricate QHE-based resistance standards) was demonstrated to be as low as 3×10^{-11} , one order of magnitude lower than the previous best quantisation tests. This was done using the quantum Wheatstone bridge technique.

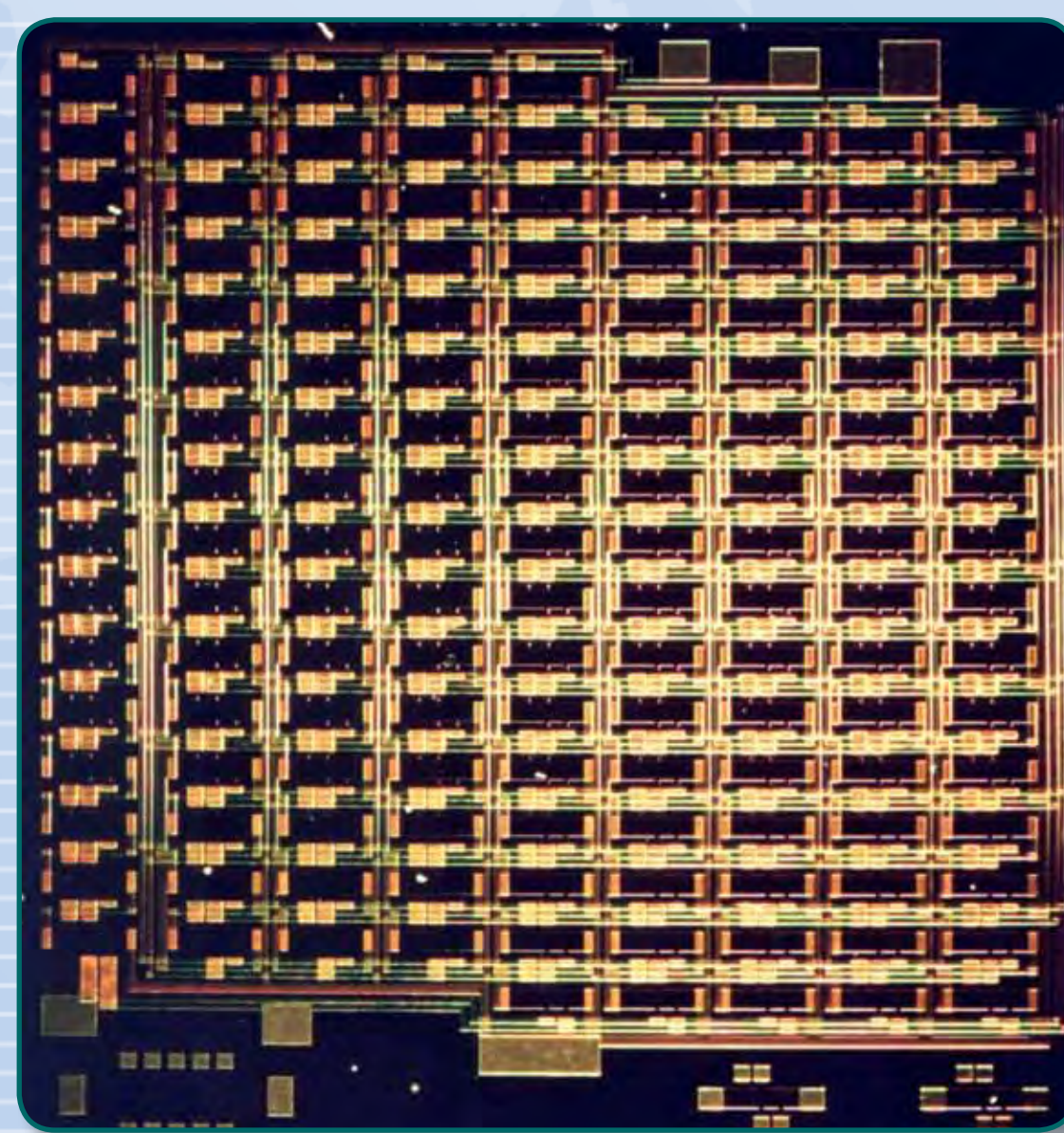
The quantisation of the Hall resistance in the fractional QHE regime was carried out for the first time in high mobility ($10^7 \text{ cm}^2/\text{V}\cdot\text{s}$) GaAs (accuracy within 3×10^{-8}).

The project pioneered the development of a reliable fabrication process of QHE devices adapted for metrological measurement from graphene exfoliated from graphite, although some limitations were found.

The project has validated the use of low value QHARS as the next generation of quantum resistance standards. These devices, based on a combination of up to 145 Hall bars in series and/or parallel arrays, have been demonstrated to present quantised resistance values ranging from 100 W to 1.29 MW within uncertainties as low as 10^{-9} . The QHARS are compatible with commercial bridges and can be used to calibrate them. During the QHARS fabrication, a process for producing double vertically stacked two dimensional electron gas (2DEG) from GaAs was also successfully developed.

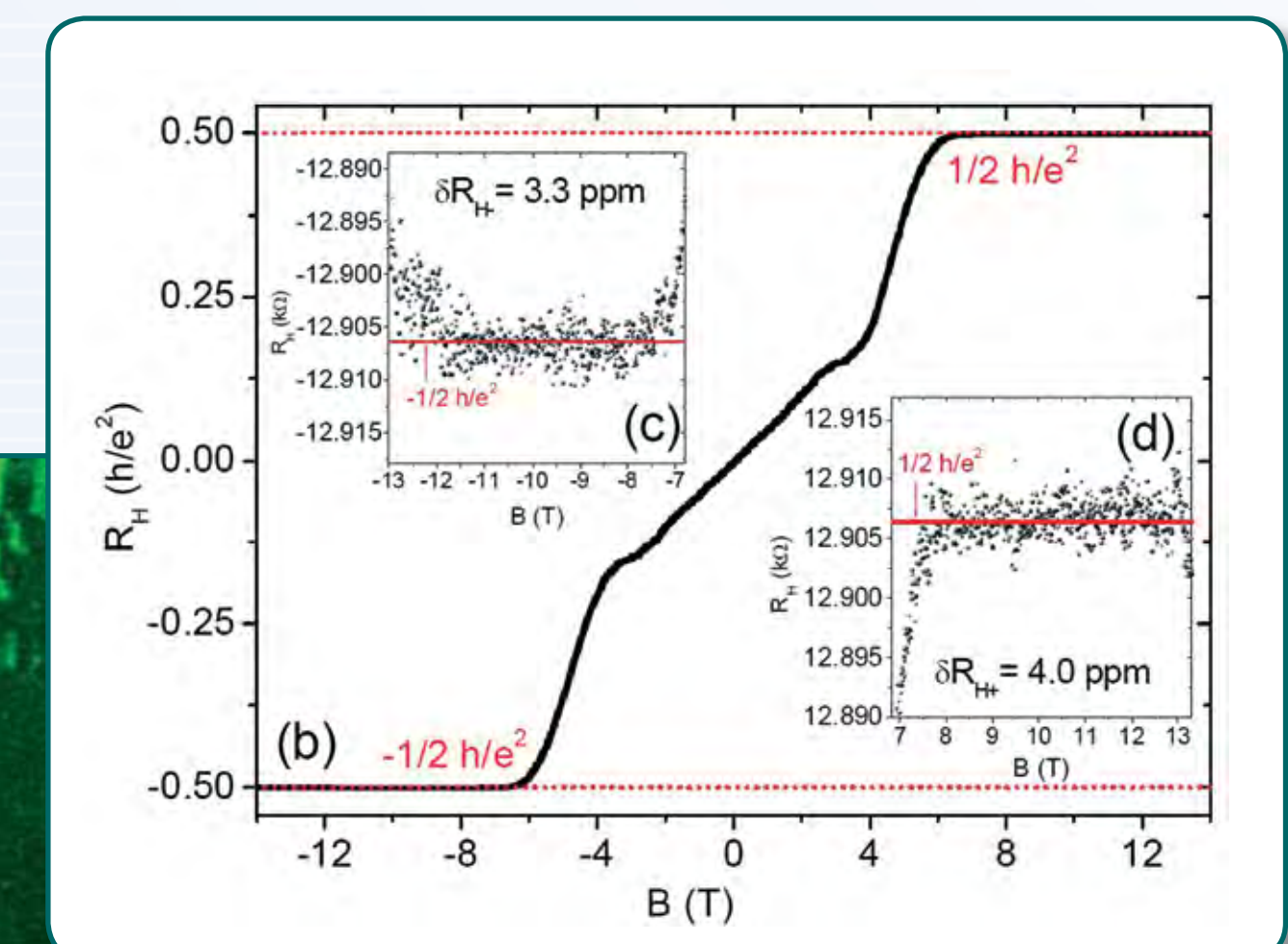
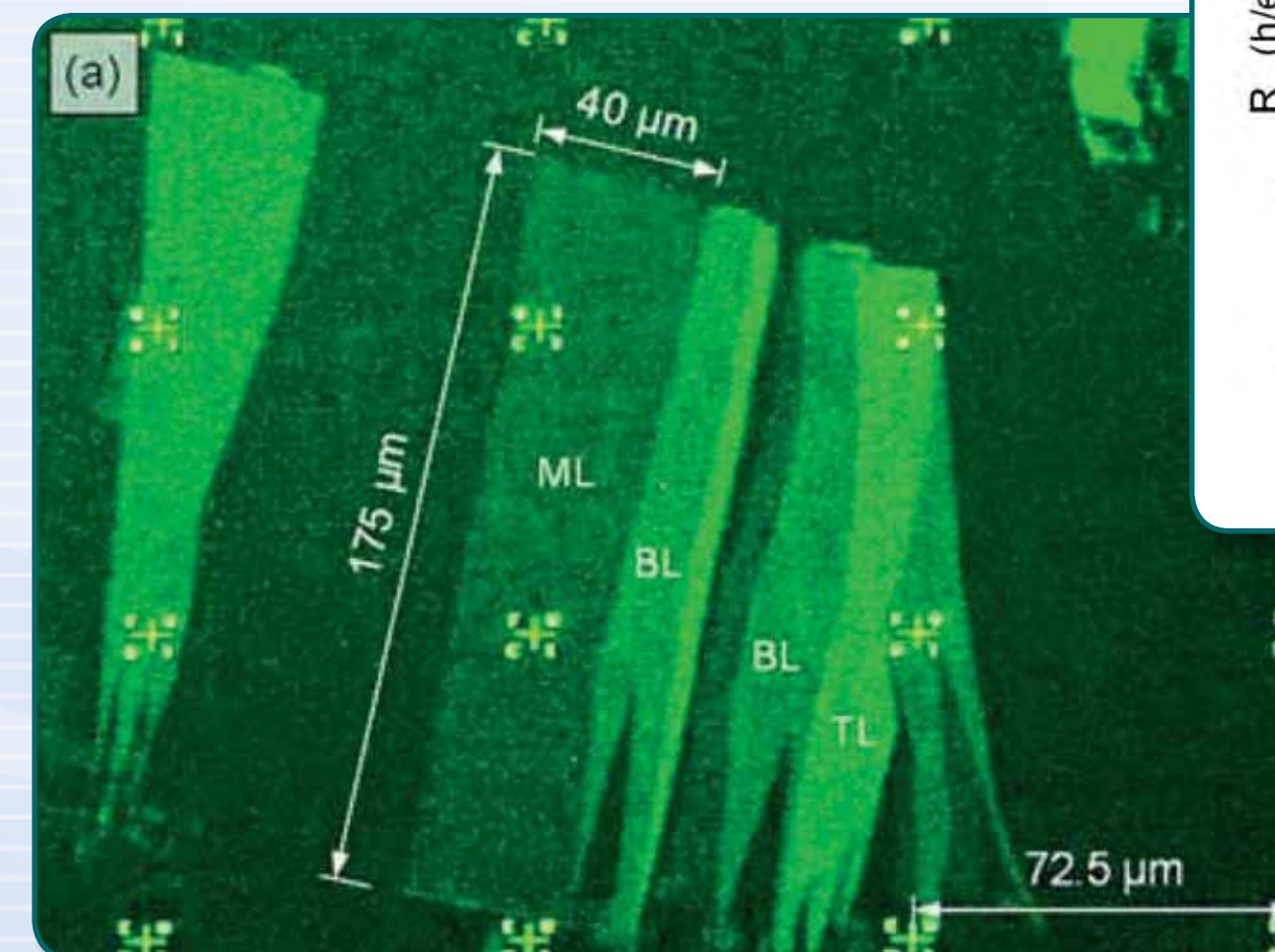


Reproducibility test of the QHE in GaAs samples by means of the quantum Wheatstone bridge technique. 1) Picture of the device. 2) Drawing of the experimental principle.



Picture of a 100 W QHARS (129 Hall bars in parallel with 16 Hall bars in series). The Hall bars are $400 \mu\text{m}$ wide. The total size of the device is 12 mm .

Optical microscopy image of graphene flakes exfoliated from graphite and deposited on GaAs/AlAs substrates.



QHE observed in a monolayer graphene-based Hall bar.

Supporting the redefinition of SI units

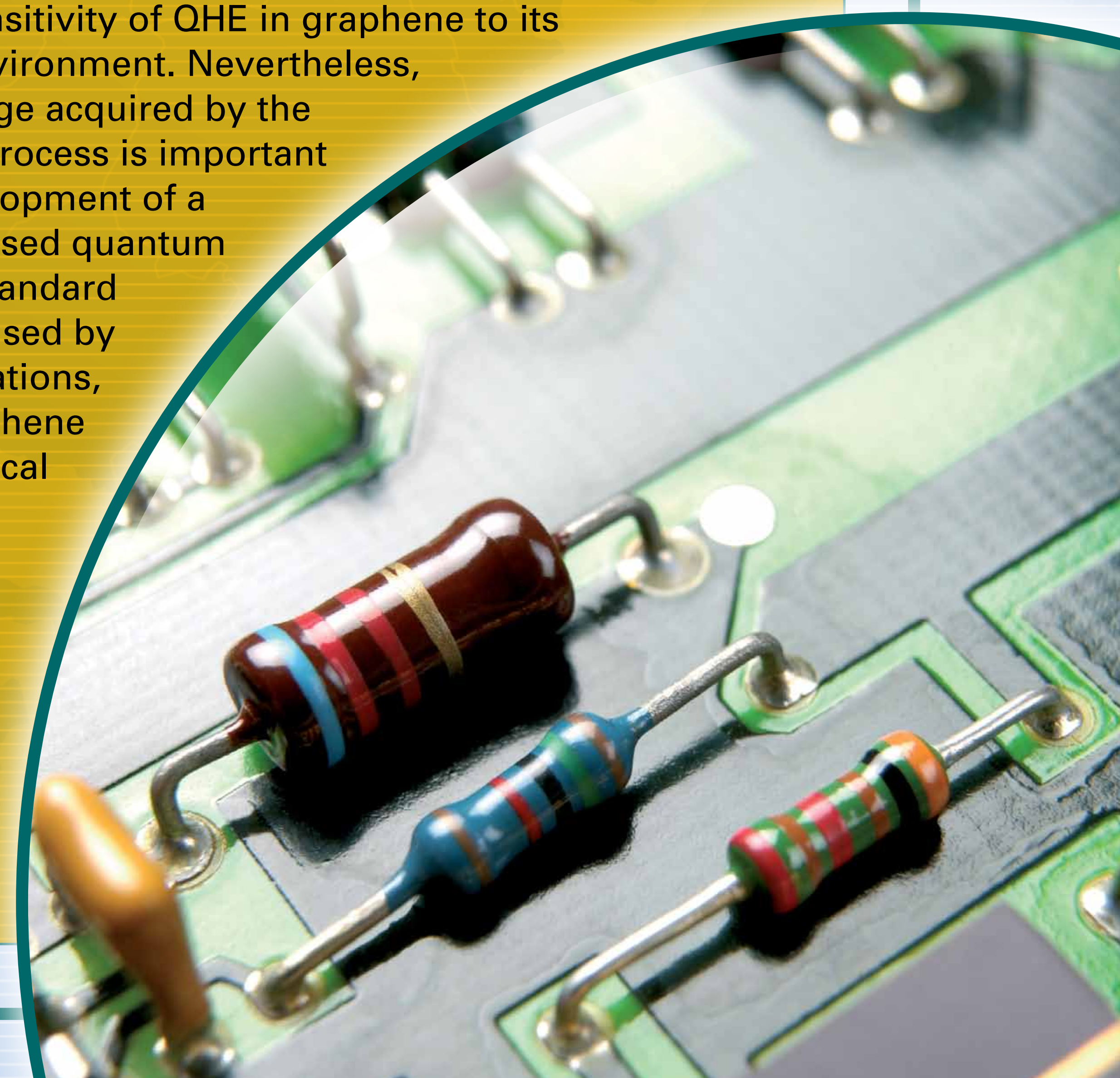
Demonstrated the reliability and robustness of QHE and its underpinning theories by reducing the measurement uncertainty. As a consequence, the project reinforced support for its use in the redefinition of the SI units e.g. the Planck constant (with the watt balance experiment) and the electron charge (with the electron charge quantum metrological triangle experiment).

New quantum resistance standards

Validated the use of QHARS as quantum resistance standards. Guidelines for the use and design of QHARS are to be targeted for National Metrology Institutes as the end-users of QHARS devices.

New graphene fabrication process

Developed a new fabrication process for QHE devices using exfoliated graphene. However, the results of the project also demonstrated that exfoliated graphene may not be the ideal choice for the application of QHE to metrology, due to low yield, the small size of the devices, moderate quality of the metallic contacts on graphene, and high sensitivity of QHE in graphene to its chemical environment. Nevertheless, the knowledge acquired by the fabrication process is important for the development of a graphene-based quantum resistance standard and can be used by other applications, such as graphene use in chemical detectors.



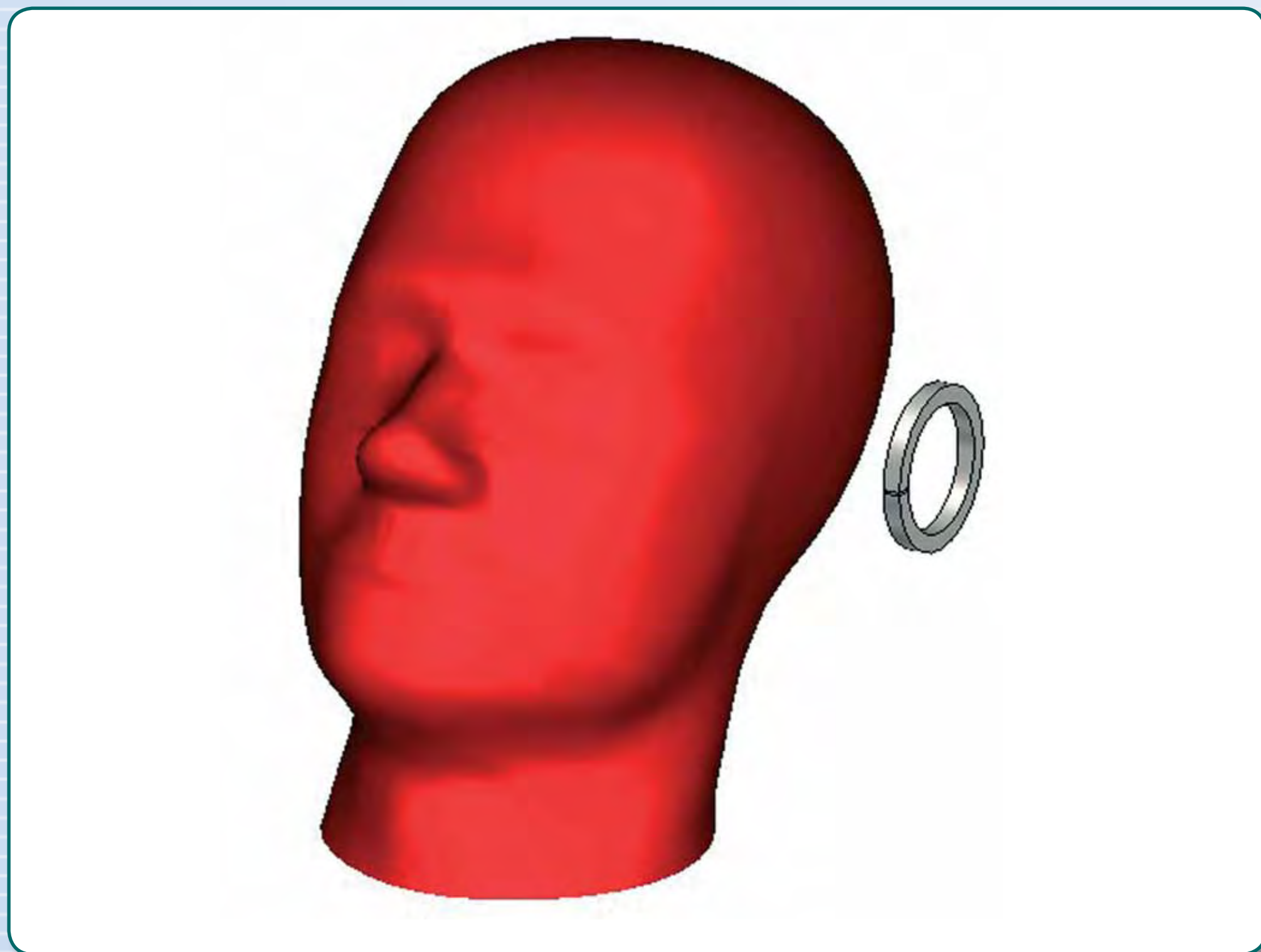
Traceable measurement of field strength and SAR for the Physical Agents Directive

The need for the project

There are minimum requirements for the protection of workers from risks arising from the exposure to electromagnetic fields (EMF) and waves – as laid down by the European Physical Agents (Electromagnetic Fields) Directive 2004/40/EC.

These requirements limit the specific absorption rate (SAR) of radio frequency (RF) power between 100 kHz and 10 GHz, and the incident power flux density (PFD) from 10 GHz to 300 GHz.

Existing standards do not comprehensively cover SAR and PFD at these ranges, and so this project aimed to provide traceable measurements of SAR and EMF strength at the most widely used frequencies.



Specific Anthropomorphic Mannequin (SAM), Filling liquid: $\sigma = 0.33 \text{ S/m}$, $\epsilon_r = 76$, $\delta = 1000 \text{ kg/m}^3$, Source: loop-antenna (radius = 25 mm) with a unitary impressed current, located 4 cm by the side of SAM, Working frequency: 100 MHz.

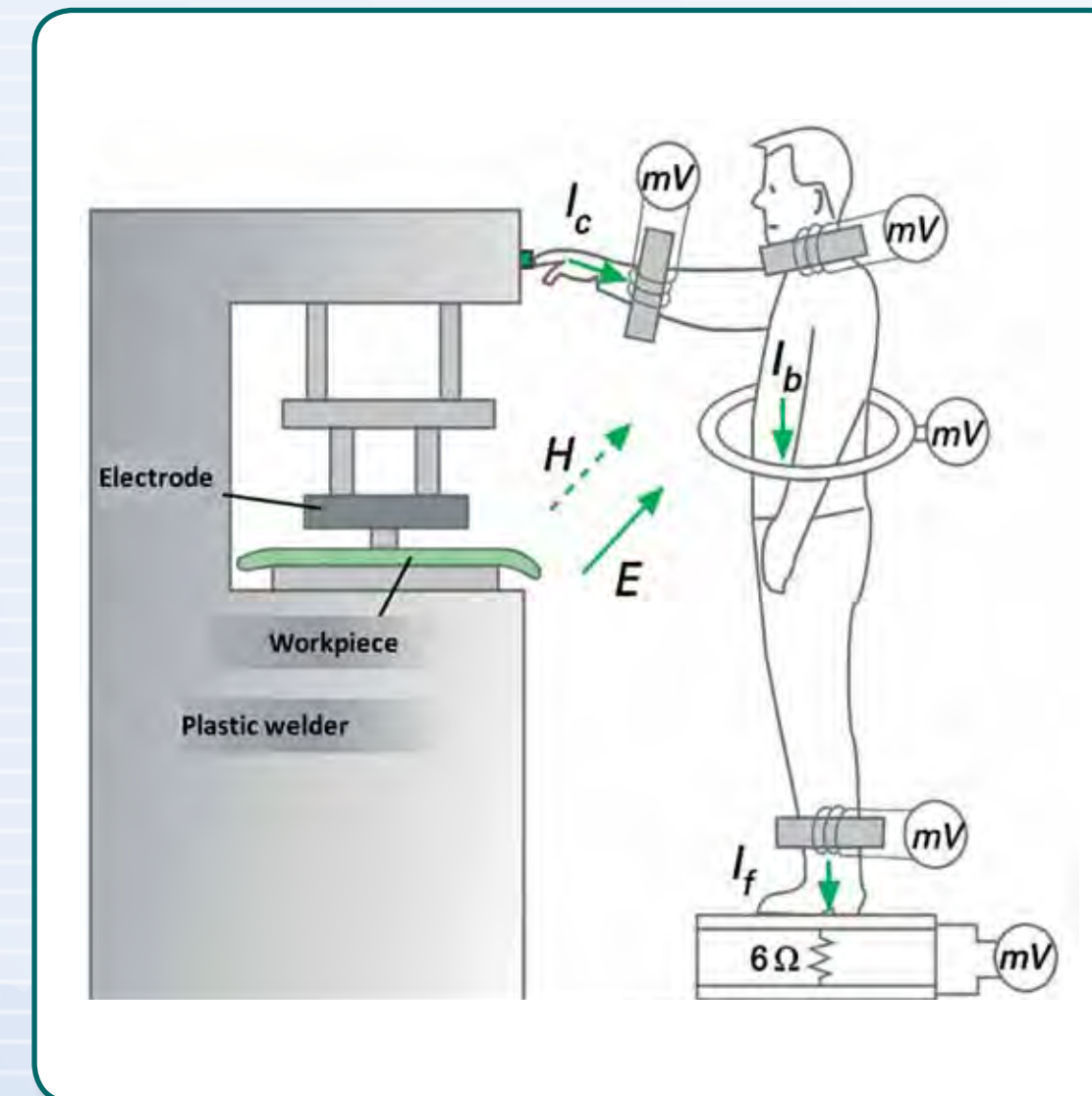
Technical achievements

Response measurements were obtained for field sensors exposed to pulsed, multi-frequency and digitally modulated signals. In addition, digital signal properties were assessed (error vector magnitude) and reference liquid and phantom material properties were characterised. These measurements are important for radar and airport EMF systems.

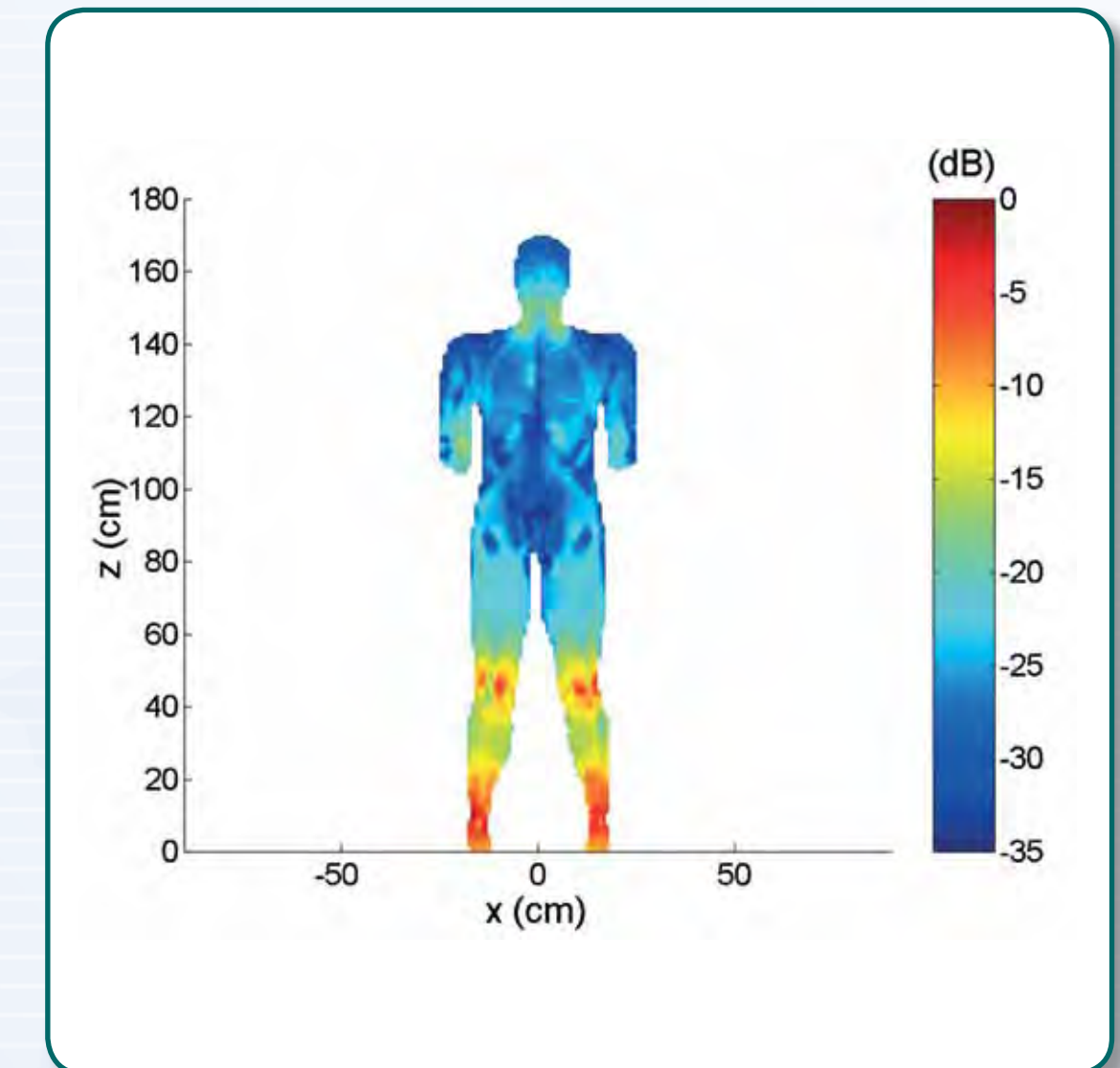
New broadband sensors, based on spiral and toothed antennas with diode sensors covering 40 GHz to 300 GHz, and thermal sensors, based on a fibre-coupled semiconductor thermometer for field strength measurements in free-space, were developed and validated.

SAR measurement setups were developed and characterised for the assessment of communication signals and reference liquid dielectric material properties. Existing measurement setups and theoretical models (e.g. relating surface currents to internal fields during MRI scanning) were optimised and inter-comparisons on SAR calibrations, on specific heat measurements and on theoretical calculations, were performed.

A field generator for a sample container containing fluorescent dyes was produced. Calculations of SAR distribution in artefact standards, phantoms and biological material monolayers, as required for micro-dosimetry, were also performed. Finally, a setup for spatially resolved measurements in a thin film on top of the coplanar waveguide was built and subsequently monitored by thermal tomography.



Measurement of induced body currents.



SAR induced by the simulated plastic welder.

New standards

Provided traceable standards, where there were previously none, for the sensor calibration of multi-frequency signals, signals with large bandwidths and pulse-modulated signals.

Produced artefact standards for SAR and dielectric properties with an extended frequency range up to 10 GHz. These will be used by wireless communication companies to demonstrate compliance of communication devices with the defined exposure limits.

New calibration facilities

Established facilities for calibrating commercial probes, and extending the frequency range from 45 GHz to 300 GHz.

Input into existing standards

Input into international standards: Institute of Electrical and Electronics Engineers, Inc. (IEEE) 1309 'Calibration of Electromagnetic Field Sensors and Probes, Excluding Antennas, from 9 kHz to 40 GHz' and national guidelines: VDI (The Association of German Engineers) VDI/VDE/DGQ/DKD 2622 'Calibration of measuring equipment for electrical quantities'.

The 'Virtual Family'

Validated the use of the computer model 'Virtual Family' for modelling EMF and SAR with human subjects – used by communications companies to design devices such as body-worn antennas.

