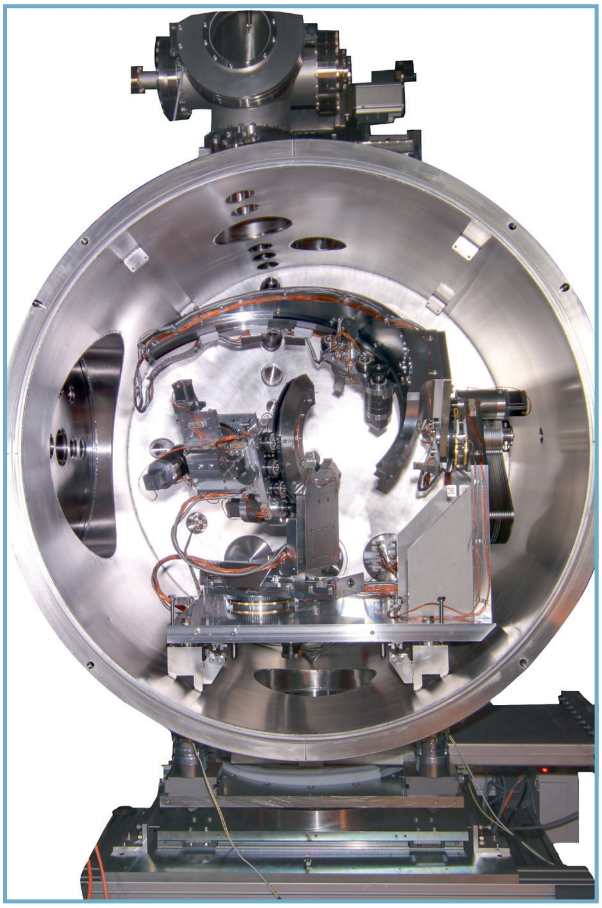
Division 7
Temperature and Synchrotron Radiation



The Division *Temperature and Synchrotron Radiation* realizes and disseminates the respective scales for a very wide measuring range in the following fields: thermometry, radiometry and vacuum metrology. Synchrotron radiation of the electron storage rings *Metrology Light Source* (MLS) and *BESSY II* is used for radiometry in the short-wave range (X-ray to UV range), but increasingly also for nanometrology and (micro)spectrometry. The measurement of thermal energy is gaining more and more importance, and the worldwide leading position in the development of SQUIDs in the field of cryogenic sensors could be substantiated in several applications (see also "News of the Year"). R&D activities are performed in all of the above-mentioned fields to an ever increasing extent, especially within the scope of the European Metrology Research Programme (EMRP). In the following, some selected developments and results will be presented.

Metrology with synchrotron radiation

At the MLS, the new measuring set-up for the calibration of radiation sources has been extensively characterized and validated. Thereby, the MLS serves as a primary source standard. When determining spectral radiant intensities and radiances of deuterium and tungsten strip lamps, very good agreement was achieved with the measurement results from BESSY II and in comparison with thermal cavity radiation.

Since October 2013, the extensive measurements carried out within the scope of the many years of cooperation on EUV lithography have, for the major part, been performed at the EUV beamline of the MLS. For this purpose, PTB's EUV reflectometer was transferred from BESSY II to the MLS in the summer of 2013. The more than 110 participants from industry and research who took part in the 273rd PTB Seminar «VUV and EUV Metrology» (which was held at the auditorium of the Helmholtz-Zentrum Berlin in Berlin-Adlershof on 24 and 25 October 2013 (Fig. 2)) could see for themselves that the EUV reflectometer had been successfully commissioned at the MLS (Fig. 1). In return, a new EUV ellipsometric scatterometer was commissioned at the EUV beamline at BESSY II (see the cover picture).

An arrangement was made with the *ISAS-Institut für Analytische Wissenschaften ("ISAS Institute of Analytical Sciences")* and the *Technical University of Berlin (TU Berlin)* to carry out joint quantitative investigations on surfaces by means of ellipsometry and electron spectroscopy at the undulator beamline of the MLS. It is envisaged to continue experiments on organometallic layer systems – which play a role in the fields of, e.g., optoelectronics and photovoltaics.

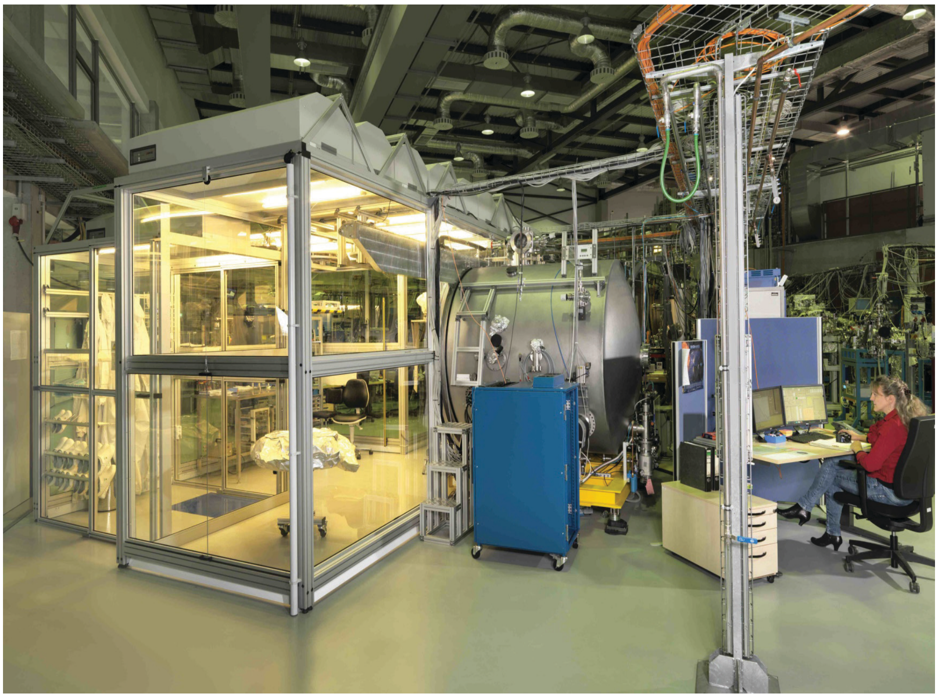


Figure 1: PTB's EUV reflectometer at the EUV beamline of the MLS

Cover picture:
PTB's EUV ellipsometric scatterometer at BESSY II

Figure 1: PTB's EUV reflectometer at the EUV beamline of the MLS

This will be done – among other things – within the scope of the new EMRP project *ThinErgy*.

PTB's worldwide unique capabilities in the field of detector-aided radiometry with synchrotron radiation were exploited to calibrate two special detector systems. On the one hand, a bolometer detector for applications in plasma diagnostics was calibrated from the UV to the hard X-ray range on five beamlines at PTB's laboratories at BESSY II and at the MLS for the IPP in Garching. On the other hand, photoionization cross sections of noble gases were measured in cooperation with DESY in Hamburg in the photon energy region between 2.1 keV and 30 keV; these are necessary to calibrate gas monitor detectors of the European X-ray laser XFEL.

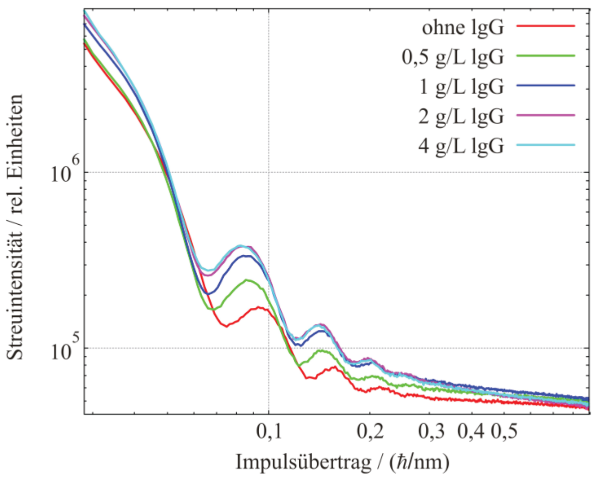


Figure 3: Scattering curves of functionalized polystyrene nanoparticles in gamma globulin (IgG) solutions of different concentrations.

After the successful completion of a doctorate on the traceable determination of the size of spherical nanoparticles at the TU Berlin in April 2013, the present activities on dimensional nanometrology via small-angle X-ray scattering with synchrotron radiation at BESSY II are now focusing on more complex systems – for example, on functionalized core-shell particles in a biological environment (Fig. 3), which are being investigated within the scope of EMRP. In addition, lattice structures, photomasks and surface roughness have been characterized in reflection geometry both in the X-ray and in the EUV range, partly within the scope of doctorates.

The «X-ray and IR Spectrometry» Working Group has been engaging successfully in the development of instrumentation for X-ray fluorescence (XRF) analysis for several years. A UHV measuring chamber for XRF analysis – which was developed at PTB and has been successfully used by the working group at BESSY II – has now been built on request for the IAEA and delivered to be operated at the ELETTRA storage ring (Fig. 4). This chamber will be available for use by the 151 member states of the IAEA to carry out XRF analysis experiments as of 2014 (IAEA XRF Newsletter No. 24, March 2013). Other chambers have been ordered by the cooperation partners TU Berlin and the French metrology institute LNE/CEA-LNHB to be used at the SOLEIL storage ring.

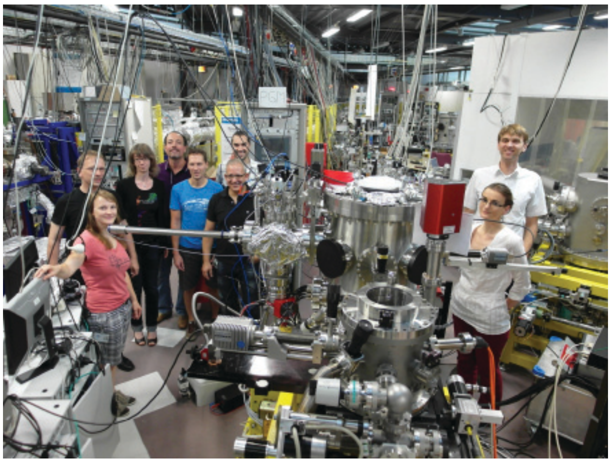


Figure 4: UHV measuring chamber of the IAEA at the PGM beamline of PTB at BESSY II

Thermometry

For the metrologists dealing with temperature, a highlight was the "Symposium on Temperature and Thermal Measurements in Industry and Science" (TEMPMEKO), which is organized every three years and took place in Madeira, Portugal. In October 2013, 300 top scientists from 42 countries met there to discuss new developments with regard to the realization and dissemination of scales, the introduction of novel, eutectic high-temperature fixed-points, and the re-definition of the kelvin within the scope of more than 350 presentations. Corresponding to their international reputation, the thermometry experts from Division 7 were involved in 32 presentations, one of which was a guest presentation on the re-definition of the kelvin.

From 4 to 7 June 2013, the national temperature congress "TEMPERATUR 2013" was held at the Berlin Institute of PTB with 124 external participants, specifically from industry and from the metrology and verification authorities. Departments 7.3 and 7.4 thus hosted this practice-oriented congress on procedures and instruments for thermal and gas humidity measurements for the fifth consecutive time. This congress is organized in close collaboration with VDI and the Temperature and Humidity Technical Committee of DKD (the German Calibration Service).

The second determination of the Boltzmann constant by PTB is a milestone on the way to the re-definition of the kelvin. In 2011, PTB published its first determination at the triple point of water using dielectric constant gas thermometry. With 4.3 ppm, the new measurements, which were published in December 2013 in the scientific journal *Metrologia*, exhibit relative uncertainties which are half as high as in the previous measurements. An important factor for this was the low deformation of the measuring capacitor under pressure due to electrodes made of tungsten carbide. The determination of the deformation (Fig. 5), the analysis of the impurities in the measuring gas helium, as well as the reproducibility of the capacitance measurements, were improved. PTB's latest results provide, for the first time, an independent measurement method to guarantee that the other values of the Boltzmann constant, which were determined by means of acoustic gas thermometry, exhibit the quality required by the international specialized committees.

The Division's effort in the EURAMET committees to obtain full entries in the BIPM's database for thermometric calibration services has, at long last, been successful. Figure 6 proves PTB's effort to provide its customers with calibrations from the lowest temperatures in the millikelvin range up to the highest temperatures which are measurable only by means of radiation thermometers. The entries are grouped into instrument categories, arranged clockwise according to increasing temperature, starting with thermometers for the low-temperature scale PLTS-2000. With a total of 112 entries at present, now each PTB calibration certificate for thermometers can be issued with a reference to the BIPM's

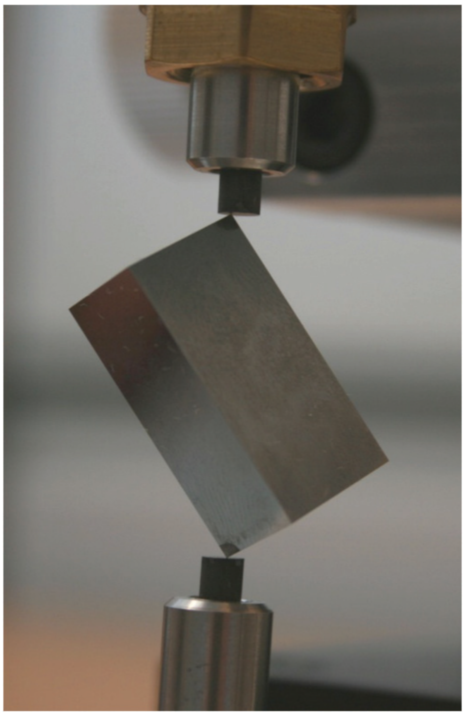


Figure 5: Ultrasonic resonance spectroscopy to determine the deformation of the capacitors of the dielectric constant gas thermometer

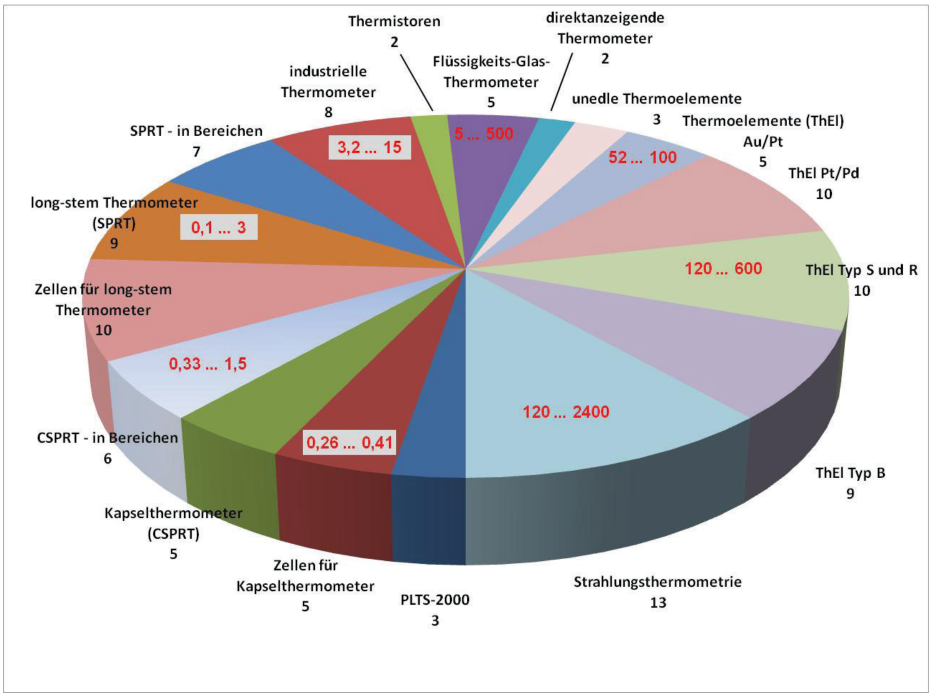


Figure 6: Number of PTB entries in the BIPM's database for thermometric calibration services, arranged according to the type of thermometer. In red font: some typical expanded uncertainties, indicated in mK.

database. This is a considerable competitive advantage for the numerous calibration laboratories for thermometry that are supervised by the German accreditation body, DAkkS, and for the worldwide leading German thermometer SMEs, as the calibration certificates for their products can, thus, be recognized abroad without any difficulty. But also at the international level, PTB occupies a leading position, both with regard to the width of the temperature range and to the uncertainties. For example, PTB is the only state institute which can provide calibrations with entries in the BIPM's database in the low-temperature range below 14 K.

When it comes to the laser hardening of high-alloyed steel materials or cast iron in the temperature range of up to 1300 °C, the temperature has to be regulated within less than a few kelvins in order to achieve the desired hardening properties of a material in a process-safe way. Within the scope of the EMRP project "High-temperature metrology for industrial applications (> 1000 °C)", a mobile calibration fixed-point radiator has been developed in collaboration with the *Fraunhofer Institut für Werkstoff- und Strahltechnik ("Fraunhofer Institute for Laser and Surface Technology"* – IWS) specially for laser-hardening applications. The good practicality of the calibration radiator was demonstrated at several hardening plants in an industrial environment.

Division 7 also takes account of the increasing importance of imaging metrology in non-contact temperature measurement, both by supporting the technical regulations to be elaborated in the relevant committees, and by developing measuring set-ups which allow imaging temperature measurement systems to be characterized metrologically with a low measurement uncertainty, in accordance with the technical regulations. This was also the context in which the constituting session of the new VDI/VDE technical committee GMA 8.16 "Temperature measurement with infrared cameras" – which took place at PTB in June 2013 – is to be seen. A novel transfer standard of variable radiation density, which is based on an integrating sphere, is now available for the radiometric characterization and calibration of imaging detector systems, in particular for instrumentations used for the remote sensing of the Earth. This transfer standard has a radiation density which can be varied over more than two orders of magnitude without experiencing significant changes in the spectral distribution (Fig. 7).

Radiometry

The Division's activities for the traceability of radiometric and radiation-thermometric remote sensing of the Earth have been successfully continued. The World Meteorological Organization (WMO) – within the scope of its recognition of the strategic research of the EMRP in the environmental field – has, vis-à-vis EURAMET, emphasized the significance of four projects, two of which ("SolarUV" and "Metrology for Earth Observation and Climate") are mainly being worked on by Department 7.3. In addition to the *radiator-based* radiometric traceability, the so-called "Calibration Home Base" (CHB), a *detector-based* traceability in the spectral range from 300 nm to 3 µm, could be guaranteed within the scope of a cooperation project with the Remote

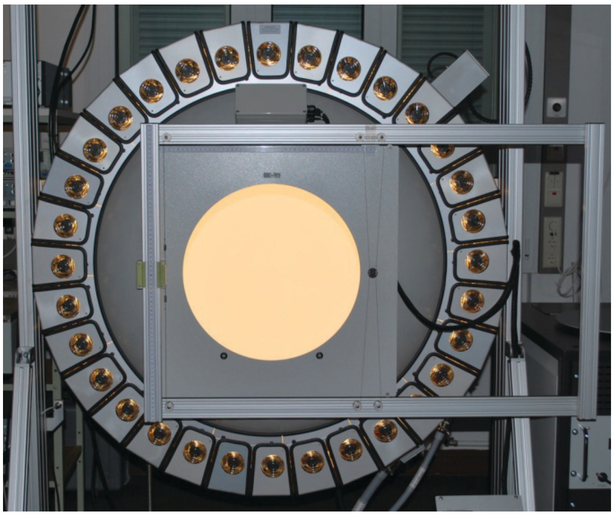


Figure 7: Illuminated integrating sphere with a radiating surface of 40 cm in diameter, used as a transfer standard of variable radiation density for the calibration of imaging radiometric and thermometric measuring systems.

Sensing Technology Institute (*Institut für Methodik der Fernerkundung – IMF*) of DLR in Oberpfaffenhofen, using five filter radiometers whose absolute calibration had been carried out at PTB. The CHB, as an essential calibration facility of DLR for the remote sensing of the Earth, is now linked up with the two primary standards of PTB (the cryogenic radiometer and the black body) via independent traceability chains.

PTB's two emissivity measuring arrangements in air and under vacuum now allow investigations of materials from 25 °C (in air) and from -40 °C (under vacuum) up to temperatures of 600 °C and wavelengths of 100 µm. Emissivity measurements at room temperature in air are important for industry-related issues such as the emissivity of textiles, or of materials in the automotive sector. Measurements at very low temperatures and high wavelengths are relevant for the characterization of coatings of reference radiation sources for the remote sensing of the Earth.

By using a reference detector specially designed for THz radiation as well as a THz molecular gas laser, THz detectors can now be calibrated in the full spectral range from 1 THz to 5 THz at standard measurement uncertainties of the spectral responsivity of less than 2 %. In order to develop a radiation detector for the FIR and for the THz spectral ranges with a far better linearity than commercial bolometers, a transition edge sensor (TES) bolometer was developed. This is based on three components that were manufactured at PTB by means of lithographic processes and then optimized: an absorber made of structured thin metal layers on a silicon nitride membrane, an aluminium/niobium bi-layer as the actual TES sensor, as well as a SQUID as a current sensor (Fig. 8). The first measurements carried out have shown a spectrally flat and linear responsivity over a large measuring range and, thus, a good suitability of the detector for quantitative FIR and THz spectroscopy.

In the field of single-photon radiometry, the use of the MLS as a radiation source with a photon flux

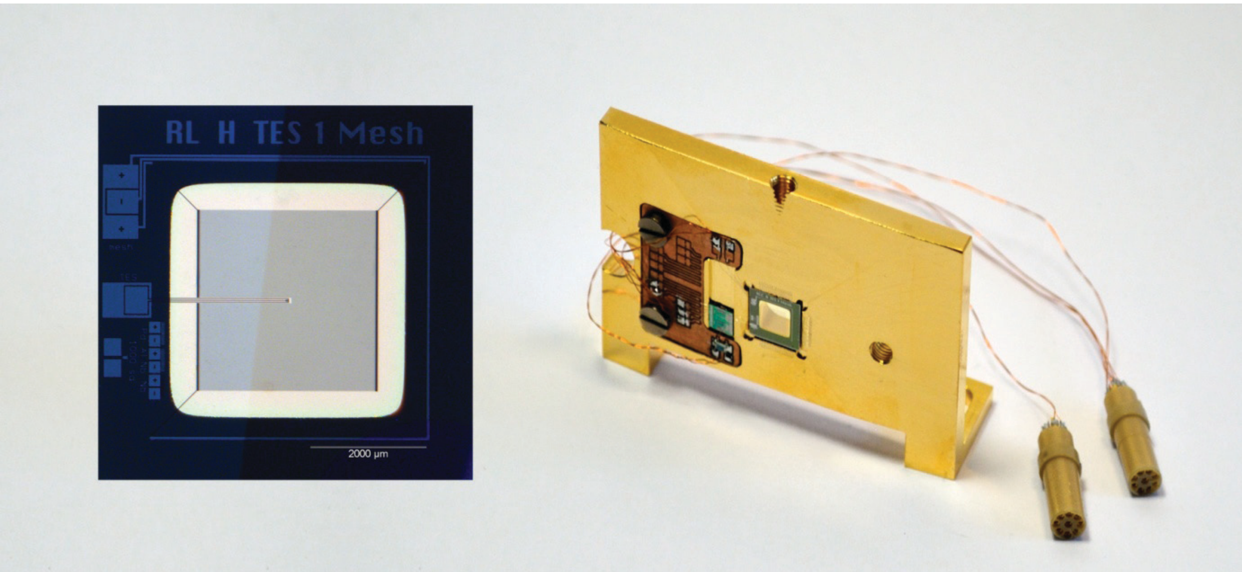


Figure 8: PTB's TES bolometer for FIR and THz spectroscopy, which is characterized by a large linear working range and a spectrally flat absorbance for radiation with a wavelength from 100 µm to more than 3 mm.

that can be adjusted over 11 orders of magnitude has been successfully continued. At the MLS, the spectral responsivity of glass-fibre-coupled, superconducting single-photon detectors was linked up in the NIR range with a cryogenic radiometer with relative measurement uncertainties below 2 % within the scope of the EMRP project "Metrology for industrial quantum communications".

Cryogenic sensors

In the past few years, the project "European Microkelvin Collaboration" was successfully completed. A total of twelve partners from eight countries were involved in this project which had a duration of four years. PTB's contributions focused on the development of SQUID systems for use in ultra-low-temperature facilities of project partners. Hence, the SQUID systems thus developed are now used, e.g., at the Royal Holloway University of London for NMR experiments and for noise thermometry. Further detector systems are used in low-temperature groups at the Aalto University of Helsinki, the University of Heidelberg, the University of Leiden, the University of Basel, and the Néel Institute in Grenoble. The association of the partners involved has durably strengthened the low-temperature community in Europe – which has, for example, led to the creation of the Low Temperature Section at the European Physical Society. The relationships which were established within the scope of this project will persist long after the process has been completed. The partners have agreed upon a follow-on project which they are planning to initiate.

Division 7 has been supporting the activities of Division 2 for several years by means of metrological developments which are needed in the field of resistance metrology and for the redefinition of the base unit *"ampere"*. Thus, a novel amplifier design, a so-called "Ultra-stable Low-noise Current Amplifier" (ULCA), has been developed and tested successfully for high-precision and traceable measurements of currents in the range of 100 pA (see also "Report of Division 2"). A patent application has been filed for this amplifier design.

Vacuum metrology

The new dynamic standard for fast varying pressures under low vacuum has been commissioned (Figure 9). Vacuum measuring instruments which control fast processes in industrial applications, for

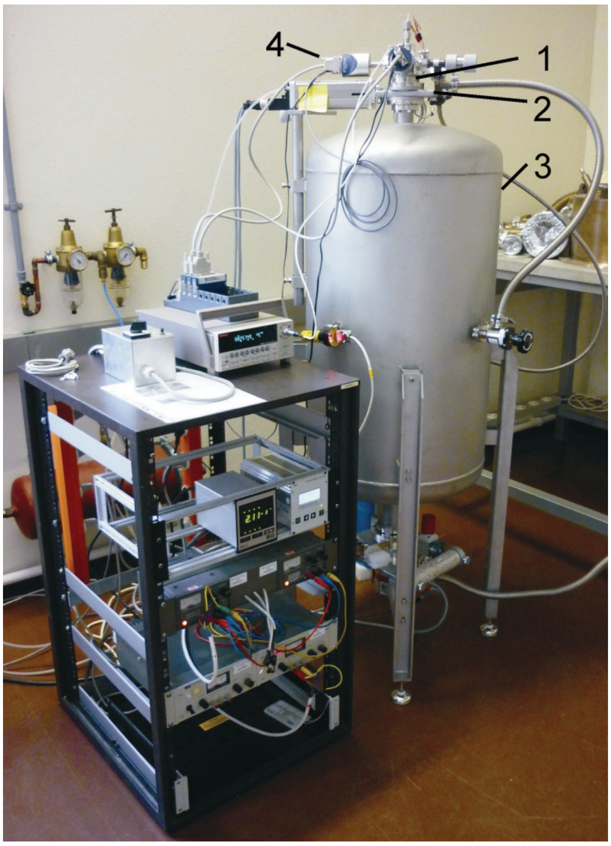


Figure 9: The new dynamic vacuum standard. Due to the gas expansion from a very small (1) into a very large volume (3) induced by a fast opening gate valve (2), the pressure in the small volume can be reduced by 3 orders of magnitude within 20 ms, and the response time of a vacuum gauge (4) can be investigated.

example gate chambers, should react immediately to changes in pressure. In order to test the responsivity of vacuum measuring instruments, a pressure drop from 100 kPa down to 100 Pa can be generated within 20 ms, or the pressure can be predictably reduced from 100 kPa to 100 Pa within a second by means of a diaphragm or by means of de Laval nozzles. To this end, sophisticated simulations were necessary. Capacitive membrane vacuum gauges of a project partner in the EMRP project IND12 "Vacuum Metrology" which are optimized for a fast reaction exhibited a response time of 1.3 ms maximum for a 1/e pressure drop. Since the measurement uncertainty of these vacuum gauges is smaller than the uncertainty of the calculation, it is envisaged to use them as reference standards for fast varying pressures, which is an excellent example of fruitful cooperation between industry and a metrology institute.

Thermal energy

A laser-optic, high-temperature flowrate standard, based on a commercial gauge-glass gate valve and a precise sonic venturi nozzle, was set up as a so-called “insert” (Figure 10). Due to two physically independent procedures, the standard allows the volume rate to be determined simultaneously – on the one hand, by means of the differential pressure procedure which has established itself in industry, and, on the other hand, by means of traceable laser-doppler anemometry (LDA). The standard is designed for temperatures up to 230 °C at operating pressures of up to 40 bar in DN150. By selecting another gauge-glass gate valve, the system can be adapted to considerably higher temperatures (600 °C) and pressures (800 bar) and therefore opens up the possibility of being used for the direct calibration of flowrate sensors under the conditions prevailing in power plants. A recalibration of the nozzle at regular intervals can be carried out by means of full-scale LDA measurements without having to remove it. For the time being, it is planned to use it as a primary standard for an existing high-temperature flowrate testing facility. But due to its robust design, the measuring system is also suitable for applications in an industrial environment.

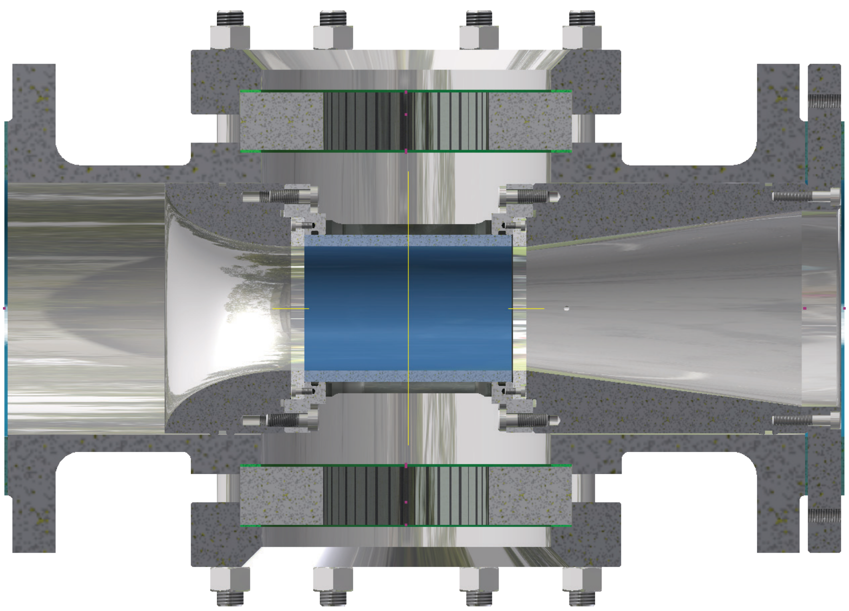


Figure 10: Gauge-glass gate valve with venturi insert as the core piece of the laser-optic flowrate standard (here: cross-sectional view). The volume rate is yielded from the integration of the laser-optically determined velocity profile across the surface of the precision glass pipe (represented in blue) and from the pressure difference over the venturi outline.

Headlines: News from the Division

Fundamentals of Metrology

Measurement of the spectral responsivity in the extreme UV (EUV)

Detector for plasma diagnostics calibrated from the UV to the X-ray range

Photoionization cross sections in the X-ray range

Measuring set-up for the calibration of radiation sources at the *Metrology Light Source*

Counting electrons at the *Metrology Light Source*

Cause of additional noise in Chopper amplifiers found

Scattered light near-field microscopy and nano-FTIR spectroscopy at the Metrology Light Source

New SAN storage system put into use

New plasma etching facility for SQUID micromanufacturing

Characterization of the undulator at the *Metrology Light Source* (MLS) for the calibration of single-photon detectors

Calibration of fibre-coupled single-photon detectors at the telecommunications wavelength of 1.55 µm

EMRP Project "New primary standards and traceability for radiometry" started

Determination of the non-linearity of terahertz spectrometers

Reduction of platinum oxide

Improved high-precision determination of the Boltzmann constant at PTB

Precise determination of the virial coefficients of helium

Improved pressure measuring technique for thermometry

"Predictable" magnetic-field-fluctuation thermometer

Dynamic standard for fast varying pressures in vacuum commissioned

Stereo Particle Image Velocimetry established as a method for flowrate diagnostics

Computational Fluid Dynamics (CFD) in flowrate measurement

Metrology for the Economy

High deflection efficiency in the case of coated laminar lattices

Comparative investigation of photomasks with EUV scatterometry and AFM

Move of the EUV reflectometer to the *Metrology Light Source*

273rd PTB Seminar

Metrology for Society

International Affairs

European comparison for low-temperature radiation thermometry (EURAMET 658)

Traceability of the emissivity for CENAM

Assessment of national institutes

"Mise en pratique" for the realization of the temperature base unit

CCT working group for the re-definition of the SI base unit *"kelvin"*