PTB news
Scientific Newsletter
1
2015

Three times a year, PTB News provides topical information from the varied spectrum of activities of the Physikalisch-Technische Bundesanstalt (PTB) consisting of fundamental research, legal metrology and PTB's various activities in the service of the economy.

Atoms for the kilogram

The measurements carried out by PTB play a decisive role in the redefinition of the units

Especially interesting for
- fundamentals of the system of units

The Avogadro constant and Planck's constant are the corner stones of the new system of units. The measurements with regard to these constants have been expedited within the scope of the international Avogadro Project; they are based on counting atoms in spheres made of pure silicon. A few months ago, Russian contracting laboratories supplied practically isotopically pure silicon so that the Leibniz Institute for Crystal Growth (Leibniz-Institut für Kristallzüchtung – IKZ) in Berlin was able to grow a single-crystal out of it; this single-crystal has now been delivered to PTB. PTB will now produce the spheres and carry out all the subsequent measurements. The declared aim is to determine the Avogadro constant with a relative uncertainty of $1 \cdot 10^{-8}$ or better.

According to the Comité International des Poids et Mesures (CIPM), the International System of Units (SI) is to be defined on the basis of fundamental constants. Especially the base units the kilogram, the ampere, the kelvin and the mole are in the focus of the redefinition. Hereby, clearly determined numerical values are attributed to the fundamental constants concerned. Prior to this, however, these values have to be measured as accurately as possible. For the redefinition of the kilogram, PTB is pursuing a very promising approach by "counting" atoms in a silicon single-crystal using the so-called "X-ray Crystal Density (XRCD) method". Hereby, the Avogadro constant is determined by measuring the crystal properties (the molar mass and the crystalline lattice parameter, among other things) as well as the sphere properties (the mass and the volume, among other things).

However, to be able to measure the molar mass with sufficient accuracy, it is necessary to use a crystal made of practically monoisotopic silicon. For this purpose, PTB concluded a contract concerning the delivery of 12 kg of ^{28}Si with the export trading company ISOTOPE in Moscow. The materials specifications stipulated a high degree of enrichment of the isotope ^{28}Si of more than 99.99 % as well as an

From the polycrystalline material from Russia, the Institute for Crystal Growth (IKZ), Berlin, grew a high-purity ^{28}Si single-crystal. It has an isotopic purity of 99.998 %. From this material, PTB produces nearly perfect spheres which will be analyzed to determine the Avogadro constant and Planck's constant.

exceptionally high chemical purity. The silicon isotope is enriched by means of centrifuges of the Electrochemical Plant in Zelenogorsk – the largest supplier of any type of isotopes worldwide. The gaseous silicon tetrafluoride ($^{28}SiF_4$) is then transformed into silane ($^{28}SiH_4$) at the Institute of Chemistry of High-Purity Substances of the Russian Academy of Sciences in Nishniy Novgorod; it is then painstakingly purified and eventually deposited as polycrystalline silicon onto a thin, electrically heated silicon rod, a so-called “slim rod”.

The first polycrystal with a mass of 6 kg was delivered from Russia in August 2014. From this polycrystal, the Leibniz Institute for Crystal Growth (IKZ) in Berlin grew a single-crystal which was handed over to PTB in March 2015. From this single-crystal, PTB will manufacture – by means of a complex production line which was especially developed for this task – a total of two spheres having a mass of approx. 1 kg each for the determination of the Avogadro constant and of Planck's constant. The production process has meanwhile been improved to such an extent that clean oxide layers and a roundness clearly better than 50 nm can be obtained. The delivery of a second polycrystal is planned for mid-2015. Then, four silicon spheres in total will be available to determine the Avogadro constant and Planck's constant with the greatest accuracy. In addition, when the system of units has been redefined, the “silicon kilogram” will allow the unit of mass to be realized and disseminated in a feasible way. ■

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Mathematical modelling of flows in cells

New findings on pattern formation in biological cells

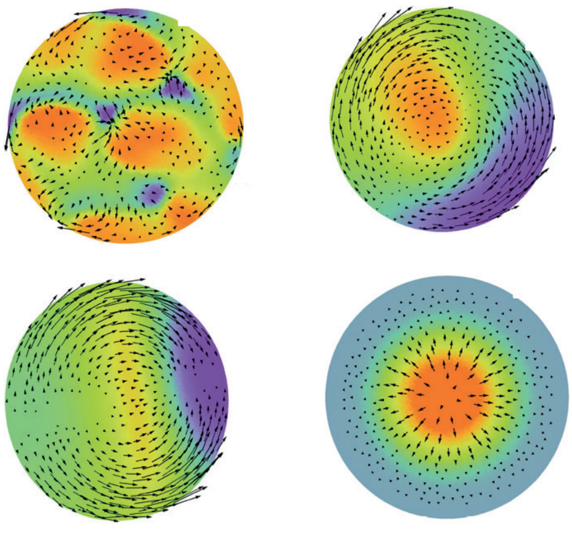
Especially interesting for
- biophysicists
- cell biologists

In a new model for the formation of biological patterns, developed by mathematicians from PTB, cells are described as active porous and elastic materials. Simulations carried out using this model were able to reproduce experimentally wave patterns that had been observed in microdrops of the slime mould *Physarum polycephalum* and allow predictions for new experiments.

Biophysicists and cell biologists have been strongly interested recently in how spatial structures form spontaneously in cells and tissues and which physical, chemical and biological mechanisms are decisive for this process. Whereas biochemical and genetic processes have long been investigated as “driving forces”, mechanical forces and the associated flow processes inside the cell have recently moved into the focus of research. Knowing the dynamics of such mechanisms and of chemical processes in cells is important for the understanding of processes such as cell motion and the development of cell tissues.

Mechanical forces are generated by the contraction of clusters of the biopolymer actin. This motion, in turn, is generated by the activity of myosin molecules that act as molecular motors. The myosin-regulating calcium ions inside the cell have to redistribute to enable this process to create biological structures; this causes a feedback reaction towards the mechanics of the actin fibre bundles.

A model which was developed in a cooperation project between PTB and TU Berlin describes the cell as a porous and elastic (“poroelastic”) medium and has now been applied successfully to the biophysical model organism *Physarum polycephalum*. Physarum cells contain a great number of cell nuclei and reach macroscopic dimensions, exhibiting frequently complex geometric shapes. If a small amount of cell plasma is taken from larger cells, cylindrical microdrops form. In these drops, many different patterns of deformation waves are observed. PTB's model allows all these patterns to be reproduced successfully. In addition, the model predicts a strong coupling of the mechanical processes to the intracellular fluid dynamics and the local calcium concentration.



These numerical simulations of the poroelastic model show the microdrops with different structures. From top right (clockwise): turbulent structures, rotating spiral waves, an antiphase oscillation, and an “even” wave running from right to left. The colours indicate regions of mechanical contraction (blue) and relaxation (red) which cause flow motions with velocities in the range from 0.1 µm/s to 1 µm/s.

Such simulations can also be used for the quantitative assessment of dynamic processes in cells and may trigger new measurements of the intracellular flows and spatial distributions of calcium and actin (e.g. by means of fluorescence methods) as well as of the mechanical and elastic properties of the cell material. ■

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Scientific publication
M. Radszuweit, H. Engel, M. Bär: An active poroelastic model for mechano-chemical patterns in protoplasmic droplets of Physarum polycephalum. PLOS One 9, e99220 (2014)

Intercontinental comparison of optical clocks – do all clocks tick the same?

For the first time, optical atomic clocks have been compared over a distance of 9000 km – directly and in real time

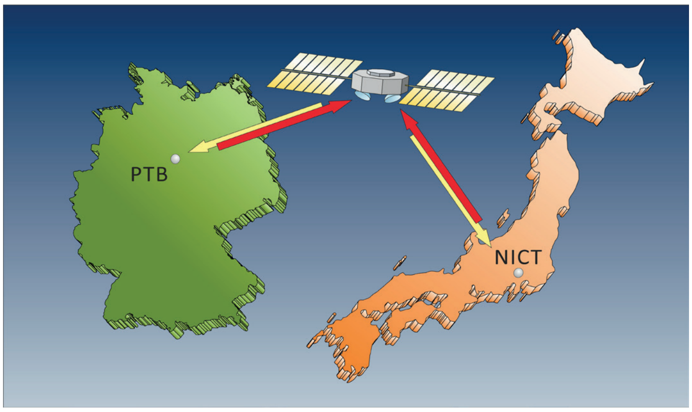
Especially interesting for
- developers of optical clocks
- geodesy
- fundamental research

Intercontinental comparisons of atomic clocks can, at present, only be realized with the aid of satellites. The measurement noise occurring hereby determines the measurement time required. To compare the optical strontium lattice clocks of PTB with those of the Japanese Institute of Information and Communications Technology (NICT) in Tokyo, a new method with improved stability, developed by NICT, has now been used.

Optical frequency standards are currently being investigated at research institutes worldwide, for example in view of their use for a new definition of the second. One of the issues hereby is to which extent atomic clocks of the same type really provide identical frequencies. Clocks of the strontium lattice type are relatively often investigated, thus, for example, at NICT and at PTB. In order to compare these two clocks, a measurement campaign was started in the spring of 2013 where a method developed by NICT was used to exploit the carrier phase in two-way frequency comparisons via satellite. Compared to previous satellite-based methods with modulated signals, this leads to lower noise and improved stability. Also, the transmission band width required is smaller. The 9000 km distance between the two institutes was bridged by means of a geostationary TV satellite which was operated continuously for a few months. The main result obtained was the proof of a considerably lower instability for frequency comparisons at short averaging times of $2 \cdot 10^{-13}$ in 1 s.

In the summer of 2013, the optical strontium lattice clocks of NICT and PTB were compared directly with each other, which was the first comparison of this kind worldwide. It was shown that the two clocks were in agreement within the total measurement uncertainty of $1.6 \cdot 10^{-15}$. Thereby, the measurement uncertainty of the strontium lattice clocks involved is so low that the total measurement uncertainty is mainly due to the comparison. This real-time method is thus an alternative to comparisons via primary caesium atomic clocks or Coordinated Universal Time and is, in principle, available also to institutes without a direct connection to the metrological infrastructure.

Further improvements by one order of magnitude seem possible if an optimized control of the ambient conditions of the emitting and receiving electronics as well as longer measuring times are used. This is particularly interesting for the further development of optical clocks, but also for fundamental experiments on the theory of relativity or for applications in geodesy where the difference in the rate of the clocks could indicate height differences. ■



Scheme of the comparison of the optical strontium lattice clocks of PTB in Braunschweig and NICT in Tokyo via satellite. The arrows represent the up- (red) and the down-link of the two-way technique which is very well suited to suppress disturbances.

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Scientific publication
H. Hachisu, M. Fujieda, S. Nagano, T. Gotoh, A. Nogami, T. Ido, S. Falke, N. Huntemann, C. Grebing, B. Lipphardt, C. Lisdat, D. Piester: Direct comparison of optical lattice clocks with an intercontinental baseline of 9000 km. Optics Letters 39, 4072-5 (2014)

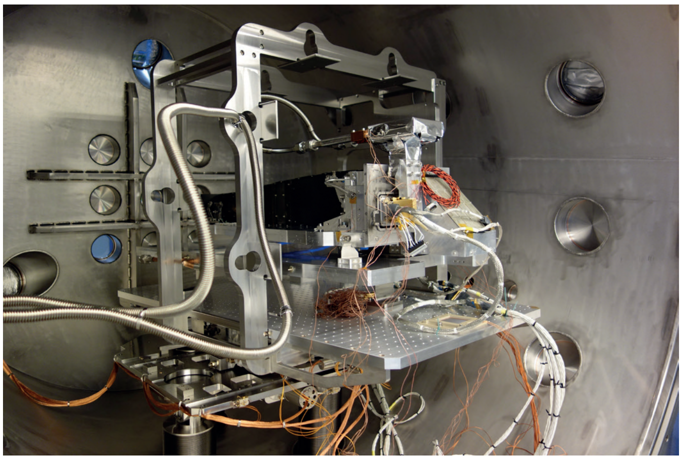
The Metrology Light Source as a standard for solar radiation

Calibration of the solar spectrometer SPICE of ESA's Solar Orbiter mission using calculable synchrotron radiation

Especially interesting for
- solar and atmospheric research
- developers of space telescopes

The SPICE (*SP*ectral *I*maging of *C*oronal *E*nvironment) solar spectrometer is part of the Solar Orbiter mission of the European Space Agency whose start is planned for 2017. Within the scope of a research cooperation project which has been concluded with the Max Planck Institute for Solar System Research (MPS) and in cooperation with the Rutherford Appleton Laboratory (RAL) in England, the SPICE engineering model (EM) has recently been calibrated in such a way that it is traceable to the primary source standard Metrology Light Source (MLS). The actual flying model will be calibrated in the summer of 2015.

In the past years, PTB has calibrated a series of telescope systems for solar observation and atmospheric research using synchrotron radiation in the UV and the vacuum UV (VUV) spectral ranges for various space missions. Hereby, either individual components or the complete systems were characterized by means of transfer radiation sources at the laboratories of the respective cooperation partner. These transfer radiation sources had been developed especially for this purpose and characterized by means of synchrotron radiation. Now that the entire device has been calibrated directly with the calculable synchrotron radiation from the electron storage ring MLS in a new, large vacuum tank, the workaround via VUV transfer sources has become unnecessary. The calibration chain is thus strongly simplified and contributes to attaining lower uncertainties in the range of a few percent. Since synchrotron radiation exhibits a continuous spectrum, all wavelengths in the spectral range of interest are accessible, contrary to the VUV transfer sources previously used which were based on a hollow cathode discharge and which only emit individual lines.



The SPICE spectrograph, mounted into the large vacuum tank at the MLS.

SPICE allows spatially resolved measurement of the VUV emission of the solar surface and the surrounding corona in the spectral range from approx. 48 nm to 105 nm and serves, among other things, to scientifically investigate the processes of creation and localization of source regions of the solar wind and of the solar magnetic field. For this purpose, the incident solar radiation is imaged onto a narrow and long entrance slit by means of a special VUV multilayer mirror and then dispersed onto two detector arrays through a grating. Spectral information is thus gathered on each array in the direction of dispersion and local information is collected in the orthogonal direction in a narrow band of the solar surface or of the solar corona. By tilting the mirror, entire solar regions can be detected.

Calibration at the MLS was performed at a reduced electron energy in order to suppress short-wavelength false light. Use was also made of the possibility of adapting the spectral radiant intensity to the limit of detection of the sensors via the electron current stored. The linearity of the sensors could thus be investigated, which is of great importance due to the strongly varying line emissions of the sun.

The SPICE Project involves the ESA Member States Germany, France, Great Britain, Norway and Switzerland; besides by ESA, it is also funded by the German Zentrum für Luft- und Raumfahrt (DLR – German Aerospace Center), the Centre National d'Études Spatiales (CNES – the French Space Agency), the UK Space Agency, the Norwegian Space Centre, and the Swiss Space Office. The measurements on the SPICE-EM model mainly served to test the calibration scheme and the handling and operation of this complex device together with the corresponding supply infrastructure at the MLS in view of the calibration of the SPICE flying model which is planned for the summer of 2015. ■

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Scientific publication
A. Fludra et al.: SPICE EUV spectrometer for the Solar Orbiter mission. Proc. SPIE 8862, 88620F (2013)

Roughness measurements inside tiny nozzles

Profilometer for roughness measurements on high-aspect-ratio surface features of microcomponents

Especially interesting for
- manufacturers of micronozzles
- manufacturers of metrological equipment

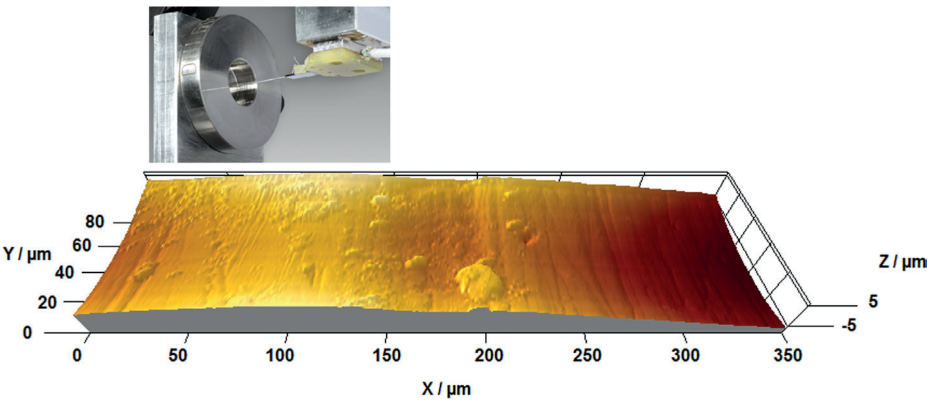
A profilometer, which was developed by PTB together with two cooperation partners, has allowed the inner surfaces of small nozzles with diameters in the micrometre range and a high aspect ratio to be successfully characterized for the first time.

Due to their long-term accuracy and excellent repeatability, small nozzles have been used in a number of industrial and scientific applications for gas and fluid flow measurements such as, e.g., calibration standards. In the case of the sonic nozzles, the flow rate remains constant over the nozzle when the differential pressure exceeds a certain minimum value. The smaller the flow rate, the smaller the diameter of the corresponding nozzle must be. With smaller nozzles, however, the topography of the inner surface has an increased influence on the flow behaviour and the flow rate. For nozzles with a diameter below 1.5 mm, significant deviations from the above-mentioned ideal relation between the differential pressure over the nozzle and the flow rate may occur, depending on the specimen considered; these deviations can turn out to be extremely disturbing in practice and their source in connection with the inner topography are not understood yet. Measuring the inner surface of such small nozzles with conventional tactile and optical measuring instruments is, however, very difficult.

PTB, in cooperation with the Forschungsinstitut für Mikrosensorik und Photovoltaik (Microsensor systems and photovoltaics research institute – CiS) in Erfurt and the Institut für Halbleitertechnologie (Institute of Semiconductor Technology – IHT) of Braunschweig Technical University, has developed a profilometer for roughness measurements on surfaces of microcomponents with a high aspect ratio. Its key component is a long silicon cantilever with an integrated tip and a full-bridge piezoresistive strain gauge for deflection detection. The cantilevers are available in different lengths (1.5 mm, 3 mm, 5 mm) and widths (30 µm, 100 µm, 200 µm) and with tip heights of up to 70 µm. The sensor is mounted into a measuring head with a travel of 800 µm × 800 µm × 250 µm. Three laser interferometers (resolution: 1 nm) are also integrated; they virtually measure the position of the tip. A coarse motorized 3D moving stage allows the precise positioning of samples. Numerous comparison measurements of step heights and of PTB roughness standards proved that the deviations of the profilometer for step heights as well as the roughness the arithmetic mean value Ra lie within ± 10 nm (2 σ).

Diverse nozzles manufactured by different methods (e.g. turning or electrical discharge machining) have been measured by the profilometer. Their diameters varied from 200 µm to 800 µm, the measuring depth from 1.5 mm to 7 mm. It is the first time that the inner surface of sonic nozzles with diameters in the micrometer range has been successfully characterized.

Hereby, in some nozzles, manufacturing defects were detected which explained the deviation of the flow rate experiments performed earlier. This also confirms the assumption that the shape and the roughness of the inner surfaces have a strong influence on the flow rate properties of the nozzles. It is expected that the calibration uncertainty and the quality control of such nozzles with diameters in the micrometre range can be improved by the measurement results of the profilometer. ■



Top: The profilometer in front of a nozzle 800 µm in diameter. The nozzle opening itself is not visible. The cantilever is 7.5 mm long. It consists of two cantilevers which are each 5 mm long and are glued together. Bottom: Measured topography of the nozzle

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Scientific publication
M. Xu, J. Kirchhoff, U. Brand: Development of a traceable profilometer for high-aspect-ratio microstructures metrology. Surf. Topogr.: Metrol. Prop. 2, 024002 (2014)

New picoamperemeter: handy, simple, extremely accurate

The Ultrastable Low-noise Current Amplifier measures pA currents with excellent accuracy

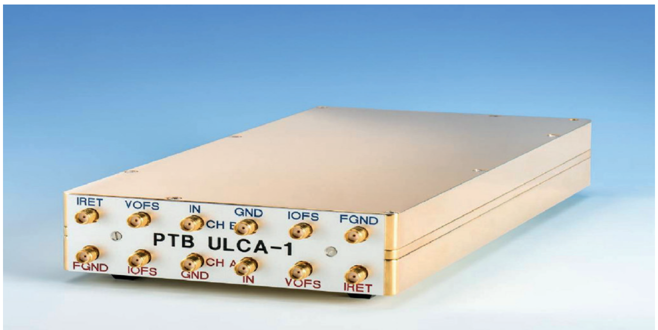
Especially interesting for
- metrology institutes and fundamental research
- calibration laboratories
- manufacturers of high-precision electronics

A handy picoamperemeter has been developed at PTB which is able to traceably measure extremely small currents with unprecedented accuracy. The Ultrastable Low-noise Current Amplifier (ULCA) exceeds the accuracy of previously available picoamperemeters by about two orders of magnitude.

Measuring currents in the range of 100 pA accurately is increasingly gaining in importance – not only for the calibration of picoamperemeters for applications in medical engineering or in the semiconductor industry, but also for fundamental research in the field of current generation based on single electrons. The accuracy of conventional commercially available measuring instruments is limited to relative uncertainties of approx. 10 parts in a million (i.e. 10^{-5}). Better accuracy could previously only be achieved by using extremely complex metrological procedures and apparatuses.

A new development, which is currently being expedited at PTB, opens up new measurement capabilities with unprecedented accuracy. The ULCA is based on a new picoamperemeter concept with a two-stage set-up whose patent is pending. The first stage amplifies the input current by a factor of 1000 while the second stage converts current to voltage. Besides being relatively easy to use, this handy device is characterized by excellent amplifier properties. The second ULCA prototype generation already showed a very low input current noise of 2.4 fA/√Hz with a white spectrum down to very low frequencies of about 1 mHz. The optimized circuit design allows the realization of an amplification factor with maximum stability and linearity (the effective transimpedance is 1 GΩ). In addition, the amplification factor can be calibrated with a relative uncertainty of better than 10^{-7} at averaging times below one hour, hereby ensuring traceability to the quantum resistance standard (quantum Hall effect) by means of a cryogenic current comparator (as used for electric resistance metrology at PTB).

The combination of these excellent properties thus allows the absolute measurement of a 100 pA current within a measuring time of only 10 hours with a relative uncertainty of one part in ten million. The ULCA hereby exceeds the accuracy of previously available picoamperemeters by about two orders of magnitude. ■



Two-channel ULCA prototype (front view with SMA connectors)

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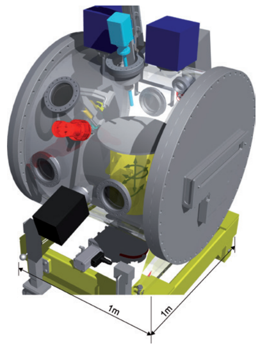
Scientific publication
D. Drung, C. Krause, U. Becker, H. Scherer, F. J. Ahlers: Ultrastable low-noise current amplifier: a novel device for measuring small electric currents with high accuracy. Rev. Sci. Instrum. 86, 024703 (2015)

X-ray analysis for 450 mm wafers

Especially interesting for
- semiconductor manufacturers
- manufacturers of X-ray test equipment

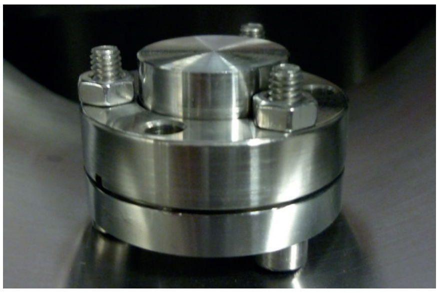
The introduction of the 450 mm technology for wafer production and the further scaling-down of critical dimensions require improved X-ray analytical methods. PTB has therefore designed a complementary metrology chamber to characterize 450 mm wafers; its core component is a multi-axis manipulator whose patent is pending. By spatially stacking all mobile components, this chamber allows several X-ray methods, which are indispensable in the production process of wafers to analyse defects, to be integrated into the smallest possible volume. (Technology offer: #307) ■

Advantages
- *comprehensive 450 mm wafer characterization in one tool with a 1 m² footprint*
- *2D scanning*
- *adjustment of the crystal orientation at any wafer location*
- *maximum reliability and reproducibility*



Reference outgassing sample

Especially interesting for
- vacuum technology
- semiconductor industry
- aerospace technologies



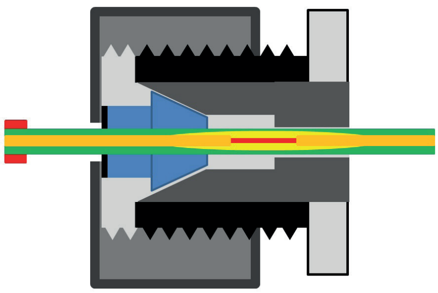
Measuring outgassing rates of vacuum components reliably and quantitatively has become increasingly important over the past few years. With PTB's new reference outgassing sample, such outgassing rate measuring systems can be calibrated and traced back to a national standard. Thanks to its compact set-up, it can be used in a variety of scenarios. It provides a precisely known gas flow which is constant or which decreases only at a constant slow rate over the years. It is designed for the outgassing of diverse gases or gas mixtures. (Technology offer: #357) ■

Advantages
- *constant outgassing rates from 10^{-8} Pa L/s up to 10^{-4} Pa L/s*
- *comparability of measurements carried out in different places*
- *traceable to a national standard*

Compact vacuum feedthrough

Especially interesting for
- manufacturers of vacuum components
- process control engineering

How can highly sensitive optical fibres be introduced into vacuum vessels so that they can be rotated in the narrowest of spaces and displaced longitudinally? A new development by PTB helps solve this problem. It enables optical fibres to be positioned and subsequently adjusted. Hereby, leakages, which may occur in conventional designs, are minimized, the risk of cracking is reduced due to a compression-free holder with integrated strain relief, and the shearing and torsion strains are prevented so that optimum reusability is possible. (Technology offer: #373) ■



Advantages
- *the fibres can be rotated and longitudinally displaced*
- *compression-free integrated strain- and torsion relief*
- *reusable*
- *leakage rate < 5 · 10^{-10} Pa L/s*

Contact person for questions about technology transfer
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Awards

Harald Telle
received the 2014 I.I. Rabi Award for his pioneer work on the utilization of optical frequency combs for phase-coherent frequency measurement.

Franziska Renner
This member of staff from Department 6.2 ("Dosimetry for Radiation Therapy and Diagnostic Radiology") was awarded the Elekta Prize of the Deutsche Gesellschaft für Medizinische Physik for her work "Benchmark experiment to verify radiation transport calculations for dosimetry in radiation therapy".

EMPIR

The European Parliament has approved a research programme, with a funding amounting to 600 million euros, which will further strengthen metrology – the science of exact measurement – to meet the new challenges of our time. The costs of the programme which was proposed by the European Association of National Metrology Institutes (EURAMET), will be borne by the states involved and the EU, both to the amount of 50 %. PTB is significantly involved in the development and implementation of the programme named EMPIR (European Metrology Programme for Innovation and Research).
EMPIR will be running for a total of 10 years; there will be calls for projects on key issues from 2014 until 2020. At the beginning of the programme, the focus will lie on innovation in industry: in this context, improved, more accurate measuring techniques are to ensure the conditions required. By the beginning of October 2014, project proposals with regard to 33 different topics had been submitted. PTB's involvement in many of these is considerable. In addition, the programme is to strengthen more junior research institutes by means of so-called "capacity building". In the further course of the programme, EMPIR will deal with urgent issues in the fields of the environment, energy and health, but also with metrological questions in connection with standardization, and will conduct fundamental research.

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The new Measures and Verification Act ("MessEG")

Since 1 January 2015, the manufacturers of measuring instruments have to adapt to a new act. From then on, the new "Mess- und Eichgesetz" (Measures and Verification Act – MessEG) came into force, and conformity assessment replaced the previous combination of national type approval and initial verification. PTB has prepared for this change and has set up a conformity assessment body in accordance with the new legislation. PTB offers conformity assessment for nationally regulated measuring instruments in the form of various modules, as is already customary for devices traded EU-wide. Promptly at the beginning of the new year, manufacturers of measuring instruments were thus be able to revert to the proven services of PTB. The service offer of PTB's conformity assessment body encompasses approx. 160 types of measuring instruments, sub-assemblies and ancillary devices, whose use has been placed under the protection of the "Mess- und Eichgesetz" (Measures and Verification Act) in Germany.

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PTB at trade fairs

5–8 May 2015
Control
Landesmesse Stuttgart. Hall 1, stand 1313

12–12 May 2015
ZMP
Leipziger Messe. Hall 1, stand 13

15–19 June 2015
ACHEMA
Messe Frankfurt

22–25 June 2015
LASER World of PHOTONICS
Messe Munich. Hall B1, stand 329/1

Contact person for all trade fairs:
Christine Haubold, (+49 (0)531 592-3007, christine.haubold@ptb.de)

Further dates

18–19 May 2015
Towards implementing the new kelvin
Kavli Royal Society International Centre, Buckinghamshire, UK.
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Ramona Peper
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27–30 May 2015
591. WE-Heraeus-Seminar on Astrophysics, Clocks and Fundamental Constants (ACFC 2015)
Physikzentrum Bad Honnef, Hauptstr. 5, 53604 Bad Honnef. Contact: Joachim Fischer
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24–28 August 2015
International Summer School on Metrology
With the kind support of the Helmholtz Fonds e. V. and in cooperation with the Braunschweig International Graduate School of Metrology. Kloster Drübeck.
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Federal Ministry for Economic Affairs and Energy

The Physikalisch-Technische Bundesanstalt, Germany's national metrology institute, is a scientific and technical higher federal authority falling within the competence of the Federal Ministry for Economic Affairs and Energy.