PTB news
Scientific Newsletter
3
2016

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.

A "light highway" for high-precision frequencies

Comparison of optical atomic clocks via new 1,400 km optical fiber link between Braunschweig and Paris – closer to goal of European network of the best clocks

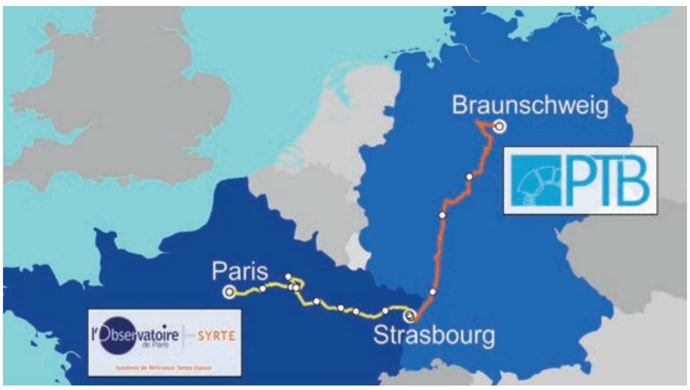
Especially interesting for
- geodesy
- fundamental research in physics

In the past few years, optical atomic clocks have made spectacular progress. They have become 100 times more precise than the best cesium clocks. So far, their precision has been available only locally, since frequency transfer via satellite cannot provide sufficient resolution. This has now changed thanks to a novel 1,400 km optical fiber link between Braunschweig and Paris. This link allows frequencies to "travel" and optical atomic clocks to be compared across national borders. In the first comparison between PTB's optical strontium clocks and those of the French LNE-SYRTE, an unrivaled fractional uncertainty of $5 \cdot 10^{-17}$ was achieved.

Comparisons of clocks at the highest resolution allow a wide range of very sensitive physical experiments to take place, such as the search for time-dependent changes of fundamental constants. In addition, the apparent rate of a clock depends on the local gravitational potential: comparing two clocks measures the gravitational redshift between them, and thus yields their height difference. This research approach is pursued jointly by physicists and geodesists in Collaborative Research Centre 1128 ("geo-Q") of the German Science Foundation (DFG).

Today's most precise atomic clocks are based on optical transitions. Such optical clocks can provide a stable frequency with a fractional uncertainty of only a few 10^{-18}. This is approximately 100 times more precise than the best cesium fountain clocks, which realize the unit of time, the SI second. However, clock comparisons using frequency transfer via satellites are limited to a frequency resolution near 10^{-16}.

For this reason, scientists from PTB and from two French institutes in Paris (Systèmes de Référence Temps-Espace, LNE-SYRTE, and Laboratoire de Physique des Lasers, LPL) have been working for several years on an optical fiber con-



The German part of the link uses commercially rented optical fibres and facilities of the German National Research and Education Network (DFN). The French part of the link uses the Network for Education and Research, RENATER, which is operated by the GIP RENATER. Approximately midway, signals from LNE-SYRTE and PTB meet at the IT Centre of the University of Strasbourg, so that the clocks of the two institutes can be compared there. (Fig.: PTB) nection between the German and French national metrology institutes, PTB and LNE-SYRTE. The 1,400 km link is now complete. Frequency shifts which are generated across the long distance are actively suppressed by up to 6 orders of magnitude, while power losses of 200 dB (10^{20}) are compensated by means of special amplifiers. This allows optical signals to be transmitted with very high stability.

The link enables fast clock comparisons with an uncertainty of less than 10^{-18}. When comparing the two strontium optical lattice clocks, frequency fluctuations of less than $2 \cdot 10^{-17}$ were observed after only 2,000 seconds of averaging time, thereby demonstrating the high stability of the clocks. The 22.7 m difference in height between the two institutes was confirmed by means of the gravitational redshift measured within the clocks' uncertainty of $5 \cdot 10^{-17}$.

This development represents progress toward the goal of a European network of optical clocks connected by optical fiber links which additional European metrology institutes could successively join. This should place them in a leading role for the dissemination of optical reference – a role which, to date, has only been available in a few metrology institutes. In addition, this work clears the path towards a redefinition of the unit of time, the SI second, now that regular international comparisons of optical clocks are possible. ■

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Scientific publication
C. Lisdat, G. Grosche et al.: A clock network for geodesy and fundamental science. Nature Communications 7:12443 (2016)

Laser optical volume flow standard

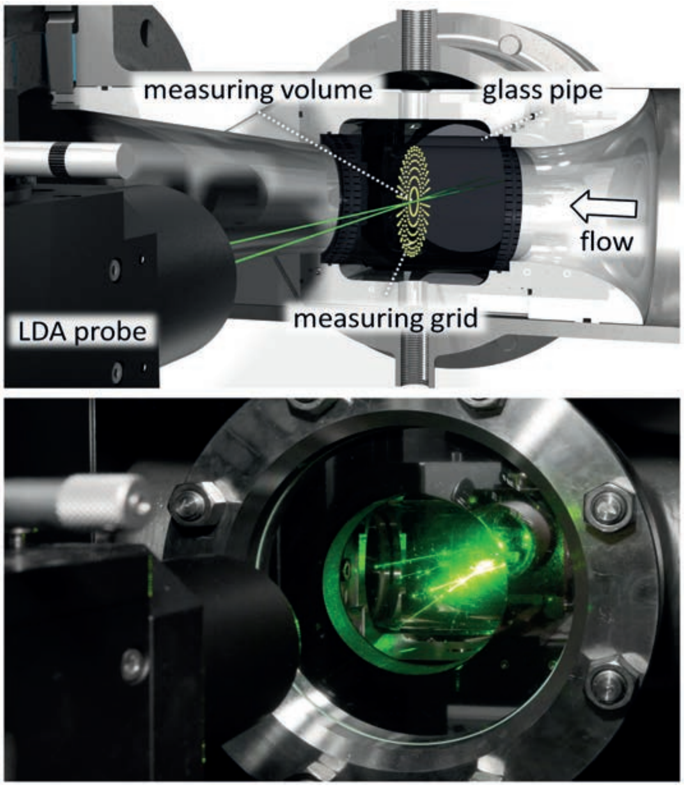
The in-situ calibration of volume flow rate measuring devices allows an increased efficiency of thermal power plants

Especially interesting for
- power plant operators
- manufacturers of volume flow rate measuring devices

The decisive factor for increasing the energy efficiency of thermal power plants is to reduce the measurement uncertainty of the hot water volume flow rate measurement. The current uncertainty of approx. 2 % is not sufficient to optimize the control of power plants, which limits their efficiency. For this reason, PTB has developed a laser optical volume flow measurement standard (LVN) which allows the calibration of measuring instruments with an uncertainty of 0.15 %.

At 2 %, the current uncertainty of volume flow rate measurements in power plants is too high. This is, in part, due to the fact that there is no test facility in the world which enables the calibration of volume flow measuring instruments under conditions that are similar to those encountered in power plants (i.e. at water temperatures of 400 °C and pressures of 300 bar). On the other hand, internal fittings such as valves or bends have an influence on the velocity profile inside a pipe, and thus on the measurement. For this reason, PTB has developed a compact laser optical volume flow rate measurement standard (LFS) which allows measuring instruments to be calibrated on site (i.e. while they are mounted and in operation) with an uncertainty of only 0.15 %.

This procedure is based on laser Doppler anemometry (LDA) which is, itself, based on the scattering of light on small water impurities. Hereby, two laser beams are made to overlap at a certain angle. At the point where the two beams cross, which is the measuring volume, an interference fringe pattern forms. An impurity particle moving through the measuring volume with the flow generates a scattered light signal whose frequency is proportional to the particle velocity. The fluid



In the case of the LDA method, two laser beams are made to overlap. At the point where the beams cross, the measuring volume forms which allows fluid velocities to be measured. The velocity profile inside the pipe is measured on a measuring grid (yellow in the top picture) through a window; by integration, this provides the volume flow rate.

velocity is measured by means of LDA at several positions which are distributed across the section of the pipe. From this data, the velocity profile is reconstructed and integrated in order to calculate the volume flow rate.

The main challenge in developing the LFS was to considerably reduce the current measurement uncertainty of 4.5 % of the LDA volume flow measurement technique. The highest uncertainty contribution hereby came from the local resolution of the measurement procedure, which corresponds to a measuring volume of approx. 2000 µm in length. Due to the extended measurement procedure, the local resolution has already been improved to reach 6 µm. For this purpose, two measuring volumes with variable interference fringe intervals are superimposed, which allows the position at which the particles cross the measuring volume to be determined more accurately. Superimposing the two measuring volumes places high requirements on the positioning of the laser beams. At each measurement point within the pipe cross section, four laser beams with a diameter of 150 µm each must be made to overlap. Measurement procedures have therefore been developed which allow the position of the laser beams to be determined for the first time with high metrological accuracy.

This method provides a measurement uncertainty of 0.15 %, i.e. improved by more than a factor of 10. A comparison measurement carried out with the heat meter test section – a gravimetric standard measurement facility used to realize the volume flow up to 90 °C with an uncertainty of 0.04 % – showed excellent agreement. ■

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Scientific publication
M. Juling: Rückgeführte Volumenstrommessung mittels ortsaufgelöster Laser-Doppler-Anemometrie. Dissertation, TU Berlin (2016), doi:10.14279/depositonce-5170

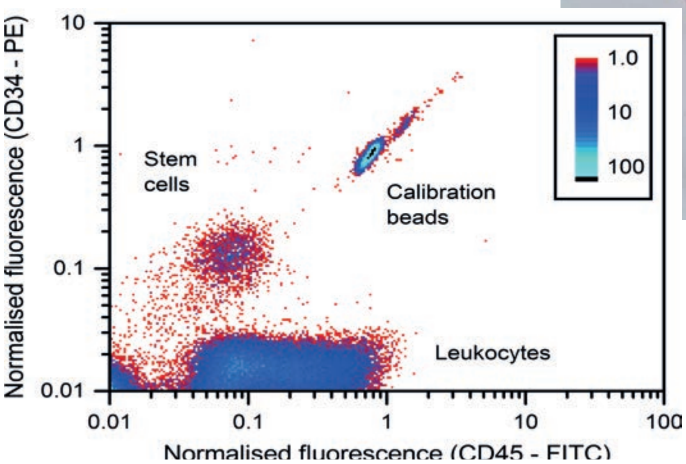
How many stem cells are there in a stem cell product?

Traceable measurements of stem cell concentrations in stem cell products by means of flow cytometry

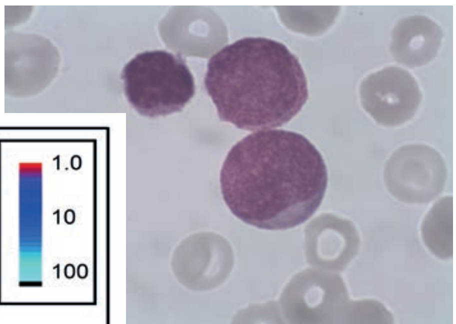
Especially interesting for
- transfusion and transplantation specialists
- oncologists
- haematologists
- specialists in laboratory medicine

When transplanting stem cells, the dose is a decisive factor. To improve the determination of the dose by the measurement of the required concentration of viable stem cells, PTB has developed a reference procedure. It has been applied for the first time for comparison with results of the two methods that are currently used.

Some malignant tumors, such as, e.g., acute myeloid leukemia, can be treated by transferring stem cells from a donor. Flow cytometry is used to determine the required dose. For this purpose, the stem cells are identified and delimited from other cells by means of their cell surface antigens (CD) using fluorochrome-marked antibodies. In a flow cytometer, the fluorescence signals of the cells are recorded individually one after the other and plotted in a two-dimensional fluorescence scatter diagram. Presently applied routine protocols rely on the determination of the stem cell concentrations either relative to the known concentration of leukocytes or relative to the concentration of fluorescent calibration particles that had been added to the sample. The disadvantage of such relative measurements is the additional systematic and statistic contributions to the measurement uncertainty. The leukocyte concentration is determined with another instrument, an automated hematology analyzer. When using calibration particles, their number in the stem cell sample varies for produc-



Fluorescence scatter diagram for the identification of the CD34-positive stem cells, of the white blood cells (leukocytes) and of the calibration particles that have been added to the sample.



Microscopic image of a blood smear of a patient with acute myeloid leukemia prior to stem cell transplantation. Apart from the biconcave red blood cells, three stained white blood cells are identified. The smaller white blood cell is a lymphocyte. The two larger cells are myeloid precursor cells of white blood cells, so-called "blasts", which are not found in the blood of healthy persons.

tion and/or pipetting reasons.

With the reference flow cytometric procedure developed at PTB, however, the sample volume is measured and the stem cell concentration directly determined from the number of stem cells. Compared with the two routine procedures used to date, the accuracy of the stem cell concentration measurement is increased by a factor of 6 (compared to the relative measurement with leukocytes) and by a factor of 2 (with respect to the relative measurement based on calibration particles).

Using such flow cytometers with integrated volume measurement for routine measurements of stem cell products would considerably simplify the analysis and, at the same time, improve its accuracy. To transfer this procedure to practical applications, systematic investigations on fresh and cryopreserved stem cell products are necessary. ■

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Scientific publication
J. Neukammer, M. Kammel, J. Höckner, A. Kummrow, A. Ruf: Referenzverfahren zur Messung von Stammzellkonzentrationen. BIOspektrum 21, 294 (2015)

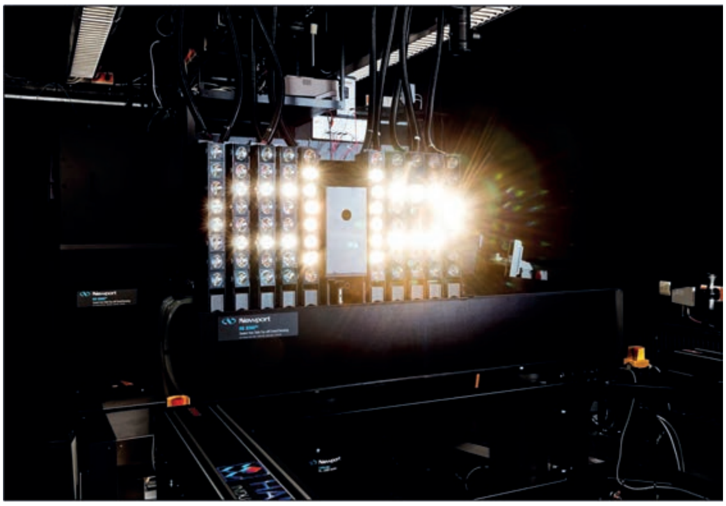
How much current does a solar cell provide?

PTB calibrates reference solar cells for real conditions by means of a novel measuring setup

Especially interesting for
- manufacturers of solar cells
- operators of solar power facilities

Conventional measurement procedures for determining the performance of solar cells make it difficult to obtain yield forecasts under real operating conditions. By means of a novel measuring setup which uses a laser-based spectral measurement procedure, PTB researchers have successfully characterized solar cells comprehensively enough that their yield can be calculated for any given climatic condition. This measuring setup also allows reference solar cells to be calibrated under the established standard test conditions (STCs) with a measurement uncertainty of less than 0.4 % – the only setup in the world to achieve an uncertainty this low.

Because of the boom in the renewable energy market, it will become increasingly important in the future to compare the performance of solar cells, as they are different in terms of their efficiency. A cell made by one manufacturer may achieve greater electrical power at a certain level of solar radiation (irradiance) than a comparable cell made by another manufacturer. Here, although electrical power can be measured with relative ease, it is considerably more difficult to determine irradiance. To this end, PTB calibrates reference solar cells whose short-circuit current is a measure of irradiance. Short-circuit current is the largest possible current which a module or a cell can produce. Calibration takes place under standard test conditions (STCs), i.e., under irradiation with a standardized solar spectrum, at 1000 watts per square meter and a solar cell temperature of 25 °C. However, in reality, the spectrum of sunlight varies depending on the time of day and on the season, as well as on the atmospheric composition; furthermore, the temperature, the angle of incidence and the irradiance are different depending on where the module/cell is installed.



In this PTB laboratory, solar cells are irradiated with white light as well as with monochromatic light (via the opening in the middle).

To be able to generate yield forecasts for these cells, which are used throughout the world, PTB has expanded its solar cell measuring setup. For comparability measurements, the so-called differential spectral responsivity (DSR) method is used; this method was recently further developed to become the laser DSR method. This method allows the yield for any given climatic condition to be calculated, such as for solar cell temperatures between 15 °C and 75 °C, and for irradiances from 0 W/m^2 to over 1100 W/m^2. In addition, the wavelength and the angle of incidence of the light can be varied. Ultimately, all of these measurements allow the performance of different solar cells to be compared.

For conventional (lamp-based) DSR procedures, white light is separated into individual wavelengths and temporally modulated by means of a monochromator and a chopper wheel, and then directed onto the solar cell via an optical element. In this way, the system can be set to all colors from ultraviolet to infrared light. At the same time, the cell is irradiated with white light, as this is the only way to achieve the 1000 W/m^2 required for the measurement. Yet the current generated by means of white light is up to a billion times greater than that generated by means of monochromatic light, often leading to a poor signal-to-noise ratio. By means of the laser-based DSR procedure, the noise problem – depending on the wavelength – has successfully been reduced by a factor of 100 to 10 000. In this way, the overall measurement uncertainty has been improved to the record value of less than 0.4 %. One additional advantage should be mentioned: Until now, only reference solar cells of a size of 20 mm × 20 mm could be calibrated. With the new facility, it has become possible to calibrate solar cells with sizes of up to 15 cm × 15 cm. The expanded portfolio of calibration services with the reduced measurement uncertainties mentioned above is passed on to testing and calibration laboratories, where these services make more precise traceability possible. ■

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Scientific publication
S. Winter, T. Fey, I. Kröger, D. Friedrich, K. Ladner, B. Ortel, S. Pendsa, F. Witt: Design, realization and uncertainty analysis of a laser-based primary calibration facility for solar cells at PTB. Measurement 51, 457–463 (2014)

PTB calibration service for airborne ultrasound

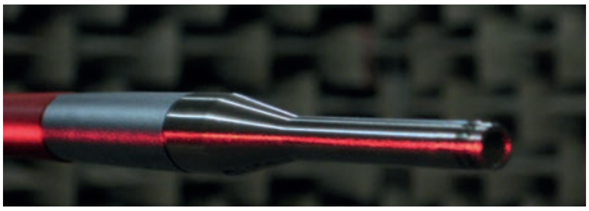
Traceability of sound pressure at frequencies over 20 kHz possible for the first time

Especially interesting for
- manufacturers of measuring and laboratory equipment
- users of ultrasound methods

At PTB, a procedure has been developed for the calibration of microphones in the ultrasound range of up to 100 kHz. By means of this procedure, reference standards and working standards can be produced for laboratories in industry and research in order to ensure the traceability of airborne ultrasound. This new calibration service is now available.

In many areas of our daily lives, the use of ultrasound technology is increasing. With it, airborne sound measurements in this frequency range are also becoming more important, such as those which are performed to check the limits of noise emissions. To this end, employers and occupational associations measure and evaluate the sound pressure level present at workplaces affected by noise pollution, such as ultrasonic welding facilities. In accordance with DIN EN 61010-1, manufacturers of measuring and laboratory equipment must furnish proof of the safety of their products by means of an airborne sound measurement up to 100 kHz.

To date, metrological traceability has not been possible for measurements of sound with frequencies of over 20 kHz for the unit of sound pressure, the pascal. The Danish Metrology Institute (DFM) recently introduced a primary standard which allows specialized measurement microphones to be calibrated as a primary transfer standard for frequencies of up to 150 kHz. As this method is limited to a certain microphone type, a procedure has been developed at PTB which is designed to enable the calibration of all types of microphones. The procedure is based on the conventional substitution procedure in accordance with DIN EN 61094-8, by means of which a controlled sound field is generated and successively measured by the primary standard and by the test piece. The substitution procedure was adapted to the specific characteristics of airborne ultrasound.



Reference microphone for airborne ultrasound (¼-inch measurement microphone mounted on an adapter to ½ inch) in the calibration setup at PTB's acoustic anechoic chamber.

As the wavelength of the sound propagated in the air amounts to only a few millimeters (3.4 mm at 100 kHz), very small areas and edges (which can be neglected in hearing sound) cause reflection and diffraction; this severely disturbs the sound field and, thus, the precision of the calibration. In addition, above approx. 10 kHz, the damping in air is no longer negligible and increases sharply in the ultrasound range; furthermore, it is heavily dependent on the ambient conditions. This places high demands on the sound source, and requires that measurement conditions be precisely controlled. Here, compared to calibrations for hearing sound, more effort and higher measurement uncertainties are involved; the uncertainties, depending on the frequency and type of the specimen, range from 0.4 dB to approx. 0.6 dB.

The new calibration procedure now allows ½-inch and ¼-inch (with adapter) measuring and working microphones to be traced to the Danish primary standard for frequencies of up to 100 kHz. They serve as secondary transfer standards in research and industry, as well as being used for measurements themselves. In the next step, the procedure will be further developed in such a way that any type of microphone, such as microphone/amplifier combinations, MEMS microphones, optical microphones or sound level meters, can be calibrated. ■

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Scientific publication
C. Kling: Microphone calibration service for airborne ultrasound. Proceedings INTER-NOISE Hamburg (2016)

Traceability for magnesium isotope ratios

PTB procedure contributed considerably to the development of reference material with a measurement uncertainty adapted to practical applications and traceable isotope ratios

Especially interesting for
- geochemistry
- isotopic analysis
- metrology

Within the scope of the Avogadro project for the redefinition of the kilogram and of the mole, PTB has developed mathematical and experimental methods to measure extremely accurate and traceable isotope ratios. These very procedures had a decisive influence on the development of three magnesium isotope reference materials which BAM – Federal Institute for Materials Research and Testing, Germany, will soon be marketing.

The new mathematical methods which were developed by PTB to determine the isotope ratios in silicon allow an analytical solution to be used for the first time for the evaluation of the measurements that are based on mass spectrometry – and thus ensure traceability to the SI. They have already contributed to considerably improving the measurement uncertainty within the scope of the Avogadro project. Now they are used to improve the measurement uncertainty also for the determination of the isotope ratios of magnesium by a factor of 10. Moreover, the magnesium isotope ratios will be comparable worldwide on an absolute scale (rather than one based on consensus) for the first time.

Geology, archeology, climate research, environmental protection, food safety and origin/authenticity tests are all areas in which isotope ratio measurements play an important role. For this purpose, an isotopic reference material (IRM) with certified isotope ratios is often necessary. For magnesium, with a natural variation of its molar mass of 1.2×10^{-3} g/mol, NIST SRM 980 has been the most widely used reference material so far; its measurement uncertainty, however, already exploits 30 % of this variation. Publications suggest that the material is so inhomogeneous that the measurement uncertainty covers nearly 50 % of the range; under such circumstances, sensible measurements seem impossible. Within the scope of EMRP project SIB09, three isotopic reference materials were developed under the leadership of BAM, together with LGC and based on PTB's methods. These exhibit a drastically reduced measurement uncertainty which, thus, lives up to practical applications for the first time. Even taking the homogeneity into account, the isotope ratio $n(^{26}\mathrm{Mg})/n(^{24}\mathrm{Mg})$ has an expanded measurement uncertainty of only 0.010 %.

This also prevents the progressive increase of the measurement uncertainties, which seemed inevitable when replacing one consensus material with another. The three magnesium IRMs developed will be available for purchase in the near future under the designations ERM®-AE143, -AE144 and -AE145 from BAM. ■

Three of the new magnesium isotopic reference materials (Photo: Dora Kolar-Bosnjak, BAM)

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Scientific publication
J. Vogl, B. Brandt, J. Noordmann, O. Rienitz, D. Malinovskiy: Characterization of a series of absolute isotope reference materials for magnesium: ab initio calibration of the mass spectrometers, and determination of isotopic compositions and relative atomic weights. Journal of Analytical Atomic Spectrometry, 31, 1440–1458 (2016)

Polishing procedures for ultraprecise silicon spheres

Especially interesting for
- manufacturers of precision spheres
- national metrology institutes

Ultraprecise spheres exhibit form errors and roughness in the nanometer range. Their crystal surfaces are free of metallic contaminations and show no crystal structure damage. These properties place extremely high requirements on their manufacturing process. Through the use of a PTB invention, it is possible to produce spheres with small form errors (on the order of 20 nm) in a reproducible manner by an ultraprecise manufacturing procedure. The invention is based on a pivoting polishing tool equipped with a membrane bearing for optimized control of the polishing operation. (Technology Offer 0404) ■

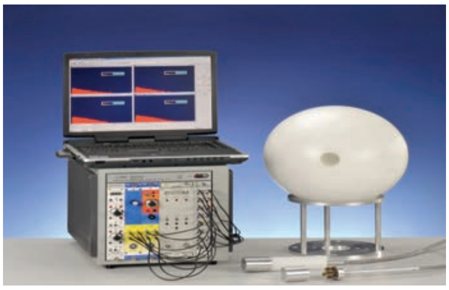
Production line of a 1 kg silicon sphere based on the example of the Avogadro project; representation of the different steps of the procedure: all the way from the ingot to the perfect sphere

Advantages
- small form errors on the order of 20 nm
- average roughness values of less than 1 nm

Neutron monitor for pulsed radiation fields

Especially interesting for
- medicine
- radiation protection

Pulsed radiation fields are used increasingly in novel medical accelerators and in fundamental research. Due to the high counting rate and detector saturation, radiation protection is confronted with new challenges. PTB's dosemeter is the first electronic radiation meter ensuring high metrological quality via high isotropy and low dead time for pulsed neutron fields. It consists of four silicon detector modules. Radiation discrimination is achieved by suitable selection of neutron-activatable or only poorly neutron-activatable substances. A US patent and an EP patent have been granted. (Technology Offer 0215) ■



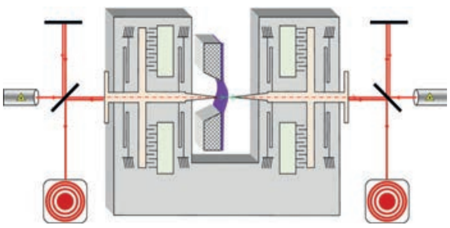
Semiconductor neutron monitor for pulsed radiation fields with a moderating sphere. The electronic unit can be easily miniaturized.

Advantages
- improved neutron dosimetry
- easily miniaturizable

Membrane thickness measurement

Especially interesting for
- biomedicine
- manufacturers of laboratory devices

Free-standing thin membranes are used in numerous scientific and industrial contexts. The thickness of these membranes is often highly relevant for their pertinent function. PTB's new measurement device allows a quick and very accurate determination of the membrane thickness. For this purpose, the membrane is contacted from both sides. By integrating two fiber interferometers into the microelectromechanical system (MEMS), low uncertainties can be attained. (Technology Offer 0361) ■



Set-up of the microelectromechanical system with two fiber interferometers

Advantages
- high precision thanks to micro-technology
- membranes made of transparent and soft materials can now be measured

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Awards

Harry Stolz,
the Managing Director of PTB's Conformity Assessment Body, was elected the new chairman of NoBoMet on 20 April 2016. NoBoMet (Notified Bodies in Metrology) is the European coordination group of notified bodies for the European Directives for Measuring Instruments (2014/32/EU) and for non-automatic weighing instruments (2014/31/EU).

Fabian Wolf,
a staff member of the QUEST Institute at PTB, received a Student Poster Award at EFTF 2016 for his work called "Quantum logic state detection for molecular ions".

Marc Bieler,
a staff member of Department 2.5 (Semiconductor Physics and Magnetism), received an Outstanding Paper Award from the journal *Measurement Science and Technology* for his article titled "Asynchronous electro-optic sampling of all-electronically generated ultrashort voltage pulses".

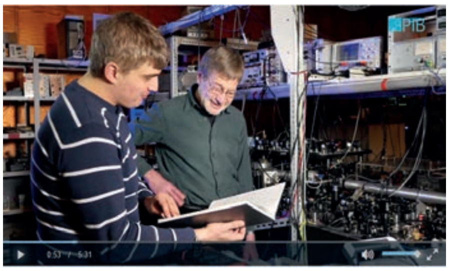
Ralf Behr, Oliver Kieler, Jinni Lee, Stephan Bauer, Luis Palafox and Johannes Kohlmann,
staff members of Department 2, had their joint work, titled "Direct comparison of a 1 V Josephson arbitrary waveform synthesizer and an ac quantum voltmeter", selected as one of the "Highlights of 2015" by the journal Metrologia.

"*maßstäbe*" Issue 13 published

The latest edition of *maßstäße*, PTB's popular-science magazine, which is available in German only, takes the reader on a journey through everyday life. No matter what we are doing or where we are, measurements are always there – and quite a few of them at that! Our reading journey begins by looking at our homes and our bodies, stops along the way to explore food & drink, and continues well beyond – down all kinds of transportation routes, into the air, into space, and into networks and grids of every kind.
"*maßstäße*" can be read online at: www.ptb.de > Press & What's New > Journals & magazines > maßstäbe
To order for free (including as a subscription or as a set for classroom use), please contact: Cornelia Land (+49 (0)531 592-9313, cornelia.land@ptb.de)

PTB News on film

Each edition of PTB News now includes a film, which will be shown online. These films will provide you with background information and a direct look at the work done in our laboratories. We began with "World record for two optical clocks" (2.2016). For this edition (3.2016), we have prepared a film for you on the topic of "How much current does a solar cell provide?" Each film can be found



in the same location as the accompanying edition of PTB News: www.ptb.de > Publications > PTB News.

Collaborative Research Centre: Development of quantum sensors

A great success for quantum metrology: On 1 July 2016, the *Deutsche Forschungsgemeinschaft* (DFG, German Research Foundation) established the new Collaborative Research Centre 1227, "Designed Quantum States of Matter (DQ-mat) – Generation, Manipulation and Detection for Metrological Applications and Tests of Fundamental Physics" at Leibniz Universität Hannover. The participating scientists will be working on exploiting the special properties of quantum-mechanical systems for better quantum sensors, such as optical clocks and accelerometers. In addition to Leibniz Universität Hannover (*Institut für Quantenoptik, Institut für Theoretische Physik and Institut für Didaktik der Mathematik und Physik*), other participating institutions are the Center of Applied Space Technology and Microgravity (ZARM) of the University of Bremen, and PTB in Braunschweig. The CRC is designed to last twelve years. Funding for the first four years will amount to slightly less than €10 million.

EMRP and EMPIR

For a few years, metrology institutes, researchers and companies have been supported by the EU to join forces in order to pool their resources and make available the measurement technology that a modern society needs. This is also reflected in the fact that a coordinated European metrology research program was launched with the support of the European Union – first within the scope of the 7th EU Framework Programme for Research and Technological Development, and thereafter as a measure under Article 185 of the Treaty on the Functioning of the European Union. At the moment, two programs are running: EMRP and EMPIR. The meetings taking place within the scope of the European Metrology Research Programme (EMRP) and of the European Metrology Programme for Innovation and Research (EMPIR) can be found on PTB's "European Metrology Research" website. http://www.ptb.de/cms/en/international-affairs/metrology-in-europe/european-metrology-research.html

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Bundesministerium für Wirtschaft und Energie

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.