PTB news 2/2010
The Science Newsletter of the Physikalisch-Technische Bundesanstalt


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Sound creates light

World first: PTB researchers transfer ultra-stable frequencies across a 480 km long optical fiber link

With a novel method of signal amplification PTB is able to transfer frequencies with highest precision to far-away users by employing optical fiber links to overcome distances of many hundred kilometers. This allows the best available optical clocks to be compared with one another, even over long distances.

Especially interesting for
- *optical clock development*
- *geodesy*
- *radio astronomy*


Photo: GasLINE

With fiber optics it is not only possible to transmit data, but also extremely accurate frequency information.

When light is not used to transmit data (as it does in optical telecommunications), but when rather a property of light itself – specifically: its frequency – is to be transferred, and that with the highest possible precision, then the classical techniques of fiber optic telecommunications reach their limits. The novel system of signal amplification utilizes stimulated Brillouin scattering (fiber Brillouin amplification). A so-called pump light with a precisely defined frequency is injected into the far end of the optical fiber, so that the pump light travels in the opposite direction to the signal light, generating sound waves (acoustic phonons) in the glass fiber. The sound waves, in turn, scatter the pump light, enabling the few already existing signal photons to stimulate the emission of many more signal photons. Thus, a photon avalanche is created which is kept going by the sound waves and brings the frequency information to the remote end of the optical fiber with extremely small losses.

If the amplification is sufficient, the light can be reflected from the remote end point and returns to the transmitter by travelling along exactly the same optical fiber path as on the way out. With this two-way information, the optical link can be stabilized. Thus, a fixed phase relationship is created between two very remote sites; with the stabilized fiber connection, frequencies are transported across long distances with extreme accuracy.

Using this technique, it was possible to characterize the optical magnesium clock at the Leibniz University of Hanover across a 73 km long optical fiber link, with the aid of a PTB frequency standard as well as of femtosecond frequency comb generators. The PTB scientists then planned a direct fiber connection from PTB to the Max Planck Institute of Quantum Optics (MPQ) in Garching – a fiber link distance of approx. 900 km, which attenuates the light by the almost inconceivable factor of 10^{20}, unless it is amplified. With the new system, even very weak signals are amplified; the signal power is increased by up to six orders of magnitude, so that only three amplifier stations are necessary instead of nine. Moreover, it is possible to selectively amplify very narrow-band light signals, which is advantageous for the testing of the narrow-band clock transitions of optical clocks. The method has already been tested on a deployed underground fiber link – in cooperation with the Deutsches Forschungsnetz (German National Research and Education Network) and the GasLINE company, which operate a German-wide fiber network. With only one intermediate amplification station, an ultra-stable frequency was transmitted across a 480 km long optical fiber link with a relative uncertainty of 2×10^{-18}.

Thus, the way is now open for a connection with MPQ in Garching in order to utilize the highly stable reference frequencies of PTB for joint experiments. Also, a connection with LNE-SYRTE, the French partner institute of PTB in Paris, now appears realistic, so that both institutes could work together on optical clocks in the future. Furthermore, applications in geodesy and also in radio astronomy are already on the horizon.

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Scientific publication
Terra, O.; Grosche, G.; Schnatz, H.: Brillouin amplification in phase coherent transfer of optical frequencies over 480 km fiber. Opt. Express 18, 16102-16111 (2010)

Please blow!

Alcohol breath analyzers can be calibrated by means of a new generator

A generator has been developed at PTB which produces water-saturated gas mixtures with a defined ethanol content and, for the first time, produces these gas mixtures in a dynamic-gravimetric way. Apart from enhancing the basis of the calibration of alcohol breath analyzers, it will improve the traceability of ethanol concentration to the SI units.

Especially interesting for
- *the police*
- *law courts*
- *equipment manufacturers*

Since 1998, evidential breath analyzers have been permitted for use in alcohol tests carried out by the police in road traffic. In Germany, they are an essential part of legal metrology and require a type approval from PTB before being used. For these tests, calibration gas mixtures are produced in PTB, which simulate the breath of a person who is under the influence of alcohol. The gas mixtures consist of air, water and ethanol in a precisely known composition and – to date – had been produced by the usual international saturation method. Here, an air stream is passed through an ethanol-water solution and enriched with ethanol and water until it is saturated. The concentration of ethanol in the gas stream is calculated via distribution coefficients which were determined empirically. In the literature, however, various values are to be found for them.

Weighing system to determine the mass flows of ethanol and water

With the new generator developed at PTB, the gas mixtures can be produced in a dynamic-gravimetric way. The core of the generator is a weighing system with which the mass flows of ethanol and water are determined by the quasi-continuous weighing of the storage containers. The air is dosed via thermal mass flow controllers. The liquid components ethanol and water are injected into the carrier gas flow made of synthetic air and vaporize there completely. As the mass flows of the components are determined individually before mixing, the composition of the gas mixture can be traced directly back to the SI base unit of mass, the kilogram. The use of empirical values from the literature is, thus, no long necessary.

The measurement uncertainty of the ethanol concentration in the gas mixture of the new generator was clearly reduced in comparison to the saturation method.

In the future, the generator can also be used to produce gas mixtures with other components e.g. acetone or carbon dioxide, to calibrate other types of sensors.

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Scientific publication
Pratzler, S.; Ulbig, P.; Scholl, S.: Einfluss der Temperatur- und Feuchtemessung auf die Herstellung von Atemalkoholkalibriergasen. Temperatur 2009: Symposium, PTB, June 2009. ISBN: 3-981002121-9-9 online advance copy: Chemie Ingenieur Technik, (02.07.2010). http://onlinelibrary.wiley.com/doi/10.1002/cite.201000093/abstract

Biomarker rapid tests improved

Primary method for the measurement of proteins in blood serum

A newly developed and now internationally recognised primary method of measurement enables PTB to offer the calibration of biomarker rapid tests as the first national metrology institute. The aim of this new method is to ensure the better reliability and comparability of clinical laboratory tests.

Especially interesting for
- *clinical chemistry laboratories*
- *doctors*

For the early diagnosis of life-threatening diseases, tests with so-called biomarkers play an increasingly large role. In this case, biomarkers are substances contained in the blood which, at certain concentrations, are typical of certain diseases. Rapid tests can detect such compounds relatively fast and with little effort. However, depending on the manufacturer and the laboratory, the results of such assays can differ considerably from one another. A comparison showed differences of more than one order of magnitude in the results of rapid tests. Such inaccuracies can lead to false diagnoses which are a psychological strain for the patient and can even be life-threatening; furthermore, they can lead to unnecessary costs for the health care system.

Therefore, together with doctors of the Medizinische Klinik – Innenstadt of the Ludwig-Maximilians-Universität in Munich, PTB has developed a novel method to measure proteins in blood serum. This method measures the human growth hormone – which also plays a role in doping. Its analytical reliability and accuracy are based on the application of isotope dilution mass spectrometry (IDMS). This principle has been applied for a long time for the determination of target values for the quality control of diagnostic markers such as cholesterol, glucose, creatinine or steroid hormones, all of which are rather "small" molecules. The recent development transfers the measurement principle (IDMS) from "small" organic molecules to "biological" macro-molecules such as proteins. Thereby, amino acid chains are broken into small fragments which are easier to handle for analytical purposes, but which are still long enough to be unmistakably recognisable as fragments of the precursor protein. Chemical analysis, thereby, uses techniques belonging to the field of proteome research, especially enzymatic proteolysis.


PTB staff performing mass-spectrometric analysis of chemically prepared protein samples

This method represents a step towards filling the gap existing due to the lack of DMS-based target values in quality assurance for routine measurements of diagnostic protein markers.

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Scientific publication
Arsene, C. G.; Henrion, A.; Diekmann, N.; Manolopoulou, J.; Bidlingmaier, M.: Quantification of growth hormone in serum by isotope dilution mass spectrometry, Analytical Biochemistry 401(2010) 228-235. Generally available as preprint: Nature Precedings (07.12.2009), http://hdl.handle.net/10101/npre.2009.4050.1

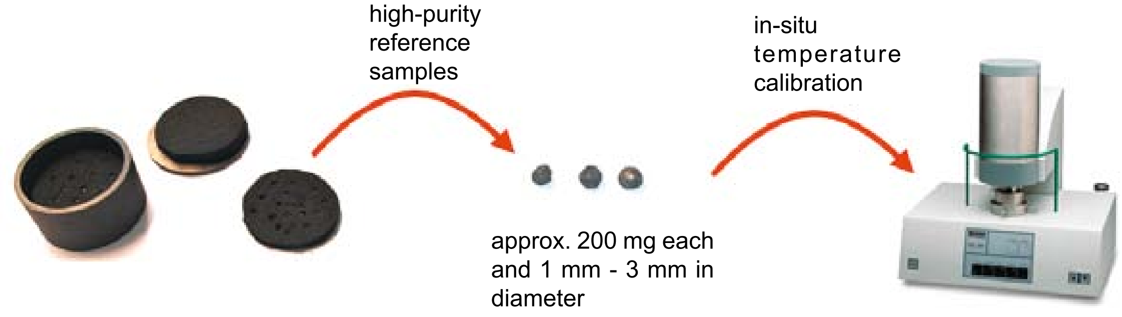
Hot fixed points

In-situ temperature calibration of dynamic scanning calorimeters above 1100 °C possible

To improve the energy efficiency of power plants, motors and thermal process engineering facilities, ever higher process temperatures are required which, however, make great demands on the materials used. For temperatures up to more than 2000 °C, the measuring methods used so far did not allow the thermo-physical properties of these materials to be determined with sufficient accuracy. In a cooperation with an industrial consortium, PTB scientists now succeeded in improving the uncertainty of the temperature measurement in devices for dynamic scanning calorimetry with the aid of high-temperature fixed points to values below 1 K.

Especially interesting for
- *material scientists*
- *production and process engineers*

Industrial and scientific applications at very high temperatures (e.g. in power plants and in aviation and aerospace for reentry and turbine technologies) require the optimization of materials with respect to energy efficiency and reliability. In such cases, the specific heat capacity is an important thermo-physical material property. In commercial instruments it is determined by measuring a differential temperature between two samples with a thermocouple. As the



stability of the thermocouple decreases at temperatures above 1000 °C, an in-situ calibration of the thermocouple is required.

Within the scope of a project funded by the BMWi, PTB is developing – in cooperation with Netzsch Gerätebau GmbH and with the support of Techno-Team Bildverarbeitung GmbH, HTM Reetz GmbH and ifa GmbH – novel calibration fixed points on the basis of eutectic metal-carbon alloys. In the project it has been possible to prove the suitability of these material combinations for fixed point standards in the temperature range from 1100 °C to 2300 °C. These fixed points now allow the in-situ calibration of the temperature of apparatuses for differential scanning calorimetry.

For temperatures up to above 1600 °C, the uncertainty of the temperature measurement in dynamic differential calorimetry has been improved to values below 1 K. Analogous to the ultrahigh-purity fixed points, these standards are being made available to the users as reference samples. A patent for the procedure has already been applied for and the method shall, in future, also be used in other devices for thermo-physical material investigations.

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Simply smooth

PTB is able to manufacture atomically smooth surfaces for testing the best microscopes

At PTB, atomically smooth silicon surfaces have been produced which are also suited to be used as reference standards for surface metrology. As they are, in addition, surrounded by mono-atomically high, but micrometre-wide steps, they can also be used for the precise calibration of the topography measuring axis of surface measuring devices.

Especially interesting for
- *developers of scanning probe microscopes and of other microscopes*
- *technicians in surface measurement technologies*

In the case of surface measuring devices, smooth surfaces can serve to measure deviations in the “image” – in the case of tactile measuring instruments they can, for example, measure the guiding errors of the movement axes and, in the case of interference microscopes, the quality of the reference surface. Corresponding surfaces can, for example, be manufactured for gauge blocks, optical flats or silicon wafers with different polishing methods. Over large areas and on the atomic scale, these surfaces are, however, rather rough.

In the case of crystalline material without dislocations, it would be ideal to produce faces as large as possible, consisting of one single lattice plane. If these atomically smooth areas are, however, surrounded, in addition, by mono-atomic steps with large terrace widths, a precise calibration of the topography measuring axis of surface measuring instruments for highest resolutions could be achieved in addition to this flatness. As a material, silicon with atomic lattice distances – i.e. step heights of 0.13 nm for Si(001) and 0.31 nm for Si(111) planes – would be well suited.

Although silicon can be manufactured without dislocations, many images which have been recorded with scanning probe microscopes show a great number of steps with terrace widths of less than 300 nm. This is due to the limited accuracy when the wafers are aligned – before cutting – with respect to their crystalline orientation (> 0.02°).

At PTB, surfaces with larger step-free areas could be produced by means of annealing silicon wafers in the ultra-high vacuum. These areas are surrounded by a series of mono-atomic steps with terrace widths of up to a few micrometres. Through a suitable treatment of the surface after annealing in vacuum, a – very thin and homogeneous – oxide layer grows on the silicon wafer which passivates these areas at the same time. Surfaces which have been produced in this way are stable over months (also in air) and can be used as reference standards for surface metrology.

These surfaces were used to determine guiding errors of the moving axes and effects of the optical detection systems for the determination of

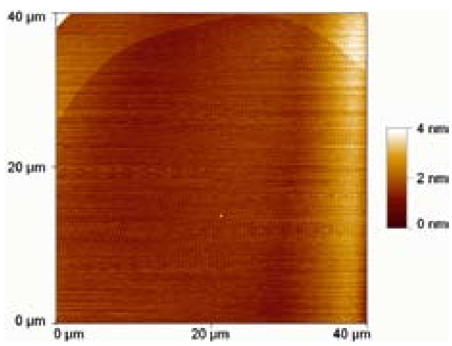

Image of a large, atomically smooth plane recorded with a scanning force microscope on a (111) silicon wafer. In the upper range, a mono-atomic step with a height of 0.31 nm can be seen. These measurements allow deviations in the measuring instrument (here: scanner) having the size of nanometer fractions to be clearly seen.

the cantilever deflection in the case of scanning force microscopes and, thus, to further optimize the scanning force microscopes. In the case of optical microscopes for topography determination, the resolving power of the topography measuring axis could be checked. In the case of an adequately exact calibration, the surface steps could, in future, also be used as a standard for the traceability of microscopes.

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Particle tracks in the cells

A new nanodosimeter enables better investigation of the effects of ionising radiation on the human body

At PTB, a nanodosimeter has been developed in collaboration with colleagues from Israel. By means of this dosimeter, measurands are to be developed which will reproduce the biological effects of ionising radiation on the cells better than the measurands used at present.

Especially interesting for
- *radiation protection*
- *medical scientists*
- *radiologists*

To determine – for radiation protection or medical radiation therapy – the effects of ionising radiation on the human body, measurands have been used up to now which are based on phenomenological processes. In nanodosimetry, the correlation with the detailed microscopic structure of the particle track – i.e. of the spatial distribution of the interaction events inside the body – is investigated. In the long run, the aim of this work is to develop new dosimetric measurands which already take the biological effects of ionising radiation into account.

To characterise the structure of the particle track, statistical measurands – such as the ionisation cluster size distribution – are used. This measurand indicates the probability that a certain number of ionisation processes is generated in a defined volume. In biological cells, e.g. in DNA segments with 10 to 20 base pairs, these are typically volumes with dimensions of a few nanometers. The effects of ionising radiation cannot be directly measured in such small volumes; they can only be determined by means of Monte Carlo simulation calculations. To link the – calculated – ionisation cluster size distributions in condensed matter (e.g. in biological cells) with the – measurable – ones in macroscopic volumes of measuring gases, a scaling procedure has been developed.

The nanodosimeter consists of a vacuum chamber which is filled with the corresponding measuring gas at a pressure in the order of 100 Pa. The cathode of a parallel plate capacitor located in the chamber has a bore through which the ions generated in the gas are extracted. A particle detector is used to detect the ions.

The nanodosimeter which has been developed in collaboration with the Weizmann Institute of Science in Israel is now used to experimentally verify the validity of this scaling procedure and to validate the Monte Carlo programs used for the simulations. For this purpose, the ionisation cluster size distributions are systematically investigated in different measuring gases for different radiation qualities. The density of the measuring gases is chosen in such a way that the gas mass in the detection volume corresponds to the mass of a DNA segment. Recently, these data have revealed inconsistencies in the material-specific datasets used in the programs, with the consequence that the corresponding cross sections need to be determined experimentally.

In the short run, it is planned to measure ionisation cluster size distributions for molecules which occur as constituents in the DNA and are therefore of great importance for the investigation of the relation between nanodosimetrical measurands and the biological activity of the radiation. In the medium term, the nanodosimeter is to be further developed for the spatially resolved detection of the primary particle in order to enable the 2D measurement of the parameters of the track structure.

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Scientific publication
Hilgers, G.: Check of the scaling procedure of track structures of ionizing radiation in nanometric volumes. Rad. Meas (2010). In print

Plastic syringes: Dosage exact to the microlitre

PTB provides precise reference values for the concentration of blood cells

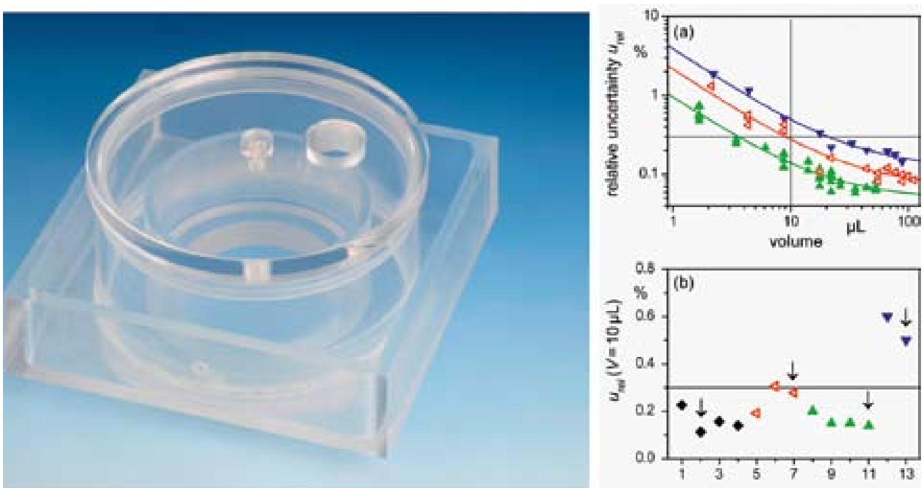
At PTB, a novel measuring set-up has been developed to calibrate syringes made of glass and plastic. Appropriate plastic syringes now provide more accurate reference values for interlaboratory tests with regard to an important analytical method of laboratory medicine – the determination of the complete blood count (CBC) or full blood count (FBC).

Especially interesting for
- *medical laboratories*
- *physicians*
- *manufacturers of dosage devices*

For external quality assurance, haematology laboratories have to take part in interlaboratory tests that are organised by medical professional associations. Thereby, PTB's task is to provide reference values for the complete blood count (CBC) or full blood count (FBC), i.e. to specify the concentration of the different blood cells present in a sample. Such values provide the physicians with valuable information about numerous diseases where, for example, the number of leucocytes is increased. Thereby, the different cell types – such as, e.g., red and white blood cells – are differentiated according to their size and structure and counted by means of a flow cytometer.

To measure the concentration of the different cell types in the sample, it is essential to precisely determine the volume of the blood sample. The fact that cells remain stuck on the walls of glass syringes and are, thus, lost for the measurement can, in the case of red blood cells, lead to a loss of up to 5 %. With plastic syringes made of polypropylene, cell loss is considerably lower; they have, however, not been used so far due to their unknown accuracy for volume determination in the microlitre range. The experiments performed by PTB have shown that, in the case of a volume of 10 microlitres, the syringes of selected manufacturers comply with the uncertainty of < 0.3 % specified in DIN standards on cell counting. They are thus suitable for the determination of cell concentrations as long as the plastic plunger is replaced with a plunger made of aluminium.

Essential features of the new measuring set-up are an integrated, newly developed evaporation trap and the application of an appropriate measurement cycle with regard to the correction for the (remaining) evaporation. It is, thus, possible to achieve measurement uncertainties of approx. 0.1 % at a volume of 10 µL. In future, the sensitivity of the arrangement will be increased to be able to determine also volumes in the range below 1 µL with low measurement uncertainties.



Evaporation trap. Right: (a) Relative uncertainty as a function of the volume for three different combinations of driven syringes. (b) Comparison of the measurement uncertainties for different combinations of driven syringes for a volume of 10 µL. The black symbols represent the results for glass syringes, the coloured symbols correspond to plastic syringes of different manufacturers.

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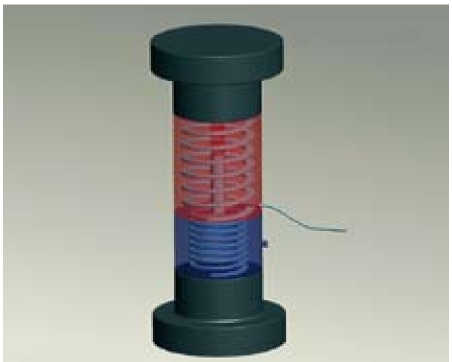
Scientific publication
Reitz, S.; Kummrow, A.; Kammel, M.; Neukammer, J.: Determination of microlitre volumes with high accuracy for flow cytometric blood cell counting. Measurement Science and Technology, Vol. 21, 074006 (9pp), 2010

Galileo: ground control gets its vital components

PTB has supplied an ensemble of caesium clocks and measuring instruments to the ground control centre of the future European satellite navigation system Galileo. The ensemble of commercial atomic clocks – which were thoroughly and comprehensively tested by PTB before being delivered – is part of the Galileo Precise Timing Facility (PTF) in Oberpfaffenhofen which was developed by the Kayser-Threde company; it is supplemented by two hydrogen masers and facilities for time comparisons. The PTF provides the exact time of the Galileo system; this time is the reference time for all systems linked within Galileo – both on ground and in orbit – and with which they are synchronised, for example for the orbit determination of the satellites or to check the time signals sent by the satellites. In principle, the PTF ensures that the Galileo components all use a time which is painstakingly the same – up to many places after the decimal point. The PTF is thus the basis for the utilisation of Galileo satellite signals for position determination.

Measuring force and displacement dynamically and with high precision

Licence partners wanted



Especially interesting for
- *mechanical engineering*
- *material testing*

Whether in the field of vibration technology or in mechanical engineering: The measurement of forces and torques is the basis for numerous applications in industry and research, e.g. material testing. So far, however, the dynamic calibration of test equipment, e.g. material testing machines, has been a problem. The measurands "force" and "displacement" are often calibrated only statically – individually and sequentially. This is not only time-consuming, but can also lead to a great measurement uncertainty in a dynamic test. The dynamic force-displacement sensor – a transfer standard developed by PTB – can measure force and displacement jointly and dynamically and can thus be used for an application-oriented in-situ calibration. The results are traceable and more precise than ever before.

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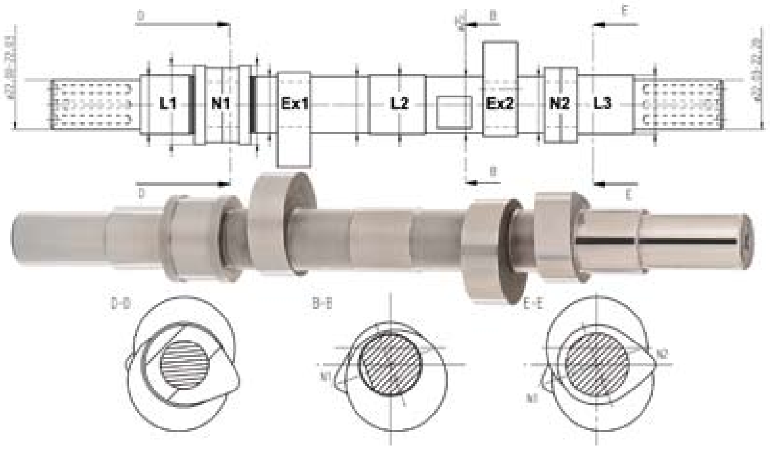
Patent No.
DE 10 2009 022 769.5 ; 2009-05-25

Novel cam standard

Further cooperation partners wanted

Especially interesting for
- *automobile industry*
- *manufacturers of coordinate measuring machines*

The dimensions of a cam shaft must be exactly complied with as already a deviation of a few hundredths of a millimetre puts its quality into question and the pollutant emission, the fuel consumption and the wear increase. But how can a reliable proof of the dimensional accuracy of a cam shaft be furnished? For this purpose, a novel cam standard has been developed at PTB in cooperation with a partner from industry. On the basis of this standard, cam measurements can be traced back with due regard to practice. In cooperation with other industrial partners, the procedures shall, in future, be adapted to the needs of calibration service providers.



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Fast and robust trace gas analysis

Licence partner wanted

Especially interesting for
- *spectroscopy*
- *industrial gas monitoring*
- *climate research*

Due to a simpler design, the production of Herriott cells could be more economical and the time-consuming adjustment could be omitted. In conventional Herriott cells the laser light is bounced back and forth between two concave mirrors. So far, the laser has had to enter and leave through a small hole in the cell, which posed some difficulties for the operator. The new component can do without the "troublesome" hole. Application and manufacture are correspondingly robust. The device is also suitable for Cavity Ring Down Spectroscopy (CRDS), in which various gas species are detected simultaneously.

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Awards

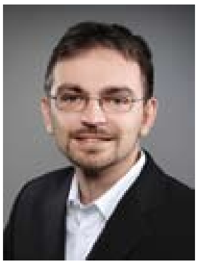
Piet O. Schmidt

The Head of the QUEST Institute at PTB received the Young Scientist Award at this year's EFTF Conference (European Frequency and Time Forum) in Noordwijk, the Netherlands, for his contribution to developing one of the currently most precise atomic clocks in the world.

Osama Terra

This PhD student in PTB Department 4.3 "Quantum Optics and Unit of Length" received the EFTF Student Award in the category "Timekeeping, Time and Frequency Transfer, GNSS and Applications" for his work "Optical frequency dissemination over a German wide-area network using Brillouin amplifiers".

Matthias Müller



This PhD student in PTB Department 7.1 "X-ray Metrology with Synchrotron Radiation" was given the Young Spectrometrist PhD Award at this year's European Conference on X-ray Spectrometry (EXRS) in Figueira da Foz (Portugal). The European X-ray Spectrometry Association (EXSA) thus honoured Müller's PhD thesis, which is on novel methodologies for quantitative wavelength-dispersive X-ray fluorescence analysis in the soft X-ray range at the Berlin electron storage ring Bessy II.

Dates

24.11.2010 Plenary Assembly for Verification Matters
Location: PTB Braunschweig. Morning: Public part, Lecture Hall in the Kohlrausch Building. Afternoon: Working session, Vieweg Building, Room 134

23.11.2010 VMPA Meeting, Expert Committee on Noise Protection
Committee meeting. Location: PTB Braunschweig, Ohm Building, Seminar Room 221. Contacts: W. Scholl; M. Witwer. Phone: +49 (0)531 592-1700, +49 (0)531 592-1701. Organizer: PTB Department 1.7

22–26.11.2010 Photometry Seminar
Location: PTB Braunschweig, Einstein Building. Contact: K. Stock. Phone: +49 (0)531 592-4100. Organizer: PTB Department 4.1

3.12.2010 Desertec – Challenges for Metrology and Technical Cooperation
258th PTB Seminar. Location: PTB Braunschweig, Lecture Hall in the Kohlrausch Building. Contacts: C. Heider, K. Stock, M. Kahmann. Phone: +49 (0)531 592-2301. Organizers: Q.52, 4.1, 2.3

6.–7.12.2010 Concluding conference "Gentoxische Effekte von THz-Strahlung in vitro?"
Location: PTB Braunschweig, Lecture Hall in the Kohlrausch Building. Contact: Chr. Jastrow. Phone: +49(0)531 592-2215. Organizer: PTB Working Group 2.21

7.–8.12.2010 Radiation Protection in Medicine, Research and Industry
Seminar of TÜV SÜD Akademie GmbH. Location: Freiburg. Contact: TÜV SÜD Akademie GmbH, Tagungen und Kongresse, Westendstraße 160, 80339 München. Phone: +49(0)89 5791-1122. Organizer: TÜV SÜD

16.12.2010 Deadline of Abstract submission: SPIE Conference on Optical Metrology
Date of the conference: 22.-26.5.2011. Location: International Congress Centre, Munich. Contact: B. Bodermann. Phone: +49(0)531 592-4222. Organizer: SPIE Europe.

10.1.2011 New Deadline of Abstract Submission: 10th IMEKO Symposium Laser Metrology for Precision Measurement and Inspection in Industry (LMPMI) 2011
Date of the Symposium: 12.–14.09.2011. Location: PTB Braunschweig. Organizer: VDI/ PTB/TU Braunschweig. Contact: H. Bosse. Phone: +49(0)531 592-5010

22.2.2011 "Advances in Coordinate Measurement Techniques for Industrial Applications"
NIMTech International Workshop. Location: vTI Braunschweig. Contact: F. Härtig. Phone: +49(0)531 592-5300. Organizer: PTB Department 5.3

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Focus: Terahertz Metrology (in German)

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Imprint

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