

Accurate Determination of Atomic Mass Differences by means of Prompt (n, γ) Spectrometry

Spectrometry of γ -rays emitted promptly after neutron capture by atomic nuclei in a sample allows a most precise determination of mass differences as well as concentrations of isotopes contained in the sample. Twenty different isotopic mass differences have been determined recently by a PTB team at the Institut Laue Langevin (ILL) in Grenoble with relative uncertainties of less than $3 \cdot 10^{-10}$. These measurements present a new route for the reduction of uncertainties in the determination of Avogadro's constant in a high-purity, perfect single crystal of natural silicon which might be used in future to replace the kilogram artifact.

Thermal neutrons irradiating a specimen will be captured by the nuclei of the atoms contained in the sample. The energetic rearrangement of the nucleons in the nucleus is accompanied by the prompt emission of γ -rays with energies which are characteristic for any species of nuclides. PTB is currently establishing this «prompt (n, γ) spectrometry» as a method to measure isotopic mass differences with highest accuracy and to determine the isotopic composition and the content of impurities in particular specimens in a quantitative and non-destructive way.

PTB has recently set up a measurement station at the high-flux reactor of the ILL in Grenoble where a well collimated beam of thermal neutrons extracted from the reactor via a neutron guide tube hits the target nuclei. The γ -rays emitted are detected

on-line and simultaneously by large Germanium detectors. From the measured γ -spectra, the isotopes are identified by their specific γ -energies while the abundances are determined by the peak areas taking into account transition probabilities, detection efficiencies, solid angles and the thermal neutron capture cross sections.



PTB employs spheres like this (mass 1 kg, diameter about 90 mm, deviations from an ideal sphere less than 50 nm) for the determination of Avogadro's constant (number of particles making up the amount of substance of 1 mole or, specifically, number of carbon atoms in 12 g of ^{12}C).

About 20 different isotopic mass differences of interest for silicon single crystals have been measured with relative uncertainties of less than $3 \cdot 10^{-10}$. The next step will be the determination of the atomic abundances of the stable Si isotopes and the impurities in the single crystals in order to determine the molar mass with highest possible accuracy.

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Traceability of Chemical Measurements to SI Units

The growing importance of certification and quality systems results in an increasing demand to ensure that measurements in chemistry are traceable to the international system of units (SI). This is particularly true for the fields of clinical chemistry and environmental analyses, for measurements which are to control the keeping of legal limits, and for long-term observations of small changes in chemical composition. In order to create reliable reference points which allow chemical measurements to be traced back to SI units just as well as measurements in general, PTB is engaged in research, development and testing of primary methods.

Accurate chemical measurements and their traceability to SI units call for new and different provisions as compared to traditional fields of metrology. The differences result, for instance, from the vast number and variety of chemical materials and from the fact that a particular chemical compound usually has to be determined as a single component in a complex environment of other substances. At present, tests of measuring instruments and methods used for chemical routine analysis are most often done with the help of reference materials, but only a few of these are traceable to SI units as yet. Therefore, PTB considers it as a main task to

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Thermoacoustic Measurement of Ultrasonic Power

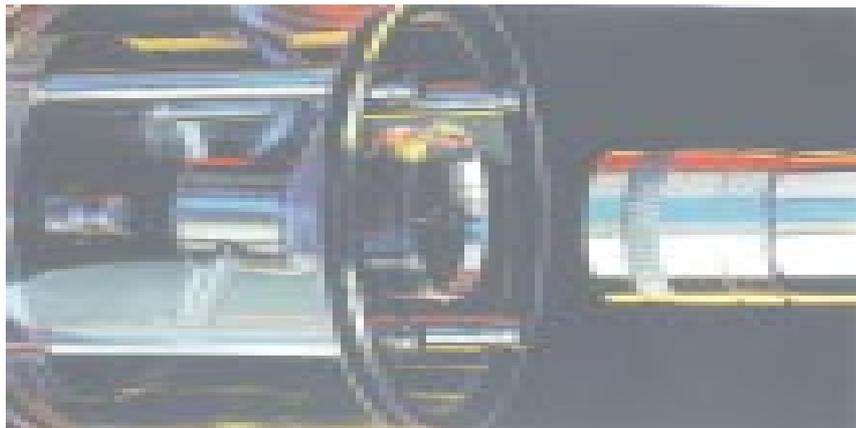
PTB has developed an ultrasonic power sensor based on the thermoacoustic principle. Due to its simplicity, the sensor is particularly well suited for routine inspections of ultrasonic equipment used in the fields of medicine, materials testing and chemical engineering.

Conventional methods of ultrasonic power measurement such as reciprocity calibration, time-delay spectrometry or the acoustic radiation force method are time consuming, complicated and require a great deal of equipment. These drawbacks are avoided with the thermoacoustic sensor. Its most essential component is a plexiglass cylinder of 1 cm length as ultrasound-absorbing element, which transforms the ultrasonic energy into heat. The temperature increase produced by the ultrasonic wave in the state of thermal equilibrium is measured by thermistors or thermocouples at the rear end of the plexiglass absorber. The absolute value of the incident ultrasonic power is then determined from the temperature increase, the thermal conductivity and the acoustic absorption coefficient of the absorber material.

The thermoacoustic sensor can measure ultrasonic powers above 1 mW in the frequency range of 1 MHz to about 20 MHz. As an example, the temperature increase produced by an ultrasonic power of 100 mW and a frequency of 5 MHz is about 3 K.

The thermoacoustic measuring system has small dimensions and requires only minimal instrumentation. Because of its simple and robust construction it is very stable and nearly maintenance-free. Systems of this type have already been put in service for calibration purposes and routine inspections of ultrasonic equipment. Main fields of application of such equipment are medicine, materials testing and chemical engineering.

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Thermoacoustic sensor (left) with transmitting transducer (right) in a water basin. Only the front surface of the plexiglass absorber (length 1 cm, diameter 3 cm) is in contact with the water.

Improved Characterization of the Hardness of Materials

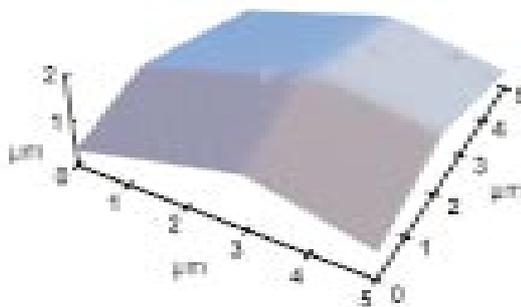
The universal hardness measuring method employs continuous synchronous measurements of test force and depth of indentation. This allows to obtain more information and higher accuracy than the conventional techniques according to Rockwell, Vickers and Brinell, in particular for microhardness.

The conventional methods of hardness measurement in the macrohardness range (test force > 2 N) are of utmost importance for quality assurance in the field of mechanical properties of materials. However, the traditional methods yield only a «single-point» hardness value reflecting the plastic behaviour of the material. The new universal hardness measuring method features measurements with increasing and decreasing test force and thus takes into account the elastic behaviour of the material in addition to the plastic behaviour.

The universal hardness measuring method proves especially useful for metals, glass, ceramics, and thin layers. The high accuracy of the method has been confirmed by intercomparison measurements with hardness testers calibrated with glass as reference material. As a basic requirement for the widespread application of universal hardness measure-

ments, reliable calibration procedures have been developed by the hardness laboratory of PTB, founded in 1996. The adoption of the new method will also be supported by standards such as DIN 50 359.

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In hardness measurements, only a volume of approximately $15 \mu\text{m} \times 15 \mu\text{m} \times 3 \mu\text{m}$ of the indenter tip takes part in the indentation process. Therefore, the geometry of the diamond indenter is a major source of uncertainty and has to be determined with high accuracy. PTB uses a calibrated scanning force microscope with three laser interferometers in its x-, y-, and z-axes to accomplish this measurement task with a measurement uncertainty of $U \approx 10 \text{ nm}$. The figure shows a 3D graph of the tip area of a Vickers indenter measured with this microscope.

More Reliable Vehicle Speed Measurements



A new PTB reference system for vehicle speed measurement ensures improved certainty and reliability of the devices used for speed limit enforcement on our roads. It thereby contributes to

a better acceptance of speed measurement results at court.

Type testing and approval of devices used for traffic speed control has to guarantee that the prescribed error limits are reliably met. An essential part of the approval procedure consists of tests in public traffic, where the speed values are compared with those of a reference system. The system used previously as a reference system is based on three piezocables as sensors to measure the average speed over a rather long distance. This system provides usable results for only about two thirds of all vehicles because it requires sufficiently constant speed.

The new reference system is based on two cross-road light barriers of known distance and works much more effectively. By measuring the time a vehicle travels between the two barriers, the speed value is determined. As a novel feature, defective scanning – which may occur if different parts of the outline of the vehicle are scanned by the two light barriers – is not only detected but also corrected. For this purpose the vehicle is monitored by a video camera as it passes each light barrier. Its position at start and stop of the time measurement is

determined with high precision by means of digital image analysis.

In summary, the advantages of the new reference system are:

- Measurements with defective scanning have no longer to be rejected during type testing and approval of speed enforcement devices.
- The tests can be performed even in critical traffic situations.
- The measurement uncertainty is reduced to 0,5 % of the measured speed.

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*Use of the reference system
for measurements of vehicle
speed in traffic conditions*



Accurate Measurements of Electrical AC Power

A novel method of accurate generation and measurement of power for alternating currents allows to calibrate measuring instruments for active, reactive and apparent power with greatly improved relative uncertainties.



With the daily sales of electrical energy in Germany amounting to a value of 250 million DM, continuous reduction of the uncertainty and increasing ease of calibration of the measuring instruments are not only important in terms

of metrology but for economical reasons as well. Therefore, PTB has developed a novel procedure for accurate calibration of instruments for the measurement of active, reactive, and apparent power with any power factor between zero and unity.

The method uses a twin source to generate the voltage U_1 and the current I_1 to be applied to the instrument to be calibrated. The current I_1 is con-

verted into another voltage U_2 by means of a current transformer and a current shunt. Both voltages U_1 and U_2 are alternately measured with a sampling voltmeter. A computer program based on discrete Fourier transform algorithms evaluates the complex ratio of the voltages U_1 and U_2 and determines the DC component, the harmonic distortion, and the relative stability of the voltages in addition. The traceability to the SI units Volt (DC voltage) and Ohm (DC resistance) is ensured by a calibrated rms voltmeter and an AC shunt. The low measurement uncertainty is due to the fact that both generation and measurement of the electrical quantities are locked to the master clock of the sampling voltmeter.

The method was demonstrated using a prototype system for 120 V and 5 A (600 VA) at 62,5 Hz. The relative measurement uncertainties for all power factors are about five parts in 10^6 . This represents an improvement by a factor of three to ten over hitherto established methods.

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Traceability of Chemical Measurements to SI Units

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provide standard measuring procedures and devices which can be used as reference points in chemical analysis.

Medical or clinical chemistry is an important but particularly difficult sector of chemical analysis. In response to the requirements of clinical laboratories to trace measurement results to the SI unit of the amount of substance, the mole, PTB has started a programme for high-accuracy determination of organic chemicals using a modified version of

$^{12}\text{C}/^{13}\text{C}$ isotope dilution mass spectrometry (IDMS). This is a primary method which allows to determine most of the organic compounds important as diagnostic constituents of human blood with improved accuracy. IDMS is also used for the determination of the diagnostically important elements. For sodium, a special ion-chromatographic method is being developed at PTB.

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Transportable Calcium Frequency Standard

PTB has set up an optical frequency standard stabilized to a narrow transition in an atomic calcium beam. The standard allows to disseminate the 456 THz radiation frequency produced by a diode laser with a relative uncertainty which is an order of magnitude smaller than that of the iodine stabilized He-Ne laser, the most accurate and most widely applied standard in length metrology at present.

Lasers with frequencies stabilized to transition frequencies of suitable atoms, ions, or molecules now can represent optical frequency standards of unprecedentedly low uncertainties. They do not only offer a potential for the development of optical atomic clocks, but they can also be used for highly accurate measurements of fundamental constants, for tests of basic theories in physics, or as wavelength standards for the realization of the metre. However, the widespread utilization of these frequencies is hampered by the fact that the most accurate optical standards are only available at a small number of institutes.

In order to facilitate the dissemination of highly accurate frequencies PTB has set up a transportable wavelength/frequency standard based on a diode laser system the frequency of which is stabilized to a narrow transition in ^{40}Ca atoms in an effusive beam. This frequency has been recommended recently by the International Committee of Weights and Measures (CIPM) as the optical frequency in the visible spectral range with the lowest uncertainty suitable for the realization of the metre. The standard allows the dissemination of 456 THz ($\lambda = 657 \text{ nm}$) radiation with a relative uncertainty of $1,2 \cdot 10^{-12}$. This is an order of magnitude smaller than the uncertainty of the iodine stabilized He-Ne laser at $\lambda = 633 \text{ nm}$, the most widely applied laser in length metrology at present.

The transportable standard has been compared with the Ca standard of the National Institute of Standards and Technology (NIST, USA) in Boulder, and good agreement of the two frequencies has been found within the combined uncertainty. A new project has recently been started to utilize PTB's transportable optical Ca standard for comparison of optical clocks and frequency measurement chains as well. As a next step, the effects caused by the high velocity of the Ca atoms, for instance, the relativistic time dilation, will be reduced significantly by employing laser cooled atoms.

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Transportable optical frequency standard comprising of a diode laser system frequency-stabilized to the intercombination transition in a Ca atomic beam

Events

(Further information can be found in the Internet/WWW via <http://www.ptb.de/>; choose "English", then "News")

The Role of Metrology in Economic and Social Development

Seminar organized by PTB together with the BIPM, the OIML, and the IMEKO

Braunschweig, 16 to 18 June 1998

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Hermann von Helmholtz Symposium 1998

The annual symposium is organized in remembrance of PTB's first president and is devoted to a topic of basic research which is of current interest and closely related to metrology. This year's symposium will be held in PTB on 26 June 1998 and deal with «Natural and Artificial Lattice Structures».

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Kryoelektronische Bauelemente Kryo '98

Braunschweig, 11 to 13 October 1998

Further information by fax: (+49 531) 592-24 05 or email: kryo98@ptb.de

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