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Titelbild

Windenergieanlagen gelten als Grundpfeiler der Energieversorgung aus regenerativen Energien. Rückgeführte Kalibrierungen und wissenschaftliche Forschung in der PTB tragen sowohl zur technischen als auch zur wirtschaftlichen Effizienz solcher Anlagen bei. (Foto: Chris Keller / bobsairport.com)
The PTB-Mitteilungen are the metrological specialist journal and official information bulletin of the Physikalisch-Technische Bundesanstalt, Braunschweig and Berlin. As a specialist journal, the PTB-Mitteilungen publishes scientific articles on metrological subjects from PTB’s fields of activity. As an official information bulletin, the journal stands in a long tradition which goes back to the beginnings of the Physikalisch-Technische Reichsanstalt (founded in 1887).
The Contribution of Metrology to the Sustainable Generation, Distribution and Utilization of Energy

Klaus-Dieter Sommer*

1 Introduction

The greatest scientific and technical challenges mankind is facing today and will also face in the coming decades are – in view of the fact that the fossil energy sources which can be exploited with reasonable effort are limited and that the carbon dioxide emissions into the atmosphere are taking on increasingly critical proportions – to ensure a secure, need-oriented and sustainable supply with energy, an intelligent distribution of energy, as well as its efficient use. In particular, there are four points which make the urgency of sustainable solutions clear:

a) According to the information provided by the Norwegian Centre for International Climate and Environmental Research, the carbon dioxide emission due to fossil fuels rose, in 2010, to 9 billion tons – a so far unprecedented number. Compared to the preceding year, this corresponds to an increase by almost 6%. In consideration of this fact, the so-called “Kyoto objective” (i.e. the limitation of global warming to a maximum of two kelvins) is probably no longer a realistic target and many regions of the world will already have to prepare themselves for extreme weather conditions and significant climatic changes in the coming decades. For the time being, a definitive end of the greenhouse gas increase is not in sight: As long ago as the Kyoto conference in 1997 (the “COP3 Conference of Parties”), important industrialized countries and countries in transition were not prepared to make commitments, and this “Kyoto protocol” – which is, of course, politically and strategically extremely important (although it is, in reality, only efficient to a limited extent) – will probably expire in 2012.

b) At the very latest since the Fukushima disaster on 11 March 2011 – when the Tōhoku earthquake and the subsequent tsunami caused core meltdowns in three reactor blocks, associated with a considerable contamination of the air, the soil and of water and food – the necessary confidence in the safety of nuclear power plant technology, which was developed several decades ago, has been lacking, in particular in the densely populated industrialized countries such as Germany. Better nuclear power plant technologies are, at present, not available and new developments are not being made – or are made only hesitantly.

c) The number of explored deposits of fossil fuels – in particular of natural gas and mineral oil – which can be exploited with a justifiable technical effort and at economically acceptable costs, has – according to the knowledge gained so far – decreased in the past decades. The exploitation of “new”, so-called unconventional fossil energy carriers – which comprise, for example, oil sands, oil shales, polar oil and gas condensates – as well as the exploitation of fossil fuels from very deep deposits by means of deep drilling engineering with force fitting of “frac fluids” (hydraulic fracturing) are, in most cases, very time- and labour-consuming and/or are associated with high risks for the environment and for the population.

d) And ultimately, we are – due to the rapid industrial advancement of the countries in transition, in particular China, Brazil, India and Russia – confronted at the global scale with increased energy consumption and, thus, with international competition for those fossil energy reserves that are difficult to exploit efficiently.

2 New approaches and general strategies

The consequences for a country with extremely little natural gas and mineral oil deposits such as Germany, which – due to the collective worry about the safety of the nuclear power plants in densely populated regions – wants to do completely without this energy source until 2022, are relatively clear and without actual alternatives:

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a) The available fossil energy sources and the renewable primary energy sources must be utilized much more efficiently. This not only applies to the efficiency of the power plants, but also to the distribution and transfer of energy. Furthermore, energy consumption in industry, in private households and in transport must become more efficient. Here, the keyword is "energy saving". Savings might be achieved by both intelligent consumption strategies – e.g. the use of current-intensive working steps at staggered intervals (supported by "smart metering"), in connection with incentives in terms of prices – and also by new, energy-saving construction and processing technologies (keywords: "Green Production", "Sustainable Living").

b) Our primary energy mix must be developed significantly towards renewable energies, in particular with respect to wind energy (which can be developed in a relatively short time), with respect to solar energy (which, in Germany, is possible to a limited degree only), and with respect to an increase in the share of biogenic fuels. To prevent cut-throat competition between "tank and dish", biogenic energy carriers must not have their sources in the same original area as food production. Consequently, only vegetable waste and such bio-waste which cannot be utilized for other purposes come into question as raw materials for energy carriers. The fuels produced in this way are referred to as "belonging to the second generation".

c) Another – quite essential – point is that the energy must be available at any time at the place where it is needed. We therefore need a significant increase in the availability of the energy generated in Germany itself and all over Europe – in particular of renewable energy – at the place of demand and as independent as possible of solar irradiation and of wind force – and this, preferably, exactly at the point in time when the need for it arises. This requires an efficient network with large transmission capacities and low losses, as well as intelligent control of the energy flows and their distribution (Smart Grid) and – in view of the dominating role of electric energy – the development and establishment of highly efficient stores for electric energy which can be used rapidly. The network is the backbone of the entire distribution system. Without an efficient network, all other activities remain fragmented.

d) In view of the fact that, on the one hand, the share of transport in energy consumption lies at almost 30 percent and the environmental burden originating from this is enormous, but that, on the other hand, the economic importance of automobile construction is of great importance for Germany as an industrial base, we need a new mobility concept which has the following focal points:

- Significant promotion of electro-mobility (including energy-efficient motors), of mobile energy storage (batteries) and of the charging infrastructure (part of the Smart Electrical Grids, Smart Metering) in individual automobile transport;
- Energy-efficient and environmentally compatible combustion engines with increasing use of renewable bio-fuels in the transport of passengers and goods on the road.

All four points mentioned so far require new technological solutions, new materials and energy carriers, new and intelligent supply concepts and networks as well as – what is all-important – broad, collective confidence in both the safety of the new concepts, technologies and substances and demonstrable fairness with respect to the costs in trade with energy and for required investments and equipment. This is the point where metrology comes in with its contribution.

According to these requirements, reliable information is needed about:

- Safety-relevant process parameters which determine the process efficiency and the material properties in the generation, transmission, storage and use of energy;
- Important variables of state which are required for the operation, capacity utilization and mathematical reconstruction of the energy supply network, including their development with time and their spatial distribution;
- Relevant characteristics of fluid energy carriers, in particular energy content, mechanical transport characteristics, material compatibility and – via indicators – proof of origin;
- Energy measurement data relevant to invoicing, including their time sequence.

Special importance is attributed to the metrologically correct determination of efficiencies, in particular, in the fields of energy generation (e.g. with respect to the efficiency of a wind park or of a specific solar cell type), the use of energy (e.g. with respect to the efficiency of motors or heating systems in buildings) and, of course, also with respect to energy storage (e.g. capacity and condition of batteries).

The provision of the above-mentioned reliable information by metrologically traceable measurements is not only important for engineering and for field surveying, but it also provides an indispensable basis for international energy research, in particular for the comparability of R&D results.
3 Metrological challenges

This section gives a survey of the concrete metrological challenges to be met by energy metrology and by the metrology for energy research, technology development and the future sustainable energy supply. As to the structure, it orientates itself on the so-called energy supply chain, consisting of the following components:

(a) Energy generation (i.e. conversion of the energy which is primarily available into a usable form, e.g. conversion of the chemical energy of natural gas into electric energy or into thermal energy)

(b) Energy transport and distribution (including any necessary conversions, e.g. to other pressure- or voltage levels)

(c) Energy storage comprises the required conversion, e.g. from electric into chemical (batteries) or mechanical (pump storage stations) energy.

(d) Energy consumption and saving (i.e. conversion of a high-grade energy form such as electric energy or gas into effective and dissipative energy, e.g. electric energy into mechanical operating energy and heat)

Figure 1 shows a graphic representation of this energy chain. In this representation, also the part Electric mobility has been subsumed with the transaction energy and the mobile energy storage (batteries).

3.1 Energy generation

Research and development in the field of energy generation has three clear main development directions:

- The development of new – as efficient as possible – technologies for the further (and economical) exploitation of renewable energies, in particular of wind energy, solar energy and bio-energy for fuels,
- A clear increase in the efficiency of conventional (large) power plants which will – for a long time still – provide the basis of our energy supply, as well as
- A significant reduction in the emission of greenhouse gases into the atmosphere and the reduction of the production of all kinds of waste in the large-scale production of energy. Here, it is of decisive importance that none of the measures towards a more sustainable energy supply will impair the supply security – not even temporarily!

Conventional (large-scale) power plants: An essential measure having a significant impact on the efficiency of the complete energy supply is the improvement of the efficiency of large conventional – and also nuclear – power plants by shifting the focal points of work towards higher temperatures and pressures. The metrological tasks
and challenges resulting from this are: the exact determination of the thermo-physical material properties of the more stable materials to be used, including the emission coefficients which are important for the achievable measurement uncertainty of radiation temperature measurements as well as a significant reduction in the measurement uncertainty. (See also the following sections).

Renewable energies (wind, sun): The primary objective is to realize correct measurements – i.e. comparable measurements which are traceable to national standards – and this not only in view of the efficiencies and the introduced primary energy, but also in view of the – in most cases less complicated – transferred energy. In the field of photovoltaics, this requires – due to the different special spectral distributions of the technology applied – the definition of an international (spectral) evaluation scale in which – in addition to PTB – the Japanese AIST, the American NREL (and in future also NIST) as well as the Chinese TIPS are involved. In the field of wind energy, the challenge is to determine the influencing flow profile (e.g. up to a height of 200 m) with a low measurement uncertainty in a completely metrological way to allow reliable statements about the efficiency of single plants and large wind parks to be made.

Liquid fuels: Conventional liquid fuels are usually refinery products of mineral oil. The risks for the environment, which cannot be denied and which became, for example, apparent in 2010 with the disastrous blow-out on the “Deep-Water Horizon” platform in the Gulf of Mexico, as well as the limited new resources which can be exploited economically to a limited degree only, lead to an increase in the use of bio-fuel mixtures with an increasing share of bio-fuel. Due to a possible competition with food production, this is not without problems. Metrology is, therefore, challenged to determine energy contents and transport properties of these gasoline blends not only reliably and traceably, but to develop also methods to prove the origin. The purpose of this is to detect cases where – for reasons of cost – a fully grown rain forest which is under protection is, for example, sacrificed for reasons of cost – a fully grown rain forest which is under protection is, for example, sacrificed for reasons of cost – a fully grown rain forest which is under protection is, for example, sacrificed for.

Bio-gases: Bio-gases are mainly produced from bio-waste mass. For this reason, they do not usually compete with food production. Bio-gas is usually generated decentrally in small plants and also fed into the grid decentrally. This causes some problems in the fair, energy-content-orientated distribution, supply and invoicing. The following parameters are to be measured very exactly and traceably to national standards:

- Loss of methane in the production phase

\textit{Note: This could have a strong negative influence on the climate footprint of bio-gas production.}

- Efficiency of the biogas plant or of the current production process. \textit{Note: This is important for both the efficiency prediction in the planning phase and for the trade with such plants and subsequent promotion and investment decisions.}

- Energy content and composition (quality) of the bio-gas fed into the network.

3.2 Energy transport and distribution

Electric energy: In the field of maximum voltage for the bridging of very large distances – e.g. from the wind energy fields on the German North Sea Coast to the industrial processing centres in the south of Germany – completely new transmission technologies – in particular the High-Voltage Direct Current transmission (HVDC) – are being tested, and the associated measuring and control techniques – which are at present still in their infancy – are being developed. This comprises in particular power measurements – which can be traced back to national standards – at the transmission/converter stations.

In addition, the increasing combination of large conventional and nuclear power plants with the decentrally fed-in electric energy from wind and electro-voltaic power stations requires an intelligently controlled network \textit{(SmartGrid)} based on measurement information which must be based on exact and comparable input information and reliable measurement values. As large electric motors – e.g. for airport ventilation systems, for the driving of roller plants or in smelters – are increasingly controlled with thyristors or special high-performance field effect transistors, the question of the so-called power quality (i.e. the share of uneven-numbered harmonics in the network) is increasingly gaining – together with its metrological characterization and its influence on the quality of other relevant measurements – in importance.

Transmission and distribution of energy gases: The so-called “energy gases” such as, for example, natural gas or natural gas-biogas mixtures are usually traded in relation to the delivered energy quantity. Due to the constantly changing gas qualities from quite different sources (North Sea, Russia, other countries, North German Plain, bio-gases, gasified LNG qualities...) in connection with the gas distribution network which is usually “under-instrumented” (i.e. only insufficiently equipped with devices), the gas quality delivered to the individual consumers must be reconstructed mathematically as exactly as possible or estimated within the scope of a legally prescribed accuracy band. The prerequisites for this are the knowledge of the distribution network, its flow characteristics, the gas qualities fed in and the available measurement data. Already today, highly industrialized national economies with distribution networks.
as closely meshed as in Germany can no longer completely control their gas distribution without such reconstruction systems. At present, a research project – financed by the DVGW – is being performed at PTB to further qualify these methods.

3.3 Energy consumption and saving

Private sector: In the private sector, the technological development towards higher efficiency in energy consumption is characterized by new energy-efficient building and insulation concepts, special domestic developments, e.g. for older generations, as well as – very importantly – by the introduction of newer, fairer and more transparent and comprehensible invoicing and payment concepts for the effectively consumed energy quantity within the scope of Smart Metering. From metrology it is expected that it will not only furnish exact in-situ measurement results and the guarantee of their safety and reliability. Metrology must also furnish the relevant quantitative material properties, e.g. for the thermal insulating behaviour – and it must develop the most suitable and cost-efficient traceability schemes.

Industrial sector: The essential tasks consist in making the efficiency, including the raw materials and semi-finished products, energetically ascertainable and measurable (energy balance, CO₂ balance: so-called “green/sustainable production”). Corresponding models, approaches and strategies must be developed, connected to the traceability hierarchies and finally be made available to industry.

For both sectors – the private and the industrial sector – new saving technologies must be developed. In addition to building restoration/-insulation, the subject of “lighting” takes priority on the agenda: The development of new, significantly energy-saving lighting technologies – such as, for example, organic LED technologies – and their maturity for large-scale production open up enormous saving potentials. Their spectral characteristic and their subjectively felt illuminating power deviate, however, significantly from traditional illuminants. This requires new, physiological approaches for their metrological assessment.

3.4 Energy storage and mobility

Energy storage on a large scale: In particular countries and national economies with a very high share of renewable energies are confronted with the necessity of storing electric energy on a large scale, e.g. with the aid of pump storage power stations. Alternative solutions for countries or regions which do not have the respective geographic prerequisites at their disposal are, at present, not known. Nevertheless, an efficient and easily accessible energy storage, associated with the exact measurement of the energy stored or withdrawn is indispensable for the future.

Batteries and fuel cells: The most important reason for the currently booming activities in the field of battery and fuel cell research – particularly in the field of the lithium-ion batteries – is their intended use in the field of electric mobility in private transport. The efficiency aimed at has not yet been achieved, not all safety questions have been clarified and there is still essential fundamental research – also with respect to fundamental material properties, thermodynamic relations and models for the assessment of the charging and life condition of the batteries – to be done. In addition, safety questions represent an important open point.

Liquid fuels for e-mobility: For long-distance trips, also electric vehicles in the private transport sector of the future will need so-called range extenders which are based on conventional fuels. For purely conventionally driven vehicles, requirements relating to the correct measurement of the exhaust gases, their components and the (soot) particles emitted will have to be met.

Energy supply infrastructure for electric mobility: The supply network/the supply networks for future e-mobility solutions will surely be part of the regional or national “Smart Grid”. For this reason, the metrological challenges will also be similar or identical (see 3.2). In addition, there are requirements for the extremely fast charging of car batteries, charging during driving, “flying battery change”, etc. This always comprises reliable onboard energy measurements and a fair invoicing of the transaction energy consumed, connected with distance reading and automated charging.

4 Current metrological projects related with the subject “energy”

Current metrological research at PTB related to the subject “energy” is being undertaken [7] within the scope of the European Metrology Research Programme (EMRP) [2]. This programme comprises the following research projects (in square brackets: the organizational unit of PTB):

- Characterization of Energy Gases [3,5]
- Metrology for Energy Harvesting [3,5]
- Metrology for Liquified Natural Gas (LNG) [3,5]
- Metrology for Smart Electrical Grids [3,5]
- Metrology for Solid-State Lighting [3,5]
- Metrology for Improved Power-Plant Efficiency [3,5]
- Metrology for High-Voltage Direct-Current Energy Transmission [3,5]
- Metrology for New Generation of Nuclear Power Plants [3,5]
- Metrology for Bio Fuels [3,5]
Emerging Requirements for Measuring Pollutants from Automotive Exhaust Emissions [4,5]. In addition, PTB performs relevant metrological research in the following fields:

- Metrology for Wind Power Stations [5]
- Model-Based Reconstruction of the Energy Content in Under-Instrumented Gas-distribution Grids (METROGAS-Projekt) [5,6]
- International Photovoltaic Scale (AIST, NREL, PTB, TIPS) [5,7].

5 Literature

Metrology for Improving the Efficiency of Conventional Power Plants

Thomas Lederer*, Steffen Rudtsch, Klaus Anhalt, Karsten Tawackolian, Stephan Krenek

The backbone of European energy supply is the generation of electric and thermal energy in conventional power plants. Metrological research aimed at improving the efficiency of power plants – so that the CO₂ emissions are reduced and natural resources are protected – is therefore a central task of PTB. To this effect, PTB is excellently interlinked in the European research landscape and is participating – as project leader, and in cooperation with nine other national metrology institutes – in a research project within the scope of the European Metrology Research Programme.

In the focus of this project are temperature and volume flow measurement techniques, as these are – as controlled variables for the operation of conventional power plants – the decisive process parameters.

In the field of temperature measurement, industrial platinum resistance thermometers are being investigated. In particular at temperatures above 600 °C, an exact determination of the temperature is difficult with an industrial resistance thermometer. A higher operating temperature promises, as a matter of principle, more efficient energy generation. Often, however, it entails higher material stress. The use of nickel-based alloys promises an increase in the working temperature to up to 1300 °C. However, precise contact-free temperature measurement often fails due to insufficient knowledge of thermo-physical material parameters – such as, e.g., the emissivity in the high-temperature range. For this reason, new measurement procedures for the determination of the spectral emissivity above 1000 °C will be developed within the scope of this research project.

The volume flow in the feed water circuit of a power plant is an elementary process quantity for controlled operation. Worldwide, however, no testing facility exists where the sensors can be calibrated for the extreme operating conditions of 300 °C and 180 bar at high volume flows. Within the scope of this project, new strategies will be developed to make this possible for the first time.

As the result of these research activities, an increase in efficiency of 2 % to 3 % in total is expected for conventional power plants. Coal-fired power plants, for example, contribute with approx. 34 % to the CO₂ emissions. The efficiency increase of these power plants could reduce the worldwide emission of CO₂ by approx. 600 to 900 million tons per year.

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Metrology for Photovoltaics – Measurement of Solar Energy
Stefan Winter*

At present, the investments in the photovoltaics (PV) industry are exponentially growing and amount – worldwide – to approx. 35 billion Euros per year (2010). The predictability of the economic yield of solar modules depends decisively on the realistic assessment of the efficiency. Even small measurement uncertainties lead to very large economic and ecologic uncertainties and barriers.

The measurements of the German photovoltaics industry are based on measurements carried out by PTB, which is the only European reference laboratory and also achieves the smallest measurement uncertainties worldwide in the primary calibration of reference solar cells. Reference solar cells are selected, specially tailored solar cells with an active area of typically 2 x 2 cm². These reference cells, which are calibrated at PTB with high precision, represent the beginning of a calibration chain which reaches up to the solar modules that are to be mounted on the roof. The procedure developed by PTB for the calibration of reference solar cells – which is internationally recognized – characterizes these cells with respect to their spectral behaviour, their temperature dependence, their non-linearity and their angular dependence.

The boom in the field of photovoltaics is also reflected by the demand for high-precision primary solar cell calibrations, which has increased tenfold in the past few years. For this reason, a completely newly designed measuring arrangement is being set up which is based on a spectrally tuneable laser source instead of incandescent lamps and xenon lamps. The monochromatic output power which, in the case of the laser-based set-up, is by up to a 1000 times higher, allows the solar cell to be irradiated more homogeneously, with a finer spectral resolution and a better signal-to-noise ratio. Due to this, it is not only possible to calibrate a greater variety of solar cell types, but to calibrate them also with a clearly improved accuracy – complying thus with the demand from industry. This project is supported with funds of the European Regional Development Fund.

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Sketch of the new measuring arrangement (partly in false colours). On the right, the beam paths of the different lasers are to be seen. Via an optical fibre (blue), the laser pulses are smoothed in time, made even more narrow-banded by means of a monochromator (green) and imaged – via an output optics (red) – homogeneously on the solar cell (red). To ensure that the solar cell is in the same operating state as in the case of solar irradiation, it is additionally irradiated with a solar simulator (black).
Metrology for Wind Turbines

Harald Müller*, Helmut Többen

In the wind energy sector, the exact measurement of the wind speed is of central importance – not only for determining wind resources to enable increased and more cost-effective wind farm assets, but also for power performance measurements of electricity-producing wind turbines. The traceability of wind velocity measurements to the SI units is a task of PTB and is realized by non-interacting laser-optical procedures, using wind tunnels for the realization and dissemination of the unit “flow velocity”.

By 2020, the share of the regenerative energies in the electricity supply is to be increased gradually to 30 percent. Here, wind energy will play a key role. In addition to the new offshore wind parks, i.e. in the North Sea and in the Baltic Sea, the development of suitable sites onshore and the modernization of wind parks by “repowering” (i.e. by replacing older wind turbines by fewer – but more efficient – new wind turbines) are constantly gaining in importance. In order to assess the efficiency of a wind park, wind resource analyses are carried out in advance which are based on long-term measurements of the wind velocity distribution in the planned wind park area. According to IEC standard 61400-12-1, for both wind speed and power performance measurements, only cup anemometers are accepted which have been calibrated by accredited calibration laboratories. As the electric energy generated by a wind energy turbine depends on the wind speed to the power of three, the exact measurement of the wind speed plays a key role for the projected energy yields.

One task of PTB’s “Gas Flow” Department is to ensure the traceability of wind speed measurements and – herewith associated – the comparability of calibration results.

For this purpose, laser Doppler anemometer systems are used as reference or transfer standards which in cooperation with the German WindGuard Wind Tunnel Services GmbH company – a leading operator and owner of large wind tunnel facilities for calibration and research tasks in the wind energy sector – have already allowed the measurement uncertainty for the calibration of cup anemometers used in the wind energy sector to be halved.

Calibration of a cup anemometer at the German WindGuard Wind Tunnel in Varel.

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Energy from the Environment – Traceable Metrology for Microgenerators

Torsten Funck*, Jürgen Melcher

In our environment, energy sources exist in the form of waste heat, vibrations and movement which are largely unused and are “fed” by human activities or natural energy flows. To tap these energy sources is the objective of the newly developed microgenerators which are to provide the operating energy, for example, for portable electronic devices or sensor systems at inaccessible places. This will allow the need for chemical batteries and – thus – the costs and the disposal problems associated with these to be drastically reduced.

The manufacturers of such microgenerators need reliable measuring techniques to compare different constructions with respect to their power and efficiency, whereas the potential users need reliable data of the different generators in order to select the generator which is suited best for their individual application. However, the data so far available have not always been comparable because traceability to national standards is lacking for many of the measurements required for this purpose. For this reason, an international project “Metrology for Energy Harvesting” – which is developing traceable metrology techniques for this purpose – was launched in September 2010 within the scope of the EMRP and under the leadership of PTB. The participants are the national metrology institutes of seven states: Germany (PTB), the Czech Republic (CMI), Italy (INRIM), France (LNE), Finland (MIKES), the United Kingdom (NPL) and Slovenia (SIQ).

Depending on the source of the environmental energy, the output signals of the observed microgenerators are in many cases very small, not sinusoidal or even not stationary. As this considerably complicates traceable measurements, the main objective of this project is to develop the required measurement procedures and a corresponding calibration infrastructure to support industry in the investigation and development of microgenerators. Here, the focal point lies on thermoelectric and vibration generators with small output powers, whereby the form of the excitation is to come as close as possible to the conditions prevailing in nature.

After a project duration of three years, a metrological infrastructure will be available in Europe which will allow both microgenerators and their materials to be characterized and the measuring instruments required for this to be calibrated.

A reduction of the battery waste would not only reduce costs, but also the environmental burden. Other than shown in the picture, the project is aimed, in particular, at reducing the use of button cells.
Plasma Diagnosis for Nuclear Fusion

Helmut Schuhmacher*, Andreas Zimbal

From nuclear fusion, i.e. from the melting of light atomic nuclei, an inexhaustible energy source is expected. With the International Thermonuclear Experimental Reactor (ITER) in France – one of the largest research projects worldwide – an important step from today's research facilities towards plants for future energy generation shall be taken.

Neutrons are important products in fusion reactions. Measurements of the neutron flux and its energy distribution provide knowledge of the development of the fusion processes in plasma. Compared to other fusion plants (e.g. the Joint European Torus (JET) in England, or the fusion experiment Wendelstein 7-X in Greifswald) in which PTB is already involved, an increase in the neutron production by several orders of magnitude is expected from ITER. This leads to new challenges for metrology, but also to a heavy strain on the material by radiation. An example of an already successful development is the compact neutron spectrometer (approx. 5 kg) with a high energy resolution, which is to replace the spectrometer used so far for plasma diagnosis (approx. 60 tons).

In cooperation with working groups from the field of plasma research, PTB is involved in projects for the development of suitable measurement techniques. Hereby, PTB’s excellent facilities for the generation of neutron radiation play an important role. The properties of the radiation fields can be selected in such a way that they largely correspond to those occurring during nuclear fusion – except for the extreme neutron fluxes to be expected at ITER. This allows measurement procedures to be tested at PTB under controlled conditions and new measuring instruments to be calibrated in a way which comes close to reality.

The most important cooperation partners are: IPP Garching and Greifswald, ENEA Frascati (IT) and JET Culham (UK). The projects are partly funded by the European Commission.

ITER will be loaded with measuring instruments for monitoring the different physical parameters of the fusion plasma.


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The addition of agro-technically generated so-called bio-fuels to fossil fuels not only requires modifications of motors and injection systems, but – for purposes under verification law, fiscal law and quota law – also a new measurement of the transport properties and of the energy content of the new mixed fuels.

In trade, liquid fuels are measured in volume units. Tax burdens and specifications of the quota law are also referred to a volume unit. To create a comparison quantity which is independent of the measurement temperature, all data related to the volume are converted to a reference temperature of 15 °C.

Therefore, the density-temperature behaviour must be known for all fuels. At present, the bio-fuel quota is defined via the energy content of the components. From 2015 on, the CO₂ saving potential will be used.

For this reason, PTB has set up measuring facilities for the precise measurement of the density and its temperature dependence and of the energy content of fuels. The density can be measured in a temperature range from −25 °C to +50 °C with an uncertainty of 0.02 kg/m³ (approx. 0.0025 %), using an electronic density measuring device which has been modified especially for this purpose. The combustion calorimeter installed for the determination of the energy content of fuels allows measurements to be carried out with an uncertainty of 0.2 %.

To guarantee European harmonization in this field, PTB is cooperating with 12 partners in a European metrological multi-sectoral project. The project is aimed, in particular, at harmonizing measurement procedures for the chemical and physical characterization of bio-fuels. In addition, international harmonization is advanced by a trilateral cooperation of PTB and the national metrology institutes from France and Brazil with the primary objective of harmonizing measurement procedures and carrying out comparison measurements.

With these measurement campaigns, PTB is continuing a tradition that goes back more than a hundred years. Whereas in the beginnings of the Physikalisch-Technische Reichsanstalt, the services for verification and tax authorities and for standardization organizations were to the fore when the technically more demanding fuels were introduced, today the environmental aspect plays the most important role.
Metrology of Non-conventional Fuel Gases

Stefan M. Sarge*

Due to the relative environmental friendliness of fuel gases rich in hydrogen, it is planned to increase their share in the future energy mix. For this purpose, the raw material base will have to be broadened. This will lead to a shifting of the quantitative and qualitative properties of natural gas, which is predominantly used at the moment. PTB is already making manifold efforts to ensure that the energy amount delivered with the fuel gas is measured and billed correctly.

To conserve the present deposits of natural gas, other fuel gas sources are increasingly being developed. Some of the alternatives are: pit gas and shale gas, methane hydrate, gas from fermentative processes (biogas, landfill gas), synthetic gas from the thermo-chemical decomposition of biomass, hydrogen through electrolysis from excess wind or solar power – possibly with subsequent methanation.

In addition to the solving of toxic problems and problems related to hygiene or to the material, the metrological infrastructure used for the quantitative and qualitative determination of the gas composition of today’s natural gas distribution grid has to be adapted to the new situation. This natural gas distribution grid consists of a few feed-in stations with high power which are embedded in a network which supplies the consumer with gas. In future, a large number of sources with low power, too, are going to feed their gas into this network. The current measurement infrastructure, which is used – on the one hand – to control and regulate the gas flows and, on the other hand, to determine and invoice the energy amount is, however, designed for a clear flow direction and for using natural gas as fuel gas.

PTB is meeting the challenges resulting from this new situation in cooperation with universities, medium-sized companies, gas suppliers, verification authorities and European metrology institutes by manifold activities, for example by

- adjusting the legal regulations for the correct measuring of the volume and the energy;
- investigating the bases for the chemical and physical characterization of the non-conventional fuel gases;
- developing modern mathematical and statistical procedures to determine the measurement uncertainty in the computational determination of gas properties in under-instrumented distribution grids;
- using its competence for the development of gas chromatographs for the quantification of carbon dioxide, hydrogen sulphide and water.

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At the Sleipner gas field in the North Sea, carbon dioxide is sequestered in a rock formation a kilometre beneath the sea floor. Credit: Dag Myrestrand / StatoilHydro
Liquefied Natural Gas (LNG) – Natural Gas Supply without a Pipeline

Horst Bettin¹, Jürgen Rauch, Markus Richter*, Reiner Kleinrahm*, Roland Span*

Today, Germany is provided with natural gas by just a few suppliers, via a widely ramified pipeline system. If participating more intensively in the worldwide trade with natural gas is desired, structures will have to be established which enable the feeding-in of natural gas which is delivered in a liquefied state (i.e. LNG).

To feed LNG into the gas distribution system, the natural gas must be liquefied at the producer’s side by cooling it down to approx. −161 °C. Then it must be transported in the liquefied state with tankers and discharged at the recipient’s side. Possibly, it must be stored temporarily, and evaporated again. Under the aspect of fair and fiscally correct trade, the transfer of the natural gas between the contracting parties is of special importance – and this delivery is usually made in the liquid state.

Whereas for pipeline natural gas, procedures have been developed which allow the energy contents to be invoiced very accurately, there is – with respect to LNG – still a need to catch up. The aim of the research project “Metrology for Liquefied Natural Gas”, which is funded by EURAMET within the scope of the EMRP programme, is to reduce the measurement uncertainty in the invoicing of LNG by half. In addition to volume flow measurement, the measurement and the calculation of LNG densities is of critical importance here. The density measuring device for LNG, which is under construction at the Ruhr University Bochum, is envisaged to be, with an uncertainty of 0.02 % ($k = 2$), more exact by a factor of approximately 10 than the data on which today’s invoicing procedures are based. Traceability to the SI units is guaranteed by PTB. In addition to this, PTB is participating within the scope of the EMRP project in the calculation of the energy content of LNG.

The central part of the density measuring device is a magnetically suspended sinker. By means of this sinker, the density can be measured according to the Archimedean principle. On the part of PTB, the volume of the sinker is determined with the utmost accuracy and is traceable to the SI units. The design of the thermostat and of the measuring cell allows boiling mixtures to be measured at cryogenic temperatures, without additional uncertainties arising with respect to the liquid density which is caused by concentration changes during the phase change.

Cooperation partners: VSL (coordinator), TUV NEL, PTB, LADG, FORCE, SP, JV, CMI, INRIM, Elengy, E.ON Ruhrgas AG, ENAGAS, Ruhr University Bochum.

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3D representation of the measuring cell and of the first thermostat stage of the new density measuring device. To be seen are the sinker (turquoise) and the magnetic suspension coupling (red) which connects the sinker – through the wall of the measuring cell – with the high-accuracy weighing machine.
In the history of public energy supply (which goes back more than 100 years), grid structures have been developed which are optimized for generating, transmitting and distributing energy in a centrally organized network. In view of the innovations that have been achieved in the past decades in the field of decentral energy resources and information and communications technology (ICT) it makes sense, however, to develop this network further in such a way that also a decentralized energy supply, which is integrated in the overall supply system by means of ICT, becomes economical.

In Germany, electricity from regenerative energy sources has already reached a share of approx. 13% of gross electricity consumption – and this share is still rising. In industrialized countries such as Germany, a reliable 24-hour supply based only on regenerative energy sources is, however, not possible without difficulty. To integrate the electricity which is produced in a regenerative way as efficiently as possible into the total electricity supply network, an information and control network has to be set up which coordinates – with regard to time and quantity – the energy flows coming from conventional generation, from regenerative generation, from the storage utilizations and from the consumption at the end user’s. The realizations of such concepts are today usually called “smart grids”. In such networks, the information which is decisive for the stability of the system is supplied by correct energy measurement values. “Correct” hereby does not only mean the “correct measurement” of electric quantities – such as, e.g., flows, voltages, frequencies and reactive and active energy – but it also – and in particular – means an unfalsified transmission of the measurement values from the meter in the supply network to the coordination points and to the monitoring authorities of the information and control network. Due to this duality, PTB experts for measurement accuracy – together with PTB experts for measurement data security – are engaging (within the scope of the national innovation initiative “E-Energy”) in the relevant standardization committees of DIN and VDE and – at the international level – in the committees set up within the scope of standardization mandates of the European Commission, e.g. the Smart Grid Coordination Group of CEN, and in CENELEC and ETSI. Due to its involvement in standardization activities and due to the experience gathered by tackling scientific problems of its own, PTB is qualified as an independent metrological consultant and as a mediator between the interests of the grid industry, on the one hand, and the interests of the State in the field of energy management, on the other hand.

The figure shows the technological approach of the Smart Grids: the energy flows and the measurement data flows are coordinated in a network in such a way that although the need for electricity produced in a non-regenerative way is reduced to a minimum, a continuous supply with energy is ensured.
A prerequisite for trading with electricity is to have suitable measurement techniques available. Experience has shown, however, that metrological infrastructures making the respective measurement techniques available – and keeping them available also permanently – are usually not developed by the markets themselves. The aim of PTB’s department “Electrical Energy Measuring Techniques” is therefore to create the metrological prerequisites which are necessary to measure electricity correctly.

Based on the different pillars of the “Law concerning the electrical units of measurement” (“Gesetz betreffend die Elektrischen Maßeinheiten” – GEM) of 1898, the Verification Act defines the tasks of the field of “Electrical Energy Measuring Techniques” assigned to the state – still today – as follows:

The first pillar is the realization of the electric unit kW. This realization is effected by means of a primary power standard which works in accordance with the sampling principle. With the aid of this standard, the unit kW can be realized with an uncertainty of $5 \times 10^{-6}$.

The second pillar is the dissemination of the unit kW. This dissemination is realized by the calibration of commercially available, high-precision power measuring devices. With these devices, measurement uncertainties of $5 \cdot 10^{-5}$ can be achieved. The users of this service are mainly the 109 test centres which have been approved by the State for testing electricity measuring instruments in Germany. The exact standard power measuring devices are used by these centres for the verification of the – approximately – 43 million electrical energy meters which are used in Germany for billing purposes. The meters may, however, only be verified if they can provide valid proof of their conformity with the construction requirements of the German Verification Law.

The furnishing of such proof then constitutes the third pillar of the field of “Electrical Energy Measuring Techniques”. Here, the customers are national and international manufacturers of electrical energy meters.

In order to measure very large energy quantities, the meters are combined with instrument transformers. For these, too, the department “Electrical Energy Measuring Techniques” offers metrological services for the realization, dissemination and type testing of billing devices. The measurand used here is not the electrical power, but the instrument transformer voltage or current ratio.
Traceable Efficiency Measurement of Electric Motor Units

Frank Lienesch*, Christian Lehrmann

The increase in the efficiency of electric motors protects the energy reserves and is, thus, regarded as a CO₂-saving potential as electric motors consume about one third of electric energy. With the imminent electrification of automobiles, the saving potential will increase still more considerably. Therefore, the European Union has, for the time being, specified minimum efficiencies for standard asynchronous motors. Compliance with these specifications will have to be ensured by market surveillance. The basis of well-functioning market surveillance is the metrological traceability of the measurands and defined measurement procedures.

The efficiency of the electric motor unit is the ratio of output power and input power. To determine the input power, the measurands "electric potential difference U", "current I" as well as their phase angle cosφ are required. In addition to the purely sinusoidal feeding, frequencies of up to 100 kHz must be taken into account for motor units with a frequency converter.

The output power is calculated from the torque and the speed (direct efficiency determination). Alternatively, the respective losses of the machine can be subtracted from the input power (indirect efficiency determination). These losses comprise stator/rotor copper losses, iron/friction losses and the so-called "additional losses". Both procedures are described by the regulation or by the standardization, respectively.

The direct efficiency determination requires the measurement of the electric and mechanical quantities with chronological synchronization. Modern power measuring devices ("power analyzers") provide this option. The dynamic fractions are important in particular for the traceability of the measurand "torque".

The comparison of the measurement results for the defined measurement procedures in accordance with the usual practice shows partly considerable differences which entail interpretation problems for an assessment of the minimum efficiency levels.

The reduction in the measurement uncertainty during the efficiency measurement of electric motor units improves the optimization possibilities for the different losses inside the machines in order to achieve the maximum saving potential.

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Representation of a motor test bed with the most important measurands for the determination of the efficiency η as the ratio of mechanical power (P2) and electric power (P1).
Introduction

There is a high likelihood of climate change already taking place at this very moment, even in Germany. What is already striking today are the longer periods of drought, stronger rainfall, violent storms and winters without snow. Different models show that the average temperature in Germany will have increased by 1 °C to 3.5 °C by the end of this century. The impacts of climate change (such as forest fires, hot spells and rising sea levels) might thus tend to increase in the future. Climate researchers expect the greatest climatic modifications caused by climate change to take effect in south-west Germany, east Germany, the Alps and the coastal regions.

The 4th IPCC climate report dated 2007 [1] has clearly shown that the increasing global warming of the atmosphere is mainly caused by the increase in the concentration of greenhouse gases due to human activities. Climate change could already take on alarming proportions in this century if the emission of greenhouse gases is not reduced rapidly and considerably worldwide. As a result of the 15th Climate Conference in Copenhagen (COP 15) in 2009, a basic level of agreement made it possible, for the first time, to agree upon the concrete goal of limiting global warming to less than 2 °C as compared to the pre-industrial level [2]. In Copenhagen, an agreement was made by the participating countries that the industrialized states were to submit and implement quantified, national emission reduction goals for 2020 by 31 January 2010.

How Does Europe – and Germany in Particular – Deal with These Challenges?

The EU agreed upon an integrated strategy in the field of energy and climate protection for the first time in December 2008. The package [3] contains, among other things, regulations aiming at reducing CO₂ emissions from new vehicles and fuels. By 2020, greenhouse emissions are to be reduced by 20 % (or by even 30 %, if an international agreement is sealed) compared to the figures recorded in 1990. An improved energy efficiency should allow the energy consumption to be reduced by 20 % of the level expected in 2020, with 20 % of the energy being supplied by means of renewable energy sources [4].

In order to limit global warming to + 2 °C, only 2.5 tons of CO₂ on average may be emitted per capita worldwide. For example, a person driving...
15,000 km per year by car with the current average emissions of 170 g/km is already emitting 2.55 t of CO₂ and, thus, already exceeds this threshold to a small extent. With an electricity consumption of 2,000 kWh and a gas consumption of 10,000 kWh for heating, an average one-person household already emits 3.5 t of carbon dioxide, with power and heating alone, and, thus, exceeds the limit of 2.5 t CO₂ by 40 % [5].

The drastic reductions of greenhouse gases by up to 95 % in the EU and 50 % worldwide by 2050 are the most significant measures to limit the impacts of climate change in the long run and to prevent dangerous and irreversible modifications of the climate. In order to implement the European goals in terms of climate protection, the European Commission presented two programmes of measures in 2011: a roadmap for moving to a competitive, but low-carbon economy by 2050 [6], and the European Energy Efficiency Plan [7] which, in Germany, is implemented through an energy concept that was agreed upon in 2010 [8]. The development of renewable energy sources is meant to contribute essentially to reducing greenhouse gas emissions.

Observation and Monitoring Systems

The EU regularly monitors the emission and absorption of greenhouse gases by means of a monitoring system [9]. In order to progressively reduce emissions, the EU has introduced a trading system for greenhouse gas emission certificates with particular rules for fluorinated greenhouse gases [10].

Besides alleviating the greenhouse effect by reducing greenhouse gas emissions, adapting to unavoidable consequences of climate change is the second pillar of climate protection. “Adaptation measures” are necessary to be able to cope with the rising average sea level, with an expected acceleration of coastal erosion, and with increasingly severe weather-related natural disasters. To this end, the EU approved a white paper on adapting to climate change in 2011 [11].

The white paper requires the creation of a solid basis of scientific knowledge on the impacts and consequences of climate change for the EU. Deciding upon the best adaptation method requires access to reliable data on the probable impacts of climate change as well as on the resulting socio-economic aspects and the costs and benefits of the different options.

(Source: Dr. W. Zhang, WMO Presentation at the 10th anniversary of the CIPM MRA, Paris, France 10-MRA)
To establish an international basis for the development of worldwide climate expertise, the World Climate Conference 3 (WCC-3) decided in September 2009 to develop the so-called “Global Framework for Climate Services (GFCS)”. Climate services require well-functioning observation systems, local climate research and the capability of developing climate models for specific applications. Another important element is communication with the users in order to offer customer-oriented climate services. The Global Monitoring for Environment and Security (GMES) is an initiative which was launched jointly by the EU and the European Space Agency (ESA) in 1998. GMES collects data obtained by means of reconnaissance satellites, ground stations and space stations in order to obtain an exhaustive picture of the state of the Earth. The GMES system is the most important European contribution to the Global Earth Observation System of Systems (GEOSS). More than 1000 stations are part of this global network of near-ground climate observation stations which are operated by four global organizations (WMO, UNESCO, the UN Environment Programme (UNEP) and the International Council for Science (ICSU)). In addition to the conventional ground- and water-bound climate and weather observation networks of fixed stations, remote sensing data – especially those coming from satellite platforms – are becoming increasingly important for the purposes of climate monitoring. The data collected by geostationary and polar-orbiting meteorological satellites are converted into geophysical data on the energy and water budget of the atmosphere by, among others, the Deutscher Wetterdienst ("German Meteorological Service"), and are plotted as time series. The database of national GMES projects will provide an overview of current and planned national GMES projects; this database has been under development since 2010 [12]. This is supposed to improve cooperation at the European level in the event of natural disasters, as well as in the event of floods or extreme drought.

**Metrological Objectives**

Together with the other metrology institutes in Europe and the rest of the world, PTB has the task of providing the metrological basis for observation systems to supply comparable local and global climate data over very long, climate-relevant periods. For climate modelling as well as for prognoses and predictions, reliable input data of known measurement uncertainty are indispensable. This applies also to the analysis of the results of climate prognoses on which political and economic decisions are based. For this purpose, close cooperation is necessary with the operators of these observation facilities and with the metrological partner institutes, research centres and standardization organizations.

The basic objective of climate-related metrological research is to establish metrological traceability and comparability for the results of measurements and observations of the most important measurands for Earth and climate observation. These are the so-called "GCOS Essential Climate Variables (ECVs)" identified in meteorology. They are classified as in-situ and satellite-based observations and structured according to the observation areas "terrestrial", "oceanic" and "atmospheric" (over Land, Sea and Ice); the table below gives an overview of the ECVs. Via a GOSIC-GCOS media wiki website, up-to-date information on the measurement and the relevance of the various ECVs is made available and, if applicable, new ECVs are identified [13].

In order to identify, among the numerous ECVs, the measurands for which establishing metrological traceability is a priority, and more generally, to put the cooperation between metrology and meteorology on a solid formal ground, the World Meteorological Organization (WMO), together with the International Bureau of Weights and Measures (BIPM), organized the "WMO-BIPM Workshop on Measurement Challenges for Global Observation Systems for Climate Change Monitoring: Traceability, Stability and Uncertainty" in 2010. During the workshop, the participants identified the climatic issues to be solved by means of metrological support and advanced measurement technology; among these are the in-situ and the satellite-based Earth and ocean observation. The National Metrology Institutes (NMIs) were asked to take the requirements of climate research into account in their future strategies. On 1 April 2010, the WMO signed a Mutual Recognition Arrangement (MRA) with the BIPM [14].
The great strategic significance of a reliable data base for environmental and climate research in Europe is also reflected by the fact that the “Environment” is a “Targeted Programme” of the European Metrology Research Programme (EMRP). The projects are, on the one hand, aimed at local environmental aspects (such as, e.g., water purity and car exhaust gases). On the other hand, they include topics of a more global nature which are aimed at better comprehending the processes of climate change, and at mitigating their impact.

PTB is actively involved in all projects of the EMRP “Environment” Targeted Programme and even leads some of them. The current research projects deal with local environmental aspects such as:

- chemical pollutants in the air;
- complete detection of pollutants contained in exhaust gases;
- ensuring compliance with the European Water Framework Directive;
- the safe disposal of radioactive waste,

but also with global metrological issues:

- improving the detection of UV radiation in the atmosphere;
- calibration of satellite-based spectral radiometers;
- ocean salinity and acidification;
- spectral reference data – fundamental for monitoring the composition of the atmosphere;
- metrology for meteorology.

The objective of the Targeted Programme “Environment” is to improve the quality of the data base for political decision-making processes, for the elaboration of new directives and for the monitoring of compliance with the directives relating to the environment. This is aimed at establishing a metrological infrastructure supporting both national and international research initiatives in Earth and climate observation as well as secure political decision-making in environmental matters [15].

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### Table:

Overview of the GCOS Essential Climate Variables (ECVs), the relevant measurands for metrological research in Earth and climate observation. Source: GOSIC – Global Observation Systems Information Center (http://gosic.org/ios/MATRICES/ECV/ecv-matrix.htm)

<table>
<thead>
<tr>
<th>ATOMICPHIC (over Land, Sea &amp; Ice)</th>
<th>OCEANIC</th>
<th>TERRESTRIAL</th>
</tr>
</thead>
<tbody>
<tr>
<td>Surface</td>
<td>Surface</td>
<td></td>
</tr>
<tr>
<td>Surface Air Temperature</td>
<td>Current</td>
<td>Carbon Dioxide Partial Pressure</td>
</tr>
<tr>
<td>Surface Precipitation</td>
<td>Ocean Acidity</td>
<td>Ground Water</td>
</tr>
<tr>
<td>Surface Radiation Budget</td>
<td>Ocean Color</td>
<td>Lakes</td>
</tr>
<tr>
<td>Water Vapour (Surface humidity)</td>
<td></td>
<td>Snow Cover</td>
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<tr>
<td>Near-Surface Wind Speed and Direction</td>
<td>Sea Ice</td>
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<tr>
<td>Upper-Air</td>
<td>Sea Level</td>
<td>Glacier and Ice Caps</td>
</tr>
<tr>
<td>Cloud Properties</td>
<td></td>
<td>Permafrost</td>
</tr>
<tr>
<td>Earth Radiation Budget (including Solar Irradiance)</td>
<td>Sea Surface Salinity (SSS)</td>
<td></td>
</tr>
<tr>
<td>Temperature</td>
<td>Sea Surface Temperature (SST)</td>
<td></td>
</tr>
<tr>
<td>Water Vapor</td>
<td></td>
<td>Leaf Area Index (LAI)</td>
</tr>
<tr>
<td>Wind Speed and Direction</td>
<td>Carbon</td>
<td></td>
</tr>
<tr>
<td>Composition</td>
<td>Current</td>
<td>Fire Disturbance</td>
</tr>
<tr>
<td>Aerosols Properties</td>
<td>Nutrients</td>
<td>Soil Moisture</td>
</tr>
<tr>
<td>Carbon Dioxide</td>
<td>Ocean Acidity</td>
<td>Soil Carbon</td>
</tr>
<tr>
<td>Methane and other Long-Lived Green House Gases</td>
<td>Oxygen</td>
<td>Ice Sheets</td>
</tr>
<tr>
<td>Ozone</td>
<td>Salinity</td>
<td></td>
</tr>
<tr>
<td>Precursors (supporting the Aerosols and Ozone ECVs)</td>
<td>Temperature</td>
<td></td>
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<tr>
<td></td>
<td>Tracers</td>
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<tr>
<td></td>
<td>Global Ocean Heat Content</td>
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</tbody>
</table>
In order to put the cooperation between metrology and meteorology on a solid formal ground, the World Meteorological Organization (WMO), together with the International Bureau of Weights and Measures (BIPM), organized a joint workshop in 2010 during which the National Metrology Institutes (NMIs) were asked to take the requirements of climate research into account in their future strategies. On 1 April 2010, the two organizations signed a joint agreement in this regard.

From left to right: Len Barrie (WMO), Andrew Wallard (BIPM), Michel Jarraud (WMO), Ernst Göbel (CIPM), and Wenjie Zhang (WMO) at the signing of the Mutual Recognition Arrangement in 2010 at the BIPM.

**Literature**


Spectral Reference Data for Atmospheric Research and Observation

Volker Ebert*, Olav Werhahn

A central European molecular-spectroscopic infrastructure at PTB

The greenhouse effect, climate warming and air pollution – these subjects are promoting the development and improvement of measurement techniques which allow harmful substances, greenhouse gases and environmental pollutants in the air to be monitored and quantified. For this purpose, infrared spectroscopy offers a great number of techniques which require a precise knowledge of molecular spectral data. Spectral parameters such as, for example, line widths or pressure broadening coefficients are required to model the spectral signature of the atmosphere and to quantify remote sensing data of corresponding measuring instruments on the ground, on satellites, balloons or airplanes.

Spectral data are of essential importance for the observation, quantification and prediction of the development of the Earth’s atmosphere, as well as for its radiation and heat budget. There is, however, a concise lack of line data of high metrological quality. To face this deficit and to develop possible solutions, PTB is leading a European research group which is concerned with the establishment of the first European metrological infrastructure for the measurement of traceable spectral reference data.

Within the scope of the project, the European central laboratory, which serves to measure the spectral data, has been established at PTB. With Working Group 3.22 (Metrology in Molecular Spectroscopy), PTB is decisively participating in setting up a high-resolution ($\Delta\nu =10^{-3}$ cm$^{-1}$) Fourier transform infrared spectrometer covering the spectral range from the visible to the mid-infrared. For quality assurance, standardized measurement protocols are being developed and validated in parallel to make all measurement results available in the same quantifiable quality. The measured spectral data will become the foundation of a future metrological database which can then, for example, be used for the so-called calibration-free laser spectrometry [1] for quantitative gas analysis [2] (see next page).

Literature


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Central laboratory of the European spectral data infrastructure at PTB: Fourier transform infrared spectrometer (Bruker IFS-125) with a spectral resolution of $\Delta\nu =10^{-3}$ cm$^{-1}$. 
The measurement of “thick air” requires precisely thin air

The traceable measurement of impurities in air increasingly requires the cooperation of metrologists, test centres and legislators. This reflects a growing consideration of the environment in legislation and the special valency of health protection in Europe. Both of these aspects require improved measurement sensitivities to ensure high-quality measurements of air pollutants such as, for example, ozone or NOx. In this context, PTB cooperates with 12 European metrology institutes to promote air pollution metrology. On the German side, the Umweltbundesamt (Federal Environmental Agency – UBA) and the BAM Bundesanstalt für Materialforschung und -prüfung (BAM Federal Institute for Materials Research and Testing) are involved in addition to PTB.

The project is concerned with the production and quantification of reference gases for atmospheric and indoor air measurements and with the development of special, miniaturized sensors. The results will contribute to the revision and restructuring of EU Directive 2008/50/EC and to the emission certification of products for domestic use (“Blue Angel”).

PTB Working Group 3.22 “Metrology in Molecular Spectroscopy” is working on the characterization of so-called zero gases. A zero gas is, for example, an air mixture with exactly known components of oxygen, nitrogen and carbon dioxide, in which the impurities contained in the atmospheric air lie far below the concentrations measured in real air. Certified zero gases are prescribed in air measurements for instrument calibration. It is the objective of PTB to quantify several substances at the same time by combining established metrological concepts of laser-spectrometric gas analysis (see PTB-Mitteilungen 115 (2005), p. 305ff). For the technology used at PTB, additionally, exact knowledge of spectral reference data of the species to be measured is decisive (see the article on the preceding page). These data are also measured in the Working Group. With continuously decreasing maximum permissible values for emissions and immissions, the production of zero gases is increasingly becoming a problem. A precise certification, i.e. production and quantification of the composition of zero gas, is of immense importance for measurements of air pollutants, because almost all instruments used at environmental metering and monitoring sites must be calibrated with zero gas. Therefore, there is a great demand for certification protocols of metrological institutes.

Traceable Measurements of Air Pollutions

Olav Werhahn*, Volker Ebert

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They can cause lung cancer and other severe diseases: the minute soot particles from the exhaust gases of diesel-engined vehicles, whose number is constantly increasing. In order to nevertheless keep the harmful effects to one’s physical health due to soot particles low, the limit values for diesel soot have been reduced drastically step by step, from 180 mg/km (EURO 1, 1993) down to 5 mg/km in EURO Standard 5. In this standard, not only the mass concentration of the soot particles but also their number is examined. Through this, more recent results regarding the implications which the incorporation of soot particles represent to health are taken into account. Monitoring of the number of soot particles emitted requires the optimization of measurement procedures and the approval of corresponding measuring instruments. To adapt the complete measurement system – from the approval tests and calibrations up to the regular exhaust gas examinations – to the new requirements, a pan-European project has now been started. In this project, several national metrology institutes are cooperating with partners from industry under the auspices of PTB. Besides the diesel soot, the project also addresses two additional materials which are problematic to health and pollute the air: the smallest particles of platinum from catalyzers and other elements from the platinum group, as well as those mercury compounds which are created in the combustion of fossil energy sources in power plants.

The objective of the project, which is to run for three years, is to compare the different approaches to further develop measuring instruments and to assure the correct traceability of measurement results – i.e. to improve the measurement procedures in all fields. To this end, internationally harmonized standard procedures are to be developed for the particle number measurements (instead of their mass, as previously), which will then facilitate the calibration and approval of measuring instruments.

The significance of this subject has also become obvious for industry through the large number of partners from the ranks of the measuring instrument manufacturers and from the automobile industry. The two other subjects also present particular challenges for metrology. In the case of the so-called platinum group elements (platinum, palladium and rhodium), these elements escape into the air as “abrasion” from catalysts and represent a problem to health. To better assess this problem, it is – first of all – necessary to exactly know the amount of the escaped platinum group elements. The same problem is encountered for the release of mercury into the environment.
Organic tin and mercury compounds, inorganic cadmium compounds and brominated fire protection agents are chemical substances which have been increasingly discussed in the press lately. Their potential danger for the environment is so high that extremely low limit values have been laid down for these substances in the EU Water Framework Directive (WFD-2000/60/EC) and that only comparable – i.e. traceable – measurement results may be used for monitoring the compliance with these limit values. To support the implementation of this requirement, PTB has initiated a European research project in which it was shown how the traceable matrix reference materials which are required for this purpose can be made available throughout Europe in an efficient and sustainable way [1].

The link-up of all chemical-analytical laboratories in the EU which want to carry out measurements in connection with the WFD requires reference materials which have been traced back correspondingly. These reference materials are, however, hardly obtainable. Within the scope of the EURAMET project – which was coordinated by PTB and realized in cooperation with the BAM Bundesanstalt für Materialforschung und -prüfung (BAM Federal Institute for Materials Research and Testing), the French Laboratoire national de métrologie et d’essais (LNE) and the Institute for Reference Materials and Measurements (IRMM) of the EU – it could be successfully demonstrated – using the example of the toxic heavy metals Pb, Hg, Ni and Cd which belong to a list of priority substances – how these can be made available easily and in an efficient way. Of central importance were laboratories which are experienced in the analysis of the priority substances and which are also entrusted with the organization of comparison measurements within the scope of national QA systems. In Germany, these were, for example, the laboratories for environmental analysis of the Federal States. One of the objectives of the project was to enable these “potential calibration laboratories” (PCL) to make traceable reference values available for the comparison measurements organized by them. This allows the participants to validate their measurement procedures metrologically and to ensure traceability to the International System of Units (SI). The link-up of the PCLs with the National Metrology Institutes was realized with the aid of a comparison measurement. [1]

European network of four national metrology institutes (NMIs) and 22 “potential calibration laboratories” (PCL) from 10 European countries (plus 2 laboratories from Israel and 2 NMIs from non-European countries) set up within the scope of the project. The NMIs developed the primary measurement procedures and provided the reference values for the samples used. The PCLs, for their part, canvassed more than 100 routine laboratories from 17 EU countries to link them up with an additional comparison measurement and certified the samples used for this purpose.

When the environment is contaminated with radionuclides, decision-makers and the public expect fast, highly sensitive and reliable measurements for the assessment of potential radiological consequences. The reliability of the measurements is achieved by tracing the measurement results back to national primary standards. The most sensitive measurements of radioactive substances in the air are carried out by the trace survey stations which thus contribute to the realistic assessment of even negligibly small, additional radiation exposures.

The "Radioactivity" Department of PTB realizes the legal unit the "becquerel" (Bq) in the form of activity standards which are prepared in accordance with the half-lives of the radionuclides contained in them and which are prepared time and again. These standards are used to calibrate measuring instruments, but also to manufacture realistic reference materials for intercomparison measurements organized by other federal authorities. For this purpose, both environmental samples doped with an activity standard solution and natural radioactive reference materials are used. The reference values of the specific activity of radioactive nuclides in these materials are determined with small uncertainty by calibration measurements of PTB.

The trace measurement or trace analysis of radioactive substances in air is the very sensitive measurement of the smallest activity concentrations. In 2011, short-lived radionuclides from nuclear reactors in Fukushima (which is approx. 9000 km away) were measured every day at PTB [2]. Under radiological aspects, the activity concentrations measured in Germany were insignificant. They were, however, used – for example – to correctly balance the approved discharges of the nuclear power plants.

Trace measurements, as they have been carried out at PTB since 1963 [1], are required for radioecological investigations or for the detection of background radiation levels. The figure shows the time-dependent evolution of the activity concentration of the natural radioactive $^{7}$Be and of the anthropogenic $^{137}$Cs. The activity concentration of $^{7}$Be varies in a constant range, whereas that of $^{137}$Cs – which originates from atmospheric nuclear weapons tests – decreased until 1986. In May of that year, it increased for a short time clearly above that of $^{7}$Be as a result of the accident at the nuclear power plant in Chernobyl. After the accident in Fukushima, the peak values were smaller by about 3 to 4 orders of magnitude.


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**Monitoring of Radioactive Substances in the Environment**

Herbert Wershofen*, Dirk Arnold

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*Time-dependent evolution of the activity concentration of the natural radioactive $^{7}$Be and of the anthropogenic $^{137}$Cs.*
Today, measurements with optical remote sensing procedures from aircrafts and satellites make an essential contribution to global Earth monitoring. They allow large-surface coverage and continuous observation, and they furnish information. On the basis of this information, political decisions can, for example, be taken on the measures to be applied to reduce a possible climate change. The harsh environmental conditions of the satellites, aircrafts or balloons, as well as the required correction of the influence of the atmosphere on the measurement results contribute to the fact that the measurement uncertainties which are at present achievable are clearly limited. On the other hand, climate models require radiometric measurement data over long periods of time with uncertainties which sometimes represent a metrological challenge even under laboratory conditions. From this it follows that for comparable and sufficiently exact remote Earth sensing data, the traceability and uncertainty of the measurement data must be determined and improved in all steps of their obtainment and assessment in accordance with metrologically validated procedures.

The activities are all the more important as the “Group on Earth Observation” (GEO), an international association of space agencies and geological research institutions, is planning to establish a global Earth observation system (GEOSS: Global Earth Observation System of Systems) by the end of 2015. To ensure the good functioning of such a comprehensive system, not only the sensors which are used by the different institutions for a measurement procedure, but also different measurement procedures, for example procedures which work in the optical and in the microwave range, must furnish comparable results. Likewise, satellite-based measurements from space, airplane- and balloon-based measurements in the atmosphere and measurement networks on the surface of the Earth must furnish consistent results to allow reliable statements to be made. Here, current research projects, which are to contribute to the achievement of this ambitious goal, set in. With its sometimes unique radiometric and thermometric measuring arrangements, PTB makes essential contributions to these research projects.

Within the scope of an international project, airborne infrared radiation standards are at present being characterized and calibrated on the measuring facility shown in the figure. These reference sources allow hyper-spectral radiometers on board aircrafts, balloons or satellites to be traced back to quantities which are relevant to the climate such as, for example, the surface temperature of the Earth or distributions of temperature and trace gas concentrations in the atmosphere.

Traceable Radiometry for Remote Sensing of the Earth

Christian Monte, Berndt Gutschwager, Jörg Hollandt*
Metrology for Oceanography

Petra Spitzer*

The spatial differences in the density of seawater caused by temperature and salinity gradients are the reason for the deep sea currents which, in turn, largely determine our climate. Even small changes in the composition of seawater can disturb the existing balance severely. This can be reflected by changed climatic conditions, but also by changes in the diversity of the species in the oceans. This is why very small changes of temperature and salinity – but also of the pH value and of the dissolved oxygen concentration – are measured, analyzed and modelled within the scope of climate research over large periods (decades, centuries).

To guarantee comparability of the data obtained worldwide and over long periods of time, it is very important for oceanographic observation systems to measure these parameters with known uncertainty and with traceability to the SI. Only measurement values with known uncertainty can serve as a basis for the wide application of seawater descriptions as contained, for example, in the new international Sea Water Standard TEOS-10.

This is the subject of a joint research project of 11 European partners from metrological institutes and universities. PTB is coordinating the project and is also heading the work package for the implementation of a traceability chain to allow the practical salinity to be traced back to density standards. It will thus be possible to measure this established measurand with traceability to the SI. PTB will also make contributions to the measurement of the sound velocity and the temperature as well as to the development of pH standards.

Another objective of the project is an improved comparability of measurements of the pH value and of the dissolved oxygen concentration of seawater as well as the reliable determination of nutrients such as nitrates, phosphates and silicates and of traces of iron species which are incorporated in oceans mainly by atmospheric dust.

The importance which is attributed to the project in the oceanographic community can be seen from the number and scope of the cooperation partners. International organizations such as, for example, the Scientific Committee on Ocean Research, the International Association for the Physical Sciences of the Oceans and the International Geotraces Project are represented as well as the Federal Maritime and Hydrographic Agency and the Leibniz Institute for Baltic Sea Research (IOW).
Metrology for New Technologies

Harald Bosse*

In the different phases of product engineering (preliminary research, development, prototype fabrication and optimised production), the design and development of products make the use of supporting metrological techniques necessary which are adapted in the best possible way to each single development phase with regard to resolution, speed, robustness, comparability, measurement uncertainty and costs. This is particularly true when it comes to the engineering of products involving new technologies. In accordance with the EU research programme NMP, these “new technologies” encompass nanotechnologies, materials and new production techniques which have been identified as being important fields of technology for transforming an industry based on resources into an industry based on knowledge. In this article, PTB’s contributions to the metrological support of these new technologies will be presented and illustrated by means of examples which will highlight certain developments in more detail.

Nanotechnologies

In its “Aktionsplan Nanotechnologie 2015” [1], the German Federal Government describes nanotechnologies as one of the key technologies for Germany, providing great potential to develop new products, procedures and services in a large range of various applications – from new materials to the medical field. The international standardisation committee which was set up in 2005 to harmonise the terminology and to elaborate specific standards in the field of nanotechnologies, ISO TC 229 “Nanotechnologies”, published the following definition: ”Nanotechnology: application of scientific knowledge to manipulate and control matter in the nanoscale (size range from approximately 1 nm to 100 nm) in order to make use of size- and structure-dependent properties and phenomena, as distinct from those associated with individual atoms or molecules or with bulk materials” [2].

The work carried out by PTB on nanotechnologies is – in accordance with PTB’s spectrum of tasks – focused on nanometrology. The term “nanometrology” encompasses all the activities related to the traceable metrological characterisation of nanoscale objects and structures, whereby reference is made to the SI, the International System of Units, for the expression of the measurement results. The aim is to obtain comparable measurement results on nanoscale objects and structures within the measurement uncertainties required for their application by developing new measurement methods and by applying improved established measurement procedures. The extremely small dimensions of the structures to be characterised represent a particular challenge in nanometrology. The appropriate measurement methods should therefore be adapted to the nanoscale test objects with regard to their resolution. The traceable determination of the concerned parameters of the nanoscale objects (such as, e.g., their dimensions, including the estimation of the corresponding measurement uncertainties) requires – also when high-resolution measurement procedures such as, e.g., scanning probe microscopy or scanning electron microscopy are applied – the interaction of the measuring probe with the test object to be explicitly taken into account. In the example of a scanning probe microscope shown below, the model for the measurement process has to take into account, among other things, the shape of the object being measured.

Figure:
The colour-processed image taken by the atomic force microscope shows polystyrene spheres. In a comparison: a hair is as thick as 300 such spheres places next to each other. For PTB, especially the symmetrical arrangement of these nano-objects is interesting for use as 2D calibration lattices: it can be used as a ruler to determine the size of other objects. This photograph, which was actually taken rather for aesthetic reasons, however, shows irregularities in the crystal lattice. (Photo: Danzebrink)
and dimensions of the nanoscale-fine probe tip as well as the probing characteristics of the probe tip at the surface of the nano-object; in the case of the scanning electron microscope, the scattering process of the primary and of the secondary electrons in the nanostructure, among other things, has to be considered. An example of the application of scanning electron microscopy to the traceable determination of the size and size distribution of nano-particles is presented in the next article of this brochure, “Characterising Nanoparticles by Means of Electron Microscopy” (p. XY) [3].

When characterising nanostructures or nanoparticles, one generally differentiates between microscopic and integral measurement methods such as, e.g., scattered-light methods. Whereas single nano-objects can be investigated with microscopic procedures, the integral methods provide information on a whole ensemble of nano-objects. Examples of integral measurement procedures used at PTB are small-angle X-ray scattering to investigate nanoparticle samples in aqueous environments [4] and scatterometric measurement procedures at different wavelengths from the deep ultraviolet (DUV) [5] to the extreme ultraviolet (EUV) [6] to characterise lattice structures on semiconductor test objects. In this case, EUV radiation with a wavelength of 13.5 nm is supplied by synchrotron radiation sources (BESSY II and MLS). The article “Characterising Nanostructures on Photomasks” (p. XY) shows the optical measurement methods used at PTB for the dimensional characterisation of the dimensions of nanostructures on photomasks which are supplemented by scanning probe microscopy and scanning electron microscopy. A special vacuum interference comparator, the so-called “nanometer comparator”, is used to measure the position of micro- and nanostructures on masks and uni-dimensional grating standards of up to 600 mm in length; it is an excellent reference measuring instrument which, on suitable test objects, achieves measurement uncertainties in the nanometer range [7].

In lithography, but also for the shaping of beams in synchrotron radiation sources, extreme requirements are placed on the roughness and the shape of the optical components used (the surface of lenses or mirrors). The dimensional tolerances to observe for their fabrication often lie in the sub-nanometer range. To determine the shape of a surface, reference-free measurement methods have been developed at PTB which are based on the measurement and assessment of local surface inclinations of the sample surface to be determined [8]. These local inclinations of the surface are measured by means of electronic autocollimators which can be calibrated with highest accuracy on PTB’s reference angle comparator beforehand [9]. To determine the roughness components on surfaces, PTB has various tactile, optical and SPM measurement methods at its disposal. To this end, a metrological scanning probe microscope with a large measuring range of 25 m × 25 mm × 5 mm makes it possible to determine spectral components of the surface deviations in the roughness, waviness and, partially, also in the shape range simultaneously [10]. The executive office of the Nanotechnologie-Kompetenzzentrum für ultrapräzise Oberflächenbearbeitung (Competence Centre “Ultraprecise Surface Figuring” – CC UPOB), which is based at PTB, shows particular commitment to this field of work [11].

One aspect of nanotechnologies that is particularly interesting for PTB is the possibility of exploiting nanotechnological developments for metrology itself. An example of such an application of nanostructures to improve the sensitivity of a measurement procedure is Surface-Enhanced Raman Scattering (SERS). By using plasmon effects on metallic nanoparticles or nanostructures, the intensity of the local electric field in the vicinity of the nanostructures can be considerably increased. Analytes which have to be investigated and are in the vicinity of the nanostructures in a solution and are, thus, located in areas of considerably enhanced local electric fields, are excited to reach much stronger Raman scattering. The intensity increase in the Raman signal, compared to conventional Raman spectroscopy, can reach a factor of approx. 1,000. This distinct sensitivity increase makes it possible to investigate analytes in the range of their natural concentration, e.g. for creatinine in blood serum [12].

To manufacture nanostructures, e.g. for the nanostructured SERS substrates, but primarily to realise electric quantum standards, PTB operates procedures for the lithographic structuring of substrates which achieve the highest possible resolution when methods of electron beam lithography are used. These lithographic manufacturing processes are complemented by methods of laser structuring as well as by methods of mechanical ultra-precision processing. An overview is given in [13].

Another field of activity of PTB is the determination of the magnetic properties of a nanoparticle ensemble as well as of single nanoparticles. Magnetic nanoparticles are, for instance, used in medical diagnostics and therapy (directed magnetic drug targeting, magnetic thermoablation). Magnetic thin films and coupled layer systems are core components for data storage in hard disks. “Spintronics” is a collective term to describe possible applications of magnetic nanostructures as non-volatile dynamic storage elements in electronics. PTB conducts investigations on the dynamics of magnetisation processes in nanostructured magnetic storage cells [14].
For length measurements traceable to the SI definition of length with target uncertainties in the nm range and less, laboratories normally use optical interferometers with wavelengths corresponding to the optical radiation in the spectral range of visible light (e.g. HeNe lasers with $\lambda = 632$ nm). To be able to use it to measure displacements in the nanometer range, the interferometer signals must be electronically interpolated. Due to residual deviations in the orientation of the interferometer components as well as non-ideal optical properties of the components, non-linearities of the interpolation may occur which cause deviations of a few nanometres in length measurements. At PTB, measurement methods are therefore being investigated and applied which exploit the potentials of crystalline volume and surface lattices for length measurement in nanometrology [15]. An example of this is the X-ray scanning interferometer, in which a shift of the mobile lamella of a monolithic Si single crystal by 0.192 nm (220 lattice level distances) already leads to a full interference signal at the detector of the X-ray interferometer.

Within the scope of a European project involving PTB, the future requirements in essential fields of nanometrology in Europe have been analysed [16]. The results of the project will be taken into account especially in the designing of the EU’s 8th research framework programme in the field of nanometrology and will, thus, also have an influence on PTB’s future activities in the field of nanometrology as a basis for nanotechnologies.

**New materials**

Material-specific issues are the primary task of PTB’s sister institute falling under the competence of the Federal Ministry of Economics and Technology (BMWi), namely the Federal Institute for Materials Research and Testing (BAM) in Berlin. When investigating certain properties of some materials which are particularly important for technical applications, PTB’s metrological expertise comes in.

An example of this is the investigation of the thermal properties and long-term stability and compressibility of low-dilatation high-tech glasses, glass-ceramics and ceramics which are frequently used both in precision-manufacturing devices such as, e.g., DUV and EUV wafer steppers for the lithographic fabrication of integrated circuits and in precision measuring instruments. On the one hand, temperature-stable, structural components in manufacturing and measuring machines and, on the other hand, substrates made of stable materials with microstructured scale divisions can be used as measuring components for precision length and angle measurements. The investigation of the stability with lowest possible uncertainties is performed on gauge-block-shaped material samples in special interferential length comparators which can achieve a targeted variation of the pressure and the temperature. For further details concerning the method used, please refer to the article “Stability of High-tech Materials” (p. X); an overview is provided in [17].

The hardness of materials as well as the hardness of the layers and layer systems applied to surfaces is a material property that is very important for a proper functioning of these. The material hardness is decisive when it comes to selecting mechanical processing methods for materials; furthermore, the hardness, as a material property, determines decisively the wear of contacting surfaces. For the traceable determination of hardness as a material property, investigations have to be carried out in which a defined indenter (material, shape, dimensions) acts on the sample surface to be examined with a certain force and in a defined manner (measuring cycle consisting of a strain and a relieving phase). In order to achieve low measurement uncertainties, the applied force, the shape of the indenter and the geometry of the indentation created by the indenter in the sample surface during the hardness-measurement test have to be measured with great accuracy.

Characterising the mechanical properties of nanostructures and of thin films in the sub-micrometre range is a particular challenge; they are investigated using the nano-indentation method [18]. For the indentation tests on such samples, only very small forces in the nanonewton range must be used; an appropriate standard measuring device for the traceability of these small (nano-) forces is currently being set up at PTB [19].

Material innovations play a decisive role also in the semiconductor industry, e.g., for the development of oxide layers having a minimal thickness but providing sufficient electrical insulation in modern semiconducting transistor components. Determining the stoichiometry and the layer thickness of ultra-thin insulation layers made of materials such as, e.g., hafnium silicon oxide, is a challenge for quantitative analytical metrology which is met at PTB using X-ray spectrometric methods based on synchrotron radiation [20].

**New production technologies**

If a national economy which is based on the production of innovative products and on the development and application of new production technologies wants to be successful, it needs a reliable metrological infrastructure. Checking the tolerated characteristics during the production of the components and products requires the use of measuring instruments that are traceable in accordance with quality assurance standards.
Metrology for the Future

Thereby, PTB – which is at the top of the calibration chain established in collaboration with manufacturers and calibration laboratories – guarantees the traceability of measured values to the SI system via the national standards. By assuming its statutory tasks, namely the dissemination of measures, PTB supports industrial production in Germany and, due to internationally intertwined production structures, also abroad.

This applies, in the first place, to the measurands and measuring ranges which are necessary for the quality control of established production processes. Providing suitable metrological traceability for new production technologies represents a particular challenge. New manufacturing procedures or the extension of the scopes of application of existing production processes place new requirements on the metrological support structure of process control. In the following, examples of certain measurands are listed for certain technological fields in which particular metrological challenges arise with regard to process control and which are dealt with at PTB:

- Dimensional measurands: on the one hand, challenges arise with regard to the characterisation of micro-components due to the small dimensions and the high aspect ratios of the structures and, on the other hand, with regard to very large components with dimensions of 10 m and more (see: “Laser-aided Measurement of Large Components” (p. XZ)) [21].
- Temperature-related measurands: improved measurement procedures for the International Temperature Scale from -60 °C to 3,000 °C and characterisation of imaging temperature-measuring systems.
- Electrical metrology: research on quantum standards which are to be used close to practice, e.g. directly in production.
- Optical metrology: full-scale characterisation of the spectral properties of novel effect lacquers which change colours depending on the angle one looks at them.
- Mechanical measurands: particular challenges arise when it comes to determining dynamic mechanical quantities; the article “Dynamic Measurement of Mechanical Quantities” (p. VX) gives a good overview of this topic [22].
- Pressure and vacuum metrology: extending the measuring ranges and reducing the measurement uncertainties, e.g. for the traceable determination of the outgassing rates of new materials.

Besides off-line measurement in a laboratory under well-defined conditions, also production-integrated measuring technology is increasingly gaining in importance for efficient and comprehensive process control. The roadmap “Fertigungsmesstechnik 2020” (“Production Measurement Technology 2020”) [23] published by VDI emphasises the importance of the so-called in-line measurement technique for fast and safe process adjustment. Furthermore, it points out that also the in-line sensors and the in-line measuring instruments have to be traceable. PTB is involved in projects and directives concerning in-line measurement techniques with [24]. To be able to manufacture measuring instruments, components and standards of great accuracy in compliance with the requirements of PTB’s experts, PTB needs an efficient Scientific Instrumentation Department. The article “Production-integrated Measuring Technology” (p. XCV) gives the example of a test mass production for a European space project which illustrates the importance of production-integrated measurement techniques for precision production/precision measurement technology [25].

Literature


Nanoparticles often exhibit properties which differ from those of “large” particles made of the same material. This exactly is what makes them so interesting for various applications – but it also bears risks for humans and for the environment. To estimate these risks – and to advance the technical development – procedures to determine the exact size of these particles are important. PTB has therefore developed a new measurement procedure for nanoparticles which is based on a scanning electron microscope (SEM) equipped with an additional transmission detector. Such equipment brings about a solution to a general problem which occurs when spherical nanoparticles are measured with high precision: the precise determination of the particle boundary which – even in high-resolution electron microscopic images – is somewhat “blurred”. The question is: At which grey scale value does the particle begin, and which image pixel still belongs to the background? To determine this signal threshold level, a deeper understanding of how the image is generated inside the electron microscope is necessary. The detector signal can be calculated by means of a Monte Carlo simulation which was developed at PTB and which takes into account the interaction of the electrons with the spherical particle and the properties of the detector. This has shown that the signal threshold level at the edge of the particle depends on the properties (material and size) of the particle. To be able to take these two properties into account, an automatic image evaluation algorithm has been developed. Iteratively for each single particle, it computes an individual signal threshold level for the particle’s edge based on the simulation results. This enables an accurate determination of the size, adapted to the corresponding particle. Despite the complexity of the procedure, hundreds of images can be analysed in less than an hour. In addition, the total time needed to fully characterise a nanoparticle sample is considerably reduced by the use of a newly developed routine which makes it possible to capture numerous images of nanoparticles automatically. Thanks to this new procedure, medium-sized particles of down to approx. 7 nm can be determined with low measurement uncertainties from 1 nm to 2 nm. The work was carried out within the framework of a European Metrology Research Project in which also an extension of the procedure to non-spherical particles is aimed at.

Characterising Nanoparticles by Means of Electron Microscopy

Tobias Klein*, Carl Georg Frase

Calculated dependence of the signal threshold level on the particle material and diameter to determine the size of the particle in the SEM image (left), and histogram of the size distribution measured on a gold nanoparticle sample.
Today, integrated circuits consist of structures which may only be a few 10 nm in size. For the lithographic fabrication of these semi-conducting structures, photomasks are used as templates which can be manufactured with an accuracy of just a few nanometres. At PTB, high-resolution optical microscopy, among other things, is being further developed to measure such nanostructures. One possibility is to use a shorter wavelength. Therefore, an optical microscope is currently being developed and characterised which has an illumination wavelength of 193 nm. The figure shows one of the first test images of a circular-lattice calibration structure.

For the exact optical measurement of periodically structured surfaces, an alternative, non-imaging measurement procedure is applied. In this procedure, the surface is illuminated with a laser. The angular distribution and the intensities of the scattered light allow the size and the form of the grating structures to be determined. At PTB, such a scatterometric measuring system has been developed, based on a 193 nm laser system. Even shorter wavelengths are available to PTB at the synchrotron storage rings BESSY II and MLS in Berlin, where measurements can be performed at a wavelength of 13.5 nm. Radiation from this wavelength is used in EUV lithography to be able to manufacture even smaller semi-conducting structures.

Besides the optical measurement techniques, other procedures such as, e.g., atomic force microscopy (AFM) and scanning electron microscopy (SEM) are used to characterise nanostructures on photomasks. These procedures have the advantage of having a better spatial resolution. Since optical methods are, however, better suited to perform fast, non-contact and gentle measurements, they are often used for, among other things, process and production checking. The results obtained by the different procedures must be comparable with each other. Fig. 2 shows an example of the result of linewidth measurements carried out using PTB’s scatterometer and a scanning electron microscope.

The accurate analysis of the measurement data requires physical models describing the experiments as precisely and realistically as possible. For this purpose, the properties of both the measuring system and the test object have to be known precisely. Currently, numerical model calculations and data analyses of optical measurement results are still limited to simple structure models. Therefore, measurements with smaller uncertainty can presently be carried out only on high-end test objects with well-defined structures. Future challenges for optical metrology will be to extend the measurement procedure to complex 3D structures, structures with a large height-/width ratio and structures with a complex layer set-up.

Figure 1: Test image of a circular lattice (spacing of the lattice lines: 200 nm) with the 193 nm microscope which is currently being set up.

Figure 2: Comparison measurements of the linewidth with a critical dimension scanning electron microscope (CD SEM) and PTB’s scatterometer on various measuring fields of a photomask; the mean values have been subtracted for both: the variation of the linewidth between the measuring fields is rendered by both systems in the same way; the deviations lie in the sub-nm range.

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Thermally stable materials play an important role in dimensional metrology and in precision manufacturing. The currently highest requirements for the thermal stability of critical components are – in EUV lithography – placed on reflection masks and mirrors. These are, therefore, based on substrates made of high-tech glass/ceramics which are to exhibit a very low thermal expansion coefficient \( \alpha (\alpha < 1 \cdot 10^{-8} \text{ K}^{-1}) \).

For the precise characterisation of gauge-block-shaped measuring objects made of high-tech materials, a precision interferometer was developed with the aim of measuring the absolute length of samples up to 400 mm length with uncertainties in the sub-nanometer range. From such accurate length measurement, the thermal expansion coefficient can be calculated as a function of the temperature with uncertainties of up to \( 2 \cdot 10^{-10} \text{ K}^{-1} \). Furthermore, quantitative statements can be made with regard to the homogeneity of the thermal expansion, the compressibility, the length relaxations and also the long-term stability of samples. Length measurements with sub-nm uncertainties demand, besides the application of frequency-stabilised lasers, the consideration of influences whose uncertainty contributions can be minimised only with difficulty. For this purpose, various methods have been developed at PTB in the past few years and these have been integrated into the measuring process. An example of this is a new autocollimation process which ensures that the lightwaves hit the surfaces of the measuring objects exactly perpendicularly. The so-called cosine error is hereby lowered to under \( 10^{-11} \). Furthermore, during the electronic evaluation of the interference pattern, the exact assignment of the sample position to the camera pixel coordinates is considered. This is particularly important when it comes to measuring objects whose end faces are non-parallel due to temperature-induced changes of the lateral sample position. By taking the temperature-related influence of the deflection of the end plate wrung to the back into consideration, the precision could be increased further. When taking thermal expansion measurements on typical samples, length measurement uncertainties of 0.25 nm are now achieved.

Stability of High-tech Materials

René Schödel*

Thermally stable materials play an important role in dimensional metrology and in precision manufacturing. The currently highest requirements for the thermal stability of critical components are – in EUV lithography – placed on reflection masks and mirrors. These are, therefore, based on substrates made of high-tech glass/ceramics which are to exhibit a very low thermal expansion coefficient \( \alpha (\alpha < 1 \cdot 10^{-8} \text{ K}^{-1}) \).

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Thermal expansion coefficient \( \alpha \) of a special glass with extremely low thermal expansion. The progress of \( \alpha \) shown here derives from a series of interferometrically measured lengths of a sample with parallel end surfaces as a function of temperature. In this example, there is a point of reversal at approximately 17.5 °C at which the thermal expansion disappears. Below this point, a reduction in temperature causes an expansion of the material.

This picture shows a typical topography of the interference phase, which includes the front surface of the body and a wrung end plate. The averaged phase values within the rectangular areas on the end plate (left/right) and on the front surface of the body (centre) are the basis for the interferometric length measurement.
Laser-aided Measurement of Large Components

Frank Härtig*, Klaus Wendt

Numerous modern measuring systems are available to monitor and assemble large workpieces and components several metres in height. Depending on the tasks to be fulfilled and the accuracy required, the systems rely on different sensors and measuring principles. Laser trackers, laser scanners and triangulation-based indoor GPS belong to the laser-aided 3D systems. These are typically used in automotive engineering, aeronautics and aviation as well as in naval architecture and crane construction, wind turbine gearboxes and numerous other technical fields in which dimension, shape and position tolerances have to be determined on large workpieces. Checking machining tolerances requires the knowledge of the task-specific measurement uncertainty. To date, this uncertainty can, however, not be specified satisfactorily with regard to traceability. The reason for this is a lack of measuring instruments and measurement procedures to calibrate standards for industry with sufficiently low uncertainty. At PTB, a portable, laser-aided measurement procedure has therefore been developed within the scope of the European research project NIMTech to calibrate large, workpiece-type components directly on-site in industry in order to use them as standards.

PTB’s new measuring system M3D3 (Mobiles Multilaterations-3D-Messsystem) mainly consists of four, or more, high-precision, auto-tracking laser interferometers, the so-called “LaserTracers”, which were developed within the scope of a cooperation involving PTB and are now commercially available. During a measurement, each LaserTracer, as an independent measuring system, follows a retroreflector which is moved within the measuring volume by a mover (e.g. a coordinate measuring machine or a robot). The tracer hereby performs continuous interferometric length measurements. From the extremely accurate length measurements, 3D coordinates of the reflector positions are calculated by evaluation based on the multilateration principle. Comparison measurements have shown that smallest uncertainties of \((0.2 \mu m + 0.2 \mu m/m \times L)\) can be achieved, regardless of the spacing between the data points. To ensure that all LaserTracers perceive the retroreflector simultaneously whilst measuring large structures by means of the M3D3 system, a special, two-step procedure has been developed within the scope of the project which is applicable to all motion systems with reproducible, systematic measurement errors.

Due to the development of the new mobile measurement procedure PTB will, in future, be able to calibrate workpiece-type standards having a physical size of up to 5 m × 5 m × 5 m on-site. This procedure was successfully tested in industry by measuring a large-scale gear standard.
In numerous fields of industrial metrology, e.g. in process control in production, in component monitoring and in safety checking, we have to deal with rapidly changing signals when measuring mechanical quantities such as force, torque or pressure. These dynamics pose new challenges to metrology, since the traceability of the measuring instruments used to national standards has, to date, been based on static procedures. The (static) scales for force, pressure and torque are realised with greatest accuracy at PTB according to the principle of the action of force of a mass in the gravitational field of the Earth.

For dynamic applications, statically determined transducer characteristics can, however, be used only with some restrictions. In dynamic systems, masses move, and the induced inertial forces of the components in motion and their coupling rigidities to adjacent mechanical structures have to be taken into account. This requires – both for the calibration and for the application – new methods of realisation and dissemination of the unit, but also a new approach to data analysis and measurement uncertainty.

In a research project which was started in 2011 within the scope of the European Metrology Research Programme and which involves 9 European metrology institutes, basic work is being done to create an infrastructure for the metrological traceability of the dynamic measurement of mechanical quantities. Already in earlier research work, PTB had acquired a leading position worldwide in the field of dynamic measuring techniques. For the measurand “force”, there are already several experimental facilities in existence for the primary dynamic calibration of transducers with periodic or pulse-shaped input quantities (see the picture). For the measurands “torque” and “pressure”, the corresponding facilities for dynamic primary calibration are currently being developed.

The aim of the EMRP project is to validate the facilities and designs, among other things, by comparing them with those of the European partners, and to develop methods to use the calibration results more effectively in industry – e.g. in DAkkS-accredited calibration laboratories. The mathematical modelling of measuring chains and measuring equipment as well as the numerical procedures used to identify (quantify) the modelling parameters hereby play a significant role. Also, developing and implementing methods to calculate and propagate measurement uncertainty components in dynamic systems is a great challenge for all parties involved.
In metrology and in scientific instrumentation, but also in industrial mass production, there is a constant trend towards increasingly complex and ever more accurate workpieces. Whereas the production accuracy of modern machine tools in one axis usually lies around 8 µm, an accuracy of a few µm may be required for the components to be manufactured on diverse geometrical elements. These requirements can only be met by production-integrated measuring technology. Within the scope of a European, satellite-aided aeronautical experiment carried out to investigate the equivalence principle of inert and gravitational mass, PTB’s Scientific Instrumentation Department has produced a certain number of cylindrical test mass protection heads of approx. 80 mm in length and 70 mm in diameter and with length, shape and position tolerances in the range between 1 µm and 5 µm. This was possible only by integrating measuring technology into the machine tools, since the clamping and unclamping of the test masses would otherwise have led to impermissible errors.

By means of the measuring arrangement shown in the figure, it was possible to measure the contours of the test mass and to determine the production steps which were still required. In order to rule out a possible axis error, this measuring arrangement was calibrated using calibrated material measures which were also integrated into the machine tool.

Production-integrated measuring technology – with the support of integrated, calibrated standards as well as additional measurement results of the manufactured components, and determined by means of special coordinate measuring machines – is the only way of meeting the extraordinary requirements placed on the production accuracy. These investigations have shown that by integrating dimensional measuring techniques in a machine tool, it becomes possible to reduce machining tolerances considerably. The measuring arrangement is universal and can also be used for other workpiece geometries. It is, for example, possible to compensate tool wear, among other things, occurring during production. In order to transfer these developments to applications in industry, special attention must be paid to machine control and workpiece cleaning before the beginning of a measurement cycle.

3 mm tactile sensors for the in-situ measurement of the position and the depth of test mass countersinks.
The issue of health constantly concerns practically every human being more or less intensively. It is a topic which goes far beyond the concern of individual people about their health and, thus, is of significance for society as a whole. It not only deals with the relation between individual people and medicine, between a patient and his doctor, but it comprises far more – e.g. how our living conditions are organized, especially the marginal conditions to which we are subjected in our working environment – and the conservation of nature. Here it is the precautionary (legal) regulations which contribute indirectly to the improvement of the health situation of each individual person.

Consequently, the issue of health is one of the important future fields of German and international research and development policy, and it has been a consistent step to identify metrology for health as one of the big challenges for future priorities for the European national metrology institutes (NMIs). And this is not just the case because the subject is “topical” at the moment, but because it has become clear that the life sciences, medicine and the medical engineering industry urgently need a metrological foundation which goes beyond traditional concepts.

Whereas for the traditional disciplines of metrology, a well-proven European structure has been in existence for decades in the form of EURAMET and its technical committees, the necessity of setting up equivalent structures for the new cross-cutting issues arose during the phase of formulation of the European Metrology Research Programme (EMRP) – which was specially designed for metrological issues.

This was realized by the creation of a focus group – “Metrology for Life Sciences” – in which the relevant expert knowledge of the European NMIs was consolidated and roadmaps were developed. Additionally, the need for metrological support in different fields was determined – such as, e.g., in politics, industry, clinical practice and in society as a whole – and taken into account. Thereby, it became clear – unsurprisingly, however – that the need is extremely multifaceted and difficult to classify.

In the first phase of the EMRP, six project proposals were chosen in 2007 by European metrology institutes from the area of health to be funded by the European Commission. Thereby, only those project proposals were selected for which the complementary expert knowledge of several NMIs could be combined in an advantageous way and where it could be expected that – for concrete metrological challenges – important contributions to medical diagnostics, therapy and early diagnosis can be achieved by means of the program from which the patients – in other words: all of us – will benefit.

**Framework conditions and challenges**

In several respects, metrology for health differs from traditional metrological approaches: After all, the measurement object is the human being. Even if – e.g. in clinical chemistry – laboratory measurements are carried out, the results and the thus derived consequences directly affect a human being. Correspondingly, ethical aspects and responsibilities must be taken particularly seriously. Not everything which is measurable must be measured.

Furthermore, the supreme maxim in this field of metrology is not – as opposed to many other fields – precision measurement reaching as far as into metrological border areas. In fact, the – literally “vital” – objective to ensure the comparability of measurements takes priority. In this spirit, often newly developed reference measurement procedures are more relevant than the value of individual physical measurands. Thus, it is inessential, for example, for the electrocardiogram signal (ECG) to determine high-precision electric potential difference and time, but it is essential to analyze the measurement signals in such a way that a comparable result will be achieved as ECG diagnosis.

Fortunately, there is no such thing as a standard human being. Therefore, a “standard” in metrology for the life sciences must take into account the aspects of the enormous biovariability, the non-repeatability and low reproducibility of measurements, imprecise definitions (what is physiological, what is pathological), and other things. Often, there are only indirect, non-invasive measurements possible which are accompanied by many uncontrollable influence quantities and unknown parameters.

In the field of “Metrology for Health”, often the most diverse scientific disciplines are involved which otherwise communicate very rarely with

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each other. This inherent diversity also became blatantly obvious in the EMRP project proposals. Research and development issues opened up, ranging from thermometry, nuclear spin resonance, laser applications, ionizing radiation and ultrasound, to chemistry and biochemistry; from the medical categories of “homecare”, diagnosis, therapy, regenerative medicine and clinical research, to “proteomics”. Among other things, disease syndromes range from cardiovascular problems and cancer, to – in addition – aspects of an ageing society. Issues are: the detection of single molecules; large-scale machines; and virtual human beings.

In contrast to this, the selection of the approved projects is considerably limited and can therefore only be considered as the establishment of first steps for an increasingly wider role of a future European-linked metrology for health. As a logical consequence of the experience gained so far, the European metrology research programme will again launch research and development projects which represent new challenges for the working groups of PTB, for which, in turn, an essential part of the resources will be used.

After a brief outline of the joint projects from the field of health, in which working groups of PTB are also significantly involved, further contributions including typical examples will follow, which give a small – but far from comprehensive – insight into the metrological work of PTB carried out in this field.

Breathing gas analysis as a diagnostic tool for the early diagnosis of diseases

In this project, the technique of laser absorption spectroscopy was examined with a view to its potential for a reliable method of early diagnosis of diverse disease syndromes. Especially the detection limits and measurement uncertainties are to be improved. For the involved NMIs, the main objective is the supply of metrologically well-founded reference measurements. In this way, a metrological basis is to be provided for spectrometrically working breath diagnosis instruments. Sub-topics are: the preparation of reference gas mixtures; line strength measurements; and the examination of the influence of interferences, especially of humidity and CO₂. On the basis of a demanding state-of-the-art measuring set-up, reference points are to be provided which can be used for instruments on a broad scale.

Improvement of the efficacy of cancer treatments by means of 3D brachytherapies

The aims of this project are, on the one hand, to develop new traceability chains to primary standards for the dose absorbed by irradiation sources in brachytherapy and, on the other hand, to elaborate contributions for a better understanding of three-dimensional dose distributions in patients. Ultimately, a relative uncertainty of less than 5 % is to be attained for the determination of the absorbed dose in the target volume in order to be able to ensure the optimum therapeutic effect, on the one hand, and patient safety, on the other hand.

Metrology for cancer radiation therapy

This multisectoral project is characterized by the fact that two completely different radiation therapies correlate metrologically on the basis of ionizing radiation (electrons, protons, heavy ions), on the one hand, and of intensive ultrasound, on the other hand. The attractiveness of this approach is the merging of two metrological cultures which can be mutually beneficial. Whereas for ultrasound therapies, many fundamental questions (e.g. temperature distributions and cavitation aspects) have to be investigated metrologically, tomotherapy by means of ionizing radiation deals with more accurately determining the deposited dose in small and irregularly formed radiation fields, and especially with ensuring the traceability of dosimetry for this application in clinical practice.

Traceability of complex diagnostic biomolecules and biomarkers

To a rapidly increasing extent, diverse biomarkers – which very often are highly complex biomolecules – are used in clinical chemistry. In this field, it is an extremely demanding challenge to ensure the comparability of the results, which is approached methodically in this project. The reference to existing standards often provides insufficient results which are dependent on the methods used. An essential step is the combining of the current WHO system of “international units” with the more fundamental SI system by means of metrologically well-founded, physico-chemical techniques, e.g. mass-spectrometric isotope ratio measurements, for the determination of the units “amount of substance” and “biological activity”.

Traceable measurement results of clinical chemistry for biospecies and ion activity

The quantification of element concentrations in blood serum is one of the most frequent examinations of clinical chemistry. Internationally recognized reference measurement procedures for the traceability of such measurements exist for the determination of the total contents of the most important elements. Additionally, what is
increasingly important is the determination of the amount of substance of the clinically active form of a certain element. Examples to be emphasized are selenium species which improve the effectiveness of cancer treatments in combination with chemotherapy. One target of the project is the quantification of the important species in the very narrow clinically effective concentration ranges.

In a similar way, sometimes only the ionized forms of an analyte are effective. Here, e.g., methods for the traceability of ionized calcium are developed in the project. Especially for critically ill people, the measurement of ionic calcium is of decisive importance.

**Metrology for regenerative medicine**

Great hopes and expectations are currently placed on the development of new treatment possibilities for injuries or for certain diseases by means of artificially manufactured cellular or tissue-like transplants. In spite of considerable research efforts and successes achieved, the potential of these promising approaches for personalized medical care could so far be only implemented to a minor degree and very slowly. This is essentially also due to the fact that there are no internationally accepted metrological procedures, approaches and standards for quality and safety controls of products of regenerative medicine. Hence, the project targets for this subject are aimed at showing exemplary ways of how these gaps can be closed. Focuses of the works include the development of flow-cytometric measurement procedures for the characterization, counting and enrichment of stem cells from peripheral blood as well as the development of quantitative 3D imaging procedures for the characterization and control of surface textures and supporting corsets for the growing of artificial tissue.
Dosimetry for Non-ionizing Radiation

Thomas Kleine-Ostmann

Also non-ionizing electromagnetic radiation whose quantum energy does not suffice to break up chemical bonds in biological molecules can be dangerous for the living organism. Beyond controversy are thermal effects which are based on the fact that the energy of the field is transformed into heat. Furthermore, it is still unclear whether non-ionizing radiation with power densities below the thermal damage threshold can have negative effects. A suitable measure for the strength of the exposition – both for the assessment of personal expositions and for exposition experiments with cell lines, is the specific absorption rate (SAR) that indicates how much power per kilogram is converted into heat.

The metrological detection of this quantity is possible to a limited extent only. To detect the effect of mobile transmitters (e.g. mobile phones), so-called “body phantoms” are used whose absorption behaviour is modelled on the human body and whose liquid content in the interior allows measurement by means of a SAR sensor. Within the scope of the project funded by the EU as part of the programme iMERA-plus “Traceable measurement of field strength and SAR for the Physical Agents Directive”, a procedure was developed at PTB for the calibration of sensors in the frequency range between 6 GHz and 10 GHz (Figure 1), and thus the frequency range was extended in which traceable measurements of this quantity are possible. At high frequencies – above approximately 10 GHz – the measurement of the SAR fails due to the size of the field sensors. In exposition experiments with cell lines – which are often cultivated in the form of very thin layers – the specific absorption rate cannot be measured in this way, not even at lower frequencies. Nevertheless, the specification of the SAR is helpful to quantitatively evaluate, e.g., the field exposition experiments. In this case, the specific absorption rate must be determined numerically by means of field calculation procedures. Within the scope of the project funded by the Bundesamt für Strahlenschutz (Federal Office for Radiation Protection) “Genotoxic effects of THz radiation in vitro”, in which two different skin cell types (HaCaT keratinocytes and primary skin fibroblasts) were exposed to continuous THz radiation at six different frequencies between 100 GHz and 2.52 THz [1], the exact field distribution could be detected in the samples (Figure 2).


Figure 1: Model for the numerical calculation of the SAR value in the calibration apparatus.

Figure 2: Calculation of the specific absorption rate at 106 GHz in a sample container for the field exposition of skin cell lines. The cells are located on a thin plastic foil at the bottom of the sample container and are irradiated from below by means of a plane wave with a power density of 2 mW/cm².
High-intensity Therapeutic Ultrasound: Local Heat for (Tumour) Cell Death

Julian Haller*, Volker Wilkens, Sven Sonntag, Christian Koch

The quantitative description of the ultrasonic fields used for therapeutic purposes is a necessary precondition to estimate the therapeutic effect, to plan the course of the therapy and to assess the health risks for neighbouring healthy tissue. Due to the high powers and temperatures, the extremely rapid heating and the danger of cavitation, non-everyday measurement methods are required for this purpose.

A first precondition for a quantitative estimation is the necessity to determine the total ultrasound power delivered into water, which can amount up to 400 W for clinical instruments. At PTB, a measurement method was developed and realized on the basis of the determination of the acoustic radiation force. By means of this method, almost all currently used sources can be measured. However, for an exact determination of the temperature distribution, knowledge of the spatial acoustic field distribution is required. Sound pressure sensors are required which can cope with the extreme conditions (Figure 1). Alternatively, sensors were developed which are able to measure the heating in a test piece and link it with the acoustic intensity.

The main obstacle for the use of the new technology is the difficulty to get information from the place of treatment about the state of the coagulation. This is why many operations are carried out under control by means of magnetic resonance imaging (MRI), whereby the temperature can be determined online. However, the accuracy is extremely dependent on the instrument and its adjustments. To be able to determine the accuracy of the MRI temperature measurement on site, a phantom was developed (Figure 2) which generates exactly defined temperature fields and heating rates that can be measured and compared.

Figure 1:
The above sensors were newly developed at PTB. They are suitable for spatially resolving measurements of high-intensity ultrasonic fields. (Left) Membrane-type hydrophone for the measurement of sound pressure distributions. (Right) Thermal sensor.

Figure 2:
Phantom for the examination of MRI instruments: a coaxial resistor (black) generates a defined temperature distribution (here: symbolized in colour) in a medium (in this case: water).

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Europe-wide, cancer is the second most common cause of death, and the search for therapies extends over a wide range of physical and chemical methods. Radiation with ionizing rays, currently very often the selected method – leads to an increased radiation burden for the neighbouring healthy tissue. This can be avoided by new methods on the basis of ultrasound which can generate a local heating of the (tumour) tissue far in excess of 60 °C. Thus, the cells die off (coagulation) and are broken down as is the case in apoptosis processes.
Water calorimetry for dosimetry in radiation therapy

Achim Krauss*

The measurand in dosimetry for radiation therapy is the so-called „absorbed dose to water, $D_W$“. At PTB, the unit of this measurand, the gray (Gy; 1 Gy = 1 J/kg), is realized by means of a water calorimeter for $^{60}$Co radiation under reference conditions with a standard measurement uncertainty of 0.2 %. Traceable to the measurements carried out by means of this primary standard, secondary dosemeters (e.g. ionization chambers), which are used in radiotherapeutic hospitals, are calibrated in units of the absorbed dose to water.

Water calorimetry is based on the measurement of the radiation-induced temperature increase $\Delta T$ at a measuring point in a water phantom, whereby $\Delta T$ only amounts to 0.24 mK/Gy. As a precondition for the precise measurement of this temperature increase, a very good thermal insulation and an active temperature stabilization of the whole calorimeter, which is operated at a water temperature of 4 °C, is required. The radiation-induced temperature increase is measured by means of temperature-calibrated thermistors, which are fused into the tip of thin glass pipettes (diameter: approx. 0.5 mm).

As water calorimetry is a measurement procedure which is, as a matter of principle, independent of radiation energy, the energy-dependent correction factors ($k_Q$ factors) of ionization chambers, for example, which are required in the dosimetry of high-energy photon and electron radiation, can be determined experimentally. Furthermore, water calorimetry provides valuable contributions to topical questions in dosimetry, e.g. for dosimetry in small radiation fields which today are used increasingly in radiation therapy.

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To protect the health of persons who are exposed to ionizing radiation at their workplace, the measurement of the dose is prescribed by law. The measuring instruments to be used – the area and personal dosemeters – are subject to the Verification Act at most places of work and therefore require a type testing and approval by PTB. If this type approval, which is only valid at the national level, is in agreement with the international standards, it will also be recognized internationally and will be considered as a proof of quality of the dosemeter. This is ensured through active participation in the international standardization bodies.

For the measurement of the dose, meanwhile electronic dosemeters with a direct display and alarm function are being increasingly used. Most of the approximately 340,000 persons who are exposed to radiation at their workplaces in Germany work in the medical sector, where most of the radiation fields are pulsed. When carrying out measurements in such fields, PTB found out in 2007 that the measurements of electronic dosemeters are reliable only to a limited extent. This is due to the method of measurement applied, which is usually of the “counting” type.

As so far, no tests or requirements have existed for dosemeters – neither nationally nor internationally – it was absolutely necessary for PTB to take action. In the case of the installations in the medical sector, the parameters of the radiation pulses are not adjusted in physical quantities like electricity and electric potential difference, but according to the required image quality. This is why the challenge for the development of the appropriate test rig was to make the physical parameters required for the examination of dosemeters – independently of each other – adjustable. The thus evolved installation is the first and only installation worldwide to permit this.

The second important step is to lay down test requirements for radiation protection dosemeters internationally in IEC standards and the respective reference or test fields in ISO standards. For both, Germany introduced standardization proposals, which were supported by the programme “Innovation with Standards (INS)”.

Figure 1 shows examples for an area and a personal dosemeter, respectively. Figure 2 shows the typical place of use of personal dosemeters in the medical field of X-ray diagnostics. It is stipulated that the dosemeter must be worn under protective clothing, which was therefore represented transparently in the area of the dosemeters.

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Radiobiological Experiments with Living Cells

Ulrich Giesen*

The ion microbeam has been established as a special facility for the research of phenomena in the low-dose range, e.g. the bystander effects [1] and the direct observation of reactions in living cells within seconds after irradiation by means of “Live-cell Imaging”.

At the PTB Ion Accelerator Facility (PIAF), the microbeam system focuses beams of protons and alpha particles down to a diameter of only 2 µm to 3 µm [2]. This allows the selective irradiation of only the nuclei (diameter about 6 µm to 12 µm) or the surrounding cytoplasm of living cells. In addition, the number of particles per cell is precisely controlled by a detection system and the fast deflection of the beam. At a low macroscopic dose, the number of hits for the usual radiation sources varies according to the Poisson distribution, e.g. from 0 to 3 particles per nucleus, whereby the radiation effect is averaged over a large range of damages. By means of microbeam technology, every nucleus can be hit as desired – for example, by exactly one alpha particle.

Important parameters for radiation therapy and radiation protection are the relative biological effectiveness (RBE) and the radiation weighting factor \( w_R \), which vary significantly for different radiation qualities [3]. By means of the microbeam, the effect of densely ionizing alpha particles with a high LET (Linear Energy Transfer) as, e.g., during radon decay, and the effect of sparsely ionizing protons with a low LET as, e.g., for X-rays [4] are examined. Typical biological measurands are the clonal survival or the evaluation of the numbers and patterns of chromosome damages.

In combination with modern methods of molecular biology, the initial reactions of cells to radiation-induced DNA double-strand breaks are currently investigated. In order to make the damages and the processes of repair visible in the cells, a repair protein is marked by genetic engineering by means of a fluorescent agent so that an exclusive observation of this protein is possible. If repair proteins accumulate within seconds or minutes after the irradiation at the double-strand breaks, they can be observed “live” under the microscope as luminous points – so-called “foci” [5]. The procedure of “Live-cell Imaging” is supposed to provide information about radiation damages and the sequence and speed [6] of different repair mechanisms in the cells, and to show how special drugs can influence these processes.

(Acknowledgements and Literature on page 55)
Clinical Ultra-high-field Magnetic Resonance Imaging (UHF-MRI) – A Metrological Challenge

Frank Seifert* 

Magnetic resonance imaging (MRI) is an attractive diagnostic method, as it provides tomographic views of arbitrary orientation of the entire body without ionizing radiation and with an excellent soft tissue contrast. At the beginning of the 1980s, the method found its way into medical routine. Currently, about 70 million MR examinations are carried out per year.

In clinical MR scanners, magnetic fields up to 3 tesla are currently used. However, the trend is moving towards much higher fields of, e.g., 7 tesla, with the promising prospect of opening up new diagnostic possibilities. The propagation phenomena of electromagnetic fields generated thereby in the human body make novel MR imaging procedures necessary, which also involves the development of new metrological concepts for ensuring patient safety.

MRI of the human body is based on the imaging of its water protons. For this purpose, an additional magnetic field is generated by means of a radio-frequency coil. However, in the case of UHF-MRI, the wavelength of this electromagnetic radiation is so short that propagation phenomena lead to inhomogeneously illuminated MRI images. Moreover, the body absorbs the radiation and heats up – an effect which is described by the specific absorption rate (SAR) that must not exceed certain limits for the patient’s safety. The inhomogeneity in the UHF-MRI image can be met with optimal multi-channel excitation methods, whereby multi-channel HF excitation coil arrays are used. However, due to the complexity of the procedure it is clearly more difficult to control the SAR. For this reason, PTB develops novel measurement procedures for the validation of simulation calculations which permit the determination of the complex time-dependent courses of electromagnetic field components in phantoms or in the human body. This will also lay the foundation for future normative regulations in the field of MR safety and extend our knowledge about propagation and impact of electromagnetic fields inside the human body.

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Low-field Magnetic Resonance

Martin Burghoff*

For many years, nuclear resonance measurements have been used successfully in medicine and research. For example, a magnetic resonance scanner can produce very high-resolving anatomic pictures of the human brain. Nuclear magnetic resonance measurements can also be carried out in very low fields. If the used fields approach the order of magnitude of the magnetic field of the Earth – or are even weaker – one should perform the measurements in a magnetic shielding (such as the Berlin Magnetically Shielded Room 2 (BMSR- 2)). For signal recording in the range of very low fields, superconducting magnetic-field detectors (SQUIDs) are used which show the required sensitivity also for resonance frequencies below one kilohertz. For this purpose, a SQUID measuring set-up has been developed in the “Biosignal” Department which enables combined measurements of the $T_1$ and of the $T_2$ relaxation time at low fields of less than 1 µmT [1, 2].

This set-up was used first to investigate the effect of the measuring system on the measured NMR lines. After these metrological effects were understood, the physical background of the NMR signal of liquids was investigated in the low field. As regards water in the neutral pH range, it has been known for a long time that the $T_1$ relaxation time changes in the low field range. This phenomenon reflects the diffusion of $\text{H}_3\text{O}^+$ and OH$^-$ ions which is largely suppressed in the case of extreme pH values. With the new measuring set-up it was possible to investigate the relaxation behaviour of water extensively over a wide frequency range by combined measurement of $T_1$ and $T_2$. These data do not only inform about the molecular dynamics in water, but they also provide background information for the NMR imaging in the low field [3], which is currently being established at PTB and which is mainly based on the NMR signal of water.

A further research objective of low-field magnetic resonance at PTB is the direct measurement of neuronal currents by means of NMR. Thereby, it is intended to use the effect that neuronal currents build up a small magnetic field in their neighbourhood which modifies the NMR spectral lines [4]. This modification is then a measure for the neuronal currents. In combination with low-field NMR imaging, a measurement method would then become accessible which immediately marks the neuronal currents in an anatomic image.

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View inside a magnetically shielded room with a system of Helmholtz coils for magnetic resonance imaging and for measuring neuronal currents in the low magnetic field. The SQUID sensor is arranged at the bottom of the aluminium foil-cladded Dewar vessel.
Continued from page 52

Radiobiological experiments with living cells
Ulrich Giesen

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Literature


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Low-field magnetic resonance
Martin Burghoff

Literature


The research area “Safety and Protection from Risks” is (and will continue to be so in future) one of the most important fields of work in which PTB conducts research, carries out measurements and renders consultancy services to promote consumer protection and to safeguard living conditions. In these fields, special tasks have been assigned to PTB by different national laws and ordinances, for example by the Precautionary Radiation Protection Act, the Radiation Protection Ordinance, the X-ray Decree, the Act on the Proof Testing of Arms and Ammunition and the Explosion Protection Ordinance.

In agreement and in cooperation with the BAM Bundesanstalt für Materialforschung und -prüfung (Federal Institute for Materials Research and Testing), PTB has taken on the fields of “Physics in Safety Engineering” and “Explosion Protection of Electrical Equipment and Apparatus” which are important for the economy and, in particular, for society [1]. These activities serve to prevent damage that might be caused to people, to material or to the environment by technical products, technical installations and technical facilities. Depending on the individual case, explosion protection is ensured by technical solutions and legal provisions. PTB makes its competence available to its customers (federal ministries and ministries of the individual federal states of the Republic of Germany, trade supervisory centres, occupational associations, standardization committees and industry) for the further development of their safety concepts and concepts for explosion protection. This also applies to the work in the fields of “Radiation Protection” and “Precautions against Radiation Damage”.

One of the most important tasks of PTB is the field of “Metrology of Ionizing Radiation”. Activities to be mentioned here are: the measurement of low radioactivities; the measurement of radionuclides in ground-level air; radionuclide analysis carried out on environmental samples, industrial products and industrial waste; and the collaboration in control centres for the surveillance of environmental radioactivity in Germany [2].

The amount of data that is collected in our environment is continuously increasing – not only in the field of measurements, but also when it comes to the control and surveillance of processes and infrastructures. Collecting, summarizing and processing these data further – taking, at the same time, their uncertainty into account – are important tasks to draw the correct conclusions from them.

In the following, you will find several short articles – e.g. on the detection of dangerous substances in cargo, on the use of THz radiation in the field of civil safety, and on data security. These are examples of PTB’s research activities in the field of safety and the protection from risks. They are followed by a detailed report on explosion protection – a subject area which plays an outstanding role in the field of safety and the protection from risk.

Literature


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Due to the increasing danger of terror\(^1\) and, as a result of this, the tightening of the safety regulations of some countries in the international trade of goods, the importance of checking cargo for dangerous substances is constantly increasing. However, the enormous worldwide exchange of goods at increasing speed renders thorough checks, e.g. during the inspection of air passenger baggage, difficult and expensive. Innovative detection procedures which are able to detect dangerous substances fully automatically offer the chance of increasing the safety in the exchange of goods and of meeting the necessary requirements without impeding or delaying the process inadequately.

In cooperation with German and Israeli partners, PTB is investigating – as the project coordinator – a new technique for the automatic detection of explosives and radioactive material in air cargo. Within the scope of the project “Automatic Cargo Container Inspection System (ACCIS)”, which is jointly financed by the research ministries of Germany and Israel, the project partners (i.e. research institutes, industry, and the end users) will investigate components that are essential for an innovative inspection system, and they will test them at laboratory scale.

In many aspects, this new procedure is similar to the conventional X-ray screening of baggage and cargo. The ACCIS project, however, combines the properties of high-resolution neutron resonance and gamma radiography and spectroscopy. The additional value of this combination is that a large number of substances (e.g. commercial and improvised explosives) can be detected due to their composition and that the analysis can, to a large extent, be automated.

Within the scope of this project, not only scientific and technological questions will be examined, but also the conditions for using this technology at airports or at border crossings. Here, especially the legal framework conditions for such an application are investigated. A special question to be examined is whether this technology will be accepted by the public as – due to the use of ionizing radiation – a high level of irritation is anticipated, in particular on the part of the public in Germany.

The aim of the project partners is to make faster and fully automatic inspection systems available in the future, which are able to reliably detect explosives and radioactive substances in cargo. This allows the security standards in air traffic and other means of transportation to be increased and reduces time-consuming manual inspection.

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\(^1\) e.g. parcel bombs in air freight in Nov. 2010 http://www.spiegel.de/thema/paketbom-ber-jemen_2010/archiv-2010309.html
Safe Measurement of Terahertz Radiation

Andreas Steiger*

Terahertz radiation, i.e. electromagnetic radiation with frequencies around 1 THz or wavelengths around 1 mm, lies in the border zone between optics and electronics. On the one hand, it is the highest radio frequency which can be generated electronically, on the other hand, it is the far infrared limit of optical radiation with special characteristics. Due to the low photon energy, the radiation is not ionizing, and many substances in this spectral field change their transmission, reflection and absorption. This allows novel technologies for the security check of passengers and cargo in airports and railway stations to be developed. Examples of current developments for a future, improved “homeland security” are actively imaging THz systems such as body scanners and THz spectrometers for the detection of forbidden dangerous substances. Such systems, which use a THz laser radiation source to illuminate the objects to be investigated, require an accurate power measurement to comply with limiting values for electromagnetic radiation. A reliable THz metrology has, however, not existed so far. To date, PTB is the only metrology institute worldwide which offers the calibration of the power sensitivity of THz detectors at 2.52 THZ with traceability to the International System of Units (SI).

As a reaction to the wishes of the customers regarding this service, PTB is developing – in cooperation with a German manufacturer of energy and power sensors for laser radiation – a terahertz detector which can easily be calibrated. It covers the wide spectral range from far infrared laser radiation down to 1 THz. The cooperation project is thus making an important contribution to closing the metrological gap in the terahertz spectral range.

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First functional model of a terahertz detector which can easily be calibrated with an adapted signal amplifier.

View into the interior of the novel terahertz detector: A compact set-up (protected by PTB patent) which serves as a radiation trap with three sensor elements detects the large reflection losses of a single sensor element which are typical in the terahertz spectral range.
Measurement Data Trustworthy
Only with IT Security

Dieter Richter*

Trustworthy measurement data can only be obtained by carrying out correct and exact measurements. The correctness and accuracy of measurements is of central importance for metrology. Today, however, measurements almost always require information processing and – with increasing frequency – the transmission of data. The question which now arises is: How can the reliability of IT and communication processes be guaranteed? And how is it ensured that electronically issued invoices in commercial transactions contain the correct – i.e. the authentic and unaltered – measured values? Finding answers to these questions is one of the tasks of legal metrology.

Checking an invoice is possible if the consumer can trace his measurement values and the tariffs applicable for invoicing back in a simple way. This is, for example, the case when it comes to classical energy consumption meters in households, and also to the fuelling of cars at petrol stations, or when goods are weighed in shops. What happens, however, if today’s smart meters allow a frequent change of tariffs, or if the electric cars of the future will be able to charge current at different suppliers of electric energy who all have different tariffs (Figure 1)?

PTB’s Department „Metrological Information Technology“ is dealing with these questions. Time and again, this department has to assess whether the latest IT solutions and – in particular – the measures provided for IT security meet the requirements of legal metrology. Besides this, new concepts are being developed in the field of metrology and IT security in cooperation with partners from industry and with experts from other institutes. Here, a main point of research and development is the question as to whether the latest back-up procedures from the field of information technology are compatible with the principles of metrology. Another point of research is tracing back the security requirements coming from the fields of metrology or legal metrology to IT security standards to be able to make use of the potentials of IT security technology in the best possible way (Figure 2). A third focal point is the quality assessment of metrological software. Erroneous software does not only affect the performance characteristics of technical systems, but is also a risk factor for security reasons.

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Figure 1: Charging station for electric cars.

Figure 2: Smart Meter: Symbolic representation of the traceability of metrological requirements to IT security standards.
Explosion Protection at PTB

Michael Beyer¹, Heino Bothe²

1 Introduction

The following article specifically focuses on explosion protection, as this field of work plays an outstanding role within PTB’s work area “Safety and Protection from Risks”.

In everyday life, industry, trade and commerce, flammable substances are often used (for example: fuels required for driving a car, flammable solvents and lacquers in the painting trade, flammable liquids and gases in the chemical and petrochemical industries, and flammable dusts – such as flour – in the food industry). Finely distributed in the air (or in other oxidants), these flammable substances may build up an explosive mixture whose ignition can have fatal or disastrous consequences. This becomes especially obvious when we hear, time and again, of disastrous accidents that have occurred somewhere in the world. Major incidents – such as, e.g., the explosion and serious fire at the fuel depot in Buncefield (UK) in December 2005, the explosion of the refinery in Texas City (in 2005), the explosion of the drilling platform Deepwater Horizon (in 2010), the fire on an ethylene pipeline in Köln-Worringen (in 2008), and disastrous flour-dust explosions in mills, as well as serious accidents in coal mines, time and again draw our attention to this risk and to the importance of preventing explosions by taking technical or organizational protection measures.

In Germany, the first legal regulations were adopted very early – first of all for coal mines, because there, unprotected working equipment all too often ignited “firedamp” below ground. As early as in 1884/1885, the Preußische Schlagwetterkommission (Prussian Firedamp Commission) had the devices which were used underground subjected to an assessment, and in the years after that, suitable protective measures and test procedures were developed. (The same was done in Great Britain.)

At the same time, it was realized that the handling of flammable liquids, too, was a source of danger, and this was countered in 1882 with a decree – the “Ordinance on the Commercial Selling and Offering of Petroleum”. For the normal industrial environment, the Chemisch-Technische Reichsanstalt (Imperial Chemical Technical Institute – CTR) in Berlin took on, in 1920, the task of testing explosion-protected and flame-arresting equipment. After the Second World War, this task passed over to the newly established Physikalisch-Technische Anstalt (Physical Technical Institute – PTA), which was the predecessor of PTB. Later on, these tasks were divided among two institutes: depending on the field of work, either to the BAM Bundesanstalt für Materialforschung und -prüfung (BAM Federal Institute for Materials Research and Testing), which was the successor organization of the CTR, or to PTB. Today, these

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Figure 1: Destruction after a serious fire at the Buncefield fuel depot north of London, 2005. © Chris Radburn/Pool/PA/Reuters/Corbis
tasks are carried out in close cooperation, in a joint field of tasks (“Physical and Chemical Safety Engineering”).

Explosion protection is a sub-field of the field of “Physical and Chemical Safety Engineering” and encompasses the following areas: characteristics of the explosive atmosphere; keeping ignition sources under control; limiting the impacts of an explosion; performance requirements for explosion-protected equipment, protective systems and installations; and operating regulations for the safety of plants and the transport of dangerous goods. The activities of PTB in these subject areas are mainly: the prevention of electrical and mechanical ignition sources, and the handling of flammable liquids.

Whereas PTB especially deals with flammable liquids, the BAM deals with flammable gases and dusts. Besides this, the BAM also deals with safety in the handling of dangerous reactive substances – such as, for example, gases, solids and explosive substances and objects (e.g. pyrotechnics) – as well as enclosures and technology for the transport and storage of dangerous substances and goods.

This division of tasks, which has existed for decades, has proven its worth. In 2005, a joint steering committee for the field of physical and chemical safety engineering was established whose task it is to coordinate the activities in this field.

The safety assessment of explosion hazards takes place in a 3-step process. The aim of the first step is to prevent the occurrence of explosive atmospheres or explosive mixtures. If this step succeeds – for example, because non-flammable substitute materials are used, or because processes are applied in which the explosion limits are not exceeded – it is not necessary to take any additional explosion protection measures. If explosive atmospheres cannot be avoided, the main priority is to keep the ignition sources under control. If it is, however, not possible to prevent ignition sources altogether, the impact of an explosion must, at least, be limited to an acceptable degree. All steps of this process are part of PTB’s spectrum of tasks, i.e. of the four PTB departments dealing with explosion protection (these being: Fundamentals of Explosion Protection, Flame Transmission Processes, Intrinsic Safety and Safety of Systems, Prevention of Ignition Sources).

2 Testing, certifying and monitoring – from national monopolist to global player

European harmonization started in the field of explosion protection much earlier than in all the other areas regulated by law. This field of work, which dealt with electrical equipment, had so far been regulated by national law. In 1976, it was the first field of work which was placed on a European basis, i.e. by means of the European Framework Directive 76/117/EEC. In addition, the respective European standards for conformity were laid down in the “Old Approach Directives” (79/196/EEC and foll.). At the beginning, particularly sensitive areas were excluded from these directives and were subjected to national type approval, with PTB as the test centre.

When the “New Approach” Directive 94/9/EC ("ATEX") came into force, this meant that all the requirements for placing autonomous protective systems and explosion-protected electrical and non-electrical equipment on the market were finally the same everywhere in Europe. This directive contains conformity assessment procedures which often require the involvement of a “Notified Body” – both for testing the products and for production control at the manufacturers. With this Directive, the Ordinance on Flammable Liquids (VbF) became void and PTB’s legal monopoly position for the areas of zone 0 came to an end in Germany. From then on, a series of "Notified Bodies" was established in Europe. PTB was one of the first bodies of this type. PTB is notified, among other things, for the assessment of explosion-protected equipment and autonomous protective systems (in the form of flame arresters) and for checking the quality management systems of manufacturers for compliance with the Directive. One of the tasks of PTB is also to determine the safety characteristics of flammable liquids. These are needed as a basis for explosion protection measures and for the transport of dangerous goods.

To be able to participate in world trade, the German manufacturers must be able to submit internationally recognized certificates. Such certificates are provided by the international IECEx system. PTB was a major force in establishing and developing the IECEx system. Furthermore, there is a partnership with the American test organization Underwriters Laboratories Inc. (UL) which offers – in addition to assessments which are in compliance with European requirements – also assessments which are in compliance with US requirements. This is of great help when it comes to activities on the North American market. In addition, further national (with VDE) and international collaborations ensure that the certificates of PTB are accepted in important industrial regions.

If conformity assessments are to be recognized worldwide, all test centres have to be equally qualified and have to apply equal assessment criteria.

To ensure this, a worldwide programme of comparison measurements has, for the first time in this field, been launched under the leadership of PTB. The aim of this programme is to compare typical test procedures for explosion-protected equipment. Meanwhile, the most important test centres of the IECEx system are taking part in this programme.
3 Transfer of knowledge to industry and society

PTB is one of the few test centres that carry out investigations and conducts research in all areas of the 3-step process of explosion protection. PTB's tasks are to investigate (i) the properties of an explosive atmosphere; (ii) electrical and non-electrical ignition sources; and (iii) the development of explosion and detonation processes. The aim of PTB's work is to conduct fundamental research on ignition processes; prenormative research for standards or regulations; and to provide a basis for technical developments. With this knowledge (especially in innovative fields of work), PTB is able to offer testing and consultancy services to the manufacturers of explosion-protected equipment and of protective systems and to the operators of plants in potentially explosive atmospheres.

3.1 Safety characteristics – a prerequisite for assessing explosion risks

In order to be able to assess explosion risks, the safety characteristics of flammable substances must be known. PTB determines these characteristics for flammable liquids. On the one hand, these characteristics are needed in order to assess whether an explosive atmosphere has built up (this assessment is possible, for example, by means of the safety characteristics “explosion limits”, “limiting oxygen concentration”, “explosion point” or “flash point”) or whether the flammable substance may be ignited (this can be found out, for example, via the “ignition temperature” (see Figure 2) or via the “minimum ignition energy”). On the other hand, these characteristics are needed to assess the impact of an explosion (this assessment is possible, for example, via the “maximum explosion pressure” and via the “maximum explosion pressure rise”). The influence of special conditions on the combustion reaction (such as high or low pressures and temperatures, or special oxidants) is investigated to allow safety statements on the current developments in process engineering to be made. The characteristics are gathered in the CHEMSAFE database which is jointly operated by the BAM, PTB and DECHEMA. The most essential feature of this database is the fact that an assessment process is carried out before the substance data are entered in the database. In this way, the information stored in this database is particularly reliable.

3.2 Prevention of an explosive atmosphere

To prevent the building up of an explosive atmosphere, PTB provides the required safety characteristics for the assessment of this measure. Furthermore, it investigates, either by experiment...
or by computation, exemplary situations which occur in reality in plants or facilities (Figure 3). In a generalized way, these then provide the basis for the determination of zones in the explosion-protection documents of plant operators or for regulations of the State or of Social Accident Insurance Institutions.

3.3 Prevention of ignition sources

In potentially explosive atmospheres, different ignition sources must be taken into account (e.g., hot surfaces, electrostatic discharges, electric and mechanical sparks, optical radiation or ultrasound). PTB deals with these ignition sources in research, tests and practical assessments.

The typical strategy for preventing ignition sources on explosion-protected equipment is to apply standardized “types of protection”. Although these are different for electrical and non-electrical equipment, they are based on the same model. The stored (electric) energy can be limited to such an extent that it is too low to ignite the explosive mixture (“intrinsic safety”), or the ignition source is prevented by an adequate construction (“increased safety” or “constructional safety”). If these measures are not successful, access of the Ex atmosphere to the ignition source can also be prevented by encapsulation (as in the case of the “pressurized apparatus”, “oil or liquid immersion”, “encapsulation” or “restricted breathing”), by nipping the ignition process in the bud (“powder filling”) or by safely enclosing a potential explosion (“flameproof enclosure”).

Due to the abundance and variety of electric equipment, it is the electric ignition sources which are the most frequent type of ignition source. Here, PTB contributes, with its research, essentially to the fundamentals of the above-mentioned types of protection, in particular for “intrinsic safety”, “increased safety” and “flameproof enclosure”. Here, the main priority is to adapt the explosion-protection concepts to new developments – e.g. to new measuring and control techniques, to new light sources and to the drive technology.

In mechanical equipment and components such as, e.g., gears, pumps, stirrers or dynamic seals, ignition sources may occur from technical defects or simply in normal operation as a result of impacts or of continuous friction. These ignition sources are either particles of high temperature which have been separated (e.g. mechanical sparks) or, in the case of friction processes, hot surfaces. In this regard, PTB and the BAM work closely together in the fields of research and testing of mechanical explosion-protected equipment.

PTB’s field of tasks also comprises special ignition sources such as, e.g., electrostatic discharges, optical radiation or ultrasound.

PTB employees are decisively involved in the development of novel technical solutions for the prevention of ignition sources (e.g. the development for an intrinsically safe energy supply concept with high electric power (Power-i/DART); fuel cells for use in explosive atmospheres; converter-fed explosion-protected motors; and novel sinter materials for flame arresters and pressure relief devices on flameproof enclosures).

3.4 Limiting explosion impacts

If ignition sources cannot be prevented altogether, explosions must be kept under control in another way. It is, for example, possible to limit the impacts of an explosion to an acceptable extent if the explosion is safely enclosed (by an explosion-pressure-resistant construction or by a flameproof enclosure) or if it is stopped (by flame arresters or explosion suppression) or if the impact of the pressure is attenuated (by explosion-pressure relief).

Here, PTB’s work is focussed on the prevention of flame transmission. Such a prevention is, for example, guaranteed by flame arresters. The prevention of flame transmission is also – besides explosion pressure resistance – a requirement for the flameproof enclosure. Apart from fundamental research, PTB also conducts other research work in this field – for example, with regard to the suitability of new materials for flame arresting elements (Figure 5) and pressure relief devices on flameproof enclosures, or with regard to the suitability of the – very small – internal dimensions of micro structured equipment as flame arresting components.
3.5 Rendering consultancy to policy makers; standardization; supporting industry

The knowledge and experience gained in research, development and testing provide the basis for the consultancy services which employees of PTB render to those groups dealing with explosion protection. Being a departmental research institution of the Federal Government, this is an important task of PTB (in cooperation with the BAM) so that it fulfils its function as a connecting link between society and the economy. This also encompasses the rendering of consultancy to the Federal Government (in the safety committees of different federal ministries, e.g. for the transport of dangerous goods (BMVBS), for safety at work (BMAS) and for the safety of plants (BMU)). Representatives of PTB participate in committees of the Social Accident Insurance Institutions and, in addition, render advisory services to the health and safety as well as the market surveillance authorities of the federal states and to other authorities, and in addition, also to industry and to the manufacturers of explosion-protected equipment and to the operators of hazardous plants.

For these industries, European and international standardization is of increasing importance as German industry depends on the worldwide trade with equipment, chemical products and engineering services. Here, harmonized European standards which give rise to the presumption of conformity with respect to the safety requirements of the European Directive 94/9/EC (ATEX) are particularly important for explosion protection. For electric explosion protection, these standards are meanwhile being elaborated almost exclusively at the international level (in the IEC/TC 31 “Equipment for explosive atmospheres”) and then adopted as harmonized European standards. For the standards of the CEN/TC 305 for non-electrical equipment, protective systems and safety characteristics (which have so far been developed exclusively at the European level), this process is just taking place in the IEC/SC 31M “Non-electrical equipment and protective systems for explosive atmospheres”.

Employees of the four PTB explosion protection departments work at all levels of this standardization process, in technical and managerial capacities. They represent safety and technological principles which have been developed in Germany and Europe over many decades and thus also support the many SMEs which are not able to participate in international standardization themselves.

The successful technology transfer achieved by PTB in the past few years has been acknowledged by high decorations in the fields of research, technical development and standardization.
Flame Propagating Safety in Micro-process Engineering

Elisabeth Brandes*

In the chemical industry, there is an increasing use of micro-processing technology. Thanks to this technology, reactions for which substances with a high oxidation potential (oxygen, dinitrogen monoxide, etc.) are used, can be better mastered.

However, if the desired reactions occur at increased temperatures and at increased pressures, and if oxidants are used which are more reactive, then devices based on micro-processing technology are – despite their small internal dimensions – not intrinsically safe (investigations of the BAM). To prevent the propagation of a flame front in equipment of that type, the characteristic properties of flame transmission must be known.

To investigate transmission processes in micro-structured components, two approaches have been chosen: detonative flame transmission through single capillary tubes (special steel or glass) with typical diameters was observed by measurement or directly (Figure 1); alternatively, characteristics such as, e.g., the detonation speed, the detonation pressure and the detonation cell width in pipes with larger diameters (10 mm) were determined in order to draw conclusions from this to the behaviour in capillary tubes. The equipment is suitable for temperatures up to 150 °C, initial pressures up to 20 bar and for oxidants with a high oxidation potential.

The investigations carried out on different hydrocarbon/oxygen mixtures confirm that capillary tubes with very small pipe diameters – as are used in micro-reaction technology – are not at all intrinsically safe against the transmission of a detonation. Although the propagation speeds (Figure 2) in these capillary tubes are clearly reduced, they lie unequivocally in the detonative range.

The “λ/3 rule” – according to which the tube/capillary diameter must amount to at least 1/3 of the detonation cell width if flame propagation is to be successful – was confirmed. The risk of a flame propagation due to micro-structured components can, thus, be assessed by determining the width of the detonation cells applying the λ/3 rule, or by direct observation of the propagation process.

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Figure 1:
Draft of the experimental set-up for the optical determination of detonations.

Figure 2:
Propagation of an ethane/O₂ detonation at 1 bar through a glass capillary tubing with a diameter of 0.5 mm. (10 displaced single images (Δ: 37 μs)).
Ignition Hazard Assessment of PEM Fuel Cells with Respect to Internal Explosive Combustion Reactions

Thomas Horn*  

To develop an explosion protection concept for a polymer electrolyte membrane fuel cell (PEM-FC), a detailed analysis of the potential ignition hazards is necessary.

During fuel cell operation, the process gases – hydrogen (H₂) and oxygen of the air – are separated. Aging of the materials used can, however, lead to internal leakages. If an effective ignition source occurs, internal explosive combustion reactions are possible (cf. Figure 1).

The impacts of an internal explosion again represent an effective ignition source for a potentially explosive atmosphere around the fuel cell. For this reason, the ignitability of an H₂/air mixture inside the fuel cell was experimentally investigated. In fuel cell test arrangements, a fuel gas transfer was simulated. In the ignition tests, explosive combustion reactions during the operation of the fuel cell could be detected only in the case of a forced ignition close to the gas inlet. The cause is the humidity and nitrogen fraction of the H₂/air mixture which is converted directly exothermally at the catalyst layer and which increases with increasing path length. In the direction of the gas outlet, this, consequently, led to an increasing inertization of the mixture [1].

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Ignition through Electric Discharges

Detlev Markus*

In many fields of daily life, electric energy is of essential importance. But in spite of its enormous benefits, also its dangers must be taken into account, for example when being used in an explosive atmosphere. To assess the probability of an explosive atmosphere igniting through an electric ignition source, several concepts have been elaborated. If one wants to use models for that purpose, it must be taken into account that an ignition depends on such a large number of chemical and physical single processes interacting with each other that its assessment on the basis of computational models alone is not yet possible. New electric energy supply concepts such as Power-i/DART or the use of converter-fed drives in potentially explosive atmospheres require, however, detailed knowledge of the respective ignition processes. Researchers of PTB are therefore investigating – by means of numerical simulation and modern laser-optical procedures – the transition from the coupling of electric energy by electric gas discharges to the self-sustaining flame propagation of an explosion. In this field, PTB closely cooperates with different institutions at home and abroad within the scope of joint research projects.

The aim of this research work is to allow – in the long term – an assessment of the ignition probability for different types of electric discharges. The research field therefore encompasses different forms of electric discharge. Partial discharges have, for example, for a long time been regarded as insignificant for potential ignition processes. In this case, the gas discharge stops before the thermalization between the gas molecules and the electrons in the discharge section is terminated. Therefore, the electric energy is largely converted in the form of kinetic energy of electrons. Besides the heating of the gas volume, the generation of different reactive species by dissociation of gas molecules due to impacts with high-energy electrons is, therefore, also of significance. If high-frequency AC voltage is used, an accumulation of this very effective energy coupling may occur over many oscillation cycles, which may finally cause the ignition of a potentially explosive atmosphere. Therefore, also partial discharge due to high-frequency compensation processes in electric mains power supplies must be taken into account for the safety assessment of potentially explosive atmospheres.

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Figure 1: Electric discharge forms: a) brush discharge, b) spark discharge, c) propagating brush discharge.

Figure 2: Chemoluminescence of partial discharges.

Figure 3: Ignition of a hydrogen/air mixture by partial discharges.