The Reference

Switzerland's metrology magazine

No 01 | 2023

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10th anniversary of being a federal institute

METAS

Editorial

Dear Reader,

It's finally here: the first edition of "The Reference.", Switzerland's metrology magazine, has arrived. Formerly known as METinfo, "The Reference." is METAS' revamped journal for metrology and returns with both a new name and a brand-new look. All of the changes we've made are designed to support the same goal – to inform the reader about developments in the wider field of metrology, ranging from the scientific (results from research and development) and industrial (services and courses) to legal metrology.

Legal notice

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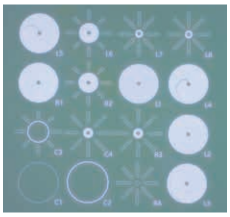
METAS employees in the analytical laboratory.

Content



Whether you prefer having the physical journal in your hands or the PDF version on your device, I invite you to give "The Reference." a quick read to find out about the latest changes and innovations at METAS. A good ten years ago, METAS was transformed from a federal office into a federal institute. We look back on a decade of METAS as an institute on page 18.

Climate change is also creating new challenges for metrology. Scientists, politicians and administrators alike need reliable and comparable data. This includes information about the presence and distribution of environmentally hazardous gases such as halogenated volatile organic substances. METAS has developed traceable reference gases for such substances. The World Meteorological Organization (WMO) recently recognised METAS' Gas Analysis Laboratory as the global reference laboratory for calibrating these gases, which you can read about on page 8.

These are just two examples of the varied content you'll find in this edition. I hope that you find "The Reference." both visually appealing and informative. I look forward to receiving your feedback.

Dr. Philippe Richard
Director
Federal Institute of Metrology METAS

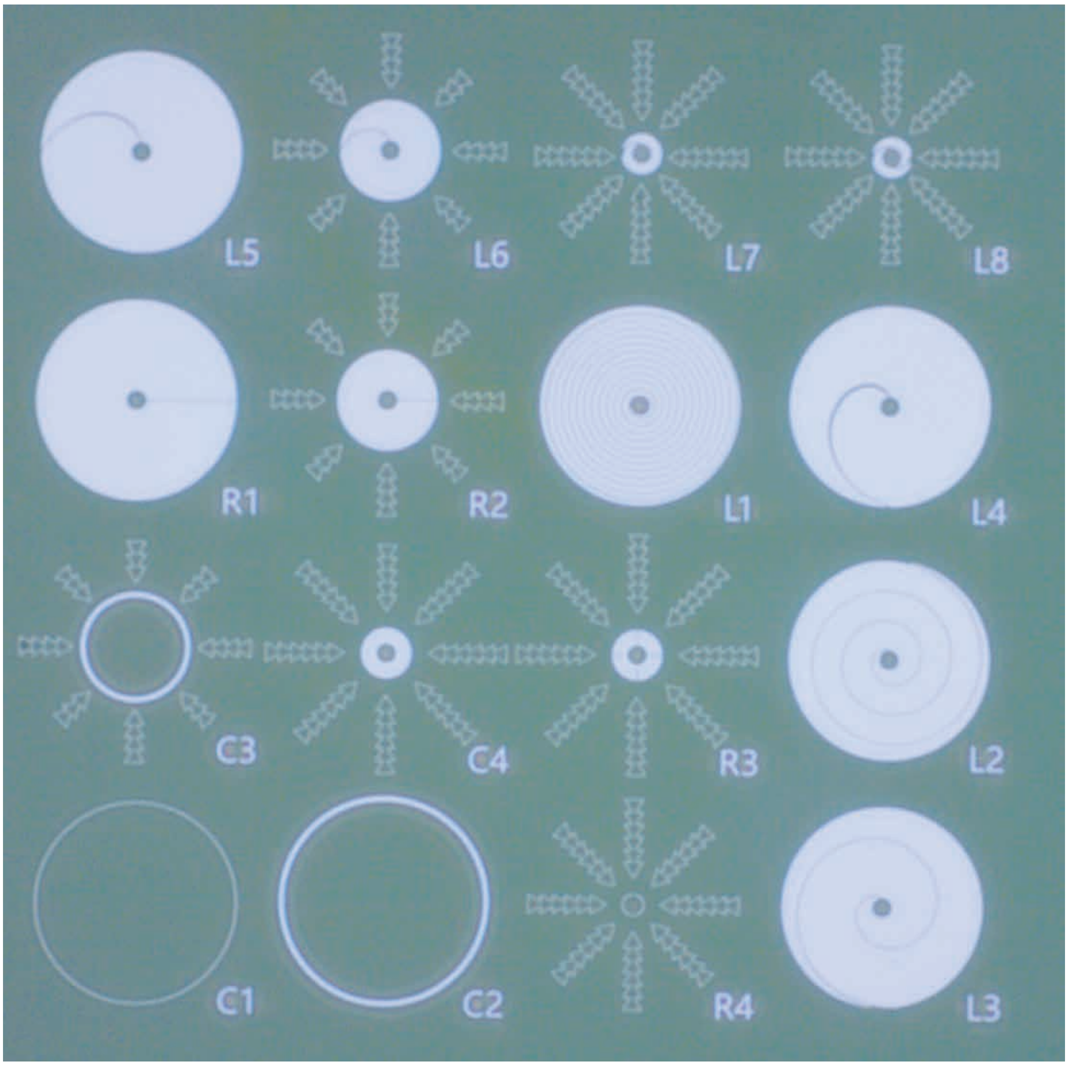


Figure 1: Standards for coaxial SMM tips on a silicon oxide membrane (white) which is mostly coated with gold (green). The labels L, R, C designate inductors, resistors and capacitors.

Research and development

Nanoelectric metrology for industry

From washing machines to quantum computers – today, almost all electronic devices contain circuits based on semiconductors. These integrated circuits in turn contain very small, nanoscale electrical components. In order to measure such components or more general materials spatially resolved, measurement systems with very fine probes are required.

Dr. Johannes Hoffmann, Sophie de Préville, Bruno Eckmann, Dr. Hung-Ju Lin, Dr. Markus Zeier

The ELENA project (ELEctrical NAnoscale metrology in industry) comes into play here and aims to make nanoscale electrical measurements simpler and more reliable. One of the goals is to ensure traceability of the results to SI units (reliability) while the other is the use in industry (simplicity). Here, two different types of scanning probe microscopy are used: the "Conductive Atomic Force Microscope" (C-AFM), which, in addition to topology, measures electrical values at direct current or in the low-frequency range, and the "Scanning Microwave Microscope" (SMM), which works with high-frequency signals in the gigahertz range. The work at METAS is on the SMM and is subdivided into the following activities: design of two new reference substrates for calibration with coaxial probes, design of cost-effective electronics for SMM measurements and set-up of a measuring station for customer measurements.

The Scanning Microwave Microscope with coaxially shielded tip

An SMM mainly consists of an atomic force microscope (AFM) with a conductive probe. The latter is coupled with a measurement electronic which transmits high-frequency signals to the tip and receives the signals reflected from there. The ratio between the reflected and the transmitted signal is called the reflection coefficient. Reflection amplitude and angle depend on the material characteristics of the substrate under the tip. This can be used to calculate values such as conductivity, permittivity, resistance, inductance, capacity and doping density. In addition, the doping type of semiconductors can be determined by superimposing a low-frequency signal. However, to feed in the low-frequency signal, the high-frequency structure of the SMM must be slightly modified.

The commercially available SMM systems all contain tips, which are only partially shielded electrically. The result of this is that structures present in the vicinity of the actual measuring point will have an interfering effect on the measuring result. The reason for this is the electrodynamic coupling between these structures and the exposed tip. If geometries and material characteristics are known, this coupling can be eliminated from the result mathematically. However, if the measurement object needs to be changed, for example, the change from a reference substrate to the actual test specimen, this influence can no longer be corrected mathematically. This type of change alters the measured signals of the SMM so profoundly that the device can only be calibrated if the calibration standards are located on the same substrate as the measurement object. In order to overcome these problems, METAS already developed a coaxially shielded tip in the past.[1]

Two new reference substrates

However, the existing reference substrates for SMM, such as the reference substrates described in [2], are suitable for non-coaxial tips in the first instance. Due to its structure, the contact surface of a coaxially shielded measuring tip is larger than the contact surface of an unshielded tip. This can lead to a short circuit during measurement. To avoid this, METAS

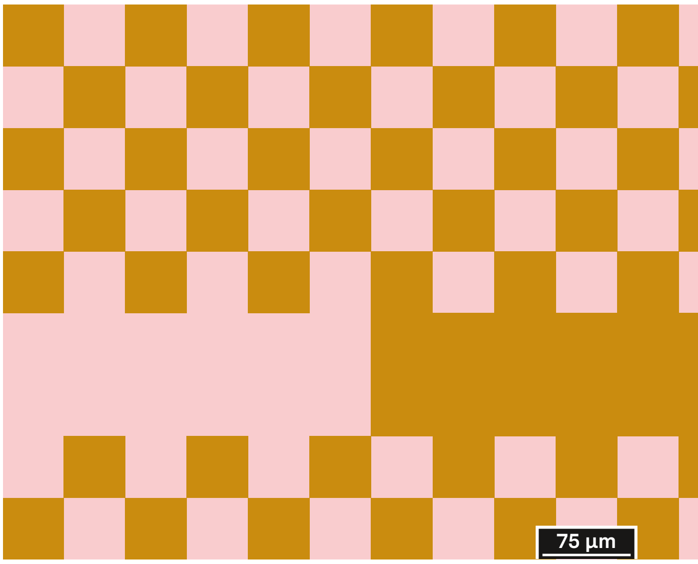


Figure 2: Chequered pattern of ITO (pink) on silicon oxide (brown). This structure is available in various different thicknesses and is used as a reference substrate for calibrating the coaxial tips of the SMM. The colours of the ITO and silicon dioxide in this image are caused by interference and background material. ITO and silicon dioxide are transparent.

Figure 3: Measurement electronics for SMM measurements. The measurement electronics consists of a “Software Defined Radio”, a “Single Board Computer”, a low-frequency signal generator and a high-frequency network which separates the wanted signals of the SMM from the background signals. The measuring tip of the atomic force microscope is connected to the plug on the front panel with a coaxial cable.

designed two new calibration substrates for coaxial tips. The first substrate is a modification of a substrate already known from the ADVENT project.[1] It consists of a silicon wafer which contains on the surface several membranes made from silicon oxide. A gold coating is applied to the membranes and different patterns are etched into the gold (see Figure 1). Different impedances are created through the geometry of these patterns: capacitors (circular area with insulation ring to outer ground plane), resistors (centre point with bridges to outer ground plane) and inductances (centre point with spiral to outer ground plane). These standards are suitable for larger coaxial tips (outside diameters approx. 20 µm).

A second type of reference substrate for coaxial tips is based on structures with “Indium Tin Oxide” (ITO). This is a relatively weakly conductive transparent oxide, which is also used in most touchscreens (such as in a mobile phone) as an electrode. The standards consist of a chequered pattern of layers of varying thickness of ITO, which are applied on different silicon wafers coated with silicon oxide (see Figure 2). Arbitrarily fine coaxial tips can thus be calibrated.

The two newly developed reference substrates have different characteristics: The membrane-based standards offer the best possible calibration accuracy for all possible measurement objects but are restricted to large coaxial tips. The ITO-based standards offer good calibration options for resistance-like measurement objects but present increased measurement uncertainties for capacitive or inductive measurement objects. However, the ITO standards are suitable for arbitrarily fine coaxial tips.

Development of more cost-effective electronic measuring equipment for industry

A vector network analyser (VNA) is normally used as the measurement electronic for SMM measurements. However, this represents a considerable cost factor, which has led to a very restrained use of SMM metrology in industry. Together with an industry partner, METAS has designed electronic measuring equipment which is specifically geared towards the requirements of the SMM and is considerably more cost-effective than a VNA. This enables cost-effective access to SMM measurements. The electronics are based on a "Software Defined Radio" (SDR), which, in simplified terms, is a generic high-frequency transmitter and receiver module with direct and very broadband digitisation of the analogue signal. This in turn requires a well-designed digital signal processing. Accordingly, a significant portion of the effort goes into programming the electronics. Two systems must be programmed for this purpose: first of all the field programmable gate array, which contains the filters and controls the high-frequency component of the circuit; and secondly the interface between the PC and the measuring instrument. METAS also developed a high-frequency network for signal routing and signal separation. With this network, the signals are conducted with as little loss as possible to the measuring tip of the instrument, and the reflected signals are isolated from background influences. This is necessary as the wanted signals are very small and require a high level of amplification. If the wanted signals are not well separated from the background signal, the resulting signal is not easy to amplify and it is difficult to draw conclusions from the measurement data.

In order to set up a measurement service with the SMM, the necessary instructions, measurement routines, uncertainty budgets and quality documents according to ISO 17025 are now being developed in the next step as part of the ELENA project. A measurement comparison will then be made between the National Metrology Institute of France *("Laboratoire national de métrologie et d'essais", LNE)* and METAS, to practically test the uncertainty budgets and measurement routines as well as to ensure comparability. Once all discrepancies have been resolved, the new measuring service can be entered in the "Key Comparison Database" (KCDB)[3] of the *"Bureau International des Poids et Mesures" (BIPM).* This gives international recognition to calibration certificates with SMM results. ●

This project is funded by the EMPIR Grant "20IND12 Elena" of the EU.

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[1] Hoffmann et al, METinfo Vol. 28, No. 1/2021

[2] LeQuang et al, Rev. Sci. Instrum. 92, 023705 (2021)

[3] KCDB: Key Comparison Database, bipm.org/kcdb

Reference material

METAS – the worldwide reference laboratory for halogenated VOCs

Since this summer, METAS' gas analysis laboratory has been the global reference laboratory for ten halogenated volatile organic compounds in the atmosphere. METAS is thus contributing to stable and comparable climate data in the long term.

Dr. Tobias Bühlmann, Céline Pascale

Well over 30 different halogenated volatile organic compounds, known as halogenated VOCs, can be found in the atmosphere. These substances have names which are barely pronounceable, such as HFO-1336mzzZ, HFC-365mfc and 1,2-dichloroethane. They come from cooling systems, refrigerators, propellant gases for aerosol cans or have been used as solvents. They are released into the atmosphere through leaks or improper handling. The halogenated VOCs include the chlorofluorocarbons, known as CFCs, which caused the ozone hole and were therefore banned under the Montreal Protocol. The CFCs were subsequently replaced by hydrochlorofluorocarbons and chlorine-free alternatives which, however, turned out to be potent greenhouse gases and were therefore likewise regulated by the Montreal and Kyoto Protocols. The halogenated VOCs contribute significantly to global warming. In 2019, they accounted for around 15 % of the total anthropogenic radiative forcing. The latest generation of halogenated VOCs, the

so-called hydrofluoroolefins (HFOs), are not potent greenhouse gases. However, it is assumed that their degradation products have detrimental effects on the environment. This highlights the importance of being able to measure all the halogenated VOCs present in the atmosphere on a global, comparable and accurate basis.

Comparable and accurate measurement of halogenated VOCs

Whether the measures implemented for reducing emissions are actually having an impact can only be verified by means of comparable and accurate measurements. However, such measurements are not easy, as these substances are found in the atmosphere in very low proportions. For example, the refrigerant HFO-1336mzzZ occurs less than once for every billion molecules in the atmosphere. It is therefore not surprising that only highly specialised institutions can carry out such measurements at all. These institutions include the "Global Atmosphere Watch" measurement stations (GAW) of the World Meteorological Organization (WMO) as well as the two networks "Advanced Global Atmospheric Gases Experiment" (AGAGE) and "National Oceanic and Atmospheric Administration" (NOAA).

However, it is not sufficient to simply measure, as the measuring instruments must be calibrated with high-precision SI-traceable reference gases. And that is exactly where METAS comes in, when it comes to SI-traceable reference gases. SI-traceable means that a measured value is related through an uninterrupted chain of calibrations to a recognised standard – in this case to the International System of Units (SI). This enables measurements to be compared over long periods of time, regardless of location and measuring method, which is particularly helpful for scientists and decision makers from the areas of politics and the administration.

Concentration unit of the halogenated VOC meter.

SI-traceable reference gas mixtures for halogenated VOCs

The European Association of National Metrology Institutes EURAMET recorded in its roadmap ten years ago that its members should occupy and maintain a niche in their area of expertise to keep the quality as high as possible and to prevent unnecessary duplication of effort. METAS' gas analysis laboratory already had many years of experience back then with the dynamic production of reference gases in the field of air pollutants. At this time, the Swiss Federal Laboratories for Materials Science and Technology EMPA, which operates measuring stations for halogenated VOCs in the atmosphere, announced the need for new reference gases. For certain halogenated VOCs, there were no references, while for others, there were non-SI-traceable references, some of which did not correspond with each other. The need for SI-traceable reference gas mixtures for halogenated VOCs was thus present and provided the perfect niche for METAS. All the more so because the required expertise was available and no other national metrology institute was working in this area. The gas analysis laboratory therefore started to expand the existing infrastructure as part of an internal project in 2014, to be able to produce reference gas mixtures for halogenated VOCs. As these substances are found in very low proportions in the atmosphere and must be present in similar portions in the reference gas mixture, the production of these types of reference gas mixtures requires special equipment.

Dynamic manufacturing process

The manufacturing process for SI-traceable reference gases starts with what is known as a permeator. This is a small tube with a membrane filled with a pure halogenated VOC. For example, if a reference gas for HFO-1336mzzZ is to be manufactured, then this tube is filled with exactly this substance. At a constant temperature and with constant pressure, the same amount of the pure halogenated VOC – in this case HFO-1336mzzZ – will always be released from the permeator. A magnetic suspension balance measures how much of this substance is released from the tube over time. Because when the substance is released from the tube, it becomes lighter. However, as this mass loss is only between 100 and 500 nanograms per minute, such measurements last several days until ultimately a stable and conclusive measured value of the mass loss is present. During the measurement, the permeator hangs in a permeation chamber, through which a defined quantity of gas is conducted. The proportion of the halogenated VOC in the reference gas is calculated from the mass loss of the permeator and this gas flow. This portion is still considerably above that in the atmosphere, which is why the reference gas needs to be further diluted over another two stages.

The smallest impurities must be identified

Impurities in the reference gas and losses must be prevented at all costs, as the smallest loss or the smallest impurity make the reference gas mixture unusable. METAS' instruments are therefore equipped with a special coating which prevents molecules of the reference gas from remaining stuck on the surfaces of the instruments and thus being lost. In addition, all the equipment was examined for potential contamination. A special measuring instrument is required for such measurements so that even the smallest traces of these substances can be measured. The Swiss Federal Laboratories for Materials Science and Technology EMPA has constructed a device for METAS, which concentrates the substances to be measured from up to six litres of air and then measures them using a gas chromatograph and a mass spectrometer. Thanks to this device, even the smallest impurities can be detected.

Filling process at –196°C

To store and transport the dynamically produced SI-traceable reference gas mixture, it is filled in stainless steel cylinders. However, this is not easy, as between two and five litres of gas per minute flow unpressurised out of the production facility. To fill these reference gases into cylinders, a “cryo filling system” was developed at METAS specially for this application and has been constantly improved. The “cryo filling system” functions according to the principle of the “cold trap”: the cylinder to be filled is placed in a vessel filled with liquid nitrogen. At –196°C, liquid nitrogen is extremely cold and liquefies the gas that flows into the cold cylinder. Thanks to the “cryo filling system”, the amount of gas filled in the cylinder can be controlled very precisely. After filling, the cylinder needs to be defrosted. Subsequently, the quality of the SI-traceable reference gas is checked and then the reference gas mixture is ready for calibrating measuring instruments.

Reference gas mixtures

As part of the project “HIGHGAS: Metrology for High Impact GreenHouse GASes”, which was implemented during the “European Metrology Research Programme” from 2014, METAS developed a set of SI-traceable reference gas mixtures for five halogenated substances. The two halogenated VOCs HFO-1234yf and CFC-13 from this set are now used by the AGAGE network for their calibrations. This was a major success for METAS and garnered much international recognition. A further set of SI-traceable reference gas mixtures for six substances has been developed in an internal METAS follow-up project as well as within the framework of the project “MetClimVOC: Metrology for climate relevant volatile organic compounds”. The latter was carried out on the occasion of the “European Metrology Programme for Innovation and Research”. Thanks to this new reference gas set, EMPA was able to quantify HFO-1336mzzZ in the atmosphere as the first and so far only institute. In addition, AGAGE uses this reference gas mixture to measure 1,2-dichloroethane in the atmosphere worldwide on a comparable basis – another success for METAS.


The shimmering colour coating prevents particles of the reference gas from sticking to surfaces.

METAS is a “Central Calibration Laboratory”

The programme “Global Atmospheric Watch” (GAW) of the “World Meteorological Organization” (WMO) is a partnership of more than 100 countries, which pursues the goal of monitoring the chemical composition of the atmosphere with a global network of measurement stations in the long term on a comparable basis. In order for this to work, high-quality references are needed. For certain substances, one specialised institution takes over the task as “Central Calibration Laboratory” within GAW. This means that this institution produces these references and makes them available to the GAW measurement stations. The Dutch Metrological Institute, for example, has taken over this task for nitrogen oxides, and the American counterpart has taken it over for ozone. Up to now, there has been no “Central Calibration Laboratory” for halogenated VOCs. After almost a decade of experience in this field, it was thus an obvious choice for the METAS gas analysis laboratory to apply for this function to the GAW programme – and it was successful. Since June 2023, METAS has held the function of “Central Calibration Laboratory” for a total of ten halogenated VOCs. This task is financed by MeteoSwiss, which coordinates the tasks of the GAW in Switzerland. With this important task, METAS laid the foundation for enabling measurement of these substances within the GAW network worldwide on a comparable basis in the future. However, the gas analysis laboratory cannot rest on its laurels, as there are still numerous halogenated VOCs in the atmosphere for which no SI-traceable references yet exist – so there is still much to be done.

METAS is strengthening its relations with the National Metrology Institute of Japan

METAS, in its capacity as the national metrology institute of Switzerland, and the Japanese national metrology institute **have signed a memorandum of understanding to strengthen mutual cooperation in the field of metrology and measuring standards.** The memorandum was concluded in Paris on 22 June 2023 on the sidelines of a meeting of the International Committee for Weights and Measures (CIPM). It was signed by the director of METAS, Dr Philippe Richard, and the Director General of the National Metrology Institute of Japan (NMIJ), Dr Takashi Usuda. The aim of this memorandum is to promote the exchange of scientific and technical knowledge between METAS and NMIJ and to expand the scientific and technical skills of the two NMIs.

Our metrology magazine has been given a new title and a makeover

After more than 15 years in the same look, it was time to rejuvenate **"METinfo".** No sooner said than done: **"METinfo"** was renamed **"The Reference."**, and the previous layout has been completely revised and updated. The magazine has been restructured and has become more readable. In order to provide you with better orientation and interesting insights, we have also created new sections: these include "In brief", "Object" and "Look behind the scenes" – you can get to know the first two in this issue. Like the previous metrology magazine, "The Reference." will also provide informative content and a wide range of topics. A digital version in the form of a PDF can be downloaded from our website at any time.

Prize in physical metrology

Since 2014, METAS has endowed a prize from the Swiss Physical Society (SPS) for outstanding work related to metrology. This year's METAS prize was awarded to Dr Mohammad J. Bereyhi for his doctoral thesis on nanomechanical quantum systems at EPF Lausanne (EPFL) at the SPS's annual conference in Basel in early September 2023. We warmly congratulate Mohammad on winning this award and wish him every success as CEO of his start-up Luxtelligence.

Bobjoseph Mathew is president of the CIML

Since 17 October 2023, Bobjoseph Mathew has been the tenth president of the ***International Committee of Legal Metrology (CIML).***

He was elected for a six-year term of office. The Head of the Department of Legal Metrology and Deputy Director of METAS is the first Swiss national to hold this post. Before that, he was vice president of this committee for four years.

He is the tenth president of the international organisation that has existed since 1955.

A trustworthy quality system

Confidence in the functioning of the quality system was extended for a further five years on 29 March 2023 on the occasion of the meeting of the Technical Committee for Quality (TC-Q), which was organised in-house by METAS this year.

METAS has been operating a quality management system since 2001 which covers its metrological activities in accordance with the standards ISO 17025 and ISO 17034. This system is regularly reviewed by the Technical Committee for Quality of EURAMET (TC-Q) within the framework of the CIPM Mutual Recognition Arrangement (CIPM MRA).

Detecting earthquakes with fibre-optic networks

A new technological breakthrough uses existing fibre-optic networks to detect earthquakes over long distances. The technology is based on active phase noise suppression and was originally developed to distribute optical reference frequencies. Applying seismic phenomena could now help to detect earthquakes more quickly and accurately, thus providing better protection against natural disasters. Dominik Husmann and Jacques Morel from METAS demonstrated this technology in collaboration with the group headed by Prof. Andreas Fichtner from ETH Zurich.

To read the scientific publication in the journal Scientific Reports, scan the QR code.

The Forwarding Department and its team in particular are a cornerstone of METAS. Without their commitment, verifications, tests and calibrations could not be carried out for our customers. The comparison campaigns would also be unthinkable without the reliable shipment of measuring devices. Between September 2022 and September 2023, our Logistics Unit accepted delivery of an impressive

11,100

consignments and delivered them to their intended recipients.

Interview

“The indirect connec to technology is still there, which is very important to me.”

After four years in the role of Head of Verification and Testing, Dr Fabiano Assi took over the management of the Physics department at the beginning of 2023 and thus his new role as a member of the Executive Board. As Head of Department, he leads over 70 employees. In the interview, Fabiano Assi looks back and explains his motivation for the change of job.

Interview with Fabiano Assi, conducted by Xavier Rappo

Fabiano, you have been a member of the METAS Executive Board since January 2023. What motivated you to take on this role?

In October 2022, the position of Head of the Physics Department became vacant, and I was asked if I would be willing to take up the post on an interim basis. It was a relatively easy decision: I saw this as an opportunity to gain valuable experience and get involved in a field that I was very interested in. During my time as interim manager between October and November 2022, I got to know the department, the operational processes and the employees better. It became clear to me during this time that this position is extremely exciting and challenging. This ultimately led me to decide to apply for the position and accept it in the end.

What is the biggest difference between your previous position as Head of Verification and Testing and your new position as Head of the Physics Department?

The biggest difference lies in the dimension and complexity of responsibilities. In my current role as Head of the Physics Department, I now manage more than 70 employees, compared to just under 20 in my previous position. As a member of the Executive Board, I also quickly realised that my decisions now have a much greater impact on the organisation. However, I can always rely on the support of my colleagues on the Executive Board and exchange ideas with my employees.

tion

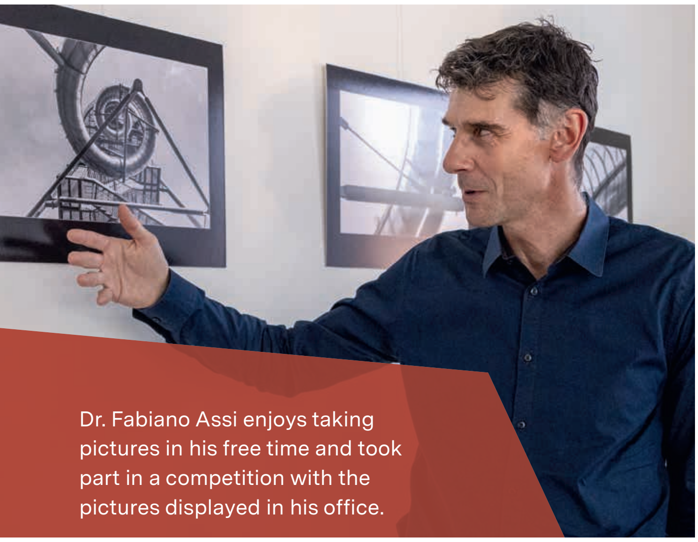
Dr. Fabiano Assi has been the new Head of the Division Physics at METAS since 1 January 2023.

Previously, you worked in materials science and innovation management which were more practice oriented. Do you not miss the practical relevance in your new role?

The practical relevance has by no means been lost – it is still omnipresent. I receive information from various projects on a daily basis and regularly exchange ideas with the employees in the laboratories and the head of department about new developments and various activities. Over and above that, I am in close contact with customers and partners. Although this no longer involves direct, practical activities in the laboratory, there is still an indirect connection to technology, which is very important to me.

You led the Verifications and Tests division for five years until the end of 2022. What are you particularly proud of having achieved during that time?

I am particularly proud of the discussions surrounding and the development of Vision METAS 2025, which took place in 2019 and 2020. I had the opportunity to help shape this vision. The reorganisation of the Verifications and Tests division was also an important step. When I took over the division in 2018, it was structured in a completely different way. I am also pleased to be involved in projects such as the revision of the approval process and the organisation of the Road Transport Conference in September 2022. All these experiences have enriched me personally and have shown how changes and shared visions can be implemented.



Dr. Fabiano Assi enjoys taking pictures in his free time and took part in a competition with the pictures displayed in his office.

And what are you less proud of?

A disappointing experience was the autonomous driving projects and applications in which I was involved as Head of Division. We took various approaches and tried to initiate joint projects with other metrology institutes. Unfortunately, our projects were not accepted. We invested time and resources without achieving the desired success.

Let's look ahead to the future: in the 2022 Annual Report, it says "Well positioned for the challenges of tomorrow". What does this mean for your department in particular? What are these challenges?

For our department, this means that we need to prepare for developments in metrology, especially with regard to more accurate and complex measuring instruments and measuring systems. It is now about understanding and building up what is needed in the future. Currently, the focus is on various topics, including digitisation, quantum physics and generally more precise measuring instruments or measuring instruments with smaller measurement uncertainties. We need to understand exactly what the technical requirements will be in the future and how metrology can help meet these needs. Our department needs to be flexible and adaptable, in order to be able to respond to these constantly evolving challenges.

And how can these challenges be overcome?

For the Physics department, preparing for the challenges of tomorrow means that we are primarily facing strategic decisions. Compared to other metrology institutes, METAS is smaller and does not have the funds and resources to follow up on all topics and projects in depth. We must therefore carefully consider which areas and projects we want to pursue. We need to make smart decisions to ensure that we do not miss the relevant developments while using our limited resources efficiently. This requires continuous assessment and adaptation of our strategies and priorities.

Where is the journey of metrology heading for the Physics department?

Overall, it is apparent that physical metrology continues to play a key role in scientific research and technological applications. However, it faces various developments and challenges: firstly, the aim is to continuously increase the accuracy of the measurements and to avoid measurement uncertainties. On the other hand, networked measuring systems and measurement intelligence are a central topic: the trend is heading towards the development of measuring systems in which the various measuring instruments are interconnected and rely on multiple sensors to carry out a precise and intelligent measurement. However, standards and methods still need to be established to test and validate such networked measuring systems.

"The biggest difference lies in the dimension and complexity of responsibilities."

The continuous improvement of accuracy and the integration of new technologies will help to make metrology even more relevant in the future.

Will metrological tests also be necessary in the future? Today, almost everything is digitised, so nothing can be measured incorrectly any more.

Metrological tests will continue to be necessary in the future, even in a digital world. For instance, natural processes, such as chemical reactions or physical phenomena, are fundamentally analogue in nature. Digitisation only occurs when these processes are measured and the data is transferred into digital formats. Therefore, metrological tests are needed to ensure the accuracy and reliability of these measurements.

In addition, metrological tests will play a role, especially for algorithms and AI systems. These systems process and analyse digital data, but the quality and accuracy of the input data is and will remain crucial. However, metrological tests are also necessary to ensure that the data processed by such systems is still reliable. Overall, the role of metrological testing in a digital world will continue to be of great importance to ensure the traceability of data and the reliability of measuring systems.

How can METAS benefit most from your experience?

Overall, I offer a wide range of skills and experience that can help to strengthen METAS in its research and development work and drive the implementation of its objectives.

My more than eleven years in the solar and semiconductor industry have allowed me to work in an extremely dynamic environment. METAS can benefit from my ability to think outside the box and find creative solutions to complex challenges.

My experience in dealing with customers from different countries and cultures also enables METAS to strengthen its customer relations and be internationally competitive. And my experience in large, cross-site projects helps to establish effective project management practices in METAS and optimise project execution.

Finally, thanks to my academic background, at universities in both Switzerland and the USA, I bring in-depth research experience to the table. This allows me to promote scientific excellence at METAS and to push ahead with research and innovation projects. ⬢

> **"The continuous improvement of accuracy and the integration of new technologies will help to make metrology even more relevant in the future."**

About METAS

10th anniversary of being a federal institute

What began in 1862 as the *Eidgenössische Eichstätte* (federal verification laboratory) is now the Swiss Federal Institute of Metrology METAS. Since 2013, METAS has been a federal institute, i.e. a decentralised unit of the Federal Administration with its own legal personality and operating on its own account. METAS celebrated its 10th anniversary as a federal institute last June. The event focused on its role as a trustworthy benchmark for accurate measurements and reliable results – in the article we look back at its beginnings and, in particular, the last ten years.

Dr. Jürg Niederhauser

In September 1862, the Federal Council decided to set up an *Eidgenössische Eichstätte* (federal verification laboratory), a predecessor institution of what is now called METAS. In the more than 160 years of its existence, METAS has always adapted to current requirements, both in terms of scientific and technical development and organisation. What began as the *Eidgenössische Eichstätte* became the *Eidgenössisches Amt für Mass und Gewicht (AMG) (federal office of measurement and weight),* then the Federal Office of Metrology (FOM), followed by the Swiss Federal Office of Metrology and Accreditation METAS, and finally the Federal Office of Metrology METAS became the Federal Institute of Metrology METAS.

The transition from a federal office to a federal institute

A good ten years ago, on 1 January 2013, the former federal office was transformed into a public institution with its own legal personality and operating on its own account. As a result, METAS has gained more independence so it can perform its tasks more efficiently. A more flexible organisational structure is essential, in order to meet as best as possible the technological challenges that a national metrology institute has to face. At the same time, the social and economic importance of a national metrology institute suggests that it should be closely linked to the government.

Ensuring reliable measurement

The basic tasks have remained the same over all these years: METAS is tasked with ensuring that measurements can be made in Switzerland with the accuracy required for business, research and society. It must also ensure that the measurements necessary for trade and transport, as well as for the protection and safety of people and the environment, can be carried out correctly and in accordance with the regulations.

Reliable metrology is the basis for creating confidence in measurements of all kinds in daily life – that was the case in the past, that is the case today and will continue to apply in the future.

New topics and areas

In order to be able to reliably characterise luminaires, metrology was and is essential in optics. Sensors are now playing an increasingly important role in lighting technology, as they contribute to energy saving. METAS set up a test laboratory three years ago to test such motion and presence detectors. This newly built test laboratory is an example of developments that have taken place in the ten years that METAS has been an institute. It is a new facility in an area in which METAS has already been successfully operating for a long time: optics. In recent years, however, METAS has also set up facilities in new fields, such as biology and laboratory medicine.

The visitors showed a keen interest in the METAS measuring equipment on the customer day.

The biology laboratory which was put into operation in the autumn of 2021.

In laboratory medicine, traceable measurements – as are common in physics and some areas of chemistry – are becoming increasingly important. METAS specialises in nucleic acid metrology in this field. In order to be available as a reliable contact person and service provider in this field, METAS has set up a biology laboratory that was commissioned in the autumn of 2021.

New tasks

The development of the institute is also reflected in the figures: in 2013, METAS had a workforce of 151 FTEs, and now that figure has risen to 239 including training positions. METAS is greatly committed to vocational training, which is reflected in the high proportion of trainees: in 2022, the proportion amounted to 8.4 % of the total workforce.

The increase in the number of staff is due, on the one hand, to the fact that METAS has taken over tasks and the associated facilities. For instance, METAS has expanded its chemical-analytical laboratory activities several times: in 2017, the customs laboratory of the then Federal Customs Administration (FCA) was integrated into METAS and at the beginning of this year, the laboratories of the Federal Food Safety and Veterinary Office (FSVO) followed suit.

Trustworthy benchmark

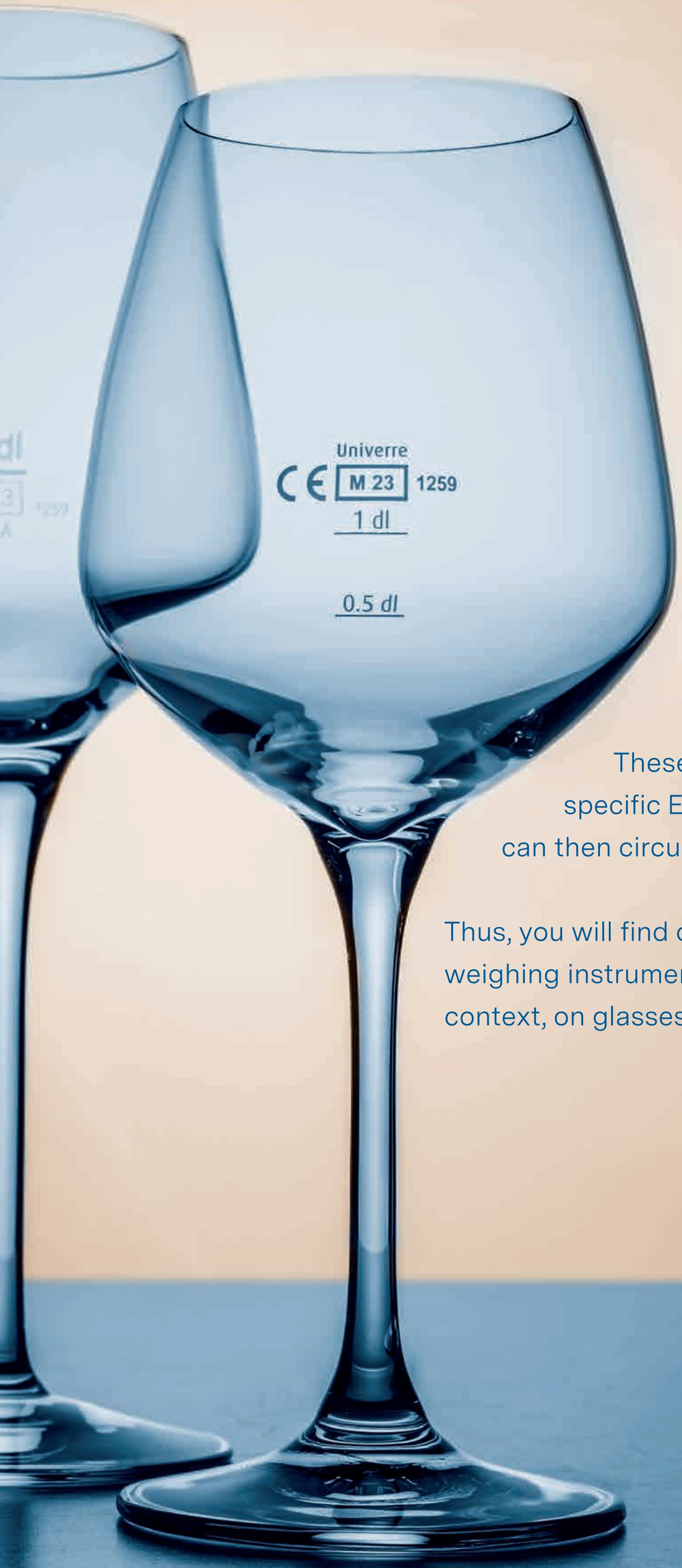
Since being founded in 1862 as the *Eidgenössische Eichstätte,* METAS has therefore always adapted to current requirements, both in terms of scientific and technical development and organisational structure. For a good ten years, in its capacity as a Swiss institute, METAS has been fulfilling its role as a trustworthy benchmark for accurate measurements and reliable results in various industries.

Adapting the organisational structure

METAS has also continuously adapted its structures and processes to meet technological and social requirements. In 2019, an independent “Chemistry” department (now called “Chemistry and Biology”) was created to express the increasing importance of metrological traceability in chemistry and biology. New divisions have been set up, such as digital transformation, data science, customer care and communications.

Object

Conformity mark



METAS-Cert certifies manufacturers and their measuring instruments so that the CE conformity marking and the metrological M can be affixed for regulated use.

These markings indicate the conformity with the specific European requirements for the product, which can then circulate freely within the European Economic Area.

Thus, you will find our number 1259 on various instruments, including weighing instruments, electrical energy meters or, in a more friendly context, on glasses.

Reference material

Certified reference materials: metrology in support of food safety

High-protein foods such as dairy products can harbour a range of contaminants that may present a health risk for the people that consume them. The certified reference material developed by METAS ensures that consumers can trust in the food safety system.

How can we guarantee that our food is safe and not hazardous to health? Our researchers recently developed a novel, whey protein-based certified reference material for determining polycyclic aromatic hydrocarbons (PAHs) and toxic elements. This makes it possible to reliably determine the concentrations of these dangerous contaminants in our food and establish metrological traceability by relating them to reference values.

Peter Rohrer, Simon Lobsiger

Food safety cannot be left to chance

Food safety is a hot topic in today's society. As awareness of healthy eating and nutrition increases, we also want to be sure that what we eat is free of harmful substances. The World Health Organization (WHO) has called for a better study of the impact of chemicals on the burden of foodborne diseases. Responding to this request will require more surveillance data on food contaminants, and this must be based on accurate, metrologically traceable measurement results.

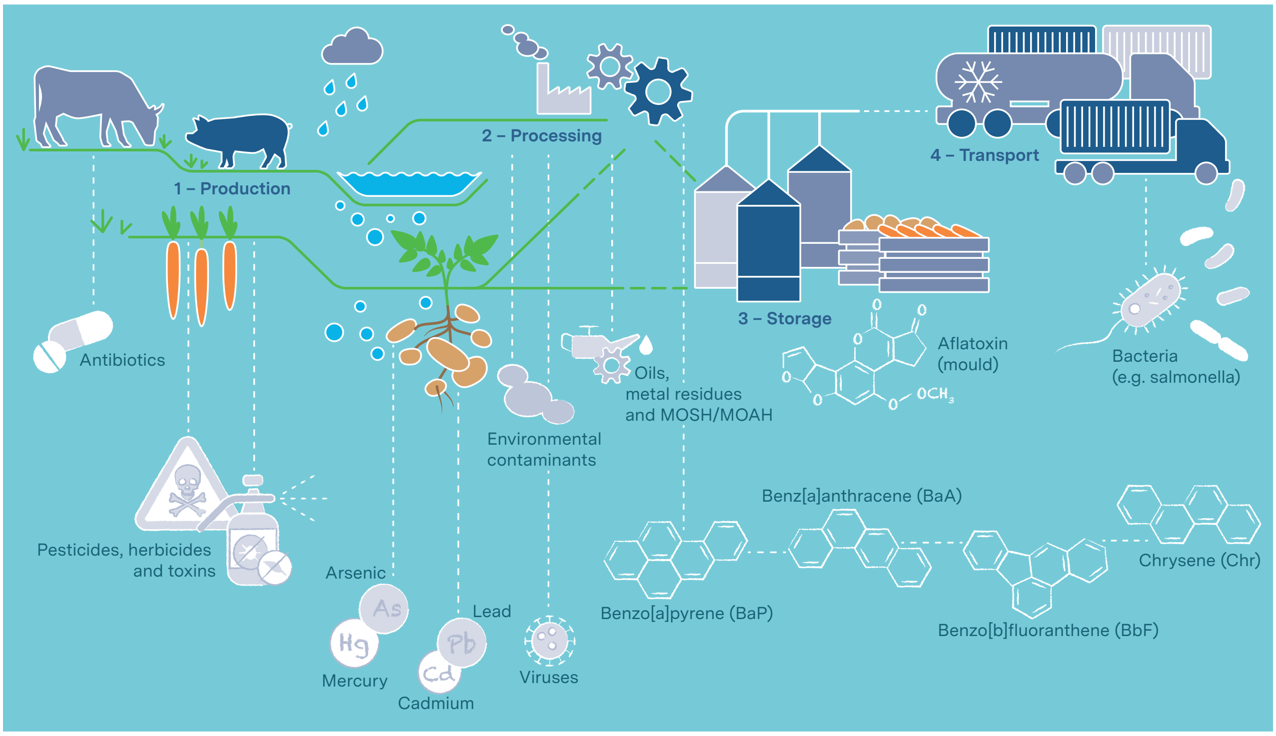
PAHs and toxic elements: hidden hazards in our food

Polycyclic aromatic hydrocarbons (PAHs) are harmful organic compounds that consist of hydrocarbons with at least two fused aromatic ring systems. They mainly form as the result of the incomplete burning of organic material. Several of these PAH compounds are classed as genotoxic (DNA-damaging) and carcinogenic (able to cause or increase the risk of cancer).[1]

Polycyclic aromatic hydrocarbons can enter a range of foodstuffs (including cereals, smoked or flame-grilled meat and fish, plant-based oils and fats, tea and coffee, etc.) and pose a risk to human health. Food can be contaminated by PAHs present in the soil, water or air as environmental pollution, during processing (either industrial or on a smaller scale, e.g. when cooking at home) or through packaging processes and packing materials. Their low solubility in water and lipophilic nature allows them to easily accumulate in food products.

The presence of toxic (poisonous) elements such as arsenic (As), lead (Pb), cadmium (Cd) and mercury (Hg) in the environment, and as a possible consequence, in our food, poses a further and no less significant hazard to human health.

To mitigate these health risks, both the European Union and Switzerland have enacted legislation[2] in order to regulate the maximum levels of the four PAHs and toxic elements – benzo[a]pyrene (BaP), benz[a]anthracene (BaA), benzo[b]fluoranthene (BbF) and chrysene (Chr), and arsenic, cadmium, mercury and lead respectively – allowed in food.



Various types of hazardous pollutants can make their way into our food.

How do we examine everything that needs to be examined?

Using precise, reliable and proven analytical methods, food analysis laboratories ensure consumer protection by analysing the chemical contaminants in food to verify that these do not exceed the regulatory threshold values. These laboratories generally require complex, matrix-dependent sample preparation and sophisticated measuring procedures, including extraction (separation, isolation), clean-up, concentration and measurement (note: the matrix is the entire portion of the sample that the target substance is embedded into; in other words, everything that does not need to be analysed).

Certified reference materials (CRMs) play a vital role in developing, validating and assessing the performance of these analysis methods, as well as in ensuring that measuring results are traceable to standardised reference, the International System of Units (SI).

The principle behind using a CRM is straightforward in of itself: if an analysis process (or at least, the device used to perform the analysis) successfully returns the certified values for the contaminants in the CRM as set by its manufacturer, and does so within the allowed measurement uncertainty, that process is deemed “fit” for use in examining real test samples. This makes it possible for the laboratory to use the CRM to review and, if necessary, adapt its entire analytical process.

In recent years, the demand for CRMs to facilitate the analysis of foodborne contaminants, including PAHs and toxic elements, has continued to rise. The current dearth of commercially available reference products of this kind has complicated laboratories’ efforts to perform quality control in the context of food safety.

METAS’ certified reference material

A team of researchers from METAS’ “Chemical and Biological Metrology” section set out to develop, produce and characterise (a process for describing/determining certain characteristics, properties or qualities in a substance) a CRM with the “catchy” designation of WP-CBR001, for use in analysing the four PAHs (BaA, BaP, BbF and Chr) and toxic elements (As, Cd, Hg and Pb) in a high-protein foodstuff – provided in this case by a matrix consisting of whey protein with a high protein content.

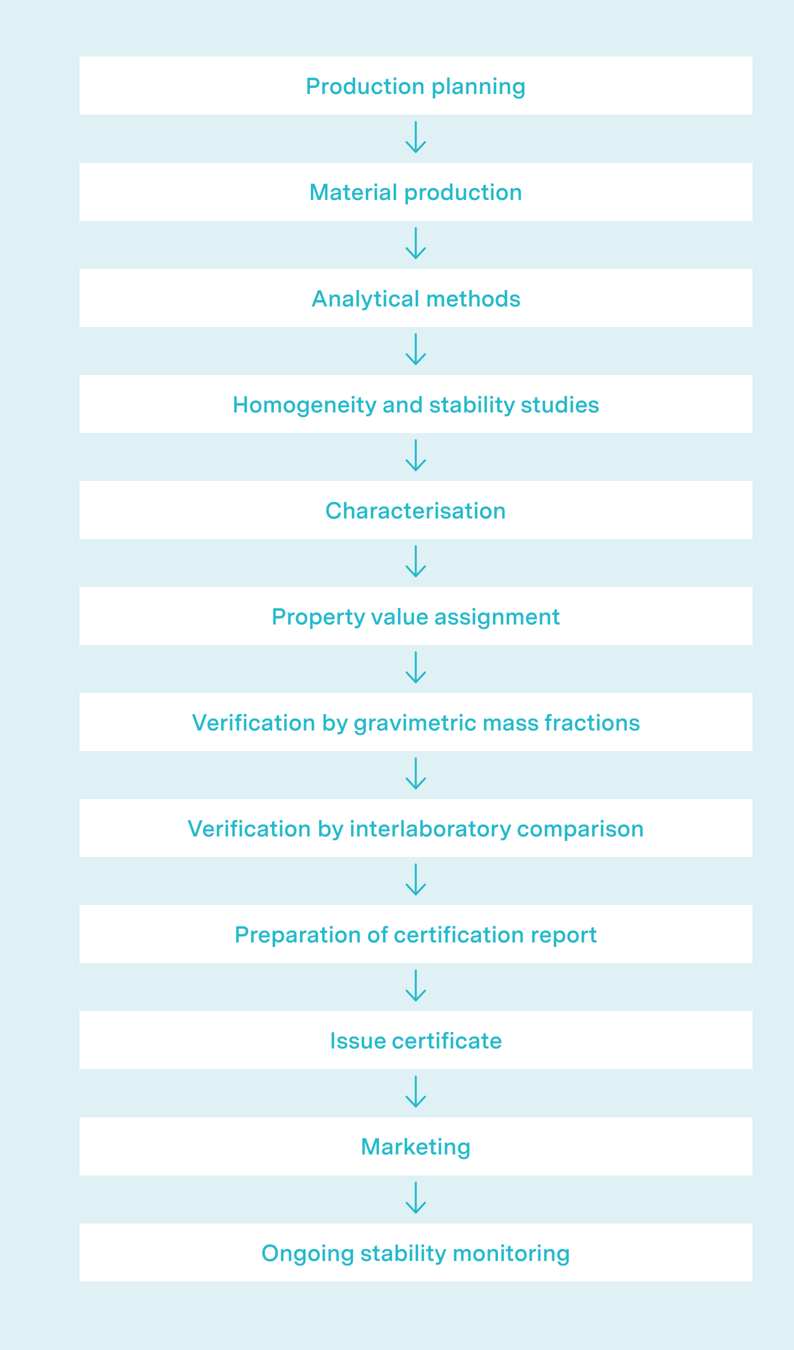
Although whey protein is not explicitly listed as a foodstuff in the regulations pertaining to PAHs and toxic elements, it can be used as a model for high-protein matrices. Whey protein powder is produced from whey, a liquid by-product of cheese manufacturing, and is therefore available in large quantities in the dairy industry. Whey protein powder has a high nutritional value due to its high content of essential elements and amino acids, and is one of the most commonly used food additives and supplements worldwide. However, as with many other food additives and food supplements, whey protein is susceptible to contamination by PAHs and toxic elements through environmental pollution, production processes or packaging materials.

Structure of the CRM development process

WP-CBR001 was developed for the determination of the target analytes BaA, BaP, BbF and Chr and the toxic elements arsenic, cadmium, mercury and lead. It was produced as part of a joint project, conducted between METAS, Sigma-Aldrich Production GmbH (a subsidiary of Merck KGaA, Darmstadt, Germany), and Hochdorf Swiss Nutrition Solutions AG, that consisted of the following main steps (based on [3] and [4]).

Production of WP-CBR001

The commercially available, industrially produced whey protein powder proved to be free of the target PAHs and target elements, which meant that liquid whey had to be actively contaminated with BaA, BaP, BbF, Chr, As, Cd, Hg and Pb before it could be processed into a CRM. The entire process for producing WP-CBR001 mirrored that used in industry, which ensured that our CRM would behave similarly to real whey protein powder with respect to sample preparation.



Sequence of the main steps involved in the development of METAS' CRM WP-CBR001.

Since the mass fractions of PAHs and toxic elements in whey protein powders are not explicitly regulated in the EU and in Switzerland, METAS' researchers made reasonable assumptions when defining the target mass fraction for each of the four PAHs and toxic elements.

The raw whey, which had been produced via a conventional industrial production process, was contaminated with each of the four respective PAHs and toxic elements by means of a spike solution. Once all of the contaminants had been introduced, the adulterated whey was then spray-dried using an industrial pilot plant. The main constituents of the resulting whey powder matched those of commercial whey protein powders produced under similar conditions as WP-CBR001.

The final product was filled into around 700 bottles in 30 g portions, with each bottle numbered according to the order of bottling, and then deep-frozen for storage.

Constituent	Mass fraction (g/100 g)
Protein	77
Carbohydrates	10
Fat	6
Water	5
Other	2

WP-CBR001 meets quality requirements

The homogeneity study was performed using ten randomly selected bottles and featured three rounds of analysis. No evidence of a statistically significant inhomogeneity was observed, either within or between the bottles, during this study.

In the stability study, selected bottles were stored separately at different temperatures (–20°C, 4°C, room temperature and 45°C) for up to twelve months and subsequently analysed. No statistically significant instability was observed during the study, even at room temperature and, in the short term, at higher temperatures. During the subsequent characterisation in METAS' laboratories, the researchers accurately determined the mass fraction (content)

of each of the eight spiked contaminants and estimated the uncertainty of each value, taking into account all associated influence quantities.

The certified values for reference material WP-CBR001 were verified by an interlaboratory comparison (a counteranalysis performed independently by selected control laboratories in Switzerland and Germany under real laboratory conditions) and gravimetric values drawn from the mass balance as identified from production data (where the exact mass of introduced contaminants in the total mass of the end product is a known value). One of the significant findings of the interlaboratory comparison was that only polar and protic solvents, such as methanol or water, were able to provide access for the complete extraction of the four PAHs. This knowledge will prove important in establishing new, standardised analysis methods in future.

An important product from METAS' laboratories

The protection of consumers from ingesting hazardous contaminants is an important task for food safety laboratories, which use many different analysis procedures to verify compliance with the statutory provisions in this area. As the required sample preparation methods vary significantly between different food matrices, it is crucial that the chosen CRM closely approximates the studied material.

WP-CBR001, the whey-protein-based certified reference material developed by METAS for determining PAHs[5] and toxic elements, closely approximates a real, potentially contaminated high-protein food matrix. WP-CBR001 can therefore be used to determine the mass fractions, of the four PAHs and toxic elements – BaA, BaP, BbF, Chr, As, Pb, Cd and Hg – in food. The product is already available on the market and supports food testing laboratories in improving or reviewing their analysis methods, to ensure that they obtain valid and comparable results.

Distribution of reference material:

Switzerland: references@metas.ch
International: sigmaaldrich.com

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[1] World Health Organization & Food and Agriculture Organization of the United Nations. Evaluation of certain food additives and contaminants. Prepared by the sixty-seventh meeting of the Joint FAO/WHO Expert Committee on Food Additives. Rome; Italy. 2007. https://apps.who.int/iris/handle/10665/43592.

[2] Commission Regulation (EU) 2023/915 of 25 April 2023 on maximum levels for certain contaminants in food and repealing Regulation (EC) No 1881/2006 (Text with EEA relevance). Off J Eur Union. 2023;119:103–57. FDHA Ordinance of 16 December 2016 on the Maximum Levels for Contaminants (Contaminants Ordinance, ContO) of December 2016 (as at 1 July 2020). SR 817.022.15.

[3] International Organization for Standardization. General requirements for the competence of reference material producers. ISO 17034. Geneva: ISO. 2016.

[4] International Organization for Standardization. Reference materials – guidance for characterization and assessment of homogeneity and stability. ISO Guide 35. Geneva: ISO. 2017.

[5] S. Lobsiger, L. Märki, S. Mallia, G. Umbricht, H. Sprecher, K. Breitruck, M. Obkircher, Development of a novel certified reference material for the determination of polycyclic aromatic hydrocarbons (PAHs) in whey protein powder, Anal Bioanal Chem (2023), https://doi.org/10.1007/s00216-023-04863-9

WP-CBR001 in front of one of around 700 bottles, each containing 30 g of METAS' new CRM.

Certified values for reference material WP-CBR001

Contaminants: PAHs	Mass fraction (µg/kg)	Uncertainty (µg/kg)
Benz[a]anthracene (BaA)	3.17	0.32
Benzo[a]pyrene (BaP)	4.18	0.48
Benzo[b]fluoranthene (BbF)	4.73	0.49
Chrysene (Chr)	2.85	0.33

Contaminants: toxic elements	Mass fraction (mg/kg)	Uncertainty (mg/kg)
Arsenic (As)	0.214	0.033
Lead (Pb)	0.494	0.032
Cadmium (Cd)	0.200	0.019
Mercury (Hg)	0.555	0.011

The measurement uncertainty specified for the certified values in both tables is the expanded uncertainty with a coverage factor of k=2, and corresponds to a confidence interval of about 95 %.

Service

A beer can be drafted in one minute or in thirty years thanks to flexible, precise flow rates

Calibration of flow devices is important in several areas of pharmaceutical, flow chemistry and microfluidic applications, where dosage of process liquids or accurate flow measurement are required. Therefore, METAS has developed facilities with piston provers designed by METAS to address the issue of measuring with process-oriented liquids non-constant flow profiles. The lower limit of flow rates corresponds to draft a beer in thirty years.

Dr. Hugo Bissig, Martin Tschannen, Dr. Marc de Huu

Dosage of process liquids or accurate measurement of the flow rate are processes applied in industry. The flow devices in use can be calibrated to gain information about the accuracy and precision of its flow rate indication and its long-term stability. The calibration of the flow meter or microfluidic device with the process-oriented liquid is important, because some sensor technologies depend on fluid properties.[1] Performing dynamic flow profile changes to simulate any dosing process gives important insight in the behaviour of these flow devices and their accuracies under non-constant flow conditions.

METAS has developed facilities with METAS piston provers to address the issue of measuring with process-oriented liquids non-constant flow profiles for flow rates. The flow rate is as low as for drafting a beer in thirty years.[2–4] The METAS piston provers allow changing the flow rate within seconds and the generated flow rate change is traceable due to the calibrated position measurement and the inner diameter of the piston. The gravimetric method of the Microflow facility has also been upgraded with a so-called capillary beaker, where a vertically mounted capillary is used to collect the water by placing the outlet needle inside the top of the capillary to minimise instabilities in weighing data during flow rate changes[3, 5] (see Figure 3).

METAS piston prover as primary standards

The METAS piston provers generate flow rates for drafting a beer in almost one minute or in thirty years, which corresponds to the range from 400 mL/min to 20 nL/min (see Figure 1).[2–4] These flow rates are achieved by using commercially available glass syringes or homemade pistons with volumes ranging from 0.05 mL to 200 mL and the corresponding speed ranges of the piston provers ranging from 0.1 mm/s to 0.1 µm/s resp. from 4.0 mm/s to 4.0 µm/s for the Microflow facility resp. of the Milliflow facility. The METAS piston provers are primary standards for volume flow because the speed of the piston respectively the inner diameter of the prover body is calibrated and traceable to length and time respectively to length. Multiplying the speed with the cross section of the prover body results in the traceable volume flow rate. The movement of the piston is controlled by a high precision linear stage with a fixed linear measuring system. The pulses sent by the linear measuring system are counted by means

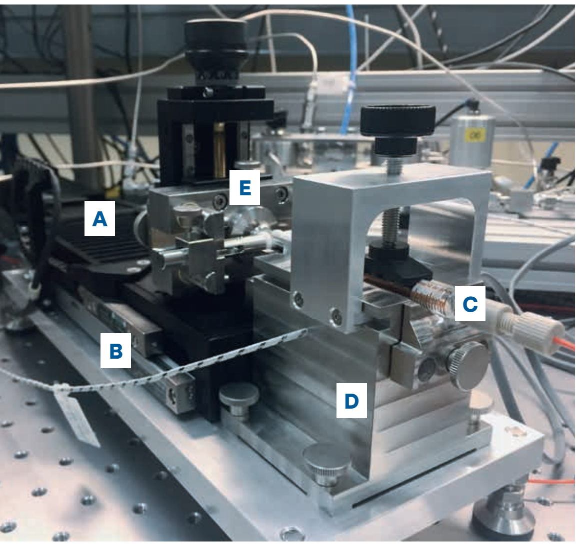

Figure 1: METAS piston prover of the Microflow facility. (A) high precision linear stage, (B) linear measuring system, (C) syringe, (D) mounting syringe body, (E) mounting and positioning for syringe plunger. The same design and components are used for the METAS piston prover of the Milliflow facility, but with a different speed range.

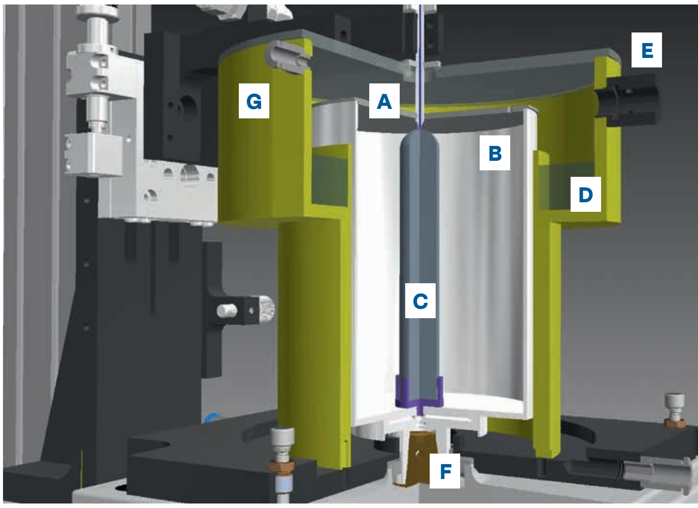

Figure 2: Weighing zone on the balance of the Milliflow facility. (A) outlet needle, (B) beaker with cover, (C) glass filter, (D) water in evaporation trap, (E) mount for T and rH sensor, (F) balance, (G) tubing for humidity exchanger.

of a Field Programmable Gate Array (FPGA). This Field Programmable Gate Array is running with hard coded program code on a defined constant cycle time of the order of 25 ns (40 MHz). For each pulse in any direction, a time stamp of the FPGA is recorded and a pair with the position and the timestamp is formed. This pair of values is then read from the main software and the real time position can be recorded. Applying a linear fit on several pairs of position data determines then the speed of the linear stage and thus of the piston.

Special beaker design for collecting the liquid

For devices generating the flow the METAS piston prover cannot be used to calibrate the delivered flow rate. Examples are high precision syringe pumps or insulin pumps. In this case the gravimetric method is used to collect the delivered liquid in a beaker on the balance.[3] The Microflow and Milliflow facilities have similar designs, where the flow of the water out of the outlet needle (see Figure 2 and Figure 3) into the beaker occurs over a water bridge between the outlet needle and the beaker contact. The beaker of the Milliflow facility contains a vertically mounted glass filter, where the outlet needle is in general positioned (200 to 50 µm) above the glass filter depending on the diameter of the outlet needle (1 to 0.3 mm) and the generated flow rate (Figure 2).[2]

The water enters the beaker by forming a water bridge between the outlet needle and the glass filter. Due to capillary forces the droplet formation at the outlet needle is avoided, which ensures a continuous water flow. The water flows through the glass filter down to the bottom of the beaker, where the water level is raising, but never reaches the top of the glass filter. Thus, the water contact between the outlet needle and the glass filter depends not on the water level inside the beaker.

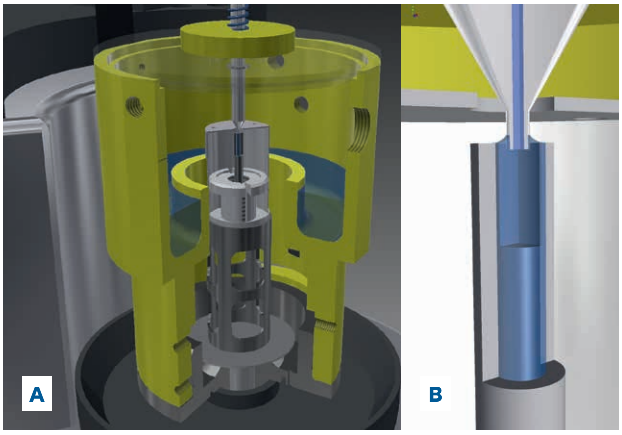

Figure 3: (A) Weighing zone on the balance of the Microflow facility showing outlet needle and beaker with cover. (B) Insight into the contact of the outlet needle in the capillary.

The capillary beaker of the Microflow facility contains a vertically mounted capillary (Figure 3). The capillary is filled with water and the outlet needle is positioned within this capillary.[3, 5] The water enters then the beaker through the capillary, and the water level in the beaker is raised without changing the capillary forces at the contact.

Measurements of dynamic flow profile changes

The piston prover and the gravimetric method use similar procedure to determine the instantaneous flow rates. For the piston prover, at each pulse of the linear measuring system the position and the timestamp are collected by the FPGA to make a pair. The weighing data are continuously collected by a Real Time system (RT), which communicates with the balance at 20 Hz and pairs the weight value directly with the time stamp of the RT. Choosing a fixed time window as short as half a second and performing the least square linear fit over the pairs of data within this time window allows to determine the instantaneous flow rates of the piston prover and the gravimetric method.

These instantaneous flow rate measurements offer the possibility to characterise the response time of flow meters. The signal of the Coriolis flow meter is recorded at a frequency of 25 Hz and averaged as well for half a second. All the instantaneous flow rates of the piston prover, the gravimetric method and the Coriolis flow meter measured at the Milliflow facility are shown in Figure 4.

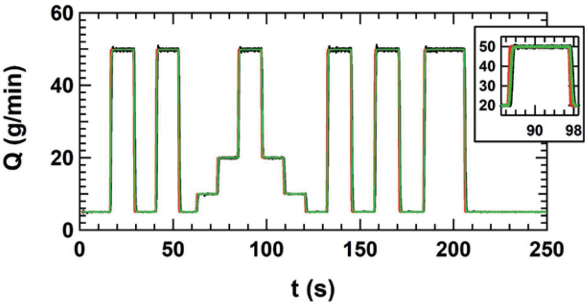

Figure 4: The determined mass flow rate Q of the gravimetric method (black line), the piston prover (red line) and the Coriolis flow meter (green line) as a function of time t.

Dynamic flow profiles at the lower limit

To measure the dynamic flow profiles at the Microflow facility the capillary beaker has been developed at METAS. Due to capillary forces, the meniscus between the capillary and the outlet needle remains almost stable even when a strong change in the flow rate occurs. A dynamic flow profile at the lowest range has been performed with a thermal flow meter to highlight the functionality of the Microflow facility. Three cycles with steps of 30 s at flow rates between 30 nL/min and 100 nL/min have been generated after a stabilisation time of 2 h as can be seen in Figure 5.

Obviously, the gravimetric method is getting to its limits for these short fixed time windows and low flow rates as can be seen by the noisy data (black line) in Figure 5. The influence of the variation of the capillary forces at the water contact between the outlet needle and the capillary gets more important than at higher flow rates. However, the data of the piston prover (Figure 5, red line) and the thermal flow meter (Figure 5, blue line) follow the same trend and the local speed fluctuations are recorded by both. This is due to the fact that the piston prover measures the effective flow rate, which is delivered.

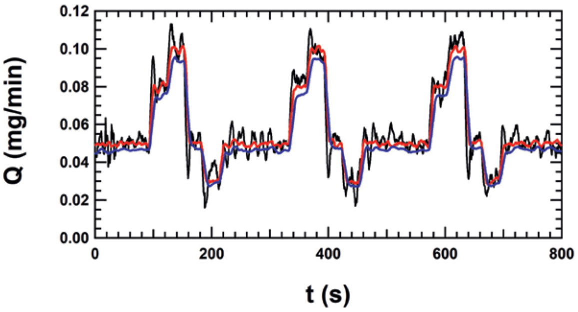

Figure 5: Dynamic flow profile generated by the piston prover (red line) and measured with a thermal flow meter (blue line). The gravimetric method (black line) is also shown.

These three cycles of data offer now different options to analyse the deviation of the thermal flow meter compared to the reference flow rate of the piston prover. First, the deviation of the flow meter is determined for each of the plateaus (constant flow rates) of the three cycles and the average of several deviations at each flow rate is calculated and shown in Figure 6 as full red circles. These deviations are compared to the deviations obtained from constant flow calibration over several hours (full blue circle), which were not performed at exactly the same flow rates. For two flow rates, the results are consistent even for these short measurement times.

Secondly, the average flow rates over a full cycle can be determined and compared to the reference flow rates. The deviations over a full cycle with respect to the reference flow rate of the piston prover respectively the gravimetric method are shown as open red circle respectively open black circle in Figure 6. These results are all consistent within the stated measurement uncertainties.

METAS piston provers guarantee traceable flow rate changes

METAS has developed facilities with METAS piston provers as primary standards to address the issue of measuring non-constant flow profiles for flow rates down to 20 nL/min with process-oriented liquids. The piston provers allow changing the flow rate within seconds and the generated flow rate change is traceable due to the calibrated position measurement and the inner diameter of the piston.

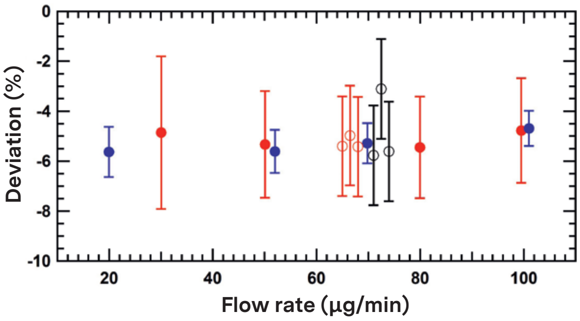

Figure 6: The deviations of the thermal flow meter. Constant flow calibration (full blue circle), plateau values from dynamic flow profile calibration (full red circle). Calibration over a full cycle with piston prover (open red circle) and with the gravimetric method (open black circle).

In addition, the gravimetric method allows the characterisation of insulin pumps or other flow generators. Moreover, the piston provers are part of a micro-pipe viscometer to determine in-line the dynamic viscosity of various liquids.[8] Keep in mind, thirty years for drafting a beer corresponds to the lowest flow rate of the facility.

Uncertainties for the flow rate and their validation

The facility uncertainties for the flow rate range from 400 L/min to 20 nL/min range from 0.07 % to 1.0 % for constant flow rates and from 0.20 % to 2.0 % for dynamic flow profiles. These uncertainties in the lower flow rates have been validated by means of an European interlaboratory comparison EURAMET project 1508 “Pilot study: Intercomparison of ultra-low liquid flow rates in range below 100 nL/min”, where the METAS calibration results of two thermal flow meters are consistent with the reference value.[4, 6]

Characterisation of Insulin pumps

The gravimetric method of the Microflow facility is also used to characterise the performance of insulin pumps, which delivers small amounts of liquids at a specific time interval to deliver the insulin quasi-continuous. The pump mechanism consists of a stepper motor that moves the plunger incrementally, which forces the piston into the container to push out the insulin. A discrete volume is often called a single dose and corresponds to the smallest delivered quantity of insulin. Depending on the basal rate (insulin flow rate), the volume of the single dose and the time interval between single doses (i.e. the cycle time) are varying. A detailed characterisation can be found in reference [7].

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Research and development

Advanced aerosol metrology for atmospheric science and air quality

Air pollution is an environmental and social issue; air pollutants are emitted from both anthropogenic and natural sources and give rise to health and climate change concerns.

B. Beckhoff, U. Winkler, S. Horender, J. Malet, M.-C. Lépy, S. Koust, S. Seeger,
J. Tompkins, K. Vasilatou *on behalf of the EMPIR AEROMET II consortium*

Accurate aerosol metrology is a prerequisite for enforcing regulations, protecting human health, and supporting research on climate change and atmospheric processes. However, air pollution is a complex challenge that currently lacks traceable measurement and characterisation of aerosols in the environment. To address this, the EMPIR AEROMET II project has provided methodological improvements.

Achievements of the EMPIR AEROMET II Project

Standardisation of soft X-ray aerosol neutralisers

Measurements of the particle size distributions using Mobility Particle Size Spectrometers (MPSS) rely on an accurate knowledge of the particle charge distribution. The most common approach – the use of radioactive charge conditioners to produce a well-known particle charge equilibrium – is not viable in many situations, mainly due to regulatory issues. During this project, novel non-radioactive alternatives underwent a thorough examination in a work package led by TROPOS (Leibniz Institute for Tropospheric Research) (Figure 2). The main focus was laid on four commercially available soft X-ray charger conditioners (SXR CC), which were kindly provided by three industrial stakeholders. The activities were directly related to the development of the standard ISO/DIS19996 "Charge conditioning of aerosol particles for particle characterization and the generation of calibration and test aerosols" within ISO TC24/SC4 Working Group 12. As a result of the long-term test, it can be stated that the particle charge equilibria produced by the tested models differed significantly from each other. With the new ISO standard it is possible to develop individual parametrisations. Besides, instrument performance also showed alterations with time which must be addressed. Within the same project, tests using non-spherical soot particles were also carried out and revealed, even for radioactive charge conditioners, some differences in the particle charge distribution compared to the state produced for spherical particles of the same size. Other tests at the UK Metrology Institute NPL concerned plasma charge conditioners which are still in development stage.

Standardisation for automatic pollen monitors

At present, there is no standardisation or available calibration procedure for automatic pollen monitors despite the fact that these instruments are about to be installed at meteorological stations across Europe. The AEROMET II project went beyond the state-of-the-art by developing traceable calibration procedures for automatic pollen monitors in the laboratory within a work package led by the Swiss National Metrology Institute METAS. For the first time, automated pollen monitors were traceably calibrated with size-certified polystyrene particles up to 20 µm. The automated pollen monitors were trained to measure pollen taxa and have been used in the field since 2021. In spring 2022 in Payerne, Switzerland and Oslo, Norway, two automated pollen

Figure 1: AEROMET II consortium at meeting at BAM, Berlin, Germany.

monitors were evaluated by comparing the measured pollen taxa to the manual reference method, i.e. the Hirst-type impactor, see Figure 3.

Calibration of TXRF spectrometers

A Benchtop-Total Reflection X-ray Fluorescence (TXRF) spectrometer from BAM was calibrated using reference standards. Full physical traceability to primary standards, such as a synchrotron-radiation based GIXRF set-up could be achieved at the German Metrology Institute PTB. Sets of reference calibration samples were produced and initial laboratory- and synchrotron-radiation-based characterisation measurements were performed. The effect of calibration on accuracy and comparability on benchtop TXRF spectrometers to reference-free SR-TXRF (Synchrotron Radiation TXRF) was demonstrated by measurements at facilities of the PTB and the French Metrology Institute LNE (in collaboration with CEA-LNHB) using reference sample sets produced during the project (Figure 4). An agreement in the order of 20 % could be achieved.

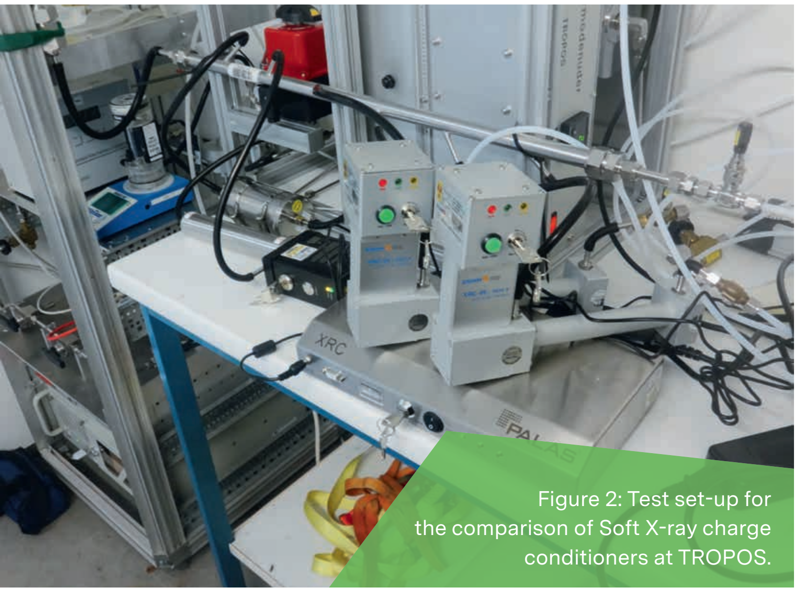

Figure 2: Test set-up for the comparison of Soft X-ray charge conditioners at TROPOS.

Calibration of portable aerosol instruments

Portable, commercially available instruments for the measurement of ambient aerosol particle number concentrations are popular because of their easy handling and affordability. However, their measurement uncertainties are not fully understood and the instruments exhibit high signal instabilities compared to more sophisticated laboratory-based instrumentation. The project compared portable, commercially available instruments with calibrated reference instruments under laboratory conditions. In addition, field studies were performed under a wide range of environmental conditions. This was the first step in quantifying the effects of a variety of environmental conditions, on the accuracy of the portable, commercially available instruments. The project also developed novel compensation algorithm procedures for the use of the portable, commercially available instruments that take into account the effects of the environmental conditions and apply relevant measurement principles.

Real-time online measurements of black carbon (BC) with portable optical instruments are also facing similar measurement quality issues related to inappropriate guidelines and the lack of traceable calibration methods. These BC-measuring instruments are currently not a mandatory component of air quality monitoring stations, therefore periodic calibration does not happen on a regular basis. Portable particle counters and BC-measuring instruments were characterised with respect to relevant key parameters such as counting efficiency, precision, and long-term reliability by performing laboratory and field campaigns (see Figure 5).

Perspectives for aerosol metrology

On June 27, 2023, a workshop on future aerosol metrology was organised by LNHB, IRSN and PTB in Saclay, France, in conjunction with the final AEROMET II project meeting. Invited contributions discussed upcoming developments in view of generally decreasing air pollution levels in Europe, novel regulations and technologies.

Mohsen Kazemimanesh (NPL) introduced synthetic ambient aerosols including challenges of measuring <10 nm or at least <30 nm particle sizes in view of future EU regulations. Furthermore, particle mass classifiers might be used as an in situ reference method for particle mass concentration measurements, since individual particles are classified by mass and not affected by composition.

David Butterfield (NPL) presented OPSS as approved devices and gave a short review of equivalence testing in the UK and in Germany involving PM_{10} and $PM_{2.5}$ automatic analysers versus gravimetric methodologies, for which deviations due to filter material were investigated. TEOM-FDMS (tapered element oscillating microbalance) is rolling out across automatic urban and rural networks at about 100 sites and the transition from mass-based automatic instrument to OPSS is being continued.

Amelle Kort (IRSN) summarised latest research on miniaturised sensors for aerosol measurements. She also stressed the need for in situ industrial facilities to measure aerosol deposition without modifying the deposit. Different kinds of sensors were of interest: acoustic wave mass sensors such as surface acoustic wave (SAW) and real-time-capable film bulk acoustic resonator (FBAR) as well as resonant mass sensors such as MEMS and NEMS.

FIgure 3: A new automated pollen monitor (left) next to a Hirst-type sampler (right, in the back).

Kostas Eleftheriadis (National Centre for Scientific Research Demokritos) presented the challenges with respect to monitoring of ultra-fine solid particles in ambient air. WHO has already formulated health concerns regarding non-regulated BC mass concentration and Ultrafine Particle (UFP) number concentration. These challenges involve the development of a thorough definition for the term “solid particle”, establishing links to the impact of the sources and the monitoring of the air quality. Open issues are UFP dynamical changes following emission, the link of health effects to specific sources and the gap in standards for tailpipe measurements and atmospheric monitoring networks.

Thorsten Streibel (Univ. Rostock) described the ULTRHAS-projects on Ultrafine Particles from Transportation and Health Assessment of Sources. Health effects of aerosols could be related to different transport modes, different fuels, non-exhaust emission, and atmospheric ageing processes, while the chemical compositions and secondary aerosols are relevant, too. He stressed that knowledge on health hazards of different sources could improve the risk assessment, drivers of toxicity needed to be identified and atmospheric aging effects could significantly change the particle size distribution.

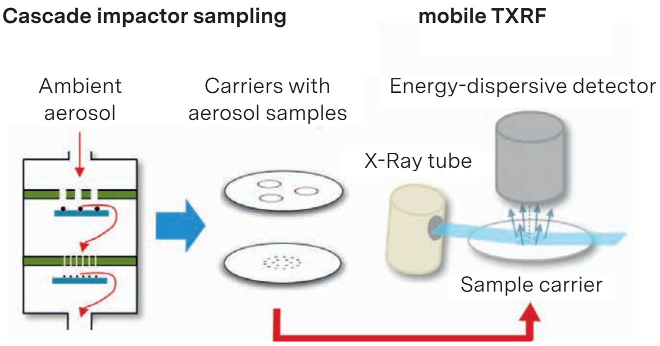

Figure 4: Schematic diagram of the experimental set-up for a portable TXRF spectrometer.

Thomas Krinke (TSI GmbH) reported on challenges in aerosol metrology from the perspectives of manufacturers and application. An overview over real-time measurements of submicrometer particles was given and the need for SMPS measurements to measure particles smaller than 10 nm was stressed. Suggestions to use an electrospray droplet generator for dispersing reference particles from solutions as well as to use water-based CPC for particles above 2 µm were discussed.

Drew Hill and Jeff Blair (AETHLABS) introduced advances in microAeth® miniaturised black carbon monitoring and source apportionment with applications for large-scale and portable Aethalometry. Microaeth instruments have been used for mobile monitoring to probe ambient and exposure concentrations, and spatial analysis of soot can be particularly important because of its high variability. Mobile monitoring facilitates the identification of spatial pollution hotspots, e.g. by comparing near-road and on-road pollutant concentrations.

Acknowledgement and references

This work was carried out as part of the EMPIR 19ENV08 AEROMET II project. This project has received funding from the EMPIR program, co-financed by the participating countries and the European Union's Horizon 2020 research and innovation program.

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The objective of the project

The overall objective of the EMPIR AEROMET II project is the traceable measurement and characterisation of aerosols in the environment. METAS has participated in several successful subprojects.

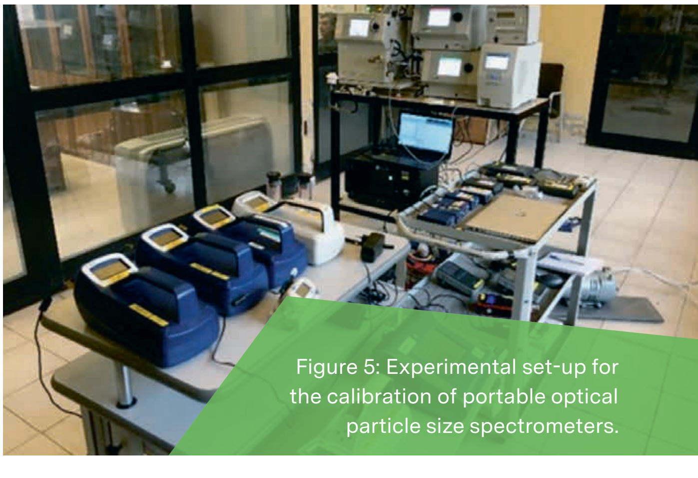

Figure 5: Experimental set-up for the calibration of portable optical particle size spectrometers.

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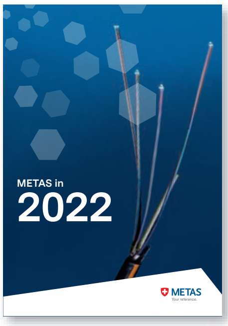

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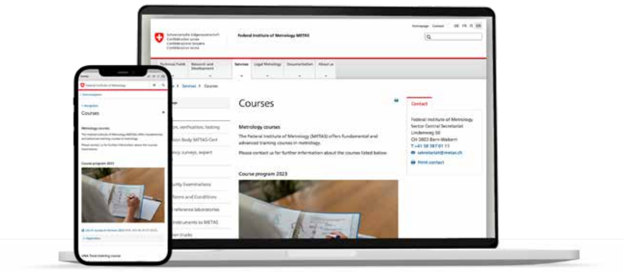

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