

# **BOOK OF ABSTRACTS**





Dear Colleagues,

Welcome to the Joint Conference of IMEKO TC8, TC11, TC24 and EUROLAB aisbl, being held in Funchal, from 11 to 13 of October 2023 in the conference facilities at Hotel VIDAMAR, in the magnificent Island of Madeira in Portugal. Following the recent success of the Joint Conference held in Cavtat-Dubrovnik, Croatia, in 2022, and strengthened by the long tradition of cooperation between IMEKO Technical Committees, RELACRE (the Portuguese Association of Accredited Laboratories) hosts the 2023 Joint Conference, organized by IMEKO TC8, TC11, TC24 and EUROLAB aisbl. This event will bring together the TIC community, academia and industry, promoting the latest advancements in science and technology in many fields of metrology.

The joint event promoted by IMEKO TC8 (Traceability in Metrology), IMEKO TC11 (Measurement in Testing, Inspection and Certification) and IMEKO TC24 (Chemical Measurements), covers different aspects of interest of the wide scientific domains, bringing experts and professionals from all over the world to share ideas and knowledge about "New challenges and opportunities in traceability", "Testing, Inspection and Certification for confidence and safety" and "New perspectives in Chemical measurements".

It is also a great pleasure to meet you in Madeira Island, a Portuguese jewel discovered in the XV Century in the Atlantic Ocean, known for the nice tropical weather, a place full of authenticity where art, culture and nature provide incredible experiences and lasting memories. We hope that you will be amazed with the exotic colours of the flowers, the blue sea and the emerald green vegetation of this archipelago, enjoying the city of Funchal, famous for its hospitality, the gastronomical heritage and the vibrant environment all over the places.

The Executive Committee and the Local Organizing Committee will do their best to offer you a memorable Conference, hoping to create a great opportunity to share knowledge and new ideas improving the growing interest in Traceability, TIC industry and Chemical measurements. We hope that you will find the event technically fulfilling and highly entertaining and that it will be an opportunity for useful interactions and communications with colleagues from all over the world. A warm welcome to all of you coming to Madeira, for the 2023 IMEKO TC8, TC11, TC24 & EUROLAB joint Conference.

On behalf of the Organizing Committee

Michela Sega	Álvaro Ribeiro	Tatjana Tomic	Laura Martin
IMEKO TC8 Chairperson	IMEKO TC11 Chairperson	IMEKO TC24 Chairperson	EUROLAB aisbl Secretary General



RELACRE was born in 1991, driven by the Portuguese Quality Institute and by some of the most relevant entities with laboratory activity in Portugal, creating conditions that did not exist until then, to act as a facilitator of synergies in a context of the growing economic activity of laboratories with accreditation. The constitution of RELACRE responded to a set of emerging challenges in the national context, following the dynamics of accreditation, and in the international context, after the foundation of EUROLAB (European confederation of laboratory associations) in 1990, constituting itself as the representative association of Portugal in this new organization and against the backdrop of the creation of the single European market. Since then, RELACRE has occupied an important space, responding to a growing need for shared resources and an independent space for dialogue and discussion of sectoral problems and potential solutions. This movement resulted in a strong institutional representation of the sector and, simultaneously, in the creation of bridges and a strong capacity for intervention in the European and international context, anticipating the importance that this aspect currently has for many of its members and for the laboratory community in Portugal.

# About IMEKO

IMEKO is a non-governmental federation of 42 Member Organizations individually concerned with the advancement of measurement technology. Its fundamental objectives are the promotion of international interchange of scientific and technical information in the field of measurement and instrumentation the enhancement of international co-operation among scientists and engineers from research and industry. Founded in 1958, the Confederation has consultative status with UNESCO and UNIDO and is one of the five Sister Federations within FIACC: Five International Associations Co-ordinating Committee, further consisting of: IFAC- International Federation of Automatic Control, IFIP International Federation for Information Processing, IFORS International Federation of Operational Research Societies and IMACS International Association for Mathematics and Computers in Simulation.

# About EUROLAB

EUROLAB was created in Brussels on April 27, 1990 on the basis of a memorandum of understanding, signed by delegations representing the private and public laboratories of 16 out of the 19 countries of the EEC and EFTA.

EUROLAB is since October 1998 a legal entity in the form of an international association under Belgian law (A.I.S.B.L. - Association Internationale Scientifique sans But Lucratif) setting it as the European Federation of National Associations of Measurement, Testing and Analytical Laboratories.





Michela Sega



**Thomas Wiedenhoefer** 



Laura Martin



Álvaro Silva Ribeiro



Marija Cundeva-Blajer



**Mladen Jakovcic** 



Tatjana Tomic



Leonardo Iannucci

# Local Organizing committee



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Ana Duarte



**Carla Estevens** 



Cila Silva



**Catarina Martins** 

# International Program Committee

TC8 - Traceability in Metrology	TC11 - Measurement in Testing, Inspection and Certification	TC24 – Chemical Measurements
Michela Sega <i>(Chair)</i> Thomas Wiedenhoefer Sergio P. Oliveira Serhii V. Pronenko	Álvaro Silva Ribeiro (Chair) Mladen Jakovcic Marija Cundeva-Blajer Honglei Yang Admer Rey C. Dablio Agne Bertasiene Antonio Shemakalu Christian Müller-Schöll Annette Röttger Rama Dasu Pittala Yoshitada Tanaka Rola Bou Khozam Shiv Kumar Jaiswal Gertrud Mamiya Ivana Ljevaković-Musladin José Luis Prieto Calviño Maria do Céu Lopes de Sousa Ferreira Paolo Emilio Roccato Sanjay Yadav Vedran Šimunovic Yao Hejun Bodan Velkovski Kruno Milicevic Gorana Baršić Mare Srbinovska Miryana Masheva Kiril Demerdziev Tzvetelin Gueorguiev	Leonardo Iannucci (Chair) Aleksandra Aleksic Carolina Andrade Emma Angelini Sandra Babic Ana Cop Leila Es Sebar Přemysl Fitl Sabrina Grassini Katarina HafnerVuk Martin Hruska Otta Jaroslav Luca Lombardo Zhechao Qu Michela Sega Christable Tan Tatiana Tomic Luisa Vigorelli

# Joint Conference of TC8, TC11 and TC24 Mottos

- TC8, New challenges and opportunities in traceability
- TC11, Testing, Inspection and Certification for confidence and safety
- TC24, New perspectives in Chemical measurements



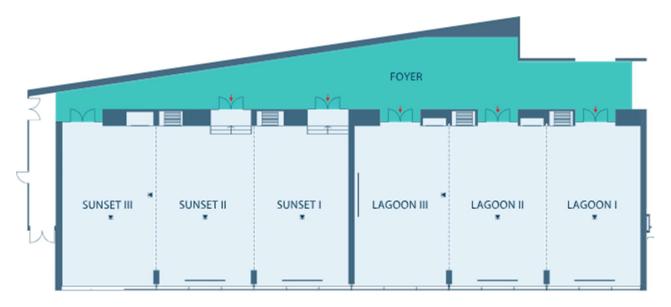
Vidamar Hotels and Resorts Madeira, Funchal, Madeira Island (Portugal)

#### VIDAMAR Resort HOTEL MADEIRA

#### Address: Estrada Monumental, 175-177 Funchal

Phone Number: (+351) 291 717 700





The 5-star Vidamar Resorts Madeira is situated on Madeira's southern coast, just few minutes' walk from central Funchal. It features 3 salt-water infinity pools for adults and one for children. The property boasts panoramic views of the Atlantic.

All rooms at Vidamar Resorts Madeira have sea or garden views from the balconies. Each room is equipped with air conditioning, cable TV, mini refrigerator, and an en suite bathroom.

The room rate includes half board plan. On-site restaurants offer local and international specialities and include the Ocean Buffet Restaurant, the Sabor a Mar Restaurant, the Casa das Espetadas Restaurant, the Koi Sushi Bar and the Italian á la carte restaurant Mamma Mia by Giovanni. There is also a piano bar and snooker lounge.



#### SUMMARY OF TOPICS TC8 Traceability in Metrology

- Digital twins
- Energy-related topics
- Traceability in AI, self learning systems and big data
- Metrological traceability in chemistry (primary reference methods and primary standards)
- Reference materials and certified reference materials
- International cooperation and SI traceability in chemistry, pharmacy and medicine
- Standardization

#### SUMMARY OF TOPICS TC11 Measurement in Testing, Inspection and Certification

- Management systems and Quality in the TIC sector
- Regulatory framework for quality, safety and security of products and services (agrifood, environment, health, cybersecurity, communication, construction, IoT, AI, among others)
- Innovation and validation of methods in testing, calibration, inspection and certification
- Sampling activities related to measurements in the TIC Sector
- Metrological traceability, measurement uncertainty and conformity assessment in TIC Sector
- Certification of reference materials and their use in laboratories
- Certification of products, processes, management systems and persons
- Digital transformation in testing, inspection and certification
- Measurements as an enabler of economic development
- Quality management and conformity assessment in TIC Sector
- SMART Metrology and TIC: Demands and new opportunities in the incorporation of Key Enabling Technologies (KET's)
- Other TIC issues

#### **SUMMARY OF TOPICS TC24 Chemical Measurements**

- Traceability and fundamental chemical metrology
- Chemical and biochemical sensors
- Separation techniques
- Gas-phase chemical measurements
- Measurements for Cultural Heritage
- Process Analytical Technology
- Validation, uncertainty and quality control in chemical measurements
- Chemical measurements for environment
- Chemical measurements for food and agriculture
- Chemical measurements for health and biomedical applications
- Data integrity and digitalization



# Damiano Pietri



"Digitization of laboratory processes: the ABC Bilance-Metricode case" Co-founder and President of Metricode s.r.l. which develops technologies to automate and digitalize industrial weighing processes by integrating them with hardware and industrial applications. Previously he was CEO in ABC Bilance s.r.l. and in charge of legal metrology and he is still the calibration laboratory manager. Damiano represented for four years an important Italian SME association being delegated for digital development issues both in Italy and Europe.

# Přemysl Fitl



"Solid-state gas sensors and their applications in the field of metrology" Přemysl Fitl has been working in field of chemical gas sensors since 2002. He is currently working at the University of Chemistry and Technology, Prague and the Institute of Physics of the CAS, where he works on the development of chemical gas sensors using thin film technologies and research on the preparation of thin films and nanostructured materials. He teaches courses focused on measurement and sensor technology. He is the author of 105 published scientific papers, 1 monograph and 2 patents. H-index 18. Since 2014 he is the Czech representative of TC24 in IMEKO organization.

## Sascha Eichstädt



"Digitalization of the Quality Infrastructure"

Dr. Sascha Eichstädt is the leader of the department "Metrology for digital transformation" at the Physikalisch-Technische Bundesanstalt (PTB) since mid-2021. He received his Diploma in Mathematics in 2008 at the Humboldt University Berlin, and his PhD in Theoretical Physics in 2012 at the Technical University Berlin. He is a passionate metrologist since 2008 when he joined the group "Mathematical modelling and data analysis" at PTB to work on measurement uncertainty for time series. In 2017 he started his adventures in digital transformation of metrology as working group leader of the group "Coordination Digitalization" in the Presidential Staff of PTB. He chaired the EURAMET working group "Metrology for digital transformation" from 2020-2022. Sascha Eichstädt is chairing the OIML Digitalisation Task Group since 2022 and the IMEKO Technical Committee on Digitalisation since 2021



### **IMEKO TC8 Traceability in Metrology**

### Digitalization in Traceability – new tools, new opportunities, new challenges

To provide a comprehensive understanding of the latest trends and technologies in digital supported traceability, challenges, and opportunities in implementing digital traceability solutions, and showcase success stories shall be presented and discussed.

In special keynote talks on topics e. g. from Blockchain Technology for Enhanced Traceability, Digital Calibration Certificates (DCC), Metrological Digital Twins (M-DT), smart supply-chain-management, AI and machine learning chances, changes and limitations of these new techniques shall be highlighted. You are invited to contribute by uploading an abstract of max. 250 words to the conference management system. Please state your interest in this special session.

We are aiming to get insights from industry experts and researchers on current developments, challenges, and barriers regarding digitalization in traceability, while uncovering opportunities for innovation and collaboration in this interactive workshop.

#### Keywords:

- •Digitalization in Traceability
- •metrological digital twins
- •smart supply-chain-management
- •digital calibration certificates
- •AI and ML

### **IMEKO TC24 Chemical Measurements**

### Gas analysis for climate change and energy transition

The rise in atmospheric greenhouse gases is the primary driver of climate change. Closely monitoring the gas phase composition of the atmosphere is therefore critically important to understand and monitor climate change. Robust metrology for greenhouse gases monitoring in the air will be vital to achieve zero-pollution and carbon neutrality, ambitions laid out in strategies by both the European Union and the United Nations.

In striving towards environmental sustainability and a reliable energy network, it is vital to address outstanding fundamental challenges to establish renewable gases (e.g., Hydrogen, Ammonia, Biomethane) as a fuel source and as an energy vector. To ensure the safety and reliability of renewable gaseous fuels it is important to have robust, accurate measurements that can be traced to established standards.

#### Keywords:

- •Gas metrology
- •Climate change and air quality
- •Low-cost sensors
- •Hydrogen
- Decarbonization



	10-Oct		
	Welcome reception		
	11-Oct		
	Registration		
	Opening ceremony		
	Damiano Pietri "Digitization of laboratory processes: the ABC Bilance-Metricode case"		
	Laura Martin "EUROLAB: an international collaboration towards the Lab of the Future"		
	Coffee & Networking		
	Sunset III	Sunset II	
11:00	Gas analysis for climate change and energy transition (part I)	Digital transformation in TIC and Digital Twins	
	#4 "Nanoporous Black Gold for Hydrogen Sensing"	#22 "Validation methods in the preparation of Digital Calibration Certificates (DCCs)"	
	#8 "Climate change – cross-disciplinary metrology	#24 "The Digital Twin for frequency transfer	
	supporting its mitigation and control"	traceability"	
	#14 "Preparation of Multicomponent Mixtures to Support	#28 "Digitalzations impact on Traceability in	
	<u>Carbon Metrology"</u> #21 "Enabling measurement of CO2 purity and	Metrology"	
	composition: NPL's role as the UK National Metrology Institute"	#34 "Progress with Digital Certificates of Analysis for Reference Materials and Traceability at NIST"	
Poster session		ession	
	Lunc	h	
	Přemysl Fitl "Solid-state gas sensors and the		
	Workshop TC24 (Sampling as laboratory acti		
	Coffee & Networking		
	Sunset III	Sunset II	
	Gas analysis for climate change and energy transition (part II)	Gas-phase chemical measurements (part I)	
	#6 "Metrological Traceability in Environmental Gas	#5 "Investigation of Surface Processes on Black	
	Analysis – From SRP to TILSAM to d-TILSAM"	Aluminium Layers for Sensor Applications"	
	#13 "Production of gaseous certified reference materials at	#18 "Properties of black metal - lanthanide doped	
	INRiM for amount of substance fraction of CO2"	oxide sensitive layer toward multivariable gas sensor"	
	#27 "Traceable in-house preparation of RM CO2/N2 gas	#17 "Surface decorated black metal active layers for	
	#27 "Traceable in-house preparation of RM CO2/N2 gas mixture using gravimetric standardized method" End of fir	gas sensors"	

	12-Oct			
08:30	Registration			
09:00	Sascha Eichstädt "Digitalization of the Quality Infrastructure"			
09:45	Invited Speaker (My Green Lab)			
10:10	Workshop TC8 (Digitalization in Traceability – n	Workshop TC8 (Digitalization in Traceability – new tools, new opportunities, new challenges)		
11:30	Coffee & Ne	Coffee & Networking		
Location	Sunset III Sunset II			
11:50	Chemical measurements for health and biology	Innovation and validation of methods in testing, calibration, inspection and certification (part I)		
	#23 "Metrological traceability of moisture content measurements in plant-origin bulk materials"	#25 "Traceability and method validation of a fully automated optical process to provide the on-site measurement of waste and bulk cargo volume"		
	#29 "Chemical measurents and their applications in health biomedical sciences"	#30 "Implementation and Validation of Calibration Methods in the Area of High Frequencies"		
	#35 "Thin films gas sensors for biomedical applications"	#31 "Extreme Impedance Calibrations: Enhancement of Metrology Infrastructure"		
	#37 "Novel approaches for measurements in dentistry"	#38"The need to sensitize inspection and certification bodies in the use of decision rule to avoid poor decision making when interpreting results from certificates of analysis"		
13:00	Lunc	h		
14:45	Conference	Conference Photo		
15:00	Cultural visit (Conference Bus from the hotel)			
18:00	End of cultural visit			
19:30	Join in for Dinner (Confere	Join in for Dinner (Conference Bus from the hotel)		
20:00	Conference Dinner			

	13-Oct		
09:00	TC8, TC11, TC24 meetings		
Location	Sunset III	Sunset II	
10:00	Gas-phase chemical measurements (part II)	Quality management, conformity assessment and use of reference materials in TIC Sector	
	#9 "Laser Direct Write Treatment of Black Metals for Gas Sensing Applications"	#2 "Characterization of electronic waste materials by Instrumental Neutron Activation Analysis for Certified	
	#33 "On site measurements for the safeguard of metallic works of art"	#15 "Quality managment abd conformity assessment"	
10:50 Coffee & Networking			
cation	Sunset III Sunset II		
11:20	Gas analysis for climate change and energy transition	Innovation and validation of methods in testing,	
1.20	(part III)	calibration, inspection and certification (part II)	
	#12 "Assessment of moisture absorption by anhydrous ethanol under different environmental conditions"	#3 "Experimental determination of the absolute roughness of concrete conducts in a water supply network"	
	#26 "Preparation of stable isotope reference mixtures of CO2 in air global atmospheric monitoring"	#16 "Development of open-source tools for the digital and machine-readable calibration of flowmeters with numerical displays."	
	#36 "Metrology for hydrogen vehicle 2: new reference	#20 "Analysis of flow rate measurement accuracy	
	materials and new sampling approaches applied to	and traceability of flowmeters in field conditions	
	hydrogen fuel"	using clamp-on ultrasonic flowmeters"	
2:30	Closing ceremony		
3:00	End of Conference		



#### 11-0ct

#### Gas analysis for climate change and energy transition (part I)

Location: Sunset III Time: 11:00 Chairs: Tatjana Tomic & Leonardo Iannucci

#### Digital transformation in TIC and Digital Twins

Location: Sunset II Time: 11:00 Chairs: Marija Cundeva-Blajer & Thomas Wiedenhöfer

#### Gas analysis for climate change and energy transition (part II)

Location: Sunset III Time: 16:30 Chairs: Zhechao Qu & Leonardo Iannucci

#### Gas-phase chemical measurements (part I)

Location: Sunset II Time: 16:30 Chairs: Sabrina Grassini & Simon Peter Mukwaya

12-0ct

#### Chemical measurements for health and biology

Location: Sunset III Time: 11:50 Chairs: Emma Angelini & Julio Brionizio

#### **Innovation and validation of methods in testing, calibration, inspection and certification (part I)** Location: Sunset II

Time: 11:50 Chairs: Catarina Simões & Marija Cundeva-Blajer

#### 13-0ct

#### Gas-phase chemical measurements (part II):

Location: Sunset III Time: 10:00 Chairs: Sabrina Grassini & Tatjana Tomic

### Quality management, conformity assessment and use of reference materials in TIC Secto

Location: Sunset II Time: 10:00 Chairs: Álvaro Silva Ribeiro & Mladen Jakovcic

#### Gas analysis for climate change and energy transition (part III)

Location: Sunset III Time: 11:20 Chairs: Francesca Durbiano & Martin Hruska

#### **Innovation and validation of methods in testing, calibration, inspection and certification (part II)** Location: Sunset II

Time: 11:20 Chairs: Catarina Simões & Marija Cundeva-Blajer



#### #2

#### Characterization of electronic waste materials by Instrumental Neutron Activation Analysis for Certified Reference Material production

Authors: Marco Di Luzio<sup>1</sup>; Luigi Bergamaschi<sup>1</sup>; Radojko Jacimovic<sup>2</sup>; Giancarlo D'Agostino<sup>1</sup>

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Electronic devices are ubiquitous within the European Union and their ever increasing demand is putting a lot of pressure on the supply chain, especially for what concerns materials defined as tech-nology critical elements (TCE). Waste from Electrical and Electronic Equipment (WEEE) can provide an accessible source to gather TCE; to this end, the European Union highly encourages practices in-volving more sensible waste management aiming to recycle those elements. However, a primary metrological obstacle to the recycling lies in the lack of certified reference material (CRM) for this heterogeneous compound which makes difficult to perform reliable analytical measurements on waste samples.

Within the European project MetroCycleEU, samples from two WEEE materials were prepared and characterized to be evaluated as candidates with the aim to produce the corresponding CRMs. In de-tail, gathered materials from Printed Circuit Boards (PCB) and Light-Emitting Diodes (LED) were grinded to powder and analyzed by different analytical techniques to quantify interesting TCE present with mass fraction greater than  $1 \mu g g^{-1}$ .

In this work the adoption of relative and  $k_0$ -standardization of Instrumental Neutron Activation Analysis (INAA) to quantify TCE in candidate samples is described. INAA provides a suitable reference method for bulk analysis preventing the dissolution of sample which is usually a challenging task for highly heterogeneous materials. The optimized INAA measurement procedures are reported and results for quantified TCE in both matrices are plotted on a sample-per-sample basis; major contributors to uncertainties are identified based on the provided uncertainty budgets, which also support SI traceability.

# Experimental determination of the absolute roughness of concrete conducts in a water supply network

Authors: Luís Martins<sup>1</sup>; Álvaro Ribeiro<sup>1</sup>; Catarina Simões<sup>1</sup>; António Pais<sup>1</sup>; Ricardo Mendes<sup>1</sup>

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Water supply networks have a huge role in our society, both in urban and rural areas. Water is afundamental and basic resource for the subsistence and development of all countries and regions worldwide, justifying a dedicated attention by United Nations (UN-Water) since the 1970's. These networks include several hydraulic elements such as reservoirs, dams, wells, pumping and treatment stations, and usually have a high extension. Conducts are essential to the water transportation be- tween the mentioned hydraulic elements, from an initial stage (collection) to a final stage (customerdelivery).

From a design point of view, the friction of the water against the inner wall of conducts is a key is- sue due to the need of pumping to overcome the corresponding pressure loss along the water supplynetwork, therefore having a direct impact in construction and operation costs. The friction factor of a conduct is directly related to its roughness, being considered a complex problem in the field of fluid mechanics, usually requiring an experimental approach under restricted conditions, to obtain an accurate solution.

This paper describes the experimental determination of the absolute roughness of concrete conducts in a pressurized water supply network related to agricultural irrigation. Both the applied mathematical model (based on the Colebrooke-White equation) and the measurement uncertainty evaluation approach (using a Monte Carlo method) are described. The discussion of results includes the out-put measurement uncertainty, the analysis of the corresponding probability distribution and the identification of the main input contributions to the obtained dispersion of values.

### Nanoporous Black Gold for Hydrogen Sensing

Author: Martin Hruska<sup>1</sup>

Co-authors: Jan Kejzlar <sup>2</sup>; Jaroslav Otta <sup>2</sup>; Přemysl Fitl <sup>2</sup>; Michal Novotný <sup>3</sup>; Jakub Čížek <sup>4</sup>; Martin Vrňata <sup>2</sup>

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In the last few years, there has been a growing trend in the field of hydrogen gas detection regarding the green deal and the use of hydrogen as a next-generation energy source. The main reason for hydrogen detection is the fact that hydrogen/air mixtures are explosive at concentrations above 4%. One approach for the simple detection of low hydrogen concentrations is the usage of highly nanoporousblack metal chemiresistor films (BMs) prepared by thermal evaporation. The fractal structure and thehigh surface-to-volume ratio of the BMs are highly beneficial for gas sensing because the nanostructured surface can provide more bonding sites and sites for physical adsorption. Another advantage

is the possibility to control the level of nanostructurization. This can be done either by controlling the working pressure during the deposition or subsequently by thermal annealing. By this procedure, it is possible to increase or decrease the sensitivity to a particular analyte.

Nanostructured black gold (BAu) layers were prepared by thermal evaporation in an inert argon at-mosphere on 2x2 mm wide alumina chips with platinum electrodes. The layers were characterised by SEM, AFM and UV-VIS spectroscopy. For further characterisation, defects and nanopores in black- metal films were studied by positron annihilation spectroscopy. The sensing properties of BAu films with different porosities were investigated at different hydrogen concentrations.

It was proven that the nanoporous BAu is a suitable active chemiresistor layer for hydrogen sensingand that the sensitivity of the BAu toward hydrogen can be controlled by the nanostructurization of the prepared layer.

# **Investigation of Surface Processes on Black Aluminium Layers for Sensor Applications**

#### Author: Petr Pokorný<sup>1</sup>

**Co-authors:** Martin Hruska <sup>2</sup>; Joris More-Chevalier <sup>3</sup>; Přemysl Fitl <sup>4</sup>; Michal Novotný <sup>5</sup>; Takayuki Kiba <sup>6</sup>; Midori Kawamura <sup>6</sup>; Jan Lančok <sup>3</sup>; Martin Vrňata <sup>4</sup>

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There is an increasing trend in the utilization of highly structured materials for chemical gas sensing, because of the large specific surface, which provides a lot of space for the chemisorption or physisorption of chemical species. One such material are black metals (BMs), highly porous and nanostructured metal surfaces with fractal structure, which absorb almost all incident light as a result of the multiple reflexion and absorption in pores and cavities. BMs are promising materials for chemical gas sensor applications due to the above-mentioned facts and also because the nansotructurization of the surface can be easily controlled by deposition con- ditions (used deposition method – thermal evaporation/magnetron sputtering, used inert gas, and deposition pressure).

In this work, three different samples of black aluminium (BAI) films were prepared by thermal evaporation, DC magnetron sputtering, and RF magnetron sputtering at low temperatures, and characterized and compared in terms of surface processes and sensing capabilities. Scanning electron microscopy (SEM), atomic force microscopy (AFM) and ultraviolet-visible (UV-VIS) spectroscopy wereused to characterize the prepared layers of BAI. For the measurement of sensing capabilities, thermal and optical stimulated exoelectron emission and thermal stimulated desorption (OSEE, TSEE, and TSD) were used.

It has been shown that the sensing capabilities of nanostructured materials strongly correlate with the specific surface area, whereas the rigidity and thermal stability, which are also crucial for sensorapplications, usually go the opposite way than the increases in the specific surface area and sensingproperties.

# **Metrological Traceability in Environmental Gas Analysis – From SRP to TILSAM to d-TILSAM**

Authors: Jan C. Petersen<sup>1</sup>; Olav Werhahn<sup>2</sup>; Ravi Fernandes<sup>3</sup>; Zhechao Qu<sup>2</sup>

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National metrology institutes (NMIs) provide metrological traceability to the international system ofunits (SI) for customers. Within the framework of the CIPM MRA, these services are internationally recognized when baked-up by calibration and measurement capabilities (CMCs) published in the BIPM key comparison database (KCDB). Each CMC is a documentation of metrological traceability available to customers. Within the area of gas analysis, there are many CMCs relevant for air quality and climate change measurements in the KCDB. However, most of them are based on very stable and accurate reference material-based gas standards. Artifacts, gravimetrically generated gas mixtures with very low uncertainty of the constituents' amount fractions. A completely different approach has been taken when ozone is measured. The mole, with the symbol mol, the SI unit of amount of substance, is realised for ozone by a Standard Reference Photometer (SRP), an instrumental standard, that provides SI traceable measurement results of ozone in air. For many other important analytes, particularly in air quality context, the production of reference materials is a challenge due to their reactivity and instability. Among those are ammonia, nitrogen dioxide, hydrogen chloride and even water. In this work we propose an alternative method for measurement of amount of substance for such species. The method relates back to the ozone SRP. The Traceable Laser-Spectrometric Amountfraction Measurement (TILSAM) relies on first principles laser spectroscopy. When realized in an appropriate spectroscopy instrument it can serve as an optical gas standard. We discuss examples and the potential of a digital d-TILSAM.

#### **#7 POSTER**

# Traceable NH3 quantification and metrological uncertainty evaluation in a shock tube

Authors: Denghao Zhu<sup>1</sup>; Sumit Agarwal<sup>1</sup>; Bo Shu<sup>1</sup>; Ravi Fernandes<sup>1</sup>; Zhechao Qu<sup>1</sup>

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Ammonia (NH3) is a promising alternative fuel due to its carbon-free feature. However, the emission of ammonia is one most unwanted drawbacks and needs to be solved by quantitative measurement of its footprint in the entire energy conversion process. As in a combustion chamber, a highly dynamic reactive environment exists generally, and it is a big challenge to accurately probe the speciation variation in it. Shock tubes are typically applied to create a quasi-instantaneously and homogeneously high-temperature and pressure environment with test times of a few hundred microseconds to several milliseconds which are suitable for characterizing dynamic gas sensors. Given this, we developed and validated an ultra-rapid spectrally resolved tunable diode laser absorption spectroscopy (TDLAS)-based spectrometer with a scan frequency of 40 kHz and coupled it withthe shock tube for dynamic NH3 quantification. Thanks to the high scan frequency, the NH3 mole fraction at various stages during the dynamic process can be quantified, covering a wide temperature range of 295-3348 K and a pressure range of 0.02-3.15 bar. In addition, considering lacking metrology in shock tubes for dynamic studies, we comprehensively evaluated the uncertainty sources and bud-gets of thermodynamic parameters and species concentration based on Guide to the expression of uncertainty in measurements (GUM). The established metrological uncertainty evaluation method for shock tube experiments can be beneficial to provide traceable and high-quality data, which is vital for chemical kinetic modeling.

# Climate change – cross-disciplinary metrology supporting its mitigation and control

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Climate change provides probably the most challenging threat to mankind and life on Earth. Mitigation of follow-up effects, measurements to control actions, and monitoring the Earth system seem mandatory. Notwithstanding that each metrology discipline has its competencies, a cross-discipline approach to climate change questions is required. The Physikalisch-Technische Bundesanstalt (PTB) is Germany's national metrology institute reaching out to its stakeholders and providing metrology support to measurement tasks related to climate change. To address the cross-disciplinarity of the climate challenge, PTB has set up an Innovation Cluster Climate & Environment (ICCE) combining its scientific disciplines to address climate change metrology. The contribution showcases the ICCE structure, its liaison with CIPM MRA activities within EURAMET and the BIPM, and discusses scientific research to foster metrology solutions to measurement tasks in chemistry, oceanography and remote sensing. Combining this research with the backbone principle of metrological traceabilityto the international system of units, metrology contributes to climate change mitigation. The contribution will also present the close interaction of ICCE topics to those of digital transformation and energy supply in Europe.

### Laser Direct Write Treatment of Black Metals for Gas Sensing Ap-plications

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In the field of gas sensing applications, many approaches on how to improve sensor sensitivity and selectivity have emerged. One method with great potential is surface laser treatment, which modifies the active surface area by the formation of nanostructures.

Black gold or black aluminium layers were prepared on fused silica and glass substrates by thermal evaporation from a tungsten boat in an inert atmosphere at process pressure in the range of 100 - 1000 Pa or by magnetron sputtering in Ar-N2 gas mixture. These layers were modified and annealedusing continuous wave laser direct write (LDW). Laser fluence was controlled by altering the laser scan speed and/or changing the laser source power.

The morphologies of laser-treated black metals were characterised by Scanning electron microscopyand Atomic Force Microscopy. With appropriate LDW parameters applied on black aluminium, the formation of nanowires was observed. Aluminium nanowires have a great potential in chemiresistive gas sensor applications. In the case of LDW treatement of black gold, plasmonic structures were achieved. Tunable distributions of gold plasmonic structures have a great potential in optical or diffractive gas sensor applications. To confirm the formation of gold plasmonic nanoparticles, UV-vis spectroscopy was used. To support experimental data, computational simulations of gold plasmonic data were in agreement with the measured absorbance spectra; the plasmonic absorption peakis shifting toward shorter wavelengths with a smaller particle diameter.

#### #10 POSTER

### Regulatory framework in Spain of LNG measurement systems in service

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Climate change is affecting all countries on all continents. It is disrupting national economies and affecting different lives. Weather systems are changing, sea levels are rising, and weather events are becoming more extreme.

The fight against climate change is essential for the future of Europe and the world. The European Climate Regulation enshrines the EU's goal of achieving climate neutrality by 2050

Being climate neutral means that, by 2050, EU countries will have to drastically reduce their green- house gas emissions and find ways to offset the remaining and unavoidable emissions to reach a balance of net zero emissions.

LNG is the fossil fuel with the least environmental impact of all those used, both in the extraction, processing and transportation stage, as well as in the utilization phase.

The need to minimize the impact of the carbon footprint of the transport industry has increased the consumption of LNG in maritime transport and heavy goods transport, due to its low polluting level and great autonomy. Emerging the need for regulation at the national level of LNG metering systems once put into service

Spanish legislation is developed in Order ICT/155/2020, of February 7, which regulates the metro- logical control of the State of certain measuring instruments, in said Order it is intended to include the regulation of the instruments phase in service of LNG metering systems.

### SACAREF Internal Project: Development of sucrose solutions in water as Certified Reference Materials (CRM)

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The interest in the development of sucrose solutions in water as Certified Reference Materials (CRM)arises, on the one hand, to comply with the traceability requirements of the verifiers and repairers of the instruments used to measure the sugar content of grape must, of concentrated musts and rectified concentrates musts, as established in Annex XVI of Order ICT/155/2020, of February 7, which regulates the State Metrological Control of certain measuring instruments in Spain and, on the other hand, the need of the food sector to have traceable reference materials.

The development of the SACAREF internal project and, in general, of the CRMs, is one of the priority objectives of the Chemistry and Health Area of the Spanish Metrology Center (CEM) and will allow it to create new commercial opportunities covering the existing needs in Spain in the State Metro- logical Control activities of the refractometers, providing verifiers and repairers with the necessary solutions to give traceability to the State Metrological Control activities on the instruments destined to measure the sugar content of the grape must, concentrated must and rectified concentrated mustand on the other hand, providing traceability to the food industry.

# Assessment of moisture absorption by anhydrous ethanol under different environmental conditions

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The use of bioethanol as automotive fuel has been considered as a renewable alternative biofuel, especially in the last few years, due to many reasons, especially those related to environmental sustainability, energy security and limited availability of the non-renewable fossil fuel. The main use of bioethanol as a fuel worldwide is by adding it in gasoline. The addition of ethanol has themain purpose of increasing gasoline octane number. The bioethanol suitable for mixing in gasoline must be the anhydrous one, which according to ASTM D 5798-09b, must contain up to 1% (w/w) of water. It is well known that ethanol is a hygroscopic substance. However, no complete study can be found in literature addressing the rates of water absorption by the ethanol and its evaporation, or showing how weather conditions can influence on these factors. The evaluation of this behavior is fundamentally important for all the ethanol production and distribution chain, in order to be estimated how long an ethanol lot can be handled still keeping adequate moisture content, or even to avoid loosing significant amount of it by evaporation causing environmental problems and financiallosses. In order to evaluate the ethanol behaviour under different weather conditions, this work simulated, in a climatic chamber, environments with different relative humidity and temperatures and studied how these factors can influence the rate of moisture absorption by an anhydrous ethanolsample and/or evaporation of ethanol. It was also evaluated whether evaporation of ethanol or moisture absorption occurs in a preferentially way.

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# **Production of gaseous certified reference materials at INRiM for amount of substance fraction of CO2**

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Carbon dioxide (CO2) is one of the greenhouse gases (GHGs) that most contribute to global warming. Its concentration has grown continuously reaching an annual average of 420.41  $\mu$ mol·mol-1 in 2023. Accurate and sound determinations of the atmospheric concentration of GHGs enable the development of models to predict future scenarios and to implement effective measures to counteract global warming. For this purpose, it is very useful to have metrological references represented by gas mixtures with CO2 concentration at the atmospheric level to ensure the reliability of the results and to have the possibility to compare them internationally.

INRiM activity deals with the development of primary methods to prepare reference gas mixtures for environmental pollutants. In addition to dedicated primary methods based on gravimetry and dynamic dilution, INRiM is working to become an official producer of certified reference materi-als (CRMs) for the amount fraction of CO2 in matrices of synthetic air or nitrogen, in accordance with the requirements of the standard ISO 17034:2016. The CRMs are prepared in 5 L aluminium alloy cylinders, accompanied by a certificate of analysis that provides the value of the CO2 amount fraction, its associated uncertainty, and a statement of metrological traceability. The experimental assessment of the stability of the amount fraction of CO2 in the gas mixture under storage conditions is also carried out.

Further work is foreseen for the development of CRMs for the isotopic composition of CO2 in air ( $\delta$ 13C-CO2), after successful participation in a specific international comparison in this measurement field.

#### #14

### Preparation of Multicomponent Mixtures to Support Carbon Metrology

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The Reference Gas Laboratory (LGR) of IPQ is participating in the project MetCCUS - Metrology for Carbon Capture Utilization and Storage under the new EPM (European Partnership on Metrology) Program. This project started on October 1, 2022, with the participation of 22 partners and will lasts for 36 months. The goal of this project is to develop a metrological infrastructure that enables monitoring and detection of carbon dioxide leaks in energy and industrial processes and in transport networks. It also aims to foster the use of carbon dioxide removal by enabling capture, utilization, and storage, supporting a better

understanding of the life cycle of carbon dioxide. The contribution of LGR involves the preparation of certified reference materials (CRM) to allow the measurement of impurities in CO2 with metrological traceability, providing support for the calibration and validation of instrumentation used in carbon capture processes. LGR had prepared the following multicomponent mixtures:

- H2S in CO2 matrix
- SO2 in CO2 matrix
- SO2+H2S+CO+O2 in CO2 matrix

These reference gas mixtures were prepared at the highest level of accuracy by gravimetric methodbased on the international standard ISO 6142-1. The composition of gas mixtures was certified by analytical reference methods according to ISO 6143. These CRM provide national and international recognition of the calibration and measurements capabilities (CMC).

This communication intends to describe the work carried out by LGR performed under the MetCCUS project that will contribute to improve the accuracy, reliability and traceability of measurements related to carbon metrology.

### Quality management and conformity assessment

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quality management is set of activities of an organism to maintain and improvement a level of excel-lence by setting a strong quality planning, quality assurance and quality improvement in establishing policies, guidance and procedures and also implementation of standards requirements.

In summary to achieve those objectives the aim of the conformity assessment is to determine whether the quality system conform to specified requirement related to product, process, system, person or body by using the different techniques like testing inspection, validation, verification, certification, and accreditation.

#15

### Development of open-source tools for the digital and machinereadable calibration of flowmeters with numerical displays.

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The Industrial 4.0 era and particularly the Industrial Internet of Things (IIoT) lead to an unprecedented ability to use different types of sensors and generate enormous amount of measurement data, requiring traceability. The heterogeneous nature of the equipment and the laboratory with inadequate Information Technology (IT) penetration, is often the bottleneck for a fully digitalized calibration process.

The development and establishment of digital processes and digital infrastructures offers enormous potential for overall calibration process efficiency. However, this process is often doomed to manual interaction and is usually labor-intensive. It is possible to reduce human interaction and increase process efficiency by increasing the infrastructure's IT penetration, towards a fully digital and machine-readable calibration process.

The current accessibility to technology combined with the maturity of open-source software, including AI, allows the introduction and interoperability of digital processes with available-of-the-shelf hardware and state-of-the-art software, with reduced financial investments. The method proposed for the calibration of flowmeters with numerical displays, aims to aggregate and automate in a single computer all the data collection and storage of a typical decentralized and manual calibration process, gathering images remotely with cameras (image synchronization is discussed), followed bypre-processing the images and obtaining the machine-readable readings from the numerical displays by means of Optical Character Recognition (OCR).

This method is scalable to virtually any number of cameras and can be applied to laboratory calibration and *in-situ* measurement processes. The software is developed with open-software solutions implemented in *Python*, with *OpenCV* for image pre-processing and *Tesseract* for OCR implementation.

### Surface decorated black metal active layers for gas sensors

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The focus of this contribution is on the nanostructured black metal active layers decorated with MXenes. The black metals are used for gas sensors functioning as chemiresistors, meaning that the change in the chemical composition of the atmosphere causes a certain shift in the electric resistivity of the sensor. The sensoric response can be further improved by decorating the black metal surface with suitable compound. For this purpose, the hybrid MXenes were chosen.

The samples of the black metals were prepared on glass, fused silica and silicon substrates using thermal evaporation in the inert atmosphere of argon (30 Pa). The MXenes were synthesised from the Ti3AlC2 MAX phase by selective etching of aluminium following the MILD etching procedure. The resulted colloidal solution of Ti3C2Tx MXene sheets was used for fabrication of Ti3C2Tx thin films. The contact gold electrodes with thickness of 200 nm were prepared by magnetron sputtering either directly onto the substrate surface or onto the prepared MXene layer.

Prepared samples were measured for their sensory properties, their surface morphology via scanning electron microscopy (SEM) and their chemical composition by energy-dispersive X-ray spectroscopy (EDXS). As model gas pollutants, NOX and NH3 gases together with water vapours were selected. The samples were also measured for their stability in the atmosphere of synthetic air.

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# Properties of black metal - lanthanide doped oxide sensitive layer toward multivariable gas sensor

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Black metals (BMs) exhibit highly porous surface (cauliflower morphology structure) resulting in large surface area. This property makes BMs interesting in utilization in chemical gas sensors, where they can improve on sensitivity because of the increased area of sorbent. BMs can be used in quartz crystal microbalance (QCM) sensors and chemiresistors. Lanthanide doped nanostructured oxides (eg. TiO2 and ZnO) may be utilized as semiconducting luminescent sensitive layer in gas sensors based on the response detection by both electrical and optical approaches. Synergic combination of the BMs and the oxide layers possess potential toward development of novel multivariable single gas sensor employing measurement of photoluminescence combined simultaneously with eitherelectrical (AC/DC) conductivity or sorption processes by QCM.

BMs were prepared by vacuum thermal evaporation or magnetron sputtering. Sm and Eu doped TiO2 and ZnO films were fabricated by pulsed laser deposition (PLD). Pulsed laser annealing (PLA) was performed to enhance luminescent optical properties. In-situ optical properties monitoring wasused to optimize PLA conditions. Multilayer structure consisted of the oxide film deposited on the BM layer.

The properties of particular layers as well as multilayer structure were characterized by AFM, SEM, XRD, thermally stimulated exelectron emission and desorption, spectrophotometry, spectral ellipsometry, Raman spectroscopy and photoluminescence. Regarding sensory properties focus was on detection of oxygen.

#### **#19 POSTER**

# Polymer ionic liquids as perspective materials for chemiresistors and QCM sensors

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Polymerized ionic liquids (PILs) have been reported for the first time in 1998. Their electrotransport properties are unique (when compared with other organic substances), as they are purely ionic conductors. Moreover, majority of them can be considered as single-ion conductors. PILs are characterized by large capacity to absorb analytes with small molecules – especially CO2 and water. When this absorption takes place, the internal volume of polymer is modified, hence the mobility of ions changes. Such a phenomenon could work as a prospective transducer mechanism in chemiresistors. This property of PILs is very valuable, because namely CO2 and numerous other analytes (whose molecule has neither redox properties, nor dipole moments) cannot be detected on chemiresistors by any other mechanism.

In the field of chemical sensing, PILs are often employed in electrochemical sensors, but much rarelyfor sorbents in QCM sensors. Surprising still, their applications in chemiresistors are at a pioneeringstage.

This contribution presents the research of chemiresistors and QCM sensors with sensitive layers

based on poly (tetrabutylphosphonium 3-sulfopropylacrylate) marked as poly(P4,4,4,4SPA) and poly(tributyloctylphosphonium 3-sulfopropylacrylate) marked as poly(P4,4,4,8SPA). The response of prepared sensors to noble gases, hydrogen, carbon dioxide, alkanes and methanol vapours was evaluated by impedance spectroscopy. The purpose was to study the consequences between the molecular weight and/or chemical reactivity of the analyte on one side and the sensing mechanism (modulation of electrotransport parameters of PIL in chemiresistor; modulation of the amount of analyte absorbed in

PIL in QCM) on the other side.

### Analysis of flow rate measurement accuracy and traceability of flowmeters in field conditions using clamp-on ultrasonic flowmeters

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Hydraulic infrastructures often have flowmeters installed in pipes with local conditions preventing their removal for calibration in the laboratory. Flow rate measurement in these places is needed to provide data for diverse tasks in water management, including commercial transactions, system controls and for calculating water balances. Flow rates are variable over time, resulting in time series whose analysis depends on data quality.

Clamp-on ultrasonic flowmeters are an alternative to assess measurement quality in the locations where measurements are needed, allowing non-invasive assessment of measurement quality of flowrate and velocity. Advantages include avoiding pressure drops in the pipe, easy installation and readjustment to the pipe. The procedure must follow some specific rules to ensure the quality of collected data using this method. These are related to fluid characteristics, installation setup, and pipe characteristics.

Flow behaviour and regimes in non-laboratory conditions are typically not under control, and there is a need to study how, under those conditions, to assess the traceability of installed equipment and how to estimate the accuracy of the measurements. In this sense, it is pertinent exploring how the process of time series of flow data contributes directly to the performance and quality of the measurement results.

In this context, measurement uncertainty plays a central role. This paper describes how the sourcesof uncertainty are characterized and how they are combined to determine the uncertainty associated with the measurement results, thus influencing the decision-making processes

#20

# Enabling measurement of CO2 purity and composition: NPL'srole as the UK National Metrology Institute

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Crucial role of carbon capture, utilization, and storage (CCUS) in achieving energy security and reduce fossil fuel based emissions is now beyond a doubt. The governments across the world and various CCUS stakeholders have fast faced their efforts to capture anthropogenic CO2 to achieve Paris climate agreement objectives and to minimize effects of climate change. Capturing CO2 using various technologies such as post/pre combustion, oxy-fuel, negative emissions have been proposed as a promising option to reduce the CO2 emissions and minimize the amount of CO2 in the atmosphere. In the UK, government has an ambition to capture and store 20-30 Mt of CO2 by 2030 and 50 Mt by 2035. However, there is a lack of traceable measurement science infrastructure to accelerate successful implementation of CCUS in the UK and beyond. Across the CCUS value chain, be itcapture, transport, utilization or storage, CO2 purity plays a significant role. To support industry to adapt to this emerging and challenging technology, NPL's Gas Metrology Group (GMG) is actively working with various stakeholders to develop necessary metrological infrastructure. GM is developing various analytical techniques, primary reference gas mixtures (PRMs) to support CO2 purity analysis. NPL is also developing materials testing platform to support stakeholders to understand capture efficiency, degradation products, material stability and CO2 purity of their technology. This presentation is going to give overview of GMG's activities in developing metrological capabilities to support CCUS industry within the UK and beyond.

# Validation methods in the preparation of Digital Calibration Certificates (DCCs)

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Calibration certificates are the heart of the science of metrology. Unfortunately, they are still paper based. Luckily, there are many efforts to change this. One of them is the Digital Calibration Certificate (DCC). The DCC is an endeavour to digitalise calibration certificates, developed by the Physikalisch-Technische Bundesanstalt (PTB) with the contributions of national and international partners. The DCC relies on XML format. Based on this, an XSD (XML Schema Definition) is devel- oped. The XSD defines the structure of the XML files. This allows validating the XML files (DCCs).Many errors in the document can be caught with schema validation. This includes unfilled manda- tory elements or incorrectly arranged XML elements. This form of validation of the structure of the XML file against an XML schema is offered by almost every advanced editor. Going beyond schema validation, there is also the possibility of validation with Schematron. Schematron is a schema lan- guage which is used to write logical rules for XML files. Therefore, with Schematron, an XML filelike the DCC can be checked for logical errors. For this, individual rules are created which can either throw a hint, a warning, or an error. This is a very elegant way to check the XML. Every DCC creator is free to write additional Schematron rules for his own use case. The PTB's DCC Team has prepared a Schematron validation tool by using open-source resources. This presentationshows which errors can be found through schema validation and which errors can be found through Schematron validation.

### Metrological traceability of moisture content measurements in plantorigin bulk materials

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The moisture content is one of the most important characteristics of plant-origin bulk materials, which is necessary to support fair trade in the grain market. To determine moisture content, industrial laboratories mostly use reference or absolute methods described in ISO standards. In the reference method, a test portion is dried at a temperature of about 130 °C for a few hours. In the absolute method a test portion is dried under reduced pressure and kept at about 50 °C for up to one month, until the constant mass is reached. The main disadvantage of reference methods is that the completeness and specificity of moisture removal is not guaranteed. Absolute methods are difficult to perform and require considerable time.

The term "moisture" is generic and, to have proper Calibration and Measurement Capabilities (CMCs) and Certified Reference Materials (CRMs), a better specification of the measurand should be given. Currently, no CMCs for moisture content measurement in the plant-origin bulk materials, as well as respective CRMs, are available in the KCDB. Undoubtedly, those CMCs and CRMs are cruciallyneeded to provide metrological traceability in this area.

Karl Fischer titration allows to measure the water content in materials by relying on a selective chemical reaction and on the end point determination of the reaction by potentiometry. This absolute method is suitable to carry out measurements having a sound metrological basis and it represents avaluable solution for the production of CRMs, providing traceability to SI units.

### The Digital Twin for frequency transfer traceability

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Ultra-stable time and frequency sources, primary time standards or even commercial time standards, contributing to the realization of the UTC, have an important role in fundamental physics and in time, frequency and other metrology areas. In Electrical Metrology, the most accurate standards are the quantum standards, in particular the Josephson voltage standard, which requires the best frequency stability to generate the volt unit.

To give traceability to the Josephson voltage standard, the Time and Frequency Laboratory install an optical fiber between the two laboratories to transfer the standard frequency from the Cs clocks to the Electricity Laboratory. In general, the frequency stability of an oscillator in free mode, with small integration times, is better than the frequency stability of an oscillator that is at the end of a transport medium, due to the noise introduced by this medium.

In this work we duplicate this frequency transfer system with is digital counterpart, the Digital Twin. To realize this, we incorporate the necessary software and hardware components that enable the digital twin to interact with the physical twin that it relates to. Moreover, all uncertainty components will be part of the digital twin, allowing us to verify if there are non-orthogonality between any of the uncertainty components or if any of the uncertainty sources have greater impact than others in the final uncertainty budget. The use of DT allows a deeper knowledge of our system performance, prevent malfunctions or failures and to increase efficiency achieving better stability in frequency transfer.

### Traceability and method validation of a fully automated optical process to provide the on-site measurement of waste and bulk cargo volume

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The circular economy close-up linked to Europe's wide climate agenda and sustainable development goals, conceptualizes resource efficiency and sustainability, promoting that waste can be turned into a resource by reusing, repairing, refurbishing and recycling existing materials and products. Identified gaps in its roadmap includes problems resulting from the non-interoperation of data (waste and bulk cargo) and the lack of accurate data for illegal discharge prediction and logistic threads on bulkcargo transportations.

The project DiCiMa - Digitalisation and Circularity for Maritime as-built information is a disruptive solution for the integration of geometrical data acquisition and data interpretation of waste and bulkcargo, within the prism of circularity, focused on the domains of waste and bulk cargo data management at Maritime Ports. This project considers the complexity and time-consuming task that is measuring and interpreting the physical environment in maritime ports, with the on-site measuringperformed by a human operator, and the data transferability subjected to manual procedures and errors.

The solution developed within the scope of the project, already applied in maritime ports, is based on the use of LIDAR combined with algorithms and mathematical models, aiming at the determination of waste and bulk cargo volume in containers in a fully automated and rigorous way, requiring metrological traceability and the development of the method validation.

Considering the contractual and legal implications of the use of this measurement process, the accuracy of the measurement approach becomes of major relevance to providing confidence and trans- parency given to clients and stakeholders.

## Preparation of stable isotope reference gas mixtures of CO2 in air for global atmospheric monitoring

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Global warming caused by excessive emissions of greenhouse gases has emerged as one of the biggest environmental challenges threatening the world. Human activities like burning fossil fuels, using of various chemicals agriculture and other industrial activities have increased concentration of green-house gases such as CO2, CH4 etc. in the atmosphere. CO2 is the largest source of green house emissions, with a share of about 72%, followed by CH4 (19%). A new metrological infrastructure is primarily necessary, since it can contribute to verification of emissions. It can be achieved by developing new stable isotope reference materials, calibration methods and performing isotopic measurements of these components as traceable to the SI [1-2]. In this study, static gas reference materials of CO2 with different isotopic compositions at 410  $\mu$ mol/mol were prepared in CO2 free air matrix. The static reference gas mixtures were produced gravimetrically in accordance with the ISO 6142-1standard [3] at three different isotopic compositions (covering the range -42 ‰ to +1.5 ‰ vs VPDBfor 13C-CO2 in treated aluminum cylinders. Pure CO2 gases from SIAD Company and air free ofCO2 provided by NPL were used in the mixture preparation. The stability measurements of the static gas mixtures were performed over the 18 months using GC-IRMS facility for the target uncertainties 0.05 ‰ for 13C-CO2 during the stability period.

## Traceable in-house preparation of RM CO2/N2 gas mixture using gravimetric standardized method

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The necessity for national and international programs to monitor the levels of carbon dioxide emissions in the atmosphere has arisen as a result of the recent large greenhouse gas emissions that cause a rise in the Earth's temperature and climate changes with severe impacts. In order to give confidence in the monitoring results and enable the proper decisions to be made regarding the supply of environmental treatment and air quality through the limiting and monitoring of emission, it is re- quired to track the measurement data to SI units. Gas measurements laboratory at SASO-NMCC uses gravimetric method to prepare a reference gas mixtures of CO2 in N2 as CRM cylinders based onuniversal gas law. The used method fully complies with ISO 6142. Description of the steps of the pro-duction process and its method verification as well as equipment used and associated uncertainty are presented in this work. The detailed description of the used processes; automatic weighing, gas filling, mixing and homogenization processes and the used equipment are shown. In accordance with the requirements of ISO 6143, a validated gas chromatography thermal conductivity detector (GC- TCD) method was selected to verify the mole fraction of the gravimetrically prepared gas mixtures. Reproducibility of the produced concentrations is demonstrated through mid-term and long-term evaluations. Six certified reference materials (CRMs) of different concentrations were used for the GC calibration to provide metrological traceability of the measurement results to SI units. Associated uncertainty budget with description of different components is presented.

### Digitalization impact on Traceability in Metrology

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In recent years, the field of metrology has undergone significant changes and advances with the rapid transformation brought about by digitalization, particularly in the area of traceability. Digital twins, virtual replicas of physical objects, have become increasingly popular in metrology, allowing for more efficient and accurate calibration processes. While some areas have been using digital twins for decades, such as virtual CMM, their development and use are rapidly growing in other areas.

Another key development in digitalization is the use of digital calibration certificates, which offer several advantages over traditional paper certificates. These digital certificates can be easily accessed and stored in a secure database, such as a blockchain-based system, reducing the risk of loss or damage. Additionally, they can be easily shared with stakeholders, such as customers or regulators, improving transparency and traceability. The talk will highlight different approaches and developments, discussing new opportunities, as well as risks and overhyped impressions.

The digitalization of metrology is also linked to the recent fundamental update of the International System of Units (SI) in 2019. This redefinition was based on the use of fundamental physical constants, providing a more stable and precise basis for metrology.

Overall, the digitalization of metrology has great potential to improve the efficiency, accuracy, and traceability of calibration processes. As technology continues to advance, it is likely that digitalization will continue to play an increasingly important role in the field of metrology.

### Chemical measurements and their applications in health and biomedical sciences

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Chemical measurements are the cornerstone of many modern techniques for diagnosis and treatment of diseases. Chemical measurements help physicians diagnose and understand diseases and analyze biological samples such as enzymes, bacteria, and blood. Results upon which doctors base to make medical decisions heavily rely on accurate and precise measurements. For instance, errors in measurement of a drug for treatment can lead to overdosage or under dosage hence delays in the right treatment. Moreover incorrect doses of radiation exposure can harm the cancer patients they were trying to treat. Consequently, it is evident that a minor deviation in the correct value of a measurement in medicine may evolve into loss of a life. It has been mentioned that the value of accurate and reliable chemical measurements has been strongly appreciated and implemented in military and industry and less so in health and biomedical field. There is need for a strong and ardent implementation of reliable and sufficient accuracy in chemical measurements in this field through properequipment maintenance and verification, correct and continued calibration done by an accredited organization that provides traceability to known standards. Moreover, information has to be availed to physicians and other biomedical scientists to guide on the need for proper consideration of accuratechemical measurements to increase trust and acceptability of results from one clinic or physician byanother. This article will shine light on the practical implications of accurate and reliable chemical measurements in health and biomedical sciences as a tool for proper implementation.

### **Implementation and Validation of Calibration Methods in the Area of High Frequencies**

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The expansion of production facilities in the automotive supply chain in recent years in the region of South Eastern Europe has increased the need for the development of testing and calibration facilities for advanced electronic components. However, the conformity assessment bodies in the field of electronic devices are insufficient in the region of South Eastern Europe. This implies the necessity of upgrading the capacities of testing and calibration infrastructure in the area of testing electronic components. One of the most demanding fields is the calibration of testing devices, such as oscilloscopes, counters and function generators, for high frequencies. In this paper, novel calibration methods for instruments for high frequencies over 100 MHz up to GHz level, in compliance with the Calibration Guide EURAMET cg-7 Version 1.0 (06/2011), developed at the Laboratory for Electrical Measurements at Ss. Cyril and Methodius University in Skopje, will be presented. The establishment of an unbroken measurement traceability chain at high frequencies is an international metrological challenge. The evaluation of uncertainty due to significant and with unknown properties contributing influential factors, represents a computationally intensive modeling task. As a results of the lack of appropriate metrological facilities, validating developed calibration methods poses further implementation obstacles. In this contribution, the methodology for overcoming these issues and the obtained metrology outputs in the Laboratory of Electrical Measurements at Ss. Cyril and Methodius University in Skopje, will be elaborated and discussed.

## **Extreme Impedance Calibrations: Enhancement of Metrology Infrastructure**

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The overall aim of the metrology is obtaining as accurate as possible measurement results and pro-viding their adequate interpretation. Metrology infrastructure is constantly enhancing in two directions: 1-enabling measurement capabilities for extreme (very high or very low) physical quantities not covered by the existing laboratory facilities, or 2- increasing the measurement accuracy through decreasing measurement uncertainty by innovation, improvement or modification of already existing measurement procedures. In South-Eastern Europe the metrology infrastructure for electrical quantities is underdeveloped i.e., certain essential areas of electrical instruments' calibrations are not sufficiently covered by the existing metrology laboratories. However, the regional economy and science are expressing evident needs for this kind of calibration and testing facilities. The paper aim is to present the boost of the calibration and measurement capabilities for extreme electrical impedance. The paper presents the challenges faced in developing this infrastructure and how they are being overcome. The accredited Laboratory for Electrical Measurements at the Ss. Cyril and Methodius University in Skopje is introducing novel calibration procedures of measurement instruments for very high and very low electrical resistance and inductance. The validation and verification procedures for establishment of traceability and the innovative techniques for estimation of measurement uncertainty for calibration of impedance instruments will be presented. The final objective of the research is the accreditation of novel calibration methods in the Laboratory for Electrical Measurements and contributing to the capacity building of the metrology infrastructure in the field of extreme electrical impedance, in particular in the region of South-Eastern Europe.

#### **#32 POSTER**

### Quartz Crystal Microbalance Sensors with Nanoporous Coatings: An Impedance Analysis Study

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Quartz crystal microbalance sensors (QCM) are characterized by many potential applications. They are widely used as biosensors, for combustion control, environmental pollution monitoring, or gas sensors. Regardless of the specific application, the recognition resolution of QCMs is determined and restricted according to the Sauerbray equation. This fact is crucial mainly for gas sensor applications where low concentrations need to be detected, and the mass of adsorbed analytes does not usually lead to a sufficient sensor response. Yet, there are some possibilities for how to enhance the QCM sensor properties and amplify their sensitivity, for instance by increasing the specific surface area and thus providing more bonding sites for the analytes. One such approach is the use of highly nanoporous metal layers, namely black metals (BMs), due to their fractal surface and high surface- to-volume ratio. However, BMs also show lower rigidity and impaired viscoelastic properties with respect to QCM oscillations, and thus they can cause damping as shown by the lower quality fac-tor. For these reasons, proper investigation of those processes is necessary to optimise the black metal preparation conditions and utilisation of black metal based QCM sensors. Herein we present a detailed study of QCM sensors with nanoporous black metal coatings using impedance analysis. The influence of the thickness of the black metal layer and the preparation method on the quality factor of the QCM is compared and discussed. Also, a novel impedance data fitting method for the Butterworth-van Dyke equivalent circuit model is presented.

### On site measurements for the safeguard of metallic works of art

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Non-invasive on site measurements are important tools to obtain valuable information on the artefact conservation state and on the environmental conditions to which the objects are exposed to, allowing long-lasting monitoring campaigns to be scheduled.

The research group CoMeTA (Corrosion Measurements Tool for Artefacts) of Politecnico di Torino performed several measuring campaigns on bronze artworks and weathering steel structures ex- posed indoor and outdoor, in Italy and abroad, by a multi-analytical approach.

Electrochemical impedance spectroscopy (EIS) is employed to study the corrosion processes affecting metallic artworks, to assess the stability of the corrosion products and the protective effectiveness of protective coatings; X-rays fluorescence (XRF) and Raman Spectroscopy are employed for chemical and microstructural characterisations. Moreover, a 3D photogrammetry survey is carried out to create a complete documentation of the artworks. At the same time, the microclimatic conditions to which the artefacts are exposed to were monitored by specifically-designed smart sensors. The combination of all these techniques allows identifying which corrosion products are present on the metallic surface, to characterise their chemical and microstructural features, together with their electrochemical stability and to correlate them to the exposure conditions, by integrating all the analysis on a virtual 3D model, that can be stored and shared with curators and conservators.

The obtained results of the on site monitoring campaigns will be presented and discussed, highlight-ing the advantages of the proposed approach and of the developed measuring devices, and discussing the challenges still open in the development of tailored safeguard methodologies for metallic artefacts.

### **Progress with Digital Certificates of Analysis for Reference Materials and Traceability at NIST**

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NIST is in the early phases of the digital transformation of metrology within its measurement ser- vices. Two key NIST services are the calibrations, of which there are over 400 services in nine different areas, and the standard reference materials (SRMs), of which NIST has more than 1000 in its catalog. Two separate but related projects are underway in each of these services: first, to develop atool to both produce human readable calibration reports and NIST digital calibration reports (NDCR); the second is to produce a digital certificate of analysis (CoA) for our SRMs. Challenges for the calibration projects include the wide variety of reports NIST produces; a standard template must be developed for the human readable reports, and the elements of the reports must also be mapped into a digital report. NIST will leverage the XML schema DCC presently under development by PTB for calibration reports as much as possible. For the digital CoAs, the metadata descriptors for reference materials are quite different and the DCC schema will need CoA-specific extensions. Demonstrating traceability to the SI is fundamental to both SRM products and calibration services and a digital representation of this traceability could be an important feature of digital certificates. Concerns regarding privacy and BII is particularly a challenge to calibration services. In this presentation, the NIST progress and status of these projects will be reported, with a perspective of some of the key challenges regarding the digital representations of traceability that are presently perceived.

### Thin films gas sensors for biomedical applications

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Metal oxide (MOX) sensors are very promising solution for gas monitoring for biomedical applications. These sensors can be manufactured at a low-cost and their structure is suitable for miniaturization of the devices, a factor that is extremely important in portable and low-power applications. Moreover, the sensing performance and the target gas of this type of sensors can be widely modified acting on the sensing material, which is typically a metal oxide compound deposited over the sensorelectrodes in the form of thick or thin layer. The recent development of nanostructured materials is quickly improving the performance of such sensors, especially in terms of sensitivity and selectivity, making them a good and flexible solution for a wide range of gas sensing applications. The sensing mechanism of MOX sensors is generally well understood, and it is based on the presence of vacancies and broken bonds on the sensing film surface. Such defects act as bonding sites for the gas molecules with the consequent modification of the band structure of the material. Therefore, the conductivity and the sensing film resistance change as consequence of the interaction between the bonding sites and the gas molecules. Among the several materials which can be employed for these category of gas sensors, the niobium oxide is a material not frequently employed and whose performance are still not fully assessed. for the development of an acetone gas sensor depositing by reactive plasma sputtering a Nb2O5 thin film deposited over a commercial alumina substrate.

## Metrology for hydrogen vehicle 2: new reference materials and new sampling approaches applied to hydrogen fuel

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Hydrogen fuel cells are an alternative power supply for electric drive trains and could represent 32% of fuel demand by 2050. To deploy fuel cell electrical vehicles, there is current regulatory barriers (ISO 14687, ISO 19880-8, ISO 19880-1, ISO 21087, OIML R139-1) that requires accurate measurements. The European funded project European project MetroHyVe 2 has provided solutions and improve- ments in the four measurements challenges (flow metering, quality control, sampling and fuel cell stack testing). The presentation will provide a comprehensive overview of the project achievements. The presentation would focus around achievements on hydrogen fuel inter-laboratory comparison (gas intercomparison challenges including preparation of batch of gas cyliunders), new reference materials for hydrogen fuels (multicompounds, 1 year stability study results, > 7 different types of gas cylinders and passivation) and the results of the first hydrogen sampling inter-comparison. The stud-ies showed that it was possible to stabilise more than 80% of the regulated compounds for hydrogen fuel into one gas cylinders at nmol/mol to µmol/mol level with 1 year stability period. It identified the challenges around the realisation of an hydrogen fuel sampling intercomparison at 700 bar in real condition and supported evolution of approaches and methodologies. The overall achievements of the projects impacted the whole realisation of hydrogen fuel quality and were already integrated into new project as HyQuality.

### Novel approaches for measurements in dentistry

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Oral diseases represent one of the most prevalent health conditions worldwide, affecting people throughout their lifetime, with important impacts on their life quality. Because of this, it is of paramount importance to develop new approaches for non-invasive measurements in dentistry. Specifically, in the last years research has focused on the development of new measurement proto- cols which avoid the use of ionising radiations. Chemical measurements can thus play an important role in this field, providing important information on the health status of dental tissues. This contribution will present some of the latest results obtained in the last years in the field of impedance spectroscopy applied to the detection of dental caries. Models to describe the electrochemical system will be shown and the possible development of ad-hoc instrumentation will be presented. Moreover, the use of Raman spectroscopy to monitor enamel demineralisation will be presented, showing some case studies on ex-vivo samples.

This work was supported in part by the Italian Ministry of Foreign Affairs and International Cooperation, grant number PGR US23GR06, in the frame of the Project 'Innovative materials and techniques for dental health (IMT4DeH)'.

# The need to sensitize inspection and certification bodies in the use of decision rule to avoid poor decision making when interpreting results from certificates of analysis

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Inspection and Certification bodies play a big role in determining products and goods that go to the markets in different countries. They use certificate of analysis from different laboratories of which a number of these certificates bear results without conformity status and that is to be determined by user of the certificate and it is worse when the testing laboratory do not include measurement uncertainties. This puts a dought on how accurately the user bodies can interpret these results, because any result must have uncertainty of measurement, a characteristic that can help in decidingwhether the sample is failing or passing depending on the decision rule stated. This rule is well catered for in the ISO/IEC 17025:2017, meaning that only laboratories can know how to use this rule leaving out the users of these certificates produced by labs not knowing how to use this rule and more so it is not mentioned anywhere in the standards being used by the certification and inspection bodies. This puts a high risk on health and environmental safety to countries that rely ondecisions made by these bodies in allowing good and product to go to the market because a number of substandard and dangerous products can be consumed by their citizens.

### Validation of a Method for the Extraction and Quantification of Water-Soluble Chloride in Cement by Ion Chromatography

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Chloride is a corrosive anion that can attack steel and other metal embedded materials in concrete structures leading to its degradation. Chloride in concrete can come from various sources and one of them is from the materials used in making concrete such as cement. Hence, it is important to have a reliable method for determining chloride in cement. Water-soluble chloride refers to the amount of chloride that is dissolved in water after extraction. This parameter is one of the commonly requested analyses in our laboratory by cement manufacturers, cement importers, and construction companies. In this study, we used the ASTM C1218/C1218M-20 method as a guide for sample ex- traction and quantification of water-soluble chloride in cement. Validation of this method beginningfrom sample preparation to instrumental determination by ion chromatography was conducted to evaluate the fitness-for-purpose of the method. Performance characteristics assessed include working range, detection limit, repeatability, intermediate precision, and trueness. Based on the statistical evaluation of validation results, it was found that the method was suitable to be used for the determination of water-soluble chloride content in cement. This method was applied in ten different cementsamples which were found to contain 33 - 84 mg/kg water-soluble chloride.

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