

INRiM activity report in the field of mass and related quantities for the years 2021 - 2023

19th meeting, Thursday 25 – Friday 26, May 2023

Working Group on Density and Viscosity (CCM-WGDV)

Main research and development activities

The thermodynamic modeling of seawater poses many problems since the system is composed by a large number of different dissolved salts in small quantities, but strongly interacting. For this reason, a complete description of the interactions as a function of the composition has been avoided in favor of the adoption of salinity as a new independent variable, defined on the basis of chemical properties.

Since 2010, the thermodynamic of seawater has been described by the Thermodynamic Equation of Seawater - 2010, or TEOS-10, in terms of absolute salinity, S_A , defined as the mass fraction of dissolved material per kilogram of seawater. However, the validity of TEOS-10 and the experimental values in the literature do not include the liquid metastable region.

In this framework, a consolidated experimental apparatus for measuring the density of liquids, such as high pressure and metastable states, was exploited to measure thermal properties of seawater ($S_A = 35.158 \text{ g kg}^{-1}$). Measurements were carried out by means of a calibrated pycnometer of known volume as a function of temperature and pressure. The mass of seawater was determined by the gravimetric method. Seawater density was obtained for temperatures between (261.15 to 313.15) K and in the pressure range from about (21 to 110) MPa, thus partially covering the supercooled region, with an estimated expanded relative uncertainty of 0.03%, with $k=2$. The experimental values were compared to the equation of state of TEOS-10 and, considering the experimental uncertainty, all the measurements are in agreement with TEOS-10 data within $\pm 0.02\%$.

Participation in the EMPIR project 20IND13 SAFEST “Sustainable advanced flow meter calibration for the transport sector” the main objective of this project is to provide the foundation for advanced flow metrology in road and maritime transport sector, leading to reliable fuel consumption measurements. Part of the project deals with the study of the effects of viscosity and density on flow measurements. In this project, density and viscosity measurements are carried out on fuels, to prepare a matrix of the transport properties of alternative fuels, e.g. bio-diesel (FAME), bio-alcohol and synthetic fuels. This should allow the transfer of calibrations between fuels and extrapolation to higher temperatures and pressures. INRiM is leader of the WP3 “Transport properties of alternative and synthetic fuels”, which addresses of these measurements.

Peer reviews (received/conducted)

- CENAM (Mexico), peer review viscosity laboratory (conducted).
- Peer review mass and density laboratories (received).

Key comparisons

- EURAMET Key Comparison EURAMET.M.D-K4.2020: Hydrometer calibration comparison from 600 kg/m^3 to 2000 kg/m^3 . Andrea Malengo *et al.* | 2022. METROLOGIA 59 07005 (INRiM Pilot laboratory).
- CCM.D-K1.2023 Density measurement of a silicon sphere (INRiM Co-pilot laboratory).

Publications

- R. Romeo, P. A. Giuliano Albo, S. Lago, *Density and derived properties of standard seawater up to high pressure in stable and metastable states*. DEEP-SEA RESEARCH PART I-OCEANOGRAPHIC RESEARCH PAPERS - 177 (2021) 103624.

Working Group on Fluid Flow (CCM-WGFF)

Main research and development activities

Participation in the EMPIR project 20IND13 SAFEST “Sustainable advanced flow meter calibration for the transport sector”. the main objective of this project is to provide the foundation for advanced flow metrology in

road and maritime transport sector, leading to reliable fuel consumption measurements. A new test bench for calibrating flow meters under dynamic conditions and with liquids other than water was designed and built at INRiM.

Participation in the EPM Project 21GRD06 MetCCUS “Metrology Support for Carbon Capture Utilisation and Storage”, which has the aim to provide metrological support for the CCUS developments.

A study was conducted to verify the feasibility of using clamp-on type ultrasonic flow meters for the periodic verification of heat meters.

Developments of the large piston prover for calibration of MFCs. New software for the small piston prover; research on the properties of calibrated leaks.

Coordination of the EURAMET project 1557 “Revision of EURAMET Calibration Guide No. 21 - Guidelines on the Calibration of Standard Capacity Measures Using the Volumetric Method”.

Key comparisons

- EURAMET Project 1525 Evaluation of repeatability measurement procedures in gravimetric volume calibrations of glassware.

Peer reviews (received/conducted)

- CENAM (Mexico), peer review volume laboratory (conducted).
- MIRS (Slovenia), peer review mass laboratory (conducted).

Publications

- G Ficco, A Frattolillo, A Malengo, G Puglisi, F Saba, F Zuena, *Field verification of thermal energy meters through ultrasonic clamp-on master meters*, MEASUREMENT 151, 107152.
- A Piccato, M Bisi, PG Spazzini, F Bertiglia, G La Piana, E Audrito, M Santiano, R Bellotti, C Francese, *Metrological features of the Large Piston Prover at INRiM*, MEASUREMENT 192, 110841.

Working Group on Force and Torque (CCM-WGFT)

Main research and development activities

Participation in the EMPIR project 18SIB08 COMTRAFORCE on continuous, dynamic and multicomponent calibration of force transducers and testing machines (development of calibration procedures). Theoretical studies on primary realization of micro and nano-force standards.

Key comparisons

- CCM.F-K23 (200 N and 500 N), Draft A.
- EURAMET.M.F-K3 (0.5 MN and 1 MN), Draft B.

Peer reviews (received/conducted)

- IPQ (Portugal), peer review of force laboratory (received).
- CEM (Portugal, peer review of force and torque laboratories (conducted).

Publications

- Prato, A; Borgiattino, D; Facello, A; Mazzoleni, F; Germak, A., *Calibration of multicomponent force and moment transducers using uniaxial force standard machines integrated with tilted plates*. MEASUREMENT SCIENCE & TECHNOLOGY vol. 33 (9), 2022, DOI: 10.1088/1361-6501/ac793c.
- Sander, J.; Mirian, D.; Frank, H.; Hassan, S.; Kumme, R.; Prato, A.; Germak, A.; Wozniak, M.; Izquie, *Validation report which details the advanced models developed to describe a) static and continuous and b) dynamic force transfer standards taking into account sensitivity stability, temperature and other parasitic influences on the measurement uncertainty (target uncertainty is 1 % up to 100 Hz and 2 % between 100 - 1000 Hz)*. 2022, DOI: 10.5281/zenodo.7113187.
- Knott, Andy; Altintas, Aykurt; Arrhén, Fredrik; Averlant, Philippe; Aydemir, Bülent; Germak, Alessandro; Miha Hiti; Rolf Kumme; Andrea Malengo; Maria Nieves Medina Martín; Dirk Röske; Lukáš

Vavrečka. *Guidelines on the Uncertainty of Force Measurements*. EURAMET Calibration Guide No. 4 Version 3.0 (02/2022) vol. 4, Version 3.0, 2022.

- Prato, Andrea; Borgiattino, Davide; Mazzoleni, Fabrizio; Facello, Alessio; Germak, Alessandro. *Theoretical insights on the influence of the experimental plan in the calibration of multicomponent force and moment transducers*. MEASUREMENT: SENSORS vol. 18 (100209), 2021, DOI: 10.1016/j.measen.2021.100209.
- Prato, Andrea; Genta, Gianfranco; Galetto, Maurizio; Mazzoleni, Fabrizio; Facello, Alessio; Germak, Alessandro. *Accurate coil springs axial and transverse stiffness measurements with multicomponent testing machines*. IEEE International Workshop on Metrology for Automotive (MetroAutomotive), 1-2 July 2021 (2021), ISBN: 978-1-6654-3906-0, DOI: 10.1109/MetroAutomotive50197.2021.9502872.

Working Group on Gravimetry (CCM-WGG)

Main research and development activities

Research activities were conducted for improving the IMG-02 Absolute Gravimeter. A project aimed at realizing a reference network for absolute gravity and at establishing the International Height Reference System/Frame (IHRS/IHRF) has been started. Studies on MEMS accelerometers in gravimetry have been carried out.

Peer reviews (received/conducted)

- none.

Key comparisons

- none.

Publications

- Prato, Andrea; Mazzoleni, Fabrizio; Facello, Alessio; Origlia, Claudio; Schiavi, Alessandro; Germak, Alessandro. *Perspectives and limits on the use of commercial low-cost digital MEMS accelerometers in gravimetry*. DOI: 10.5194/egusphere-egu21-10360. In EGU General Assembly 2021, 2021.

Working Group on Hardness (CCM-WGH)

Main research and development activities

Research activities were carried out in the field of Instrumented Indentation Test (IIT), as well in the regular Rockwell, Vickers, Knoop and Brinell scales. A new micro-Vickers primary hardness machines has been metrologically characterized.

Peer Reviews (received/conducted)

- none.

Key comparisons

- EURAMET.M.H-K1.b Vickers (HV1) (INRiM Pilot). Approved, and final report available;
- EURAMET.M.H-K1.c Vickers (HV30) (INRiM Pilot). Approved, and final report available;
- EURAMET.M.H-S2.a Brinell (HBW1/30) (INRiM Pilot). Approved, and final report available;
- EURAMET.M.H-S2.b Brinell (HBW2.5/187.5) (INRiM Pilot). Approved, and final report available.

Publications

- Kuzu, C.; Germak, A.; Origlia, C.; Pelit, E., *Result analysis of EURAMET Vickers comparison between INRiM and UME (EURAMET.M.H-K1.b.c)*. MEASUREMENT: SENSORS vol. 18 (100268), 2021. DOI: 10.1016/j.measen.2021.100268.
- Fidelus, J. D.; Germak, A.; Origlia, C., *Bilateral comparison in Rockwell C hardness scale between INRiM and GUM*. MEASUREMENT: SENSORS vol. 18 (100360), 2021. DOI: 10.1016/j.measen.2021.100360.

- Germak, A; Kuzu, C; Origlia, C; Pelit, E., *EURAMET key comparison between INRiM and UME in Vickers hardness scales (HV1 - HV30)* - EURAMET.M.H-K1.b and c. METROLOGIA vol. 58 (1A), 2021. DOI: 10.1088/0026-1394/58/1A/07002.
- Germak, A; Kuzu, C; Menelao, F; Origlia, C; Pelit, E. *EURAMET supplementary comparison between INRiM, UME and PTB in Brinell hardness scales (HBW1/30 - HBW2.5/187.5)* - EURAMET.M.H-S2.a. METROLOGIA vol. 58 (1A), 2021. DOI: 10.1088/0026-1394/58/1A/07003.
- Schiavi, Alessandro; Origlia, Claudio; Germak, Alessandro Franco Lidia; Prato, Andrea; Genta, Gianfranco, *Indentation Modulus, Indentation Work and Creep of Metals and Alloys at the Macro-Scale Level: Experimental Insights into the Use of a Primary Vickers Hardness Standard Machine.* MATERIALS vol. 14, 2021. DOI: 10.3390/ma14112912
- Germak, A.; Kuzu, C.; Origlia, C.; Pelit, E.; Menelao, F., *Result analysis of EURAMET Brinell comparison between INRiM, UME and PTB (EURAMET.M.H-S2.A.B).* MEASUREMENT: SENSORS vol. 18 (100251), 2021. DOI: 10.1016/j.measen.2021.100251.
- Low, S.; Germak, A.; Knott, A.; Machado, R.; Song, J., *Developing definitions of conventional hardness tests for use by National Metrology Institutes.* MEASUREMENT: SENSORS vol. 18 (100096), 2021. DOI: 10.1016/j.measen.2021.100096.

Working Group on Mass (CCM-WGM)

Main research and development activities

A new laboratory for atomic scale length metrology is under construction and will be fully operational by the end of the year. The equipment for measuring the lattice parameter has been moved to the new laboratory but is not operational. Considering the absolute measurement of the unit cell volume for the kilogram realisation, we developed new models of the X-ray interferometer and performed numerical simulations to quantify or exclude systematic errors. For experimental verification of these predictions, we extracted data from the archive of the lattice parameter measurements. In particular, systematic errors introduced by the defocusing of the X-ray interferometer have been investigated and a novel method for an online measurement of the miscut angle (angles between optical and atomic surfaces of the X-ray interferometer mirrors) has been reported in a dedicated paper.

Participation in the EMPIR project SRT-r03 "RealMass, Improvement of the realisation of the mass scale" (2020-2023). INRiM is the WP3 leader "Development of mathematical and software tools". In 2022, a training course in Sarajevo on the use of software for the realisation of the mass scale was provided to the participants of the project. In 2021, the development of a software in the Matlab environment with a graphic user interface for the realisation of the mass scale has started. Two estimators can be used: Gauss Markov and the Lagrange multiplier method. The software is designed to make it easy to implement the system of equations to be solved. The work was presented at the CIM2023 (07-10 March 2013) congress in Lyon. The executable file of the software will be made available on the INRiM website in the middle of 2023.

Peer reviews (received/conducted)

- IPQ (Portugal), peer review mass laboratory (conducted).
- MIRS (Slovenia), peer review mass laboratory (conducted).

Key comparisons

- Beste Korutlu, Christos Mitsas, Andrea Malengo, Christian Wüthrich, Sungjun Lee, Saud Alqarni, Aisha Al-Abdulhadi, Abeer I Al-Qattan, Khalid Sadee Mahmoud. Final report for GULFMET. MM-K4: key comparison of 1 kg stainless steel mass standards. METROLOGIA 57 1A 07027.
- EURAMET Project 1556 Study Comparison for the realization of the mass scale.

Publications

- C P Sasso , G Mana and E Massa, *Defocused travelling fringes in a scanning triple-Laue X-ray interferometry setup* J. Appl. Cryst. (2021). 54. DOI: 10.1107/S1600576721007962.
- C P Sasso , G Mana and E Massa, *Measurement of miscut angles in the determination of Si lattice parameters.* METROLOGIA 58 (2021) 034004. DOI: 10.1088/1681-7575/abef23.

- Z Zelenka, S Alisic, B Stoilkovska, R Hanrahan, I Kolozinsky, G Popa, D Pantic, V Dikov, J Zuda, M Coenegrachts, A Malengo, *Improvement of the realisation of the mass scale*. Acta IMEKO 9 (5) 4-6 2020.
- Z Zelenka, S Alisic, R Hanrahan, I Kolozinsky, G Popa, J Zuda, A Malengo, *Why and how to improve the subdivision technique in mass metrology*. MEASUREMENT: SENSORS 18, 100228 2021.
- A Malengo, W Bich, *Conformance probability in the assessment of Calibration and Measurement Capabilities*. MEASUREMENT 192, 110865 2022.
- Andrea Malengo and Davide Torchio, *Development of a comprehensive software application for realization and dissemination of the mass scale*. MEAS. SCI. TECHNOL. (2023) 34 074001.

Working Group on Pressure and Vacuum (CCM-WGPV)

Main research and development activities

A pioneering system for pressure measurement, based on the Rayleigh scattering of gas molecules was realized at INRiM. A novel version, named RAY, was characterized in terms of resulting intensity of scattered light as function of gas pressure for three different gases, helium, argon, and nitrogen, paving the way for the realization of a transfer standard based on this technique, which can be achieved through a simple experimental setup, and it is able to provide fast measurement, in principle, at low cost.

A novel optical pressure standard operating up to 120 kPa, based on an unbalanced homodyne interferometer (UINT) was realized at INRiM and aims to propose an alternative route to current Fabry-Perot cavity-based techniques. The most important factors influencing the performance of UINT were studied, including the control of the temperature at millikelvin level and the measurement in vacuum of the unbalance of the interferometer, fulfilling the aim the main goal of measuring pressure with a relative uncertainty of 10 ppm at pressure above 10 kPa. The UINT optical pressure standard was also compared with INRiM primary standards, namely a force balanced piston gauge for pressures up to 10 kPa, and a pressure balance for pressures above 10 kPa to 120 kPa: The results demonstrated the agreement of the UINT system and the INRiM conventional pressure standards within their related uncertainty ($k=1$).

UINT and RAY systems were developed within EMPIR project 18SIB04 “QuantumPascal”, in which INRiM participated and led the WP2 (alternative non-Fabry P erot based approaches for the realisation of absolute and partial pressure standards).

After the renewal and the re-commissioning of static expansion system (SES), two comparisons for validating the improved SES were performed: a comparison between SES and INRiM primary standard FPG in the range between 1 Pa and 1000 Pa and a further comparison between SES and INRiM continuous expansion system (CES) in the range between $3 \cdot 10^{-4}$ Pa and $9 \cdot 10^{-2}$ Pa. Both the comparisons demonstrated the equivalence of the involved INRiM primary standards, supporting the successful reinstatement of the INRiM CMC, absolute pressure, gas medium, vacuum gauge from $9 \cdot 10^{-2}$ Pa to 1000 Pa.

Participation in the EU Twinning Project GEOSTM.

Participation in a circular comparison between a transportable Fabry-Perot refractometer and the conventional primary standards available at INRiM, LNE, PTB and RISE-UmU. Four inter-laboratory comparisons with accredited Italian laboratories were performed.

Peer reviews (received/conducted)

- none.

Key comparisons

- none.

Publications

- Mari Domenico; Pisani Marco; Francese Claudio. *Rayleigh scattering for pressure assessment*, MEASUREMENT: SENSORS 18, 100253, 2021.
- Mari, D.; Pisani, M.; Astrua, M.; Zucco, M.; Pasqualin, S.; Egidi, A.; Bertinetti, M.; Barbone, A. *Realisation of an optical pressure standard by a multi-reflection interferometric technique*. MEASUREMENT 211, 112693, 2023.