

# **INRIM activity report in the field of mass and related quantities for the years 2019 - 2020**

**18<sup>th</sup> meeting, Thursday 20 – Friday 21 May 2021**

## **Working Group on Density and Viscosity (CCM-WGDV)**

### Main research and development activities

The standard seawater-density was measured at different absolute salinities from (10.044 – 38.178) g kg<sup>-1</sup>, in the temperature range (278.15 – 313.15) K at atmospheric pressure. The measurements were carried out using a commercial vibrating tube densimeter, following the substitution method (alternating samples of seawater and water) and under conditions typical of most oceanographic laboratories capabilities. An accurate analysis of the uncertainty of density was performed, obtaining a relative expanded uncertainty of 0.003% (k=2). Measurements were compared with the density values provided by the international reference equation of state, the Thermodynamic Equation of SeaWater-2010, TEOS-10.

In this framework, an already existent single sinker hydrostatic weighing densimeter was adapted to work with seawater. The absolute density of a sample of Standard Sea Water (IAPSO) has been measured, and results compared with those predicted by TEOS-10. These results have been presented to the IAPWS Annual Meeting (29 September – 6 October 2019) in Banff, Canada.

Most salinity calculations are based on conductivity measurements. The review paper describes other techniques which are used or could be used to assess the absolute salinity of seawater and questions the measurand of these techniques and the possibility to redefine the concept of salinity from physical properties.

### Peer Reviews (received/conducted)

- INMETRO (Brasil) Peer Review density laboratory.

### Key Comparisons

- EURAMET Key Comparison 1031 (EURAMET.M.D-K1.1) - solid density comparison. Horst Bettin et al. METROLOGIA 57 07028 (2020);
- EURAMET Key Comparison "EURAMET.M.D-K4.2020" on hydrometers, started in 2020, INRIM is the pilot laboratory.

### Publications

- "Density of standard seawater by vibrating tube densimeter: analysis of the method and results". R. Romeo, P. A. Giuliano Albo, S. Lago, DEEP-SEA RESEARCH PART I-OCEANOGRAPHIC RESEARCH PAPERS - ISSN:0967-0637, 154 (2019);

- "The absolute salinity of seawater and its measurands". M. Le Menn, P. A. Giuliano Albo, S. Lago, R. Romeo and F. Sparasci. METROLOGIA, 56 (2019) 015005.

## **Working Group on Fluid Flow (CCM-WGFF)**

### Main research and development activities

Developments on MICROGas test rig for small flow rates aimed at reducing the uncertainty through following the piston profile. Development of automated software for acquisitions on the various INRiM gas test rigs. Development of a large piston prover for increasing its usability, and dimensional calibration of the piston.

An improved dynamic method for liquid flow rate measurements has been investigated to be considered the primary standard for water flows lower than  $40 \text{ kg h}^{-1}$ . It is based on the immersed jet approach. Thanks to the submerged tube's constant immersion depth, it has the advantage that measurements do not need corrections for hydrostatic buoyancy. The proposed method was compared against a typical dynamic method based on the submerged jet approach to evaluate the measurement capabilities at lower flow rates. The comparison was carried out in the water flow range from  $10 \text{ kg h}^{-1}$  to  $60 \text{ kg h}^{-1}$ . It pointed out a good degree of equivalence between the two dynamic methods, highlighting the lower uncertainty of the proposed method, particularly for flow rates lower than  $25 \text{ kg h}^{-1}$ , where the uncertainty of 0.006% was obtained.

New methods have been studied to improve the accuracy of the heat accounting systems used in residential buildings supplied by centralised heating systems. The on-field accuracy of the developed methods has been experimentally compared to the traditional ones using a specially designed experimental mockup, simulating a virtual four-storey/eight-apartments building.

### Key Comparisons

- CCM.FF-K6-2017: draft A sent by the pilot and discussed between participants during 2020, Draft B expected in 2021;
- EURAMET 1325 (registered in KCDB as EURAMET.M.FF-S10): final report published online in November 2019;
- EURAMET project 1395 / EURAMET.M.FF-K4.1.2016: volume comparison at 20 L. A Malengo et al 2020 METROLOGIA 57 07021. INRiM is the pilot laboratory;
- AFRIMETS.M.FF-S1, comparison on the gas flow between NMISA, INRiM and MIKES-VTT started in 2020, measurements ongoing.

### Peer Reviews (received/conducted)

- DPM (Albania) Peer Review volume laboratory.

### Publications

- "A dynamic gravimetric standard for liquid flow measurements". F Saba, A Malengo, M Santiano. METROLOGIA 58 015007 (2021)
- "Field verification of thermal energy meters through ultrasonic clamp-on master meters". G. Ficco, A. Frattolillo, A. Malengo, F. Saba, F. Zuena, G. Puglisi. MEASUREMENT 0263-2241. - 151(2020), p. 107152;
- "A novel measurement method for accurate heat accounting in historical buildings". M. Dell'Isola, G. Ficco, B. Di Pietra, F. Saba, M. C. Masoero.

## Working Group on Force and Torque (CCM-WGFT)

### Main research and development activities

The participation in the EMPIR project 18SIB08 COMTRAFORCE contributed to the calibration of multicomponent force transducers and machines (development of calibration procedures). Study of a new control system for improvement of the primary force standard deadweight machines (1.3 kN, 30 kN, 100 kN and 1 MN) and design of a new 100 kN deadweight machine.

### Key Comparisons

- CCM.F-K23 (200 N and 500 N), ongoing.

### Peer Reviews (received/conducted)

- Peer Review of force and torque laboratory, INMETRO (Brazil).

### Publications

- "Self-calibration of the 1 MN deadweight force standard machine at INRiM". Prato, A., Mazzoleni, F., Facello, A., Germak, A. ACTA IMEKO - ISSN:2221-870X vol. 9 (5) 2020, pp.100-105.;
- "Final report on the force key comparison CCM.F-K3". Kumme, R.; Averlant, P.; Bartel, T.; Germak, A.; Knott, A.; Man, J. et al. METROLOGIA - ISSN:1681-7575 vol. 56 (Number 1A, Technical Supplement 2019) 2019 pp.1-27.

## Working Group on Gravimetry (CCM-WGG)

### Main research and development activities

Design of new launch system and modified Jamin interferometer for the IMGC-02 Absolute Gravimeter.

### Key Comparisons

- None.

### Peer Reviews (received/conducted)

- The gravity laboratory was peer-reviewed by CENAM (Mexico).

### Publications

- "Design of New Launch and Interferometer Systems for the IMGC-02 Absolute Gravimeter". Prato, A., Desogus, S., Origlia, C.; Bisi, M., Germak, A., INTERNATIONAL ASSOCIATION OF GEODESY SYMPOSIA - ISSN:0939-9585 (2020);

- “Final report of EURAMET.M.G-K3 regional comparison of absolute gravimeters”. R Falk, V Pálinkáš, H Wziontek, A Rülke, M Val'ko, Ch Ullrich, H Butta, J Kostelecký, M Bilker-Koivula, J Näränen, A Prato, F Mazzoleni, C Kirbaş, Coşkun, M Van Camp, S Castelein, J D Bernard, A Lothhammer, M Schilling, L Timmen, D Iacovone, G Nettis, F Greco, A Messina, R Reudink, M Petrini, P Dykowski, M Sękowski, J Janák, J Papčo, A Engfeldt and H Steffen; METROLOGIA, Volume 57, Number 1 (2020);
- "A new modified Jamin interferometer for the IMGC-02 transportable rise-and-fall absolute gravimeter". Prato, A., Desogus, S., Origlia, C., Germak, A., Proceedings of EGU General Assembly, 7-12 April 2019, Vienna, Austria (2019).

## Working Group on Hardness (CCM-WGH)

### Main research and development activities

Research activities were carried out in the field of Instrumented Indentation Test (IIT):

- I. indentation modulus at macro-scale level measured from Brinell and Vickers indenters by using the primary hardness standard machine at INRIM,
- II. indentation modulus, indentation work and creep of metals and alloys at the macro-scale level: experimental insights using a Primary Vickers Hardness standard machine (a paper on this subject has been submitted to an international peer-review journal).

Other activities were devoted to implementing a new procedure for the calibration of Knoop indenter using the Vickers-addressed optical system.

A new primary micro-Vickers hardness standard machine was designed and realised in cooperation with an industrial partner. The main characteristics are:

- I. force applied using direct weights with masses from 20 g to 3 kg;
- II. testing cycle controlled by an electro-mechanical system that detects the applied force using a force transducer and the displacement employing a laser measuring system;
- III. frictions minimised using air bearing system to drive the application of the force;
- IV. positioning, hardness testing, and indentation reading fully automated and controlled through a microscope, video camera, and 4-axis motorised displacement system (X, Y, Z and rotation);
- V. calibrations of Vickers reference blocks according to ISO 6507-3 (its application is also being evaluated for the instrumented indentation test according to ISO 14577).

### Key Comparisons

- EURAMET.M.H-S1.a Rockwell (HRA), (INRIM Pilot). Approved, and Final Report available;
- EURAMET.M.H-S1.b Rockwell (HRB) (INRIM Pilot). Approved and Final Report available;
- EURAMET.M.H-S1.c Rockwell (HRC) (INRIM Pilot). Approved and Final Report available;
- EURAMET.M.H-K1.b Vickers (HV1) (INRIM Pilot). Final Draft in publication;
- EURAMET.M.H-K1.c Vickers (HV30) (INRIM Pilot). Final Draft in publication;
- EURAMET.M.H-S2.a Brinell (HBW1/30) (INRIM Pilot). Final Draft in publication;
- EURAMET.M.H-S2.b Brinell (HBW2.5/187.5) (INRIM Pilot). Final Draft in publication.

### Peer Reviews (received/conducted)

- The Hardness laboratory was peer-reviewed by CENAM (Mexico);
- Peer Review of hardness laboratory, INMETRO (Brazil).

### Publications

- "CCM pilot study overview: geometrical measurement of the Rockwell diamond indenter". Machado, R. R.; Low, S.; Germak, A. ACTA IMEKO - ISSN:2221-870X vol. 9 (5) 2020. pp.250-255.
- "EURAMET supplementary comparison between INRIM and UME in Rockwell hardness scales (HRA - HRBW - HRC) - EURAMET.M.H-S1.a.b.c. ". Germak, A.; Kuzu, C; Origlia, C; Pelit, E, DOI:10.1088/0026-1394/57/1A/07018. pp.07018. In METROLOGIA - ISSN:0026-1394 vol. 57 (1A), 2020.;
- "Preliminary results of EURAMET Rockwell comparison between INRIM and UME (EURAMET.M.H-S1.A.B.C)". Kuzu, C. Germak, A. Origlia, C. Pelit, E. ACTA IMEKO - ISSN:2221-870X vol. 9 (5), 2020. pp.256-260.
- "A suitable geometrical model for the verification of Knoop indenters with Gal-Indent optical system". Prato, A.; Origlia, C.; Germak, A.; ACTA IMEKO - ISSN:2221-870X vol. 9 (5), 2020 pp.216-220.
- "Verification of Knoop indenters with a Vickers-addressed optical system". Prato, A.; Origlia, C.; Germak, A.; MEASUREMENT - ISSN:0263-2241 vol. 163 (107928); 2020. pp.1-8.
- "Indentation modulus at macro-scale level measured from Brinell and Vickers indenters by using the primary hardness standard machine at INRIM". Schiavi, A.; Origlia, C.; Germak, A.; Barbato, G.; Maizza, G.; Genta, G.; Cagliero, R.; Coppola, G.; ACTA IMEKO - ISSN:2221-870X vol. 8 (1), 2019. pp.3-12;
- "A correction method for Vickers indenters squareness measurement due to the tilt of the pyramid axis". Prato, A.; Galliani, D.; Origlia, C.; Germak, A.; MEASUREMENT - ISSN:1536-6367 vol. 140, 2019. pp.565-571.

## **Working Group on Mass (CCM-WGM)**

### Main research and development activities

A new laboratory for atomic-scale length-metrology is expected to be operational at the end of the year. The apparatus for measuring the lattice parameter is no more operational; it will be moved to the new lab without significant upgrades. X-ray phase-contrast tomography was used to investigate the strain sensitivity of x-ray interferometry to the finishing, annealing, and coating of the interferometer crystals. An analytical model of differential wavefront sensing was developed to investigate the origin of fake tilts observed in the combined x-ray and optical interferometer. In collaboration with NMIJ and PTB, INRIM was determined by finite element analysis of the self-weight correction of the Si sphere's volume for the kg realisation. The sphere volume might be overestimated from  $0.3 \times 10^{-9}$  to  $5.1 \times 10^{-9}$  (relative terms), depending on the crystal orientation and measurement procedure. An experimental check of the finite element analysis predictions seems possible.

A voltage balance prototype based on a parallel plate capacitor suitable for measuring small mass standards up to 1 g was realised. The characterisation of the actuator is made by changing the distance between the electrodes while keeping fixed the beam of the balance (constant force) and measuring the capacitance changes of the same. This approach greatly simplifies the mechanical design at the expenses of a more complex model for calculating the force. Preliminary results in the milligram range show the uncertainty of the order of  $10^{-3}$ .

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 2020, a 4-day training course on the realisation of the mass scale was provided to participants.

### Key Comparisons

- GULFMET.M.M-K7: Key Comparison of 5 kg, 100 g, 10 g, 5 g and 500 mg mass standards. Beste Korutlu et al 2020 METROLOGIA 57 07005. INRIM Link laboratory;
- GULFMET.M.M-K4: Key Comparison of 1 kg stainless steel mass standards. Beste Korutlu et al 2020 METROLOGIA 57 07027. INRIM Link Laboratory

### Peer Reviews (received/conducted)

- DPM (Albania) Peer Review mass laboratory;
- INMETRO (Brasil) Peer Review mass laboratory.

### Publications

- "Gravity and anisotropy effects in the volume determination of Si spheres for the kilogram realisation", Mari, D; Nicolaus, A; Kuhn, E; Kuramoto, N; Mana, G; Massa, E. - METROLOGIA. - ISSN 0026-1394. - **57**: 4 (2020), p. 045004;
- "X-ray phase-contrast topography to measure the surface stress and bulk strain in a silicon crystal", Massa, E.; Sasso, C. P.; Fretto, M.; Martino, L.; Mana, G. - JOURNAL OF APPLIED CRYSTALLOGRAPHY. - ISSN 1600-5767. - **53**: 5 (2020), pp. 1195-1202;
- "The Measurement of the Silicon Lattice Parameter and the Count of Atoms to Realise the Kilogram", Massa, Enrico; Sasso, Carlo Paolo; Mana, Giovanni. - MAPAN. JOURNAL OF METROLOGY SOCIETY OF INDIA. - ISSN 0970-3950. - (2020);
- "Bayesian model selection applied to linear regressions with weighted data", Mana, Giovanni; Massa, Enrico; Sasso, Carlo Paolo. - METROLOGIA. - ISSN 0026-1394. - **56**: 2 (2019).
- "Fake tilts in differential wavefront sensing" Massa, Enrico; Sasso, Carlo Paolo; Mana, Giovanni. - OPTICS EXPRESS. - ISSN 1094-4087. - **27**: 24(2019), p. 34505-34518;
- "Improvement of the realisation of the mass scale" Z. Zelenka, S. Alisic, B. Stoilkovska, R. Hanrahan, I. Kolozinsky, G. Popa, D. Pantic, V. Dikov, J. Zuda, M. Coenegrachts, A. Malengo. ACTA IMEKO Vol 9, No 5 (2020);
- "A Simple Electrostatic Balance for the Milligram Range" Pisani M, Malengo A, Santiano M, Saba F, Torchio D. IEEE Transactions on Instrumentation and Measurement, Vol 68, 6, 2019.

## **Working Group on Pressure and Vacuum (CCM-WGPV)**

### Main research and development activities

The research activities were focused on developing two different systems for pressure measurement: an optical pressure standard based on refractive index measurement through a multi-reflection interferometric technique (UINT) and an experimental set-up based on the measurement of Rayleigh scattering of gas molecules (RAY). Upgrades of systems UINT and RAY are in progress in the framework of EMPIR 18SIB04 QuantumPascal (2019-2022), where INRIM participates and leads the WP2 (Alternative non-Fabry Pérot based approaches for the realisation of absolute and partial pressure standards).

INRIM also carried out a FEM study of pressure-induced deformations of optical cavities for pressure measurement in the framework of EMPIR 18SIB04 project. Four partners independently performed this study. They demonstrated that the various partners could perform simulations with minor discrepancies and that methodological mistakes contribute to a sub-ppm uncertainty in the assessments of refractivity of N<sub>2</sub>.

The improvement of the INRiM static expansion system has been completed, extending its lower limit from 90 mPa to 0.5 mPa. The next step will be participating in an international comparison. Five inter-laboratory comparisons with accredited Italian laboratories were performed.

Peer Reviews (received/conducted)

- INRiM (Italy, visited NMI), periodical on-site peer review pressure laboratory, December 2019 (EURAMET project 1123);
- IPQ (Portugal, visited NMI) periodical on-site peer review pressure laboratory, December 2020 (EURAMET project 1123).

Publications

- "Simulation of pressure-induced cavity deformation – the 18SIB04 QuantumPascal EMPIR project". Zakrisson J, Silander I, Forssén C, Silvestri Z, Mari D, Pasqualin S, Kussicke A, Asbahr, P, Rubin, T, Axner, O., ACTA IMEKO - ISSN 2221-870X 9:5 (2020), 281-286;
- "Time stability characterization of quadrupole mass spectrometers". Elkatmis Alper, Kangi Rifat, Becker Ute, Jousten Karl, Mari Domenico, Boineau Frederic, Vicar Martin, Ruiz, Salustiano, Setina, Janez. MEASUREMENT 165 (2020), 108143;
- "Towards the realization of an optical pressure standard". Mari D, Pisani M, Zucco M. 132 MEASUREMENT 132 (2019), 402-407;
- "Improvement of INRiM static expansion system as vacuum primary standard between  $10^{-4}$  Pa and 1000 Pa". Milena Astrua, Domenico Mari and Stefano Pasqualin. 9<sup>th</sup> International Congress of Metrology (2019), 27007.