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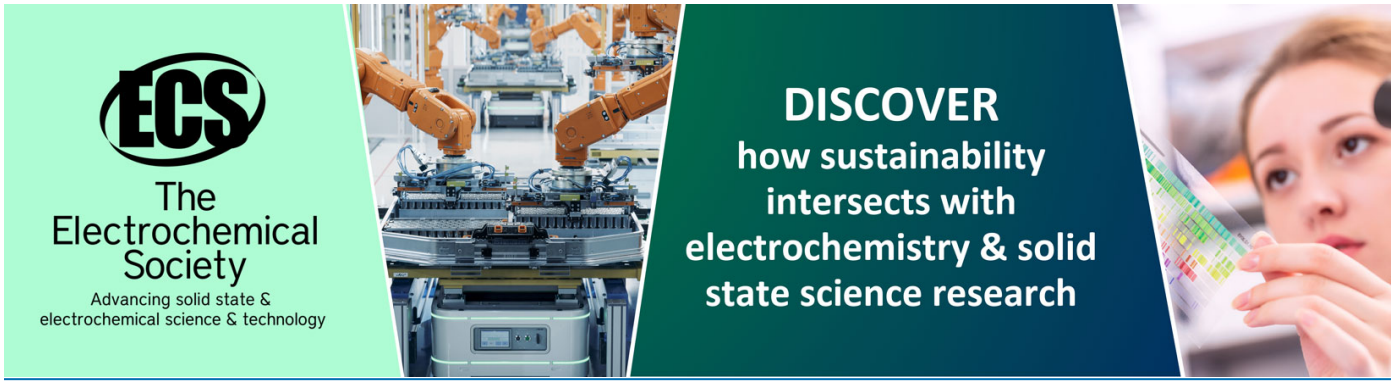

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Establishing traceability for liquid density measurements in Europe: *17RPT02-rhoLiq* a new EMPIR joint research project

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Abstract. The aim of this EMPIR project is to establish traceability for liquid density measurements at emerging 8 National Metrology Institutes. This will enable them to provide high-level measurement and calibration services, and to produce density reference materials for national stakeholders, e.g. food, chemical, pharmaceutical and petroleum industries. The international recognition of these NMIs in this metrological field will indirectly lead to the reinforcement of mutual confidence and cooperation at regional and international level. This project will facilitate compliance with economically relevant EU Directives, e.g. Directive 2007/45/EC, and it will further reinforce the competitiveness of production industries.

1. Need for this project

Density bridges two classical quantities, mass and volume, which are the major driving elements in economic transactions with important goods, ranging from fuels to liquid food products, which are commonly sold as pre-packaged goods, . The actual consumption of high-value liquids, such as wine, olive oil and fuel, can be considered of major importance to the European economy.

In Europe, only four National Metrology Institutes (NMI), BEV-PTP, GUM, MKEH, and PTB, currently possess the appropriate expertise to perform liquid density measurements at the primary-level, i.e. with hydrostatic weighing apparatuses with a level of accuracy and uncertainty that meets national (e.g. to fulfil national laws) and international (e.g. to fulfil European Directives and standards)

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needs. As a consequence the national traceability chain of liquid density measurements, down to the second and third level, which are used in industry and in research laboratories, is often compromised in less experienced European countries. For these reasons, emerging countries are keen to prepare their markets, for integration into the EU single market, by harmonising their national legislation in order to the meet the standards set out in EU directives, e.g. Directive 2007/45/EC for prepacked products, as well as for the control of fuel production and use [1].
Liquids can differ in density and viscosity, can show non-Newtonian behaviour, can contain dissolved gas (e.g. carbon dioxide in beverages), or particles (e.g. pulp in juice), and may be handled at high temperatures and pressures. This wide variation in properties and conditions means that density meters need to have adequate calibration procedures. Metrological information about the robustness of these density measurement methods, in this range of conditions, is needed as it is not currently described in standards documents. In fact, there is also a lack of EURAMET guides on liquid density measurements and existing international standards [2, 3] and the reference documents used in Legal Metrology [4, 5] are outdated and incomplete. The European Directive [1] regarding the mandatory control of the volumes of the liquids contained in prepacked products for consumer protection, as well as for the introduction of these products onto the market, has intensified the need for NMIs to provide the proper measurement and calibration services. Improvements in the quality of liquid density measurements will have a direct impact at industrial sites, leading to a reduction in the number of products that do not conform with volume requirements. Therefore, this will lead to more competitive national industries and it will allow a free circulation within the EU market. Having the proper methods to control the volume inside a prepacked liquid will boost confidence in the market.

2. Objectives

The overall objective of this project is to develop the national metrological capacity in liquid density metrology in 8 emerging NMIs (BRML, CMI, DMD, IMBiH, INM, IPQ, JV and TUBITAK), with the target of achieve the lowest measurement uncertainty possible when using state-of-the-art density measuring systems. This will include coverage of the quantities that influence the measurement of density, i.e. temperature, pressure, viscosity and surface tension. These objectives will include a review and upgrade of the existing capabilities and needs, validation of the existing measuring systems and, if required, the development of new systems, with the support of 3 more experienced NMIs (BEV-PTP, GUM and PTB), for density, temperature and pressure intervals relevant for scientific and industrial needs, i.e. [600, 1 700] kg/m^3, [5, 60] °C and [1, 600] bar, respectively. The specific objectives of this project are:

- To develop first-level liquid density measurement capabilities by the hydrostatic weighing method at atmospheric pressure, in the temperature interval from 5 °C to 60 °C, with an uncertainty from 0.002 kg/m^3 to 0.005 kg/m^3.
- To develop second-level liquid density measurement capabilities performed with oscillation-type density meters for liquids at atmospheric pressure with dynamic viscosity at least up to 2 000 mPa·s, in the temperature interval from 5 °C to 60 °C, with an uncertainty from 0.01 kg/m^3 to 0.05 kg/m^3, and for pressures up to 600 bar with an uncertainty from 0.1 kg/m^3 to 0.5 kg/m^3.
- To establish the degree of equivalence of the density measurements performed by the emerging NMIs with the developed first and second-level measuring systems via comparisons.
- To study the robustness of first-level, i.e. hydrostatic weighing method, and second-level, i.e. oscillation-type density meter, liquid density measurement methods regarding the influence of the liquid's physical properties: viscosity (from 1 mPa·s to 10 000 mPa·s); surface tension from 20 mN/m to 72 mN/m and viscoelasticity (behaviours other than Newtonian); and of independent properties (temperature (from 5 °C to 60 °C) and pressure (from atmospheric pressure up to 600 bar).
- To develop new guidance documents for first and second-level liquid density measurement methods and for the production of certified reference liquids for use in density measurements, such as EURAMET guides and a good practice guide, for the metrological and industrial communities. To

provide input for the revision of relevant reference documents (ISO 15212 [2, 3], OIML Guide 14 [4] and WELMEC Guide 6.4 [5]).

- To develop the emerging NMI's individual strategies for the long-term operation of the capacity developed, including new/upgraded measurement and calibration services; the production of certified reference liquids; the provision of training to disseminate best practices; the provision of national metrological infrastructures and proficiency testing schemes to support accreditation bodies and field laboratories.

3. Progress beyond the state of the art and expected results

3.1. Developed/improved liquid density measurement capabilities at first and second-level in 8 emerging NMIs and developed capabilities for producing CRM for liquid density

First-level traceable measurement capabilities for liquid density will be established by developing the hydrostatic weighing method in emerging 8 NMIs. This will allow the dissemination of liquid density from the SI base quantities, mass and length, by establishing the first step of the liquid density traceability chain and it will enable the production of Certified Reference Materials (CRM) for density, targeting the lowest uncertainty in the interval from [0.002, 0.005] kg/m^3, to be used for the dissemination of the liquid density quantity to a second-level of the traceability chain.

Second-level traceable measurement capabilities for liquid density will be established in the emerging NMIs, using standard oscillation-type density meters at ambient pressure and high pressure (up to 600 bar), with an uncertainty in the interval from [0.01, 0.50] kg/m^3 and [0.1, 0.5] kg/m^3, respectively. The emerging NMIs will be able to offer new/better calibration services with a high level of accuracy; to determine a liquid's thermal expansion coefficient and compressibility coefficient; and to produce and certify reference materials (RM) with an uncertainty level sufficient to be used by accredited calibration laboratories and industrial companies. Additionally, some of these NMIs will be able to produce accurate and traceable density measurements of liquids at high-pressure (up to 600 bar), and for high viscosity liquids (with viscosity (ideally) up to 10 000 mPa·s).

3.2. Establishment of the level of equivalence of liquid density measurements of the emerging NMIs, by means of comparisons, with publication of CMC in the KCDM/BIPM

The establishment of the degree of equivalence of the density measurements performed with the developed first and second-level measuring systems by means of comparisons in the emerging NMIs, will enable the recognition and demonstration of the competence of the these NMI's measurements capabilities in accordance with European rules, i.e. CIPM MRA. This will enhance the negotiation of wider agreements for international trade and regulatory affairs.

3.3. Enhanced scientific knowledge regarding liquid density measurement methods which are able to give input for international guides and standards

The NMIs will be able to develop strategies for accurately measuring the density of liquids with non-classical physical properties, i.e. with physical properties that differ from water or hydrocarbons which are normally used as standard liquids. This will include robustness studies using liquids with high viscosity, with viscoelastic behaviour, and with dissolved gases or suspended particles. These kind of liquids are the most commonly measured by the end-users (food, chemical, pharmaceutical, petroleum, biofuels, etc.), therefore, the knowledge gained about possible interferences and corrections, will be crucial to obtain accurate and traceable density measurement results. These kinds of measurements can be performed under limited conditions and will often result in larger uncertainties. This knowledge will also be disseminated in international guides and standards for scientific, applied (via 3 new EURAMET guides and the revision of existing ISO standards [2, 3]) and legal (via revision of existing OIML [4] and WELMEC [5] documents) metrology and this will address the lack of documentation on this issue.

3.4. Creation of new and recognised measuring capabilities for the end-user community
The implementation of the emerging NMI's individual strategies for the long-term development of their research capability in liquid density measurements will result in them being able to offer calibration services from their established facilities to their own country and to neighbouring countries.

4. Longer-term economic, social and environmental impact
The project will lead to the establishment of a proper and recognised traceability chain for liquid density measurements which will be mandatory to increase national industry confidence and competitiveness in many fields such as food, chemical, pharmaceutical, petroleum, biofuels, etc. The growth in the free circulation of these goods on the European market, according to European Directives [1], regarding the mandatory quantity control of prepacked products for means of consumer protection has intensified the requests of these industries to the NMIs to provide proper calibration services in their own country in the future.
Boosting liquid density metrology will lead to an increased protection of European citizens and markets in terms of legal metrological control. Confidence in the products coming from these emerging countries will grow, leading to an increase in the free circulation of goods. The support for European protection of products and enterprises against defrauders will have secondary effects such as an economic effect in terms of boosting industry competitiveness, but also environment protection. Better control of fuels will also contribute to environmental protection.

5. References
[1] Directive 2007/45/EC of the European Parliament and of the Council of 5 September 2007, laying down rules on nominal quantities for prepacked products, repealing Council Directives 75/106/EEC and 80/232/EEC, and amending Council Directive 76/211/EEC.
[2] ISO 15212-1:1998. Oscillation-type density meters - Part 1: Laboratory instruments.
[3] ISO 15212-2: 2002. Oscillation-type density meters - Part 2: Process instruments for homogeneous liquids.
[4] OIML G 14: 2011 (E) - Density measurement.
[5] WELMEC Guide 6.4 - Guide for packers and importers of e-marked prepacked products.

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