

Publishable Summary for 17RPT02 rhoLiq Establishing traceability for liquid density measurements

Overview

The aim of this project is to establish traceability for liquid density measurements at emerging National Metrology Institutes (NMIs). 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, and it will further reinforce the competitiveness of production industries.

Need

Density bridges two classical quantities, mass and volume, which are the major driving elements in economic transactions with important goods, ranging from liquid food products, which are commonly sold as pre-packaged goods, to fuels. The actual consumption of high-value liquids, such as wine, olive oil and fuel, is of major importance to the European economy. In Europe, few NMIs possess the appropriate expertise to perform liquid density measurements at the primary-level, i.e. with hydrostatic weighing apparatuses, and at the secondary level, i.e. with oscillation-type density meters, 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), needs. Therefore, the national traceability chain of liquid density measurements, down to the third-level, which is used in test, calibration, and research laboratories as well in industry, 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 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.

Liquids may differ in density and viscosity, may show non-Newtonian behaviour, may 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. Therefore, metrological information regarding the robustness of these density measurement methods, in this range of conditions, is needed as it is not currently adequately described in standards documents. In fact, there is also a lack of EURAMET guides on liquid density measurements and the existing international standards (ISO 15212) and reference documents used in Legal Metrology (OIML Guide 14 and WELMEC Guide 6.4) are outdated and incomplete. The European Directive (Directive 2007/45/EC) regarding the mandatory control of the volume 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.

Objectives

The overall objective of this project is to develop the national metrological capacity in liquid density metrology in the less experienced partners, with the target of achieving 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. This will also 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 the most experienced NMIs for density, temperature and pressure intervals that are relevant for scientific and industrial needs, i.e. [600, 1 700] kg/m³, [5, 60] °C and [1, 600] bar, respectively. The specific objectives of this project are:

1. **To develop first-level liquid density measurement capabilities using the hydrostatic weighing method in the less experienced partners**, at atmospheric pressure, and in the temperature interval from 5 °C to 60 °C, with an uncertainty from 0.002 kg/m³ to 0.005 kg/m³.

2. **To develop second-level liquid density measurement capabilities using oscillation-type density meters in the less experienced partners**, for liquids with a dynamic viscosity of at least up to 2 000 mPa s (ideally up to 10 000 mPa s), in the temperature interval from 5 °C to 60 °C, at atmospheric pressure, with an uncertainty from 0.01 kg/m³ to 0.05 kg/m³, and for pressures up to 600 bar, with an uncertainty from 0.1 kg/m³ to 0.5 kg/m³.
3. **To establish the degree of equivalence of the density measurements** performed by the emerging NMIs via comparisons of the first and second-level measuring systems that will be developed.
4. **To study the robustness of the first-level, i.e. hydrostatic weighing method, and the 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, such as yield point); and of independent properties (temperature (from 5 °C to 60 °C) and pressure (from atmospheric pressure up to 600 bar).
5. **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, OIML Guide 14 and WELMEC Guide 6.4).
6. **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 in liquid density metrology; the provision of national metrological infrastructures and proficiency testing schemes to support accreditation bodies and field laboratories.

Progress beyond the state of the art

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

These measurement capabilities will become available in the most relevant intervals: of density from [600, 1700] kg/m³; of viscosity from [1, 10 000] mPa·s; and temperature from [5, 60] °C, i.e. for the liquids most commonly measured by the end-users.

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 saturated hydrocarbons which are normally used as standard liquids. This will include robustness studies, of the first and second-level liquid density measurements methods, using liquids with high viscosity, with viscoelastic behaviour, and with dissolved gases and with suspended particles. These types 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 they 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) and legal (via revision of existing OIML and WELMEC documents) metrology and this will address the lack of documentation on this issue.

Establishment of the level of equivalence of liquid density measurements of the emerging NMIs, by means of comparisons, with publication of CMCs in the KCDM/BIPM and creation of new and recognised measuring capabilities for the end-user community

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 these NMI's measurement capabilities in accordance

with European rules, i.e. CIPM MRA. This will enhance the negotiation of wider agreements for international trade and regulatory affairs.

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.

Results

1. *To develop first-level liquid density measurement capabilities using the hydrostatic weighing method in the less experienced partners, at atmospheric pressure, and in the temperature interval from 5 °C to 60 °C, with an uncertainty from 0.002 kg/m³ to 0.005 kg/m³.*

The reference liquids that to be used in the diagnostic comparison for liquid density determination, by hydrostatic weighing, were selected, and measured by the pilot laboratory (PTB) by hydrostatic weighing at three temperatures (15 °C, 20 °C and 25 °C), at atmospheric pressure. The selected liquids were: deuterated ultra-pure water (1.5 %, air-saturated) and n-Dodecane. The technical protocol of the diagnostics density measurement comparisons was prepared and reviewed by all the partners. The liquids used in the comparison were checked for homogeneity and stability by the pilot laboratory (PTB). The measurements of the diagnostic comparison were finished in December 2019 and the report is under preparation.

2. *To develop second-level liquid density measurement capabilities using oscillation-type density meters in the less experienced partners, for liquids with a dynamic viscosity of at least up to 2 000 mPa·s (ideally up to 10 000 mPa·s), in the temperature interval from 5 °C to 60 °C, at atmospheric pressure, with an uncertainty from 0.01 kg/m³ to 0.05 kg/m³, and for pressures up to 600 bar, with an uncertainty from 0.1 kg/m³ to 0.5 kg/m³.*

The reference liquids to be used in the two diagnostic comparisons for liquid density determination, by oscillation-type density meters were selected, and measured, tested for homogeneity and stability by the pilot laboratory (PTB) by hydrostatic weighing at three temperatures (15 °C, 20 °C and 25 °C), at atmospheric pressure. The selected liquids were: deuterated ultra-pure water (air-saturated) and n-Dodecane. The measurements of the diagnostic comparison were finished in December 2019 and the report is under preparation.

3. *To establish the degree of equivalence of the density measurements performed by the emerging NMIs via comparisons of the first and second-level measuring systems that will be developed.*

Establishing the degree of equivalence of liquid density measurements, via comparisons, will enable the emerging NMI's measurement capabilities to be recognised and demonstrated in accordance with the CIPM MRA with the publication of CMCs in the KCDB/BIPM.

The chairperson of the Technical Committee for Mass and Related Quantities of EURAMET as well as the chairperson of the Working Group on Density and Viscosity (CCM-WGDV) of CIPM were contacted in order to get approval to register two EURAMET Key-Comparisons on the density of liquids using the hydrostatic weighing method and oscillation type density meters. The consortium has been authorised to register two Key-Comparisons at the EURAMET level by the Working Group on Density and Viscosity (CCM-WGDV) of CIPM.

4. *To study the robustness of the first-level, i.e. hydrostatic weighing method, and the 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, such as yield point); and of independent properties (temperature (from 5 °C to 60 °C) and pressure (from atmospheric pressure up to 600 bar).*

Knowledge on the robustness of first-level (i.e. hydrostatic weighing method) and second-level (i.e. oscillation-type density meter) liquid density measurement methods will be obtained in relation to 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, such as yield point); and of independent properties (temperature (from 5 °C to 60 °C) and pressure (from atmospheric pressure up to 600 bar).

5. *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, OIML Guide 14 and WELMEC Guide 6.4).

New guidance documents will be prepared including EURAMET guides for first and second-level liquid density measurement methods and for the production of certified reference liquids for density, and a good practice guide for the metrological and industrial communities. The project's results will also be provided for the revision of several reference documents (ISO 15212, OIML Guide 14 and WELMEC Guide 6.4).

6. *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 in liquid density metrology; the provision of national metrological infrastructures and proficiency testing schemes to support accreditation bodies and field laboratories.*

After the conclusion of the project's 3 training activities (i.e., a one-week Seminar at PTB, a 2-day Workshop at Anton Paar, and 1 to 2-weeks of Individual Training at PTB), each partner prepared and delivered a roadmap describing the improvements required for their own hydrostatic weighing apparatuses and oscillation-type density meters, in alignment with the national needs. The final strategy report with input from the proceeding tasks is in preparation by PTB. The NMI's strategies for the long-term operation of the developed measurement capabilities will include: new/upgraded measurement and calibration services; the production of certified reference liquids; the provision of training to disseminate best practices in liquid density metrology; the provision of national metrological infrastructures and proficiency testing schemes to support accreditation bodies and field laboratories.

Impact

Just after project's kick-off meeting a one-week Seminar took place at the PTB - Braunschweig (Germany) (15-18 May 2018) where the theoretical aspects related to the methods of determining the density of liquids were discussed. Next, a Workshop held at the Anton Paar, in Graz, Austria (12-13 June 2018), brought the opportunity to exchange experiences among the producers of this type of measuring instruments and metrology experts in this area. To finalize the training each project partner had visited PTB, for a 1-2-week training (May 2018 to February 2019) on hydrostatic weighing apparatus and oscillation-type density meters. Project's progress and first results concerning viscoelastic samples in oscillation-type density meters were communicated and disseminated to the scientific and industrial end-users by the publication of 3 papers in open access journals and by presentations in 6 international events. Both diagnostic comparisons measurements were completed, and Draft reports will be issued by February 2020.

Impact on industrial and other user communities

This project will support national industries, and those in neighbouring countries, through the implementation of a recognised traceability chain for liquid density measurements. This will be achieved through the implementation / development of liquid density measurement capabilities at emerging NMIs and through the production of reference documents, e.g. a good practice guide for liquid density measurements in industry, as well as the revision of outdated reference documents, such as ISO 15212, OIML Guide 14 and WELMEC Guide 6.4.

The beneficiaries will be the production and distribution industries that use density measurements for the quality (e.g. food, chemical, pharmaceutical, petroleum, biofuels, etc.) and quantity control of their products (particularly liquid packagers, e.g. of food products and distributors, e.g. of petroleum products). The project's outcomes will significantly advance expertise on density determination at all levels of measurement down to the measurements undertaken in the production and distribution lines. An important aspect will be to provide the means to fulfil the legal metrological aspects for e.g. prepacked products. This will allow the free circulation of goods and, thus, this will have a huge economic impact not only for the internal market of these countries but also for free circulation in the EU market. This project provides the basis for the implementation of the necessary measures for the public protection of consumers. In addition, this project will produce training to disseminate best practice in liquid density metrology by means of the organisation of at least 2 national courses for end-users of these measurement methods and of the organisation of one international workshop for stakeholders, where all of the project's conclusions will be shared.

Impact on the metrological and scientific communities

The early impact of this project relates to the opportunity for a large group of less experienced NMIs and a density meter manufacturer to establish/improve their liquid density measurement capabilities at first and second-levels, i.e. by using a hydrostatic weighing apparatus and oscillation-type density meters, respectively. At the end of this project these partners will be able to establish a complete national density traceability chain at a level of uncertainty close to the more experienced NMIs (from 0.005 kg/m³ down to 0.002 kg/m³ for the hydrostatic weighing method, from 0.05 kg/m³ down to 0.01 kg/m³ for the oscillation-type density meter at atmospheric pressure and from 0.5 kg/m³ down to 0.1 kg/m³ for pressures up to 600 bar). Additionally, these NMIs will have the opportunity to establish the degree of equivalence of their liquid density measurements by means of comparisons leading to their recognition and demonstration of competence in accordance with the CIPM MRA.

The development of liquid density measurement capabilities will empower these partners to offer new/improved calibration services and to produce CRM for density, fulfilling the needs of national industries and services. They will also be able to support research activities in this field. Additionally, accredited bodies and calibration laboratories will benefit immediately from this traceability chain in their own country. Beneficiaries will include the food, chemical, pharmaceutical and petroleum industries, as well developers of new fuels and e.g. oceanographers.

Impact on relevant standards and other reference documents

The outcomes of this project will contribute to the revision of outdated ISO standards regarding the use of oscillation-type density meters (ISO 15212 [1; 2]) and it will also complement the information contained in legal metrology documents such as: OIML Guide 14 [3] regarding density measurement and the WELMEC Guide 6.4 [4] which is a guide for packers and importers of e-marked prepacked products.

Together with the EURAMET TC-M Subcommittee Density, the consortium will develop 3 EURAMET guides for hydrostatic weighing, for oscillation density meters at ambient and high pressure, and for the production of adequate CRMs for density. Targeting the end-users, the consortium will also develop a Good Practice Guide for Industry (e.g. oil, pre-packages, distilleries, etc.) addressing the potential problems arising when measuring complex fluids, e.g. two-phase fluids like carbonated beverages.

Longer-term economic, social and environmental impact

In the longer term, this project will lead to the establishment of a proper and recognised traceability chain for liquid density measurements. This will increase the confidence and competitiveness of national industries in many fields, such as: food (e.g. alcoholic and non-alcoholic beverages, drinking water, fruit and vegetable beverages, milk, food oils, etc.), chemical, pharmaceutical, petroleum, biofuels, etc. The free circulation of these goods on the European market has increased in recent years and these goods are required to meet the consumer protection requirements of European Directives, e.g. Directive 2007/45/EC, regarding the mandatory quantity control of prepacked products. This situation has intensified the number of requests to the NMIs, from these industries and from test and calibration accredited laboratories, to provide proper measurement and calibration services and CRMs. In the longer term, the emerging NMIs will be able to address these requests in their own countries. These new capabilities have the potential to create substantial economic impact in the longer term due to the vast quantities of these products on the European market. For example, according to European Commission statistics, Europe consumed approximately 60 G€ of wine (corresponding to 17 GL) in 2015 and around 6 T€ of olive oil in 2017. In addition, the economic impact of density measurements in the field of fuels can be exemplified with Norway, where over 4.2 GL of car fuels were sold in 2016 corresponding to an annual average amount of 5 G€. The tax authorities in Norway, like in the all other European countries, require reports from the fuel trade in "standard volume" which in turn requires the density of the fuel to be known.

Boosting liquid density metrology will lead to an enhanced 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.

List of publications

1. Furtado, A., Pereira, J., Schiebl, M., Mares, G., Popa, G., Bartos, P., Bebic, J. Lenard, E., Alic, A.,

- Alisic, S., Neuvonen, P., Wolf, H., Sariyerli, G., Bescupschii, A., & Laky, B. (2018). Establishing traceability for liquid density measurements in Europe: 17RPT02-rhoLiq a new EMPIR joint research project. *Journal of Physics: Conference Series* (Vol. 1065, No. 8, p. 082013). IOP Publishing. <http://iopscience.iop.org/article/10.1088/1742-6596/1065/8/082013>
2. Furtado, A., Pereira, J., Quendera, R., & Cidade, M.T. (2019). Robustness studies of oscillation-type density meters with viscoelastic fluids. In *19th International Congress of Metrology (CIM2019)* (p. 13002). EDP Sciences. DOI: <https://doi.org/10.1051/metrology/201913002>
3. Furtado, A., Gavina, J., Napoleão, A., Pereira, J., Cidade, M.T & Sousa, J. (2019) Density measurements of viscoelastic samples with oscillation-type density meters. *J. Phys.: Conf. Ser.* 1379 012020. <https://doi.org/10.1088/1742-6596/1379/1/012020>

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Internal Funded Partners:	External Funded Partners:	Unfunded Partners:	
1 IPQ, Portugal	11 INM, Republic of Moldova	12 Anton Paar, Austria	
2 BEV-PTP, Austria			
3 BRML, Romania			
4 CMI, Czech Republic			
5 DMDM, Serbia			
6 GUM, Poland			
7 IMBiH, Bosnia and Herzegovina			
8 JV, Norway			
9 PTB, Germany			
10 TUBITAK, Turkey			
RMG1: IPQ, Portugal (Employing organisation); BEV-PTP, Austria (Guestworking organisation)			