VSL Report to the CCM

April 2023

Main research and development activities in the fields of Mass, Pressure, Density & Viscosity, Fluid Flow, Chemistry.

The Mass related quantities department of VSL has expanded over the past few years to accelerate into international research in mass related metrology and ensure sufficient backup in personnel. Since then, new areas for development are being investigated. The bigger technologies of VSL remain Fluid Flow, Chemistry, and electricity, but we are also looking into options to expand in the more fundamental departments. In the field of Pressure, we are considering expansions towards both extremes of the pressure range due to new developments and questions from the field. In the department of Mass, we try to remain at a high level in calibration and are looking for collaborations in areas that are financially less demanding than most current research around the development of the Kibble balance. As VSL we are largely funded by the Ministry of Economic Affairs and Climate Policy, and have the duty to ensure metrology for enabling fair trade. That means that we combine research demands of society with current metrology themes like energy transition. For this reason, most research is within the Fluid Flow and the Chemistry department. Next to the dissemination of the measurement units themselves, we also focus on the dissemination of knowledge. Internally, where different departments come together to collaborate under the same roof on each other's challenges. We strongly believe in successful multidisciplinary metrology as the society is helped by excellent metrology for all quantities. And externally, to keep raising the importance of metrology in society and companies to make it an indispensable part of measurements.

1. Mass

The VSL mass lab remains of good quality to provide traceability to internal mass related quantities and external calibration laboratories. We recently purchased a new mass robot as a replacement for one of our current robots. We are looking to get into research topics in the field mass but most research currently is around Kibble balances which exceeds our budget. We would look forward to cooperating with other NMIs to help solve subproblems related to the redefinition of the kg. Our interest especially goes to the area of dissemination of the redefinition to represent and help NMIs outside the CCM that have equally limited budget in the mass department.

2. Pressure

In the department of pressure, we are currently looking to expand at both ends of the spectrum. The vacuum facility will be renewed with higher vacuum at improved stability. Application for accreditation in vacuum will be further investigated. On the other end of the spectrum, we are looking to expand to a standard for high pressure gas calibrations. Mainly for hydrogen applications where contamination of our current oil-based calibrations is undesirable.

Revision of pressure calibration guides EURAMET cg-3 and cg-17 (EURAMET project 1570, 2022-

2024) Recently, we joined EURAMET project 1570 for a revision of the EURAMET Calibration guidelines no. 3 (Calibration of Pressure Balances) and no. 17 (Guidelines on the Calibration of Electromechanical and Mechanical Manometers). Hereby, we look forward to contributing to the metrological pressure society and exchange knowledge on pressure calibrations. Together with our

international colleagues we will be working on a formal revision of the guidelines and look for the possible inclusion of new calibration techniques for which no calibration guideline is written yet.

EMPIR DynPT (17IND07, 2018-2021): VSL has been part of this project for the development of primary dynamic pressure calibrations. Currently, VSL is co-author in the current call of the European Partnership on Metrology for the follow-up project "Metrology for Green Maritime Shipping: Emission control through traceable measurements and machine learning approach ". The VSL departments for Chemistry, Humidity, and Data science also take part in this SRT to optimise our interlaboratory cooperation, having all these technology in one building.

3. Liquid Density and Viscosity

For Liquid Density we are looking to improve our measurement methods for viscous fluids by gravimetric density determination. Traceability for our density standard, doubly distilled demineralised water is well arranged and compared with CRMs from PTB. This is done by Oscillation type density meters. For fluids with different viscosities than that of water, dampening corrections are used which have large influence on the uncertainty. Therefore, the new methods will be developed in the upcoming year together with our volume laboratory using pycnometers.

For viscosity, we have been doing a research project to investigate a calibration service for easy to use, tabletop, rotor viscosity meters. The demand from Dutch laboratories was present, but traceable calibrations of these devices are hard to find.

4. Fluid Flow

Fluid flow, one of the biggest technologies in VSL, contains the following subsections:

- Low pressure gas flow
- High pressure gas flow
- Liquid flow & Volume
- LNG

The main developmental activity of the fluid flow group has been the build of a new facility for Liquid flow & Volume, the European Centre for Flow Measurements (ECFM). The grand opening of the new building has recently taken place together with organising the European Flow Measurement Workshop where VSL has been welcoming up to 175 participants from 33 countries all over the world. This new facility ensures calibration of big industrial liquid flow meters with improved precision and reduced uncertainty. Scalability in flow is always a tricky task and therefore the new facility creates trust in industrial metering of liquids where measurement errors can have great financial consequences. The Fluid flow & Volume calibrations are traceable to our Mass and Liquid Density laboratories where we collaborate to improve each other's calibration capabilities.

The main research activities of the fluid flow departments are given below.

Metrology for Decarbonising the Gas Grid, Decarb (20IND13, 2021 – 2024): VSL participates in the flow work package of the EMPIR project Decarb which aims to (I) develop new primary standards and traceable flow calibration facilities for metering of hydrogen (mixtures) and carbon dioxide, and (II) assess the performance of various flow measurement technologies with hydrogen (mixtures) and carbon dioxide. VSL will participate into an intercomparison for hydrogen gas flow using its mercury sealed piston prover and VSL will perform tests of flow meters with hydrogen enriched natural gas and carbon dioxide.

Improved vehicle exhaust quantification by portable emission measurement systems, MetroPEMS (19ENV09, 2020 – 2023): Within EMPIR project MetroPEMS VSL is leading the work package 3 on flow metrology of exhaust flow meters (EFM), which are embedded in portable emission measurement systems (PEMS). PEMS are used in on-road type approval tests as prescribed in real driving emission (RDE) legislation. VSL will perform calibrations of EFMs in varied circumstances applicable to on-road testing to investigate uncertainty sources and develop PEMS EFM calibration procedures.

Metrology infrastructure for high-pressure gas and liquified hydrogen flows, MetHyInfra (20IND11, 2021 – 2024)

Within EMPIR project MetHyInfra VSL is leading work package 4 in which primary standards will be developed for critical nozzle calibration at high pressures (pressures up to p_{max} = 3 MPa) and the establishment of SI-traceable measurement uncertainty of liquefied hydrogen gas flow measurement. VSL performed calibrations of liquified hydrogen (LH2) flow meters (1000 kg/h to 5000 kg/h, p_{max} = 3 MPa) with alternative (cryogenic) fluids and performed an LH2 flow measurement test campaign with the same flow meters. First steps in providing SI-traceability to LH2 flows have been made.

Metrology for the hydrogen supply chain, Met4H2 (21GRD05, 2022 - 2025)

The "Metrology for the hydrogen supply chain" project is designed to provide new and improved standards for safe hydrogen application, flow measurement, hydrogen quality assessment, and custody transfer. VSL coordinates the project. As part of the project, VSL will co-organize an intercomparison for high pressure flow measurement of hydrogen-enriched natural gas, and is developing a test facility for domestic gas meters with hydrogen.

Metrology Support for Carbon Capture Utilisation and Storage, MetCCUS (21GRD06, 2022 – 2025)

VSL is coordinating the "Metrology Support for Carbon Capture Utilisation and Storage" project. Its technical activities are focusing on: developing flow traceability for gaseous CO_2 flows, developing primary gaseous reference materials for impurities in CO_2 , validation and uncertainty assessment of the effect of impurities on the CO_2 equations of state and how this affects flow metering. VSL plans to perform calibrations with CO_2 of flow meters at pressures and flow rates directly applicable to the CCUS industry and will investigate the current state of the art of SI traceable liquid CO_2 flow measurements. The developed primary gaseous reference materials will be validated to determine stability and uncertainty, and will be used to assess the capabilities of commercial online CO_2 analysers.

LNG facility (ongoing)

VSL has performed systematic research into LNG flow and composition measurement systems providing data to establish measurement device errors directly under process conditions. The research enables to establish trust in the trade of LNG. Results are continuously communicated through VSL's media channels, conference contributions, and trade journals. VSL's primary system is traceable to mass and VSL's LNG facility is able to calibrate flow meters with a CMC of 0.17 % on mass flow rate up to 150 m³/h.

5. Chemistry

Chemistry is part of the CCQM, and the focus area is gas analysis. However, the VSL chemistry laboratory ensures SI traceability of the amount of substance through mass and purity determination. The primary preparation method of the primary standard gas mixtures is based on gravimetry and, for this, high resolution mass comparators and weighing robots are used. The research projects relevant to the CCM are given below. To perform these measurements, the

automation software of the VSL Mass laboratory is also implemented in the Chemistry laboratory to obtain accurate measurements without human intervention.

Metrology for trace water in ultra-pure process gases, PrometH2O (20IND06, 2021 – 2024): Within the PrometH2O project (www.prometh2o.eu) VSL is developing a method to generate moisture in the range of 50 nmol/mol up to 5 μ mol/mol based on permeation (ISO 6145-10) and dynamic dilution with thermal mass-flow controllers (ISO 6145-7). For this goal, a new magnetic suspension balance has been installed in the VSL laboratories that will be used to accurately determine the mass loss in time of the water permeation tube.

Metrology for oxidised mercury, MercOx (16ENV01, 2017 - 2020): VSL was leading the work package for the development of calibration methods for oxidized mercury. The methods were based on the primary mercury gas standard VSL developed in the last decade. The primary mercury gas standard works according to ISO 6145-8 the diffusion method. Using specially designed diffusion cells, elemental mercury is vaporized under well-controlled conditions (temperature, flow and pressure). By weighing the diffusion cells at regular time intervals with a high-resolution balance, an accurate mercury diffusion, or mass flow rate, traceable to the SI, is obtained.

After mixing the mass flow of mercury with accurately measured mass flows of nitrogen and air, a primary mercury gas standard has been obtained. During the project a calibration method has been developed using a dual channel analyzer for the analysis of total mercury and elemental mercury. Both channels of the analyzer were calibrated with elemental mercury. After calibration, the analyzer can be used to calibrate the output of oxidized mercury gas generators. (www.mercox.si)

Metrology for traceable protocols for elemental and oxidised mercury concentrations, SI-Hg

(19NRM03, 2020 – 2023): VSL is coordinating this normative project. In the project protocols for the calibration of mercury gas generators are developed and validated. In the past decade primary mercury gas standards and SI-traceable calibration methods for mercury concentrations in air have been developed. Notwithstanding these efforts there are no standardized procedures that ensure the dissemination of the metrological traceability from primary to working standards among calibration and testing laboratories and in emission sources. Scientifically sound calibration protocols, to assess the performance of elemental and oxidized mercury gas generators in the form of formally accepted documentary standards, are of fundamental importance to guarantee the accuracy and comparability of the mercury measurement results in air in Europe and globally. The calibration protocols developed in the project contribute to establishing a traceability chain from a primary mercury gas standards to measurement data obtained of mercury in air. The protocol thereby replaces other references currently used in mercury concentration measurements. The validation data contributes to the incorporation of the calibration protocol into new and existing documentary standards. (www.SI-Hg.eu)

Participation in relevant comparisons

Comparison ID	Subfield	Number of participants	Pilot laboratory	Status	Years
CCM.D-K5	Density of liquids	15	BEV	Measurements in progress	2017-2023
CCM.V-K4.A	Viscosity	12	CENAM	Report in progress, draft B	2018-2023
CCM.V-K4.B	Viscosity	12	CENAM	Report in progress, draft B	2018-2023
EURAMET.M.FF-S12	Air flow low pressure up to 400 m ³ /h	5	VSL	In progress	2019-2023
CCM.M.FF-K5C	Harmonized reference values for volume and volume flow rate of high-pressure natural gas	4	РТВ	In progress	2015-2023
EURAMET.M.FF-S14	Calibration and calibration method of a 1000 L proving tank.	16	MIRS	Approved	2019-2023
EURAMET.M.FF-S15	Gravimetric standards for hydrogen refuelling stations	3	LNE-LADG	In progress	2023
EURAMET.M.FF-S16	Volume 50 ml burette	14	DMDM	Measurements in progress	2022-2023
EURAMET.M.FF-S19	Volumetric glass flasks 500 mL and 5000 mL	-	VSL	Planned	2022-2024

List of relevant publications

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Schakel, M. (2021). MetroPEMS: Metrology for portable emission measurement systems; project goals and preliminary findings [abstract]. 24th ETH-Conference on Combustion Generated Nanoparticles.

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