LABORATOIRE NATIONAL DE MÉTROLOGIE ET D'ESSAIS

Report related to the CCM activities of the French metrology at LNE & Cnam

1 Main research and development activities

1.1 Pressure and vacuum

The research domain in vacuum and pressure extends from a few 10^{-7} Pa to 1 GPa; for gas flow rates from a few g/year to 100 ml/min.

The main highlights concern the studies conducted on the primary range between 1 Pa and several MPa. In these ranges, the primary static pressure references are based on the principle of piston gauges The relative uncertainty is a few 10^{-6} at 100 kPa and a few 10^{-4} at 1 Pa. Several approaches to new references based on a "thermodynamic" definition of pressure are being developed in the laboratory: an optical method based on an absolute refractometer and a method based on a superconducting microwave cavity. Both approaches are based on the determination of the molar density of a gas of which some parameters can be calculated ab initio (helium).

1.2 Mass

Kibble balance

LNE continues the developments on the Kibble balance to realise the kilogram with an aimed uncertainty of a few parts in 108 in vacuum.

After a number of various optimizations, dynamic phases were performed in vacuum. These dynamic phases "2021" show a significant improvement of the noise compared to 2017 for the determination of the field profile (the rejection factor has indeed been improved by a factor of 3). This result is the outcome of several improvement of the experimental process, notably: hollow

retroreflectors for interferometers, pillar structure for mechanical structure of the set-up, commercial nanosecond trig generator, new amplified photodetectors with higher gain and lower noise.

An automated system for the control of the optical frequency of the Nd:YAG laser (intended to measure the velocities) has been realized. It completes the optical device and the PLL, allowing locating the lines of the hyperfine structure of the di-iode used for the realization of the meter. A system of control identifies the line of interest and locks the frequency of the laser on this target. In case of loss of the servo due to an external event, the return to the frequency line is also automatic.

The force model determined by the double pendulum model was used again and carefully checked. A comparison was made with other possible force estimates: by calculating the forces from the displacement of other segments of the suspension, but also by the position of the coil with respect to the magnetic circuit. These 3 groups of force estimation are based on simple physical models, and are driven by several numerical parameters (like the length and masses of the different suspension segments for example). Their comparison allows to refine the value of these numerical parameters (known theoretically) and especially to remove most of the possible errors of instrumentation, since each model uses different information.

In case of a non-horizontal beam used as force comparator, horizontal parasitic forces exerted on the coil can cause of bias. A method of measuring the position of the beam has been developed. It has the advantage of being carried out with the habitual instrumentation of the balance, using in particular a commercial interferometer (measuring the vertical position of the end of the beam) and the Gaussian sensors (measuring the position of the coil with respect to the horizontal plane). Under the sole assumption of the verticality of the Gaussian sensor beams, it is possible to determine the inclination of the beam axis of rotation with respect to the horizontal and the inclination of the beam longitudinal axis with respect to the horizontal plane.

Various vacuum measurements were performed from July 2021 to September 2022. Especially, mass measurements were carried out for the comparison CCM.M-K8.2021: the mass standard was a 500 g iridium, identified as DB1. DB1 was use to calibrate 500 g iridium mass DB2 and the stack 1 kg PtIr JM15 with mass comparator. Its mass was determined with a type-uncertainty of 108 µg. This uncertainty should be considered as the outcome of the first dry run under vacuum of the LNE Kibble balance.

Electrostatic force balance

LNE has begun the conception and machining of an electrostatic force balance, aiming at realizing the mass unit at the milligram level in the International System of Units (SI).

The most important parts are designed, machined and assembled: a mass balance mechanism, two circular capacitance actuators (for balancing the force to be measured and for changing the vertical position). The balance mechanism is a fully monolithic parallelogram with 40µm-thick flexure hinges. The two actuators are fixed on the same side of balance mechanism, with their own monolithic adjustment system. Special attention has been paid to the overall symmetry of the system and to cost efficiency.

By using a commercial interferometer, an 8.5 digits voltmeter and ±1000V-source driven by a custom digital PID, the study of the vertical stiffness was done, in particular its dependence (from 8 N/m to lower than 0.01 N/m) relative to the position of the mass centre of the balance mechanism. A very rough weighing of a 2mg-mass was carried out.

The next steps will be to lower the stiffness by a non-gravitational method, to study the influence parameters for mass measurement standard deviation and to measure the capacitance gradient.

1.1 Force and torque

1.1.1 Force deadweight standard machine at 5 kN

Method for re-calibration of the masses, without removing them from the machine has been applied. Results are satisfying for the whole set of masses as they have not significantly drifted since they were first calibrated in 2000. This method leads to a higher uncertainty compared to the original weighing. The global uncertainty on the forces (F) generated is then increased: 2.0 x 10^{-5} F (k=2) but is still competitive with the best uncertainties observed at the international level. The calibration for each mass individually will be made for the 5 kN machine.

1.1.2 Climatic chamber for the 5 kN force standard machine

A climatic chamber for the 5 kN force standard machine has been developed and received. Like the climatic chambers for the 50 kN and 500 kN force standard machines, it will allow to perform measurements of force transducers in a controlled thermic environment from -15°C to + 50°C.

1.2 Participation in research projects

1.2.1 EURAMET (EMRP)

- JRP 18SIB04: Towards quantum-based realizations of the pascal

The project started in 2019. The overall objective is to develop two new primary pressure standards based on a quantum approach directly traceable to the SI, and to improve absolute pressure uncertainties especially in the range 1 Pa to 10 kPa.
The new standards developed by LNE-CNAM will be based on a Fabry-Perot cavity refractometer and a micro-wave cavity.
Three departments were involved in this project: pressure, length and temperature.
The project ended in Dec. 2022.

- JRP 21GRD05: Metrology for the hydrogen supply chain (Met4H2)

The project led by the VSL, started on October 1, 2022. LNE will work on two methods for hydrogen leak detection:

- Development of a flowmeter for leakage rates relative to the atmosphere in the range of 10-6 to 10-9 mol/s using the technique of measuring the pressure change in a known volume.
- Evaluation of a refractometry-based method for leak detection at atmospheric pressure in the range 10^{-7} to 10^{-9} mol/s.

The two methods will be compared in their common measurement range. The design of a new Fabry-Perot refractometer is in progress and should be based on a Fabry-Perot refractometer with a double cavity in Invar.

2 Participation in relevant comparisons

2.1 Euramet Comparisons

- EURAMET M.F K3: Force comparison at 1 MN

PTB pilots this comparison. Measurements at LNE were carried out in 2014. Sixteen laboratories participate. Draft B has been submitted to the participants in august 2022. The results are satisfying for LNE.

- EURAMET M.T-S4 (Project 1304): Torque comparison from 0.1 up to 50 N·m.

Measurements with PTB have been performed in 2017. Uncertainties claimed by LNE, lower than CMCs, were not supported. But LNE registered CMCs were supported. The final report is underway. A study comparison was carried out with CEM. The results are satisfying and confirm the uncertainty claimed of the 50 N·m LNE reference.

- EURAMET M.T-S5 (Project 1428): Torque comparison up to 5000 N m.

In order to verify the validity of the protocol, a preliminary study comparison was carry out with CEM and NMIJ. Results are satisfying. The official comparison with PTB have been registred. Measurements will start when M.T-S4 and M.T-S6 will be completed.

- EURAMET M.T-Sx: Torque comparison up to 5 N m.

A preliminary study comparison was carried out with NMIJ to validate the protocol. Results were not satisfying (instability on NMIJ measurements), a new preparatory comparison will be planned, with PTB.

- EURAMET M.T-S6: Torque comparison up to 500 N m.

A preliminary study comparison was carried out with CEM to validate the protocol.

- EURAMET F-1479: supplementary comparison, calibration of a 1000 L proving tank

The aim of this project was to compare results and method calibration of a 1000 L proving tank. The results are satisfying

2.2 CIPM comparisons

- Key comparison in the field of R-134a refrigerant leaks.

During the CCM pressure meeting in February 2014, LNE proposed to pilot a key comparison for leaks flowing to the atmosphere (R-134a gas, for leak rates around 3 grams per year (g / a)). This

comparison has been initiated in 2020, the characterization of the transfer standard is currently under study.

- CCM.P-K16, CCM.P-K17, CCM.P-K18

These key comparisons piloted by the CENAM and the PTB are under way, with drafts expected in 2023.

LNE carried out its measurements in July and August 2022. The transfer standard is a Fluke PG7601 pressure balance equipped with two sets of 10 and 200 kPa/kg cylinders.
The results show a consistency between our pressure standards better than 2ppm.

- EURAMET M.F K23 : Force comparison at 200 and 500 N

METAS pilots this comparison. LNE reference transducers are used. Measurements are completed and draft A has been submitted to the participants, results are satisfying for LNE.

- CCM.T-K3: Torque comparison at 50 N·m:

PTB pilots this comparison. The redaction of the protocol is ongoing.

- CCM.F-K1: Force comparison at 5 kN

TUBITAK UME pilots the comparison. The measurements at LNE were completed in January 2023, the comparison is ongoing.

- CCM.M-K8.2021: key comparison of kilogram realizations. Published 26 January 2023.

BIPM piloted this comparison. LNE measurements were made from November 2021 to February 2022.

3 List of relevant publications of the French metrology

- Pierre Otal, Étalons de pression, Les Techniques de l'ingénieur, 2022

- K. Jousten, F. Boineau & al Evaluation and metrological performance of a novel ionisation vacuum gauge suitable as reference standard, Measurement 2023

- F. Boineau, M. D. Plimmer, A technique to improve low leak rate measurement with a constant-volume flowmeter, IMEKO International Conference 2022

- Méthode de mesure du volume d'un solide et pycnomètre associé ; F. Boineau ; Brevet LNE

- T. Rubin, D. Mari, Z. Silvestri, D. Bentouati, O. Axner, M. Zelan et al., "Quantum-based realizations of the pascal' status and progress of the EMPIR-project: Quantumpascal", Joint IMEKO TC3, T5, TC16 and TC22 International Conference, DOI: 10.21014/tc16-2022.103.

- Hautes pressions Domaine de 50MPa à 2 GPa, Les Techniques de l'ingénieur, Pierre Otal, Jean-Claude Legras, 2021

- Metrological performance of a novel ionisation vacuum gauge with respect to that of the commercial gauges commonly used as reference standards, F. Boineau & al. CIM 2021

- Méthode de mesure du volume d'un solide et pycnomètre associé ; F. Boineau ; Brevet LNE; date dépôt : 14 avril 2021

- B. Blanc, F. Beaudoux, “Monte Carlo method applied to gravimetric leak calibration”IMEKO 24th TC3, 14th TC5, 6th TC16 and 5th TC22 International Conference 11 – 13 October 2022, Cavtat-Dubrovnik, Croatia

- M. Stock et al, Final report CCM key comparison of kilogram realizations CCM.M-K8.2021, 2023 Metrologia **60** 07003