CCM Working Group Mass Standards

Report to CCM on activities from 2002 to 2005

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1 Research, development, calibrations

1.1 Research and development

1.1.1 Surface investigations

NPL: Surface studies on Silicon, platinum-iridium and stainless-steel in air and in vacuum have been made.

BNM/INM: Some new experiments have been developed in last years in order to increase the knowledge of the behaviour of standard mass: thermal adsorptions spectrometry (a new system is built and the results in junction with a mass change have been obtained), a new system for roughness measurement using X-ray techniques.

METAS: A surface analysis project using combined methods inside a 1 kg vacuum mass comparator has been started in March 2004. This method should allow to perform quantitative analysis of the chemical composition of adsorbed layers (depth profiles) on mass standards. This analysis with Angle Resolved X-Ray Photoelectron Spectroscopy (ARXPS) will be performed under the same environmental conditions used for the weighing (gravimetric method), directly on mass standards up to 1 kg.
The developed instrumentation will be directly applied to the characterization of possible new alloys, candidates for the test mass of the Watt balance experiment. The results obtained would allow to validate the final choice of this material and insure the traceability of the mass scale between air and vacuum measurement.
The coupling method (ARXPS and gravimetric method) should also deliver useful information on the short- and long-term stability of mass standards, on the reproducibility of the mass for air-vacuum cycles and for different environmental conditions.
The new 1 kg vacuum mass comparator is already built (Figure 1). A vacuum level of 1×10^{-5} Pa was already achieved in the mass comparator vacuum chamber. The surface analysis system with a goniometer to move the mass standards is expected to be ready before the end of 2005.

Figure 1 The new METAS 1 kg vacuum mass comparator coupled to the surface analysis system

BNM-INM participated in and coordinated the EUROMET Project 551 related to the measurement techniques to characterise the roughness of mass standards. Measurements were made on two OIML weights (500 g and 2 kg), on a 50 g cylindrical mass in stainless steel and on a 100 g cylindrical mass in alacrite. Five laboratories participated in this project: BNM-INM, NAWC (US), SP (SE), LPUB (FR) and NIST (US)). Five systems are involved (3 based on optical methods and 2 on contact-type profilometer (Talystep and near-field microscope). This project is now completed and the final report (BNM-INM/CNAM/Ma-2003-01) sent to the participants. Following this project, two publications are submitted to Metrologia.

KRISS: Adsorbed mass on the national prototype of the kilogram (Pt-Ir) has been estimated.

INMETRO: Investigation of water adsorption on metal surfaces is planned.

1.1.2 Air density

NPL: Work on large volume (600 cm^3 difference) air density artefacts with a view to further improving the uncertainty of air density measurement has been completed.

CNAM/INM: The transparency of dew point hygrometers associated with enclosures of mass comparators has been checked and quantified by means of an hygrometer based on absorption of a specific wavelength of the vapour of water. The influence of switching on/off the dew point hygrometer has been measured ; the corresponding change in water vapour pressure and in the density of air has been estimated to – $0{,}6 \times 10^{-4}$ kg/m^3 due to vapour condensation on the mirror.
The argon content in air has been measured using a mass spectrometer. The results obtained in 2004 show values in agreement with previous measurements but with an uncertainty too high to bring a conclusion.

KRISS: Experimental determination of the air density by using buoyancy artefacts have shown a significant discrepancy in respect to the CIPM formula.

NIM: To determine the air density by experimental method, a pair of air artifact is under production in China. It will be used to determine the air density. The result will be compared with that of the climate kit, A30V after the installation of the M-one mass comparator.

Comparisons between buoyancy artefacts for improving the buoyancy correction have been made in EUROMET project 144 with the participants PTB (coordinator), NPL and BIPM and in a bilateral comparison between PTB and KRISS. The results of the measurements have shown a significant deviation of the air density from the value of the CIPM formula. The supposed wrong value of the Ar content in air used with the CIPM formula has been confirmed by a new determination of the Ar content at KRISS.

1.1.3 Magnetism

NIM: To study the influences from the magnetic properties (magnetization and susceptibility) of mass standards, a BIPM susceptometer was delivered in June 2004. The measurements of the influences from the magnetic properties of weights have been carried out since that. At the first beginning, the measurement was focused on the Class E_1 and E_2 weights with density of 8000 kg/m^3 and austenitic stainless steel. From September 2004 to January 2005, different weights with different magnetic properties were used to do measurements on varies kinds of balances, including mechanical Balances and electronic balances. But the results were not clear. In October, 2004, further measurements were carried out on different types of susceptometer, the BIPM one and another one produced by Sartorius, Germany. A new paper will be released on APMF'2005 meeting in Korea.

CENAM: A susceptometer has been manufactured and the magnets have been characterized for a secondary laboratory in Mexico with the support of R. Davis/BIPM. This laboratory offers the approval model for new weights in Mexico.

1.1.4 Other activities

NPL: Works have been done on stability of fraction mass standards, extension of the mass scale below 1 mg and also measurement of nano-forces.
A comparison of silicon artefacts has recently been completed with BIPM.
Monitoring of the gold plated copper weights used for the NPL Watt balance experiment is ongoing.
Weighing of silicon spheres on the NPLone primary vacuum balance (as part of a comparison piloted by CSIRO) were made.
Properties of materials for use in Watt balance constructional components have been investigated (particularly the coil former).

BNM-INM: A calibration of the national prototype (kilogram no. 35) took place in April 2002. The value of the mass allows to know the drift of this artefact

since the third periodic comparison. It shows a good agreement between the value measured and the predicted value using the law given by Girard in his paper (Girard in Metrologia 31 (1994), 317-336).

PTB: After calibrations of the Pr-Ir prototypes no. 55 and no. 70 at BIPM and their comparison with the national prototype no. 52 at PTB an excellent agreement with the corrected mass value of no. 52 has been found. A correction is made at any time of use, based on known mass changes in the past.
In the frame of the Avogadro project, the mass of the silicon sphere AVO#1 has been determined, whereby the buoyancy correction has been done by use of the PTB buoyancy artefacts. The standard uncertainties between 19 µg and 25 µg are considerably less than those with the use of the CIPM formula (43 µg). Comparisons between AVO#1 and AVO#2 were made with standard uncertainties of 5 µg and 6 µg.
For the ion accumulation experiment aimed at monitoring and redefining the kilogram a new ion source for bismuth has been installed and modified. A current of about 5 mA of the ion beam has been measured, more than one order of magnitude larger than the maximum achievable current with the former gold ion source. A new accumulation experiment is under preparation.

CNAM/INM, LNE and many other laboratories are involved in the French Watt balance project. INM is in charge of the main following points:

- Mass (alloy, characterisation of surface with the techniques previously developed as TDS, mirage effect, optical roughness meter, X-ray roughness meter, change in mass during the vacuum to air and air to vacuum process, in cooperation with LNE)
- The force comparator in charge to established the equality between the gravitational force and the electric force due to the interaction between the current and the magnetic field
- The mechanical link between the comparator and the coil to ensure the same beam length for the weight and the electric force.
- Mechanical system to ensure vertical translation during the dynamic phase

METAS: For the project "Surface analysis using combined methods" METAS purchased in 2005 a pair of Pt-Ir surface (or sorption) artifacts. The solid cylinder is the prototype Nr. 89. The second artifact, which is a 1 kg stack of 8 Pt-Ir disks, will be also manufactured at BIPM.
METAS hosted in November 2004 the third annual Watt Balance Technical Meeting. All active teams in this field were represented (NPL, NIST, BNM, METAS and BIPM). The main subjects were: status of progress report from each experiment, data acquisition and results processing, alignment and surface effect on mass standards.

IMGC: A number of comparisons were carried out on a set of six 1 kg stainless steel standards, both in air and in vacuo. The results are under evaluation.
In the framework of the Avogadro project, a number of silicon density standards were directly compared to the Pt-Ir kilogram 76. The typical standard uncertainty of this measurement is 50 µg.

The third-generation prototype (ground-model) of an Inertial Centrifugal Balance (BIC 3) was constructed. This instrument is intended for operation in a microgravity environment, such as the International Space Station. It can weigh any kind of objects, including liquids, powders or gel, up to 100 g with a standard deviation of 1 mg and an expanded uncertainty of 3 mg. The measurement is carried out according to an original method which avoids the knowledge of the centre of gravity of the object (Fig. 1).

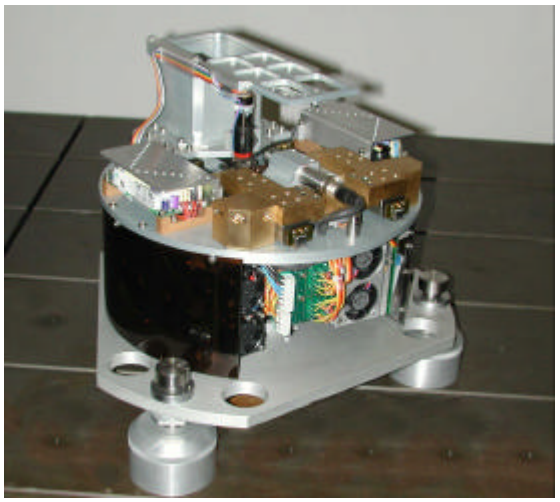

Fig.1. The BIC 3 Inertial Centrifugal Balance of IMGC.

VNIIM: The collaboration with MIKES/Finland exploiting recent developments in growth of niobium films on large (100 cm^2) monocrystalline sapphire substrates in continuing. The system will consist of a superconducting levitated frame weighing 100 g which is guided magnetically. The frame lifts a separate, spherical, mass standard of 100 g. They have built the interferometric system for measuring the vertical displacement of the levitated body. The project has a target uncertainty of a few parts in 10^{-8}. At present, details of the time required to reach the target are not available. The project has a total staff of five: three of MIKES and two of VNIIM.

NIM: The prototype balance C1000S has been improved. The comparison between the national prototype kilogram and a stainless steel weight is carried out under ambient conditions using C1000S. To improve the measurement accuracy, and for other studies, a vacuum mass comparator, M-one, with 6 positions, will be installed at the beginning of March, 2005. A set of weights, with combination of 5, 5, 2, 2, 1, 1 and of cylindrical shape have been produced and are under stability test since October, 2004. The density of the weights is quite close to 8000 kg/m^3, and the magnetic susceptibility of each weight is less than 0.0004.

CENAM: As part of the research to improve the uncertainty of the national mass scale, mass measurements were carried out with the reference standards of stainless steel and the national prototype no. 21 using the HK 1000 MC comparator. The air density inside the chamber is kept constant. In order to validate the uncertainty estimation of the calibration procedures used (Gauss Markov and Minimum Variance Methods) a Numerical Simulation Method (Monte Carlo Method) was applied as part of this project obtaining excellent results. A report of the results and procedure used was published in

proceedings of Simposio de Metrologia 2004, Queretaro Mexico.
A similar sealed chamber was set up for the C1000S Balance.
With the purpose of monitoring the mass stability of the national prototype no. 21, CENAM purchased the new Pt-Ir prototype no. 90 from BIPM which arrived at CENAM in January 2005.

KRISS: Calibration designs for mass standards for two comparators in a decade have been studied.

INMETRO: Realization of the mass scale from 1 mg to 1 kg of stainless-steel weights with the link to the national prototype no. 66 is in process.

INMS/NRC: Drifts of standards with time have been assessed.
Aerostatic estimates of the densities of weights have been made for weights smaller than 100 g.

1.2 Calibrations

NPL: Calibrations of mass standards from 1mg to 500 kg are ongoing

NIST: Disseminations from primary Pt-Ir standard to stainless steel working standards, automated mass measurements from 1 mg to 50 kg, measurements at constant pressure/vacuum with a 1 kg comparator are ongoing.

2 Mass laboratories and equipment

2.1 Staff

METAS: Dr. Peter Fuchs is the new head of mass and weighing instruments laboratory. The total staff of the mass and weighing instrument laboratory includes now 3.5 persons.

PTB: Dr. M. Borys is the new head of the mass working group and Dr. M. Gläser is now head of the department Solid Mechanics (force, torque, periodic force and ion accumulation)

2.2 Laboratory rooms

NPL: The mass area is due to move into new facilities in June 2005.

METAS: A new calibration laboratory (for verification offices) was established with new mass comparators including a robotic system for 20 kg weights (Mettler-Toledo M10-AX32004). This activity moved from the Legal Metrology section to the Section Mechanics.

NIST: New mass laboratories in Advanced Measurement Laboratory (AML) moved in August 2004. Special features of AML labs: tight temperature ($\pm$ 0.01 ^{0}C and $\pm$ 0.1 ^{0}C) and humidity ($\pm$ 2 %) controls, clean room class 1000 and vibration isolation.

NMi: The offices of NMi VSL have moved to the new building. The new visiting address is :

Thijsseweg 11
2629 JA Delft
The Netherlands

P.O box, telephone, fax, etc. have remained the same.
The laboratories of Mass, Pressure and Viscosity are not yet moved due to problems with the air conditioning. It is expected that these laboratories will be moved soon.

2.3 Equipment

METAS: Two new air tight chambers were installed for a 100 g mass comparator (Mettler-Toledo AT106H) and for a 10 kg mass comparator (Mettler-Toledo AT10005).
A total of four air tight chambers, equipped with membrane pumps and new software for pressure regulation, allow mass determinations at constant air density in the range from 10 g to 10 kg. This way the best possible uncertainty of measurement could be achieved for example for the calibration of the 100 g test mass used in the Watt balance experiment.

IMGC: The 1 kg and 10 kg balances were both equipped with home-designed air tight enclosures. The balances were suitably modified and re-aligned for operating in vacuo.

Nmi: Developments in the field of mass are :
- new automatic weighing device (1 kg to 10 kg) in co-operation with R. Spurny
- new project aerostatic weighing project started in 2004 (will end in 2006)
- new vacuum calibration facility (10^{-8} hPa - 10 hPa)

GUM: In 2004 following equipment arrived:
A 10 kg mass comparator Sartorius CC 10000 U-L, $s \leq 0{,}05$ mg
A 50 kg comparator balance Mettler-Toledo
Ten pieces of 1 kg stainless-steel mass standards
Five pieces of 10 kg stainless-steel mass standards
18 pieces of 50 kg stainless-steel mass standards (class F_1)
A 1000 kg mass standard (class F_2)

2.4 Quality system

NPL: Accreditation by the United Kingdom Accreditation Service (UKAS) is completed.

NIST: Development and implementation of ISO 17025 quality system is completed. Internal assessment is completed and accepted by SIM Quality System Task Force Group.

INMS/NRC: The Mass Standards Program underwent a third party accreditation visit according to ISO 17025 in November 2004.

CENAM: In May of 2004, CENAM has been Peer Reviewed for the Mass and Density CMC's. As conclusion of the Peer Evaluation, CENAM is able to support its CMC Claims. The results of the Peer Review were presented for review and approved in the SIM Quality System Task Force Working Group (SIM QSTF) on November 2004.

INMETRO: In February, INMETRO has been Peer Reviewed for mass.

3 News from the NMIs

LNE: A French Metrology Reform has taken place. The Government has just decided to reform the management organization of the French metrology to optimise the means and to carry much weight at the European and international level in the context of the creation of networks of excellence. A joint decree of the Ministries of Industry and Research has just performed the dissolution of the "Bureau national de métrologie" and the transfer of the control task of metrology to LNE.
On that occasion, the LNE's name has been changed into « Laboratoire national de métrologie et d'essais", while keeping the well-known initials LNE. The BNM's teams became integrated into the LNE. Luc ERARD, BNM's former director, has recently become appointed director of this new LNE directorate.
The LNE will ensure the monitoring of the French metrology with the support of a metrology committee of fourteen people.
The new organization will provide more flexibility. Joint units or common laboratories in particular shall be created, which would enable to provide work on the same site to scientific teams from different business enterprises relying on pooled reference facilities.

Nmi: Organizational changes are that Mass & Related Quantities is now merged with Chemistry, Flow and Temperature into one large department MCST. Also the general director of NMi, Mr. C. Gouwens has retired and Mr. J. de Ridder has taken over his job.

PTB: PTB has been re-organized in 2003. The number of departments has been increased and their staff was reduced to numbers between 15 and 25. The former laboratories were re-named to working groups. Dr. M. Borys is now head of the working group 1.11, Realization of Mass. Dr. M. Gläser is head of the department 1.2, Solid Mechanics (force, torque, periodic force and ion accumulation).

4 CIPM key comparisons

The CIPM key comparisons

CCM.M-K1 (1 kg, pilot: BIPM)
CCM.M-K2 (10 kg, 500 g, 20 g, 2 g, 100 mg, pilot: PTB)
CCM.M-K3 (50 kg, pilot: LNE)

have been finished. The results and final reports are implemented in the KCDB and are published in Metrologia Technical supplement.

CCM.M-K4 (1 kg, pilot: BIPM) is under preparation. The travelling standards, four sets each comprising two pieces 1 kg stainless steel standards are being tested for stability at BIPM. Provisionally starting time will be in 2005.

CCM.M-K5 (2 kg, 200 g, 50 g, 1 g, 200 mg, pilot: NMIJ/AIST): Measurements are completed, Draft A report is under preparation.

Considering the 10 years repetition interval, decided by the WGM, the next CIPM key comparison will start in 2011 with CCM.M-K6 (50 kg) followed by CCM.M-K8 (5 kg, 100 g, 10 g, 5 g, 500 mg) in 2012 and by CCM.M-K7 (1 kg) in 2015.

5 RMO key comparisons

EUROMET.M.M-K1 (1 kg, project 215, pilot: NPL): Measurements have been completed in 1999, Draft B report is available, final report is under preparation.

EUROMET.M.M-K2 (10 kg, 500 g, 20 g, 2 g, 100 mg, project 445, pilot: SP): Measurements are completed, final report is under preparation.

EUROMET.M.M-K2.1 (10 kg, 500 g, 20 g, 2 g, 100 mg, project 786, pilot: SP) is under preparation. This KC will comprise those EUROMET countries that didn't participate in EUROMET.M.M-K2.

EUROMET.M.M-K4 (1 kg, project 510, pilot: NPL): Measurements are completed, Draft B is available, final report is under preparation.

EUROMET.M.M-S1 (500 kg, project 461, pilot: CMI): Measurements are completed, Draft A is available.

APMP.M.M-K1 (1 kg, pilot NIMT) has been finished. The results and final report are implemented in the KCDB and are published in Metrologia Technical supplement.

APMP.M.M-K2 (10 kg, 500 g, 20 g, 2 g, 100 mg, pilot NPLI) is under preparation.

COOMET.M.M-K1 (1 kg, pilot SMU): Measurements are completed. Two sets of results are available. The discussion on the reference value took place. Draft A is under preparation.

COOMET.M.M-K2 (10 kg, 500 g. 20 g. 2 g. 100 mg, pilot VNIIM): The protocol is available in Russian language and is being translated into English. VNIIM is preparing two complements of five comparison sets of mass standards of nominal values: 200 mg, 1 g, 50 g, 200 g, 2 kg with different densities and volumes made of steel. VNIIM is determining the density and volume of the standards as well as calibrating each of the sets.

SADCMET.M.M-K5 (2 kg, 200 g, 50 g, 1 g, 200 mg, pilot: CSIR-NML). The protocol is available.

SIM comparisons

SIM CAMET mass comparison (SIM.7.16.b, 2 kg, 1 kg, 200 g, 50 g, 1 g, 200 mg, pilot: CENAM): Measurements have been completed in 2003, final report is published in Proceedings of Simposio de Metrologia 2004, Queretaro, Mexico.

Three bilateral mass comparisons (2 kg, 1 kg, 200 g, 50 g, 1 g, 200 mg) between CENAM and INTI (SIM.M.BK5.c), NRC and INMETRO (SIM.M-BK5.b) and between NIST and LATU (SIM.M-BK5.a). Measurements CENAM-INTI were completed in 2003, a common report is under preparation.

Three bilateral comparisons (SIM.7.15.P, 1 kg) between LATU and NIST (SIM.M-BK4.a), INMETRO and NRC (SIM.M-BK4.b) and CENAM and INTI (SIM.M-BK4c). Reports are in progress.

A bilateral comparison (1 kg) between INDECOPI and LCN-CESMEC. Report is in progress.

SIM ANDIMET mass comparison (1 kg, 100 g, 20 g, 5 g, 100 mg, pilot: CENAM): Measurements are ongoing.

Inter regional SIM mass comparison (2 kg, 1 kg, 200 g, 50 g, 1 g, 200 mg, pilot: CENAM) with one participant of each of the subregions CARIMET, CAMET, ANDIMET, SURAMET and NORAMET: Measurements are ongoing.

Bilateral comparison (500 mg, 200 mg (2), 100 mg) between CENAM and INIMET (Cuba, COOMET): Measurements have been completed in 2004, report is under preparation.

Further comparisons: SIM.7.1, SIM.7.11.P, SIM.7.12.P, SIM.7.13.P, SIM.7.17 (1 g – 1 kg)

6 Other international comparisons

An intercomparison on magnetic susceptibility and remanent magnetism has been initiated by the CCM WG Mass Standards with NRC as pilot laboratory. The measurements are about to be finished. A report is under preparation.

EUROMET organized a comparison with two Pt-Ir standards of NPL and PTB comprising 18 participants (EUROMET project 509, pilot: NPL). The measurements are finished and the results are being evaluated.

INMETRO participated in a Accredited Laboratories (IAAC) comparison (1 kg, 100 g, 10 g, 100 mg, 1 mg) with 15 further laboratories of North-, Middle- and South-America

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