

## **BIPM Physical Metrology Department**

**Director: M. Stock**

**(1 January 2015 to 31 December 2015)**

In October 2015 the staff and the activities of the Electricity and Mass Departments were merged into the new Physical Metrology Department. The work in the electricity laboratories is described in Section 1, that of the mass group in Section 2.

### **1. Electrical metrology**

#### **1.1 Electrical potential difference (voltage)**

##### **1.1.1 Compact NIST 10 V programmable Josephson voltage standard for ac voltage measurements (S. Solve, R. Chayramy)**

During the secondment of Dr Solve to the NIST, from July 2012 to July 2013, a programmable Josephson Voltage Standard (PJVS) with compact electronics, which was designed to be transportable, was developed for the BIPM under a cooperative research and development agreement. This new system is intended to be used to develop a future comparison scheme for AC Josephson voltage standards. In 2015, it was used for the first time for an on-site comparison, mainly to test its suitability for transportation and use in another laboratory. The comparison was performed with the NMIJ, Japan, at 10 V DC against one of their programmable Josephson voltage standards. This was the first direct comparison of programmable primary voltage standards of different technologies; it led to an excellent agreement of a few parts in  $10^{12}$ . The BIPM PJVS was found to be very stable in terms of the repeatability of its biasing parameters. It was also found to be robust against trapping of magnetic flux.

Based on the work of 2014 on the leakage resistance to ground during the comparison with the NMIJ, different grounding configurations of the measurement setup and their influence on the measurement noise were investigated. It could be demonstrated that the capacitors of the filters on the precision measurement leads can introduce a systematic error of up to a few parts in  $10^{10}$  depending on where the leakage current flows. Precautions must be taken in the implementation of filters on PJVSs in order to limit the leakage resistance error.

The current version of the new BIPM transportable quantum voltage standard is not yet fully operational for the generation of AC stepwise approximated signals. The generation of such signals is already possible, but they cannot yet be synchronized in phase with an external AC signal.

##### **1.1.2 Quantum voltage reference for the watt balance (S. Solve, R. Chayramy)**

The measurement of the induced voltage in the BIPM watt balance will be carried out using a programmable Josephson voltage standard. If the watt balance is operated in the special one-phase mode, where the force and velocity modes are carried out simultaneously, a second Josephson system will be needed for the determination of the coil current, measured as the voltage drop over a calibrated resistor. The work on these systems is described in Section 2.4 on the watt balance.

### 1.1.3 Upgrade of the automatic Zener measurement system for measurements at 10 V (S. Solve, R. Chayramy)

Work continued on the implementation of the automated Zener measurement setup throughout the year. The final aim is to replace the conventional primary voltage standard based on underdamped Josephson junctions and its associated measurement setup. To test the correct functioning of the new automated system, the 12 BIPM Zener secondary voltage standards were successively calibrated against the two measurement setups several times. For most of the standards, the results obtained with the two different measurement systems were in good agreement, with differences varying from 5 nV to 90 nV for the 10 V output. However, the difference can reach up to 500 nV for the remaining standards, which is not acceptable.

Calibrations of the 1 V outputs of the same Zener standards did not show a similar difference. The assumption of a leakage error at the level of the 10 V output of the Zeners was investigated by changing the grounding configuration of the PTB-SNS array in the automatic system but no evidence of leakage could be identified. A number of other tests were carried out but could not explain the observed offset. The Josephson voltage standards used for the two calibration systems were directly compared and showed agreement within a few parts in  $10^{11}$ . The output voltages of the Zener standards are corrected for changes of their internal temperature. The correction coefficients had been determined more than 10 years ago and there is the possibility that they might have changed with time. A measurement setup to re-determine the temperature coefficients has been developed and a measurement campaign will be carried out in 2016. The results are also of importance for the comparison of Zener calibrations, BIPM.EM-K11, which the BIPM offers to NMIs and for which the Zener standards are used.

## 1.2 Electrical impedance (resistance and capacitance)

### 1.2.1 DC resistance and quantum Hall effect (N. Fletcher, P. Gournay, B. Rolland)

The quantum Hall resistance (QHR) was realized several times during 2015, as required to maintain traceability for resistance calibration and comparison services and also the link to capacitance. Continuing improvements have been made to the BIPM's set of transportable equipment for on-site QHR comparisons. The last of a set of three transportable thermo-regulated resistance standards has been brought into service, and regular verifications of the critical 1 Hz-dc coefficients of these new standards have been undertaken to establish confidence in their use with the transportable 1 Hz bridge.

In collaboration with MIKES/VTT and PTB a new generation of low frequency current comparators have been investigated. The new comparators, built at MIKES, offer some improved characteristics over those presently used at the BIPM, and gave good results when substituted in our existing bridge electronics.

Some graphene QHR samples (developed in a collaboration between MIKES-VTT and Aalto University (Finland) using SiC wafer technology) were investigated at the BIPM, and demonstrated equivalence to the existing GaAs reference for calibration work. Further sample improvements are required to achieve an accurately quantized QHR plateau under simplified experimental conditions (e.g. 4.2 K temperature and 5 T magnetic field, as compared to the existing requirement of 1.5 K and 10 T). A commercial graphene sample that promises to meet these conditions has been obtained, and will be tested in early 2016. If successful, this could greatly simplify the logistics and cost of performing on-site QHR comparisons.

### 1.2.2 Calculable capacitor (P. Gournay, N. Fletcher, L. Robertsson)

A new stabilized laser source for the interferometer has been developed to replace the previous source which had shown instabilities. Unlike the previous source, this one has been fully developed at the BIPM and allows fine control of all tuning parameters, leading to a much better stability of the laser frequency. Moreover, a frequency

beat can now be carried out with the stabilized laser source of the watt balance by creating interference between beams derived from both sources. During capacitance measurements with the calculable capacitor this beat frequency is now recorded to identify eventual frequency jumps, which lead to step changes of the realized capacitance.

A new series of measurements carried out with the new laser source have confirmed its good functioning and have allowed the BIPM to perform the first tests of the voltage dependence of the calculable capacitor. The results are encouraging but not yet conclusive. The limit is currently our knowledge of the voltage coefficient of the standard capacitors implied in these measurements, which need to be determined more precisely.

As evidenced by the characterization measurements performed in 2014 and confirmed in 2015, the present alignment accuracy of the electrode bars and a few other minor problems (requiring disassembly of the calculable capacitor) limit the uncertainty of the determination of the von Klitzing constant to about 2 parts in  $10^7$ . The weak point of the aligning method has been identified and a new aligning tool, designed by the NMIA, Australia, has been constructed and calibrated during 2015 at the BIPM. It will be used in early 2016 to re-align the electrode bars. Before re-alignment, the calculable capacitor will be disassembled and moved into a new laboratory with a floor comprising a large concrete block. This should limit significantly the effect of ground vibrations currently seen on the bridge balance signal of the calculable capacitor bridge.

The expected improvements from this work should allow the BIPM to measure the von Klitzing constant to better than 5 parts in  $10^8$  in 2016.

### 1.2.3 Maintenance of a reference of capacitance (P. Gournay, R. Chayramy, N. Fletcher)

The unit of capacitance is maintained at the BIPM by a group of four 10 pF standard capacitors. They are calibrated every six months against the dc-QHR through a quadrature transfer. During 2015 this transfer was carried out a third time in-between the two normal ones due to the participation of the BIPM in the EURAMET.EM-S31 comparison of capacitances at 10 pF and 100 pF. In effect, following the significant discrepancies found after the first circulation of the standards in 2011, this comparison is still ongoing and a second circulation started in 2015. This time it includes the NMIA in order to obtain at least one result traceable to a Thompson-Lampard capacitor. As the BIPM received the standards two months apart, before and after the measurements in Australia (to keep track of possible drift of the standards during transportation), the reference group of capacitors was calibrated once more against the QHR to achieve the best possible measurement uncertainty. This also required verification of the entire measurement chain from the QHR to the 10 pF reference standards, including in particular:

- the frequency corrections of the standard transformers at the operating frequencies (measurements were carried out at 1027 Hz, 1541 Hz and 3082 Hz),
- the frequency corrections of the ac-resistors of the quadrature bridge against a coaxial and calculable Haddad resistor,
- the drift of the 10 pF reference group of capacitors,
- the frequency dependence of the voltage injection devices.

The final results of this second circulation should be known in the first half of 2016.

As usual, the reference group of capacitors has also been used during 2015 for calibrations for NMIs (see Section 1.4) and for a bilateral comparison of capacitances at 10 pF and 100 pF (BIPM.EM-K14.a and BIPM.EM-K14.b) which started with NIS, Egypt, at the end of 2015 and should be finished in February 2016.

### 1.3 Comparisons of electrical standards

#### 1.3.1 BIPM ongoing key comparisons in electricity (R. Chayramy, N. Fletcher, P. Gournay, B. Rolland, S. Solve, M. Stock)

The work programme in Electricity is focused on a comparison programme to validate national primary standards for fundamental electrical quantities: voltage, resistance and capacitance. To validate the high intrinsic accuracy of electrical quantum standards, the BIPM organizes on-site comparisons of Josephson voltage standards (BIPM.EM-K10) and of quantum Hall resistance standards (BIPM.EM-K12). For these comparisons, the BIPM quantum standards are transported to the laboratory of the participating NMI. BIPM staff and NMI staff then carry out a joint direct comparison of the BIPM and the NMI quantum standards.

The BIPM also organizes comparisons of voltage (BIPM.EM-K11), resistance (BIPM.EM-K13) and capacitance (BIPM.EM-K14) by sending secondary standards (Zener voltage standards, resistors, capacitors) to the participating NMI. In the case of voltage and resistance, the uncertainties in these comparisons are significantly higher than in the on-site comparisons which allow a direct comparison of the quantum standards.

On-site comparisons of Josephson voltage standards were carried out with the DMDM, Serbia, and the NIMT, Thailand. The report on the comparison with the PTB, Germany, in 2014 was published. As described in Section 1.1.1 a first comparison with the new programmable AC Josephson system was carried out with the NMIJ, Japan.

An on-site comparison of quantum Hall standards was organized with the VSL, the Netherlands. Unfortunately, the VSL system did not function properly during the comparison and no definitive result could be obtained.

Comparisons of Zener calibrations were organized for the NSAI NML, Ireland, and the JV, Norway. The reports for three comparisons undertaken during 2014 were published.

Bilateral comparisons of resistance measurements for 1  $\Omega$  and 10 k $\Omega$  were carried out with NSAI NML, Ireland, NIMT, Thailand, and CMI, Czech Republic. Reports are currently being written.

A bilateral comparison of capacitances at 10 pF and 100 pF has started with NIS, Egypt, at the end of 2015 and should be finished in February 2016.

#### 1.3.2 CCEM-K4.2016 (P. Gournay, N. Fletcher, M. Stock)

The key comparison CCEM-K4 of capacitance at 10 pF was last carried out during 1996 to 1999. In 2012 a decision was made by the Working Group on Low-Frequency Quantities (WGLF) of the Consultative Committee for Electricity and Magnetism (CCEM) to repeat this comparison and the general principles were discussed and adopted at its meeting in June 2015.

The new CCEM-K4 comparison will be organized as a large-scale star comparison. It will consist of a large number of bilateral comparisons between the participating NMIs and a reference institute during the same time frame. The BIPM has been designated by the CCEM as the reference institute and also the pilot for the comparison.

In this comparison scheme, all the participating institutes will be asked to send their own standards to the BIPM where they will be compared during the same time period. Before and after this measurement period at the BIPM, each institute should calibrate their own standards, again, preferably during the same time period. Two or three 10 pF standards capacitors could be sent to the BIPM and also an optional 100 pF standard in order to address the question of the scaling process from 10 pF.

For comparability, the capacitance measurements should be reported using the SI unit farad. This means that institutes realizing their traceability from a quantized Hall resistor should use the value of the von Klitzing constant ( $R_K$ ) from the latest CODATA fundamental constants adjustment and not its 1990 conventional value

$R_{K-90}$ . Capacitance measurements will be carried out under the usual ambient conditions ( $23\text{ }^{\circ}\text{C} \pm 1\text{ }^{\circ}\text{C}$  and 50 %  $\pm 10\text{ }%$  relative humidity) and preferably at 1592 Hz and 100  $V_{\text{rms}}$  (10  $V_{\text{rms}}$  for 100 pF capacitors).

Analysis of the results will be performed by the BIPM with the support of a group of experts from NMIs appointed by the CCEM. A statistical method that will allow, to address at best, the problems of drifts and step changes due to transportation of the standards will be developed and agreed before starting the comparison. The BIPM will fulfil the dual role of serving as reference laboratory (to compare all participants' standards against a set of reference capacitors) and of participating in the comparison with its own primary reference. The key comparison reference value (KCRV) will be derived by statistical techniques using all the participants' results and will not be taken as the BIPM result (contrary to the programme of bilateral comparisons BIPM.EM-K14).

The first draft of the technical protocol of the new CCEM-K4 comparison has been developed in 2015. It will be submitted for review and approval by the support group in early 2016 and the comparison is expected to start in the second half of 2016.

#### 1.4 Calibrations of electrical standards (R. Chayramy, N. Fletcher, P. Gournay, B. Rolland, S. Solve, M. Stock)

During the period from January to December 2015 the Electricity Department calibrated the following standards:

1  $\Omega$ , 100  $\Omega$  or 10 k $\Omega$  resistors were calibrated for: BEV (Austria), NMISA (South Africa), INMETRO (Brazil), MSL (New Zealand), SMD (Belgium), NMC (Singapore), BIM (Bulgaria), NIMT (Thailand), RSE KazInMetr (Kazakhstan). In total 43 certificates and two study notes were issued for nine NMIs.

1 pF, 10 pF and 100 pF capacitors were calibrated for: CEM (Spain), BEV (Austria), NIMT (Thailand), NMISA (South Africa), CMI (Czech Republic), SMD (Belgium), RSE KazInMetr (Kazakhstan), CENAM (Mexico) and INTI (Argentina). In total 33 certificates and two study notes were issued for nine NMIs.

Three Zener voltage standards were calibrated for UTE (Uruguay) and KIM-LIPI (Indonesia).

As every three years, the measurement services of the Electricity Department have been subjected to an external peer audit in 2015. As has been the case previously, services provided to the Member States of the BIPM are in compliance with the BIPM Quality Management System, based on the requirements of ISO/IEC 17025.

## 2. Mass Metrology

### 2.1 Measurement Services in Mass

#### 2.1.1 Calibrations

##### 2.1.1.1 *Extraordinary calibrations with respect to the IPK in anticipation of the redefinition of the kilogram (P. Barat, E. de Mirandés, M. Stock, M. Milton)*

In 2014 an extraordinary campaign of calibrations was initiated at the BIPM to provide improved traceability to the international prototype of the kilogram (IPK) for those NMIs that are carrying out experimental determinations of the Planck constant to support the forthcoming redefinition of the kilogram. During the first phase of the calibration campaign the IPK was used to recalibrate its six official copies and the BIPM working standards. This work was completed in 2014 and the results for the IPK and the official copies have been published [1]. In Phase two of the calibration campaign, mass standards of eight NMIs involved in measuring the Planck constant were calibrated against the BIPM working standards. The calibration certificates were sent to the NMIs in April 2015, providing them with improved traceability to the IPK, with an uncertainty of 3.5  $\mu\text{g}$ .

During Phase one, in 2014, it had been observed that the BIPM as-maintained mass unit, traceable to the IPK and which had been measured previously during the 3rd Periodic Verification, 1988-1992, had drifted away from the IPK by 35  $\mu\text{g}$ . It had also been observed that all working standards had lost mass since 1992, with losses ranging from 19  $\mu\text{g}$  to 88  $\mu\text{g}$ , which appeared to be correlated with the intensity of use. In the light of these experimental findings, the BIPM has conducted an analysis of all the available data from internal mass comparisons among the ten BIPM working standards since the 3rd Periodic Verification in order to understand the origin and evolution of this offset. A set of deterministic mathematical models for the mass evolution of the mass standards, capable of describing the experimental weighing data set has been developed. The model which describes the data best, suggests that a wear phenomenon, associated with one of the BIPM mass comparators, which was modified in-house, was responsible for a collective mass loss experienced by all the BIPM working standards, principally during the years 2004-2010. Since the IPK was not accessible between the 3<sup>rd</sup> Periodic Verification and 2014, a part of this collective mass loss went undetected, which explains the offset observed in 2014. This mass comparator was taken out of service in 2010. It is planned to re-activate the comparator to test the hypothesis that it was the origin of the mass losses.

As a consequence of the undetected mass changes, mass values attributed in mass calibrations since 2004 were generally overestimated, up to 35  $\mu\text{g}$  in 2014. The mathematical model allows determination of the evolution of this mass offset over time. This has allowed the BIPM to calculate corrected mass values which have been provided to its calibration customers. Revised calibration certificates for Pt-Ir standards were sent in March 2015 and for stainless steel standards in May 2015. Institutes involved in precision measurements of the Planck constant and the Avogadro constant had already been informed of the revised mass values at the end of 2014, so that they could provide updated measurement results for the CODATA 2014 fundamental constants adjustment.

As a result of these findings, a new strategy for the use of the working standards was developed, with the objectives of limiting the potential mass loss of the standards and to provide the possibility to detect such changes, if they were to occur. This new approach is described in the following section.

#### 2.1.1.2 *Calibration of 1 kg Pt-Ir prototypes and stainless steel standards (P. Barat, M. Stock)*

As described in section 2.1.1.1, during the calibration campaign using the IPK in 2014, it had been observed that the BIPM working standards had changed mass and that the full amount of mass change had not been detected. In order to ensure a stable BIPM mass unit in the future, two measures have been presented to the CCM during its meeting held in February 2015 (see Report of the Consultative Committee for Mass and Related Quantities, 15th meeting (2015), 2015, p.19): (1) that, henceforth the BIPM mass calibration service is no longer continuously available but restricted to two periods of the year and (2) that the working standards shall be used in a hierarchical approach, with different levels of usage.

Only two calibration periods are now open to the NMIs: March to May and September to November. At the beginning and the end of each period, the stability of the BIPM working standards will be checked. As a consequence, the calibration certificates are issued only after the end of each calibration period.

Furthermore, a new hierarchy has been established among the BIPM working standards. This new hierarchy is described in figure 1.

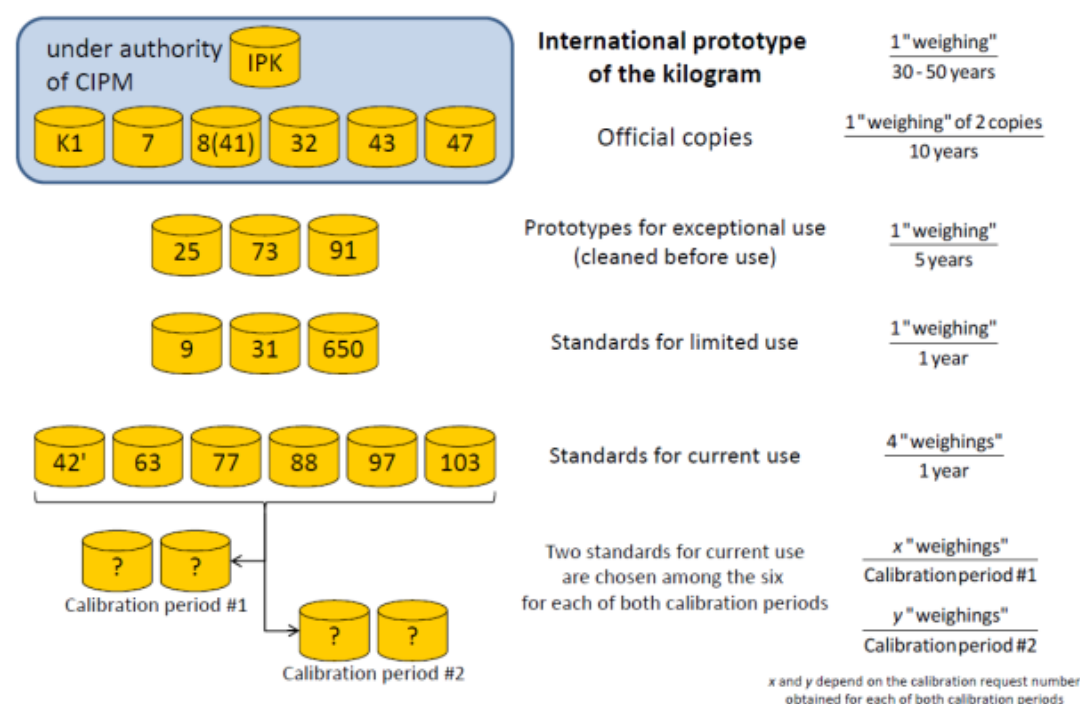


Figure 1. Hierarchy of BIPM prototypes and working standards

During 2015, 41 mass calibration certificates and one study note have been issued:

- Thirteen calibration certificates issued in the framework of the Extraordinary Calibrations using the IPK (see section 2.1.1.1).
- Sixteen certificates issued for the first calibration period from February to June 2015 (due to the Extraordinary Calibrations, the first calibration period for 2015 was extended from February to June) for the calibration of prototypes Nos. 35 (France), 44 (Australia), 46 (Indonesia), 56 (South Africa), 71 (Israel), 78 (Chinese Taipei), 89 (Switzerland), for 1 kg mass standard in platinum-iridium designated "A" (NPL, UK) and for 1 kg mass standards in stainless steel from the BIM (Bulgaria), the INPL (Israel), the KazInMetr (Kazakhstan), the LATU (Uruguay) and the VSL (the Netherlands).
- Ten certificates issued for the second calibration period from September to November 2015 for the calibration of prototypes Nos. 24 (Spain), 48 (Denmark), 64 (China), 76 (Italy), 80 (Thailand), for 1 kg mass standard in platinum-iridium designated "B" NPL (UK) and for 1 kg mass standards in stainless steel from the NIMT (Thailand), the NSAI NML (Ireland) and the VSL (the Netherlands).
- Two certificates and one study note issued for the calibration of new prototypes which have been provided to Member States: Prototypes Nos. 93 SASO (Saudi Arabia), 108 INM (Colombia) and 109 PTB (Germany).

### 2.1.1.3 Mass determinations of the AVO28 spheres (P. Barat, M. Stock)

In 2014 the BIPM contributed to the work of the Avogadro consortium by weighing the two isotopically pure Si-spheres AVO28-S5 and AVO28-S8. The result of the new determination of the Avogadro constant was published in March 2015 [2]. For the CODATA fundamental constants adjustment it is important to know by how much this new result is independent from the previous determination, published in 2011. Both

determinations have been made with the same spheres, however in-between they were etched and re-polished to eliminate metal contamination and to improve roundness.

To make both results usable for the least squares adjustment, the correlation between both results has been determined. Owing to the surface re-polishing and the upgrades of many of the measurement technologies, the correction is only 17 %. This analysis was published in July 2015 [3].

#### 2.1.1.4 *Volume calibrations of mass standards (D. Bautista)*

New software has been developed with a view to using silicon density standards in the hydrostatic apparatus. This should also simplify the use of the apparatus and allow the measurements to be carried out with minimum intervention. This work is now nearly finished and tests will follow in early 2016.

It has been observed that the hydrostatic balance leaves marks on the surface of the new prototypes under fabrication. This complicates the final polishing, as these marks need to be removed. Studies have been carried out to investigate the origin of this surface damage and to reduce it. To achieve this, the alignment of the weight exchangers and pans has been improved and a new system for the thermal treatment of the suspension wire has been designed.

The measurement capability for stacks of disks has been re-established, following measurements carried out on a stack for which the density was previously determined by the NPL. The difference between the results is consistent with the uncertainties, at the 95 % level of confidence, and proves the validity of our new measurements.

#### 2.1.1.5 *Pressure calibrations (F. Idrees)*

No internal pressure calibrations were carried out in 2015 because the pressure calibration facility was not operational. The piston-cylinder assembly of the pressure balance has been damaged and the manufacturer has been contacted to resolve the issue. The calibration campaigns have been postponed until the whole system is functional again.

#### 2.1.2 *CCM pilot study of primary realizations of the kilogram (M. Stock, P. Barat, S. Davidson (NPL, UK))*

One of the preparatory steps of the joint CCM and CCU Roadmap towards the redefinition of the kilogram is a "Pilot Study" of primary realizations of the kilogram. This Pilot Study shall compare realizations of the unit of mass at the level of 1 kilogram obtained with primary realization experiments (watt/joule balances, XRCD experiment), which are capable of determining mass at an uncertainty level of better than 2 parts in  $10^7$ , that is 200  $\mu\text{g}$  at the level of 1 kg. The BIPM will act as the pilot laboratory.

The two main objectives of the Pilot Study are (1) to test the uniformity of kilogram realizations based on different primary realization experiments and (2) to test the continuity between the kilogram realized according to the future definition and according to the present definition. The technical protocol has been designed so that primary realizations of the kilogram obtained by different primary realization experiments may be compared as directly as possible.

The Pilot Study will be carried out by using two sets of 1 kg travelling standards (provided by each participant) to be calibrated by the participants as follows:

Set 1: One Pt-Ir standard and optionally one standard of the participant's choice (e.g. Si-sphere, tungsten cylinder, second Pt-Ir standard), to be calibrated as directly as possible with the primary realization experiment (in general under vacuum);

Set 2: Two stainless steel standards, to be calibrated in air, traceable to the primary realization experiment.



Each participant shall calibrate its travelling standards assigning to each of them a mass and an uncertainty, based on an exact value of the Planck constant, stated in the comparison protocol. The BIPM will afterwards collect the travelling standards from all participants and carry out mass comparisons among all of them. Results from these comparisons will be compared with the mass and uncertainties claimed by the participants. This will assess the capability of future primary realizations to realize and disseminate a consistent unit of mass (uniformity) world-wide. The traceability of the BIPM working standards to the IPK, re-established in 2014 (see 2.1.1.1), will allow investigations of the agreement between kilogram realizations based on the future and the present definition of the kilogram (continuity). Following the measurements at the BIPM, the NMIs will recalibrate the standards to verify that their mass has not changed.

The measurements at the NMIs are planned for January - March 2016 and July - September 2016. The comparison measurements at the BIPM are foreseen for the period of April - June 2016. The Final Report is expected by March 2017.

### 2.1.3 Maintenance of the mass comparators and associated equipment (P. Barat, T. Lavergne)

The M<sub>one</sub> 6V-LL mass comparator is now equipped with an Artefact Transfer Device (ATD) developed by Mettler-Toledo AG. The latter allows the transfer of artefacts, stored in inert gas or under vacuum, into the comparator without contact with the ambient laboratory air by means of an Artefact Storage and Transport Vessel (ASTV).

Henceforth, with two mass comparators equipped with transfer systems for mass artefacts, CCL1007 from Sartorius (with the Vacuum Transfer System – VTS) and M<sub>one</sub> 6V-LL from Mettler Toledo (with the ATD), the BIPM is equipped with an important capability for the future work for the Ensemble of Reference Mass Standards.

A new device is being developed for the transfer of mass artefacts from the Automatic Loadable Container (ALC, Sartorius) to the Artefact Storage and Transport Vessel (ASTV, Mettler Toledo) and vice versa. This will allow determinations of the average mass of the Ensemble of Reference Mass Standards with both mass comparators, i.e. the CCL1007 and the M<sub>one</sub> 6V-LL.

### 2.2 Manufacturing 1 kg artefacts in Pt-Ir for NMIs (F. Boyer - BIPM Workshop)

The manufacture of three new 1 kg mass prototypes has been completed in 2015: one for Pakistan (the one previously allocated to Pakistan has been re-allocated to Saudi Arabia), one for Colombia and one for the PTB (Germany).

### 2.3 Ensemble of Reference Mass Standards (E. de Mirandés, F. Idrees, P. Barat)

In 2015 progress was made on the inert gas storage network of the ensemble of mass standards. The containers were already maintained under nitrogen and mass holders designed for each material have been inserted in the containers through the glovebox facility. This has led to a higher humidity level in the containers. In order to reach a lower level, all metallic parts of the containers were heated in the oven connected to the glovebox. A second heat treatment is planned at a higher temperature to reduce the humidity level further and to get it closer to the level of a blank measurement. The flow rates and the pressure levels inside the containers were adjusted to achieve a more uniform flow distribution inside each line. Software for monitoring the system and for detecting anomalies has been developed.

It has been observed that the vacuum level was too high when all the containers of the vacuum network were connected. To solve this problem a new series of containers has been designed which are under fabrication.

#### 2.4 Watt balance (H. Fang, F. Bielsa, A. Kiss, T. Lavergne, R. Chayramy, Y. Lu<sup>1</sup>, E. de Mirandés, B. Rolland, L. Robertsson, S. Solve, M. Stock)

A watt balance is being developed at the BIPM as a means for the practical realization of the expected new definition of the kilogram in terms of the Planck constant. A new apparatus has been developed with the objective of reducing in particular the type B measurement uncertainties related to misalignment. Its assembly is being finalized at the end of 2015. In addition to the use of several new elements and measurements facilities, the new apparatus allows the application of various alignment techniques already developed earlier, but not easily implemented inside the previous apparatus.

The radial magnetic field produced by the magnetic circuit has been aligned horizontally using two experimental procedures [4]. The upper cover segments of the magnet structure are presently not yet in place, thereby allowing relatively unrestricted access to the air gap. Both techniques make use of a 3-axis Hall probe mounted eccentrically on a high precision rotation stage. The axis of the rotation stage serves as a vertical reference while the magnetic probe is used to locally sense the orientation of the magnetic field by measuring its vertical component (which disappears for horizontal alignment). The first technique can be applied to the present open circuit configuration where the Hall probe can be rotated continuously around the gap. The magnetic plane at the central position inside the gap was aligned to better than  $10\ \mu\text{rad}$  and a planarity better than  $15\ \mu\text{rad}$  has been observed. The alignment uncertainty was within  $20\ \mu\text{rad}$ , which was mainly limited by the instability of the tiltmeter used for vertical alignment of the rotation axis. Constancy of the magnetic plane inclination was verified at the  $20\ \mu\text{rad}$  level over a length of 40 mm in the vertical direction. This is particularly important for the BIPM watt balance where the electromagnetic force is exerted on the coil while the coil moves vertically through the magnetic gap. The geometry of the magnetic circuit was also characterized which confirms the suitability of the magnetic circuit for use in the BIPM watt balance at the  $10^{-8}$  uncertainty level. Capacitive sensors were used to measure a decentring between the inner and outer poles of the yoke of  $14\ \mu\text{m}$  and a cylindricity of each pole surface better than  $1\ \mu\text{m}$ . A variation in the magnetic flux density of 0.6 mT in amplitude observed in the gap is well correlated with the variations of the measured gap width. For the centring of the coil, necessary for the watt balance operation, this variation in magnetic flux density corresponds to a coil decentring below 0.25 mm which is largely within the space available in the gap. A second alignment method, which had been developed in 2014, has been refined and can also be applied to the circuit in the future closed configuration, for which the first technique is not applicable. An uncertainty of the horizontality alignment of about  $25\ \mu\text{rad}$  was achieved using this technique. Regarding the technique developed for an open circuit, the dominant uncertainty source is the instability of the tiltmeter. The second most important source of uncertainty is the imperfect cancellation of the planar Hall effect of the magnetic probe at each location of the circuit. The two techniques yielded consistent results when applied to the present open configuration of the circuit. The accuracies of the two experimental procedures fulfil the requirements for the BIPM watt balance. They could easily be improved further by using a tiltmeter with higher precision, a Hall probe compensated for the planar Hall effect, and a stiffer mechanical support.

A new support structure was mounted to support the weighing cell and the whole suspension hanging from the weighing cell. The new structure has an open design allowing easy access to key components of the watt balance in order to facilitate their alignment. The suspension including the dynamic alignment mechanism and the inductive coil was assembled. The integration of a new mass loading device, mass exchanger and new interferometers is in progress. Electrical and optical connections have been totally remade. A lot of care has been taken to adequately shield the cables that supply high voltages and the measurement signals as well as to avoid ground loops. Special precautions have been taken with the induced voltage signal in order to minimize thermal electro-motive forces. Improvements have also been made to the current source to minimize current leakage and to improve its reliability. All the control and measurement units have been transferred to an adjacent laboratory. This laboratory also accommodates the new green YAG laser source for velocity measurement and the new red

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<sup>1</sup> Seconded from NIM, China until 30 January 2015.

diode laser source for position sensors. In total, twelve fibre-coupled optical feedthroughs have been installed to transport the laser beams into the watt balance apparatus enclosure located in the adjacent room. Servo-control of the vertical coil motion along the air gap is being optimized. New compact position sensors have been developed and implemented inside the apparatus to determine the linear and angular displacements of the coil along its travel. Work will start soon to align the electric plane of the moving coil and its centre with respect to the magnetic circuit.

The BIPM continued the development of two Josephson voltage standards (JVSs), one for the voltage and one for the current measurements. To improve the flexibility of the JVS used for the current measurement, a multi-channel bias source capable of independently biasing the 13 segments of the Josephson junction array is being developed. The bias source is designed to supply a current adjustable between  $-30$  mA and  $+30$  mA for each segment. Special precautions have been taken to minimize the electrical noise and to avoid ground loops. Fabrication of a prototype source is under way and it is expected to be finalized by the end of 2015. Its operation will be tested on the Josephson array before fabrication of the whole multi-channel source.

The JVS dedicated to the induced voltage measurement was tested on its own. A voltage ramp from  $0.6$  V to  $0.7$  V was realized with a resolution corresponding to one Josephson junction ( $38$   $\mu$ V). This corresponds to the succession of 2615 combinations of the biasing configuration of the 13 subarrays. After each voltage step, the polarity was reversed to extract the linear evolution of the thermal EMFs. This test is very demanding on the equipment and therefore allows identification of weaknesses in the measurement setup.

The original design of the bias source, powered from a set of batteries, needed to be changed. The original bias source, tested on an array of resistances was successful, but behaved unpredictably when used with the array of Josephson junctions. We suspect that the problems encountered are related to both the structure of the impedance presented by the array and the galvanic isolation of the source. The generation of the voltage ramp using linear power supplies instead of the batteries was challenging but successful. The experimental results were as expected but exhibited a high sensitivity to magnetic flux trapping.

Development of the new interferometer is quite advanced. The frequency stabilization of the  $532$  nm laser source was further improved by refining the servo-control loop to compensate frequency drift in time. The frequency stability was characterized by measuring a beat note against a second similar laser system used for the calculable capacitor. The short-term stability was a few parts in  $10^{12}$  for an integration time of several seconds which is mainly limited by the frequency modulation applied to the laser for the servo-control. Laser mode-leakage was also evaluated. No extra longitudinal modes were observed above the detection noise which was  $55$  dB below the laser mode. This should lead to an error of less than  $10$  pm on position measurement. The interferometers were aligned and characterized on a test bench. The output signals exhibit a signal-to-noise ratio of about  $70$  dB in a  $1$  kHz bandwidth, and a rejection of the cross-talk at the level of  $60$  dB, which should lead to very low phase noise and extremely low non-linear error. The whole experimental set-up has been transferred to the watt balance laboratories, where alignment of the interferometers inside the watt balance apparatus will start soon.

### 3. Activities related to the work of Consultative Committees

M. Stock is the Executive Secretary of the Consultative Committee for Electricity and Magnetism (CCEM) and a member of several of its working groups.

N. Fletcher is a member of the CCEM working group on proposed modification to the SI (WGSI). The CCEM and its working groups met in March 2015 at the BIPM.

M. Stock is the Executive Secretary of the Consultative Committee for Photometry and Radiometry (CCPR) and a member of its working groups. The working groups on key comparisons (WG-KC) and on strategic planning (WG-SP) met in October 2015 at the NIM, in Beijing. A workshop on comparison analysis was held at the same time. The new *mise en pratique* for the definition of the candela and associated derived units for photometric and

radiometric quantities has been published on the BIPM web site in July 2015. On 1 January 2016, J. Viallon from the BIPM Chemistry Department will become the new Executive Secretary of the CCPR.

H. Fang has been the Executive Secretary of the Consultative Committee for Mass and Related Quantities (CCM) since 1 January 2015. She is a member of several working groups (WGs) and task groups (TGs) of the CCM. The CCM and its working groups met at the BIPM in February 2015.

E. de Mirandés is Executive Secretary of the Consultative Committee for Units (CCU) which will hold its next meeting in June 2016.

#### 4. Activities related to external organizations

M. Stock is a member of the Executive Committee of the Conference on Precision Electromagnetic Measurements (CPEM). H. Fang, N. Fletcher, S. Solve and M. Stock are members of the Technical Committee of the CPEM 2016.

M. Stock is the contact person for the BIPM liaison with the International Commission on Illumination (CIE).

P. Gournay represented the BIPM on the Organizing Committee and the Scientific Committee of the International Congress of Metrology, CIM 2015, held in Paris.

The BIPM is an external collaborator on the EMRP projects “Quantum Resistance based on Graphene (GraphOhm)” and “Automated Impedance Metrology extending the Quantum Toolbox for Electricity (AIMQuTE)”. The BIPM is also an external collaborator of the project ACQ-PRO, part of Joint Research Project to the European Metrology Programme for Innovation and Research (EMPIR), for the years 2015 to 2019.

M. Stock acted as the BIPM liaison with the International Avogadro Coordination project, the EURAMET Technical Committee for Mass and Related Quantities (TC-M), the Technical Committee for Photometry and Radiometry (TC-PR) and the Technical Committee for Electricity and Magnetism (TC-EM), and for the European Metrology Research Programme (EMRP) joint research projects SIB-05 (NewKILO) and SIB-03 (kNOW).

E. de Mirandés is a member of the CODATA Task Group on Fundamental Constants.

#### 5. Publications

1. Stock M., Barat P., Davis R.S., Picard A., Milton M.J.T., Calibration campaign against the international prototype of the kilogram in anticipation of the redefinition of the kilogram part I: comparison of the international prototype with its official copies, *Metrologia*, 2015, **52**(2), 310-316.
2. Azuma Y., Barat P., Bartl G., Bettin H., Borys M., Busch I., Cibik L., D'Agostino G., Fujii K., Fujimoto H., Hioki A., Krumrey M., Kuetsgens U., Kuramoto N., Mana G., Massa E., Meeß R., Mizushima S., Narukawa T., Nicolaus A., Pramann A., Rabb S.A., Rienitz O., Sasso C., Stock M., Vocke Jr R.D., Waseda A., Wundrack S., Zakel S., Improved measurement results for the Avogadro constant using a  $^{28}\text{Si}$ -enriched crystal, *Metrologia*, 2015, **52**(2), 360-375.
3. G. Mana, E. Massa, C.P. Sasso, M. Stock, K. Fujii, N. Kuramoto, S. Mizushima, T. Narukawa, M. Borys, I. Busch, A. Nicolaus, A. Pramann, The Correlation of the  $N_A$  Measurements by Counting  $^{28}\text{Si}$  Atoms, *J. Phys. Chem. Ref. Data*, 2015, **44**(3), 031209-1 – 031209-8.
4. F. Bielsa, Y. F. Lu, T. Lavergne, A. Kiss, H. Fang, M. Stock, Alignment of the magnetic circuit of the BIPM watt balance, *Metrologia*, 2015, **52**(6), 775-782.

5. Davis R.S., What is a kilogram in the Revised International System of Units (SI)?, *J. Chem. Ed.* 2015, **92**(10), 1604-1609.
6. Nielsen L., Davis R.S., Barat P., Improving traceability to the international prototype of the kilogram, *Metrologia*, 2015, **52**(4), 538-551.
7. N. Fletcher, R.S. Davis, M. Stock, M. Milton, Modernizing the SI – implications of recent progress with the fundamental constants, *arXiv*:1510.08324v1.
8. Fletcher N., Götz M., Rolland B., Pesel E., Behavior of 1  $\Omega$  resistors at frequencies below 1 Hz and the problem of assigning a dc value, *Metrologia*, 2015, **52**(4), 509-513.
9. Gournay P., Fletcher N., Robertsson L., Stock M., Progress on the Thompson-Lampard calculable capacitor project at BIPM, CIM 2015, DOI: 10.1051/metrology/20150012001.

### Comparison Reports

10. Power O., Moran A., Fletcher N., Goebel R., Stock M., Bilateral comparison of 10 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.a) between the NSAI-NML, Ireland, and the BIPM, March-August 2011, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01017.
11. Power O., Moran A., Fletcher N., Goebel R., Stock M., Bilateral comparison of 100 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.b) between the NSAI-NML, Ireland, and the BIPM, March-August 2011, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01018.
12. Saleem M., Ansari M.A., Saxena A.K., Fletcher N., Goebel R., Stock M., Bilateral comparison of 10 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.a) between the NPLI and the BIPM, July 2010-May 2011, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01015.
13. Saleem M., Ansari M.A., Saxena A.K., Fletcher N., Goebel R., Stock M., Bilateral comparison of 100 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.b) between the NPLI and the BIPM, July 2010-May 2011, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01016.
14. Sapunova I., Fletcher N., Goebel R., Stock M., Bilateral comparison of 10 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.a) between the BIM, Bulgaria, and the BIPM, April 2012 to September 2012, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01012.
15. Sapunova I., Fletcher N., Goebel R., Stock M., Bilateral comparison of 100 pF capacitance standards (ongoing BIPM key comparison BIPM.EM-K14.b) between the BIM, Bulgaria, and the BIPM, April 2012 to September 2012, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01013.
16. Solve S., Chayramy R., Stock M., Mageed H.M.A., Aladdin O.M., Raouf M.H.A., Bilateral comparison of 1 V and 10 V standards between the NIS (Egypt) and the BIPM, August to September 2014 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01011.
17. Solve S., Chayramy R., Stock M., Palafox L., Behr R., Comparison of the Josephson Voltage Standards of the PTB and the BIPM (part of the ongoing BIPM key comparison BIPM.EM-K10.b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01025.
18. Solve S., Chayramy R., Stock M., Pantelic-Babic J., Sofranac Z., Zivkovic V., Bilateral comparison of 1 V and 10 V standards between the DMDM (Serbia) and the BIPM, January to March 2014 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01006.
19. Solve S., Chayramy R., Stock M., Pimsut S., Bilateral Comparison of 1 V and 10 V Standards between the NIMT (Thailand) and the BIPM, October to December 2014 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01019.

20. Solve S., Chayramy R., Stock M., Power O., Bilateral Comparison of 10 V Standards between the NSAI - NML (Ireland) and the BIPM, March 2015 (part of the ongoing BIPM key comparison BIPM.EM-K11.b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01021.
21. Solve S., Chayramy R., Stock M., Sengebusch F., Bilateral Comparison of 1 V and 10 V Standards between the JV (Norway) and the BIPM, January to February 2015 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01022.
22. Solve S., Chayramy R., Stock M., Simionescu M., Cîrneanu L., Comparison of the Josephson voltage standards of the INM and the BIPM (part of the ongoing BIPM key comparison BIPM.EM-K10.b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01009.
23. Solve S., Chayramy R., Stock M., Vlad D., Bilateral Comparison of 1 V and 10 V Standards between the SMD (Belgium) and the BIPM, October to December 2014 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 01020.

## 6. Travel in 2015 (conferences, lectures and presentations, visits)

M. Stock to:

- NPL, Teddington (UK), 16 January, to discuss possible collaborations in the field of mass metrology.
- Sarajevo (Bosnia-Herzegovina), 15-17 April, to attend the EURAMET TC-M meeting and the EMRP NewKILO meeting.
- PTB, Braunschweig (Germany), 28-29 May, to visit the mass laboratories.
- NIM, Beijing (China), 26-28 October, to attend CCPR working group meetings and the CCPR workshop on comparison analysis.

P. Gournay and N. Fletcher to:

- LNE, Paris, 4-5 March, for EURAMET workshop on capacitance traceability and bridges, presentations on the BIPM quadrature chain and calculable capacitor.

P. Gournay to:

- International Congress on Metrology, CIM 2015, Paris, 24 September, to give a presentation on the calculable capacitor.

S. Solve to:

- Ecole Supérieure de Métrologie, Douai (France), 24 April, to give a talk on “voltage metrology within the BIPM mission”.
- SP, Borås (Sweden), 19-20 May, to attend the EURAMET Electrical Power and Energy Expert meeting.
- METAS, Bern (Switzerland), 27-29 May, to attend the EURAMET Expert meeting DC & Quantum Metrology.
- La Défense, Paris (France) to attend three LabVIEW<sup>®</sup> software courses, May and September.

S. Solve and R. Chayramy to:

- DMDM, Belgrade (Serbia), 17-25 June, to carry out an on-site BIPM Josephson voltage standard comparison.

- NIMT, Klong Luang, Pathumthani (Thailand), 3-12 November, to carry out an on-site BIPM Josephson voltage standard comparison.
- NMIJ, Tsukuba (Japan), 2-12 December, to perform the first direct comparison between the new BIPM Programmable Voltage Standard and the NMIJ Programmable Voltage Standard.

N. Fletcher, P. Gournay, B. Rolland to:

- VSL (the Netherlands), 2-9 October, to carry out an on-site comparison of quantum Hall resistance standards.

F. Bielsa, H. Fang, A. Kiss and T. Lavergne to:

- METAS, Bern (Switzerland), 8 July, to visit the watt balance and for technical discussions.

P. Barat, F. Idrees and E. de Mirandés to:

- PTB, Braunschweig and Berlin (Germany), 4-6 May, to visit the mass laboratories.

P. Barat to:

- Académie des Sciences de Paris, Paris (France), 7 July, to deliver prototype No. 34 which was calibrated during the Extraordinary Calibrations using the IPK.

## 7. Visitors in 2015

Electricity laboratories

- Dr Nabil Ben Hsouna and Dr Ghislain Boloma, students at ESM (France), to visit the voltage metrology laboratories, 11 February.
- Dr. Yang Yan and Dr Lu Huang (NIM), to visit the electricity laboratories, 6 March.
- Dr Ralf Behr and Dr Luis Palafox (PTB) to discuss the comparison report on the Josephson voltage standard comparison with PTB in October 2014, 9 March.
- Dr Ilya Budovsky (NMIA) and Dr Jon Pratt (NIST), to visit the voltage metrology laboratories, 9 March.
- Dr Yi-hua Tang (NIST), to visit the voltage metrology laboratories and to discuss future collaboration work, 10 March.
- H.E. Mr Nabil A. Molla, Omar S. Kanakrieh, GCC Standardisation Organization (GSO), Saudi Arabia, Mohamed Ahmed Al Mulla, GULFMET President, United Arab Emirates, to visit the calculable capacitor and the watt balance, 19 March.
- Amer Ali, Graphensic AB, Sweden, for discussions on graphene samples, 9 June.
- Dr Alexandre Satrapinski, MIKES/VTT, Finland, for investigations on low-frequency current comparators, 22-26 June.
- Dr Martin Götz, PTB, Germany, for investigations on low-frequency current comparators, 23 June.
- Group of 30 visitors from KASTO (Korean Association of Standards and Testing Organization), Republic of Korea, to visit the electricity and mass laboratories, 29 June.
- Dr Barry Wood (NRC), for an external audit of the electricity measurement services, 2-3 September.
- M. Mohamad Syahadi (KIM-LIPI) to bring a Zener voltage standard for calibration and visit the voltage laboratories, 7 September.

- Group of five scientists (KIM-LIPI), for a laboratory tour, including the electricity laboratories, 9 September.
- Dr Samuel Benz (NIST) to visit the voltage metrology laboratories and discuss future collaboration work, 10-11 September.
- Dr Chiharu Urano (NMIJ) to visit the voltage metrology laboratories and discuss the technical details of the comparison of programmable Josephson voltage standards at NMIJ, 14 September.
- Dr Sophie Djorjevic (LNE) to discuss issues on the operation of programmable arrays of Josephson junctions, 23 September.
- Khosro Madanipour (President NMCI), Ahad Mohammadi Livari (Deputy), Omidali Masoudi (General Director Public and international relations), NMCI(ISIRI), Iran, for a laboratory tour including the mass and electricity laboratories, 23 October.

#### Mass laboratories including the watt balance

- Dr Stephan Schlamminger (NIST), to deliver the watt balance made from LEGO bricks, 24 February.
- Ms Sheila Preste (LATU), to visit the watt balance, 25 February.
- Dr Ilya Budovsky (NMIA), to visit the watt balance, 9 March.
- Dr Dae-im Kang and Won-gyu Lee (KRISS), to visit the watt balance, 12 March.
- P. Pinot, P. Otal and F. Beaudoux (LNE/Cnam), to visit the Mass laboratories and to collect prototype No. 35, 18 March.
- H.E. Mr Nabil A. Molla, Omar S. Kanakrieh, GCC Standardisation Organization (GSO), Saudi Arabia, Mohamed Ahmed Al Mulla, GULFMET President, United Arab Emirates, to visit the calculable capacitor and the watt balance, 19 March.
- L. Cagliioni and M. Kliebenschädel, Mettler-Toledo, Greifensee (Switzerland), to install the Artefact Transfer Device, 20-22 April.
- Mr Fernando Ferrer Margaleff and Mr Angel Robles Carbonell (CEM), to discuss the outcome of the Extraordinary Calibrations and to visit the watt balance, 23 April.
- Dr Sergio Zagier and Ms Claudia Urruty (Zagier&Urruty publications, Argentina) to visit the watt balance, 24 April.
- Mr Leon Chan (NIST), to visit the watt balance, 17 June.
- J. Pereira, Mettler-Toledo, Viroflay (France), to visit the mass laboratories, 24 June.
- Group of 30 visitors from KASTO (Korean Association of Standards and Testing Organization), Republic of Korea, to visit the electricity and mass laboratories, 29 June.
- Dr Wynand Louw (NMISA), to visit the watt balance 15 July.
- Mr Seog Hwan Hong and Dr Jookeun Park (KRISS), to visit the watt balance, 20 July.
- Group of NMI Public Relations experts to visit the watt balance, 21 July.
- Mr Ilya Victorov (INPL), to visit the watt balance, 24 July.
- S. Cantelou, Mettler-Toledo, Viroflay (France), to visit the mass laboratories, 29 July.
- Working Group CCTF-WGTWSTFT, to visit the watt balance, 8 September.



- Group of five scientists (KIM-LIPI), for a laboratory tour including the fabrication of mass standards, 9 September.
- Dr Ricardo José De Carvalho and Msc Hamilce Simas Iozzi Coda Santos (ON/DSHO), to visit the watt balance, 14 September.
- Dr Chiharu Urano (NMIJ), to visit the watt balance, 17 September.
- Ms Changqing Cai and Dr Jiang Wang (NIM), to visit the watt balance, 22 September.
- R. Farley, S. Wettstein, MBW, Wettingen (Switzerland) and E. Georjin, CETIAT, Lyon (France), to visit the mass laboratories, 23 September.
- Dr Paul Gale (Vatican Observatory), to visit the watt balance, 5 October.
- Khosro Madanipour (President NMCI), Ahad Mohammadi Livari (Deputy), Omidali Masoudi (General Director Public and international relations), NMCI(ISIRI), Iran, for a laboratory tour including the mass and electricity laboratories, 23 October.
- Mr Thomas Mautjana (NMISA), to visit the watt balance, 13 November.
- R. Sukhon (NIMT), to have a training session on mass metrology and to collect prototype No. 80 and one 1 kg mass standard in stainless steel, 1-2 December.
- Mr Sylvain Lucas and Mr Yann Moysan (SHOM), to carry out gravimetric measurements in the watt balance laboratory, 1-2 December.
- Dr Eran Tal, Univ. of Cambridge, to discuss the philosophical aspects of the redefinition of the kilogram, 7 December.
- Mr Alfredo Esparza (CENAM) and Mr Jorge Arturo Arzate Flores (National Autonomous University of Mexico, UNAM), to visit the watt balance, 14 December.
- 38 visitors to deliver and to collect mass standards, at the BIPM for calibration.

## BIPM Time Department

Director: E.F. Arias

(1 January 2015 to 31 December 2015)

### 1. International Atomic Time (TAI), Coordinated Universal Time (UTC) and Rapid UTC (UTCr) (E.F. Arias, A. Harmegnies, Z. Jiang, H. Konaté, G. Panfilo, G. Petit and L. Tisserand)

The reference time scales, International Atomic Time (TAI) and Coordinated Universal Time (UTC), are computed from data reported regularly to the BIPM by the various timing centres that maintain a local UTC; monthly results are published in *Circular T*. The UTC rapid solution (UTCr) is published every Wednesday at 18 h UTC at the latest. All information related to the publication of UTC and UTCr can be accessed at [www.bipm.org/en/scientific/tai/ftp\\_server/introduction.html](http://www.bipm.org/en/scientific/tai/ftp_server/introduction.html).

The *BIPM Annual Report on Time Activities for 2014*, volume 9, provides the definitive results for 2014 and is available on the BIPM website at [www.bipm.org/en/bipm/tai/annual-report.html](http://www.bipm.org/en/bipm/tai/annual-report.html).

### 2. Algorithms for time scales (G. Panfilo, G. Petit, A. Harmegnies, L. Tisserand and F. Parisi<sup>2</sup>)

The algorithm used to calculate the time scales by the Time Department is an iterative process that starts by producing a free atomic scale (*Échelle atomique libre*, EAL) from which TAI and UTC are derived. Research into time-scale algorithms is ongoing in the department, with the aim of improving the long-term stability of EAL and the accuracy of TAI.

After the implementation of the new weighting algorithm, based on the concept of clock frequency predictability, the behaviour of UTC is routinely and carefully monitored to trap and fix unexpected anomalies although none were observed throughout the year. An improvement in the short- and long-term stability of EAL is already visible after the application of the new weighting algorithm.

Within the framework of a six month placement with a student from the University of Torino (Italy), which began in 2014, the use of the Kalman Filter (a very powerful statistical tool) has been tested to build an independent time scale. The results are very promising and encouraging for its continued investigation and development for its application in UTC.

The revision of the algorithm for the calculation of the uncertainties reported in Section 1 of *Circular T* is in progress. The current algorithm underestimates the uncertainty values for the pivot laboratory (at present PTB) because correlations have not been fully considered. The result of the revision study was presented at the 20th meeting of the CCTF, and the implementation of the new algorithm, which will provide the correct uncertainty estimations, is expected within the next 2 years.

#### 2.1 EAL stability

Some 87 % of the clocks used in the calculation of UTC are either commercial atomic clocks with high performance caesium tubes or active hydrogen masers. The number of hydrogen masers operated at the participating laboratories has increased by 24 % in the last two years, without any significant increase in the number of caesium standards. The weighting procedure involved in the time scale computation guarantees the long-term stability of EAL. To prevent domination of the scale by a small number of very stable clocks, a

<sup>2</sup> Department of Mathematics, University of Torino, Italy, on a six-month secondment from 1 November 2014

maximum relative weight is used each month which depends on the number of participating clocks. On average, about 11 % of the participating clocks were at the maximum weight during 2015; almost all of these were hydrogen masers. The new weighting algorithm, based on the predictability of the clock's frequency, enhanced the influence of the hydrogen masers on the resulting time scale; 40 % of the contributing hydrogen masers were, on average, at the maximum weight in 2015, whilst only 0.1 % of the caesium clocks reached the maximum weight.

UTC implicitly relies on the hydrogen masers in the short term and on caesium clocks in the long term, which was an aim of the new weighting procedure. The stability of EAL at the end of 2015, expressed in terms of an Allan deviation, is about three parts in  $10^{16}$  for averaging times of one month.

## 2.2 TAI accuracy

To characterize the accuracy of TAI, estimates are made of the relative departure, and its uncertainty, of the duration of the TAI scale interval from the SI second, as produced on the rotating geoid, by primary and secondary frequency standards. Since January 2015, individual measurements of the TAI frequency have been provided by thirteen primary frequency standards, including eleven caesium fountains (SYRTE FO1, SYRTE FO2, NIST F1, NIST F2, IT CSF2, SU CSFO2, NPL CSF2, PTB CSF1, PTB CSF2, NPLI CSF1 and NIM 5), and by a rubidium secondary frequency standard (SYRTE FORb). Reports of the operation of the primary and secondary frequency standards are regularly published on the BIPM website and collated in the [BIPM Annual Report on Time Activities](#).

Since January 2015, the global treatment of individual measurements has led to a relative departure of the duration of the TAI scale unit from the SI second on the geoid ranging from  $+0.83 \times 10^{-15}$  to  $-0.44 \times 10^{-15}$ , with a maximum standard uncertainty of  $0.39 \times 10^{-15}$ . No steering correction has been applied since October 2012, confirming that the new algorithm maintains a positive impact on the accuracy of TAI.

## 2.3 Independent atomic time scales: TT(BIPM)

TAI is computed in 'real-time' and is subject to operational constraints; as a result it does not provide an optimal realization of TT, the time coordinate of the geocentric reference system. The BIPM therefore computes an additional realization, TT(BIPM), in post-processing, which is based on a weighted average of the evaluation of the TAI frequency by the primary frequency standards. The Time Department provided an updated computation of TT(BIPM) in January 2015, known as TT(BIPM14), valid until December 2014, which had an estimated accuracy of about 2-3 parts in  $10^{16}$  over recent years. Moreover, the Time Department provides a formula to extend TT(BIPM14) based on the most recent TAI computation. Such an extension is useful for pulsar analysis pending the yearly updates of TT(BIPM). Studies to improve the computation of TT(BIPM) are ongoing, in order to keep it in line with improvements in the primary and secondary frequency standards.

## 2.4 Local representations of UTC in national laboratories as broadcast by the GNSS

The Time Department continues to calculate and publish the differences between the predictions of UTC(USNO) and UTC(SU) (as broadcast by GPS and GLONASS) and UTC in BIPM *Circular T*. As a consequence of the alert made by the BIPM on the offset of GLONASS time and the broadcast prediction of UTC(SU) with respect to UTC, work has been developed with the VNIIFTRI, Russian Federation, and the GLONASS authorities on the absolute calibration of a BIPM receiver.

### 3. **Primary frequency standards and secondary representations of the second** (E.F. Arias, G Panfilo, G. Petit and L. Robertsson)

Members of the BIPM Time Department actively participate in the work of the CCL-CCTF Frequency Standards Working Group (WGFS), and the Consultative Committee for Time and Frequency (CCTF) Working Group on Primary and Secondary Frequency Standards (WGPSFS). These Working Groups seek to encourage comparisons, knowledge-sharing between laboratories, the creation of better documentation, the use of high-accuracy primary frequency standards (Cs fountains) and secondary frequency standards for TAI.

The WGFS maintains a list of recommended values of standard frequencies for applications including secondary representations of the second. Updates of frequency values and their respective uncertainties for secondary representations of the second in the list have been recommended by the CCTF in September 2015, and have been adopted by the CIPM in Recommendation 2 (CI-2015).

#### *Secondary representations of the second reported in BIPM Circular T*

Since January 2012 the LNE-SYRTE has reported frequency measurements of the Rb microwave transition obtained with a double Cs-Rb fountain (FORb). Twelve measurement reports of FORb were submitted in 2015 and have been officially used for the accuracy of TAI.

### 4. **Time links used for UTC** (E.F. Arias, A. Harnegnies, Z. Jiang, H. Konaté, G. Panfilo, G. Petit, L. Tisserand, and W. Wenjun<sup>3</sup>)

At the end of 2015, 74 time laboratories supplied data for the calculation of UTC at the BIPM. The laboratories are equipped with GNSS receivers and some of them also operate two-way satellite time and frequency transfer (TWSTFT) stations.

Data from three independent techniques are included in the process of comparison of laboratories' clocks based on tracking GPS and GLONASS satellites, and TWSTFT.

The GPS all-in-view method is widely used and takes advantage of the increasing quality of the International GNSS Service (IGS) products (clocks and IGS time). Clock comparisons for UTC are implemented using C/A code measurements from GPS single-frequency receivers, or dual-frequency, multi-channel GPS geodetic-type receivers (P3). The GPS phase and code data provided by time laboratories which operate geodetic-type receivers is processed each month using the Precise Point Positioning (PPP) technique. The Time Department also regularly computes combined GPS/GLONASS links resulting in improved link uncertainty. About five GPS/GLONASS links are regularly computed for *Circular T*.

Nine laboratories operate TWSTFT stations and officially submit data for use in the computation of UTC, representing 8 % of the time links. No TWSTFT contributions from the laboratories in the Asia-Pacific region were possible in 2015 due to an interruption of the satellite service. The combination of TWSTFT and PPP (so called TWPPP) has been used whenever possible. This combination takes advantage of the small noise of the GPSPPP and of the accuracy of the TWSTFT links.

GPS PPP alone or in combination with TWSTFT are in use for UTC clock comparisons in almost 60 % of the links, where the statistical uncertainty of time transfer is well below the nanosecond, the best value is 0.3 ns for 51 % of the time links.

<sup>3</sup> Chinese Academy of Sciences, NTSC (Xi'an, China), on a one-year secondment starting 3 June 2014

#### 4.1 Global Positioning System (GPS) and Global Navigation Satellite System (GLONASS)

All GNSS time and frequency transfer data are corrected for satellite positions using IGS and the Information and Analysis Centre of Navigation (IAC) of the Mission Control Centre in Russia. The measurement data obtained by using single-frequency receivers are corrected for ionospheric delays using maps of the total electron content of the ionosphere provided by the Centre for Orbit Determination in Europe (CODE).

Techniques that use dual-frequency, GNSS carrier-phase measurements in addition to the codes, are widely used by the geodetic community and have been adapted to the needs of time and frequency transfer. This topic is studied within the framework of the IGS Working Group on Clock Products, which has a physicist from the Time Department as a member.

Data from world-wide geodetic-type receivers are collected for UTC computation, using procedures and software developed in collaboration with the Observatoire Royal de Belgique (ORB). These P3 time links are routinely computed and compared to other available techniques, notably two-way time transfer. The software producing iono-free has been implemented in some receivers, and these now automatically produce both formatted GPS and GLONASS P3 code results. These newly available data will be used in multi-GNSS system time links, but further studies on GLONASS inter-frequency biases have to be carried out first.

The NRCan's PPP software is used for the time link calculation. The current version of the software is capable of processing both GPS and GLONASS data but only GPS results are used operationally. Comparisons with other PPP software have been carried out. Studies are continuing to improve long-term stability, using new processing techniques, in collaboration with software developers at NRCan, the ORB, the *Centre National d'Études Spatiales* (CNES) and also with other institutes. A novel PPP technique using integer phase ambiguities (IPPP) has been successfully developed within the framework of a post-doctoral project. It significantly improves the stability in the medium term (several hours) and mostly in the long term (days). In 2015 the IPPP technique moved to a pre-operational stage and it is used regularly to compare IPPP results to the few available optical fibre links.

Comparisons of the different possible links on a baseline linking two contributing laboratories are computed and published monthly on the Time Department's ftp server.

#### 4.2 Two-way satellite time and frequency transfer

One meeting of the TWSTFT participating stations was held during 2015 at the IFCS/EFTF meeting in Denver, USA, on 12-16 April. The 23rd annual meeting of the CCTF WG on TWSTFT was held at the BIPM on 7-8 September 2015. The outcomes of these meetings that impact the Time Department's activities are: the approval and implementation of TWSTFT Calibration Guidelines; and the organization of calibration trips between TW stations, where the BIPM is charged with the validation of the reports and introduction of the calibration parameters in the calculation of UTC. The BIPM is also involved in the calibration of two-way time-transfer links by comparison with the corresponding GPS links. This is necessary to maintain stability of the TWSTFT links, in case of a loss of their direct calibration.

The TWSTFT technique is currently operational in eleven European, two North American and nine Asia-Pacific time laboratories. Eight TWSTFT links had been used in the computation of UTC in 2015; they are combined with GPS PPP solutions. Due to the interruption of the satellite service, no TW data contribution from institutes in the Asia-Pacific region had been possible during the year. Some of the TWSTFT links involved in the computation of UTC are used in the experiment 'Time Transfer by Laser Link' (T2L2). The BIPM aims to develop studies on this technique, which could be used to validate less accurate time links and their calibrations.

Campaigns with a travelling calibration station were organized and funded by the participating laboratories in 2015. The parameters obtained have been implemented for UTC computation following validation of the results by the Time Department.

Results of the time links and link comparison using GNSS single-frequency, dual-frequency and TW observations are published monthly on the Time Department's ftp server (<ftp://tai.bipm.org/TimeLink/LkC>).

#### 4.3 Calibration of delays of time-transfer equipment and time links

The characterization of the delays (so-called "calibration") of time transfer equipment in the contributing laboratories is necessary to improve the uncertainty of  $[UTC-UTC(k)]$  and for the accuracy of UTC dissemination.

Following a recommendation by the CCTF, the Time Department has issued the *BIPM Guidelines for GNSS calibration*. This document is intended for Regional Metrology Organizations (RMOs) and establishes a permanent cooperation for sharing the organization of campaigns to determine the relative delays of time transfer equipment and links in UTC contributing laboratories. The *Guidelines* are under continuous improvement, and this has led to a revised edition of the *Guidelines* being produced in September 2015.

In 2015 the BIPM concluded the first calibration campaign to the "Group 1" laboratories in APMP, EURAMET, SIM and COOMET, and expects that regional calibration trips to "Group 2" laboratories will be implemented in 2016 by the RMOs in accordance with the *BIPM Guidelines*. By repeatedly applying this new procedure time transfer accuracy is expected to improve by a factor of 2.

The BIPM Time Department is not directly involved in specific TWSTFT calibration trips, but is responsible for the validation of the calibration reports and implementation of the results in the calculation of UTC. It also provides support whenever necessary to maintain a TW calibration by alignment with a calibrated GPS link (see section 4.2).

Results of the differential calibration exercises are made available on a dedicated web page ([www.bipm.org/jsp/en/TimeCalibrations.jsp](http://www.bipm.org/jsp/en/TimeCalibrations.jsp)), where past calibration results are also provided.

The Guidelines for TWSTFT link calibration, elaborated by the CCTF WG on TWSTFT, were approved at the 20th meeting of the CCTF in September 2015.

#### 4.4 Advanced time and frequency transfer

Data from two fibre links between UTC contributing laboratories in Europe are regularly submitted and compared with the corresponding links by GNSS time transfer techniques. The aim of the Time Department is to include the fibre links in the computation of UTC in the future, and in this direction the CCTF WG on Coordination of the Development of Advanced Time and Frequency Transfer Techniques (WGATFT) has established a study group to develop the strategy for the use of these very accurate links in UTC. The terms of reference of this study group include the establishment of standards for data transmission and the validation of the compatibility of the different techniques.

### 5. Key comparisons (E.F. Arias, H. Konaté, G. Panfilo, A. Harmegnies and L. Robertsson)

#### *Key comparison in Time CCTF-K001.UTC*

Results of the key comparison in time, [CCTF-K001.UTC](#), involving the time laboratories participating in the CIPM MRA, have been published monthly in the BIPM key comparison database (KCDB) since March 2015.

### *Key comparison of stabilized lasers CCL-K11*

Following a decision at the 98th meeting of the CIPM in 2009 the BIPM continues to support the [CCL-K11](#) key comparison by participating in measurement campaigns and by providing general advice whenever solicited. This comparison is the internationally recognized traceability chain to the SI metre and is supervised by the CCL. In 2015, BIPM staff supported the key comparison on issues relating to the development of the measurement campaigns and reporting.

#### **6. Rapid UTC (A. Harmegnies, G. Panfilo, G. Petit and L. Tisserand)**

Since January 2013 the Time Department has published a UTC rapid solution 'UTCr', that is, daily values of  $[UTCr - UTC(k)]$  evaluated on a weekly solution on one-month batches of data. About 48 laboratories that are traceable to UTC contribute to UTCr, together representing 60 % to 70 % of the clocks participating in UTC.

UTCr attained the expected quality, providing a weekly solution which is consistent within 1.1 ns RMS and  $\pm 3$  ns peak to peak with the values published monthly in BIPM *Circular T*. The results (<ftp://tai.bipm.org/UTCr>) have been published every Wednesday, without interruption since the end of February 2012.

UTCr does not change the procedures for the monthly calculation of UTC, which remains the only key comparison on time. However, UTCr favourably impacts on the quality of the local representations UTC(k) in national laboratories, and on the steering of GNSS times to UTC via some UTC(k).

#### **7. New proposed definition of UTC (F. Arias)**

The BIPM has actively participated in discussions about a possible redefinition of UTC without leap seconds since 2000. This proposal favours systems that require precise time synchronization and does not allow a discontinuity in the time scale that they use as a reference.

The BIPM contributed to this process at the International Telecommunication Union (ITU), and participated in the World Radiocommunication Conference held in Geneva, Switzerland, from 2-27 November 2015 (WRC15). A resolution of the WRC15 stresses the responsibility of the BIPM on the definition and maintenance of the reference time scale, and of the ITU on its dissemination by time signals and frequency services. The resolution also recommends strengthening the cooperation that the ITU has with the BIPM and other international organizations and delaying the decision on the adoption of a continuous reference time scale until WRC23. In the meantime, further studies are to be developed on the impact of possible reference time scales.

#### **8. Pulsars (G. Petit)**

Collaboration continues with radioastronomy groups that observe pulsars and which analyse pulsar data to study the possibility of using millisecond pulsars as a means of sensing the very long-term stability of atomic time. The Time Department provides these groups with its post-processed realization of Terrestrial Time, TT(BIPM). Additionally it participates in a Working Group on pulsars and time scales established by the International Astronomical Union (IAU).

#### **9. Space-time references (E.F. Arias and G. Petit)**

Activities related to the realization of reference frames for astronomy and geodesy are ongoing in cooperation with the International Earth Rotation and Reference Systems Service (IERS). In these domains, improvements in

accuracy will increase the need for a full relativistic treatment and it is essential to continue to participate in international working groups in this field.

Cooperation continues on the maintenance of the international celestial reference system within the framework of the activities of a working group created by the IAU in August 2012. This working group met within the period, and submitted a report on the features of the next realization of the International Celestial Reference Frame (ICRF3) to the IAU General Assembly held in Honolulu, USA, in August 2015, with a view to the submission of the catalogue with the set of coordinates in ICRF3 in 2018.

As part of its participation in the Conventions Centre of the IERS, the BIPM maintains the web and ftp sites for the *IERS Conventions* (<http://tai.bipm.org/iers/>). The Conventions describe the latest realizations of the celestial and terrestrial reference frames, and the model for the transformation between them. They also describe conventional models for the gravitational field, the displacement of markers on the Earth's crust and for the propagation of electromagnetic signals. In addition, the *Conventions* now provide a complete set of associated conventional software. Since the completion of the latest reference edition, *IERS Conventions* (2010) in December 2010, work is continuing with the help of an Editorial Board to provide updates to the *Conventions* (2010) which are posted on the website (<http://tai.bipm.org/iers/convupdt>).

## 10. Comb activities (L. Robertsson)

The BIPM comb activities are limited to the maintenance of the BIPM frequency comb for internal use related to laser applications only and in other departments when needed.

## 11. Publications

### External publications

1. Fey A, Arias E.F., *et al.*, The second realization of the International Celestial Reference Frame by Very Long Baseline Interferometry, *Astron. J.*, 2015, **150**, 58.
2. Petit G., Arias F., Panfilo G., International atomic time: Status and future challenges, *Comptes Rendus Physique*, 2015, **16**(5), 480-488.
3. Jiang Z., Czubla A., Nawrocki J., Lewandowski W., Arias E.F., Comparing a GPS time link calibration with optical fibre self-calibration with 200 ns accuracy, [\*Metrologia\*, 2015, \*\*52\*\*\(2\), 384-391.](#)
4. Defraigne P., Petit G., CGGTTS-V2E: an upgraded standard for GNSS Time Transfer, [\*Metrologia\*, 2015, \*\*52\*\*\(6\), G1.](#)
5. Petit G., Conventional reference systems, models and parameters for space geodesy, in *Encyclopedia of Geodesy*, E. Grafarend Editor, Springer, to be published.
6. Petit G., Arias E.F., Panfilo G., International atomic time: Status and future challenges, *Comptes Rendus de Physique*, 2015, **16**(5), 480-488.
7. Petit G., Kanj A., *et al.*,  $1 \times 10^{-16}$  frequency transfer by GPS PPP with integer ambiguity resolution, [\*Metrologia\*, 2015, \*\*52\*\*\(2\), 301-309.](#)
8. Luzum B., Petit G., *et al.*, IAU Working Group for Numerical Standards of Fundamental Astronomy (NSFA): Past Efforts and Future Endeavors, *IAU General Assembly*, 2015.
9. Jiang Z, Czubla A, Nawrocki J, Lewandowski W and Arias F (2015), Comparing a GPS time link calibration to an optical fibre self-calibration with 200 ps accuracy, [\*Metrologia\*, 2015, \*\*52\*\*\(2\), 384-391.](#)



10. Jiang Z. (2015) Link calibration or receiver calibration for accurate time transfer? *Proc. EFTF/IFCS2015*, April, Denver, US
11. Yao J., Skakun I., Jiang Z. and Levine J. A Detailed Comparison of Two Continuous GPS Carrier-Phase Time Transfer Techniques, *Metrologia*, 2015, **52(5)**, 666-676.
12. Matsakis D., Jiang Z. Wu W (2015) Carrier Phase and Pseudo-range Disagreement as Revealed by Precise Point Positioning Solutions, *Proc. EFTF/IFCS2015*, April, Denver, US.
13. Esteban H., Galindo J., Bauch A., Polewka T., Cerretto G., Costa R., Whibberley P., Uhrich P., Chupin B., Jiang Z. (2015) GPS Time Link Calibrations in the Frame of EURAMET Project 1156, *Proc. EFTF/IFCS2015*, April, Denver, US.

#### BIPM publications

14. *BIPM Annual Report on Time Activities for 2014*, 9, 131 pp., available only at <http://www.bipm.org/en/bipm/tai/annual-report.html>.
15. *Circular T* (monthly), 8 pp.
16. *Rapid UTC (UTC<sub>r</sub>)* (weekly), 1 pp.

## 12. Activities related to the work of Consultative Committees

E.F. Arias is Executive Secretary of the Consultative Committee for Time and Frequency (CCTF). She is the Secretary of the CCTF Working Group on TAI (WGTAI) and the CCTF Working Group on Strategic Planning (WGSP).

Z. Jiang is Secretary of the CCTF Working Group on TWSTFT (WGTWSTFT).

G. Panfilo is Secretary of the CCTF Working Group on the CIPM MRA (WGMRA) and the CCTF Working Group on Time Scale Algorithms (WG-ALGO). She has been appointed in November 2015 Secretary of the Consultative Committee for Acoustics, Ultrasound and Vibration (CCAUV).

G. Petit is Secretary of the CCTF Working Group on Primary and Secondary Frequency Standards (WGSPFS) and the Working Group on Global Navigation Satellite Systems (WGGNSS).

L. Robertsson is Executive Secretary of the Consultative Committee for Length (CCL), a member of the CCL Working Group on Strategic Planning (WG-S) and of the Discussion Group DG-11 (Lasers). He is the BIPM representative on the CCM Working Group on Gravimetry (WGG). He is also Secretary for the CCTF WG on Coordination of the Development of Advanced Time and Frequency Transfer Techniques (WGATFT) and shares the secretariat of the CCL-CCTF Frequency Standards WG (WGFS) with E.F. Arias.

## 13. Activities related to external organizations

E.F. Arias is a member of the IAU and participates in its working group on the International Celestial Reference Frame (ICRF); she had been vice-president of Commission 31 (Time) until mid-2015, when the IAU put in place a new set of commissions. She has been elected a member of the Steering Committee of IAU Division A on Fundamental Astronomy and a member of the Division A Working Group on the Third Realisation of the International Celestial Reference Frame. She is an associate member of the IERS, a member of its International Celestial Reference System Centre, and of the Conventions Centre. E.F. Arias is a member of the International VLBI Service (IVS). She is the BIPM representative to the Governing Board of the International GNSS Service (IGS). She is the BIPM representative to the UN sponsored International Committee on GNSS (ICG) and the chairperson of its Task Force on Time References. E.F. Arias is a member of the IAG Global Geodetic

Observing System (GGOS) Steering Committee representing the BIPM. She is a member of the Argentine Council of Research (CONICET) and an associate astronomer at the LNE-SYRTE, Paris Observatory. She is a corresponding member of the *Bureau des longitudes* and the BIPM representative to the Working Party 7A of Study Group 7 of the International Telecommunication Union – Radiocommunication Sector (ITU-R).

G. Petit is co-director of the Conventions Centre of the IERS. He is an associate member of the IGS and member of the IGS Working Groups on Clock Products and on Bias Calibration. He is a member of the IAU Working Groups on Numerical Standards in Fundamental Astronomy and on Pulsar Time Scale.

G. Panfilo collaborates with the Joint Committee for Guides in Metrology (JCGM) Working Group 1 (WG1) on the Expression of Uncertainty in Measurement (GUM) to provide a section on uncertainty of time measurements for the new version of the GUM.

#### 14. Travel in 2015 (conferences, lectures and presentations, visits)

E.F. Arias to:

- Paris (France), 29 January, for the Journée GNSS et Science at the CNES, with an invited lecture;
- Vienna (Austria), 17-18 March, for the meeting of the EURAMET Technical Committee on Time and Frequency for coordinating on GNSS calibrations and presenting the Time Department activities;
- Geneva (Switzerland), 23 March to 1 April, for the 2nd Conference Preparatory Meeting for the ITU World Radiocommunication Conference 2015;
- Gran Canaria (Spain), 18-20 May, for the URSI Atlantic Radio Science Conference, to chair a session and make a presentation, and to participate to the business meeting of URSI Commission A;
- Geneva (Switzerland), 20-26 May, for the meeting of the Working Party 7A at the ITU;
- Bordeaux (France), 16 June, for a PhD dissertation, acting as rapporteur and member of the jury;
- Saint Mandé (France), 17 June, to the Journée Scientifique de l'IGN;
- Honolulu (Hawaii, USA), 4-14 August, for the IAU General Assembly 2015, for the relevant commission and working group meetings, including presentations and reports;
- London (UK), 28 October, for the Workshop on UTC Traceability to the Financial Sector, with an invited lecture;
- CCTF and associated meetings.

Z. Jiang to:

- Xian (China), 13-15 May, for the China Satellite Navigation Conference 2015;
- Beijing (China), May, for a visit to NIM gravity and time laboratories;
- Xian (China), May, for a visit to NTSC time laboratory;
- Denver (Colorado, USA) 13-17 April, to attend the FCS/EFTF 2015 and the TWSTFT Participation Stations meeting;
- CCTF and associated meetings.

G. Panfilo to:

- Turin (Italy), 16-21 April, for the master's degree panel for Federica Parisi, and for planning future work in collaboration between the BIPM Time Department and the Department of Mathematics at the University of Turin;

- CCTF and associated meetings.

G. Petit to:

- Paris (France), 29 January, to attend the “Journée GNSS et science”;
- Denver (Colorado, USA) 13-17 April, to attend the FCS/EFTF 2015 meeting, to give three oral presentations, and to attend two CCTF WG meetings;
- Besançon (France), 30 June-1 July, to give two lectures at the European Frequency and Time Seminar;
- Prague (Czech Republic), 24-27 June, to attend the UGGI General Assembly, with one invited presentation;
- Potsdam (Germany), 12-14 October, for the 8th Symposium on frequency standards and metrology, with one presentation;
- Boulder (Colorado, USA), 2-6 November, for the tenth meeting the International Committee on GNSS (ICG), with chair of task force and presentations;
- Toulouse (France), 18-19 November, for the GRGS Workshop G2 with a presentation, and for discussions with the CNES/CLS groups on the IPPP technique;
- Bern (Switzerland), 30 November-1 December, for the ISSI workshop HISPAC, with an invited presentation;
- CCTF and associated meetings.

**15. Visitors, secondees**

- W. Wu (NICT, Chinese Academy of Sciences) on a one-year secondment starting on 3 June 2014, for activities on time transfer and calibration;
- F. Parisi from the University of Torino (Italy) to study an independent time scale based on the Kalman Filter, 1 November 2014 – 6 March 2015, 1-30 June 2015 and 1-18 September 2015;
- S. Zagier from Zagier and Urruty Publications for discussions on the historical evolution of clocks, 23 April 2015;
- J. Park and S.H. Hong (KRISS, Republic of Korea) for a visit to the Time Department and laboratory, 20 July 2015;
- Long-Sheng Ma from the East China Normal University (China) for discussions on optical metrology, 9-13 September 2015;
- P. Gabor from the Vatican Observatory for discussions on time scales for astronomy, 5 October 2015;
- K. Madanipour, A.M. Levi and O. Masoudi (Iran) for a visit to the Time Department and laboratory, 23 October 2015;
- D. Rovera (LNE-SYRTE) for the QMS external audit of the Time Department, 10 December 2015.

**BIPM Ionizing Radiation Department**  
**Director: J.M. Los Arcos**  
**(1 January 2015 to 31 December 2015)**

**1. X- and  $\gamma$ -rays (D.T. Burns, C. Kessler, S. Picard and P. Roger)**

**1.1 Dosimetry standards and equipment**

The project to develop an absorbed-dose standard for medium-energy x-rays is nearing completion. Modifications were made to the water phantom positioning mechanism to allow more accurate depth positioning, a critical parameter in this domain. The chamber depth can now be set to better than 0.02 mm. Repeat measurements were made of the transfer chamber responses in air and water; some observed instabilities remain under investigation and a fourth transfer chamber is currently being characterized. A pilot comparison with the PTB, Germany, was conducted in November 2015. The results were satisfactory, but indicated that more work was necessary on photon attenuation coefficients. This additional work is in progress. A first full comparison is anticipated mid-2016, giving rise to a new series of comparisons and calibrations in this domain.

The Department coordinated the ninth comparison in the series BIPM.RI(I)-K6 for absorbed dose to water in high-energy photon beams, with the NMIJ/AIST, Japan. The measurements were made in the 6 MV, 10 MV and 15 MV beams of the NMIJ/AIST Elekta accelerator from 9 to 23 April 2015 and the corresponding Monte Carlo calculations (for a depth of 10 g cm<sup>-2</sup>) were made at the BIPM using photon spectra supplied by the NMIJ/AIST. The BIPM equipment was shipped to the NMIJ/AIST in advance, with the exception of the calorimeter core and ionization chambers which were carried by hand. This comparison enabled the NMIJ/AIST to verify robustly the present Japanese primary standard to realize absorbed dose to water in accelerator beams. This comparison presented no particular problems and the draft B report is in progress.

The reports of the previous comparisons with the NPL, UK, were published, and the report of the previous comparison with the VSL, the Netherlands, is currently being reviewed by the CCRI.

The design study to establish a new laboratory for the reference air kerma rate facility for HDR <sup>192</sup>Ir brachytherapy sources is complete and is being implemented to allow the continuity of the comparison series BIPM.RI(I)-K8 carried out at the NMIs. This laboratory will also house the x-ray radiograph equipment. The support mechanism for the source transfer and for the radiographic unit has been constructed and the robotic arm installed. A further comparison in the series was carried out with the NMIJ in April 2015. The results and reports of two previous comparisons were published.

Primary measurements and reference chamber calibrations have continued in all of the reference x- and  $\gamma$ -ray beams. Comparisons and calibrations are underpinned by a significant effort in equipment calibration and maintenance, as required by the BIPM Quality System. This system was successfully subject to a peer review external audit in December 2015.

**1.2 Dosimetry comparisons**

In summary, eight comparisons were carried out in terms of air kerma or absorbed dose to water using the BIPM x- and gamma-radiation beams with the CMI (2), ENEA, ININ, ITN, NIM, NMIJ and the PTB. In addition, one high-energy absorbed-dose-to-water comparison BIPM.RI(I)-K6 was carried out with the NMIJ using their own accelerator facility and another on-site comparison for the reference air kerma rate for HDR <sup>192</sup>Ir brachytherapy sources BIPM.RI(I)-K8 was also carried out with the NMIJ using the Japan Radioisotopes Association (JRIA) facility.

Thirteen comparison reports were approved and published in the *Metrologia Technical Supplement* for the BEV (3), ENEA (2), ININ, METAS, NIM, NMIJ, NPL (2), NRC and the PTB, plus one comparison with the LNHB published as a BIPM report (see publication list below).

### 1.3 Characterization of national standards for dosimetry

Eighteen characterizations of national secondary standards were carried out for the CIEMAT (four in the  $^{60}\text{Co}$  radiotherapy beam), CRRD (four in the radioprotection beams), HIRCL (two in the  $^{60}\text{Co}$  radiotherapy beam and two in x-rays), IAEA (four in the mammography beams) and the ININ (two in the  $^{60}\text{Co}$  radiotherapy beam).

In addition, the IAEA/WHO dosimetry assurance programme continues to be supported by reference irradiations, which involved only one series of irradiations in 2015 for the radiotherapy level in the  $^{60}\text{Co}$  beam.

## 2. Radionuclides (J.M. Los Arcos, S. Courte, C. Michotte, M. Nonis and G. Ratel)

In parallel to the scientific activities, the department undertook a considerable amount of work with the ampoules containing radioactive solutions received over the last 35 years from radionuclide metrology laboratories around the world and stored in an obsolete facility. All the ampoules were checked before being transferred to a new room that has been specially designed for the storage of radioactive substances. They have been rearranged using a more rigorous and systematic scheme.

### 2.1 International Reference System (SIR) for $\gamma$ -ray emitting radionuclides

#### 2.1.1 SIR submissions in 2015

Within the radionuclide measurements programme, the Système International de Référence (SIR) received during 2015 ten ampoules of six different radionuclides from eight NMIs –  $^{57}\text{Co}$  (BEV and NMISA),  $^{68}\text{Ge}$  (IRA-METAS, LNE-LNHB and NIM),  $^{65}\text{Zn}$  (IRD-LNMRI),  $^{110\text{m}}\text{Ag}$  (PTB),  $^{131}\text{I}$  (BEV and POLATOM-RC) and  $^{134}\text{Cs}$  (IRD-LNMRI), all oriented to generate equivalence values in the ongoing BIPM.RI(II)-K1 comparison. The three ampoules of  $^{68}\text{Ge}$  received from three laboratories will serve to evaluate the key comparison reference value (KCRV) for this radionuclide and to provide a link for the CCRI key comparison [CCRI\(II\)-K2.Ge-68](#).  $^{223}\text{Ra}$  ( $T_{1/2} = 11.43$  d,  $u = 0.03$  d) is a promising radionuclide for the therapy of some cancers, for which two ampoules from two laboratories have been evaluated in 2015, constituting a new SIR entry. An earlier submission of  $^{111}\text{Ag}$  has been evaluated and the draft A report issued following receipt of the corresponding reporting form. An ampoule of  $^{57}\text{Co}$  arrived at the BIPM slightly deteriorated. The solution will be reconditioned in an undamaged ampoule to allow measurement in the SIR chambers and it will be sent back to the producing laboratory for checking.

#### 2.1.2 SIR reports

Updated final reports of four BIPM.RI(II)-K1 comparisons were published in the *Metrologia Technical Supplement* covering  $^{65}\text{Zn}$ ,  $^{85}\text{Sr}$ ,  $^{207}\text{Bi}$  and  $^{166\text{m}}\text{Ho}$ , including linked results from the CCRI(II)-K2.Zn-65 comparison. In addition, a final report for  $^{56}\text{Mn}$  was published with the first KCRV for this nuclide. There are 42 SIR results awaiting publication in the KCDB (five in the draft A stage and 37 in the draft B stage) and every effort will be made to ensure that reports are published as quickly as possible, particularly when NMIs make submissions that are to replace outdated results that have already been removed from the KCDB. Finally, reporting forms for ten measurements are still with the NMIs concerned.

Four SIR results for  $^{241}\text{Am}$  are pending publication and its KCRV update was proposed and discussed at the KCWG(II) meeting in November 2015. Some potential outliers were identified where the activity in the ampoule

was not sufficiently high in view of the background current of the SIR. However, it was decided that further measurements would be carried out at the BIPM and at the LNE-LNHB in order to confirm whether or not there is a bias.

## 2.2 Gamma spectrometry

Measurements of potential impurities in SIR ampoules have been suspended since the failure of the Ge(Li) spectrometer in July 2013. Following the contribution of Dr Antohe, who had been on secondment from IFIN-HH, Romania, in 2014, the efficiency curves of the replacement HPGe spectrometer and the uncertainty budget need to be finalized. New procedures will be drafted and the entire process validated before re-offering the gamma-ray spectrometry service to the SIR participants.

## 2.3 Extension of the SIR to short-lived radionuclides

Monte Carlo simulations of the SIR Transfer Instrument (SIRTI) response for  $^{18}\text{F}$  as a function of the ampoule shape and filling height were finalized for the evaluation of the corresponding uncertainty components of the SIRTI measurements. The first three results in the [BIPM.RI\(II\)-K4.F-18](#) ( $T_{1/2} = 1.8$  h) key comparison (VNIIM, NPL and ENEA-INMRI in 2014) were presented at the ICRM-2015 conference in Vienna, Austria, on 8-11 June 2015 and accepted for publication in *Applied Radiation and Isotopes*. The uncertainty of the SIRTI measurements at the NPL is mainly due to the potential bias related to the presence of a large drop in the ampoule neck. This is evaluated by Monte-Carlo simulation but significant uncertainty arises because the volume of the drop is difficult to estimate. Consequently, it was decided that for future SIRTI comparisons a centrifuge would be sent to the NMI for the comparison, if not already available on-site.

The [BIPM.RI\(II\)-K4.Tc-99m](#) ( $T_{1/2} = 6.0$  h) key comparisons using the SIRTI at VNIIM and ENEA-INMRI in 2014 are in the draft A stage. The  $^{99\text{m}}\text{Tc}$  and  $^{18}\text{F}$  SIRTI comparisons took place at the NMISA, South Africa, in November 2015 and the results are pending.

The first calibration measurements of the SIRTI against the SIR for  $^{64}\text{Cu}$  ( $T_{1/2} = 13$  h) were carried out by measuring a solution from the CNRS-Orléans, France, in both systems. A second series of  $^{64}\text{Cu}$  calibration measurements will take place in early 2016 prior to the first SIRTI comparison of  $^{64}\text{Cu}$  at NIST. As  $^{64}\text{Cu}$  is mainly a  $\beta^+$  emitter like  $^{18}\text{F}$ , the uncertainty budget for SIRTI measurements of  $^{64}\text{Cu}$  is taken to be identical to that of  $^{18}\text{F}$ .

## 2.4 Extension of the SIR to pure beta emitters

Due to a failure of the micro balance used by the radioactivity group, the pilot study for the extension of the SIR to beta emitters had to be postponed. A new balance has been purchased and installed in a dedicated room that has been refurbished specifically for the purpose. The balance is undergoing tests before it can be used intensively for preparing suitable liquid-scintillation samples to resume the exercise in the second half of 2016.

## 2.5 Radionuclide comparisons, reports and quality assurance.

In summary, twelve radionuclide activity comparisons were undertaken, ten BIPM.RI(II)-K1 ongoing comparisons for several radionuclides and NMIs (see 2.1) and two BIPM.RI(II)-K4 comparisons for  $^{99\text{m}}\text{Tc}$  and  $^{18}\text{F}$  carried out on-site at the NMISA.

Five updated reports of BIPM.RI(II)-K1 comparisons were published in the *Metrologia Technical Supplement* covering  $^{56}\text{Mn}$ ,  $^{65}\text{Zn}$ ,  $^{85}\text{Sr}$ ,  $^{207}\text{Bi}$  and  $^{166\text{m}}\text{Ho}$ , and the first three results in the BIPM.RI(II)-K4.F-18 comparison were submitted for publication in *Applied Radiation and Isotopes*. In addition, the radioactivity group

contributed to three chapters in the *Metrologia* special issue on 'Uncertainties in Radionuclide Metrology' as well as to the guest editors' team (see references 1, 5, 15 and 24).

Finally, a new simplified scheme for radionuclide K2-comparison reports was proposed and accepted at the meetings of the Key Comparisons Working Group (KCWG(II)) and CCRI(II) in March 2015. Consequently, reports for CCRI.RI(II)-K2.H-3 and CCRI.RI(II)-K2.Sr-89 were circulated through the CCRI(II) for comments. The KCWG(II) finally approved the new scheme in November 2015 and this will be implemented to expedite the delayed reports as well as future reports.

All SIR measurements, including the extension to short-lived radionuclides, are covered by the BIPM Quality Management System which was successfully subjected to a peer review external audit in December 2015.

### 3. Thermometry (S. Picard, M. Nonis)

From 2010 until 2014 the Ionizing Radiation Department provided internal calibration services for thermometry at the BIPM under the terms of the BIPM Quality Management System. The BIPM high-precision resistance bridge failed during 2014 and needed to be replaced. For economic reasons, it was decided to stop this activity in 2015 and the calibration of SPRTs and work thermometers has been out-sourced.

### 4. Publications

1. Amiot M.N., Chisté V., Fitzgerald R., Juget F., Michotte C., Pearce A., Ratel G., Zimmerman B.E., Uncertainty evaluation in activity measurements using ionization chambers, *Metrologia*, 2015, **52**(3), S108-S122.
2. Burns D.T., Kessler C., Steurer A., Tiefenboeck W., Bauer M., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the BEV, Austria and the BIPM in low-energy x-rays, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06001.
3. Burns D.T., Kessler C., Steurer A., Tiefenboeck W., Bauer M., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the BEV, Austria and the BIPM in medium-energy x-rays, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06004.
4. Burns D.T., Kessler C., Tanaka T., Kurosawa T., Saito N., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the NMIJ, Japan and the BIPM in low-energy x-rays, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06008.
5. Karam L., Keightley J., Los Arcos J.M., Practical implementation of uncertainty analysis in radionuclide metrology, *Metrologia*, 2015, **52**(3), S1-S2.
6. Karam L. and Ratel G., Consultative committee on ionizing radiation: Impact on radionuclide metrology, *Appl. Radiat. Isotopes* (2015), <http://dx.doi.org/10.1016/j.apradiso.2015.11.085> (in press).
7. Kessler C., Allisy-Roberts P.J., Selbach H.J., Comparison BIPM.RI(I)-K8 of high dose-rate Ir-192 brachytherapy standards for reference air kerma rate of the PTB and the BIPM, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06005.
8. Kessler C., Burns D., Roger P., Toni M.P., Pinto M., Bovi M., Cappadozzi G., Silvestri S., Key comparison BIPM.RI(I)-K7 of the air-kerma standards of the ENEA-INMRI, Italy and the BIPM in mammography x-rays, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06025.

9. Kessler C., Burns D.T., Alvarez Romero J.T., De la Cruz Hernández D., Cabrera Vertti M.R., Tovar-Muñoz V.M., Key comparison BIPM.RI(I)-K5 of the air kerma standards of the ININ, Mexico and the BIPM in  $^{137}\text{Cs}$  gamma radiation, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06020.
10. Kessler C., Burns D.T., Li D., Wang P., Key comparison BIPM.RI(I)-K5 of the air kerma standards of the NIM, China, and the BIPM in  $^{137}\text{Cs}$  gamma radiation, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06009.
11. Kessler C., Burns D.T., Steurer A., Tiefenboeck W., Bauer M., Key comparison BIPM.RI(I)-K7 of the air-kerma standards of the BEV, Austria and the BIPM in mammography x-rays, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06003.
12. Kessler C., Burns D.T., Vörös S., Hofstetter-Boillat B., Key comparison BIPM.RI(I)-K4 of the absorbed dose to water standards of the METAS, Switzerland and the BIPM in  $^{60}\text{Co}$  gamma radiation, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06002.
13. Kessler C., Downton B., Mainegra-Hing E., Comparison BIPM.RI(I)-K8 of high dose-rate Ir-192 brachytherapy standards for reference air kerma rate of the NRC and the BIPM, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06013.
14. Kessler C., Pinto M., Cappadozzi G., Silvestri C., Bovi M., Toni M.P., Key comparison BIPM.RI(I)-K1 of the air-kerma standards of the ENEA-INMRI, Italy and the BIPM in  $^{60}\text{Co}$  gamma, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06023.
15. Kossert K., Broda R., Cassette P., Ratel G. and Zimmerman B., Uncertainty determination for activity measurements by means of the TDCR method and the CIEMAT/NIST efficiency tracing technique, *Metrologia*, 2015, **52**(3), S172-S190.
16. Los Arcos J.M., Stock M., Wielgosz R., Arias F. and Milton M., News from the BIPM laboratories: 2014, *Metrologia*, 2015, **52**(1), 155-162.
17. Michotte C., Ratel G., Courte S., Bobin C., Moune M., Update of the BIPM comparison BIPM.RI(II)-K1.Bi-207 of activity measurements of the radionuclide  $^{207}\text{Bi}$  to include the 2010 result of the LNE-LNHB (France), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06012.
18. Michotte C., Ratel G., Courte S., Dziel T., Listkowska A., Update of the BIPM comparison BIPM.RI(II)-K1.Sr-85 of activity measurements of the radionuclide  $^{85}\text{Sr}$  to include the 2009 result of the POLATOM (Poland), *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06022.
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20. Michotte C., Ratel G., Courte S., Keightley J., Participation of the NPL in 2008 in the BIPM.RI(II)-K1.Mn-56 comparison of activity measurements of the radionuclide  $^{56}\text{Mn}$ , *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06018.
21. Picard S., Burns D.T., Roger P., Duane S., Bass G.A., Manning J.W. Shipley D.R., Key comparison BIPM.RI(I)-K6 of the standards for absorbed dose to water at  $5\text{ g cm}^{-2}$  and  $7\text{ g cm}^{-2}$  of the NPL, United Kingdom, and the BIPM in accelerator photon beams, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06010.
22. Picard S., Burns D.T., Roger P., Duane S., Bass G.A., Manning J.W. Shipley D.R., Key comparison BIPM.RI(I)-K6 of the standards for absorbed dose to water at  $10\text{ g cm}^{-2}$  of the NPL, United Kingdom, and the BIPM in accelerator photon beams, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06021.
23. Picard S., Burns D.T., C. Kessler, Roger P., Delaunay F., Daures J., Donois, M., Ostrowsky A., Report of a Comparison between the LNE-LNHB and the BIPM of Absorbed Dose to Graphite in a Co-60 Reference beam, *BIPM Report-15/03*.



24. Ratel G., Michotte C., Bochud F.O., Uncertainty of combined activity estimations, *Metrologia*, 2015, **52**(3), S30-S41.
25. Ratel G., Michotte C., Courte S., Kossert K., Update of the BIPM comparison BIPM.RI(II)-K1.Ho-166m activity measurements of the radionuclide  $^{166\text{m}}\text{Ho}$  for the PTB (Germany), with linked results for the EURAMET.RI(II)-K2.Ho-166m comparison, *Metrologia*, 2015, **52**, *Tech. Suppl.*, 06006.

## 5. Activities related to the work of Consultative Committees and RMOs.

J.M. Los Arcos is the Executive Secretary of the CCRI, an *ex-officio* member of all CCRI working groups, Coordinator of the CCRI(II) Working Group on the Extension of the SIR to beta-emitters (ESWG(II)) and Rapporteur of the CCRI RMO Working Group (RMOWG). During 2015, a complete series of meetings of the CCRI(I), CCRI(II), CCRI(III), CCRI and CCRI RMOWG was held in March and the Key Comparisons Working Group (KCWG(II)) met in March and November.

D.T. Burns is a member of the CCRI(I) Key Comparisons Working Group (KCWG(I)) and the Brachytherapy Standards Working Group (BSWG(I)). He is also a member of an *ad hoc* group evaluating the effect of excess charge on the value for  $W_{\text{air}}$  (work that will be reported this year in the ICRU Report on Key Data). Since 2009 he has been *rapporteur* at annual meetings of the CCRI.

C. Kessler is the Coordinator of the CCRI(I) Brachytherapy Standards Working Group (BSWG(I)).

C. Michotte is a member of the Key Comparisons Working Group (KCWG(II)) which met in March and November 2015.

S. Picard is the Executive Secretary of the Consultative Committee for Acoustics, Ultrasound and Vibration (CCAUV) which held its 10th meeting on 25-27 November 2015. She is the Executive Secretary of the Consultative Committee for Thermometry (CCT). Susanne Picard became KCDB Coordinator on 1 July 2015. She was invited to participate in the APMP-TC-AUV, APMP-TC-T, and APMP-TC-IR meetings in November.

G. Ratel is a member of the CCRI(II) Working Group on the Extension of the SIR to beta-emitters using liquid scintillation (ESWG(II)) and of the KCWG(II), the latter of which met in March and November 2015.

For ionizing radiations, seven comparison reports from the CCRI(III), APMP.RI(II), EURAMET.RI(I) (2), EURAMET.RI(II) (2) and SIM.RI(I) were reviewed technically and editorially prior to its circulation through the respective CCRI section for approval and were eventually published in the *Metrologia Tech. Suppl.* series. Within the CCAUV activities, two comparison reports from COMET.AUV.W and EURAMET.AUV.V and a pilot study report from APMP.AUV.V were processed for approval and published in the *Metrologia Tech. Suppl.* series. As well, in the CCT field, eight comparison reports from the CCT (2), COOMET.T (2) and SIM.T (4) were processed for approval and published in the *Metrologia Tech. Suppl.* series.

## 6. Activities related to external organizations

J.M. Los Arcos had been proposed and was elected in June 2015 as an Associate Member of the International Committee for Radionuclide Metrology (ICRM). He also evaluates scientific projects for the Spanish National Evaluation and Foresight Agency (ANEP) and is a technical auditor for the Spanish accreditation body.

D.T. Burns is a Fellow of the Institute of Physics (FInstP) in the UK, an elected Commissioner of the ICRU and Chairman of the ICRU Committee on Fundamental Quantities and Units. He is a member of the ICRU Report Committee on Key Data for Dosimetry and is Commission Sponsor for three ICRU reports (Key Data for Dosimetry, Operational Quantities for Radiation Protection, and Small and Non-Standard Fields). He is a

member of the Scientific Committee of the IAEA/WHO Network of Secondary Standards Dosimetry Laboratories.

C. Michotte is the Scientific Secretary and *rapporteur* for the JCGM-WG1 meetings, which were held in June and October 2015. She was the Scientific Secretary of the Organizing Committee for the BIPM Workshop on Measurement Uncertainty which took place the 15-16 June 2015 and was attended by over 100 scientists from more than 40 countries. In addition a number of other registered participants were able to see and hear the presentations live over the internet as part of the first ever webcast transmitted by the BIPM. She also gave an invited talk at the workshop, entitled "Feedback from NMIs and JCGM MOs to the circulated JCGM 100 and 110 Committee Drafts".

G. Ratel is the BIPM representative at the ICRM and is the President of the ICRM Nominating Committee. He is a member of the Scientific Committee for the 20th International Conference on Radionuclide Metrology and its Applications (ICRM 2015), held in Vienna (Austria) on 8-11 June 2015.

## 7. Travel in 2015 (conferences, lectures and presentations, visits)

D.T. Burns to:

- Tsukuba (Japan), 16-24 April, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams of the NMIJ/AIST.

C. Kessler to:

- Tsukuba (Japan), 15-22 April, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams of the NMIJ/AIST.

C. Kessler and P. Roger to:

- Lyon (France), 3 July, to visit the Inno-Robot exhibition on developments in robotics, for a demonstration of the robotic arm necessary for the new brachytherapy installation.

S. Picard to:

- Lisbon (Portugal), 26 February, to participate at the EURAMET TC-T meeting and to present recent news from the BIPM and the CCT.
- Tsukuba (Japan), 7-17 April, to carry out the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the NMIJ/AIST at their medical accelerator facility.
- Toronto (Canada), 7-12 June, to participate at the World Congress on Medical Physics and Biomedical Engineering, where she gave a presentation entitled "Results from the on-going key comparison BIPM.RI(I)-K6 : What have we learned ?" (co-author D. T. Burns, BIPM).
- Beijing (China), invited by the NIM (China), 1-6 November, to visit the NIM and participate at the APMP-TC meeting in Acoustics, Ultrasound and Vibration where she gave a presentation "The BIPM and recent activities of the CCAUV", the APMP-TC meeting in Thermometry where she gave a presentation "The BIPM and recent activities of the CCT", and the APMP-TC meeting in Ionizing Radiation where she gave the presentation "The BIPM and recent activities of the Ionizing Radiation Department". She also gave a presentation at the NIM entitled "BIPM KCDB: today and tomorrow".

C. Michotte to:

- Vienna (Austria), 8-11 June, to attend the 20th ICRM conference and make a presentation entitled "Comparison of  $^{18}\text{F}$  activity measurements at the VNIIM, NPL and ENEA-INMRI using the SIRT1 of the BIPM".

C. Michotte and M. Nonis to:

- Cape Town (South Africa), 16-28 November, to carry out activity comparisons of  $^{99m}\text{Tc}$  (BIPM.RI(II)-K4.Tc-99m) and  $^{18}\text{F}$  (BIPM.RI(II)-K4.F-18) at the NMISA using the SIR Transfer Instrument.

G. Ratel to:

- Vienna (Austria), 8-12 June, to attend the 20th ICRM conference, chair two sessions of the ICRM conference and to attend the ICRM General Meeting.

P. Roger to:

- Tsukuba (Japan), 7-24 April, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the NMIJ/AIST.
- Paris, 23 September, to visit the Enova exhibition for new developments in metrology and electronics.

## 8. Visitors in 2015

A number of delegations from different countries or organizations visited the Ionizing Radiation Department in 2015:

- Mr F. Ferrer, Director and Dr J.A. Robles, Director of the Scientific and International Relations Division, Centro Español de Metrología (CEM), Spain, 23 April.
- Dr P. Oropesa, Head, Radionuclide Metrology Department, Centro de Isótopos (CENTIS), Cuba, 18 June.
- Dr U.W. Lee, President, Dr Y.K. Park, Dean of Academic Affairs, Mr K.T. Park (Academic Affairs) and J.I. Park (Global Cooperation), Korean University of Science and Technology, Republic of Korea, 17 July.
- Dr K. Madanipour, President, Dr A.M. Livari, Deputy and Dr O. Masoudi, President's Advisor, National Metrology Center of Iran (NMCI), Iran, 23 October.
- Dr T. Yamada, Planning Section Manager, Japan Radioisotope Association (JRIA), Japan, 9 December.

## 9. Guest workers in 2015

- Dr L. Rodríguez (ex-CIEMAT, Spain), 1 January to 7 March.
- Dr J.T. Alvarez (ININ, Mexico), 27 February to 5 March.
- Dr M. Pinto (ENEA-INMRI, Italy), 22-26 June.
- Dr T. Tanaka (NMIJ/AIST, Japan), 7-11 September.
- Dr P. Avilés and C. García (CIEMAT, Spain), 14-18 September.
- Dr V. Sochor (CMI, Czech Republic) 1-3 December.
- Dr J. Cardoso (ITN, Portugal), 14-17 December.

**BIPM Chemistry Department****Director: R.I. Wielgosz****(1 January 2015 to 31 December 2015)****1. Gas metrology programme** (J. Viallon, E. Flores, P. Moussay, F. Idrees, T. Choteau and R.I. Wielgosz)**1.1 Greenhouse gases standards****1.1.1 Key comparison on methane standards (CCQM-K82)**

A paper demonstrating equivalence between standards made in whole and synthetic air and measured by cavity ring-down spectroscopy (CRDS) and gas chromatography – flame ionization detection (GC-FID) for atmospheric monitoring applications was published in *Analytical Chemistry* in February 2015, completing activities related to [CCQM-K82](#).

**1.1.2 Preparation for the key comparison on carbon dioxide (CCQM-K120)**

The BIPM developed innovative software to control, perform spectral acquisition and on-line analysis using Fourier Transform Infrared Spectroscopy (FTIR) and the MALT programme, and purchased a CO<sub>2</sub> Isotope Ratio Infrared Spectroscopy (IRIS) system. Both FTIR and IRIS systems were validated successfully using a set of 23 standards produced by NIST, NPL, and NOAA (the WMO-GAW Central Calibration Laboratory, CCL, for CO<sub>2</sub>). The standards were produced using five different sources of CO<sub>2</sub> resulting in a rich diversity of  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  values which were useful for verifying both techniques under conditions that could be expected during an international comparison. The isotopic composition in at least one standard per CO<sub>2</sub> source was value assigned on the VPDB/J-RAS06 scale by the Max Planck Institute for Biogeochemistry (MPI-BGC) in Jena, Germany, being the WMO-GAW CCL for stable isotopes in CO<sub>2</sub>, and/or the Institute of Arctic and Alpine Research - Geography University of Colorado Boulder (INSTAAR). A new paper describing this work entitled “FTIR and IRIS calibration strategies for accurate CO<sub>2</sub> in air mole fraction,  $\delta^{13}\text{C}$  and  $\delta^{18}\text{O}$  measurements” is being drafted and will be submitted to *Analytical Chemistry* in 2016.

In parallel, the assembly of the new manometric system for carbon dioxide measurements continued with the all-glass ensemble which was installed inside an oven maintained at 40°C and connected to a glass trap immersed inside a temperature controlled cryostat. Series of tests were performed, starting with leak detection and sealing, improvement of the carbon dioxide cryogenic trapping using the Residual Gas Analyser to monitor efficiency, and first measurements of the carbon dioxide amount separated from a known mixture in air. This work was supported by the secondment of Dr Steven Maxwell to the BIPM from the NIST, USA.

**1.2 Air quality gas standards****1.2.1 Ongoing ozone photometer comparison and calibration programme (BIPM.QM-K1)**

In 2015, three laboratories brought or sent their ozone national standards to the BIPM for comparison with the BIPM-SRP27 reference standard as part of the key comparison [BIPM.QM-K1](#): the NIST in July, and the VSL, the Netherlands, in November. An additional comparison was performed with the Office of Environment and Heritage NSW, Australia, and published as a BIPM report. All reports of comparisons performed in 2014 have been published.

### 1.2.2 *Ozone absorption cross-section value*

The new value of the ozone absorption cross-section measured by spectroscopy on pure ozone samples at the BIPM in 2014 was published in early 2015 in *Atmospheric Measurement Techniques*. Results were presented to the CCQM Working Group on Gas Analysis (GAWG), together with a proposal to create a task group to review all published values of ozone absorption cross sections at 254 nm, and provide a recommendation on the best value and uncertainty to be used, as a first step in the process of implementing a new cross section value globally. Dr Joseph Hodges (NIST) has accepted the role of chairing the group, with an expected starting date of early 2016.

Meanwhile a paper giving details of the results obtained in 2015 with the gas phase titration (GPT) system is in preparation. All tests and measurements have been completed using the new reaction chamber, including FTIR analysis of the standard mixtures of nitrogen monoxide (NO) and nitrogen dioxide (NO<sub>2</sub>) purchased in 2014. The presence of nitric acid was observed in nitrogen dioxide mixtures, at mole fractions larger than certified. Taking this correction into account, the titration of ozone traceable to either NO or NO<sub>2</sub> gravimetric standards resulted in good agreement, whilst the disagreement with ozone measured by UV photometry was confirmed. Final results are being used to deduce a GPT-based value of the ozone cross-section, with an uncertainty comparable to measurements performed in 2014 on pure ozone samples.

### 1.2.3 *Key comparison on formaldehyde standards (CCQM-K90)*

The comparison started in December 2014 with the measurement of the formaldehyde mole fraction in all 14 transfer standard cylinders against the BIPM dynamic generation system based on permeation of paraformaldehyde. After two series of measurements, two more were performed against the second system based on trioxane diffusion. In April 2015 a subset of eight cylinders were selected and sent to participants. Meanwhile the other six cylinders were regularly measured at an average frequency of one series of measurements per month in order to monitor their stability. Participants performed their own measurements between June and October. Cylinders are to be returned to the BIPM, where they will again be analysed together with the other six, against the two dynamic generation systems. The results will be presented to the GAWG in April 2016.

### 1.2.4 *Preparation for a repeat key comparison on nitrogen dioxide (CCQM-K74.2018)*

Following on from the initial discussion on “Future comparisons and pilot studies for the GAWG - Core KC (NO) and spectroscopy PS”, in April 2015 at the BIPM, the GAWG decided that the pilot study on spectroscopy is to be moved to a comparison timetable to coincide with the NO<sub>2</sub> key comparison planned for 2017. The NO<sub>2</sub> key comparison is to have the number [CCQM-K74.2017](#). This comparison will be designed to evaluate the consistency of primary NO<sub>2</sub> gas standards from National Metrology Institutes at the  $\mu\text{mol mol}^{-1}$  level.

### 1.2.5 *Preparation for the key comparison on nitrogen monoxide (CCQM-K137)*

Planning for a key comparison on nitrogen monoxide (NO) at 30-70  $\mu\text{mol/mol}$  in nitrogen, to commence in late 2016, was discussed in April 2015 within the GAWG. The comparison will be a repeat of the earlier CCQM-P73 pilot study that was coordinated by the BIPM. The facility developed for the pilot study has been re-validated in 2015 after the acquisition of new NO standards to underpin the GPT system. An agreement better than  $0.05 \mu\text{mol mol}^{-1}$  between a set of ten standards from two different NMIs prepared between 2009 and 2015 was demonstrated. Twenty laboratories have expressed an interest in participating in the future comparison.

### 1.2.6 Gas metrology programme quality system

A new procedure covering the comparison and calibration of nitrogen monoxide in nitrogen standards was added to the Quality System that underpins the Gas Metrology work programme. The system was reviewed during an internal audit undertaken in November 2015. No major non-conformities in the documentation and implementation were reported by the auditors. A series of actions to address minor non-conformities and observations raised in the audit report will be undertaken in 2016.

## 2. Organic analysis programme (S. Westwood, R. Josephs, N. Stoppacher, S. Maniguet, A. Daireaux, T. Choteau, and R.I. Wielgosz)

### 2.1 Purity methodology and small molecule purity analysis

The approach to the use of data from organic purity assignment comparisons coordinated by the BIPM described in a "White Paper" prepared by the BIPM was implemented in 2015 by the CCQM Working Group on Organic Analysis (OAWG) and the CCQM Working Group on Key Comparisons and CMC Quality (KCWG).

The BIPM has continued to coordinate an International Union of Pure and Applied Chemistry (IUPAC) working group, with members from 12 NMIs and two international organizations. Two further meetings were held in 2015 to draft technical guidelines on 'Methods for the SI Value Assignment of the Purity of Organic Compounds for use as Primary Reference Materials and Calibrators'. The BIPM organized and hosted the third meeting of the working group in April 2015. The final Technical Report from this working group will be produced in 2016.

### 2.2 NMR work programme

Following the official opening of the BIPM qNMR facility in November 2014 and handover of the 400 MHz spectrometer by the manufacturer, qualification and acceptance testing of the spectrometer were successfully undertaken at the BIPM at the beginning of 2015. An extended visit to the BIPM by Dr Takeshi Saito, NMIJ, Japan, in February 2015 discussed the BIPM-NMIJ collaboration in this area and provided further training to BIPM staff.

Scientists from NIM, China, and UME, Turkey, have worked on secondment at the BIPM in 2015 to develop and validate methods for performing high accuracy qNMR measurements in various deuterated solvents. As a result of this ongoing programme the measurement equation for organic purity determinations by qNMR is being investigated and the major influence factors for qNMR analysis identified.

The qNMR facility was used internally for the first time to assign a purity value to the [CCQM-K55.d](#) comparison material, folic acid.

### 2.3 Organic programme quality system

The actions to address minor non-conformities and observations raised as a result of an External Audit in 2014 were implemented and finalized in 2015. The Quality System that underpins the Organic Work Programme was reviewed during an Internal Audit in December 2015. No non-conformities were identified as a result of this latest Audit.

#### 2.4 Purity comparison CCQM-K55.d [Folic acid]

The characterization of the [CCQM-K55.d](#) candidate material (folic acid) was completed at the BIPM using both mass balance and qNMR methods. The comparison protocol and call for participation was circulated to the OAWG in July 2015. Seventeen NMIs and DIs have registered to participate in CCQM-K55.d and an additional six participants, including three authorized guest laboratories, have registered for the CCQM-P117.d parallel pilot study. The comparison samples were distributed to participants in September 2015 and the submission of results is scheduled for January 2016.

#### 2.5 Calibration solution comparison CCQM-K78 [Amino acids in aqueous solution]

Validation studies for methods for the assignment of the mass fraction content of amino acids in solution using LC-UV, LC-CAD, LC-MS/MS and IC were completed in 2015. These were required for the CCQM-K78 comparison that will be coordinated by the BIPM. The validation studies were successfully completed during a project undertaken at the BIPM in 2015 by a scientist on secondment from INMETRO, Brazil.

A candidate material consisting of a batch of 200 ampoules of a multi-component amino acid solution has been prepared and will be evaluated for suitability as the CCQM-K78 comparison material in 2016.

#### 2.6 Organic large molecule purity – Angiotensin I, Insulin, Heparin, Oxytocin and Calcitonin model studies

The development and validation of a range of analytical methods for the purity determination of the intact decapeptide angiotensin I (ANG I) and insulin (INS) was completed by the BIPM. The BIPM has successfully finalized the cross-validation of different approaches for the purity mass fraction value assignment for both model peptides ANG I, in collaboration with the NIST, and INS. The methods developed are to be used in the CCQM key comparison on peptide purity. One external publication describing the development and comparing mass spectrometric methods for the quantification of ANG I was published. External publications on the final results of both ANG I and INS are in preparation.

Ms P. Bros from the LNE joined the BIPM as visiting scientist, as part of her PhD study, to work on the method development for high resolution mass spectrometry coupled to liquid chromatography (LC-hrMS) for the purity determination of heparin, a key regulator of iron homeostasis and a promising clinical biomarker for brain iron deposition, which is implicated as a potential cause of Alzheimer's disease.

In collaboration with the NIM, China, high-purity oxytocin and calcitonin, both synthetic therapeutic peptides, have been produced, and will be used to investigate their suitability to serve as future key comparison materials as potential model systems for small peptides with disulfide bonds.

#### 2.7 Organic large molecule purity – Human C-peptide

The first CCQM key comparison on peptide purity ([CCQM-K115/P55.2](#)) coordinated by the BIPM in collaboration with the NIM, China, was launched. Dr M. Li from the NIM joined the BIPM as a visiting scientist to continue the work on method development and to study material characterization for the key comparison.

The assignment of the mass fraction content of high-purity C-peptide (hCP) has been accepted as the most appropriate choice for a first CCQM key comparison that will investigate competencies to perform peptide purity mass fraction assignment. hCP was chosen as a model system from which performance of other molecules can be inferred, whilst simultaneously focusing on a material directly relevant to existing CMC claims.

hCP is an important clinical and forensic analyte in its own right, for which accurate reference measurement systems are required. It is a chemically-synthesized linear peptide of known sequence, without cross-links, that contains 31 amino acids. It will directly support NMI services and certified reference materials (CRMs) which are currently provided by NMIs.

The impurity profile of the batch and the outcome of the homogeneity and stability studies have been found to be appropriate for the purposes of the comparison. The study samples were shipped to the participants and the key comparison and a parallel pilot study began in early December 2014.

The participants reported their assignments of the mass fraction content of the high-purity hCP prior to September 2015. Different methods were used by the participants. The PCAA analysis approach, requiring quantification of constituent amino acids following hydrolysis of the material and correction for amino acids originating from impurities, has been used by the majority of participants. Peptide related impurities have been determined mainly by LC-hrMS. The coordinating laboratories (BIPM and NIM) have used the mass balance approach employing a variety of analytical methods, such as LC-hrMS, GC-MS, KFT, TGA and IC. In addition, qNMR and CHN elemental analysis, both with correction for amino acids originating from impurities, have been used.

A first discussion of the results took place during the fall meeting of the new CCQM Working Group on Protein Analysis (PAWG) in October 2015.

### 3. **Activities related to the JCTLM (S. Maniguet and R.I. Wielgosz)**

Dr Wielgosz is the Executive Secretary of the Joint Committee for Traceability in Laboratory Medicine (JCTLM), the leader of its review team on Quality Systems and Implementation, and a member of the JCTLM Working Group for Education and Promotion on Traceability (WG-TEP). Dr Maniguet coordinates the development of the JCTLM Database, and is a member of the review team on Quality Systems and Implementation, and the Secretary of the JCTLM WG-TEP.

In February 2015, the WG1 Cycle 11 reference materials, and measurement methods, and WG2 Cycle 9 reference measurement laboratory services approved by the Executive Committee during its 13th Annual Meeting in December 2014 were published in the database.

As of December 2015 the JCTLM Database contained:

- 295 available certified reference materials that cover 11 categories of analytes. Of these reference materials, 33 are in List II, which includes reference materials that are value-assigned using internationally agreed protocols, and three are in List III, which covers reference materials with nominal properties;
- 176 reference measurement methods or procedures that represent about 80 different analytes in nine categories of analytes;
- 133 reference measurement services, delivered by fourteen reference laboratories and two NMIs in eight countries and which cover seven categories of analytes.

The WG1 Cycle 12 call for nominations of higher order reference materials and reference measurement methods or procedures, and the WG2 Cycle 10 call for nominations of reference measurement laboratory services were announced on the JCTLM website in February 2015, and email notifications were sent to about 400 potential contributors to the JCTLM. As of July 2015, 19 nominations for materials, six nominations for methods, and 27 nominations for services had been received and sent to the review teams for their evaluation.

The second issue of the JCTLM Database Newsletter was edited in collaboration with the ILAC Marketing Office, and distributed by email in March 2015 to the JCTLM contact list maintained at the BIPM. Future editions of the Newsletter will benefit from the contributions of the JCTLM TEP-WG.



A web-based form was made available on the database website from December 2014 to January 2015 to determine the profile of users of the database. A total of 85 responses were collected and evaluated, of these 34 % were from clinical laboratories, 20 % from IVD manufacturers, 20 % from Reference Laboratories, 8 % from NMIs/ DIs, 3 % from accreditation (regulatory) bodies, and the remainder from other organizations.

The database log-on analysis carried out over 2015 indicated that the number of visits to the website was 1400 on average per month.

The 14th and 15th meetings of the Executive Committee of the JCTLM were held at the BIPM on 25-26 June 2015, and on 3-4 December 2015, respectively. During its meeting in December, the Executive approved the revised text of the Declaration of Cooperation (DoC) between the BIPM, IFCC and ILAC, and its Appendices. The amendments made to the text followed the recommendation from the *ad hoc* Working Group on JCTLM Governance to open the Executive to more international organizations and bodies with a clear interest in promoting reliable, comparable and traceable measurements in clinical chemistry and laboratory medicine. It aims to create a new JCTLM Members Stakeholder category in addition to the National and Regional Members category, and to merge the WG1 (materials and methods) and WG2 (services) into a unique Database Working Group structured according to specific group of analytes.

The JCTLM Members' and Stakeholders' meeting held at the BIPM on 31 November and 1 December 2015 brought together 65 attendees from the *In Vitro* Diagnostic Industry, as well as from the clinical chemistry and laboratory medicine community. The meeting included five sessions: "Update on JCTLM activities", "JCTLM Member activities", "Developments in Traceability Requirements around the Globe", "Identifying future priorities in Traceability in Laboratory Medicine", and "New Challenges for Traceability in Laboratory Medicine".

The inaugural meeting of the JCTLM Working Group for Education and Promotion on Traceability, and the annual meeting of the JCTLM Working Group 1 and 2 were held at the BIPM on 2 December 2015. The new structure of the database WG was trialled during the review process in 2015, and the vice-Chairs of the WG together with the leaders of the review teams reported directly during the meeting on their recommendations resulting from their technical assessment of the material, method, and service nominations. The next step will be to revise the procedure documents of the JCTLM Quality Manuals to describe the process.

ISO TC 212 WG2 is continuing the revision of two normative standards of particular importance to the JCTLM processes, notably ISO 17511 and ISO 15195. The BIPM participates actively as a liaison organization to ISO TC 212.

#### 4. Publications

1. Viallon J., Moussay P., Idrees F., Wielgosz R.I. and Ross G., Comparison of Ozone Reference Standards of the OEH and the BIPM, April 2015, [Rapport BIPM-2015/02, 19 pp.](#)
2. Viallon J., Moussay P., Idrees F., Wielgosz R. and Zhou Z., Final report on ongoing key comparison BIPM.QM-K1, ozone at ambient level, comparison with NIM (July 2014), [Metrologia, 2015, 52, Tech. Suppl., 08012.](#)
3. Viallon J., Moussay P., Idrees F., Wielgosz R., Sanchez C. and Gomez P.M., Final report, ongoing key comparison BIPM.QM-K1, ozone at ambient level, comparison with ISCIII (December 2014), [Metrologia, 2015, 52, Tech. Suppl., 08014.](#)
4. Viallon J., Moussay P., Idrees F., Wielgosz R., Konopelko L. and Kustikov Y., Final report on ongoing key comparison BIPM.QM-K1, ozone at ambient level, comparison with VNIIM (June 2014), [Metrologia, 2015, 52, Tech. Suppl., 08010a.](#)

5. Viallon J., Lee S., Moussay P., Tworek K., Petersen M. and Wielgosz R.I., Accurate measurements of ozone absorption cross-sections in the Hartley band, [Atmos. Meas. Tech., 2015, 8, 1245-1257.](#)
6. Flores E., Viallon J., Choteau T., Moussay P., Wielgosz R.I., Kang N., Kim B.M., Zalewska E., van der Veen A.A. and Konopelko L., International comparison CCQM-K82: Methane in Air at Ambient level (1800-2200) nmol/mol, [Metrologia, 2015, 52, Tech. Suppl., 08001.](#)
7. Flores E., Rhoderick G.C., Viallon J., Moussay P., Choteau T., Gameson L., Guenther F.R. and Wielgosz R.I., Methane Standards Made in Whole and Synthetic Air Compared by Cavity Ring Down Spectroscopy and Gas Chromatography with Flame Ionization Detection for Atmospheric Monitoring Applications, [Anal. Chem., 2015, 87\(6\), 3272-3279.](#)
8. Stoppacher, N., Josephs, R.D., Daireaux, A., Choteau, T., Westwood, S., Wielgosz, R.I., Accurate quantification of impurities in pure peptide material – angiotensin I: Comparison of calibration requirements and method performance characteristics of liquid chromatography coupled to hybrid tandem mass spectrometry and linear ion trap high-resolution mass spectrometry, [Rapid Comm. Mass Spectrom., 2015, 29 \(18\), 1651-1660.](#)
9. Josephs R.D., Daireaux A., Choteau T., Westwood S., Wielgosz R.I., Normal phase-liquid chromatography-tandem mass spectrometry with atmospheric pressure photoionization for the purity assessment of 17 $\beta$ -estradiol, [Anal. Bioanal. Chem., 2015, 407\(11\), 3147-3157.](#)

## 5. Activities related to the work of Consultative Committees

The CCQM held its 21st meeting on 20-21 April 2015 at the BIPM. It was preceded by meetings of the CCQM Working Groups.

R.I. Wielgosz is the Executive Secretary of the CCQM and a member of the CCQM Strategic Planning Working Group (SPWG).

J. Viallon is a member of the CCQM Working Group on Gas Analysis (GAWG).

E. Flores is a member of the CCQM Working Group on Gas Analysis (GAWG).

S. Westwood is a member of the CCQM Working Group on Organic Analysis (OAWG).

R. Josephs is a member of the CCQM Working Group on Protein Analysis (formerly part of the CCQM Working Group on Bioanalysis (BAWG)), the CCQM Working Group on Organic Analysis (OAWG) and the *ad hoc* Steering Group on Microbial Measurements (MBSG).

S. Maniguet is a member of the CCQM Working Group on Key Comparisons and CMC Quality (KCWG).

## 6. Activities related to external organizations

R.I. Wielgosz is a BIPM representative to the International Union of Pure and Applied Chemistry, Interdivisional Committee on Terminology, Nomenclature and Symbols (IUPAC ICTNS), ISO TC 212, Clinical laboratory testing and *in vitro* diagnostic test systems, Working Group 2 on Reference Systems, and ISO TC 146 on Air Quality, and is a member of the editorial board of Accreditation and Quality Assurance. He is a member of the World Meteorological Organization (WMO)-BIPM Joint Liaison Group.

J. Viallon is the BIPM representative at ISO TC 146/SC 3 on Air Quality – Ambient Atmospheres.

S. Westwood is the chair of the IUPAC Project 2013-025-2-500: Methods for the SI Value Assignment of Purity of Organic Compounds, the BIPM liaison to both the ISO/REMCO and the REMCO/CASCO Joint Working Group 43 and a member of the World Anti-Doping Agency (WADA) Laboratory Expert Group.

R. Josephs is the BIPM representative to the Inter-Agency Meeting and the Codex Committee on Methods of Analysis and Sampling (CCMAS) of the Codex Alimentarius Commission.

## 7. Travel in 2015

R.I. Wielgosz to:

- Malta, 5-6 February, to present on BIPM activities at the EURAMET METCHEM meeting.
- Münster (Germany), 23-25 February, to give an invited lecture on the JCTLM at the European Winter Conference on Plasma Spectrochemistry (EWCPS).
- NIM (China), 2-6 March 2015, invited visit to the NIM laboratories and for mutual presentations of activities in chemistry metrology.
- College of American Pathologists, Chicago (USA), 7-9 April, to participate in ISO TC 212 WG2 meetings.
- Paris (France), 24 June, to give JCTLM lecture at the Euromedlab conference.
- Rotterdam (the Netherlands), 10-12 June 2015, to Chair a session on Metrology and Standardization at the GAS2015 conference.
- Boston (USA), 19-20 August, to give presentations on CCQM activities and the redefinition of the mole at the ACS conference.
- Max-Planck-Institute for Biogeochemistry, Jena (Germany), 26-27 August, to visit the Institute and to give an invited lecture.
- NIST, Gaithersburg (USA), 19-20 October 2015, to contribute to the CCQM PAWG/OAWG meetings.
- IRMM, Geel (Belgium), 10-13 November, for ISO TC 212 Plenary and WG2 meetings.
- UNIDO, Vienna (Austria), 17 December, to give a presentation on the Mycotoxin Metrology CB&KT programme.

J. Viallon to:

- OECD, Paris (France), 4 February 2015, to attend the Meeting of the Working Party on Manufactured Materials (WPMN).
- Rotterdam (the Netherlands), 10-12 June 2015, to attend the GAS2015 conference.
- NIM (China), 2-6 March 2015, invited visit to the NIM laboratories and for mutual presentations of activities in chemistry metrology.
- SIO, La Jolla, (USA), 13-17 September 2015, to attend the 18th WMO/IAEA Meeting on Carbon Dioxide, Other Greenhouse Gases, and Related Measurement Techniques (GGMT-2015).

E. Flores to:

- Rotterdam (the Netherlands), 10-12 June, to attend GAS 2015 and to present the lecture: Use of FTIR for accurate comparisons of gas standards.
- Max-Planck-Institute for Biogeochemistry, Jena (Germany), 26-27 August, to visit the Institute.
- CENAM, Queretaro (Mexico), 23 September, to give the lecture: Use of FTIR for accurate comparisons of gas standards.

- CENAM, Queretaro (Mexico), 23-25 September, theoretical and practical training course on FTIR and MALT 5.
- Universidad Nacional Autónoma de México (UNAM), Mexico City (Mexico), 28 September, to present the lecture: “*Comparaciones Internacionales de mezclas de gases primarios (CO<sub>2</sub>, CH<sub>4</sub> y HCHO), realizadas con las técnicas FTIR, CRDS y GC-FID en el BIPM (Bureau International des Poids et Measures)*”.

S. Westwood to:

- Beijing (China), 2-6 March, for discussions on the BIPM and NIM metrology in chemistry programmes and potential future collaborations.
- Pretoria (South Africa), 14-17 June, for the 38th meeting of the ISO-REMCO.
- Geneva (Switzerland), 9-11 July, for the second meeting of the REMCO/CASCO Joint Working Group 43.
- National Harbour (USA), 11-15 October, to attend the 14th BERM Conference and give an oral presentation on the BIPM qNMR programme;
- Gaithersberg (USA), 16-20 October, for the meeting hosted at the NIST of the IUPAC Organic Purity Technical Report and CCQM Organic Analysis Working Groups.
- Montreal (Canada), December, for a meeting of the WADA Laboratory Expert Group.

N. Stoppacher to:

- Frankfurt (Germany), 7-9 April, for training on the theory and application of NMR.

R. Josephs to:

- IAM, Budapest (Hungary), 20 February, to represent the BIPM at the Inter-Agency Meeting of the Codex Alimentarius Commission.
- Foresight Centre, Liverpool (UK), 7-8 July, to participate in the Ionmobility-Mass Spectrometry meeting and workshop of British Mass Spectrometry Society (BMSS).
- BERM14, National Harbour (USA), 11-15 October, to give a presentation at the 14th Biological and Environmental Reference Materials conference.
- IUPAC, NIST, Gaithersburg (USA) 16 October, to contribute to the IUPAC purity meeting.
- NIST, Gaithersburg (USA), 19-20 October, to contribute to the CCQM PAWG/OAWG meetings.
- USP, Rockville (USA), 2-4 November, to attend the therapeutic peptide workshop and expert panel of the US Pharmacopeia.

## 8. Visitors in 2015

- T. Saito (NMIJ), 9-20 February 2015, for discussions on the BIPM-NMIJ qNMR project.
- K. Chiba (NMIJ), 12-13 February 2015.
- O. Rabin and V. Ivanova (WADA), 19 March 2015, for discussions on BIPM WADA collaborations.
- S. Assonov (IAEA), 15 April 2015.
- D. Griffiths (University of Wollongong), 20-21 April 2015.
- M. Ramonet, O. Laurent and L. Rivier (LSCE), 5 May 2015.

- J. Wheeler (NIBSC), 21 May 2015.
- J. Norris (NIST), 6-10 July 2015.
- T. Jacksier and W. Weterings (Air Liquide), 8 July 2015.
- V. Delatour and S. Vaslin-Reiman (LNE), 28 July and 21 September 2015, for discussions on the BIPM-LNE secondment.
- T. Suematsu (JEOL) and A. Mairu (WAKO), 25 September 2015, to discuss the BIPM-NMIJ qNMR project.
- O. Rabin and V. Ivanova (WADA), 28 October 2015, to discuss planning for the BIPM-WADA joint symposium on Anti-Doping Analysis in 2016.
- D. Heikens (VSL), 2-4 November 2015.

## 9. Guest workers in 2015

- M. Li, NIM (China), 1 January to 31 October.
- B. Garrido, INMETRO (Brazil), 2 February to 29 May.
- T. Huang, NIM (China), 9 March to 4 September.
- P. Bros, LNE (France), since 1 July.
- I. Un, UME (Turkey), 1 September to 27 November.
- S. Maxwell, NIST (USA), since 1 September.