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Director: M. Stock

(1 January 2016 to 31 December 2016)

1. Electrical metrology

1.1 Electrical potential difference (voltage)

1.1.1 AC Josephson voltage standard (S. Solve, R. Chayramy)

The first on-site comparison using the new BIPM ac quantum voltage standard (provided by the NIST) was carried out at the NMIJ (Japan) in December 2015. This was the first direct comparison of arrays that use (NbN)-based and amorphous niobium silicon (Nb_xSi_{1-x}) Josephson junctions. At 10 V dc, a relative agreement of 5×10^{-12} was obtained. An attempt to perform an ac comparison at the NMIJ was unsuccessful because, at that time, the trigger signal used for the generation of the approximated sine wave could not be properly extracted from the bias source of the BIPM PJVS. This prevented a correct phase adjustment between the two PJVS signals from being carried out, which is a requirement to obtain meaningful results.

The bias electronics of the BIPM PJVS were successfully updated in April 2016 in collaboration with Dr Alain Rüfenacht (NIST) who visited the BIPM electricity laboratory for this purpose. During Dr Rüfenacht's visit, the new bias source was tested and a calibration of an ac sine waveform of 65 Hz with an amplitude of 1 V rms, produced by a multifunction calibrator, was carried out. The silicium-based generator was connected in series-opposition to the PJVS and the sampler was connected between the two positive poles of the sources. Once a phase adjustment was completed, the sampler measured the voltage difference between the two signals and the signal was reconstructed from these readings. The influence of different variables was investigated: the number of quantum steps for the approximated waveform; the width of the integration window of the null detector; and the time delay before the opening of the integration window.

A first pilot study comparing two quantum voltage stepwise approximated sine waves was carried out in September 2016 with CENAM (Mexico). A direct dc comparison of the two quantum standards was successfully performed at 10 V, achieving a relative agreement of a few 10^{-11}. Then, the two independent measurement setups were arranged to calibrate a 0.7 V and 7 V rms ac sinusoidal signal at 50 Hz, delivered by a commercial calibrator. This device served as a transfer standard to compare the two quantum voltage standards. The approximated sine wave was set to N=64 samples per period for both voltages. The relative difference between the two quantum standards was measured to 2×10^{-7} and 7×10^{-7} at 1 V and 10 V, respectively, with a relative Type A uncertainty of 3×10^{-7} for both voltages.

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

The Quantum voltage project at NIST provided the BIPM with two new programmable arrays: a 1.2 V array embedded on a "flexboard" chip carrier and a 2.5 V array embedded on the newly designed copper chip carrier.

The arrays were cooled down and a table representing the optimal biasing parameters, current and rf power, achieving the largest operation margins, was derived from the investigations performed on each device.

Two calibration campaigns were performed to fully characterize the gain and linearity of five precision multimeters which are operated on the watt balance to measure the current sent to the coil and the induced voltage produced by the coil.

1.1.3 Temperature sensitivity coefficient of Zener voltage standards (R. Chayramy, S. Solve)

In order to minimize the uncertainty achievable in the BIPM ongoing bilateral comparison (BIPM.EM-K11) of calibrations of Zener-based secondary voltage standards, it was decided to re-evaluate the temperature coefficients of the BIPM travelling standards involved. The corresponding relative voltage corrections ranged from 1 part in 10^9 to 75 parts in 10^9, which in extreme cases can be comparable to the Type A uncertainty in a comparison. It was therefore considered worthwhile to re-determine these coefficients, 15 years after their original determination was carried out between 1998 and 2002, particularly since no investigations have been performed on the possible evolution of these coefficients.

The non-infinite thermal gain of the temperature regulation of the enclosure in which the Zener diode is confined, and the imperfect compensation of the temperature coefficient of the Zener diode are responsible for the dependence of the output voltage on the surrounding temperature.

Temperature coefficients are determined by applying a bilinear regression of the voltage of the Zener being tested with respect to the resistance of the built-in thermistor and time (because Zeners exhibit a linear drift with time). To avoid accidental correlation between temperature and time, temperature variations are applied in uniform steps in up and down cycles. The range of temperature variations is typically between 18 °C and 28 °C. Following a temperature change, the time required to achieve temperature equilibrium in the enclosure is six to ten hours. A complete cycle requires several days.

A commercial thermo-regulated enclosure with 15 mK temperature stability, which could hold two Zeners at the same time, was used for the study. The 1 V outputs were measured against a very stable standard cell, while the 10 V measurements were carried out against a Fluke 732A Zener standard kept at a constant temperature.

The voltage differences between the outputs of the Zeners and the reference standards were measured using two nanovoltmeters. Since these measurements are purely relative and the voltage variations due to temperature or pressure changes are small, no detailed calibration of the nanovoltmeters was necessary. Nevertheless, periodic verifications were carried out against a JVS. An isolation transformer was used to decrease the coupling of mains-borne interference to the instrumentation.

It is important to note that the output voltages were corrected for atmospheric pressure changes using the sensitivity coefficients determined in 2002. The total uncertainty of the temperature coefficients was dominated by the Type A uncertainty of the least-squares regressions of the voltage with respect to time and temperature (expressed as the resistance of the internal thermistor).

The new thermal enclosure allowed the BIPM to achieve faster changes and better temperature stability when compared to those obtained during the previous determination. The uncertainty on the determination of the sensitivity coefficient has been divided by a factor of three and they are now of the same order of magnitude as the coefficients themselves.

For half of the Zeners tested, the temperature coefficients have not changed significantly. In other cases, the changes were up to several times the uncertainty of the measurements. The resistance of the thermistors measuring the temperature inside the Zeners, measured at a room temperature of 23 °C, has also changed in some cases, mainly towards higher values. Possible reasons for this are a change of the thermistor, or of the set point of the Zener internal temperature regulation. The observed changes correspond to changes in the oven temperature of between −0.05 °C and −0.38 °C.

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 2016, as required to maintain traceability for resistance calibration and comparison services and the link to capacitance. Routine measurements were made with the BIPM's existing GaAs devices, but a series of investigations on a commercial graphene sample were also conducted.

The graphene sample obtained from the manufacturer consisted of a 7 mm × 7 mm chip of SiC wafer with a graphene mono-layer grown by sublimation. On top of this, 10 Hall bars with varying geometries were patterned by lithography, including metallic contact pads. In this type of material, the density of free electrons due to intrinsic doping of the substrate is too high to make the quantum Hall plateau accessible at low magnetic fields (<10 T). A final layer of polymer is used on top of the graphene, which is capable of absorbing electrons to tune the carrier density. In tests conducted previously in 2015, in collaboration with MIKES, small amounts of ammonia gas were used to treat the polymer layer and effect this tuning. With the latest sample, an alternative technique of electrostatic discharge near the device, a method successfully demonstrated by the NPL in collaboration with Linköping University and Chalmers University (Sweden), was used.

Several of the Hall bars were tested and showed the expected quantized Hall resistance curves characteristic of mono-layer graphene. We were able to successfully use the polymer layer to control the carrier density, and thus to extend the quantized plateau down to magnetic fields as low as 2 T. However, in more detailed measurements, at these low fields the breakdown current (the maximum current for fully quantized operation) was found to be too small for use in precision resistance calibrations (<10 µA, compared to the 40 µA or 50 µA required). In addition, it was found that a large proportion of the metal-graphene contacts had too high a resistance (>10 Ω) for precision metrological use. We were able to perform precision measurements on some of the Hall bars, and successfully calibrate a 100 Ω resistor. The value thus obtained from the graphene reference was in agreement with the existing GaAs reference, within a relative uncertainty of $< 1 \times 10^{-8}$. However, these measurements were only possible with the graphene sample at fields >8 T and a temperature of 1.3 K.

In conclusion, it was possible to reproduce some of the results obtained by NMIs (particularly NPL and LNE) that have successfully shown the benefits of graphene for resistance metrology, but using a commercially available sample. However, it was not possible to obtain full quantization with the combination of higher temperature and lower magnetic field that was being sought (a target of 4 K and 5 T will allow significant simplification of the BIPM's QHR equipment, especially for on-site comparisons). The potential impact of graphene QHR is clear, but the sample technology is still immature and is not yet ready for reliable use in resistance metrology. It is hoped that more devices will be tested over the next few years, and it is highly likely that graphene will soon replace GaAs as the material of choice for QHR references, bringing a significant reduction in complexity and cost for this primary standard.

1.2.2 Calculable capacitor (P. Gournay, N. Fletcher, B. Rolland)

Characterization measurements of the calculable capacitor carried out in 2015 demonstrated that the limited alignment accuracy of the electrode bars following the initial assembly of the device was the most important source of systematic error and limited the uncertainty on the determination of the von Klitzing constant to about 2 parts in 10^7. A decision was taken to dismantle the capacitor and realign the bars, and a new alignment tool has been constructed.

During 2016, the calculable capacitor was dismantled and its bars realigned. Following its disassembly, the capacitor was relocated to a newly renovated laboratory with a floor consisting of a massive concrete block. This concrete block provides better vibration isolation for the device, an issue that had been detected during the first series of measurements.

During the dismantling process, a non-negligible amount of dust was found to have been deposited on the surface of the electrodes. It was therefore decided that before the capacitor was reassembled in the new room, a removable clean room enclosure should be built around it so that precision alignment of the electrode bars could be carried out in a clean environment.

Following a delicate cleaning procedure, the electrode bars have since been reassembled and aligned. Alignment was performed using a part of the set of the capacitive probes used for the first alignment, plus the new probe. The latter was used mainly to measure the residual skew angle of the bar assembly and also to carry out redundant measurements of the diagonal distance between bars in order to verify that they are consistent with those obtained with another probe dedicated to this measurement (with a better sensitivity). The electrode bars have been aligned such that the contributions of the residual misalignments are now close to 2 parts in 10^9, compared to 2 parts in 10^7 previously.

The next step in the reassembly of the calculable capacitor will be the alignment of the top and bottom guard electrodes and the interferometer laser beam with the axis of the set of four main bars.

The new stabilized laser source, which was built in 2015, was made more compact and installed on a smaller optical breadboard during 2016. This source, which is currently located in the laser building (laser light is provided through a 25 m optical fibre between this building and the calculable capacitor), could be then used in the new laboratory when needed.

Completion of the calculable capacitor reassembly is planned for early 2017. As the measuring chain from the calculable capacitor to the quantum Hall effect is already operational, a new series of measurements (characterization of the calculable capacitor and final measurement of R_K) could quickly follow depending on the time available.

1.2.3 Maintenance of a reference of capacitance (P. Gournay, R. Chayramy, N. Darpentigny[1], N. Fletcher, B. Rolland)

Every year, the group of four 10 pF standard capacitors used to maintain the farad at the BIPM is calibrated twice. This was carried out in January and July 2016. Calibration is performed against the dc-QHR through a multi-frequency quadrature bridge. For the second consecutive year, the calibration was carried out at both 1027 Hz and 1541 Hz, as it has been observed that the 10 pF reference used for calibration and comparison services experiences a small drift.

Maintenance on the various measuring chain bridges used to establish traceability of the farad to the dc-QHR was performed to maintain optimum operating conditions and to provide the best uncertainties for services to Member States. In particular, the two-terminal pair bridge has been equipped with a new injection device and pre-amplifier, both of which were designed and built at the BIPM. The computer part of the bridge (hardware and software) is currently being upgraded.

The reference group of capacitors were used in 2016 for calibrations for seven NMIs (see section 1.4) and three bilateral comparisons BIPM.EM-K14.a and BIPM.EM.K14.b (see section 1.3.2).

The supplementary comparison of capacitance and capacitance ratio, EURAMET.EM-S31, has been completed in 2016. Two circulations of the travelling capacitance standards and one circulation of ac standard resistors were necessary due to unsatisfactory results at the end of the first capacitance circulation, mainly due to systematic errors in the measuring bridges of several participants. Later corrections proposed by the participants have led to satisfactory results for both capacitor circulations. Draft A of this work has been approved by the participants, and the draft B stage is in progress.

[1] Intern from Centrale Supélec, Gif sur Yvette, France, from 4 July 2016 to 6 January 2017

1.3 Comparisons of electrical standards

1.3.1 CCEM comparison of capacitance calibrations, CCEM-K4.2017 (P. Gournay, M. Stock)

The CCEM decided, at its meeting in March 2015, that the comparison CCEM-K4, last carried out between 1996 and 1999, be repeated. The BIPM was designated as coordinator of the new comparison and prepared a comparison protocol during 2015. This protocol was submitted to the support group for discussion and then to the declared participants in early 2016.

In this comparison, the measurand is a 'two-terminal pair 10 pF capacitance' measured at a frequency of 1592 Hz and a voltage of 100 V (rms). An optional 100 pF capacitance measurement at the same frequency and 10 V is also possible. The comparison scheme adopted by the CCEM is a large-scale 'star-comparison' with a large number of bilateral comparisons being carried out simultaneously, piloted by the BIPM. In the original version of the protocol, the comparison measurements were scheduled to take place between August 2016 and January 2017.

Only two points were discussed during the review process of the protocol: (1) the starting date of the comparison and (2) the mandatory operating frequency. Regarding the first point, it appeared that most of the participating NMIs would not be ready to start the comparison at the date originally planned in the protocol (August 2016). A consensus has been reached to postpone the start date to late February 2017. As the cumulative measurement and transport time is planned as six months, the last comparison measurements should be completed in September 2017.

Concerning the second point, one participant requested a change to the mandatory working frequency from 1592 Hz to 1233 Hz. This frequency may be more appropriate for NMIs realizing the traceability of the farad from the quantized Hall resistance through a mono-frequency quadrature bridge, using transfer resistors and capacitors of nominal values equal to $R_K/2$ and 10 nF, respectively. A consensus was reached by adding 1233 Hz as an optional working frequency while keeping 1592 Hz as the mandatory frequency.

The protocol was approved in July 2016 by the eight participants: LNE, METAS, NIM, NIST, NMIA, NPL, PTB, VNIIM and the BIPM. The LNE has subsequently withdrawn from the comparison.

Assuming that the protocol schedule is met, the first version of the Draft A report could be sent to participants for review by the end of 2017.

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

An on-site comparison of Josephson Voltage Standards (BIPM.EM-K10) was performed with JV (Norway) in June 2016. Satisfactory results were not achieved due to interferences between the two JVSs that were so strong; stability of the quantized voltages of both arrays was lost.

Two bilateral Zener comparisons (BIPM.EM-K11) were carried out with DEFNAT (Tunisia) and with NSAI NML (Ireland). Satisfactory results for both comparisons were achieved with a relative voltage difference of 3.8×10^{-8} and 2.2×10^{-8} at 10 V, respectively.

An on-site comparison of QHR systems (BIPM.EM-K12) was organized with METAS (Switzerland). The BIPM developed thermostated resistors (1 Ω, 100 Ω and 10 kΩ) for use in this comparison. These were transported to METAS at the beginning of September 2016, several weeks before the planned comparison date, to allow stabilization. METAS subsequently encountered problems with their QHR reference in preparation for the measurements, and the comparison was eventually cancelled.

For BIPM.EM-K13, the ongoing bilateral comparison of 1 Ω and 10 kΩ standards, the results of a comparison with the CMI (Czech Republic) completed at the end of 2015 were analyzed and reported. For the first time a correction term was included for the variations of the value of 1 Ω standards with the rate of current polarity reversals. Extra characterizations of the 1 Ω travelling standards carried out at CMI confirmed the effects previously studied in a collaboration between the BIPM and PTB (assumed to be due to the Peltier effect in the junctions of the resistor construction). A correction applied on the basis of these characterizations allowed a better comparison of the 1 Ω standards under equivalent measurement conditions. This Peltier effect increasingly appears to be a limiting factor in the results achievable with 1 Ω standards; the comparison at 10 kΩ is free from this limitation. A comparison at 1 Ω and 10 kΩ was started with SMD (Belgium), with the measurements expected to be completed in early 2017.

In 2016, two bilateral comparisons of 10 pF and 100 pF capacitance standards (BIPM.EM-K14) at 1000 Hz and 1592 Hz were carried out with NIS (Egypt) and NMISA (South Africa). A third comparison at 10 pF and 1000 Hz was carried out with NSAI-NML (Ireland). The comparison reports, draft B, were finalized and are being reviewed by the CCEM chairperson.

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

During January to December 2016 the following standards were calibrated in the electricity laboratories:

1 Ω, 100 Ω or 10 kΩ resistors were calibrated for: UTE (Uruguay), DFM (Denmark), NIS (Egypt), NMIM (Malaysia), INM (Colombia), IPQ (Portugal), GUM (Poland), DMDM (Serbia), MKEH (Hungary), NMC (Singapore). In total 35 certificates were issued for ten NMIs.

1 pF, 10 pF and 100 pF capacitors were calibrated for: NMC (Singapore), BIM (Bulgaria), MIKES (Finland), EIM (Greece), INMETRO (Brazil), IPQ (Portugal), NMIM (Malaysia). In total 21 certificates and one study note were issued for seven NMIs.

Three Zener voltage standards were calibrated for INM (Colombia) and SMD (Belgium).

2. Mass Metrology

2.1 Measurement services in Mass

2.1.1 CCM Pilot Study of future realizations of the kilogram (P. Barat, M. Stock)

One of the essential activities on the joint CCM-CCU Roadmap towards the redefinition of the kilogram is the CCM Pilot Study of future realizations of the kilogram. The redefinition of the kilogram, based on the value of the Planck constant will lead to the fundamentally new situation in mass metrology, that in principle any National Metrology Institute (NMI) will be able to realize the kilogram. One of the objectives of the CCM Pilot Study is to quantify the consistency of mass calibrations carried out with different experiments which will be used, in the future, to realize the kilogram. The study will also test the continuity between the kilogram according to its present definition (as the mass of the International Prototype of the Kilogram, IPK) and the future definition. The experience gained during the Pilot Study will allow optimization of the technical protocol for a future key comparison of kilogram realizations.

The BIPM has been chosen as the pilot laboratory for this comparison. All NMIs working on primary methods have been invited to participate in the Pilot Study, on the condition that they will realize the kilogram with a relative uncertainty not larger than 2 parts in 10^7, equivalent to 200 µg at the 1 kg level. Five NMIs participated in the comparison: the LNE (France), the NIST (USA), the NRC (Canada), the NMIJ (Japan) and the PTB (Germany). The LNE, NIST and NRC used Kibble balances, the NMIJ and the PTB used the ^{28}Si-spheres AVO28-S5c and AVO28-S8c from the International Avogadro Coordination as the basis for their calibrations.

The Pilot Study used two independent sets of 1 kg travelling standards, provided by each of the participants. Set 1 consisted of one Pt-Ir standard and optionally of a second 1 kg standard of the participant's choice. These standards had to be calibrated under vacuum as directly as possible with respect to the primary method.

The masses of the standards of Set 1 had to be calculated by all participants using the same value of the Planck constant, for which the value from the 2014 CODATA fundamental constants adjustment had been chosen.

The second set of travelling standards consisted of two 1 kg stainless steel standards which had to be calibrated in air, traceable to the primary method. This required transferring the primary mass standard from vacuum into air by making a correction for surface sorption and applying any necessary buoyancy correction. The aim was to assess the equivalence of the mass scale when unilaterally disseminated from individual realization experiments.

All participants sent their travelling standards to the BIPM during March and April 2016, together with their calibration results. During May to July 2016 the mass standards were compared at the BIPM with each other and with two BIPM working standards: the standards of Set 1 under vacuum and those of Set 2 in air. The masses of the BIPM working standards were known in terms of the mass of the IPK. For each travelling standard, the difference between its mass determined by the participant and the BIPM was calculated. This allowed the BIPM working standards to serve as a stable reference to compare the calibration results of the participants. The final results were presented as differences between each participant's results and the key comparison reference value, calculated as the weighted mean of all five participants' results. The Draft A report was sent out to participants in December 2016.

Excellent agreement was found for both sets of standards between the four NMIs with the smallest uncertainties. The result with the largest uncertainty was somewhat offset, but is still in agreement with the others, at the level of two standard deviations. The uncertainty of the weighted mean of all five results was about 10 µg for the calibrations in air and under vacuum. The weighted mean of the five kilogram realizations was in agreement with the as-maintained BIPM mass unit, traceable to the IPK measured in 2014. The comparison in terms of mass calibrations was consistent with a comparison in terms of a determination of the Planck constant, which is a strong indication that the comparison procedure itself, including the shipment of the standards, did not significantly influence the results of the Pilot Study.

2.1.2 Calibration of 1 kg Pt-Ir prototypes and stainless steel standards (P. Barat)

Following the new procedure for the use of the BIPM working standards, which was adopted after the Extraordinary Calibrations using the IPK, the six working standards for current use were recalibrated in March 2016 with respect to the three BIPM working standards for limited use. These had not been used since March 2015.

Due to the work involved in the Pilot Study, no regular mass calibrations for NMIs were carried out in the first half of 2016. During September and October calibrations were made for Pt-Ir prototypes of the PTB (Germany) and of SCL (Hong Kong (China)) and for stainless steel standards of CESMEC (Chile), NMCI (Iran), MIRS (Slovenia) and VSL (the Netherlands). The new Pt-Ir prototype delivered to NIM (China) in December 2016 was also calibrated.

2.1.3 Volume calibrations of mass standards (D. Bautista)

The density of two samples from the Pt-Ir ingot received in 2016 has been determined as a quality control, leading to the acceptance of this material. Three new prototypes (n° 110 for the NIM, n° 111 for the KRISS and n° 112, which is not yet attributed) have been manufactured from this ingot and have had their density calibrated.

The density determination of a new stack of disks in stainless steel has been carried out. They will be used as a sorption artefact by the BIPM.

New software for the use of silicon density standards and FC40 liquid is now complete. The software has been designed to carry out the whole process of hydrostatic weighing on one single interface. In addition, it allows the automatic centering of the weights, direct visualization of the weights and exchanger position, and direct reading of every environmental parameter. Test measurements have been successfully performed for weighing in air with an automatic determination of the comparator sensitivity.

Mr Damien Bautista was seconded to the NPL (UK) in October 2016 for knowledge transfer by the NPL on the use of solid density standards, FC40 as transfer liquid, and the density measurement of silicon spheres. During his secondment Mr Bautista was also trained in the use of the M_one mass comparator for weighings in air and under vacuum. The BIPM thanks Mr James Berry and Dr Stuart Davidson of the NPL for their contribution to the success of this secondment.

2.1.4 Pressure calibrations (F. Idrees)

At the end of 2015, the internal calibration service for pressure gauges around the atmospheric pressure range was stopped temporarily because the piston-cylinder assembly of the pressure balance became stuck. In early 2016 a careful intervention on the assembly allowed both parts to be separated without damage to the polished surfaces. The calibration service has been resumed and two calibration campaigns have been carried out.

2.1.5 Surface investigations on Si-spheres (D. Bautista, P. Barat, M. Stock)

Within the framework of the Avogadro project, the mass of the ^{28}Si-enriched spheres AVO28-S5 and AVO28-S8 had been measured by the PTB, BIPM and NMIJ, in this order between November 2013 and November 2014. Following the measurements carried out by the BIPM in February and March 2014, the spheres were sent to the NMIJ. On arrival, Dr Shigeki Mizushima inspected the surface of the spheres using optical microscopy and identified damage in the form of groups of regularly arranged dots. Initially, the BIPM was suspected to be the origin of this deterioration.

The spheres AVO28-S5 and AVO28-S8 had been weighed at the BIPM using the CCL 1007 mass comparator. Following the NMIJ's findings, a study was carried out, which consisted of scanning an annular zone of three silicon spheres belonging to the BIPM using optical microscopy, before manipulation and after measuring them in the CCL 1007. The measurement sequence was the same as that used for AVO28-S5 and AVO28-S8. During these measurements, the annular zone that had been inspected previously was in contact with the support pins. No additional surface damage has been detected by microscopy after weighing in the CCL 1007.

Further observations have been carried out on the complete surface of the three spheres. Several 'clouds' of dots of about 0.5 mm in diameter have been detected, some of which had already been observed before the tests in the CCL 1007. These defects show a characteristic structure, which is very similar to those observed by the NMIJ on the Avogadro spheres, indicating a possible common origin. The computer reconstruction of the sphere surfaces further revealed that the 'clouds' were arranged in straight lines and with a relatively constant distance of around 4 mm. Hypothetically, such structures could be caused by the rotation of a wheel. These results were presented to a meeting of the members of the Avogadro Coordination, during which one of the participants confirmed that they had seen similar structures on Si-spheres which had never been at the BIPM. The exact origin of the damage is currently unknown, but according to the results of the study described above it did not occur at the BIPM.

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

A new 1 kg prototype was delivered to the NIM (China) in December 2016. A prototype for the KRISS (Republic of Korea) is being fabricated. A stack of eight Pt-Ir disks and 21 Pt-Ir spacers has been fabricated for the NPL (UK) from a Pt-Ir sample provided by the NPL.

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

In 2016 the BIPM ensemble of mass standards reached a crucial step whereby the first standards have been placed inside their definitive containers with their characteristic environments. In particular, the argon, nitrogen and air networks are fully operational. Only the vacuum network remains to be completed.

All the standards from the ensemble have participated in the comparison of future realizations of the kilogram (see 2.1.1) carried out in 2016. This was a successful rehearsal for the future BIPM key comparison of mass standards. During this key comparison the standards of the ensemble will be calibrated with respect to travelling standards, which are traceable to the primary realizations.

The first mass measurements have been carried out for the three operational networks, with the standards kept under their storage conditions while being weighed.

Results from the 2014 extraordinary calibrations using the IPK evidenced the importance of establishing a hierarchy among the standards composing the ensemble (as has already been done for the Pt-Ir working standards in 2015). Establishing a hierarchy means that the frequency of measurement of a standard diminishes as the standard belongs to a higher level within the hierarchy. This fact had not been foreseen in the initial concept for the ensemble. At present, the standards of the ensemble are organized in a two-level hierarchy with weighing frequencies which are, respectively, one weighing every 5 years, and one every year.

2.4 Watt balance (H. Fang, F. Bielsa, A. Kiss, Y. Gao[2], S. Li[3], R. Chayramy, A. Dupire, B. Rolland, K. Shahvar[4], S. Solve, M. Stock)

The BIPM is continuing to develop a Kibble balance, also known as a watt balance, as a means for the practical realization of the expected new definition of the kilogram in terms of the Planck constant. An improved version of the apparatus was assembled, with the best alignment to date. As a consequence, the relative uncertainty on the Planck constant determination was reduced from 5×10^{-5} to 3×10^{-6}. The uncertainty is now dominated by the residual misalignment of the electromagnetic force due to the limited mechanical stability of the suspension. The design of a refined suspension, which should allow further reduction of the type B uncertainty, has been completed.

The control and measurement programs have been upgraded using field programmable gate arrays (FPGAs). A new data acquisition and synchronization scheme, including the use of time interval analyzers (TIAs) has been set up. The new interferometer has been integrated and aligned inside the apparatus. The electromagnetic force exerted on the current-carrying coil has been more accurately aligned by adjusting the inclination and centering of the coil with respect to the pre-aligned magnetic field, to avoid parasitic forces and torques. Both types of misalignment can be detected by the presence of linear and angular motion of the coil when a current is injected into it. With the present suspension, the two alignments are partially correlated and require a large number of iterative adjustments due to the limited mechanical stability and alignment sensitivity. Although a significant improvement has been achieved when compared to previous measurements, the coil alignment was still not optimal with a residual inclination of about 2 mrad and a decentering of about 1 mm. The design of a more stable and easily adjustable suspension has been completed. The correlation between the coil inclination and decentering is significantly reduced, facilitating the alignment procedure and minimizing residual coil misalignment.

A small set of complete watt balance measurements in air was carried out in June 2016 using a 100 g tungsten mass provided by the NPL (UK). The relative standard deviation of the mean voltage-to-velocity ratio for ten vertical coil displacements was about 6×10^{-6}. The integration time for each measurement was 200 ms.

[2] On secondment from NIM (China) from 4 January to 3 June.

[3] Research Fellow since 15 September

[4] Intern from Institut Universitaire de Technologie d'Evry from 18 April to 24 June

The signal-to-noise ratio (S/N) was improved by a factor of about five due to the much reduced non-linearity of the new interferometer. The S/N ratio of the force-to-current ratio, deduced from one current polarity reversal, was dominated by the noise on the force signal with a relative standard deviation of several parts in 10^5. Each series of complete measurements typically consisted of a repetition of ten current polarity reversals. The Planck constant was determined with a relative uncertainty of about 3×10^{-6}. This uncertainty was dominated by the type B contribution from the non-optimized coil alignment. The second largest contribution came from the type A uncertainty on the force measurement.

Preparations for a further refined apparatus operating under vacuum are underway. The vacuum operation of the present apparatus has already been investigated. The whole set-up has worked as designed, except for a change in the magnetic circuit position, caused by the large force exerted upwards on the support of the magnet as a consequence of the differential pressure between the outside and the inside of the vacuum chamber. This force slightly lifted the 150 kg magnetic circuit, which resulted in a change in its position. As a result, a more rigid and adjustable mechanical support has been designed and fabricated. The new support is based on a highly-stable hexapod structure which includes an independent adjustment system, used during the alignment phase. The whole apparatus has been dismantled and the magnetic circuit has been re-installed on the new support. The magnetic plane of the circuit has been re-aligned using a rotating Hall probe. The geometry of the circuit has been re-characterized using capacitive sensors. The results are consistent with those obtained in 2015 within the measurement uncertainty. The magnetic plane at the central position in the gap is aligned horizontally within 20 µrad, with an uncertainty of 25 µrad.

A stiffer mass loading and exchange system has been designed to allow manipulation of mass standards up to 1 kg. This has the advantage of reducing the relative uncertainties contributed by the residual misalignment and by the noise on the force signal by a factor of about ten. A study to investigate ways of reducing the type A uncertainty on the force measurement was conducted. Using long integration times of the force signal acquisition and correspondingly larger sample times for the interferometric signal would not resolve the problem due to aliasing. A promising solution may be to add an analogue low-pass filter at the output of the measurement circuit and to sample the force signal at a high frequency before applying a second-order low-pass digital filter.

A bifilar coil based on a ceramic former has been wound. A pair of glued 0.2 mm copper wires was used for the coil winding. The coil is composed of 30 layers of about 40 turns each. The detection part of the interferometric system is being improved with the objective of further increasing the S/N ratio of the velocity determination. The data acquisition and synchronization scheme is also being adapted to suppress the dead time between two consecutive measurements. Moreover, the new scheme will allow the integration time of measurements to be selected as needed without any modification to the hardware or software.

For electrical measurements, two boxes of low electromotive force switches were built and tested. These will facilitate the calibration of the standard resistor for the current measurement as well as the induced voltage measurement, alternating between the two windings of the bifilar coil. The development of two Josephson voltage standards (JVSs) continued, one for the voltage and one for the current measurement. To improve the flexibility of the JVS used for the current measurement, a prototype four-channel bias source has been developed and successfully tested with the JVS. Unfortunately, the JVS array carrier (NIST flexboard) was damaged following too many temperature cycles between liquid He temperature and laboratory temperature. A new 2.5 V SNS programmable array, based on a new board design, was provided by the NIST in April 2016. The control program has been refined to avoid the high transient currents during changes of the bias currents, which can lead to the trapping of magnetic flux. The definitive bias source with sixteen channels is being fabricated. The first four channels have been successfully tested with the new JVS array.

In parallel, studies were conducted to better understand and assess any potential perturbing effects on the experiment. A finite element analysis of the position-dependence of the force on a current-carrying coil inside the magnetic circuit, due to the position-dependence of the self-inductance was carried out. The analysis shows that this reluctance force can explain the dependency of the voltage-to-velocity ratio on the current and its polarity and on the vertical position in the gap as experimentally observed with the presently open circuit.

The simulation also suggests a vertically-shifted central measurement position, at which the reluctance force disappears. The self-attraction effect due to the watt balance apparatus on the local gravitational acceleration was evaluated. It was modelled by treating the gravitational field as an electrical field in an electrostatic finite element analysis. The result yields a total correction of (4.7 ± 0.5) µGal at the central trajectory for a 1 kg Pt-Ir mass standard. Half of the correction is contributed by the magnetic circuit. The model was experimentally checked by measuring the gravity change due to the displacement of a massive body of 200 kg. The vertical gradient of the self-attraction field has also been analyzed. This information, together with the results of a previous gravity mapping in the watt balance room, led to a determination of the local gravitational acceleration along the mass movement trajectory with a relative uncertainty of 4.6 µGal.

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 (WGs).

N. Fletcher is a member of the CCEM Working Group on Proposed Modification to the SI (WGSI).

H. Fang is the Executive Secretary of the Consultative Committee for Mass and Related Quantities (CCM). She is a member of several working groups and task groups (TGs) of the CCM.

E. de Mirandés is the Executive Secretary of the Consultative Committee for Units (CCU) and the CIPM Task Group for the Promotion of the SI.

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 were members of the Technical Committee of the CPEM 2016.

P. Gournay represents the BIPM on the Organizing Committee and the Scientific Committee of the International Congress of Metrology, CIM 2017, to be held in Paris.

The BIPM is an external collaborator on the EMRP project "Quantum Resistance based on Graphene (GraphOhm, 2013-2016)" and the EMPIR project "Towards the propagation of ac quantum voltage standards" (ACQ-PRO, 2015 to 2019).

M. Stock acts as the BIPM liaison with the International Avogadro Coordination project, the EURAMET Technical Committee for Mass and Related Quantities (TC-M) and the Technical Committee for Electricity and Magnetism (TC-EM).

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

5. Publications

1. M. Stock, R. Davis, F. Arias, J.M. los Arcos, M. Milton, "News from the BIPM laboratories – 2015", *Metrologia*, 2016, **53**, 103-107.
2. E. de Mirandés, P. Barat, M. Stock, M. Milton, "Calibration campaign against the international prototype of the kilogram in anticipation of the redefinition of the kilogram, part II: evolution of the BIPM as-maintained mass unit from the 3rd Periodic Verification to 2014", *Metrologia*, 2016, **53**, 1204-1214.
3. R. Davis, P. Barat, M. Stock, "A brief history of the unit of mass: continuity of successive definitions of the kilogram", *Metrologia*, 2016, **53**, A12-A18.

4. P. Richard, H. Fang and R. Davis "Foundation for the redefinition of the kilogram" *Metrologia*, 2016, **53**, A6-A11.

5. Gournay, N. Fletcher, L. Robertsson and M. Stock, "Progress Report on Measuring the von Klitzing Constant at the BIPM", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540713.

6. P. Gournay, N. Fletcher, and M. Stock, "A New CCEM-K4 Comparison of Capacitance at 10 pF", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540687.

7. N. Fletcher, P. Gournay, B. Rolland, M. Götz, S. Novikov, N. Lebedeva, and A. Satrapinski, "Experimental Study of Carrier Density Tuning in Epitaxial Graphene QHR Devices", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540515.

8. A.Satrapinski, M. Götz, E. Pesel, N. Fletcher, P. Gournay, and B. Rolland, "Testing the New Generation of Low-Frequency Current Comparators", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540594.

9. H. Fang, F. Bielsa, A. Kiss, T. Lavergne, Y.F. Lu, L. Robertsson, E. de Mirandés, S. Solve, M. Stock, "Progress on the BIPM watt balance", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540540.

10. F. Bielsa, L. Robertsson, T. Lavergne, A. Kiss, H. Fang, M. Stock, "The new interferometer of the BIPM watt balance", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540576.

11. S. Solve, R. Chayramy, "Temperature sensitivity coefficients of the BIPM secondary voltage standards", Digest *CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540702.

12. S. Solve, R. Chayramy, M. Maruyama, C. Urano, N. Kaneko, "A direct DC 10 V comparison between the NMIJ and the BIPM programmable Josephson voltage standards", *Digest CPEM 2016, 2016 Conference on Precision Electromagnetic Measurements*, DOI: 10.1109/CPEM.2016.7540719.

Comparison Reports

1. Solve S., Chayramy R., Ben Salah B., Mallat A., Abene L., Stock M., "Bilateral comparison of 1 V and 10 V standards between the DEFNAT (Tunisia) and the BIPM February to March 2016 (part of the ongoing BIPM key comparison BIPM.EM-K11.a and b)", *Metrologia*, 2016, **53**, Tech. Suppl., 01004.

2. Solve S., Chayramy R., Power O., Stock M., "Bilateral comparison of 10 V standards between the NSAI - NML (Ireland) and the BIPM, February 2016 (part of the ongoing BIPM key comparison BIPM.EM-K11.b)", *Metrologia*, **2016**, 53, Tech. Suppl., 01007.

3. Solve S., Chayramy R., Stock M., Pantelic-Babic J., Sofranac Z., Cincar Vujovic T., "Comparison of the Josephson Voltage Standards of the DMDM and the BIPM (part of the ongoing BIPM key comparison BIPM.EM-K10.b)", *Metrologia*, 2016, **53**, Tech. Suppl., 01005.

4. Solve S., Chayramy R., Stock M., Pimsut S., Rujirat N., "Comparison of the Josephson Voltage Standards of the NIMT and the BIPM (part of the ongoing BIPM key comparison BIPM.EM-K10.b)", *Metrologia*, 2016, **53**, Tech. Suppl., 01006.

5. A. Quiroga1, A. Bermudez, F. Kornblit, F. Garcia, H. Fang, J. Caceres, L. M. Peña, O. Ramos, A. Viaggio and C. Santo "Determination of the magnetic susceptibility and the magentic polarization of weights by means of the susceptometer method" *Metrologia*, 2016, **53**, Tech. Suppl., 07012.

6. Travel in 2016 (conferences, lectures and presentations, visits)

M. Stock to:

- Budva (Montenegro), 10-12 May, to attend the EURAMET TC-M meeting.
- Ottawa (Canada), 8-16 July, to attend the CPEM conference, and for meetings of the CCEM and CCM working groups, the CODATA TGFC and the CPEM Executive Committee.
- BEV, Vienna (Austria), 13-14 October, to attend the EURAMET TC-EM meeting.
- LNE, Trappes (France), 20 October, for a meeting of the scientific council for the LNE watt balance.

S. Solve and R. Chayramy to:

- JUSTERVESENET, Kjeller (Norway), 7-14 June, to carry out a direct on-site Josephson comparison.
- CENAM, Queretaro (Mexico), 13-19 September, to carry out a pilot comparison of ac voltage measurements with the new transportable BIPM quantum voltage standard.

S. Solve to:

- Ottawa (Canada), 10-15 July, to attend the CPEM conference, to give two oral presentations and to attend the meetings of the CCEM and EURAMET working groups, EMPIR ACQ-PRO ("Towards the propagation of ac quantum voltage standards").
- NIST, Boulder (USA), 18-22 July, to attend the 2016 NIST PJVS Workshop organized by the NIST Quantum Voltage Project.
- CENAM, Queretaro (Mexico), 20-23 September to perform a peer review of the voltage calibration laboratory and to give a lecture to the *2016 Symposio de Metrologia*.

F. Bielsa and H. Fang to:

- Ottawa (Canada), 8-15 June, to attend the CPEM conference, and for meetings of the CCEM and CCM working groups.

P. Gournay and N. Fletcher to:

- Ottawa (Canada), 8-15 June, to attend the CPEM conference.

P. Gournay and B. Rolland to:

- METAS (Switzerland), 6 to 8 December 2017, for test measurements within the framework of the ongoing comparison BIPM.EM-K12.

P. Barat and D. Bautista to:

- PTB, Braunschweig (Germany), 22-23 June, to attend the Si-kg Workshop.

D. Bautista to:

- NPL, Teddington (UK), 3-30 October, for a secondment on knowledge exchange and training in the field of mass and density determination.

7. Visitors in 2016

Electricity laboratories

- Dr Sophie Djorjevic, LNE, 20 January to discuss cryocooler setups to be operated on Josephson Voltage Standards.
- Dr Alain Rüfenacht, NIST, 11-15 April to update the electronics of the new BIPM-PJVS.
- Delegation from QSTA, Hunan Province (China), led by the Director General Mr Biao Quyang to visit the watt balance and the electricity laboratories on 15 April.
- Dr John Pratt (NIST) to visit the calculable capacitor on 25 April.
- John Wilson (National Laboratory Ass. of South Africa), to visit the electricity laboratories on 30 June.
- Dr Rasha Sayed M. Ali (NIS, Egypt) to visit the electricity laboratories, 27-29 July.
- Group of NMI directors, during the NMIs Director Meeting held on 24 October.
- Group of CIPM members, during the CIPM meeting held on 26 October.
- Group of 18 attendees of the CBKT course "Leaders of tomorrow" to visit the electricity laboratories on 8 November.
- Mr Peter Scheibenreiter (BEV, Austria) to visit the electricity laboratories, 14 December.

Mass laboratories including the watt balance

- Dr Leonid Vitushkin (VNIIM) to visit the watt balance on 21 January.
- Dr Victor Jose Lizardi Nieto (CENAM) to visit the watt balance on 22 January.
- His Excellency Dr Rashid Ahmed Bin Fahad (Minister of Environment and Water, Chairman of Emirates Authority for Standardization and Metrology), His Excellency Mr Maahad Hareb Meghair Alkhyeli (Ambassador of UAE in France), Eng. Abdulla Al Maeeni (Director General Emirates Authority for Standardization and Metrology), Eng. Amina Hassan Zainal (Director of Metrology Deptartment), to visit the watt balance on 3 February.
- Dr. Zhang (ISO President), Mr McKinley (ISO Secretary General ad interim), Mr Peyrat (ISO Vice-President, Director General of AFNOR), Mr Poupet (Head of Int. Affairs Dept. AFNOR), Mr Zhu (Director of Int. Standards Section, ANSTEEL Group), Mr Ma, to visit the watt balance on 26 February.
- Delegation of QSTA, Hunan Province, China, led by the Director General Mr. Biao Quyang to visit the watt balance and the electricity laboratories on 15 April.
- Mr Renaud Degoy and Mr Gérard Mathiotte (Institut Géographique National) to carry out gravimetric measurements in the watt balance laboratory on 25 April.
- Dr John Pratt (NIST) to visit the watt balance on 25 April.
- Mr Tim Folger (Scientific American magazine) for interviews for an article about the redefinition of the

kilogram and to visit the watt balance on 17-18 May.

- Dr Jin Wan Chung, Mr In Tae Lim, Mr Ki Lyong Jeong (KRISS), to visit the mass laboratories on 24 May.
- Dr Stephan Schlamminger (NIST) to visit the watt balance on 16 June.
- Mr Renaud Degoy and Mr Gérard Mathiotte (Institut Géographique National) to carry out gravimetric measurements in the watt balance laboratory on 12 August.
- Dr Leonid Vitushkin and Dr Oleg Orlov (VNIIM) to visit the watt balance on 5 September.
- Ms Susanne Porch (NIST) to visit the watt balance on 8 September.
- Film crew sent by AQSIQ (China) for filming in the mass laboratory on 29 September.
- Mr Thomas Fehling, Mr Tony Kowalski and Mr Yingfeng Xu (Sartorius) to visit the watt balance on 11 October.
- Group of 18 attendees of the CBKT course "Leaders of tomorrow" to visit the mass laboratories on 7 November.
- Mr Gregory Strouse (NIST) to visit the watt balance on 17 November.

BIPM Time Department

Director: E.F. Arias

(1 January 2016 to 31 December 2016)

1. International Atomic Time (TAI), Coordinated Universal Time (UTC) and Rapid UTC (UTCr)

(E.F. Arias, A. Harmegnies, Z. Jiang, H. Konaté[1], 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 various timing centres that maintain a local UTC; monthly results are published in *Circular T*. The UTC rapid solution (UTCr) is published every Wednesday by 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.

The structure and content of BIPM *Circular T* were updated in 2016 with the introduction of an interactive version, which gives complete access to the information hosted on the BIPM website and ftp server. The dissemination of information has significantly improved following the development and launch of the 'BIPM Time Department Data Base'. Time Department services can be accessed at http://www.bipm.org/en/bipm/tai/.

The *BIPM Annual Report on Time Activities for 2015*, volume 10, provides comprehensive data for 2015 and is available on the BIPM website at http://www.bipm.org/en/bipm-services/timescales/time-ftp/annual-reports.html

2. Algorithms for time scales (G. Panfilo, G. Petit, A. Harmegnies and L. Tisserand)

The algorithm used by the Time Department to calculate time scales 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 algorithms (prediction and weights) for UTC calculation, behaviour is routinely and carefully monitored to trap and fix unexpected anomalies, although none were observed throughout 2016.

The results concerning the uncertainties of [*UTC-UTC(k)*] reported in Section 1 of *Circular T* have been analysed to evaluate the possibility of revising the algorithm. There are two main issues concerning the current algorithm: firstly, the statistical uncertainty, indicated with uStb in *Circular T*, of the pivot laboratory (at present PTB) is underestimated and unrealistic and secondly, the strict correlation of the uncertainty given by calibration (indicated by uCal). In fact the value of the uncertainty for uncalibrated laboratories (now arbitrarily fixed to 20 ns) affects the uncertainties of the ensemble in a significant way, including the calibrated laboratories. The particular case of the United States Naval Observatory (USNO) should be mentioned due to its important weight in the calculation of UTC; a small change in its calibration uncertainty affects, with the current algorithm, the ensemble of uncertainties in a critical way. A different approach to the problem is being studied, taking into account correlations that are not fully considered in the current algorithm.

2.1 EAL stability

Some 89 % of 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 by contributing laboratories is increasing and represented 28 % of the participating clocks in 2016. The weighting procedure involved in time

[1] Retired on 31 October 2016

scale computation guarantees the long-term stability of EAL. To prevent domination of the scale by a small number of very stable clocks, a maximum relative weight is used each month, which depends on the number of participating clocks. On average, about 12 % of the participating clocks were at the maximum weight during 2016; almost all of these were hydrogen masers. The weighting algorithm, which has been in use since 2014, is based on the predictability of the clock's frequency. It enhances the influence of hydrogen masers on the resulting time scale; 42 % of the contributing hydrogen masers were, on average, at the maximum weight in 2016, whilst no caesium clocks reached the maximum weight.

UTC implicitly relies on 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 2016, 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 2016, individual measurements of the TAI frequency have been provided by nine primary frequency standards, including seven caesium fountains (SYRTE FO2, NIST F1, IT CSF2, SU CSFO2, PTB CSF1, PTB CSF2 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 2016, 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 -0.30×10^{-15} to -1.45×10^{-15}, with a maximum standard uncertainty of 0.33×10^{-15}. A steering correction $+0.3 \times 10^{-15}$ had been applied in December 2016; this was the first time it had 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 2016, known as TT(BIPM15), valid until December 2015, 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(BIPM15) 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 primary and secondary frequency standards.

An accurate analysis of a different approach to the calculation of TT that is more adapted to the case of the secondary frequency standards required a more flexible calculation interval and is ongoing.

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*. Following the project to correct the large offset between UTC and GLONASS time, the absolute calibration of a BIPM receiver was concluded at the VNIIFTRI. This receiver has been used as the reference for the relative calibration of receivers at the Astrogeodynamical Observatory of the Space Research Centre (AOS, Borowiec, Poland) which provides data for computing this offset.

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.

Following an invitation by the CCTF to systematically study frequency ratios of ultra-high precision frequency standards, a somewhat different approach is needed to generate the update of the frequency list. Redundant measurements in this scheme allow for consistency studies but also require a non-linear least squares approach for complete evaluation. Such resources have been developed at the BIPM based on graph theory concepts. This method is a simplified approach, which is an alternative but complementary method to that developed at the NPL. Numerical validation of the two approaches is under way in order to be ready for the next recommended frequency list update in 2017.

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. In addition, measurements of two Sr optical lattice clocks were reported for the first time by the LNE-SYRTE. They will be introduced following the advice of the CCTF Working Group on Primary and Secondary Frequency Standards.

4. Time links used for UTC (E.F. Arias, A. Harmegnies, Z. Jiang, H. Konaté, G. Panfilo, G. Petit and L. Tisserand)

At the end of 2016, 77 time laboratories supplied data for the calculation of UTC at the BIPM. The laboratories are equipped with GNSS receivers and some 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 the tracking of 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 monthly 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*.

Thirteen laboratories operating TWSTFT stations officially submitted data in 2016 for use in the computation of UTC, representing 15 % of the time links. The number of TW links increased during the year with the re-incorporation of laboratories in the Asia Pacific region, which had been absent due to an interruption of the satellite service in the region in 2015. 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 being 0.3 ns.

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 includes a physicist from the BIPM Time Department as a member.

Data from world-wide geodetic-type receivers are collected for UTC computation, using procedures and software that was 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 that produces 'iono-free' solutions 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 ongoing 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), but mostly in the long term (days). Since 2015 the IPPP technique moved to a pre-operational stage and it is now used regularly to compare IPPP results to the few available optical fibre links.

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

4.2. Two-way satellite time and frequency transfer

The TWSTFT participating stations held a meeting at the 30th European Frequency and Time Forum (EFTF) in York, UK, on 6 April 2016. The 24th annual meeting of the CCTF WG on TWSTFT was held at the NIST on 7-8 September 2016. Two major issues discussed at the meetings were the realization of a pilot project involving the BIPM and the WG on TWSTFT: one on the impact of the Software-Defined Radio (SDR) receiver on the uncertainty of the TWSTFT time links; and the establishment of a task group to study the long-term comparison of GPS and TWSTFT links. One output of the pilot study could be the integration of data from SDR-equipped TW stations in the computation 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. Ten TWSTFT links had been used in the computation of UTC in 2016 in Europe-USA-Asia;

they are combined with GPS PPP solutions. Some of the TWSTFT links involved in the computation of UTC are used in the experiment 'Time Transfer by Laser Link' (T2L2). The BIPM plans to develop studies on this technique, which could be used to validate less accurate time links and their calibrations.

Campaigns with a TW travelling calibration station were organized and funded by the participating laboratories in 2016. The parameters obtained have been implemented for UTC computation after 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://ftp2.bipm.org/pub/tai/timelinks/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.

The *BIPM Guidelines for GNSS calibration* are intended for Regional Metrology Organizations (RMOs) and establish 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; the latest revision was issued in March 2016.

The BIPM started the second calibration campaign of the "Group 1" laboratories in APMP, EURAMET, SIM and COOMET in 2016, following the planned periodicity of two years. Several regional calibration trips concerning 27 "Group 2" laboratories have been initiated by the RMOs in accordance with the *BIPM Guidelines* and the first results have been implemented in 2016. By repeatedly applying this new procedure, time transfer accuracy is expected to improve by a factor of at least 2 with respect to the pre-2015 situation.

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), where past calibration results are also provided.

4.4 Advanced time and frequency transfer

Data from two fibre links between UTC contributing laboratories in Europe are regularly submitted to the Time Department and compared with the corresponding links by GNSS time transfer techniques. The Time Department aims to include fibre links in the computation of UTC in the future, and for this purpose 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 that participate in the CIPM MRA, are published monthly in the BIPM key comparison database (KCDB). The number of participants at the end of 2016 was 59, and they constitute a subset of the participants to BIPM *Circular T*.

Key comparison of stabilized lasers CCL-K11

Following a decision at the 98th meeting of the CIPM (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-CCTF Working Group on Frequency Standards, which submits results to the CCL for formal approval. In 2016, BIPM staff members supported the key comparison on issues relating to the development of the measurement campaigns and reporting. Two final reports of the results of this ongoing key comparison, which involved measurements by nine institutes in 2014 and 2015, have been published in 2016.

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, which together represent 60 % to 70 % of the clocks that participate in UTC.

UTCr attained the expected quality, providing a weekly solution which is consistent within 1.1 ns RMS and ±3 ns peak to peak with the values published monthly in BIPM *Circular T*. The results (http://www.bipm.org/en/bipm-services/timescales/time-ftp/Rapid-UTC.html) 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 has a favorable impact 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 meeting of Study Group 7 (Science services) and Working Party 7A (Time signals and frequency standard emissions) in April 2016.

The CCTF established a task group for proposing definitions of TAI and UTC to be submitted to the CGPM in 2018. This was in response to an invitation by the World Radiocommunication Conference 2015 (WRC15) to strengthen the cooperation between the ITU and the BIPM on this matter, and in preparation for the discussions scheduled for the WRC23. This task group, which includes two Time Department staff members, continued to draft the text of the recommendation during 2016 for consideration by the CCTF in June 2017.

8. 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 continued participation in the international working groups in this field is essential.

Cooperation continues on the maintenance of the international celestial reference system within the framework of the activities of a working group created by the International Astronomical Union (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 the 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*. 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).

With the development of optical clocks accurate at the 10^{-18} level, "relativistic geodesy" is the subject of numerous developments, which suggest the full potential of these clocks for the measurement of terrestrial gravity potential and the definition of systems of altimetry references. A physicist from the Time Department is co-chairing a new IAG working group on this subject. In 2016, international collaborations led to publications on the implications of such "chronometric geodesy" for geodesy and the definition of time scales and on the calculation of the relativistic frequency shift with the required accuracy for optical clocks.

9. Comb activities (L. Robertsson)

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

10. Publications

External publications

1. Denker H., Timmen L., Voigt C., Weyers S., Peik E., Delva P., Wolf P., Petit G., Geodetic methods to determine the relativistic redshift at the level of 10^{-18} in the context of international timescales – A review and practical results; *J. Geodesy*, submitted.

2. Hachisu H., Petit G., Ido T., Absolute frequency measurement with uncertainty below 1×10^{-15} using International Atomic Time, *Appl. Phys. B*, 2017, **123**(1).

3. Jiang Z., (2016) Final report of the BIPM Pilot Study on UTC time link calibration, *PTTI Proc.* 20-26, Monterey, CA, USA, 2016.

4. Jiang Z., Matsakis D., Zhang V., Esteban H., Piester D., Lin S.Y., Dierikx E., A TWSTFT calibration guideline and the use of a GPS calibrator for UTC TWSTFT link calibrations, *PTTI Proc.* 231-242, Monterey, CA, USA, 2016.

5. Jiang Z., Piester D., Schlunegger C., Dierikx E., Zhang V., Galindo J., Matsakis D., The 2015 TWSTFT calibration for UTC and related time links, Proc. 30th EFTF meeting, York, UK, 2016.

6. Matus M., Gavalyugov V., Tamakyarska D., Ranusawud M., Tonmueanwai A., Hong F.-L., Ishikawa J., Moona G., Sharma R., Hapiddin A., Boynawan A.M., Alqahtani N., Alfohaid M., Robertsson L., Report on on-going CCL Key Comparison for the year 2014 Comparison of optical frequency and wavelength standards CCL-K11, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 04001.

7. Matus M., van den Berg S., Czulek D., Seppä J., Robertsson L., The CCL-K11 ongoing key comparison. Final report for the year 2015, *Metrologia*, 2016, **53**, *Tech. Suppl.*, 04007.

8. Panfilo G. The Coordinated Universal Time, *IEEE Instrumentation and Measurement Magazine*, June 2016, **19**(3), 28-33.

9. Parisi F., Panfilo G., A new approach to UTC calculation by means of the Kalman Filter, *Metrologia*, 2016, **53**(5), 1185-1192.

10. Petit G., Defraigne P., The performance of GPS time and frequency transfer: comment on 'A detailed comparison of two continuous GPS carrier-phase time transfer techniques', *Metrologia*, 2016, **53**(3), 1003-1008.

11. Riedel F., *et al*. (G. Petit), Remote optical and fountain clock comparison using broadband TWSTFT and GPS PPP, Proc. 30th EFTF meeting, York, UK, 2016.

12. Robertsson L., On the evaluation of ultra-high-precision frequency ratio measurements: examining closed loops in a graph theory framework, *Metrologia*, 2016, **53**(6), 1272-1280.

13. Visser PNAM., Müller J., Lon G., Panet I., Kopeikin S.M., Petit G., Dirkx D., High performance clocks and gravity field determination, Proc ISSI Workshop HISPAC, Space Science Reviews, to be published.

14. Wielgosz R., Arias F., Stock M., Los Arcos J.-M., Milton M., News from the BIPM laboratories – 2015, *Metrologia*, 2016, **53**, 103-107.

BIPM publications

15. *BIPM Annual Report on Time Activities for 2015*, 10, 137 pp., available only at http://www.bipm.org/en/bipm-services/timescales/time-ftp/annual-reports.html

16. *Circular T* (monthly)

17. *Rapid UTC* (*UTCr*) (weekly)

11. 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 Two-Way Satellite Time and Frequency Transfer (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 is the Executive Secretary of the Consultative Committee for Acoustics, Ultrasound and Vibration (CCAUV). She is Secretary of the CCAUV Working Groups for Key Comparisons (CCAUV-KCWG), for RMO coordination (CCAUV-RMO) and on Strategic Planning (CCAUV-SPWG).

G. Petit is Secretary of the CCTF Working Group on Primary and Secondary Frequency Standards (WGPSFS) 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.

12. 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 is 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 until end 2016 of the Conventions Centre. E.F. Arias is a member of the International VLBI[2] 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. She is a member of the Technical Advisory Committee of International Union of Radio Science (URSI) Commission A. 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 and a member of the IERS Directing Board until the end of 2016. He is an associate member of the IGS and member of the IGS Working Groups on Clock Products and on Bias Calibration. In 2016 he represented the BIPM at the United Nations International Committee on Global Navigation Satellite Systems (ICG), and has acted as chairperson of the Task Force on Time References. He is co-chair of the IAG Joint Working Group on Relativistic Geodesy and a member of the IAU Working Groups on Numerical Standards in Fundamental Astronomy and on Pulsar Time Scale.

13. Travel in 2016 (conferences, lectures and presentations, visits)

E.F. Arias to:

- Teddington (UK), 13-14 January, for the meeting of the Quantum, Electromagnetics and Time Programme Expert Group;
- Monterey (USA), 25-29 January, for the PTTI 2016 to chair a session and participate to CCTF Working Group meetings;
- Espoo (Finland); 1-2 March, for the meeting of the Technical Committee on Time and Frequency of EURAMET to give reports on BIPM activities and to coordinate calibrations;
- Geneva (Switzerland), 4-8 April, for the meeting of Study Group 7 and Working Party 7A at the ITU;
- Cambridge (Massachusetts, USA), 6-10 June, for the Symposium on the Science of Time to give an invited talk, and oral contribution;
- Brussels (Belgium), 19-21 September, for the Workshop "Understanding the Earth core and nutation";
- Teddington (UK), 7 October, for the meeting of the Quantum, Electromagnetics and Time Programme Expert Group;
- Haystack (USA), 17-19 October, for the meeting of the IAU Working Group on the third realisation of the International Celestial Reference Frame.

[2] Very Long Baseline Interferometry (VLBI)

Z. Jiang to:

- Monterey (California, USA) 25-28 January, for the PTTI meeting with one oral presentation and a poster and for the TWSTFT participation station (PS) meeting;
- York, UK, 4-8 April, to attend the EFTF meeting with an oral presentation and a poster, to the TWSTFT participation station (PS) other CCTF WG meetings;
- NIST, Boulder, USA, 7-8 September, to the 24th Annual meeting of the CCTF WG on TWSTFT and to give oral reports on the WG activities;
- Shangsa, China, 17-21 May, for the China Satellite Navigation Conference 2016 to co-chair the Session on time and frequency and one oral presentation;
- Xian, China, 17-18 October, to attend the Ceremony of the 50th anniversary of the NTSC foundation and to give an invited presentation;
- NIM, Beijing China, 20 October – 3 November, to audit the Gravity and Time laboratories and activities.

G. Panfilo to:

- Espoo (Finland); 1-2 March, for the meeting of the Technical Committee on Time and Frequency of EURAMET to give reports on UTC algorithms and existing guidelines of the CCTF WGMRA.

G. Petit to:

- Monterey (California, USA) 25-28 January, to give a tutorial course and attend the PTTI meeting, with one oral presentation;
- Villetaneuse (France), 24 March, to attend the General Assembly of the Labex "FIRST-TF";
- York (UK) 4-8 April, to attend the EFTF 2016 meeting, to give one oral presentation, to attend four CCTF WG meetings, and to attend the Workshop "Optical clocks: Quantum engineering and international timekeeping";
- Vienna (Austria), 17 April, to attend the Directing Board of the IERS;
- Besançon (France), 28-29 June, to give two lectures at the European Frequency and Time Seminar;
- Paris (France), 10 October, to attend the *Journée thématique* "Miniature atomic clocks";
- Sochi (Russian Federation), 7-11 November, for the eleventh meeting the International Committee on GNSS (ICG), with chair of a task force and presentations;
- Paris (France), 30 November, to attend a PhD jury as reviewer.

L. Robertsson to:

- York (UK) 4-8 April, to attend the EFTF 2016 meeting, to attend four CCTF WG meetings, and to attend the Workshop "Optical clocks: Quantum engineering and international timekeeping".

14. Visitors, secondees in 2016

- V. Lizardi, CENAM (Mexico), for a visit to the Time Department and laboratory, 10 January;
- T. Ido, NICT (Japan), for a discussion on Secondary Frequency Standards, 25 February;
- X. Zhang accompanied with a delegation from ISO for a visit to the Time Department and laboratory, 26 February;
- B. Ouyanj and a delegation from NIM/QTSA (China), for a visit to the Time Department and laboratory, 15 April;
- J.W. Chung, I.T. Lim and K.L. Jeong from KRISS (Republic of Korea), 24 May;

- J. Feng, NIM (China), for training on the implementation of CCM.G-K2, 6 July to 6 August;
- P. Koppang, USNO (USA), for a discussion on algorithms, 27 September;
- A. Bauch, PTB (Germany), L. Erard (CCTF), P. Koppang, USNO (USA) and ITU WP7A, D. Rovera, LNE-SYRTE (France), P. Tavella, INRIM (Italy), P. Whibberley, NPL (UK) for the meeting of the CCTF WGTAI Task Group on Time Scale Definitions, 28 September;
- D. Matsakis, USNO (USA), for a collaboration on the evaluation of uncertainties of *[UTC-UTC(k)]* reported in Section 1 of *Circular T*, 10 October to 8 November.

BIPM Ionizing Radiation Department

Director: J.M. Los Arcos

(1 January 2016 to 31 December 2016)

1. X- and γ-rays (D.T. Burns, C. Kessler, S. Picard[1] and P. Roger)

1.1 Dosimetry standards and equipment

Eight ongoing comparison series, BIPM.RI(I)-K1 to BIPM.RI(I)-K8, are currently supported within the dosimetry programme. The project to develop a new absorbed-dose standard for medium-energy x-rays, which will form the basis for a ninth comparison series, BIPM.RI(I)-K9, was completed, with further measurements of photon attenuation in air and the characterization of three more transfer instruments. Following a pilot comparison with the PTB (Germany) in late 2015, measurements for the first full comparison, also with the PTB, were made during November and December 2016. The new standard will be presented to the CCRI(I) at its 2017 meeting, with the aim of gaining approval to launch the new comparison series and to publish the results of the PTB comparison. Comparison with the PTB will enable the results to be linked to those of a previous EURAMET comparison piloted by the PTB. The new standard will also enable a new calibration series for national standards of absorbed dose to water for x-rays in the 100 kV to 250 kV x-ray range.

For the BIPM.RI(I)-K6 comparison series for absorbed dose to water in high-energy photon beams, new software for data acquisition and analysis was developed and an update was made to the Technical Protocol that now offers two options to determine the dose conversion factor $C_{w,c}$ from graphite to water. One is based on Monte Carlo calculations for a depth of 10 g cm^{-2}, which can still be made at the BIPM using the photon spectra supplied by the NMI in question. However, a quadratic fit to twenty complete determinations of $C_{w,c}$ generated between 2009 and 2015 shows deviations consistent with the typical statistical standard uncertainty of 5 parts in 10^{-4}. For this reason, as an alternative, it is now possible to obtain $C_{w,c}$ by interpolation using the values for $TPR_{20,10}$ measured at the participating NMI.

The tenth comparison in the BIPM.RI(I)-K6 series, was carried out on-site at the NIM (China). The measurements were made in the 6 MV and 10 MV beams of the NIM Elekta accelerator from 15 to 29 November 2016, and the NIM chose to use the new interpolation option for determining the $C_{w,c}$ values. The measurements for higher energies could not be made because of the long delays necessary to fulfil appropriate conditions for radiological protection. This comparison enabled the NIM to verify robustly the present Chinese primary standard to realize absorbed dose to water in accelerator beams and the draft A report is in progress.

The report of the previous comparison with the NMIJ/AIST (Japan) was published. The report of the comparison with the VSL (the Netherlands) which took place at the NPL (UK) accelerator in October 2014, is currently under review by the Key Comparison Working Group KCWG(I) of CCRI(I).

The new laboratory to support measurements for the BIPM.RI(I)-K8 comparison series for high dose rate (HDR) ^{192}Ir brachytherapy sources was completed. A new system for digital radiography was installed in the laboratory, and the recently installed robotic arm was programmed to operate with both systems. The low-energy x-ray laboratory was refurbished, necessitating the removal of the complete measuring system; after re-installation, the reference plane for each of the standards was re-measured and a series of quality assurance (QA) measurements were performed to assure the stability of the standards.

Primary measurements and reference chamber calibrations have continued in all the reference x- and γ-ray beams. The programme to update the dosimetry services' computer hardware and software continued. Minor suggestions from the peer review external audit, which was carried out in December 2015, were implemented and the system was subject to a successful internal audit in December 2016.

[1]Shared time with the ILC Department as KCDB Coordinator

1.2 Dosimetry comparisons

During 2016, seven dosimetry comparisons were carried out in terms of air kerma or absorbed dose to water using the BIPM x- and gamma-radiation beams, with the IST-LPSR (Portugal), MKEH (Hungary), NIST (USA), PTB, SCK-CEN (Belgium) and two for the VSL. In addition, one high-energy absorbed-dose-to-water comparison BIPM.RI(I)-K6 was carried out with the NIM using their own accelerator facility.

Ten comparison reports were approved and published in *Metrologia Technical Supplement* for the CMI (Czech Republic, two reports), NIM, NMIJ (three reports), NRC (Canada), PTB and the VSL (two reports) (see § 3).

1.3 Characterization of national standards for dosimetry

Twenty-eight characterizations of nineteen national secondary dosimetry standards were carried out for the KRISS (Republic of Korea), CIEMAT (Spain), NMISA (South Africa), IAEA, SMU (Slovakia), STUK (Finland), SSM (Sweden), GUM (Poland) and the IST-LPSR.

In addition, the International Atomic Energy Agency (IAEA)/World Health Organization (WHO) dosimetry assurance programme continues to be supported by reference irradiations. This involved two series of irradiations in 2016, for the radiotherapy level in the ^{60}Co beam and for the radiation protection level in the ^{137}Cs beam.

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

2.1 International Reference System (SIR) for γ-ray emitting radionuclides

The *Système International de Référence* (SIR) was implemented at the BIPM almost 40 years ago and constitutes a unique tool to provide world-wide equivalence of radionuclide measurements on demand from NMIs/DIs. It is based on the ^{226}Ra reference sources and includes two ionization chambers to measure gamma emitters and a transfer instrument. The latter is used to carry out measurements on-site at an NMI/DI facility, when the half-life of a radionuclide is too short to allow shipment of ampoules to the BIPM.

This combination supports the ongoing comparisons BIPM.RI(II)-K1 and BIPM.RI(II)-K4. Both activities are subject to the BIPM Quality Management System and the system underwent a successful internal audit in December 2016.

2.1.1 SIR measurements and reports

Within the radionuclide measurements programme, the SIR received four ampoules of three different radionuclides, ^{88}Y, ^{133}Ba and ^{134}Cs from three laboratories (LNE-LNHB (France), NRC and PTB): all of them were sent by the laboratories to generate equivalence values in the ongoing BIPM.RI(II)-K1 comparison.

Measurements of potential impurities in SIR ampoules have been suspended since the failure of the Ge(Li) spectrometer in July 2013. The efficiency curves and uncertainty budget for the replacement high-purity germanium (HPGe) spectrometer are to be finalized.

Updated final reports of three BIPM.RI(II)-K1 comparisons were published in *Metrologia Technical Supplement* for ^{18}F, ^{113}Sn and ^{228}Th. The latter two reports include an evaluation of the first key comparison reference values (KCRVs) for these nuclides. The updated report for ^{60}Co was also submitted to *Metrologia Technical Supplement.* There are 37 SIR results in the draft B stage awaiting publication in the BIPM key comparison database (KCDB). Reporting forms for five measurements are yet to be received from participating NMIs.

Historic data on the stability of the SIR ionization chambers with measurements of the ^{226}Ra sources over 15 years were compiled and analysed for a joint research study, with contributions from 14 NMIs. This study is concerned with analysing the potential solar influence on nuclear decay constants, an effect that is claimed from time-to-time by some authors. The results obtained are extremely robust: they were supplied by 14 metrology laboratories that specialize in radioactivity measurements and refute such an influence. The results have been published in *Physics Letters B* and constitute a definitive reference article for the future.

2.1.2 Extension of the SIR to short-lived radionuclides

The second series of calibration measurements of the SIR Transfer Instrument (SIRTI) against the SIR were carried out for ^{64}Cu ($T_{1/2}$ = 13 h) by measuring a solution from the NPL in both systems. The analysis of results is in progress.

In 2016, the SIRTI comparisons (BIPM.RI(II)-K4) took place on-site at the NIST for ^{18}F, ^{64}Cu and at POLATOM (Poland) for ^{18}F, ^{64}Cu and ^{99m}Tc. These laboratories were the first participants in the BIPM.RI(II)-K4.Cu-64 comparison. The comparison reports are in the draft A stage.

The BIPM.RI(II)-K4.Tc-99m ($T_{1/2}$ = 6.0 h) key comparisons using the SIRTI at VNIIM (Russia) and ENEA-INMRI (Italy) in 2014 were published in the *Metrologia Technical Supplement.* The KCRV, which is defined in the framework of the BIPM.RI(II)-K1.Tc-99m comparison (SIR), was updated to include SIRTI results for the first time following a decision of the CCRI in 2015.

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 have been published in *Applied Radiation and Isotopes.* The three results linked to the BIPM.RI(II)-K1.F-18 comparison (SIR) agree with the KCRV within one or two standard uncertainties showing, for the first time, the usefulness of the SIRTI for comparing ^{18}F activity measurements world-wide.

The ^{99m}Tc and ^{18}F SIRTI comparisons which took place at the NMISA in November 2015 were submitted for publication in *Metrologia Technical Supplement.*

2.1.3 Extension of the SIR to pure beta emitters

An extension of the SIR to beta emitters using liquid scintillation counting (LSC) techniques is being studied by the CCRI(II) working group ESWG(II). A pilot study is in progress to test the two LSC methods proposed: the Universal Cross-Efficiency method and the Apparent Activity method.

The pilot study for the extension of the SIR to beta emitters by liquid scintillation counting resumed with the collaboration of Dr Laureano-Perez, a secondee from the NIST. A newly purchased balance has been used to prepare 10-ml sources of ^{3}H with three scintillators from the six ampoules previously received from the IFIN (Romania), IRMM, NIM, NMIJ, POLATOM and PTB. Measurements have been completed in the Beckman LSC TA 1000 counter and are in progress in the Quantulus 1220. Measurements with the Perkin Elmer TriCarb 2910 TR and with the BIPM-designed Triple-to-Double Coincidence Ratio (TDCR) counters are under way.

2.2 Primary measurement facilities

The coincidence systems using atmospheric and pressurized proportional counters were re-setup following reorganization of the laboratories in the Marie-Curie building. The atmospheric system has restarted and the pressurized system's electronics await checking. A monitoring system and alarm for CH_4 levels in the room has been installed.

2.3 Radioprotection and regulatory activities.

In parallel to the programme of measurements, the French *Autorité de Sécurité Nucléaire* (ASN) was provided with the necessary documents to renew the authorization for working with and the import and export of radioactive substances for a new five-year term. Additional documents to obtain these authorizations were requested by the ASN and are in the process of being completed.

3. Publications

1. Andreo A., Bergstrom P., Burns D.T., Fernandez-Varea J., Krajcar-Bronic I., Ross C., Salvat F., Seltzer S., Key Data For Ionizing-Radiation Dosimetry: Measurement Standards And Applications, *ICRU Report 90*, 2016.
2. Bich W., Cox M. and Michotte C., Towards a new GUM—an update, *Metrologia*, 2016, **53,** S149.
3. Burns D.T., Kessler C., de Pooter J.A., Jansen B.J., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the VSL, The Netherlands and the BIPM in medium-energy x-rays, *Metrologia*, 2016, **53**, *Tech. Suppl.*, 06016.
4. Burns D.T., Kessler C., Mainegra-Hing E., Shen H., McEwen M.R., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the NRC, Canada and the BIPM in medium-energy x-rays, *Metrologia*, 2016, **53**, *Tech. Suppl.*, 06008.
5. Burns D.T., Kessler C., Sochor V., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the CMI, Czech Republic and the BIPM in low-energy x-rays, *Metrologia*, 2016, **53**, *Tech. Suppl.*, 06009.
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4. 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 using liquid scintillation (ESWG(II)) and Rapporteur of the CCRI RMO Working Group (RMOWG). During 2016, meetings of the CCRI(II) Key Comparisons Working Group (KCWG(II)) were held 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 two *ad hoc* groups within the CCRI(I), one evaluating the effect of excess charge on the value for W_{air} (work that was incorporated into the International Commission on Radiation Units and Measurements (ICRU) report 90 on key data [publications, reference 1]) and the second to report on the implementation of the ICRU Key Data recommendations.

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

C. Michotte is a member of the CCRI(II) Key Comparisons Working Group (KCWG(II)), which met in March and November 2016.

S. Picard is the Executive Secretary of the Consultative Committee for Thermometry (CCT). She was invited to participate in the EURAMET-TC-T meeting in February.

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 the KCWG(II). The latter met in March and November 2016.

The department's cooperation work with the RMOs included two comparison reports for ionizing radiation (APMP.RI(I) and EURAMET.RI(I)), and six comparison reports for thermometry (CCT, the APMP.T (4) and the SIM.T) being reviewed prior to circulation to the CCRI or CCT for approval. The reports are published in *Metrologia Technical Supplement.*

5. Activities related to external organizations

J.M. Los Arcos is 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. In 2016 he was appointed as a member of the *Comité Scientifique Rayonnements Ionisants du Laboratoire national d'essais* (France), which met in June and November to evaluate the progress and the proposals for new research projects.

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 attended the annual ICRU Commission meeting in Rome in April 2016. He is a member of the ICRU Report Committee on Key Data for Dosimetry, which published its ICRU Report 90 in 2016 [Publications, reference 1] 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, which met in Vienna in March 2016 and reported to the IAEA its recommendations for the future work programme. He is also a consultant to the IAEA on the revision of the international code of practice on Absorbed Dose Determination in External Beam Radiotherapy (IAEA-TRS-398) and attended a consultants meeting in Vienna in August 2016.

C. Michotte is the Scientific Secretary and *rapporteur* for the JCGM-WG1 meetings, which were held in May and November 2016.

G. Ratel is the BIPM representative at the International Committee for Radionuclide Metrology (ICRM) and is the President of the ICRM Nominating Committee. He is a member of the Scientific Committee for the 21st International Conference on Radionuclide Metrology and its Applications (ICRM 2017), which will be held in Buenos Aires (Argentina) on 15-19 May 2017. He also acted as a member of the Scientific Committee for the Low-Level Radioactivity Measurement Techniques (LLRMT) Conference to organize the submitted abstracts and to review four papers presented at the conference before publication in *Applied Radiation and Isotopes*.

6. Travel in 2016 (conferences, lectures and presentations, visits)

D.T. Burns to:

- Vienna (Austria), 14 to 18 March, to participate in the biennial meeting of the Scientific Committee of the IAEA/WHO Network of Secondary Standards Dosimetry Laboratories.
- Rome (Italy), 15 to 19 April, to participate in the annual meeting of the ICRU Commission.
- Vienna (Austria), 8 to 12 August, to participate in a consultants meeting on the revision of the international Code of Practice TRS-398 for external-beam radiotherapy.

C. Kessler to:

- Buenos Aires (Argentina), 19 to 21 October, to perform a technical audit of the Secondary Standard Dosimetry Laboratory of Argentina (CRRD)
- Beijing (China), 14 to 30 November, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water with the NIM in the reference photons beams of its accelerator facility.

P. Roger to:

- Beijing (China), 14 to 30 November, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water with the NIM in the reference photons beams of its accelerator facility.

S. Picard to:

- Berlin (Germany), 26 to 27 January, to participate at the EURAMET TC-IR meeting and to present recent news from the BIPM and the CCRI.
- Valetta (Malta), 25 to 26 February, to participate at the EURAMET TC-T meeting and to present recent news from the BIPM and the CCT.
- Zakopane (Poland), 29 June to 1 July, to attend at the TEMPMEKO conference and participate in a number of working and task group meetings linked to the CCT.
- Beijing (China), 16 to 22 November, to carry out the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the NIM at its medical accelerator facility.

C. Michotte to:

- Otwock (Poland), 17-28 October, to carry out activity comparisons of ^{99m}Tc (BIPM.RI(II)-K4.Tc-99m), ^{18}F (BIPM.RI(II)-K4.F-18) and ^{64}Cu (BIPM.RI(II)-K4.Cu-64) at the POLATOM using the SIR Transfer Instrument.

C. Michotte and M. Nonis to:

- Gaithersburg (USA), 18-28 May, to carry out activity comparisons of ^{64}Cu (BIPM.RI(II)-K4.Cu-64) and ^{18}F (BIPM.RI(II)-K4.F-18) at the NIST using the SIR Transfer Instrument.

G. Ratel to:

- Rome (Italy), 2-4 November, to attend the Scientific Committee of the 21th ICRM conference to be held in Buenos Aires (Argentina) in 2017 and the Executive Board Meeting of the ICRM.

7. Visitors in 2016

- Dr Zhang, ISO President; Mr McKinley, ISO Secretary-General ad interim; Mr Peyrat, ISO Vice-President and Director General of AFNOR; Mr Poupet, Head of the International Affairs Department, AFNOR; Mr Zhu, Director of the International Standards Section, ANSTEEL Group ; Mr Ma, Secretary to Dr Zhang. 26 February.
- Visit by Mr A.A. Haj (ESMA) and Mr M. Al Mulla (EMI) Abu Dhabi, Dr M. Alharthi (SASO) and Mr O. Kanakrieh (GSO) Riyadh, and Mr S. Al-Remaihi (QS) Doha: 17 March.
- Dr Sylvie Pierre (LNE-LNHB, France) with Alfredo Lopez Ferreira (IRD, Brazil) and Liu Haoran (NIM, China), 15 December.

8. Guest workers in 2016

- Dr V. Sochor, CMI (Czech Republic), 29 February.
- Mr L. Czap, International Atomic Energy Agency IAEA, 13-24 June.
- Dr N. Durny, Slovak Institute of Metrology SMU (Slovakia), 22-24 August.
- Dr R. Nylund and Dr J. Huikari, Radiation and Nuclear Safety Authority STUK (Finland), 12-15 September.
- Dr L. Laureano-Perez, National Institute of Standards and Technology NIST (USA), 13 September 2016 – 5 March 2017.
- Dr C. Mihailescu, Belgian Nuclear Research Centre, *Studiecentrum voor Kernenergie - Centre d'Etude de l'Energie Nucleaire* (Belgium), 26-30 September.

BIPM Chemistry Department

Director: R.I. Wielgosz

(1 January 2016 to 31 December 2016)

1. Comparison coordination and supporting laboratory activities

Laboratory activities supported the coordination of seven key comparisons in the field of metrology in chemistry and biology during 2016, with the assistance of nine visiting scientists from NMIs.

In the area of air quality measurement standards, new values of the ozone absorption cross-section, based on gas phase titration have been published in *Analytical Chemistry*[1]. The BIPM contributed to the review and recommendations on absorption cross-sections of ozone in the ultraviolet and visible spectral regions published in the *Journal of Molecular Spectroscopy*[2] as part of an initiative of the International Ozone Commission (IO3C), the World Meteorological Organization (WMO) and the IGACO (Integrated Global Atmospheric Chemistry Observations) O3/UV subgroup. This initiative will study, evaluate, and recommend the most suitable ozone absorption cross-section laboratory data to be used in atmospheric ozone measurements. The BIPM is contributing to the CCQM GAWG Ozone Cross Section Task Group, created within the CCQM Gas Analysis Working Group (GAWG), which will review and recommend the best value and uncertainty for the ozone cross-section to be used in the key comparison BIPM.QM-K1. Measurements for the CCQM-K90 comparison on formaldehyde standards were completed. The final report is in preparation and will be published in 2017.

In the area of greenhouse gas standards, validation work was completed for the CCQM-K120 comparison (carbon dioxide in air), with Fourier transform infrared spectroscopy (FTIR), isotope ratio infrared spectrometer (IRIS) and gas chromatography – flame ionization detector (GC-FID) methods being fully characterized. A method for measuring isotopic abundances in CO_2, which was required for corrections to be applied to the comparison method, was developed. A paper '*Calibration strategies for FTIR and other IRIS instruments for accurate $\delta^{13}C$ and $\delta^{18}O$ measurements of CO_2 in air*', in collaboration with the University of Wollongong (Australia) has been submitted to *Analytical Chemistry*. Forty-six standards will be compared as part of CCQM-K120, with measurements expected to be completed by the end of May 2017. Development and validation of a manometric system for CO_2 measurements has progressed with secondments from the NIST and the SP (Sweden). This work demonstrated that the first all-glass prototype can produce results within 1 % of certified gravimetric values. Encouraging results on treated steel surfaces have permitted the design of a more robust second prototype, with testing and validation of the system planned for the first half of 2017. Preparation for a comparison on CO_2 isotope ratio standards, coordinated jointly by the BIPM and the International Atomic Energy Agency (IAEA), has started with the development of a stable isotope reference mixture generation facility at the BIPM, supported by a secondee from the NIM (China).

Within the BIPM's organic primary calibrator programme, the CCQM-K55.d comparison on folic acid purity was completed. The final report will be published in 2017. The comparison demonstrated the increasing use of quantitative NMR (qNMR) at NMIs for the value assignment of this category of standard. The universal calibrator programme for qNMR at the BIPM, an activity initiated together with the NMIJ (Japan), was supported by secondees from the NIM and INMETRO (Brazil), with characterization of the performance of standards that are soluble in deuterated chloroform and methanol. Preparations for the CCQM-K78.a comparison on multi-component amino acid calibration solutions were completed. Ampouled samples were distributed to participants in November-December 2016, with results expected in the first quarter of 2017. The final report of the key comparison on C-peptide purity (CCQM-K115/P55.2), coordinated by the BIPM in collaboration with the NIM, was completed. A paper written in collaboration with the University of Missouri (USA), NIM, NMIJ and NIBSC (UK) on the evolving calibration hierarchies for C-peptide measurements has been submitted to *Clinical Chemistry*. The general approach to pure peptide calibrator value assignment was presented at the

Protein and Peptide Therapeutics and Diagnostics: Research and Quality Assurance (PPTD-2016) workshop, which was held in Chengdu (China) in June 2016. This workshop was organized jointly by the BIPM and the NIM.

The work undertaken on *Impurity Determination for Hepcidin by Liquid Chromatography – High Resolution and Ion Mobility Mass Spectrometry for the Value Assignment of Candidate Primary Calibrators*, which was supported by a secondee from the LNE (France), has been submitted to *Analytical and Bioanalytical Chemistry*. Development of methods to characterize calcitonin calibrators was undertaken by a secondee from the HSA (Singapore), with the material intended as a future candidate key comparison material for small peptides with disulfide bonds.

The BIPM hosted a joint symposium with the World Anti-Doping Agency (WADA) on 28-29 September 2016. One hundred invited experts from National Measurement Institutes, the clinical chemistry and laboratory medicine community and from WADA and the WADA-accredited anti-doping laboratories discussed the latest initiatives in areas of metrology for organic and biological analysis relevant to this high-profile measurement sector. One of the major recommendations of the meeting was the requirement to produce a prioritized list of pure substance and matrix CRM requirements including: prohibited list analytes currently lacking a suitable pure substance reference materials (RM); threshold Substances; phase II/III and long-term metabolites (LTMs) of anabolic steroids; peptides/proteins; and reference materials for gene doping. WADA in consultation with the World Association of Anti-Doping Scientists (WAADS) and the anti-doping laboratories are expected to develop the list in 2017.

The department's work in the area of supporting measurements for key climatological observables advanced with the publication of a paper in *Metrologia* [3].

2. Capacity Building and Knowledge Transfer activities in Metrology for Safe Food and Clean Air

Laboratory programmes for capacity building and knowledge transfer (CBKT) in Metrology for Safe Food and Clean Air have started in the BIPM Chemistry Department.

The Mycotoxin Metrology CBKT programme started in April 2016, with Dr Xiuqin Li, from the NIM (China) joining the BIPM as a visiting scientist for a period of 1 year to develop methods for the characterization of aflatoxin B1 and zearalenone pure materials. These will form the basis of calibration solutions that will support training and skills-broadening secondments on mycotoxin calibrant preparation and characterization methods, which will organized at the BIPM from 2017 to 2019.

The issue of food safety and trade is a major concern for countries developing metrology and quality assurance systems. Ensuring the safety of food has been a major focus of recent international and national action. Both microbiological and chemical hazards are of concern, including the contamination of food and feed by mycotoxins (toxic metabolites of fungi), which are significant sources of food-borne illnesses. The knowledge that mycotoxins can have serious effects on human and animal health has led many countries to establish regulations on mycotoxins in food and feed over the last decades to safeguard the health of humans, as well as the economic interests of producers and traders. Over 100 countries have implemented specific regulatory limits for mycotoxins in foodstuffs and feedstuffs, and these need to be supported by a sound measurement infrastructure for mycotoxin analysis in order to enforce and verify products, protect populations and avoid technical barriers to trade in foodstuffs. This project has been designed to allow the BIPM and NMIs to work together to strengthen the mycotoxin metrology infrastructure; provide knowledge transfer to scientists who are developing capabilities in this area; and to enable NMIs to provide mycotoxin calibrants and matrix reference material and proficiency test materials to support mycotoxin testing laboratories within their countries.

The capacity building programme will be extended with visiting scientist training programmes expected to be implemented at the NIM on mycotoxin matrix certified reference materials as well as training on analytical methods for mycotoxins at the NMISA (South Africa). In addition to providing staff resources to support the programme, the NIM has also donated the pure materials required to deliver the programme. Training secondments for three visiting scientists from NMIs to the BIPM in 2017 are being supported by the PTB (Germany).

The startup meeting for the CBKT programme for "Metrology for Safe Food and Feed", focusing on mycotoxin metrology and standards, was held at the BIPM in April 2016, with the participation of INMETRO, INTI (Argentina), KEBS (Kenya), NIM, NIMT (Thailand), NMISA (South Africa), PTB, UME (Turkey) and the United Nations Industrial Development Organization (UNIDO). Laboratory activities at the BIPM have allowed pure materials for aflatoxin B1 and zearalenone (ZEN) to be characterized. The mass fraction of ZEN in the pure material was determined by qNMR and structurally-related impurities were quantified by liquid chromatography-ultraviolet spectroscopy-charged aerosol detection (LC-UV-CAD) and liquid chromatography-tandem mass spectrometry (LC-MS/MS) techniques. Stock and calibration solutions of ZEN have been ampouled for homogeneity and stability testing, in preparation for the capacity building and knowledge transfer activities that will take place at the BIPM in 2017.

A capacity building and knowledge transfer programme in 'Metrology for Clean Air' has been established at the BIPM. The programme started in June 2016, when Mr Manuel de J. Avila from the CENAM (Mexico) joined the BIPM on a 'skills broadening' secondment on FTIR operation and the analysis of FTIR spectra for gas analysis. This was funded in part by the PTB. The BIPM comparison programme on air quality and greenhouse gas standards has relied on Fourier Transform Infrared (FTIR) methods for the analysis of NO_2, NO, HCHO, CO_2 and CH_4 gas standard concentrations. The comparison and CBKT programme both underpin the development of traceable gas concentration standards in NMIs, in response to regulations on air quality and requirements for monitoring gaseous pollutant concentrations.

3. Activities related to the JCTLM

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

In February 2016 the Cycle 12 reference materials and measurement methods and the Cycle 10 reference measurement laboratory services, which were approved by the JCTLM Executive Committee during its 15th Annual Meeting in December 2015 were published in the JCTLM database.

As of December 2016 the JCTLM Database contained:

- 293 available certified reference materials covering 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;
- 180 reference measurement methods or procedures that represent about 80 different analytes in nine categories of analytes;
- 146 reference measurement services, delivered by fifteen reference laboratories and two NMIs in eight countries, which cover seven categories of analytes.

The latest JCTLM call for nominations for Cycle 13 reference materials and reference measurement methods and procedures, and Cycle 11 for reference measurement laboratory services, was announced on the JCTLM website in February 2016. An email notification was sent to 600 potential contributors to the JCTLM. As of July 2016, 11 nominations for materials, seven nominations for methods, and 39 nominations for services had been received and sent out to the review teams for evaluation.

The third issue of the JCTLM Database Newsletter was distributed in February 2016. Future editions of the newsletter will include contributions from the JCTLM-TEPWG.

The 16th and 17th meetings of the Executive Committee of the JCTLM were held in Chengdu (China) on 4 June 2016 and at the BIPM on 6-7 December 2016, respectively. During the December meeting, the Executive approved the revised JCTLM Quality Manual documents including the set of procedures of the Executive, Secretariat and newly formed Database Working Group. Changes introduced in the procedures include the new organizational structure described in the Declaration of Cooperation between the BIPM, the International Federation of Clinical chemistry and Laboratory Medicine (IFCC) and the International Laboratory Accreditation Cooperation (ILAC) which was signed by the three sponsoring organizations in April 2016, and the new Database Working Group policies for soliciting and reviewing nominations for materials, methods, and services.

The Database Working Group meeting was held at the BIPM on 5 December 2016. The JCTLM review team leaders reported on the assessment of the nominations for materials, methods or services, and presented an overview of potential developments in their areas.

The JCTLM Working Group on Traceability: Education and Promotion (JCTLM-TEPWG) organized two electronic meetings in April and September 2016. The JCTLM-TEPWG identified six work streams to describe traceability and its role in laboratory medicine. In addition it has made progress on producing various educational support materials, a glossary of commonly used definitions and a 'mini-presentation' to explain scientific concepts of traceability in laboratory medicine. The group has also drafted reviews in journals or presentations for symposia at professional society meetings, with the aim of raising awareness of the impact of metrological traceability to manufacturers, professionals and regulators.

4. Publications

1. Viallon J., Moussay P., Flores E. and Wielgosz R.I., Ozone cross-section measurement by gas phase titration, *Anal. Chem.*, 2016, 88(21), 10720-10727.
2. Orphal J., (Moussay P., Viallon J., Wielgosz R.I.,) *et al*, Absorption cross-sections of ozone in the ultraviolet and visible spectral regions: Status report 2015, *J. Mol. Spectrosc.*, 2016, 327, 105-121.
3. Feistel R., Wielgosz R., *et al*, Metrological challenges for measurements of key climatological observables: oceanic salinity and pH, and atmospheric humidity. Part 1: overview, *Metrologia*, 2016, **53**(1), R1-R11.
4. Viallon J., Moussay P., Wielgosz R., Bebic J., Norris J.E., Guenther F., Final report, on-going key comparison BIPM.QM-K1: ozone at ambient level, comparison with DMDM, July 2015, *Metrologia*, 2016, **53**, *Tech. Suppl*., 08006.
5. Westwood S., Wielgosz R.I., Report on the BIPM-WADA Workshop: Standards and Metrology in support of Anti-Doping Analysis, *Rapport BIPM-2016/01*, 11 pp.

5. Activities related to the work of Consultative Committees

The CCQM held its 22nd meeting on 20-21 April 2016 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 and the CCQM Working Group on Organic Analysis (OAWG). He is Chairman of CCQM Working Group on Protein Analysis (PAWG) Focus Group I on peptide/protein purity

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. He is a member of the editorial board of Accreditation and Quality Assurance and 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. He is also 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 2016

R.I. Wielgosz to:

- Teddington (UK), 18-19 January to participate in the UK National Measurement System (NMS) Optical, Gas and Particle Metrology Programme Expert Group (PEG) meeting.
- IRMM, Geel (Belgium), 4-5 February, to present BIPM activities and CIPM decisions at the EURAMET METCHEM meeting.
- Teddington (UK), 15-16 February to participate in the UK NMS Chemical and Biological Metrology Programme Expert Group (PEG) meeting.
- Berlin (Germany), 22 February, to participate in a mid-term review of EMPIR Metrology Research Projects.
- Mexico City (Mexico), 24-26 February, invited speaker at a workshop on 'Quality Infrastructure for Traceable Measurements of Greenhouse Gases to Support their Measurement, Report and Verification Strategies'.
- Frankfurt (Germany) 30 March, to present JCTLM activities at the ILAC Accreditation Committee meeting.
- Washington DC (USA), 19-20 May, to give a presentation on development of Surface Ozone Standards at the Optical Society (OSA) meeting.
- Chengdu (China), 1-3 June, as co-chair, organizer and speaker at the BIPM-NIM Protein and Peptide Therapeutics and Diagnostics Workshop.
- Teddington (UK), 9 June to participate in the UK Measurement Strategy Delivery Plan Workshop and 20-21 June to participate in the ISO TC 212 WG2 meetings.

- Varenna (Italy), 27-29 June, as a lecturer at the Varenna Metrology Summer School.
- Braunschweig (Germany), 11-12 July, to participate in the 'kick off' meeting of European Metrology Programme for Innovation and Research (EMPIR) project 15HLT02 as a member of the project advisory board.
- NIST, Gaithersburg (USA), 12-13 September, to present 'Measurement Science in Forensics and Metrologia' at the US National Committee on Forensic Science meeting.
- IPQ, Lisbon (Portugal), 11-14 October, to participate in CCQM GAWG meetings and workshops.
- WHO, Geneva (Switzerland), to participate in the meeting of the Executive Committee on Biological Standards.
- INRAP, Tunis (Tunisia), 3-4 November, as an expert advisor for the development of a Chemical Metrology Programme within Tunisia.
- Da Nang (Vietnam), 8-11 November, invited speaker at two APMP workshops on 'Mycotoxins in Grain' and 'Metrology for Climate Change'.
- Milan (Italy), 18 November, invited speaker to present JCTLM activities at the 10th International CIRME meeting.
- IAEA, Vienna (Austria), 21-25 November, to chair a session and give a presentation at the IAEA Technical Meeting on the Development of IAEA Stable Isotope Reference Products.
- WCO Headquarters, Brussels (Belgium), 20 December, for an introductory meeting between the BIPM and the World Customs Organization (WCO).

E. Flores to:

- IPQ, Lisbon (Portugal), 11-14 October, to participate in CCQM GAWG meetings and workshops and to give lectures on: CCQM-K74.2018 and CCQM-K120 (ambient level carbon dioxide) and FTIR and IRIS calibration strategies for accurate CO_2 in air $\delta^{13}C$ and $\delta^{18}O$ measurements.

R. Josephs to:

- Chengdu (China), 1-3 June, as co-chair, organizer and speaker at the BIPM-NIM Protein and Peptide Therapeutics and Diagnostics Workshop.
- Winnipeg (Canada), 6-9 June, as a speaker at the World Mycotoxin Forum, which combined the 9th World Mycotoxin Forum and the XIVth International Symposium on Mycotoxins.

S. Maniguet to:

- Chengdu (China), 1-3 June, to participate at the BIPM-NIM Protein and Peptide Therapeutics and Diagnostics Workshop and JCTLM Executive Meeting.

N. Stoppacher to:

- NIM, Beijing (China), 1 July to 31 August, for a secondment on qNMR and ion mobility mass spectrometry.

J. Viallon to:

- VSL, Delft (Netherlands), 9 March, to attend the HIGHGAS stakeholder meeting.
- LNE, Paris (France), 21 April, to give a presentation on CCQM-K90 at the KEY-VOCs mid-term stakeholders meeting.
- Edinburgh (UK), 5-9 September to present a poster on ozone cross-section measurements by gas phase titration at the Quadrennial Ozone Symposium.

- IPQ, Lisbon (Portugal), 11-14 October, to participate in CCQM GAWG meetings and workshops.

S. Westwood to:

- Lausanne (Switzerland), 12-14 March 2016, for a WADA Laboratory Expert Group meeting.
- Geneva (Switzerland), 25-27 April 2016, for a meeting of the CASCO-REMCO Joint Working Group drafting ISO 17034.
- Montreal (Canada), 20-22 June 2016, for a WADA Laboratory Expert Group meeting.
- Montreal (Canada), 5-7 December 2016, for a WADA Laboratory Expert Group meeting.

8. Guest workers in 2016

- P. Bros, LNE (France), until 31 January.
- S. Maxwell, NIST (USA), until 29 February.
- F. Arrhén, SP (Sweden), 15 January to 15 July.
- L. Wong, HSA (Singapore), 1 February to 30 April.
- W. Zhang, NIM (China), 15 February to 15 August.
- XQ Li, NIM, (China), from 15 April.
- B. Garrido, INMETRO (Brazil), from 1 September.
- M. Avila, CENAM (Mexico), 1 June to 30 November.
- T. Zhang NIM (China), from 1 November.

9. Visitors in 2016

- V. Delatour (LNE), 28 January, for discussions on the BIPM-LNE hepcidin secondment.
- D. Heikens (VSL), 29-31 September, for an ozone comparison.
- T. Suematsu (JEOL), 31 October for discussions on the BIPM-NMIJ qNMR project.
- V. Delatour, G. Matos (LNE), 7 September, for discussions on the BIPM-LNE hepcidin secondment.
- Q. Liu (HSA), 6 December, for discussions on the HbA1c hexapeptide collaboration.
- V. Delatour, G. Matos, N. Clouet (LNE), 12 December, for discussions on HbA1c.