# Director's Report on the Activity and Management of the International Bureau of Weights and Measures

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**Bureau International des Poids et Mesures** 

#### **BIPM Physical Metrology Department**

#### Director: M. Stock

#### (1 January 2017 to 31 December 2017)

#### 1. Electrical metrology

#### 1.1 Electrical potential difference (voltage)

1.1.1 AC Josephson voltage standard (S. Solve, R. Chayramy, M.-S. Kim<sup>1</sup>)

#### 1.1.1.1 On-site pilot comparison of ac quantum voltage standards

An on-going on-site comparison of dc Josephson voltage standards at 1.018 V and 10 V (BIPM.EM-K10) in which nearly 30 NMIs have participated has been organized by the BIPM for many years. As a consequence of the development of ac Josephson voltage standards in many NMIs, it is planned to extend these comparisons to ac voltages for frequencies below 2 kHz and up to 7 V rms using the differential sampling technique.

A pilot comparison was carried out at the PTB (Germany) in August 2017 within the framework of the EURAMET EMPIR project ACQ-PRO: "Towards the propagation of ac quantum voltage standards". The BIPM is an external collaborator in this project. The BIPM portable quantum voltage standard was compared to the PTB ac quantum voltage standard using an ac signal generator as a transfer standard. The comparison has been performed for low frequency sinewaves, at 1 V rms and 6.5 V rms and at frequencies of 62.5 Hz and 125 Hz. Different ac sources were used as transfer standards: a commercial calibrator and an Aivon-Mikes source.

The transfer standards were connected alternatively to each measuring system and the measurements compared. The first comparison results did not converge because of issues originating from ground loops and electromagnetic interferences. A lot of effort was required to identify the best configuration of the measurement setup using the commercial calibrator. A relative agreement  $\Delta U/U = 0.3$  ppm at 6.5 V and  $\Delta U/U = 0.2$  ppm at 0.7 V with an associated relative Type A uncertainty of 0.3 ppm and 0.9 ppm, respectively, could be obtained.

The Aivon-Mikes transfer standard had a phase stability of 20  $\mu$ rad, which is 100 times better than that of the commercial calibrator. However, despite this higher stability, it was not possible to achieve an agreement better than 1 part in  $10^6$ .

The comparison helped both participants to better understand the metrological behaviour of their own measurement setup. A number of parameters were identified which will have to be taken into account in the design of the protocol for future world-wide comparisons of ac quantum voltage standards. In the present comparison, the approach for the phase adjustment was different for the two participants, which is crucial, because a mismatch can easily lead to systematic errors in the final result.

It appears that the development of a dedicated ac source to be used as a transfer standard that can be operated on-site will be required: such an ac transfer standard source must offer better specifications than a traditional calibrator in terms of frequency and voltage stability, with a target repeatability of 0.1 ppm or better in the rms value within an hour and a phase stability of 10  $\mu$ deg. Detailed technical specifications for such a source have been developed and technical solutions will be investigated.

It is important to note that it was the third time that the new BIPM Programmable Josephson Voltage Standard (PJVS), based on the NIST PJVS, was shipped to an NMI (following visits to NMIJ (Japan) and CENAM (Mexico)) and the system has consistently demonstrated its robustness and reliability in different electromagnetic environments. Another pilot comparison is planned with the NPL (UK) for 2018.

<sup>&</sup>lt;sup>1</sup> M.-S. Kim is on secondment from KRISS (Republic of Korea) from 1 October 2017 until 30 September 2018.

#### 1.1.1.2 Indirect comparison of ac quantum voltage standard with thermal converters

An indirect comparison of low-frequency ac voltage measurements was carried out between a Programmable Josephson Voltage Standard at the BIPM and thermal converters at the NMIA (Australia). The measurements were conducted at frequencies from 62.5 Hz to 625 Hz and voltages from 0.8 V to 7 V using a sampling digital voltmeter as a transfer standard.

The sampling voltmeter was a Keysight 3458A equipped with software that allowed a full sampling of an ac signal to be performed. Whilst both the ac and dc performance of the DVM are affected by the drift in its internal reference, the drift is cancelled when the ac-dc difference is calculated, resulting in long-term repeatability of the order of 0.1  $\mu$ V/V. For this reason the ac-dc difference of the sampling DVM was chosen as the comparison quantity. The absolute levels of ac-dc difference within the frequency range of the comparison were several microvolts per volt.

At the NMIA, the sampling DVM was calibrated for ac-dc difference against NMIA primary or secondary thermal converters using the standard ac-dc transfer measurement setup. The measurement process involves sequential application of ac and positive and negative dc signals to the reference thermal converter and the DVM connected in parallel and results in the ac-dc difference of the DVM calculated as

$$\delta_{\text{ac-dc}} = (U_{\text{ac}} - U_{\text{dc}}) / U_{\text{dc}}$$
<sup>(1)</sup>

where  $U_{dc}$  is the dc voltage which, when reversed, produces the same temperature rise of the thermal converter as  $U_{ac}$ .

At the BIPM, the sampling DVM was first calibrated against the PJVS at positive and negative dc voltage. Special care was taken to reduce the effect of the dc offset of the DVM, which is prevalent in the sampling mode. The sampling DVM was then calibrated on ac by simultaneously measuring the output voltage of a precision calibrator with the sampling DVM and with the PJVS setup. From these two sets of measurements the ac-dc difference of the DVM was calculated according to (1) and compared with the corresponding NMIA value.

For ac operation, a step-wise approximation of the ac signal is formed by the PJVS and a Keysight 3458A digital multimeter is used to perform differential sampling between the PJVS voltage and the voltage of the ac calibrator synchronized with the PJVS.

The results show that the relative difference between the BIPM and the NMIA linearly increases from  $0.1 \times 10^{-6}$  to  $3 \times 10^{-6}$  as the frequency increases from 62.5 Hz to 625 Hz.

The comparison has helped ascertain the suitability of both the BIPM PJVS-based ac voltage measurement system and the sampling DVM as a transfer standard for ac voltage inter-laboratory comparisons at the lowest attainable levels of uncertainty.

#### 1.1.1.3 Investigations on different samplers

Dr Mun-Seong Kim, KRISS (Republic of Korea) joined the electricity laboratory for a one-year secondment starting on 1 October 2017, to support its work and investigations into the field of differential sampling measurements of ac voltage signals. The influence of the input bandwidth of two different samplers on the differential sampling technique used with programmable Josephson voltage standards has been investigated.

The input bandwidth of an integrating sampler, Keysight 3458A is specified to be about 100 kHz in the DCV sampling mode, while the maximum sampling rate for an NI 5922, adopting an analogue-to-digital converter with 15 MS/s, corresponds to 7.5 MHz bandwidth. Since the difference between the input bandwidths of the two samplers is huge, it is expected that they will show different responses to the input signals in a high frequency region. In the case of measurements with the large-bandwidth sampler, the RMS amplitude does not show any specific tendency with respect to the frequency. In the full sampling, for an rms voltage of 1 V an offset close to

 $30 \ \mu V$  in the data exists, which reflects a gain error of the sampler. The contribution of the offset to the differential sampling becomes irrelevant due to the much smaller signal levels.

For the low-bandwidth sampler, the RMS amplitude obtained by the full sampling deviates from the nominal input voltage with increasing frequency. The deviation around 1 kHz and 1 V is over 50  $\mu$ V. Similar behaviour was observed even in the case of the differential sampling, although the deviation is somewhat diminished compared to that in the full sampling.

#### 1.1.2 Pressure sensitivity coefficients of Zener voltage standards (R. Chayramy, S. Solve)

BIPM has been maintaining a bench of ten secondary voltage standards since 1992 which are used in an ongoing bilateral comparison of Zener voltage calibrations (BIPM.EM-K11) with National Metrology Institutes (NMIs). A determination of the temperature and pressure sensitivity coefficients of these standards was conducted at the BIPM between 1998 and 2002. At the Conference on Precision Electromagnetic Measurements (CPEM) 2016, the results of the re-evaluation of the temperature sensitivity coefficients for all standards were presented. The relative voltage corrections in Zener calibrations typically range from 1 part in 10<sup>9</sup> to 1 part in 10<sup>8</sup> and are comparable, in extreme cases, to the Type A uncertainty. In 2017 a re-evaluation of the pressure sensitivity coefficients of the BIPM Zener standards was carried out to complete this investigation.

The measurement setup, designed at the BIPM, is based on a pressure chamber, which can receive two 732B standards at the same time. The pressure of this setup can be varied by means of a small membrane pump and four solenoid-driven valves, operated by computer-controlled relays. The measurement procedure is comparable to that followed for the previous determination campaign, with the exception of the voltage outputs, which are corrected for the Zeners' temperature dependence, using the coefficients determined in 2016. The standards are operated using an additional external battery for the entire duration of the measurements, which consists of two pressure cycles from 850 hPa to 1050 hPa and back again with an increment of 50 hPa. The two output voltages of each standard are measured by a differential measurement method, using a Weston-type standard cell (1 V) and a 732A Zener (10 V) as references.

The pressure coefficient is calculated from a least-squares fit of the Zener output voltage as a function of two variables: the pressure settings inside the chamber and the measurement time. The dispersion of the measurements is taken into account in the calculation and the corresponding uncertainty of the coefficient obtained from the least-squares fit adjustment (Type A). The influence of all the other sources of uncertainty is negligible. The standard uncertainty of the measured pressure coefficients is thus dominated by the Type A uncertainty and is of the order of  $6 \times 10^{-11}$ /hPa. The newly obtained relative pressure coefficients and Type A uncertainties are in very good agreement with the previous values determined in 2002.

#### 1.2 Electrical impedance (resistance and capacitance)

#### 1.2.1 DC resistance and quantum Hall effect (B. Rolland, N. Fletcher<sup>2</sup>, P. Gournay,)

The quantum Hall resistance (QHR) was realized four times during 2017, as is required to maintain traceability for resistance calibrations and comparison services, to derive the capacitance unit, and also to prepare on-site comparisons (BIPM.EM-K12) scheduled in 2017. For the on-site comparisons, the resistance is assumed to be measured at dc, although in practice the measurement system needs to use polarity reversals to eliminate offsets and drifts. Moreover, the transportable BIPM bridge operates with sinusoidal current at 1 Hz whereas the reference for the comparison remains the BIPM Cryogenic Current Comparator (CCC) bridge, which has a full-cycle duration of 340 s, corresponding to 3 mHz ('dc'). Linking both bridges and determining frequency coefficients remains a key issue for successful comparisons. The link between the CCC and the 1 Hz bridge was

<sup>&</sup>lt;sup>2</sup> N. Fletcher left the BIPM on 31 August 2017.

made at every QHR implementation and the frequency coefficients of several standard resistors were verified at the same time.

#### 1.2.2 Calculable capacitor (P. Gournay, N. Fletcher<sup>2</sup>, B. Rolland)

Work on the calculable capacitor was suspended in 2017 as a consequence of the resignation of a staff member and the importance of focusing the remaining staff resources on services to Member States.

As a result, the current state of assembly of the calculable capacitor remains as it stood at the end of 2016. The capacitor cell is assembled with its four main electrode bars aligned. Contributions of the residual misalignments on the capacitance variation produced by the calculable capacitor are close to 2 parts in  $10^9$ . The alignment of the bars was checked in 2017 and found to be stable.

The next steps in this programme are the assembly of the two guard electrodes and their alignment with both the capacitor and the interferometer axis. This work will be addressed in 2018 when a new staff member is recruited.

#### 1.2.3 Maintenance of a reference of capacitance (P. Gournay, R. Chayramy, B. Rolland, N. Sakamoto<sup>3</sup>)

The capacitance reference group of the BIPM includes four standard capacitors kept in an oil bath thermo-regulated at 25 °C. This group is usually calibrated every 6 months against the BIPM's dc quantum Hall reference. In 2017, owing to the CCEM-K4.2017 comparison, the calibration was carried out a further two times to verify the reference capacitors before and after the comparison measurements.

The measuring chain implemented to link the dc-QHR to the reference group of capacitors includes a set of ac coaxial impedance comparison bridges, including a multi-frequency quadrature bridge. Within the framework of the services to Member States, routine maintenance of these bridges, as well as of the transfer standards equipping them, is regularly performed to achieve the best uncertainty on capacitance calibration measurements. In particular, the triennial verification of the frequency dependence of the ac-resistors of the quadrature bridge started at the end of 2017. On this occasion, a major renovation of the four-terminal-pair resistance bridge, which is used to compare those resistors to a calculable resistor, was initiated. It is anticipated that this work will be completed during the first trimester of 2018.

The reference group of capacitors was used in 2017 for the calibration of the primary standards of nine NMIs (see section 1.4) and for the key comparison CCEM-K4.2017 commissioned by the CCEM (see section 1.3.1).

#### 1.3 Comparisons of electrical standards

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

The CCEM-K4 comparison of capacitance standards was repeated in 2017 with the BIPM as the pilot laboratory. The previous (and first) time this key comparison had been carried out was between 1996 and 1999.

This repeat comparison represented the first time a CCEM comparison has been organized using the 'star scheme': all the participating NMIs calibrated a set of their own capacitors, sent them to the BIPM for comparison and verified the stability after the return of the standards. This scheme allows the comparison to be completed much faster and is more robust against potential transport problems.

Seven NMIs from four RMOs participated in the CCEM-K4.2017 comparison, in addition to the BIPM. Two capacitance values were measured, the key reference value of 10 pF and an optional one, 100 pF. The addition of this second capacitance value allows, in particular, the comparison to be performed at the 100 pF/10 pF ratio.

<sup>&</sup>lt;sup>3</sup> N. Sakamoto is on secondment from NMIJ (Japan) from 1 October 2017 until 30 September 2018.

All the participating institutes measured two of their own 10 pF standards and, for most of them, an additional two 100 pF standards were measured.

The comparison started in March 2017 with the initial series of measurements by the NMIs and finished with the return series in September 2017. In the interim between May and June 2017 the BIPM measured the standards of all the institutes. No particular issues were encountered during transportation of the standards. Out of a total of 27 travelling standards, only three showed instabilities and these were removed from the calculation of the comparison results. Redundancy of the standards of the same value sent to the BIPM by each of the NMIs allowed all the participants to remain in the comparison.

All of the participants' measurement reports were received by the pilot by the end of November 2017. Analysis of the comparison results and drafting of the comparison report started in early 2018.

### 1.3.2 BIPM participation in GULFMET.EM.BIPM-K11 (S. Solve, R. Chayramy, S. Yang<sup>4</sup>)

Dr Steven Yang (SCL, Hong Kong (China)) joined the BIPM voltage laboratory for a secondment of two months to share his experience of the calibration of secondary Zener voltage standards using Josephson voltage standards.

The SCL is the pilot laboratory for the comparison of Zener calibrations, GULFMET.BIPM.EM-K11. The BIPM is a participant and a member of the support group for this comparison and provides a link to the equivalent BIPM.EM-K11 comparison. The transfer standards, two Fluke 732B Zener standards, provided by the EMI (UAE) and the SASO-NMCC (Saudi Arabia), were measured at the BIPM from August to September 2017 during the first loop of the star-scheme comparison. They will be measured a second time at the BIPM towards the end of the comparison in early 2018.

Two important sensitivity coefficients for the standards are needed to calculate corrections due to the atmospheric pressure and the temperature of the instrument. The sensitivity coefficients for temperature and pressure of the transfer standards were determined by the SCL. The BIPM also measured these in order to cross-check the results. To our knowledge, this is the first time that determinations of Zener sensitivity coefficients have been compared by two laboratories. The BIPM determined the pressure sensitivity coefficients during the first measurement series, using the setup described in section 1.1.2. The results agreed with those of the SCL within the Type A uncertainty of 0.7 nV/hPa at 10 V and 0.07 nV/hPa at 1 V. The temperature coefficients will be determined at the BIPM during the second measurement series in 2018.

During his secondment to the BIPM, Dr Yang also compared results of Zener calibrations carried out with three different measurement setups based on three different Josephson voltage standards:

- Superconductor-Insulator-Superconductor (SIS) Josephson junctions-based array and EM-N11 analogue detector;
- Superconductor-Normal Metal-Superconductor (SNS) Josephson junctions-based array operated at 70 GHz and a Keysight 34420A nanovoltmeter;
- Superconductor-Normal Metal-Superconductor (SNS) Josephson junctions-based array operated at 18.5 GHz and a Keysight 34420A nanovoltmeter.

At 1 V, for all Zeners equivalent results were obtained with the three measurement setups. However, at 10 V, a repeatable discrepancy, in some cases larger than 100 nV, appears for some of the Fluke 732B Zeners, depending on the measurement setup. On the contrary, for the Fluke 732A Zeners the results were consistent within 20 nV for all three setups. We suspect an effect of the output impedance of the 10 V output of the 732B Zeners to be responsible for the observed discrepancies. Further investigations will be carried out in the future to confirm this hypothesis.

<sup>&</sup>lt;sup>4</sup> S. Yang was on secondment from SCL (Hong Kong (China)) from 9 January 2017 until 13 March 2017.

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## BIPM ongoing key comparisons in electricity (R. Chayramy, N. Fletcher<sup>2</sup>, P. Gournay, B. Rolland, S. Solve, M. Stock)

Two on-site comparisons of QHR systems (BIPM.EM-K12) were organized with the CMI (Czech Republic) and METAS (Switzerland). A newly fabricated bridge, based on the same principle as the one used in the first series of on-site comparisons (1993 to 1999), new thermo-regulated resistance standards (1  $\Omega$ , 100  $\Omega$  and 10 k $\Omega$ ) and other measurement instrumentation were fully operational and demonstrated to be robust and efficient for on-site use in other laboratories. In April 2017 the BIPM measurement setup was transported to the CMI. Measurements of a 100  $\Omega$  standard in terms of the conventional value of the von Klitzing constant,  $R_{K-90}$ , agreed to 6 parts in  $10^{10}$  with a relative combined standard uncertainty  $u_c = 25 \times 10^{-10}$ . Measurements of the 10 k $\Omega/100 \Omega$  and  $100 \Omega/1 \Omega$  ratios agreed to 11 parts in  $10^{10}$  with  $u_c = 22 \times 10^{-10}$  and to 33 parts in  $10^{10}$  with  $u_c = 32 \times 10^{-10}$ , respectively. The influence of the current reversal time has been systematically investigated in order to take into account possible errors due to the Peltier effect, which may be particularly large in 1  $\Omega$  standards. The on-site comparison with METAS was carried out in December 2017. The measurement report is being prepared and will be available around mid-2018.

Two bilateral comparisons of calibrations of 1  $\Omega$  and 10 k $\Omega$  standards (BIPM.EM-K13) were carried out with the SMD (Belgium) in early 2017 and the NMISA (South Africa) in mid-2017. Satisfactory results were achieved in both comparisons. For SMD the difference was smaller than the relative expanded uncertainty (95 % confidence, k = 2) of  $8.0 \times 10^{-8}$  for 1  $\Omega$  and  $6.8 \times 10^{-8}$  for 10 k $\Omega$ . The draft B report for the comparison with the NMISA has been submitted to the CCEM for approval.

The bilateral comparisons of capacitance standards of 10 pF and 100 pF (BIPM.EM-K14) carried out with NIS (Egypt), NMISA (South Africa) and NSAI-NML (Ireland, 100 pF only) were finalized during the first semester of 2017. All three comparisons led to satisfactory results.

A bilateral comparison of Zener voltage standard calibrations at 1.018 V and 10 V (BIPM.EM-K11) was carried out with NMISA (South Africa) between April and June 2017 and the comparison report was published on the KCDB database.

## 1.4 Calibrations of electrical standards (R. Chayramy, N. Fletcher<sup>2</sup>, P. Gournay, B. Rolland, S. Solve, M. Stock)

During the period from January to December 2017 the following standards were calibrated in the electricity laboratories:

1  $\Omega$ , 100  $\Omega$  and 10 k $\Omega$  resistors were calibrated for: A-STAR (Singapore, two resistors), BEV (Austria, four), EIM (Greece, four), INM (Romania, four), MSL (New Zealand, two), NMCI/ISIRI (Iran, three), SIQ (Slovenia, three), SMD (Belgium, six). Eight internal certificates were provided for other BIPM services.

1 pF, 10 pF and 100 pF capacitors were calibrated for: BEV (Austria, three capacitors), CEM (Spain, four), CENAM (Mexico, four), NIMT (Thailand, six), NPLI (India, two), NRC (Canada, two), SIQ (Slovenia, one), SMD (Belgium, four), UME (Turkey, six).

Zener voltage standards were calibrated for: BIM (Bulgaria, one Zener), SASO (Saudi Arabia, two), SIQ (Slovenia, three).

In total, the electricity laboratories issued 74 calibration certificates.

#### 2. Mass Metrology

#### 2.1 Measurement services in mass

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

The BIPM served as the pilot laboratory for the CCM Pilot Study of future realizations of the kilogram. The first objective was to test the consistency of future independent realizations of the kilogram based on different Kibble balances and x-ray crystal density (XRCD) experiments. The second objective was to test the continuity between traceability to the present definition (the mass of the International Prototype of the Kilogram, IPK) and to the future definition (the numerical value of the Planck constant). All NMIs working on primary methods were invited to participate, on the condition that they would realize the kilogram with a relative uncertainty not larger than 2 parts in  $10^7$ , equivalent to 0.2 mg. Participants were the LNE (France), the NIST (USA), the NRC (Canada), which used Kibble balances, and the NMIJ (Japan) and the PTB (Germany), which used spheres made of <sup>28</sup>Si from the International Avogadro Coordination. Two sets of 1 kg mass standards were calibrated using their realization experiments and sent to the BIPM for comparison. 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. Set 2 of the 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 calibration uncertainties for the standards of Set 1 ranged from 0.015 mg to 0.140 mg, those for Set 2 from 0.016 mg to 0.140 mg.

All measurements were carried out during 2016, the final report was published in June 2017 and a paper has been published in the *Metrologia Focus Issue* on the redefinition of the kilogram. The results for both sets are very similar: four of the participants agreed within the standard uncertainties and one result agreed within the expanded uncertainty (k = 2). The weighted mean of the five results has an uncertainty of 0.010 mg (for both sets) and is in good agreement with the calibration result traceable to the IPK.

#### 2.1.2 Calibration of 1 kg Pt-Ir prototypes and stainless steel standards (D. Bautista, M. Stock)

In June 2017 the six 1 kg Pt-Ir working standards for current use were recalibrated against the three 1 kg Pt-Ir standards for limited use. The latter had not been used since the previous comparison in March 2016. The mass differences between the three standards for limited use has remained unchanged since 2016 within the repeatability of the weighings, 0.0003 mg. The weighing scheme has been further optimized by reducing the number of weighings of each standard, without increasing the Type A uncertainty, to improve the stability of their masses.

Following the recalibration of the working standards for current use, a number of 1 kg mass standards for NMIs were calibrated.

Prototypes in Pt-Ir: No. 54 (UME, Turkey), No. 72 (KRISS, Republic of Korea), new prototype No. 107 (NPSL, Pakistan), new prototype No. 111 (KRISS, Republic of Korea).

Mass standards in stainless steel: LATU (Uruguay, one standard), RCM-LIPI (Indonesia, three standards), EIM (Greece, two standards), EMI (United Arabic Emirates, two standards), HMI (Croatia, two standards), DMDM (Serbia, one standard).

#### 2.2 Manufacturing 1 kg artefacts in Pt-Ir for NMIs (F. Boyer - BIPM Workshop, D. Bautista, M. Stock)

A new 1 kg prototype in Pt-Ir, No. 111, has been provided to the KRISS (Republic of Korea). The new prototype

for Pakistan, No. 107, which is still at the BIPM, has been recalibrated, and the mass was found to be the same as that of two years ago. The fabrication of one more prototype, which is not yet attributed to an NMI, is well advanced.

The density of two samples, from the new Pt-Ir ingot received in 2017, was determined as a quality control which led to the acceptance of this material.

A new mass comparator was purchased for the hydrostatic balance to prevent dysfunction due to the age of the one used previously and to improve the measurement process due to the larger weighing range.

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

The BIPM ensemble of reference mass standards (ERMS) reached a crucial step in 2017 when all the standards were placed inside their definitive containers with their characteristic environments. At present, the air, argon, nitrogen and vacuum networks are fully operational. In total, the ensemble is composed of twelve 1 kg mass standards plus four 1 kg stacks of disks.

All the standards of the ensemble participated in the pilot comparison of future realizations of the kilogram in 2016 (section 2.1.1) and were calibrated with respect to the IPK at the same time. All these mass comparisons were carried out in air, before the standards were placed in their definitive containers. After being placed in their containers, a first mass comparison of the standards was carried out among all the standards of the ensemble using a technique that will ensure that throughout the comparison the environment surrounding each standard is properly preserved; both during the actual weighing and during the handling phase. This process involves the use of transfer standards. These allow linking of the mass of the standards stored in different environments by weighing each of them sequentially together with the standards of each group. The masses of these transfer standards had been previously been monitored to assess their short-term stability during the passages through the different media. First mass measurements in the respective environments appear to indicate no significant mass change of the standards due to storage in the specific media. It should be noted that more conclusive confirmations will only be possible after the experiment has been in operation for several years.

Weighing of the ensemble standards has been carried out according to the protocol that was established as a consequence of the results of the 2014 extraordinary calibrations. This protocol ensures a particular hierarchy among the working standards that prevents the group from experiencing a collective wear that would be very difficult to detect.

The storage environments of the standards are permanently monitored and their chemical composition is compared to that of the source gas. These measurements are principally used as a sensitive tool to identify potential gas leaks or malfunctions in the storage systems at an early stage so that they can be fixed rapidly. The system is equipped with an automated analysis facility that compares current concentration levels with reference values and alerts the user immediately in case of a substantial mismatch.

## 2.4 Kibble balance (H. Fang, F. Bielsa, A. Kiss, S. Li, R. Chayramy, A. Dupire, B. Rolland, S. Solve, M. Stock)

The BIPM is continuing to develop a Kibble balance, also known as a watt balance, for the practical realization of the expected new definition of the kilogram in terms of the Planck constant. The apparatus is now capable of working with a mass of 1 kg and fully operational for measurements under vacuum. The day-to-day repeatability is at the  $10^{-7}$  level with a type B uncertainty of several parts of  $10^{-7}$ . The latter is still limited by the residual misalignment of the electromagnetic force and the horizontal coil velocity. The design of an improved alignment mechanism, which should allow further reduction of the type B uncertainty, is complete.

The suspension inside the mechanical support structure of the Kibble balance apparatus was significantly modified. A large 2-axis translation stage has been integrated on top of the support structure. It allows fine adjustment of the position of the whole suspension, including the moving coil, with respect to the magnetic circuit. Several gimbal blocks with two perpendicular coplanar axes have been designed and now replace the previously used, manually mounted, flexure-strips. The gimbal blocks ensure a more repeatable mechanical behaviour of the suspension. The coil assembly is connected with the suspension via a set of two gimbal blocks in order to facilitate the alignment of the electromagnetic force exerted on the coil. A stiffer device for mass loading and exchange has been integrated, which allows working with a 1 kg mass standard. This has the advantage of reducing the relative uncertainty on force measurement and the uncertainty due to misalignment. Optical rulers have replaced electrical switches to provide a continuous and high-resolution detection of the horizontal and vertical positions of the mass loading stage and the angular position of the mass exchanger. This leads to a more secure and reliable control during the loading of mass standards.

After reduction of the periodic non-linear error, the detection of the interferometric signals was further improved to increase the signal-to-noise ratio of the velocity determination. For electrical measurements, two programmable Josephson voltage standards (PJVSs) were assembled and successfully tested. The Josephson arrays, capable of providing up to 1.2 V and 2.5 V, were provided by the NIST. The first standard uses a home-made multichannel bias source based on an original current-driving scheme. The development of the source is completed. Its correct operation has been successfully tested by an indirect comparison with another PJVS standard, using a Fluke 732A voltage reference as transfer standard. The comparison showed a good agreement between the two systems within the 10 nV measurement noise. The reliability and immunity of the bias source to magnetic flux trapping during a large number of fast reconfiguration operations was verified. The second Josephson array system uses a NIST-type bias source. The system was used to calibrate the digital voltmeters which measure the induced voltage and the current.

The control and data acquisition programs have been further improved. The control of the stepper motors for the mass loading and exchange system was optimized to ensure a smooth mass loading and mass transfer in a reduced time. The data acquisition and synchronization scheme has been modified to suppress the dead time between two consecutive measurements. Moreover, this scheme allows selection of the integration time of measurements, as needed, without any modification to the hardware or software.

Significant efforts have gone into ensuring that the operation of the apparatus is as reliable as possible. Several groups of complete Planck constant measurements were carried out in air and in vacuum to check the proper operation of the apparatus and to identify the limiting contributions on measurement uncertainty. A 1 kg stainless steel mass standard was used. The apparatus was first operated using the original BIPM one-mode one measurement phase (OMOP) scheme (simultaneous weighing and moving operations). It was found that the measurements could be significantly biased up to the  $10^{-6}$  level due to the influence of the coil acceleration (resulting from vibrations) on the force signal measured by the commercial weighing cell. In addition, the type A uncertainty on the force measurement remained relatively large, even after applying appropriate filtering. The one-mode two measurement phase (OMTP) scheme was then adopted. Similarly to the conventional two-mode two measurement phase scheme (TMTP), the force and the velocity are separately measured in this scheme. Compared to the TMTP scheme, a coil current is present in both the force and velocity phases. This guarantees that the magnetic field acting on the coil, including the effect of the coil current on the field, is the same in both phases. In the force phase, the force signal is determined in static mode so that there are no influences of the coil motion. Moreover, this scheme allowed the coil velocity to be increased by a factor of five which led to a reduction of the uncertainty of both the velocity and induced voltage determinations. In each set of measurements, a current is driven successively through the first winding and then the second winding of a bifilar coil, whereas the induced voltage is measured with the winding which is free of current. The current polarity is reversed once for each winding accompanied by weighings with and without the test mass. For each configuration, a number of up and down cycles of coil displacement are carried out. Results deduced from different integration times were compared to help assess the type A uncertainty and its origins. The data

processing program is now able to compute the measurement data obtained using either the OMOP or OMTP measurement scheme. A second and independent data processing program was developed to verify the correctness of the calculations.

The measurements in vacuum showed a day-to-day repeatability of the Planck constant determination at the  $10^{-7}$  level in relative terms. The relative type B uncertainty was several parts of  $10^{-7}$ , dominated by the misalignment. Although the alignment of the electromagnetic force had been improved, it was still not optimal. It was not possible to independently align each element of the upper suspension above the coil assembly properly at the required accuracy. A new alignment procedure was established. The alignment sensitivity of each step was evaluated and optimized when possible by modelling. The design of a revised upper suspension, including several adjustable elements was completed. Parasitic coil angular velocity due to the current flowing in the velocity phase is being considered and needs to be reduced.

Studies were continued to understand and asses any potential biasing effects on the experiment. In particular, the coil-current effect on the magnetic field has been further investigated. Theoretical and experimental studies showed that the effect on the magnetic field in the velocity measurement is twice that deduced from the weighing measurement in a conventional one-mode Kibble balance. The exact size of the current effect depends on the specific geometry and dimensions of the magnetic circuit, but in general has to be taken into account at the present level of uncertainty of Kibble balances. It was demonstrated that in the OMOP Kibble balance, the averaged magnetic profiles with positive and negative coil currents have the same slope in both the weighing and velocity phases. Hence this scheme is independent of a current asymmetry between the mass-on and mass-off measurements [2]. Three experimental methods were proposed to determine the magnetic field dependence on the current in order to apply corrections in a two-mode balance [S. Li et al., *Metrologia* **55** (2018) 75]. A compensation method was also developed to minimize this effect in the BIPM-type magnet.

#### 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. The CCEM and its working groups met at the BIPM in March 2017. The CCEM organized a one-day workshop on new measurement challenges for electromagnetic metrology.

N. Fletcher<sup>5</sup> was 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 (WGs) and task groups (TGs) of the CCM. The CCM and its working groups met at the BIPM in May 2017.

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. The CCU met in September 2017. The CIPM task group for the promotion of the SI met in January 2017. The CCU organized a one-day workshop on fundamental constants and their role in the redefinition 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, P. Gournay, E. de Mirandés and S. Solve are members of the Technical Programme Committee of the CPEM 2018.

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 EURAMET and APMP Technical Committees for Electricity and Magnetism (TC-EM).

<sup>&</sup>lt;sup>5</sup> N. Fletcher left the BIPM on 31 August 2017.

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

The BIPM is an external collaborator on the EMPIR project "Towards the propagation of ac quantum voltage standards" (ACQ-PRO, 2015 to 2019).

E. de Mirandés is a member of the CODATA Task Group on Fundamental Constants and of the "Comité Science et Metrologie" of the French Academy of Sciences.

#### 5. Publications

- 1. Li S., Bielsa F., Kiss A., Fang H., Self-attraction mapping and an update on local gravitational acceleration measurement in BIPM Kibble balance, *Metrologia*, 2017, **54**(4), 445-453
- 2. Li S., Bielsa F., Stock M., Kiss A., Fang H., A permanent magnet system for Kibble balances, *Metrologia*, 2017, **54(5)**, 775-783
- 3. Stock M., Davidson S., Fang H., Milton M., de Mirandes E., Richard P., Sutton C., Maintaining and disseminating the kilogram following its redefinition, *Metrologia*, 2017, **54**(6), S99-S107
- Satrapinski A., Götz M., Pesel E., Fletcher N., Gournay P., Rolland B., New Generation of Low-Frequency Current Comparators Operated at Room Temperature, <u>IEEE Transactions on</u> <u>Instrumentation and Measurement</u>, 2017, 66(6), 1417-1424
- Khan M.S., Séron O., Thuillier G., Thévenot O., Gournay P., Piquemal F., Development of a programmable standard of ultra-low capacitance values, <u>*Review of Scientific Instruments*</u>, 2017, 88(5), 055109, https://doi.org/10.1063/1.4983337

#### **Comparison Reports**

- Gournay P., Helmy M., Raouf A., Hamed H.A.M., Eliwa Gad A., Bilateral comparison of 10 pF and 100 pF standards (ongoing BIPM key comparisons **BIPM.EM-K14.a** and **14.b**) between the NIS (Egypt) and the BIPM, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01008
- 2. Gournay P., Khoza M., Bilateral comparison of 10 pF and 100 pF standards (ongoing BIPM key comparisons **BIPM.EM-K14.a** and **14.b**) between the NMISA (South Africa) and the BIPM, *Metrologia*, 2017, **54**, Tech. Suppl., *01010*
- Gournay P., Power O., Bilateral comparison of 100 pF standards (ongoing BIPM key comparison BIPM.EM-K14.b) between the NSAI-NML (Ireland) and the BIPM, *Metrologia*, 2017, 54, *Tech.* Suppl., 01009
- Gournay P., Rolland B., Kucera J., Vojackova L., On-site comparison of Quantum Hall Effect resistance standards of the CMI and the BIPM: ongoing key comparison BIPM.EM-K12, *Metrologia*, 2017, 54, *Tech. Suppl.*, 01014
- 5. Rolland B., Fletcher N., Khumthukthit N., Jassadajin C., Bilateral comparison of 1  $\Omega$  and 10 k $\Omega$  standards (ongoing BIPM key comparisons **BIPM.EM-K13.a and 13.b**) between the NIMT (Thailand) and the BIPM, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01002
- 6. Rolland B., Fletcher N., Kučera J., Chrobok P., Vojčkov L., Bilateral comparison of 1  $\Omega$  and 10 k $\Omega$  standards (ongoing BIPM key comparisons **BIPM.EM-K13.a and 13.b**) between the CMI (Czech Republic) and the BIPM, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01007

- 7. Rolland B., Fletcher N., Power O., Bilateral comparison of 1  $\Omega$  and 10 k $\Omega$  standards (ongoing BIPM key comparisons **BIPM.EM-K13.a and 13.b**) between the NSAI NML (Ireland) and the BIPM, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01003
- 8. Rolland B., Fletcher N., Tenev A., Hadzhistoykova R., Bilateral comparison of 1  $\Omega$  and 10 k $\Omega$  standards (ongoing BIPM key comparisons **BIPM.EM-K13.a and 13.b**) between the BIM (Bulgaria) and the BIPM, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01001
- Rolland B., Fletcher N., Vlad D., Bilateral comparison of 1 Ω and 10 kΩ standards (ongoing BIPM key comparisons BIPM.EM-K13.a and 13.b) between the SMD (Belgium) and the BIPM, *Metrologia*, 2017, 54, *Tech. Suppl.*, 01015
- Schurr J., Fletcher N., Gournay P., Thévenot O., Overney O., Johnson L., Xie R., Dierikx E., Final report of the supplementary comparison EURAMET.EM-S31 comparison of capacitance and capacitance ratio, *Metrologia*, 2017, 54, *Tech. Suppl.*, 01016
- 11. Kyu-Tae Kim, Michitaka Maruyama, Stephane Solve, Louis Marais, Abdul Rashid B Zainal Abidin, Sze Wey Chua, Dennis Lee, Chun-feng Huang, Sittisak Pimsut, Seksembayev Nurlan, **APMP key comparison of DC voltage** at 1.018 V and 10 V, *Metrologia*, 2017, **54**, *Tech. Suppl.*, 01012

#### 6. Travel in 2017 (conferences, lectures and presentations, visits)

M. Stock to:

- EURAMET TC-M, Espoo (Finland), 10-12 May, to present the work of the mass laboratory and news from the BIPM.
- VNIIM, St Petersburg (Russia), 14-15 June, to give an invited talk on the revised SI at the celebrations for the 175th anniversary of VNIIM.
- *Congrès de Metrologie*, Paris (France), 19 September, to give a talk on the changes of the electrical units in the revised SI.
- EURAMET TC-EM, Caparica (Portugal), 13-14 October, to present news from the BIPM.
- APMF conference, Krabi (Thailand), 20-22 November, to give an invited talk on the revised SI.

F. Bielsa and H. Fang to:

• Technical University of Ilmenau (Germany), 28 February, to visit the Institute for Process Measurements and Sensor Techniques and to discuss technical issues on the use of high precision weighing cells.

F. Bielsa, H. Fang and S. Li to:

• NIM, Beijing (China), 23-26 October, to attend the Kibble Balance technical meeting and the workshop on absolute gravimetry.

P. Gournay and B. Rolland to:

- CMI, Prague (Czech Republic), 21-29 April, for an on-site comparison of quantum Hall resistance standards.
- METAS, Bern (Switzerland), 1-9 December, for an on-site comparison of quantum Hall resistance standards.

B. Rolland to:

• Congrès de Metrologie, Paris (France), 19 September, to give a talk on the "On-site BIPM key comparison of quantum Hall effect resistance standards".

S. Solve to:

- ESM-Douai (France), 17 February, to give a talk on the role of the BIPM to the students of the promotion 2016-2017.
- PTB (Germany), 13-26 August, to carry out a pilot study in AC voltage using the differential sampling method.
- LNE-Trappes (France), 12 December to give a talk on the BIPM activities in voltage metrology.

#### 7. Visitors in 2017

- Mr Rami Tzabar, Editor CrowdScience, BBC World Service, to visit the Kibble balance, 17 January.
- Ed Watkins, producer of the film "The last artifact", to visit the Kibble balance, 19 January.
- Dr Barry Wood (NRC), to visit the Kibble balance and to discuss the work in the electricity laboratories, 20 January.
- Dr François Piquemal (LNE), to visit impedance metrology, 2 February.
- Dr Bobjoseph Mathew (METAS, head of legal metrology division), to visit the Physical Metrology Department, 3 February.
- 18 participants of the EURAMET "TC Leadership" course, to visit the Kibble balance and the electricity laboratories, 7 and 8 February.
- Mr P. Scheibenreiter (BEV), to visit impedance metrology and the Kibble balance, 16 February.
- Dr Ilya Budovsky (NMIA), to visit the Kibble balance, the calculable capacitor, and to discuss future collaborations in voltage metrology, 19 20 March.
- Dr Gregory Kyriazis (INMETRO) to visit the voltage laboratory, 20 March.
- Dr Carlos Sanchez (NRC) and Dean Jarrett (NIST), to visit the Kibble balance, 20 March.
- Dr Yan Yang and Dr Wang (NIM), to visit impedance metrology and the Kibble balance, 12 April.
- Dr Véronique Dehant (Royal Observatory of Belgium), to visit the Kibble balance, 19 June.
- Dr Nadine de Courtenay (Univ. Paris Diderot) and Dr Patrice Delon, to visit the Kibble balance, 26 June.
- Chinese delegation from Guangdong Provincial Bureau, to visit the Kibble balance, 3 July.
- Dr Frederic Overney and Dr Alepth Hain Pacheco (CENAM), to visit the impedance metrology laboratory, the calculable capacitor and the Kibble balance, 5 July.
- Dr Huang Lu (NIM), to visit the impedance metrology laboratory and calculable capacitor, 13 July.
- Dr Kwang-Cheol Lee (KRISS), to visit the Kibble balance and discuss technical issues, 24 July.
- Prof. Dr. Pavel Neyezhmakov (COOMET Vice-President, General Director of Nat. Science Centre "Institute of Metrology") and Prof. Leonid Vitkin (Director Technical Regulation Dept. of Ministry of Economy and Trade) Ukraine, to visit the Kibble balance, 25 July.
- Ed Watkins and his film team to do some filming for the film "*The last artifact*" in the laboratory for the reference mass standards, 27 28 July.
- Dr Payagala (NIST), to visit the impedance metrology laboratory and calculable capacitor, 18 September.

- Dr Alain Rüfenacht (NIST), to upgrade the software which operates the BIPM PJVS, 27 September
- Dr Alejandra Tonina (INTI), to visit the voltage laboratory, impedance metrology laboratory and calculable capacitor, 27 September
- Dr Jan Kucera (CMI), to visit the impedance metrology laboratory, 28 September.
- Mrs. Honghui Li (NIM) to visit the voltage laboratory, 28 September
- Mr Laporte (CNAM), to visit the Kibble balance, 14 November.
- Mr Olman Ramos Alfaro (Lacomet), to visit the Kibble balance, 16 November.
- 24 participants of "Sound beginning in the CIPM MRA" course, to visit the electricity laboratories and the Kibble balance, 20 and 21 November.
- Dr Ilya Budovsky (NMIA), to carry out a bilateral comparison study in AC voltage metrology, 20 and 22 November.
- Dr Hugo Gasca Aragon (CENAM), to discuss on kilogram redefinition and future mass dissemination, 7 December.

#### **BIPM Time Department**

### Director: E.F. Arias (retired end of November 2017), P. Tavella (since 1 December 2017) (1 January 2017 to 31 December 2017)

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

(E.F. Arias, A. Harmegnies, J. Gonçalves, Z. Jiang, , G. Panfilo, G. Petit, L. Tisserand, and P.Tavella)

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 time scale; 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 <a href="https://www.bipm.org/en/bipm-services/timescales/time-ftp.html">https://www.bipm.org/en/bipm-services/timescales/time-ftp.html</a>.

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 Department Data Base'. Time Department services 'BIPM Time can be accessed at https://www.bipm.org/en/bipm/tai/.

The *BIPM Annual Report on Time Activities for 2017*, volume 12, provides comprehensive data for 2017 and is available on the BIPM website at https://www.bipm.org/en/bipm/tai/annual-report.html, as well as previous annual reports,

#### 2. Algorithms for time scales (G. Panfilo, G. Petit, A. Harmegnies, J Gonçalves, 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, the behaviour of UTC is routinely and carefully monitored to detect and fix unexpected anomalies. In late 2016 and early 2017 a frequency drift was observed that affected EAL behaviour. A revision of the algorithm has been undertaken to fix the problem, which could have affected the performance of UTC. The studies showed that the algorithm's weakness was due to the length of the window over which past data was used for estimating the frequency drift of the atomic clocks participating in UTC. After appropriate tests and evaluations, the window length was optimized and the problem was resolved. The time scale is now free from the frequency drift and is stable and accurate, as is required for an international reference.

The algorithm for the evaluation of the uncertainties of [UTC-UTC(k)], reported in Section 1 of *Circular T*, has been revised and the work is being concluded. With the proposed formalism, the correlations among different measures can be taken into account, and the uncertainty of the PTB (pivot laboratory) is more realistic. The uncertainty of uncalibrated laboratories now equals to a more realistic value (arbitrarily fixed to 20 ns) without affecting the uncertainties of the ensemble and the calibrated laboratories. The proposed algorithm represents a first step towards the use of redundant time links in UTC calculation.

Until now only two independent techniques (GNSS and TWSTFT) were used for UTC calculation. A complete set of redundant measurements is available but is not yet used. The core concept is to introduce a redundant time link system to take full advantage of all the available measurements.

This is a significant evolution in time link calculation introducing important changes in terms of algorithms and checks of the results. For this reason, it was decided to proceed step-by-step to control each detail to achieve a robust solution. The first step of the planned work consists in the development of the algorithm for redundant time links and in its application to the uncertainty evaluation of the offset [UTC-UTC(k)].

#### 2.1 EAL stability

A subset of 89 % 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 32 % of participating clocks in 2017. The weighting procedure involved in time scale computation guarantees the long-term stability of EAL. To prevent domination of the scale by a small number of very stable clocks, a maximum relative weight is used each month, which depends on the number of participating clocks. On average, about 13 % of participating clocks were at the maximum weight during 2017; almost all of which 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; 45 % of the contributing hydrogen masers were, on average, at the maximum weight in 2017, 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 2017, 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, the relative deviation of the duration of the TAI scale interval from the SI second, as produced on the rotating geoid, by primary and secondary frequency standards is estimated together with its uncertainty. Since January 2017, individual measurements of the TAI frequency have been provided by eight primary frequency standards, including six caesium fountains (SYRTE FO2, IT CSF2, SU CSFO2, PTB CSF1, PTB CSF2 and NIM 5), and by a rubidium secondary frequency standard (SYRTE FORb). A few measurements by strontium lattice secondary frequency standards (SYRTE Sr2 and SrB) have also been reported for the first time in 2017, but were not used in estimating the duration of the TAI scale unit following advice by the working group on PSFS. 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 2017, 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  $-1.46 \times 10^{-15}$  to  $+0.39 \times 10^{-15}$ , with a maximum standard uncertainty of  $0.28 \times 10^{-15}$ . A steering correction of  $+0.3 \times 10^{-15}$  has been applied each month from January to May 2017. A procedure has been developed to estimate the duration of the TAI scale unit on any interval. This is provided upon request to laboratories developing secondary standards in order for them to get optimal traceability to the SI second.

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

TAI is computed almost in 'real-time' and is subject to operational constraints; as a result, it does not provide an optimal realization of TT, the Terrestrial Time, i.e. 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 2017, known as TT(BIPM16), valid until December 2016, which had an estimated accuracy of about 2-3 parts in 10<sup>16</sup> over recent years. Moreover, the Time Department provides a formula to extend TT(BIPM16) 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.

#### 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 calibration of receivers at the Astrogeodynamical Observatory of the Space Research Centre (AOS, Borowiec, Poland) which provides data for GLONASS, the large offset that used to exist between UTC and GLONASS time was corrected on 1 March 2017.

### **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 CCTF Working Group on Primary and Secondary Frequency Standards (WGPSFS). These Working Groups seek to encourage comparisons, knowledge-sharing among laboratories, the creation of better documentation, the use of high-accuracy primary frequency standards (Cs fountains) and secondary frequency standards in 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 has been successfully carried out and they have been used for the elaboration of the new recommended frequency list examined by the WGFS at meetings on 3-4 May and 6 June 2017. The new frequency list was adopted by the CCTF at its 21st meeting in 2017.

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

Since January 2012 the LNE-SYRTE has regularly reported frequency measurements of the Rb microwave transition obtained with a double Cs-Rb fountain (FORb). In addition, measurements of two Sr optical lattice clocks were reported for the first time by the LNE-SYRTE in October 2016. After approval by the CCTF Working Group on Primary and Secondary Frequency Standards, they were published in *Circular T* in February 2017.

## **4. Time links used for UTC** (E.F. Arias, A. Harmegnies, Z. Jiang, J. Gonçalves, G. Panfilo, G. Petit and L. Tisserand)

At the end of 2017, 80 time laboratories supplied data to the BIPM for the calculation of UTC. The laboratories are equipped with GNSS receivers and some of them also operate a two-way satellite time and frequency transfer (TWSTFT) station.

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 quality of the International GNSS Service (IGS) products (clocks and IGS time). Clock comparisons are obtained by 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 are processed monthly using the Precise Point Positioning (PPP) technique. The Time Department also regularly computes combined GPS/GLONASS links, some of which are used for *Circular T*.

Thirteen laboratories operating TWSTFT stations officially submitted data in 2017 for use in the computation of UTC, representing 16 % of the time links. The number of TW links decreased during the year with the lack of satellite service for laboratories in the Asia Pacific region. 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 were 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 updated to cover all GNSS and implemented in some receivers and these now automatically produce formatted code results for GPS or for all available GNSS. These newly available data will be used in multi-GNSS system time links after validation of such processing.

The NRCan's PPP software is used for the time link calculation using phase and code measurements. 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 and is being further improved 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 has been used regularly to compare IPPP results to the few available optical fibre links.

Comparisons of the different links available between pairs of contributing laboratories, are computed and published monthly on the Time Department's ftp server.

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

The 25th annual meeting of the CCTF Working Group on Two-Way Satellite Time and Frequency Transfer (WGTWSTFT) was held on 18-19 May 2017 at the National Time Service Center (NTSC) of the Chinese Academy of Sciences in Xi'an (China).

Two major issues were discussed at the meetings concerning the time link calibrations carried out by the laboratories and the realization of a pilot project involving the BIPM and the WGTWSTFT.

The first topic was presented by each participating station in its annual report. From 2017, all the TW laboratories have been asked to report the TWSTFT and GNSS calibrations performed in their laboratories to the WGTWSTFT to better facilitate tracking and monitoring of the UTC calibrations and their long-term variations. This request was one of the outputs of the Task Group, established by the 2016 TW WG meeting at NIST, to study the long-term comparison of GPS and TWSTFT links. The Task Group was discontinued following a conclusion report given by the Chair of the WG, Victor Zhang.

The second issue is to summarize the status of the BIPM-WGTWSTFT pilot project on the use of the Software-Defined Radio (SDR) receiver technique in UTC generation. Dr Jiang of the BIPM gave the status report. The results are encouraging and the WGTWSTFT decided to make a Recommendation to the CCFT meeting in June 2017 to use SDR in UTC computation. An *ad hoc* group composed of TL, NIST, OP, PTB and BIPM experts was set up to handle the practical issues for the implementation of SDR in UTC computation.

The *ad hoc* group met at the 31st European Frequency and Time Forum (EFTF) in Besancon (France) in July 2017 and a roadmap was established. The first step is to use the SDR link as the backup UTC link: this has been carried out since October 2017.

The TWSTFT technique is currently operational in ten European, two North American and several Asia-Pacific time laboratories. Most of the TWSTFT links had been used in the computation of UTC in 2017 in Europe, USA and Asia; they are often 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). In the long-term, the BIPM plans to develop studies on this technique, which could be used to validate less accurate time links and their calibrations.

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) with whom a permanent cooperation is established 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 continued the second calibration campaign of the "Group 1" laboratories in SIM and COOMET in 2017, following the planned periodicity of two years. Regional calibration trips concerning "Group 2" laboratories continued in the RMOs in 2017 in accordance with the *BIPM Guidelines* and the results have been implemented in *Circular T*. By repeatedly applying this new procedure, time transfer accuracy is expected to improve by a factor of at least two 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 (<u>www.bipm.org/jsp/en/TimeCalibrations.jsp</u>), 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. This Study Group has made progress on this subject and has been merged into the CCTF WGATFT.

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

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

Results of the key comparison in time, <u>CCTF-K001.UTC</u>, 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 2017 was 63, 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 <u>CCL-K11</u> 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 2017, BIPM staff members supported the key comparison on issues relating to the development of the measurement campaigns and reporting.

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

Since January 2013 the Time Department has published a UTC rapid solution 'UTCr', that is, daily values of [UTCr - UTC(k)] evaluated on a weekly solution on one-month batches of data. More than 50 laboratories that are traceable to UTC contribute to UTCr, which together represent some 70 % of the clocks that participate in UTC.

To improve the stability of rapid UTC, a new weighting algorithm was introduced. As a result, since July 2017, the weekly UTCr solution is consistent within 2 ns peak to peak with the UTC values published monthly in BIPM *Circular T*. The results (<u>ftp://ftp2.bipm.org/pub/tai/Rapid-UTC</u>) 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. Revision of the definition of UTC (F. Arias, P.Tavella)

The BIPM has actively participated in discussions about a possible revision of the definition 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 October 2017.

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, drafted a text of the recommendation discussed and endorsed by the CCTF in June 2017 and successively by the CIPM in November 2017. This recommendation will be submitted to the CGPM in November 2018.

#### 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 to 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.

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. A first meeting of this working group was held in May 2017 and 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<sup>-18</sup> in the context of international timescales A review and practical results; *J. Geodesy*, 2017, doi:10.1007/s00190-017-1075-1.
- 2. Hachisu H., Petit G., Nakagawa F., Hanado Y., Ido T, SI-traceable measurement of an optical frequency at the low 10<sup>-16</sup> level without a local primary standard, *Opt. Express* **25**, 8511-8523, 2017.
- 3. Jiang Z., Zhang V., Parker T.E., Yao J., Huang Y.-J., Lin S.-Y., (2017) Accurate TWSTFT time transfer with indirect links, *Proc. PTTI 2017*, 30 January 2 February 2017, Monterey, California, USA.
- Jiang Z., Arias F.E., (2017) Pilot study on the validation of the Software-Defined Radio Receiver for TWSTFT -- BIPM contribution to the validation of the SDR for TWSTFT, *Proc. PTTI 2017*, 30 January - 2 February 2017, Monterey, California, USA.
- 5. Jiang Z., Matsakis D., Zhang V., (2017) Long-term instability in UTC time links, *Proc. PTTI 2017*, 30 January 2 February 2017, Monterey, California, USA.
- 6. Jiang Z., Lin S.-Y., Tseng W.-H., (2017) Fully and optimally use the redundancy in a TWSTFT network for accurate time transfer, *Proc. ETFT-IFCS2017*, 9-13 July Beçsançon, France, 367-380.
- 7. Lin S. Y., Jiang Z., (2017) GPS All in View Time Comparison using Multireceiver Ensemble, *Proc. ETFT-IFCS2017*, 9-13 July Beçsançon, France, 362-365.
- 8. Liang K., Zhang A., Yang Z., Tisserand L., Jiang Z., Petit G., Arias E.F., (2017) Experimental research on BeiDou time transfer using the NIM made GNSS time and frequency receivers at the BIPM in Euro-Asia link, *Proc. ETFT-IFCS2017*, 9-13 July Besançon, France, 788-797.

- Liang K., Zhang A., Yang Z., Wang J.-L., Lin H.T., Wang Y., Yang F., Yang J., Gotoh T., Cai Z., Jiang Z., Mou W., (2017) UTC time link calibration in the frame of APMP Campaigns in 2014-2016, Proc. ETFT-IFCS2017, 9-13 July Beçsançon, France, 826-834.
- 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.
- 11. Müller J., Dirkx D., Kopeikin S.M., Lion G., Panet I., Petit G., Visser P.N.A.M., High performance clocks and gravity field determination, *Space Sci. Rev.*, 2018, **214**:5.
- 12. Petit G., Leute J., Loyer S., Perosanz F., Sub 10<sup>-16</sup> frequency transfer with IPPP: Recent results, Proc. 2017 Joint EFTF/IFCS, 2017, 784-787.
- 13. Wielgosz R., Arias F., Los Arcos J.-M., Stock M., Milton M., News from the BIPM laboratories 2016, *Metrologia*, 2017, **54**, 148-151.

#### **BIPM** publications

- 14. *BIPM Annual Report on Time Activities for 2017*, 12. available only at http://www.bipm.org/en/bipm-services/timescales/time-ftp/annual-reports.html
- 15. <u>Circular T</u> (monthly)
- 16. 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<sup>6</sup> 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 an associate member of the IGS and member of the IGS Working Groups on Clock Products and on Bias Calibration. He represents the BIPM at the United Nations International Committee on Global Navigation Satellite Systems (ICG), where he chairs 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.

P. Tavella is Vice Chair of the EFTF Executive Committee, she is a member of the AdCom of the IEEE UFFC Society, a member of the Technical Advisory Committee of International Union of Radio Science (URSI) Commission A, and a member of the ION PTTI (Precise Time and Time Interval) Advisory Committee.

#### 13. Travel in 2017 (conferences, lectures and presentations, visits)

E.F. Arias to:

- Monterey (USA), for PTTI Tutorials on 30 January to give a lecture and for the PTTI 2017 from 31 January to 2 February to chair a session and participate to CCTF Working Group meetings;
- Madrid (Spain); 19-29 March, for lecturing at PhD courses in Engineering, Mathematics, Statistics and Research, Universidad Complutense y Politécnico de Madrid;
- San Fernando (Spain), in 8-9 March, the Technical Committee on Time and Frequency of EURAMET to give reports on BIPM activities and to coordinate calibrations;
- Teddington (UK), in 23-24 February, for the meeting of the Programme Expert Group on Digital;
- London (UK) for the UTC Traceability Workshop, 28 February;
- Beijing (China) for a visit to NIM and validation of facilities for the key comparison of absolute gravimeters 2017, 14-19 May;
- Shanghai (China) for the 8th China Satellite Navigation Conference, invited to give a lecture, 22 25 May;
- Besancon (France), 9-13 July, for the EFTF-IFCS 2017 congress and related CCTF WG meetings;
- Montreal (Canada), 19-26 August, for attending the General Assembly and Scientific Symposium of the International Union of Radio Science, chairing a session and presenting a paper;
- Xian (China), 7-8 September, to the 25th Annual meeting of the CCTF WG on TWSTFT and to give oral reports on the WG activities;
- San Fernando (Spain), 15 September, for participating to the inauguration of the new building of ROA devoted to Time and Frequency metrology
- Teddington (UK), 11-12 October, for the meeting of the Programme Expert Group on Digital;

<sup>&</sup>lt;sup>6</sup> Very Long Baseline Interferometry (VLBI)

- La Plata (Argentina), 25 October for the workshop on time and frequency at the Argentinian-German Geodetic Observatory (AAGO), to give a lecture;
- Buenos Aires (Argentina) 26-27 October for the technical peer review of the time and frequency activities at the Instituto Nacional de Tecnologia e Industria (INTI);
- Geneva (Switzerland), 25 October 1 November, for the meeting of Study Group 7 and Working Party 7A at the ITU;
- Valencia (Spain), 27 October, for the 22nd meeting of the Galileo Scientific Advisory Committee (GSAC).

P. Tavella to:

• Bruxelles (Belgium), 23 November, for a meeting of the Executive Committee of the European Time and Frequency Forum (Vice Chair).

Z. Jiang to:

- Besancon (France), 9-13 July, for the ETFT-IFCS 2017 meeting and the meeting of the *ad hoc* for the SDR;
- Xian (China), 7-8 September, to the 25th Annual meeting of the CCTF WG on TWSTFT and to give oral reports on the WG activities and the status of the pilot project on SDR;
- NIM, Beijing (China), 17-25 October, for the International Comparison of Absolute Gravimeters 2017 (ICAG 2017), co-chair the workshop on ICAG and Watt Balance held during the comparison.

G. Petit to:

- San Fernando (Spain), 8-9 March, to attend the Euramet TC-TF meeting, with one presentation;
- Hannover (Germany), 15-16 May, to attend the meeting of the IAG Working Group 2.1 on relativistic geodesy, as co-chair of the WG;
- Paris (France), 30 May, to attend the Journées CNES "GNSS and science", with one presentation;
- Besançon (France), 27-28 June, to give two lectures at the European Frequency and Time Seminar;
- Paris (France), 5-7 July, to attend the IGS Workshop, to chair one session and give one presentation;
- Besançon (France), 10-13 July, to attend the EFTF 2017 meeting, to chair one session and give two oral presentations;
- Paris (France), 13 October, to attend the *Journée thématique* "50<sup>th</sup> anniversary of the definition of the second" to give one oral presentation.

#### 14. Visitors, secondees in 2017

- H. Hachisu, NICT (Japan), for a discussion on the use of Sr lattices for UTC, 13 January.
- K. Liang, NIM (China) to work on calibration and time transfer with the Beidou system, 9 January to 20 December.
- H. Yuan, NTSC (China), for discussions on the BeiDou system, 4 July.
- J. Leute, CNRS (France), to develop methods and tools for the IPPP technique, 1 Aug 2017- 31 July 2019
- D. Matsakis, USNO (USA), for a discussion on uncertainty of [UTC-UTC(k)], 11 September.

### BIPM Ionizing Radiation Department Director: J-M Los Arcos (replaced by S Judge on 1 September 2017) (1 January 2017 to 31 December 2017)

#### 1. X- and $\gamma$ -rays (D.T. Burns, C. Kessler, S. Picard<sup>1</sup> and P. Roger)

#### 1.1 Dosimetry standards and equipment

About half of all cancer patients receive some type of radiation therapy during treatment. The effectiveness of the radiotherapy relies on accurate measurements of the radiation dose delivered. To achieve the required accuracy, the international traceability scheme is short so that there are as few steps as possible from the primary standard to the user: the BIPM primary standards are used as the reference standard for NMIs and DIs, who in turn calibrate clinical dosimeters directly or through a third party. There is an established rolling programme of comparisons, in which those NMIs or DIs with primary standards, carry out comparisons with the BIPM standards once every ten years.

The BIPM supports a series of eight ongoing comparisons, <u>BIPM.RI(I)-K1</u> to <u>BIPM.RI(I)-K8</u>, in the dosimetry programme. There have been two significant changes to the series during the year. First, a new comparison service was launched for absorbed dose to water for medium-energy x-rays (BIPM.RI(I)-K9). Approval to launch the service was given at the CCRI(I) meeting in June 2017, after reviewing the detailed analysis of results from a comparison with the PTB (Germany). The impact of the new service is that it will reduce the uncertainties in clinical reference dosimetry for medium-energy x-rays from 3 % to 0.7 %.

Second, the comparison service for dosimetry for high energy photons (BIPM.RI(I)-K6) has been improved. An agreement was signed with the *Commissariat à l'énergie atomique et aux énergies alternatives* (CEA) to use the LINAC at the new DOSEO facility, located near to the BIPM. The aim of the agreement was to enable comparisons to be run on demand, following the same process as other comparisons. After initial scoping studies, the beam from the LINAC was characterized and the BIPM primary standard was set up at the DOSEO site. The first comparison was carried out in November with the KRISS (Republic of Korea), and the intention is to publish the results in 2018; comparisons with other NMIs/DIs will follow.

Primary measurements and reference chamber calibrations have continued in all the reference x- and  $\gamma$ -ray beams. The programme to update the dosimetry services' computer hardware and software continued, new software for the high-voltage regulator was developed and validated. Additional lead shields were installed in the laboratory to comply with radiation protection regulations.

#### 1.2 Dosimetry comparisons

During 2017, twelve 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 KRISS, NIM (China), SMU (Slovakia), NPL (UK) and the VSL (the Netherlands).

In addition, one high-energy absorbed-dose-to-water comparison BIPM.RI(I)-K6 was carried out with the KRISS using the DOSEO accelerator facility.

Eight comparison reports were approved by the CCRI (I) and sent for publication in *Metrologia Technical Supplement* for the NIM (China) (two reports), VSL, NIST (USA), SCK.CEN (Belgium), SMU (two reports) and the IST-LPSR (Portugal) (see § 3).

#### 1.3 Characterization of national standards for dosimetry

Twenty-nine characterizations of twenty national secondary dosimetry standards were carried out for the IST-LPSR, NIS (Egypt), CRRD (Argentina), SSM (Sweden) and the IAEA. 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 2017, for the radiotherapy level in the <sup>60</sup>Co beam and for the radiation protection level in the <sup>137</sup>Cs beam.

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

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

International equivalence of radionuclide standards is equally important, driven by an expansion in molecular radiotherapy for cancer treatment and demands for accurate measurement for environmental protection, for trade (food safety) and for nuclear security. The BIPM does not hold the primary standards in this field; instead, the international measurement system is based on very stable, very precise, comparators to enable the equivalence of primary standards from NMIs/DIs to be demonstrated.

The cornerstone of the international measurement system for radionuclide metrology is the *Système International de Référence* (SIR) which enables NMIs/DIs to demonstrate equivalence of radioactivity standards of gammaemitting radionuclides on demand, avoiding the need to organize large scale comparisons and associated problems with shipping radioactive sources. The SIR comprises two ionization chambers and a set of <sup>226</sup>Ra sources, and it has been shown to be stable to better than 0.1 % over 40 years.

The SIR is suitable for long-lived gamma-emitters; this covers many of the radionuclides needed for environmental monitoring, for instrument calibration and for some medical applications. Shorter-lived radionuclides, such as those used for Positron Emission Tomography, cannot be measured using this approach. A transportable version of the SIR was therefore developed in 2009, based on a scintillation detector rather than an ionization chamber; the 'SIRTI' is used to carry out measurements of standards of radionuclides with short half-lives on site at an NMI/DI.

The SIR and SIRTI are used for the ongoing comparisons BIPM.RI(II)-K1 and BIPM.RI(II)-K4 respectively. Both activities are subject to the BIPM Quality Management System and the systems underwent a successful internal audit in December 2017.

There were some disruptions to the availability of the SIR (and to other facilities) during the year while actions were being taken to ensure that the BIPM complied with the increasingly stringent local regulations on the use of sealed radioactive sources.

#### 2.1.1 SIR measurements and reports

In 2017, the SIR received twelve ampoules covering eleven different radionuclides, (<sup>54</sup>Mn, <sup>60</sup>Co, <sup>68</sup>Ge, <sup>113</sup>Sn, <sup>131</sup>I, <sup>133</sup>Ba, <sup>137</sup>Cs, <sup>152</sup>Eu, <sup>166</sup>Ho, <sup>166m</sup>Ho and <sup>231</sup>Pa) from six laboratories: CMI-IIR (Czech Republic), LNE-LNHB (France), NMISA (South Africa), NPL, PTB and TAEK (Turkey) (who used the SIR for the first time). It was also the first time that <sup>166</sup>Ho and <sup>231</sup>Pa were measured in the SIR. These measurements should enable a link to the EURAMET.RI(II)-K2.Ho-166 and CCRI(II)-K2.Pa-231 comparisons. It is worth noting that although the SIR has been running for more than 40 years, it is still the basis of a dynamic comparison programme, with new participants joining and new radionuclides added regularly.

In preparation for the CCRI meeting in June 2017, updates to KCRVs were calculated and put forward for <sup>57</sup>Co, <sup>59</sup>Fe, <sup>109</sup>Cd, <sup>131</sup>I, <sup>134</sup>Cs, <sup>177</sup>Lu, <sup>223</sup>Ra and <sup>18</sup>F, including results linked to the SIRTI for the first time. The CCRI(II) decided not to update the KCRV for <sup>67</sup>Ga as the spread in the results was greater than would be expected from

the uncertainty budgets stated by participants. The reason for this was investigated, including checking the correction factors used, but some of the information was not available as some entries date back to the 1980s. The CCRI(II) therefore decided that participates should be asked for more details about measurement methods and correction factors when sending <sup>67</sup>Ga standards in the future to enable the reason for the spread in the results to be identified, in order to re-evaluate a robust KCRV.

Updated final reports of two BIPM.RI(II)-K1 comparisons were published in *Metrologia Technical Supplement* for <sup>60</sup>Co and <sup>241</sup>Am, covering, in total, seven independent SIR results. There are 43 SIR results in the draft A or B stage awaiting publication in the BIPM key comparison database (KCDB). Reporting forms for three measurements are yet to be received from participating NMIs.

#### 2.1.2 Extension of the SIR to short-lived radionuclides (SIRTI)

Although the comparator for short-lived radionuclides (SIRTI) can be used as an independent instrument, linking the results to the SIR combines measurements taken on both the SIR and the SIRTI; this linking procedure enables an NMI/DI to demonstrate equivalence of national standards to all the NMIs/DIs that have participated in either SIR or SIRTI comparisons.

During 2017, the linking factor was determined for <sup>64</sup>Cu ( $T_{1/2} = 13$  h) for the first time, by measuring samples of the radionuclide in solution from the CNRS/CEMHTI (France) and from the NPL in both instruments. The linking factor was then used to evaluate the result from the first <sup>64</sup>Cu SIRTI comparison (<u>BIPM.RI(II)-K4.Cu-64</u>) carried out at the NIST in 2016, and it was confirmed that the NIST result agrees with the KCRV within the standard uncertainty. The result of the <sup>18</sup>F ( $T_{1/2} = 1.8$  h) (<u>BIPM.RI(II)-K4.F-18</u>) comparison carried out at the NIST also agreed with the KCRV within the standard uncertainty. Use of the SIRTI has therefore enabled the NIST to demonstrate equivalence of primary standards of two radionuclides widely used for Positron Emission Tomography – this would not have possible without the SIRTI. The results were presented at the Life Science Working Group session of the 2017 International Conference on Radionuclide Metrology in Buenos Aires (Argentina) and have been published in the *Metrologia Technical Supplement*.

The SIRTI comparisons (BIPM.RI(II)-K4) which took place on-site at the NMISA (South Africa) for <sup>18</sup>F and <sup>99m</sup>Tc in 2015 were published in the *Metrologia Technical Supplement*.

In 2017, SIRTI comparisons took place on-site at the NRC (Canada) for <sup>18</sup>F, <sup>64</sup>Cu, <sup>99m</sup>Tc ( $T_{1/2} = 6$  h) and, for the first time, for <sup>11</sup>C ( $T_{1/2} = 20$  min), and at the ANSTO (Australia) for <sup>18</sup>F, <sup>64</sup>Cu and <sup>99m</sup>Tc. These comparisons have the status of "Report in progress, draft A" and "Measurements completed", respectively.

#### 2.1.3 Extension of the SIR to pure beta emitters

Pure beta-emitting radionuclides are also important for medical applications and for environmental protection. Work has continued on the development of a comparator with a similar specification for stability and reproducibility for such radionuclides. Two secondees (one from NIST, one from NIM) worked with BIPM staff to continue the study of a technique based on liquid scintillation counting, the "Universal Cross-Efficiency technique", which is similar to the SIR in that only the half-life of the radionuclide needs to be known. Extensive reproducibility tests were carried out to check whether the proposed method met the specification – samples of four different radionuclides were measured in three different liquid scintillation cocktails, using two different volumes and five levels of quench. The results were inconclusive and the differences between the measurements were outside the specification. Independent studies have shown that the results may depend on the instrument used and on the chemistry of the scintillation cocktail. Given the potential impact of a comparator for pure beta emitters, the work will continue in 2018 following the recruitment of a new member of staff who will investigate alternative approaches.

#### 2.2 Radioprotection and regulatory activities.

As mentioned above, regulations concerning the use of ionizing radiation are becoming more stringent. During 2017, significant administrative and practical work was necessary to ensure compliance.

#### 3. Publications

- 1. Andreo P., Burns D.T., Nahum A.E., Seuntjens J. and Attix F.H., Fundamentals of Ionizing Radiation Dosimetry ISBN: 978-3-527-40921-1 (Wiley-VCH Verlag GmbH & Co: Weinheim, Germany).
- 2. Burns D., Kessler C., Jinjie W., Peiwei W., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the NIM, China and the BIPM in medium-energy x-rays, *Metrologia*, 2017, **54**, Tech. Suppl., 06008.
- 3. Burns D.T., Kessler C., O'Brien M., Minitti R., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the NIST, USA and the BIPM in medium-energy x-rays, *Metrologia*, 2017, **54**, Tech. Suppl., 06006.
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- 5. Kessler C., Burns D., Cardoso J., Key comparison BIPM.RI(I)-K1 of the air-kerma standards of the IST-LPSR, Portugal and the BIPM in <sup>60</sup>Co gamma radiation (to be published in 2018).
- 6. Kessler C., Burns D., Durný N., Key comparison BIPM.RI(I)-K5 of the air-kerma standards of the SMU, Slovakia and the BIPM in <sup>137</sup>Cs gamma radiation (to be published in 2018).
- 7. Kessler C., Burns D., Durný N., Key comparison BIPM.RI(I)-K1 of the air-kerma standards of the SMU, Slovakia and the BIPM in <sup>60</sup>Co gamma radiation (to be published in 2018).
- 8. Picard S., Kessler C., Roger P., Burns D.T., Wang K., Wang Z., Fan Y., Jin S., Zhang J., Key comparison BIPM.RI(I)-K6 of the standards for absorbed dose to water at 10 g cm<sup>-2</sup> of the NIM, China and the BIPM in accelerator photon beams, *Metrologia*, 2017, **54**, Tech. Suppl., 06009.
- 9. Picard S., Burns D.T., Roger P., de Prez L.A., Jansen B.J., Pooter J.A., Key comparison BIPM.RI(I)-K6 of the standards for absorbed dose to water of the VSL, Netherlands and the BIPM in accelerator photon beams, *Metrologia*, 2017, **54**, Tech. Suppl., 06005.
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- Michotte C., Ratel G., Courte S., Dzeil T., Listkowska A., Csete I, Rózsa K., Szücs L., Zsinka A., Fréchou C., Bobin C., Pierre S, Update of the BIPM comparison BIPM.RI(II)-K1.Am-241 of activity measurements of the radionuclide <sup>241</sup>Am to include the 2009 results of the POLATOM (Poland), MKEH (Hungary) and the 2011 results of the LNE-LNHB (France) *Metrologia*, 2017, **54**, Tech. Suppl., 06010.
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- 16. Pommé S., Stroh H., Paepen J., Van Ammel R., Marouli M., Altzitzoglou T., Hult M., Kossert K., Nähle O., Schrader H., Juget F., Bailat C., Nedjadi Y., Bochud F., Buchillier T., Michotte C., Courte S., van Rooy M.W., van Staden M.J., Lubbe J., Simpson B.R.S., Fazio A., De Felice P., Jackson T.W., Van Wyngaardt W.M., Reinhard M.I., Golya J., Bourke S., Roy T., Galea R., Keightley J.D., Ferreira K.M., Collins S.M., Ceccatelli A., Verheyen L., Bruggeman M., Vodenik B., Korun M., Chisté V., Amiot M.-N., On decay constants and orbital distance to the Sun–part I: alpha decay, <u>Metrologia, 2017, 54(1), 1-18.</u>

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

The Consultative Committee for Ionizing Radiation (CCRI) was restructured in 2017 by merging the committees that cover the fields of dosimetry, radioactivity and neutron metrology. Discussions on issues related to specific fields continue to take place at meetings of the three sections of the CCRI. The first meeting of the combined CCRI was held in June 2017 and a workshop was also held to discuss the long-term strategy.

J.M. Los Arcos was 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). These responsibilities were handed over to S. Judge in September 2017, since when, a meeting of the CCRI Section II Key Comparison Working Group has been held.

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) 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 2017.

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 2017.

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). These reports are being reviewed prior to circulation to the CCRI or CCT for approval. The reports are published in *Metrologia Technical Supplement*.

C. Michotte gave a presentation on Measurement Comparisons (Planning and Monitoring) at the Capacity Building and Knowledge Transfer Programmes entitled "2017 BIPM-EURAMET 'TC Leadership' course" in February 2017 and "2017 Sound beginning in the CIPM MRA course" to delegates from AFRIMETS, APMP, COOMET, GULFMET and SIM in November 2017.

#### 5. Activities related to external organizations

J.M. Los Arcos was an Associate Member of the International Committee for Radionuclide Metrology (ICRM). He also evaluated scientific projects for the Spanish National Evaluation and Foresight Agency (ANEP) and was a technical auditor for the Spanish accreditation body.

D.T. Burns is a Fellow of the Institute of Physics (FInstP) in the UK, an elected Commissioner of the ICRU and Chairman of the ICRU Committee on Fundamental Quantities and Units. He attended the annual ICRU Commission meeting in Mexico City in March 2017. He is a member of the ICRU Report Committee on Key Data for Dosimetry, which published its ICRU Report 90 in 2016 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. 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 October 2017.

S. Judge is also a Fellow of the Institute of Physics (FInstP) in the UK.

C. Michotte continued as the Scientific Secretary and *rapporteur* for the JCGM-WG1 meetings, which were held in May and November 2017, at the BIPM and at the National Laboratory Association South Africa in Pretoria.

G. Ratel continued as the BIPM representative on the International Committee for Radionuclide Metrology (ICRM) including chairing the sessions on international metrology and intercomparisons at the 21st International Conference on Radionuclide Metrology and its Applications (ICRM 2017), held in Buenos Aires (Argentina) on 15-19 May 2017.

#### 6. Travel in 2017 (conferences, lectures and presentations, visits)

D.T. Burns to:

- Mexico City (Mexico), 31 March to 5 April, to participate in the annual meeting of the ICRU Commission.
- Vienna (Austria), 9 to 13 October, to participate in a consultants meeting on the revision of the International Code of Practice (IAEA TRS-398) for external-beam radiotherapy.

C. Kessler to:

• Buenos Aires (Argentina), 21 to 23 December, to perform a technical audit of the Secondary Standard Dosimetry Laboratory of Argentina (CRRD).

P. Roger to:

• Mâcon (France), 28 to 29 August, to participate in a course on radiation protection for the operation of LINACs (in relation to the work at DOSEO).

S. Picard to:

• Mâcon (France), 28 to 29 August, to participate in a course on radiation protection for the operation of LINACs (in relation to the work at DOSEO).

C. Michotte to:

• Pretoria (South Africa), 27 November to 1 December, to attend the JCGM-WG1 meeting held at the National Laboratory Association South Africa and to visit the NMISA dosimetry laboratories.

C. Michotte and M. Nonis to:

- Ottawa (Canada), 5 to 16 June, to carry out activity comparisons of <sup>11</sup>C (BIPM.RI(II)-K4.C-11), <sup>99m</sup>Tc (BIPM.RI(II)-K4.Tc-99m), <sup>64</sup>Cu (BIPM.RI(II)-K4.Cu-64) and <sup>18</sup>F (BIPM.RI(II)-K4.F-18) at the NRC using the SIR Transfer Instrument.
- Lucas Heights (Australia), 2 to 10 November, to carry out activity comparisons of <sup>99m</sup>Tc (BIPM.RI(II)-K4.Tc-99m), <sup>64</sup>Cu (BIPM.RI(II)-K4.Cu-64) and <sup>18</sup>F (BIPM.RI(II)-K4.F-18) at the ANSTO using the SIR Transfer Instrument.

G. Ratel to:

• Buenos Aires (Argentina) 15 to 18 May to attend the 21th ICRM conference and the Executive Board Meeting of the ICRM.

S. Judge to:

• PTB (Germany) 12 to 13 October and LNHB (France) 6 November to discuss BIPM strategy.

#### 7. Visitors in 2017

• Delegates from the Capacity Building and Knowledge Transfer Programme.

#### 8. Guest workers in 2017

- Dr Chul-Young Yi and Dr Hyun-Moon Kim KRISS (Korea), 13 to 24 February.
- Dr Wu Jinjie and Dr Wang Peiwei NIM (China), 6 to 9 March.
- Dr N. Durny, Slovak Institute of Metrology SMU (Slovakia), 12 to 16 June.
- Mr L. Czap, International Atomic Energy Agency IAEA, 3 to 11 August.
- Dr Martin Kelly NPL (UK), 23 to 24 October.
- Dr L. Laureano-Perez, National Institute of Standards and Technology NIST (USA) 13 September 2016 to 4 March 2017.
- Dr Haoran Liu, National Institute of Metrology NIM (China) 3 February to 31 July.

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### BIPM Chemistry Department Director: R.I. Wielgosz (1 January 2017 to 31 December 2017)

#### 1. Comparison coordination and supporting laboratory activities

Laboratory activities supported the coordination of four comparisons in this period, involving ninety three participations in these studies by NMIs, with the assistance of eight visiting scientists from NMIs. Seven comparison reports were published as well as five papers in peer reviewed journals.

Within the BIPM's small organic primary calibrator programme, the final report of the CCQM-K55.d comparison on folic acid purity was approved by the CCQM Working Group on Organic Analysis (OAWG). Measurements for the CCQM-K78.a comparison on multi-component amino acid calibration solutions were completed, and the results presented to the working group, with the Draft A report in preparation. The BIPM submitted results to the CCQM-P150.b comparison on qNMR, as well as completing characterization of samples and their homogeneity and stability for the CCQM-K148.a comparison (bisphenol A calibrator purity).

The first reference data document on 'Internal Standards for qNMR' was completed for maleic acid, and will be published on the BIPM website. This was an output of the universal calibrator programme for qNMR at the BIPM, an activity initiated together with the NMIJ (Japan), and supported in 2017 by secondees from the NMIJ, and INMETRO (Brazil), with characterization of the performance of four standards in three different solvents.

Activities to support comparability of primary standards for peptide and protein analysis have continued. The final reports of the key comparison and associated pilot study on C-peptide purity (CCQM-K115/P55.2) were published in *Metrologia* and the BIPM KCDB. A paper on the evolving calibration hierarchies for C-peptide measurements was published in *Clinical Chemistry*<sup>1</sup>. Preparative work for the next comparison of peptide calibrant value assignment facilities, focusing on oxytocin (CCQM-K155.b) in collaboration with NIM (China) has continued with the secondment of a visiting scientist from NIM. Methods for pure peptide characterization to be used in future comparisons have been studied during secondments by visiting scientists from the LGC (UK), studying BNP calibrators, and from the NIBSC (UK) studying short-peptide tryptic digest calibrators. The methods used to characterize pure peptide calibrators were published in *Analytical and Bioanalytical Chemistry*<sup>2</sup>, *Journal of Chemical Metrology*<sup>3</sup> and *Trends in Analytical Chemistry*<sup>4</sup>.

In the area of air quality measurement standards, the BIPM continued to contribute to the CCQM-GAWG Ozone Cross-Section Task Group, organizing the review of input data by the group and drafting of the first version of the paper summarizing the recommended best value and uncertainty for the ozone cross-section to be used in the key comparison BIPM.QM-K1. Six NMIs: VSL (the Netherlands), NPL, ISCII (Spain), INE (Mexico), NIST (USA) and CHMI (Czech Republic) sent their ozone standards to the BIPM and participated in BIPM.QM-K1, with two reports of the comparison published in *Metrologia* and the BIPM KCDB. Collaboration with the NIST on the upgrade of the electronic module for the Ozone SRP continued, with a prototype electronics module successfully constructed and tested at the BIPM, and the components and design for the final version agreed. The final report of the CCQM-K90 comparison on formaldehyde in nitrogen standards at 2  $\mu$ mol/mol was completed and published in *Metrologia* and the BIPM KCDB. Measurements on 24 standards from NMIs submitted to the BIPM as part of CCQM-K137 (NO in N<sub>2</sub> at 30 and 70  $\mu$ mol/mol) were completed, with standards returned to participating NMIs for stability assessment. The protocols for the comparison of NO<sub>2</sub> in N<sub>2</sub> standards at 10  $\mu$ mol/mol (CCQM-K74.2018) and the pilot study of HNO<sub>3</sub> measurements in such standards (CCQM-P172) were agreed with the CCQM Working Group on Gas Analysis (GAWG), with 14 NMIs electing to participate in the comparisons.

In the area of greenhouse gas standards, measurements on 46 standards of  $CO_2$  in air submitted for the CCQM-K120 comparison were completed at the BIPM, including Fourier transform infrared spectroscopy (FTIR), isotope ratio infrared spectrometer (IRIS) and gas chromatography with a flame ionization detector GC-FID methods for mole fraction and isotope ratio value assignment' and the Draft A report of the comparison prepared. The method developed for measuring isotopic abundances in CO<sub>2</sub> with optically based instruments was published in Analytical Chemistry<sup>5</sup>, and presented to the WMO-IAEA GGMT experts meeting in Switzerland in September 2017. This demonstrated the measurement standards and methods that could be used for such instruments in the future. Development and validation of a manometric system for CO<sub>2</sub> measurements, in support of a future planned ongoing comparison of CO<sub>2</sub> standards (BIPM.QM-K2), has progressed with a second secondment from the NIST. The first all-glass prototype was replaced with a coated stainless steel version, with much improved mechanical stability. The measurement protocol was optimized, including the identification and elimination of biases caused by trace gases within the system: new results are expected in early 2018. Preparation for a comparison on  $CO_2$  isotope ratio standards, coordinated jointly by the BIPM and the International Atomic Energy Agency (IAEA) continued, with an IRIS system for isotope ratio measurements integrated into the SIRM-GEN facility and the first blending experiments for CO<sub>2</sub> gases have been carried out. Validation work on optical tuneable diode laser spectroscopy (TDLS) and gas chromatography with electron capture detection (GC-ECD) systems in preparation for CCQM-K68.2019 (N<sub>2</sub>O in air, ambient level) were undertaken during a 3 month secondment by a visiting scientist from KRISS (Republic of Korea), with linearity and measurement uncertainty of the optical instrument verified.

#### 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 continued in the BIPM Chemistry Department, attracting twelve visiting scientists from NMIs, spending between 3 months and 1 year at the BIPM.

Three visiting scientists undertook the Metrology for Clean Air Course (NPL (UK), LNE (France) and NPLI (India)) on FTIR Measurements on Gas Standards (NO<sub>2</sub>, HCHO, HNO<sub>3</sub>, CO<sub>2</sub>) in the BIPM laboratories, and were trained on the use of B-FOS software for use with FTIR in gas metrology applications, with the software being made available later for use within the participating NMIs.

The second 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 2017. The laboratory programme on Mycotoxin Standards was supported by three visiting scientist from NIM (China) in related structure impurity analysis and calibration solution characterization and from UME (Turkey) and INMETRO (Brazil) in qNMR analysis of pure mycotoxin materials. In addition, five visiting scientists from INMETRO, INTI (Argentina), KEBS (Kenya), NIMT (Thailand), NMISA (South Africa), undertook three-month training secondments on mycotoxin calibration solution production, characterization and value assignment. The training programme was performed on zearalenone (ZEN). Pure materials for aflatoxin B1 and have been characterized, and are available for future training programmes and comparisons.

#### 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 2017 the Cycle 13 reference materials and measurement methods and the Cycle 11 reference measurement laboratory services, which were approved by the JCTLM Executive Committee during its 17th Annual Meeting in December 2016 were published in the JCTLM database.

As of December 2017 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;
- 184 reference measurement methods or procedures that represent about 80 different analytes in nine categories of analytes;
- 161 reference measurement services, delivered by seventeen reference laboratories and two NMIs in eight countries, which cover seven categories of analytes.

The latest JCTLM call for nominations for Cycle 14 reference materials and reference measurement methods and procedures, and Cycle 12 for reference measurement laboratory services, was announced on the JCTLM website in February 2017. 46 nominations for materials, 11 nominations for methods, and 2 nominations for services had been received as a result of the call.

The fourth issue of the JCTLM Database Newsletter was distributed in March 2017.

The 18th and 19th meetings of the Executive Committee of the JCTLM were held in Athens (Greece) on 11 June 2017 and at the BIPM on 7-8 December 2016, respectively, with the JCTLM Database Working Group meeting at the BIPM on 6 December 2017.

The biennial meeting of the Joint Committee for Traceability in Laboratory Medicine (JCTLM), which was held on 4-5 December 2017 at the Bureau International des Poids et Mesures (BIPM), attracted 117 delegates from 27 countries. The two-day programme for the meeting included presentations on: Why traceability matters to patients; Traceability in external quality assessment; Traceability and the IVD industry: the manufacturers' role; Traceability in the investigation of infectious diseases; Traceability: a global perspective and JCTLM update.

The second day of the programme focused on the clinical challenge of neurodegenerative disease with contributions on the requirements for and development of high-quality biomarker assays for the investigation of Alzheimer's disease, Parkinson's disease and other debilitating conditions.

The activities of the JCTLM Working Group on Traceability: Education and Promotion (JCTLM-TEPWG) led to the launch of the www.jctlm.org website in early 2017, to act as a source of information on metrological traceability for the laboratory medicine community. Thanks to the efforts of the working group, there has been a steady rise in membership of the JCTLM, with 54 Member Organizations (19 National and Regional Members; 35 Stakeholder Members).

#### 4. **Publications**

- Little R.R., Wielgosz R.I., Josephs R., Kinumi T., Takatsu A., Li H., Stein D., Burns C., Implementing a 1. reference measurement system for C-peptide: Successes and lessons learned, Clin. Chem., 2017, 63(10), 1447-1456.
- Bros P., Josephs R.D., Stoppacher N., Cazals G., Lehmann S., Hirtz C., Wielgosz R.I., Delatour V., 2 Impurity determination for hepcidin by liquid chromatography - high resolution and ion mobility mass spectrometry for the value assignment of candidate primary calibrators, Anal. Bioanal. Chem., 2017, 409(10), 2559-2567.
- 3. Josephs R.D., Stoppacher N., Westwood S., Wielgosz R.I., Li M., Quaglia M., Melanson J., Martos G., Prevoo D., Wu L., Scapin S., Öztug Senal M., Wong L., Jeong J.-S., Chan K.W.Y., Arsene C.G., Park S.-R.

Concept Paper on SI Value Assignment of Purity - Model for the Classification of Peptide/Protein Purity Determinations. *J Chem Metr.* 2017; 11(1):1-8.

- Josephs R.D., Stoppacher N., Daireaux A., Choteau T., Lippa K.A., Phinney K.W., Westwood S., Wielgosz R.I., State-of-the-art and trends for the SI traceable value assignment of the purity of peptides using the model compound angiotensin, https://doi.org/10.1016/j.trac.2017.09.026.
- 5. Flores E., Viallon J., Moussay P., Griffith D.W.T., Wielgosz R.I., Calibration strategies for FT-IR and other isotope ratio infrared spectrometer instruments for accurate  $\delta^{13}$ C and  $\delta^{18}$ O measurements of CO<sub>2</sub> in air, *Anal. Chem.*, 2017, 89(6), 3648-3655.
- Josephs R.D., Li M., Song D., Daireaux A., Choteau T., Stoppacher N., Westwood S., Wielgosz R., Xiao P., Liu Y., Gao X., Zhang C., Zhang T., Mi W., Quan C., Huang T., Li H., Melanson J.E., Ün I., Gören A.C., Quaglia M., Warren J., Pilot study CCQM-P55.2 on peptide purity – synthetic human C-peptide, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08011.
- Josephs R.D., Li M., Song D., Westwood S., Stoppacher N., Daireaux A., Choteau T., Wielgosz R., Xiao P., Liu Y., Gao X., Zhang C., Zhang T., Mi W., Quan C., Huang T., Li H., Flatschart R., Borges Oliveira R., Melanson J.E., Ohlendorf R., Henrion A., Kinumi T., Wong L., Liu Q., Oztug Senal M., Vatansever B., Ün I., Gören A.C., Akgöz M., Quaglia M., Warren J., Key comparison study on peptide purity–synthetic human C-peptide, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08007.
- Viallon J., Flores E., Idrees F., Moussay P., Wielgosz R.I., Kim D., Kim Y.D., Lee S., Persijn S., Konopelko L.A., Kustikov Y.A., Malginov A.V., Chubchenko I.K., Klimov A.Y., Efremova O.V., Zhou Z., Possolo A., Shimosaka T., Brewer P., Macé T., CCQM-K90, formaldehyde in nitrogen, 2 μmol mol<sup>-1</sup> Final report, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08029.
- Viallon J., Idrees F., Moussay P., Wielgosz R., Lin T.-Y., Norris J.E., Hodges J.T., Final report, on-going key comparison BIPM.QM-K1, ozone at ambient level, comparison with CMS-ITRI, June 2016, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08031.
- Viallon J., Moussay P., Wielgosz R., Hodges J., Norris J.E., Final report, on-going key comparison BIPM.QM-K1, ozone at ambient level, comparison with NIST, July 2015, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08006.
- 11. Viallon J., Moussay P., Wielgosz R., Idrees F., Heikens D., van der Veen A., Final report, on-going key comparison BIPM.QM-K1, ozone at ambient level, comparison with VSL, September 2016, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08025.
- Viallon J., Moussay P., Wielgosz R., Idrees F., Sanchez C., Morillo Gomez P., Final report, on-going key comparison BIPM.QM-K1, ozone at ambient level, comparison with ISCIII, March 2017, *Metrologia*, 2017, 54, *Tech. Suppl.*, 08024.

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

The CCQM held its 23rd meeting on 19-20 April 2017 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 the Executive Secretary of the CCPR and 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 Chair 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.

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. Staff

Dr N. Stoppacher left the BIPM in June 2017. Dr G. Martos joined the Department in October 2017. Mr F. Idrees transferred fully to the Department.

#### 8. Travel in 2017

R.I. Wielgosz to:

- Teddington (UK), 1-2 February and 4-5 October, to participate in the UK National Measurement System (NMS) Optical, Gas and Particle Metrology Programme Expert Group (PEG) meeting.
- Teddington (UK), 4-5 May to participate in the UK NMS Chemical and Biological Metrology Programme Expert Group (PEG) meeting.
- Minneapolis (USA), 22-25 May, to participate ISO TC 212 WG2 meetings on Reference Measurements Systems in Laboratory Medicine, and lead redrafting of ISO 15195.
- Athens (Greece), 11 June, to participate in the JCTLM Executive Committee meeting.
- Rotterdam (the Netherlands), 13-15 June, to Chair Sessions at the Gas2017 conference.
- EMPA Dübendorf, (Switzerland), 28-30 August, to participate in the GGMT-2017 WMO/IAEA Meeting on Carbon Dioxide, Other Greenhouse Gases, and Related Measurement Techniques, presenting activities on CO<sub>2</sub> isotope ratio measurements and standards.
- INRIM (Italy), 26-27 September, to participate in the meeting of the CCQM Inorganic Analysis Working Group.
- Beijing (China), 9-13 October, to give keynote lectures at the 17th Beijing Analysis Conference and Exhibition and APEC workshop on Capacity Building of Measurement Standards and Technologies in Grain Food Safety.
- VSL, Delft (the Netherlands), 24-27 October to participate in the CCQM Gas Analysis Working Group and Isotope Ratio Task Group Meetings.

- NPL, New Delhi (India), 24-28 November, to participate in APMP Focus Groups and the TCQM meetings.
- Brussels (Belgium), 30 November, to participate ISO TC 212 WG2 meetings on Reference Measurements Systems in Laboratory Medicine, and lead redrafting of ISO 15195.

E. Flores to:

- Paris (France), 29 May 2 June, Universite Pierre et Marie Curie, To attend the NDACC-IRWG & TCCON Meeting and to present the poster: Calibration strategies for FTIR and other IRIS instruments for accurate δ<sup>13</sup>C and δ<sup>18</sup>O measurements of CO<sub>2</sub> in air.
- Rotterdam (the Netherlands), 13-15 June, as a speaker at GAS 2017.
- Dübendorf (Switzerland) EMPA, 28-31 August, as a speaker at the 19th WMO/IAEA Meeting on Carbon Dioxide, Other Greenhouse Gases, and Related Measurement Techniques (GGMT-2017). Lecture: Calibration strategies for FTIR and other IRIS instruments for accurate  $\delta^{13}$ C and  $\delta^{18}$ O measurements of CO<sub>2</sub> in air.
- Delft (the Netherlands), VSL, 24-25 October to participate in CCQM GAWG meetings and workshops.

R. Josephs to:

- Budapest (Hungary), 6 May, for the Inter-Agency Meeting (IAM) of the CCMAS of the CODEX Alimentarius Commission.
- Paris (France), 11 May, to the LNE as a member of the examination board for the PhD of Ms Noémie Clouet Foraison.
- Ottawa (Canada), 27 September 29 September, to the NRC to contribute to the CCQM PAWG and OAWG meetings and related workshops.

S. Maniguet to:

• Athens (Greece), 11 June, to participate in the JCTLM Executive Committee meeting,

J. Viallon to:

- Boston (USA), 21–23 February, to undertake a training on an optical tuneable diode laser spectroscopy (TDLS) analyser for N<sub>2</sub>O,
- INRIM, Torino, (Italy), 17 March, to meet the CCPR president M.L. Rastello as of 1 January 2017 together with the former president T. Usuda.
- WMO Headquarters, Geneva, (Switzerland), 10–13 April, to give presentations at the symposium of the WMO Global Atmosphere Watch programme on the concept of traceability.
- NOAA and NCAR, Boulder, (USA), 23–26 May, to give presentations at the NOAA/ESRL Global Monitoring Conference (on calibration strategy for Isotope Ratio Infrared Spectrometer) and the WMO–GAW VOC Expert Workshop (on traceability of measurements).
- Rotterdam (the Netherlands), 13–15 June, to give a presentation on the BIPM PVT system at the GAS 2017 conference.
- VSL, Delft (the Netherlands), 24-27 October to participate in the CCQM Gas Analysis Working Group meeting.

S. Westwood to:

- Brussels (Belgium), 19-20 January, 2017 to give a presentation on Reference Measurement services supporting Customs and Border Security at the WCO Scientific Sub-Committee Meeting.
- Lausanne (Switzerland), 9-12 March 2017, for a WADA Laboratory Expert Group meeting.
- Nantes (France), 18-21 June 2017, for a WADA Laboratory Expert Group meeting.
- Berlin (Germany), 26-30 June 2017, for the 40th meeting of ISO/REMCO.
- Baveno (Italy), 20-21 September 2017, to give a presentation on Reference Materials for qNMR at the SMASH qNMR Workshop.
- Ottawa (Canada), 25 September 1 October 2017 to participate in the SIM CMWG and CCQM OAWG meetings and workshops.
- Montreal (Canada), 18-21 November 2017, for a WADA Laboratory Expert Group meeting.

#### 9. Guest workers in 2017

- C. Meyer, NIST (USA), from 16 January.
- T. Zhang, NIM (China), to 30 April.
- XQ Li, NIM, (China), to 14 April.
- XM Li, NIM, (China), from 14 February.
- J-S. LIM, KRISS (Republic of Korea), from 1 May to 31 July
- F. Torma, LGC (UK), from 1 February to 14 April
- M. Li, NIM, (China), from 20 April 2017
- B. Garrido, INMETRO (Brazil), to 28 February.
- T. Yamazaki, NMIJ (Japan), from 1 March to 30 November.
- M Fang, NIBSC (UK), from 1 September to 30 November.
- D. Preevoo, NMISA (South Africa), from 1 May to 31 August.
- Un, UME (Turkey), from 1 September to 30 November.
- S. Marbumrung, NIMT (Thailand), from 1 May to 31 July.
- E.C. Rego, INMETRO (Brazil), from 1 February to 30 April.
- M.E. Simon, INTI (Argentina), from 1 February to 30 April.
- Mugenya, KEBS (Kenya), from 1 May to 31 July.
- R. Soman Radha, NPLI (India) from 1 September.
- M.Ward, NPL (UK), from 1 September to 30 November.
- C. Sutour, LNE, (France), from 15 September to 30 November.
- Z. Guo, NIM, (China), from 1 December.

#### 10. Visitors in 2017

- C. Schneider, A. Bristow, P. Matejtschuk, J. Wheeler (NIBSC), 24 January, for discussions on the CIPM MRA and BIPM activities.
- Dr Suematsu (JEOL), Mr Miura and Mr Sabri (WAKO), 30 June for discussions on BIPM-NMIJ activities in qNMR.
- Dr Hugo Gasca Aragón (CENAM) December 6, Discussions on statistics applied to gas metrology.
- B. Sweeney (NPL) 24–26 April, to take part in BIPM.QM–K1 comparison.
- Dr H. Abe (NMIJ), 30 May, to visit Gas Metrology laboratories.
- J. Norris and G. Cula (NIST), 11–15 September, to take part in BIPM.QM–K1 comparison and work on the upgrade of the electronic module for the Ozone SRP.
- M. Vokoun (CHMI), 18-22 September, to take part in BIPM.QM-K1 comparison.
- J. Walden and S. Karri (FMI), 27–30 November, to take part in BIPM.QM-K1 comparison.
- M. Bailey, M. Fang, J. Wheeler (NIBSC), 17 November, for discussions on the CIPM MRA, BIPM and NIBSC activities.