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

Supplement: scientific Departments

(1 January 2012 – 31 December 2012)

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Bureau International des Poids et Mesures

BIPM Mass Department

Director: A. Picard

(1 January 2012 to 31 December 2012)

1.1 Calibrations

1.1.1 Certificates (P. Barat, H. Fang and A. Picard)

From 1 January to 31 December 2012, certificates were issued for the following 1 kg prototypes in platinum-iridium (Pt-Ir): No. 48 (Denmark), No. 57 (India), No. 72 (Republic of Korea) and Nos. 79 and 85 (USA). Calibration of a 1 kg prototype for Romania is under way.

Certificates for 1 kg standards in stainless steel were issued: one for the CEM (Spain), one for the CESMEC (Chile), two for the EIM (Greece), four for the HMI (Croatia), two for the INTI (Argentina), three for the KIM-LIPI (Indonesia), one for the SMD-ENS, SPF Economie (Belgium) and four for the VSL (Netherlands). Calibration is under way for a 1 kg standard in stainless steel for the INM (Romania) due towards the end of 2012, and for two 1 kg standards in stainless steel for the NML-SIRIM (Malaysia).

Determinations of magnetic susceptibility were made for six 1 kg standards in stainless steel: four for the HMI (Croatia), one for the KIM-LIPI (Indonesia) and one for the SMD-ENS/SPF Economie (Belgium).

1.1.2 Mass determination for the ensemble of reference mass standards (P. Barat)

So far, the ensemble of reference mass standards (ERMS) comprises four 1 kg Pt-Ir cylinders, four 1 kg stainless steel cylinders and four 1 kg natural silicon spheres. In addition to these 12 mass standards two surface artefacts, one in Pt-Ir and the other in stainless steel, each consisting of a stack of 8 discs, complete the ensemble.

As was mentioned in the Director's Report 2011, volume determination was performed at the BIPM for mass standards in Pt-Ir and in stainless steel. Therefore, during 2012 mass determination in air was undertaken for these mass standards. The mass stability of these artefacts will be investigated in 2013.

A delay occurred in the manufacturing process for the silicon spheres. Volume determination will be carried out by the NMIJ/AIST (Japan) and surface characterization by the PTB (Germany). It is anticipated that mass determination will recommence at the BIPM during the first half of 2013.

1.1.3 Volume of mass standards above 300 g (C. Goyon-Taillade)

Within the framework of calibration services offered to Member States of the BIPM, volume was determined for one 1 kg mass standard in stainless steel belonging to the HMI (Croatia) during 2012.

Within the framework of the ERMS development, calibration was performed for one stack of eight disks in stainless steel. In addition, determination of the volume of one stack of eight disks in Pt-Ir belonging to the NIST (USA) is in progress.

To improve the hydrostatic facility, temperature measurements of doubly distilled water, used as a density reference, have been improved to enable determination of the vertical thermal gradient of water in the bath.

Fabrication of a new prototype for use as a BIPM working standard was completed in the BIPM workshop.

1.1.4 Volume of mass standard below 100 g (C. Goyon-Taillade and F. Idrees)

Volume measurement of one mass of 52 g in Pt-Ir has been performed as a check for the BIPM Quality Management System. Twenty one rods in Pt-Ir and in stainless steel, which are part of two surface artefacts made up of a stack of eight disks, have been calibrated for the ERMS.

1.2 **Mass comparators to support the development programme** (P. Barat)

During 2012, several studies have been carried out with the following 1 kg mass comparators: M_one 6V-LL from Mettler Toledo and CCL1007 from Sartorius, in order to integrate them into the BIPM Quality Management System for mass calibrations carried out in air.

New software was developed to determine the mass and the associated uncertainty of the different mass standards involved in the mass comparisons carried out with the M_one 6V-LL mass comparator and the CCL1007 mass comparator.

Determination of the uncertainty due to positional errors for the M_one 6V-LL mass comparator taken in air was undertaken using two mass standards in stainless steel of the same volume so as to minimize the differential air buoyancy correction between both standards. The uncertainty obtained was 0.7 µg.

This determination had previously been carried out in 2009 for the CCL1007 mass comparator using the same mass standards. At that time the uncertainty due to positional errors was estimated to be below 0.6 µg.

Currently, mass calibrations provided for NMIs of Member States are performed with the Metrotec mass comparator. To check the consistency of measurements made using the three mass comparators operated by the BIPM, several mass comparisons in air using the same standards were conducted to compare results obtained from the M_one 6V-LL, the CCL1007 and the Metrotec mass comparators. Preliminary results indicated that the M_one 6V-LL and CCL1007 mass comparators both meet the requirements for calibrating mass standards of NMIs.

The M_one 6V-LL and CCL1007 mass comparators will be integrated into the BIPM Quality Management System for mass calibrations carried out in air in early 2013.

1.3 **BIPM susceptometer** (H. Fang)

At the request of the LISA-Pathfinder team (European Space Agency, ESA); the Mass Department measured the magnetic susceptibility of a 2 kg test mass made of gold-platinum alloy. Measurements were carried out to the very tight schedule required by the LISA project. Magnetic susceptibilities measured on the six faces of the test mass are similar to those obtained on similar samples which were measured in 2006 and 2007.

1.4 **Pressure** (P. Barat, C. Goyon-Taillade, F. Idrees)

Calibrations of BIPM manometers, with respect to the pressure balance maintained in the Mass Department were carried out twice in 2012. Eight internal certificates were issued.

1.5 **Support for NMIs** (P. Barat)

With a view to a future redefinition of the kilogram, a method, now well-known, was developed by the BIPM to calibrate the mass of test artefacts used in vacuum against mass standards in Pt-Ir maintained in air, such as the BIPM working standards. The method uses two additional 1 kg Pt-Ir surface artefacts, one having three and a half times more surface area than the other but both have the same volume and surface finish of a classical 1 kg prototype. These surface artefacts are used to link weighings made in air to those carried out under vacuum. They do this by taking into account the amount of water desorption between the two artefacts measured under ambient conditions in air and vacuum.

Two experiments have succeeded in linking fundamental constants of physics indirectly to the mass of the International Prototype of the Kilogram with a relative uncertainty below 5 parts in 10^8: these are the x-ray crystal density method (usually referred to as the Avogadro method) and the watt balance method. The realization of the new kilogram definition by either of these two methods must be made under vacuum. As a consequence, the laboratories involved in these experiments which will lead to a new definition of the kilogram are required to evaluate the water desorbed from air to vacuum by the test artefacts used in the experiments. If the laboratories are not able to characterize their own test standard between air and vacuum conditions, the BIPM is able to provide this service using its surface artefacts. At the request of the NIST, the BIPM conducted a study of the water sorption effect of the 1 kg prototype No. 85 following air/vacuum transfers.

1.6 International Avogadro Coordination project (P. Barat and A. Picard)

The BIPM Director's Report 2011 described within the framework of the Avogadro project, an experiment to measure the amount of irreversibly adsorbed water on a silicon sphere by deducing the mass difference of the sphere under vacuum before and after baking it under vacuum for two hours at 400 °C without any further contact with moist air.

Several problems were encountered during this study. The first involved the presence of electrostatic charges due to the dry surface of the sphere after baking. To prevent this from occurring, the BIPM acquired a HAUG discharging system to neutralize electrostatic charges. This device is installed inside a MBraun nitrogen-filled glove box which is attached to the oven. The glove box enables the transfer of the sphere to be made after baking without any contact with moist air (< 0.1 ppm of water and oxygen contents), to an automatically loadable container which is used to transfer the sphere to the Sartorius CCL1007 mass comparator. In addition to the undesirable electrostatic charge effects observed, the silicon sphere used for the study, Silo2, loaned by the PTB, must be weighed together with an additional weight in the form of a ring to be within the electronic weighing range of the mass comparator. As a result, handling the sphere while simultaneously avoiding contact with moist air was extremely complicated. The results obtained with this sphere were unsatisfactory and it was decided that despite the time already spent on this experiment the Silo2 sphere should be replaced by another more convenient silicon sphere.

The study has been repeated using a BIPM 1 kg natural silicon sphere (S2). The reversible component of adsorbed water on the surface of the sphere (the physical water sorption effect) was determined by weighing the sphere in moist air followed by measurements under vacuum (1 mPa) then the amount of irreversibly adsorbed water (the chemical water sorption effect) was determined. The S2 sphere was transferred from the mass comparator to the oven attached to the glove box, baked at 400 °C for two hours under vacuum then transferred back (again without contact with moist air) to the mass comparator, while remaining under vacuum. The weighings under vacuum before and after baking should allow the determination of the irreversible part of water desorption. The results obtained are not as expected and the study will continue.

1.7 Ensemble of reference mass standards (P. Barat, F. Idrees, E. de Mirandes, and A. Picard)

During 2012, great progress was made at the BIPM with the ensemble of reference mass standards (ERMS) experiment. We report the major achievements.

In 2012, components of all three storage networks (nitrogen, argon and vacuum) were completed and assembled. At present, the argon gas is supplied from ultrapure argon bottles (level of impurity ~50 nmol mol^{-1}). To supply nitrogen, either nitrogen bottles (~50 nmol mol^{-1} of impurities) or a nitrogen generator (5 µmol mol^{-1} of moisture and 2 µmol mol^{-1} of oxygen) are used. An automatic switch has been designed and installed to transfer between empty and full gas bottles. A similar device to switch automatically between empty nitrogen gas bottles and the nitrogen generator has also been installed.

Analysis of the background contaminants in the gas (oxygen, water, and hydrocarbons) has been fully automated. To accomplish this, oxygen and moisture analysers and a gas chromatograph (GC) coupled to a flame ionization detector (FID), were purchased, installed and characterized. Additionally, a hydrogen gas generator and a filtered air generator for the GC and the FID have been purchased, installed and made operational. Periodic internal calibrations of oxygen and moisture analysers were conducted. An exchange with gas-analysis experts at the NPL organized during 2012 improved the BIPM's understanding of these technologies.

An analysis of the background gases in each argon and nitrogen container has been running uninterruptedly for six months. So far, the purity measured for the gases flowing through the containers is within 2×10^{-6} mol mol^{-1} and 0.06×10^{-6} mol mol^{-1} for the moisture and oxygen contaminants, respectively.

With respect to the vacuum storage network, a new vacuum pump has been purchased and connected. The purity of the vacuum for each vacuum container is automatically and continuously monitored with a residual gas analyser (RGA). Between July and December 2012 the purity of the vacuum network has been monitored automatically using the RGA and the maximum partial pressure detected was below 1.0×10^{-4} Pa.

At present, some of the gas and vacuum containers are empty, some only contain a mass holder and others contain both a mass holder and a test mass in stainless steel. Gas measurements showed no evident differences between these three cases. The next step will be to introduce the mass standards constituting the ERMS.

1.8 Comparisons

1.8.1 Comparison in mass (P. Barat, C. Goyon, and A. Picard)

The BIPM is the pilot laboratory for the key comparison CCM.M-K4 which has 16 participants organized in four petals. Each participating NMI has determined the masses of two 1 kg mass standards in stainless steel. The eight travelling standards were sent to the first four participants in September 2011 and the last of the mass standards were returned to the BIPM in April 2012. All participants have reported their results and the Draft A report is in preparation.

1.8.2 Comparison in magnetic properties (H. Fang)

The BIPM participated in the Inter-American Metrology System (SIM) supplementary comparison SIM.M.M-S9, for the determination of the susceptibility and magnetic polarization of weights. The susceptometer method which was originally developed within the BIPM Mass Department is used in this comparison. There are nine participating laboratories, with the INDECOPI (Peru) acting as the pilot. The exercise was expected to be completed by the end of 2012. Measurements were carried out by the BIPM in March 2012. Three years ago the BIPM participated in TC-M project 1110, a EURAMET "Cooperation in Research" on the magnetic properties of mass standards that is still ongoing. Participation of the BIPM in this project should make it possible to compare the performance of participants in both of these regional comparisons.

1.9 **Trilateral cooperation among the NPL, the METAS and the BIPM** (S. Davidson (NPL), P. Fuchs (METAS) and A. Picard)

The BIPM continued the trilateral cooperation among the BIPM, the NPL and the METAS in 2012 to support the *mise en pratique* of the new definition of the kilogram. Different studies of cleaning efficiency using different techniques, the rate of recontamination after cleaning and the mass stability during and after cycles of weighing in air followed by weighing in vacuum have been carried out on different materials such as silicon, Pt-Ir and stainless steel. As part of the trilateral collaboration, the NPL investigated the sorption characteristics of silicon, Pt-Ir and stainless steel surfaces. Sorption artefacts made from the three materials have been investigated over a pressure range from 1 mPa to 0.1 MPa (ambient). Four measurement cycles were performed and additional results at three intermediate pressures were taken. The METAS and the NPL investigated the use of a plasma cleaning apparatus. This apparatus allows direct comparison of UV/Ozone cleaning, previously investigated two years ago at the NPL and the BIPM, to be directly compared with cleaning by hydrogen and oxygen plasmas. The aim is to provide an automated alternative to the BIPM cleaning-washing technique. The METAS first made a combined measurement using X-Ray Photoelectron Spectroscopy (XPS) and gravimetry methods on recontamination by hydrocarbons and gold after the H_2-plasma cleaning. It was demonstrated that recontamination is minimal (< 0.5 µg/year) but with linear long-term growth (more than two and half years). Nor did recontamination depend on which of the three storage conditions (argon, nitrogen and vacuum) were used. The METAS compared the effectiveness of UV/Ozone cleaning compared to H_2-Plasma cleaning methods. The comparison shows that both cleaning methods produce a highly reproducible and reversible chemical state and the spectra obtained by both methods perfectly coincide.

The use of both gravimetric measurements and surface analysis using XPS and ellipsometry allows an evaluation of the effectiveness of both new techniques not only in terms of cleaning but also regarding surface chemistry and mass stability after cleaning. Therefore, the NPL continues to develop ellipsometry models for oxide/carbonaceous/water over layers of metallic surfaces (stainless steel and Pt-Ir) with a view to using this as a tool to monitor the stability of mass standards. In addition, studies of different storage conditions are ongoing at the METAS and the BIPM (with the BIPM ERMS).

Under the umbrella of the trilateral cooperation various studies are being carried out using small samples (10 mm diameter and 1 mm thick). The BIPM and the METAS also undertook a study focused on cleaning effects at the 1 kg level of mass standards in Pt-Ir. This study is based on three cleaning methods: the *nettoyage-lavage* technique (BIPM method), the hydrogen low-pressure plasma cleaning (METAS method) and the UV/Ozone cleaning (NPL method).

Comparison of these three methods is based on two parameters: on the one hand, the amount of contaminants removed after the cleaning step and on the other hand the mass stability of the 1 kg mass standard in Pt-Ir after cleaning.

A part of this study is being carried out with the help of the METAS. Indeed, the hydrogen low-pressure plasma cleaning and the UV/Ozone cleaning are performed using their equipment. Chemical analysis of the surface of the standard is done before and after the cleaning method, using their XPS. Mass measurements to determine the stability of the 1 kg mass standard in Pt-Ir are carried out at the BIPM.

So far, only hydrogen low-pressure plasma cleaning has been applied to the 1 kg mass standard in Pt-Ir. The mass stability study is under way.

The BIPM appreciates and gives its thanks to the METAS, the NPL and other participants in this collaboration, which permitted the BIPM Mass Department to augment its programme of work, particularly for the expected redefinition of the kilogram. This cooperation will continue in 2013, and is expected to provide more results for use in the preparation of the *mise en pratique*.

These studies will continue in 2013 outside the framework of the formal trilateral cooperation, which has ended. This work continues under the umbrella of the European Metrology Research Programme (EMRP) as a Joint Research Programme (JRP) designated as SIB-05 (NewKILO): "*Developing a practical means of disseminating the redefined kilogram*". The Programme runs from June 2012 to May 2015.

1.10 Publications

- Baumann H., Bettin H., Bielsa F., Eichenberger A., Geneves G., Kuramoto N., Mana G., Massa E., Pereira dos Santos F., Picard A., Pinot P., Schindler A., Realization of the anticipated definition of the kilogram. *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM),* 2012, 340-341.

1.11 Activities related to the work of Consultative Committees

- A. Picard is Executive Secretary of the Consultative Committee for Mass and Related Quantities (CCM) and the Consultative Committee for Thermometry (CCT) and is a member of several working groups (WGs) and task groups (TGs) of these Consultative Committees (CCs), such as the CCM Working Group on Mass Standards (WGM) TG1 and TG2; the CCM Working Group on Changes to the SI kilogram (WGSI-kg); the CCM Working Group on Strategy (WGS); the CCT Task Group on the SI (CCT TG-SI); the CCT Working Group on Strategy (WGS); and the CCT Working Group on Thermodynamic temperature determinations and extension of the ITS-90 to lower temperatures (WG 4).
- E. de Mirandés has been involved in the CCM Working Group on Mass Standards (WGM) TG2 and the CCM Working Group on Strategy (WGS) in 2012.

1.12 Activities related to external organizations

- A Picard acts as the BIPM liaison with the International Avogadro Coordination project (IAC), IMEKO Technical Committee 3 (TC3) and EURAMET Technical Committee of Mass and Related Quantities (TC-M) and Technical Committee of Thermometry (TC-T).
- A. Picard is coordinator for mass measurements in the former International Avogadro Coordination project/CCM Working Group on the Avogadro Constant (WGAC).
- A. Picard is the contact person for the European Metrology Research Programme (EMRP) joint research project SIB-05 (NewKilo) and SIB-03 (kNOW).

1.13 Travel (conferences, lectures and presentations, visits)

A. Picard to:

- Euramet TC-M meeting, Vienna (Austria), 29 February to 2 March 2012, to attend satellite meetings and to give a talk on the new definition of the kilogram and its *mise en pratique*.
- 9th International Temperature Symposium (ITS9), Los Angeles (USA), 19-23 March 2012, to attend the meeting.
- Euramet TC-T meeting, Istanbul (Turkey), 18-20 April 2012 to attend satellite meetings.
- CCM WG gravimetry, Istanbul (Turkey), 29-30 May 2012, to participate to the meeting as CCM executive secretary.

- LNE, Paris (France), 20-21 June 2012, to participate in the EMRP kickoff meeting for the Joint Research Programme SIB 05 "Developing a practical means of disseminating the new kilogram".

P. Barat to:

- METAS, Bern-Wabern (Switzerland), 15-25 June 2012, to work on the first part of the study of the cleaning effect on 1 kg mass standards in platinum-iridium.

C. Goyon-Taillade to:

- MathWorks, Meudon (France), 20-22 March 2012, to attend a training course on "*Fondamentaux MatLab*".

E. de Mirandés to:

- NPL, Teddington (UK), 7-8 June 2012, to visit experts in humidity and gas analysis, accompanied by F. Idrees.
- Akita University, Akita (Japan), 20-23 August 2012, to attend the SICE 2012 conference and to deliver a keynote speech on "The possible future revision of the International System of Units, the SI".

1.14 Visitors

- C. J. Williams (NIST), to collect prototype No. 79, 24 January 2012;
- F. García (CESMEC), to collect one 1 kg mass standard in stainless steel, 4 February 2012;
- S. Lee (KRISS), to deliver prototype No. 72, 5 March 2012;
- A. Kumar (NPLI), to deliver prototype No. 57, 13 March 2012;
- J. Nicolas (SMD-ENS, SPF Economie), to collect one 1 kg mass standard in stainless steel, 4 April 2012;
- D. W. Chung (KRISS), to collect prototype No. 72, 20 April 2012;
- H. Yoon (NIST), to collect prototype No. 85, 26 May 2012;
- N. Medina (CEM), to collect one 1 kg mass standard in stainless steel, 14 June 2012;
- L. Nielsen (DFM), to collect prototype No. 48, 14 June 2012;
- P. Gatti (INTI), to deliver two 1 kg mass standards in stainless steel, 22 June 2012;
- R. Nater (Mettler Toledo) and F. Mohammed Ismail (Dubai Central Laboratory Department (DCL)), to visit the Mass Department, 22 June 2012;
- G.F. Popa (INM), to deliver prototype No. 2 and one 1 kg mass standard in stainless steel, 30 July 2012;
- M. Schreiber (Sartorius), to carry out some modifications on the CCL1007 mass comparator, 27 to 30 August and 2 to 5 October 2012;
- E. Dierikx (VSL), to deliver two 1 kg mass standards in stainless steel, 5 September 2012;
- A. Sengupta (NPLI), to collect national prototype No. 57, 14 September 2012;
- S. Tu (CMS) to visit the Mass laboratories, 12 November 2012;
- J. Cáceres (LATU), to visit the Mass laboratories, 20 November 2012;
- I. van Andel (VSL), to collect two 1 kg mass standards in stainless steel, 22 November 2012;
- J Ullrich (Director of the PTB), 4 December 2012;
- M. Sawi (NML-SIRIM), to deliver two 1 kg mass standards in stainless steel, 13 December 2012.

BIPM Time Department
Director: E.F. Arias
(1 January 2012 to 31 December 2012)

2.1 International Atomic Time (TAI) and Coordinated Universal Time (UTC) (E.F. Arias, A. Harmegnies, Z. Jiang, H. Konaté, W. Lewandowski, G. Panfilo, G. Petit and L. Tisserand)

The reference time scales, International Atomic Time (TAI) and Coordinated Universal Time (UTC), are computed from data reported regularly to the BIPM by the various timing centres that maintain a local UTC; monthly results are published in *Circular T*. The *BIPM Annual Report on Time Activities for 2011*, volume 6, provides the definitive results for 2011 and is available electronically on the BIPM website www.bipm.org/en/publications/time_activities.html.

2.2 Algorithms for time scales (Z. Jiang, W. Lewandowski, G. Panfilo and G. Petit)

The algorithm ALGOS used for the calculation of the 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 continues in the Department with the aim of improving the long-term stability of EAL and the accuracy of TAI.

Since September 2011 the clock frequency prediction model in ALGOS takes into account the frequency drift which affects most of the participating atomic clocks. The frequency drift of each clock is estimated with respect to Terrestrial Time (TT) computed at the BIPM and which represents the best reference for frequency. As a consequence, the drift of -1.3×10^{-17} /day observed in EAL with respect to TAI has been completely removed, with an improvement in the long-term stability of EAL.

2.2.1 EAL stability

Some 87 % of the clocks used in the calculation of time scales are either commercial caesium clocks of the Symmetricom/HP/Agilent 5071A type or active, auto-tuned hydrogen masers. To improve the stability of EAL, a weighting procedure is applied to clocks where the maximum relative weight each month depends on the number of participating clocks. On average during 2011, about 15 % of the participating clocks were at the maximum weight. The change of the frequency prediction model has no impact on the weight of clocks. In order to allow a correct weighting of the hydrogen masers, and for better distribution of the weight among the caesium clocks and hydrogen masers, studies were undertaken to develop a new weighting procedure based on the concept that a good clock is not a stable clock but instead is a predictable clock. At the end of 2012 this procedure had been tested on six previous years of collected data and proved to establish a good distribution of weights; as a consequence, the stability of EAL will be improved. This new prediction model will be implemented in ALGOS during the first few months of 2013. The stability of EAL, expressed in terms of an Allan deviation, is about 3 parts in 10^{16} for averaging times of one month.

2.2.2 TAI accuracy

To characterize the accuracy of TAI, estimates are made of the relative departure, and its uncertainty, of the duration of the TAI scale interval from the SI second, as produced on the rotating geoid, by primary frequency standards. Since January 2012, individual measurements of the TAI frequency have been provided by ten primary frequency standards, including eight caesium fountains (LNE-SYRTE FO1, LNE-SYRTE FO2, LNE-SYRTE FOM, NIST F1, NPL CSF1, NPL CSF2, PTB CSF1 and PTB

CSF2). Reports on the operation of the primary frequency standards are regularly published on the BIPM website and collated in the *BIPM Annual Report on Time Activities*.

Since 2012, measurements of the TAI frequency by secondary frequency standards have been reported in *Circular T*. They have not been used for TAI steering so far, but will be after the implementation of the CIPM 2012 recommendations.

Since January 2012, 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 $+3.5 \times 10^{-15}$ to -0.3×10^{-15}, with a standard uncertainty of maximum 0.5×10^{-15}. Between January and December 2012, ten steering corrections have been applied, giving a total correction to $[f(EAL) - f(TAI)]$ of -0.043×10^{-15}. Since the introduction of the new prediction algorithm in September 2011, monthly steering corrections of -5×10^{-16} and -3×10^{-16} have been applied. Since the drift affecting EAL has completely disappeared no corrections to the frequency of EAL have been applied since November 2012.

2.2.3 Independent atomic time scales: TT(BIPM)

Because TAI is computed in 'real-time' and has operational constraints, it does not provide an optimal realization of TT, the time coordinate of the geocentric reference system. The BIPM therefore computes an additional realization, TT(BIPM), in post-processing, which is based on a weighted average of the evaluation of the TAI frequency by the primary frequency standards. The Time Department provided an updated computation of TT(BIPM), named TT(BIPM11), valid until December 2011, which had an estimated accuracy of about 3 parts in 10^{16} over recent years. Moreover, the Time Department provides a monthly extension of TT(BIPM11) based on the most recent TAI computation. Such an extension is useful for pulsar analysis pending the yearly updates of TT(BIPM). Studies aimed at improving the computation of TT(BIPM) are ongoing, in order to keep it in line with improvements in the primary and secondary frequency standards.

2.2.4 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*.

2.3 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 Frequency Standards (WGPFS), seeking to encourage comparisons, knowledge-sharing between laboratories, the creation of better documentation, and the use of high-accuracy primary frequency standards (Cs fountains) for TAI.

The CCL/CCTF Frequency Standards Working Group maintains a list of recommended values of standard frequencies for applications including secondary representations of the second. At its meeting in September 2012 it proposed additions and updates to microwave and optical atomic transitions in the list. The latest changes to the list, containing frequency values and uncertainties for transitions in Rb, Hg^+, Yb^+, Yb, Sr^+, Sr and Al^+ were recommended by the CCTF in September 2012 as secondary representations of the second.

Secondary representations of the second reported in BIPM Circular T

Since January 2012 the LNE-SYRTE has reported frequency measurements of the Rb microwave transition obtained with a double Cs-Rb fountain (FO2Rb). Reports with data since November 2009 onwards have been reviewed and approved by the CCTF WGPFS only for comparison via *Circular T* with the frequency of TAI. From May 2012 a new table has been included in *Circular T*; 21 measurement reports of FO2Rb were submitted in 2012.

Advanced time and frequency transfer

The Time Department has not particularly developed activities in this field, but instead has followed the evolution of the techniques for optical clock comparison by contributing to the CCTF WG on Coordination of the Development of Advanced Time and Frequency Transfer Techniques (WGATFT) and by participating in meetings of experts, mainly on the use of optical fibres.

2.4 **Time links** (E.F. Arias, A. Harmegnies, Z. Jiang, H. Konaté, W. Lewandowski, G. Panfilo, G. Petit, and L. Tisserand)

At the end of 2012, 72 time laboratories participated in the calculation of TAI at the BIPM. The laboratories are equipped with GNSS receivers and/or operating two-way satellite time and frequency transfer (TWSTFT) stations.

Significant improvements have been made within the Time Department to the time links used for the calculation of TAI; data from three independent techniques are included in the process of comparison of laboratories' clocks based on tracking GPS and GLONASS satellites, and TWSTFT.

The GPS all-in-view method is widely used and takes advantage of the increasing quality of the International GNSS Service (IGS) products (clocks and IGS time). Clock comparisons are possible using C/A code measurements from GPS single-frequency receivers, or dual-frequency, multi-channel GPS geodetic-type receivers (P3). The older GPS single-channel single-frequency receivers have almost disappeared, replaced by either multi-channel single- or dual-frequency receivers. The GPS phase and code data provided by time laboratories is processed each month using the Precise Point Positioning (PPP) technique. Fifteen TWSTFT links are officially submitted for use in the computation of TAI, representing 21 % of the time links. Since 2011, the combination of TWSTFT and PPP (so called TWPPP) is used whenever possible. This generally concerns about a dozen links for which the two techniques are available.

Since January 2011 the Time Department started computing combined GPS/GLONASS links resulting in improved link uncertainty. About four GPS/GLONASS links are regularly computed for *Circular T*.

Testing continues on other time and frequency comparison methods and techniques.

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

Geodetic-type receivers also provide raw phase measurements which may be used, along with the code measurements, to compute time links. Since October 2007, the BIPM has computed its own solutions for such time links, using GPSPPP software from Natural Resources Canada (NRCan), and these links have been introduced into the TAI regular computation since September 2009. In 2011 a new version of the NRCan PPP software was installed. It is capable of processing both GPS and GLONASS data. Comparisons with other PPP software have been carried out. Studies are continuing to improve long-term stability, using new processing techniques, in collaboration with software developers at NRCan, the *Observatoire Royal de Belgique* (ORB), the *Centre National d'Études Spatiales* (CNES) and also with other institutes.

2.4.1 Global Positioning System (GPS) and Global Navigation Satellite System (GLONASS) code measurements

All GNSS links are corrected for satellite positions using IGS and ESA post-processed, precise satellite ephemerides, and those links using single-frequency receivers are corrected for ionospheric delays using IGS maps of the total electron content of the ionosphere.

2.4.2 Phase and code measurements from geodetic-type receivers

Techniques using 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. A study is being conducted under the framework of the IGS Working Group on Clock Products, which has a physicist from the Time Department as a member.

The method developed to perform absolute calibration of the Ashtech Z12-T hardware delays allows the BIPM to use this receiver for differential calibrations of similar receivers world-wide; calibration campaigns began in January 2001. Calibration results have also been issued for other receivers: the Septentrio PolaRx2 since 2006 and the Dicom GTR50 and Javad JPS E-GGD since 2009. Other types of receivers are being investigated in collaboration with the laboratories equipped with them. Since 2009, the BIPM travelling receiver for differential calibrations is a GTR50. In all cases, at least two receivers remain at the BIPM to serve as a local reference to which the travelling receiver is compared between calibration trips. Results of the differential calibration exercises are made available on a dedicated web page (www.bipm.org/jsp/en/TimeCalibrations.jsp), where past calibration results are also provided.

Data from geodetic-type receivers world-wide are collected for TAI computation, using procedures and software developed in collaboration with the ORB. These P3 time links are now routinely computed and compared to other available techniques, notably two-way time transfer. After one year of work, the software producing GPS P3 (iono-free) data has been upgraded and is now able to produce GLONASS P3. It will be implemented in some receivers to automatically produce both formatted GPS and GLONASS P3 code results. In the future, these newly available data are likely to be used in multi-GNSS system time links, but further studies on inter-frequency biases have to be carried out.

2.4.3. Two-way time transfer

Two meetings of the TWSTFT participating stations were held during 2012. The annual meeting of the CCTF WG on TWSTFT was held at the BIPM headquarters in September 2012.

The TWSTFT technique is currently operational in eleven European, two North American and nine Asia-Pacific time laboratories. Fifteen TWSTFT links are routinely used in the computation of TAI; thirteen of them are combined with GPS PPP solutions. Some of them are used for particular studies such as the Time Transfer by Laser Link (T2L2) experiment. The TWSTFT technique applied to clock comparisons in TAI is at present reaching its maximum potential with sessions scheduled every two hours.

The BIPM is also involved in the calibration of two-way time-transfer links by comparison with GPS.

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

2.4.4 Uncertainties of TAI time links

The values of Type A and Type B uncertainties of TAI time links are published in *Circular T*, together with information on the time links used in each monthly calculation. The values of u_A have been individually updated when deemed necessary, depending on the noise level present in the links. Due to upgrading of time transfer equipment at participating laboratories, the Time Department has refined the methods for clock comparison, and a global re-evaluation of u_A values, using the latest evaluation tools, has been made and published in *Circular T* of December 2011.

2.4.5 Calibration of delays of time-transfer equipment

The BIPM continues to organize and run campaigns for measuring the relative delays of GPS time equipment in time laboratories that contribute to TAI. The BIPM also supports TWSTFT calibration trips, using a GPS receiver from its time laboratory.

Work on the absolute calibration of GNSS receivers has been carried out by a Ph.D student through a collaboration co-financed with the CNES, and involving the LNE-SYRTE. The doctoral thesis "Contribution to the absolute calibration of a GNSS reception chain" was defended in November 2011, completing the planned programme of work. It focused on the development and optimization of a method of absolute calibration to independently determine the electrical delay of each element in a GNSS reception chain (time receiver, antenna and antenna cable) with an overall uncertainty of less than one nanosecond. The absolute calibration method can be used to characterize performance and environmental sensitivity of each component of the acquisition system.

Cooperation continued with EURAMET to obtain regional support for GNSS equipment calibration in contributing laboratories. This action follows a Recommendation of the CCTF (2009) and opens up the possibility of future interaction with other RMOs.

2.5 **Key comparisons** (E.F. Arias, H. Konaté, Z. Jiang, W. Lewandowski, G. Panfilo, G. Petit, L. Tisserand, A. Harmegnies and L. Robertsson)

Key comparison in Time CCTF-K001.UTC

Results of the key comparison in time, CCTF-K001.UTC, involving the time laboratories participating in the CIPM MRA, are published regularly in the form of the monthly BIPM *Circular T*. The CCTF approved updates to the existing guidelines and new guidelines on the evaluation of the uncertainty in frequency and on the evaluation of the uncertainty in the prediction of [*UTC*-*UTC*(*k*)] in September 2012. These guidelines had been prepared by staff of the Time Department to support the CCTF Working Group on the CIPM MRA (WGMRA).

Key comparison of stabilized lasers CCL-K11.UTC

The BIPM continues to support the CCL-K11 key comparison in terms of participation in measurement campaigns as well as by providing general advice. This follows a decision at the 98th meeting of the CIPM in 2009. During 2012, staff from the Time Department were only involved in the reporting of measurement results and no BIPM presence for measurement campaigns took place.

Key comparison of absolute gravimeters CCM.G-K1

The campaign of comparison of absolute gravimeters ICAG-2009 concluded the contribution of the BIPM to the maintenance of the world global gravity network. Since then the key comparison CCM.G-K1 has been defined as part of the ICAG. This activity has been transferred to the NMIs, and the next ICAG in 2013 will take place in Luxembourg piloted by the METAS. As agreed, the BIPM provided support to the organization of the future ICAG during 2012 and staff from the Time Department contributed to the CCM Working Group on Gravimetry (WGG). A series of relevant publications related to ICAG-2009 have been published, including the reports of the key comparison and a scientific article. Studies based on gravity measurements on the BIPM watt balance site were ongoing at the end of 2012, and a scientific article on the characterization of gravity for the watt balance experiment is expected to be published in the first three months of 2013.

2.6 **Rapid UTC** (F. Arias, A. Harmegnies, G. Panfilo, G. Petit, L. Tisserand)

At the end of 2011 the Time Department called for expressions of interest to participate in a pilot experiment for producing a "rapid UTC" (UTCr), that is, daily values of [UTCr – UTC(k)] evaluated on a weekly solution. With the full support of the time laboratories and of the CCTF, the pilot experiment started in January 2012 with the participation of about 40 laboratories that contribute approximately 60 % of the clocks in UTC. The first results (ftp://tai.bipm.org/UTCr) were published at the end of February 2012. Since then, the weekly solutions have been published every Wednesday without interruption.

A report was submitted to the CCTF in September 2012 and the pilot experiment is expected to become operational in 2013.

The new product does not change the procedures for the monthly calculation of UTC, which remains the only key comparison on time.

2.7 **New proposed definition of UTC** (F. Arias, W. Lewandowski)

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

The actions of BIPM delegates during this process have been critical at the International Telecommunication Union (ITU), and also in disseminating information and promoting decision making at the level of national representatives. A delegate from the BIPM attended the Radiocommunication Assembly 2012 (RA-12) which was held in Geneva, Switzerland, on 16 to 20 January 2012, where the ITU member states discussed the adoption of modified UTC. It was decided to postpone the decision until the World Radiocommunication Conference 2015 (WRC-15) to be held in Geneva on 2 to 27 November 2015. In the meantime investigations will continue on the feasibility of a continuous timescale to be used as the international time reference. Staff from the Time Department contributed to two Radiocommunication Seminars in St Petersburg, Russian Federation (June 2012) and Manta, Ecuador (September 2012), respectively. Staff from the Time Department attended regular meetings of Study Group 7 and Working Party 7A of the ITU-R.

2.8 **Pulsars** (G. Petit)

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

2.9 Space-time references (E.F. Arias and G. Petit)

The BIPM maintains the web and ftp sites for the *IERS Conventions* (tai.bipm.org/iers/). The Conventions describe the latest realizations of the celestial and terrestrial reference frames, and of the model for the transformation between them. They also describe conventional models for the gravitational field, the displacement of markers on the Earth's crust and for the propagation of electromagnetic signals. In addition, the *Conventions* now provide a complete set of associated conventional software. These tasks are carried out with the help of the Advisory Board for the *IERS Conventions* updates, including representatives from all groups involved in the International Earth Rotation and Reference Systems Service (IERS). After the completion of the new reference edition, *IERS Conventions* (2010) in December 2010, work is continuing to provide updates to the *Conventions* (2010) which are regularly posted on the website (tai.bipm.org/iers/convupdt).

Activities related to the realization of reference frames for astronomy and geodesy are being developed in cooperation with the IERS. In these domains, improvements in accuracy will increase the need for a full relativistic treatment and it is essential to continue to participate in international working groups on these matters. Cooperation continues for the maintenance of the international celestial reference system within the framework of the activities of a working group created by the IAU in August 2012.

2.10 Comb activities (L. Robertsson)

As a result of the reorganization of activities in the Time Department, BIPM comb activities are limited to the maintenance of the BIPM frequency comb for internal use related to laser applications only and in other sections when needed.

2.11 Publications

External publications

1. Arias E.F., Jiang Z., Robertsson L., Vitushkin L., Ruess D., Ullrich C., Inglis D., Liard J., Robinson I., Ji W., Shuqing W., Lee C., Palinkas V., Mäkinen J., Pereira Dos Santos F., Bodart Q., Merlet S., Mizushima S., Choi I.-M., Baumann H., Karaböce B., Final report of key comparison CCM.G-K1: International comparison of absolute gravimeters ICAG2009, *Metrologia,* 2012, **49**, *Tech. Suppl.,* 07011.
2. Arias F., Harmegnies A., Jiang Z., Konaté H., Lewandowski W., Panfilo G., Petit G., Tisserand L., UTCr: a rapid realization of UTC, *Proc. EFTF 2012*, 2012, 24-27.
3. Bauch A., Beutler G., Petit G., Time and Frequency Metrology and its use for Navigation: Status and Proposed Future Research Themes, Galileo Science Advisory Committee, 2012.
4. Francis O, Rothleitner Ch., Jiang Z., Accurate determination of the Earth Tidal Parameters at the BIPM to support the Watt balance project, *Proc. IAG Symposium*, **139**, 2012.
5. Jiang Z., Becker M., Jousset P., Coulomb A., Tisserand L., Boulanger P., Lequin D., Lhermitte F., Houillon J.L., Dupont F., High precision levelling supporting the International Comparison of Absolute Gravimeters, *Metrologia,* 2012, **49**(1), 41-48.
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11. Jiang Z, Lewandowski W., Inter-comparison of the UTC time transfer links, *Proc. EFTF 2012,* 2012, 126-132.
12. Jiang Z., Pálinkáš V., Francis O., Jousset P., Mäkinen J., Merlet S., Becker M., Coulomb A., Kessler-Schulz K.U., Schulz H.R., Rothleitner Ch., Tisserand L., Lequin D., Relative Gravity Measurement Campaign during the 8th International Comparison of Absolute Gravimeters (2009), *Metrologia,* 2012, **49**(1), 95-107.
13. Jiang Z., Pálinkáš V., Arias F.E., Liard J., Merlet S., Wilmes H., Vitushkin L., Robertsson L., Tisserand L., Pereira Dos Santos F., Bodart Q., Falk R., Baumann H., Mizushima S., Mäkinen J., Bilker-Koivula M., Lee C., Choi I.M., Karaboce B., Ji W., Wu Q., Ruess D., Ullrich C., Kostelecký J., Schmerge D., Eckl M., Timmen L., Le Moigne N., Bayer R., Olszak T., Ågren J., Del Negro C., Greco F., Diament M., Deroussi S., Bonvalot S., Krynski J., Sekowski M., Hu H., Wang L.J., Svitlov S., Germak A., Francis O., Becker M., Inglis D., Robinson I, The 8th International Comparison of Absolute Gravimeters 2009: the first Key Comparison (CCM.G-K1) in the field of absolute gravimetry, *Metrologia,* 2012, **49**(6), 666-684.
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19. Petit G., Panfilo G., Comparison of frequency standards used for TAI, *IEEE T. Instrum. Meas.,* 2012, **99**, 1-6.

BIPM publications

20. BIPM Annual Report on Time Activities for 2011, **6**, 105 pp., available only at www.bipm.org/en/publications/time_activities.html.
21. *Circular T* (monthly), 7 pp.
22. Liard J., Pálinkáš V., Jiang Z., The self-attraction effect in absolute gravimeters and its influence on CIPM key comparisons, *Rapport BIPM*-2012/01, 12 pp.

2.12 Activities related to the work of Consultative Committees

E.F. Arias is Executive Secretary of the Consultative Committee for Time and Frequency (CCTF). She is a member of the CCTF Working Group on Two-Way Satellite Time and Frequency Transfer (TWSTFT), the CCTF Working Group on Primary Frequency Standards (WGPFS) and the CCTF Working Group on TAI (WGTAI).

Z. Jiang is a member of the CCTF Working Group on TWSTFT.

W. Lewandowski is Secretary of the CCTF Working Group on TWSTFT and Secretary of the CCTF Working Group on Global Navigation Satellite Systems Time-Transfer Standards (CGGTTS).

G. Panfilo is a member of the CCTF Working Group on Primary Frequency Standards (WGPFS) and of the Sub-Group on Algorithms of the CCTF Working Group on TAI and collaborates with the CCTF Working Group on the CIPM MRA (WGMRA).

G. Petit is a member of the CCTF Working Group on TAI and its Sub-Group on Algorithms, of the WGPFS, and of the CGGTTS.

L. Robertsson is Executive Secretary of the Consultative Committee for Length (CCL) and 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).

2.13 Activities related to external organizations

E.F. Arias is a member of the IAU and participates in its working group on the International Celestial Reference System, she co-chairs the working group on the redefinition of UTC. She is an associate member of the IERS, a member of its International Celestial Reference System Centre, and of the Conventions Centre of the IERS. She is a member of the International VLBI Service (IVS), and of its Analysis Working Group on the International Celestial Reference Frame. She is the BIPM representative to the Governing Board of the IGS. She is the BIPM representative to the International Committee for GNSS and she is the chairperson of the Task Force on Time References. She is a member of the Global Geodetic Observing System (GGOS) Steering Committee representing the BIPM. She is a member of the Argentine Council of Research (CONICET) and an associated astronomer at the LNE-SYRTE, Paris Observatory. She is a corresponding member of the *Bureau des longitudes*. She is the BIPM representative to the Working Party 7A of the Study Group 7 of the International Telecommunication Union – Radiocommunication Sector (ITU-R).

W. Lewandowski is the BIPM representative to the Civil GPS Service Interface Committee and chairman of its Timing Sub-Committee. He is a member of the Scientific Council of the Space Research Centre of the Polish Academy of Sciences. He is also a member of a consultative Group on the Reform of Metrology at the Polish Ministry of Economy, an adviser to a Parliamentary Group on Space, and a member of the Committee on Research on Space Techniques of the Polish Academy of Sciences. He is member of European Commission Advisory Group on Galileo Time Infrastructure. Together with E.F. Arias, he is the BIPM representative to the Working Party 7A of the Study Group 7 of the ITU-R, and the UN International Committee on GNSS (ICG).

G. Petit is co-director of the Conventions Centre of the IERS. He is president of the IAU Commission 52 'Relativity in Fundamental Astronomy', member of the IAU Working Group on Numerical Standards in Fundamental Astronomy, of the IGS Working Group on Clock Products, of the GNSS Science Advisory Committee of the ESA, and of the Fundamental Physics Group of the CNES.

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

2.14 Travel (conferences, lectures and presentations, visits)

E.F. Arias to:

- Geneva (Switzerland), 16-20 January 2012, for the Radiocommunication Assembly 2012 at the International Telecommunication Union;
- La Plata (Argentina), 19-23 March 2012, for the ADeLa 2012 meeting as invited lecturer and to organize a panel discussion on the future of UTC;
- Gothenburg (Sweden), 24 to 27 April 2012, for the European Frequency and Time Forum (EFTF) and for the meetings of the CCTF WGs on Strategic Planning (as the secretary), on Coordination of the Development of Advanced Time and Frequency Transfer Techniques and on TWSTFT;
- Baltimore (USA), 22 to 24 May 2012, for the IEEE-IFCS 2012 and to give an invited lecture;
- Beijing (China), 22 to 31 August 2012, for the XXVIII General Assembly of the International Astronomical Union, including the Joint Discussion 7 on Reference Systems, and meetings of Commissions 8, 19 and 30, and to visit the NTSC (Lintong) and the NIM;
- Manta (Ecuador), 20 to 29 September 2012, for the ITU Seminar for the Americas, invited to give a lecture and for the meeting of the Working Party 7A as delegate of the BIPM;

Z. Jiang to:

- Vienna (Austria), 13-15 February 2012, for the Joint discussion meeting of IAG JWG2.1 and JWG2.2 at the BEV, with a presentation on the ICAG 2009;
- Gothenburg (Sweden), 24 to 27 April 2012, for the European Frequency and Time Forum (EFTF) with oral and poster presentations, and for the meeting of the CCTF WG on TWSTFT, (acting as secretary);
- Istanbul (Turkey), 29 and 30 May 2012, for the CCM-WGG meeting;
- Reston (USA), 26-29 November 2012, for the 44th PTTI, invited presentations and for a meeting of TW participant laboratories;

W. Lewandowski to:

- Vienna (Austria), 13 February 2012, for the preparatory meeting of the 7th ICG;
- Braunschweig (Germany), 17 April 2012, for the meeting of the Galileo FOC Timing Interface Working Group;
- Warsaw (Poland), 28 June to 3 July 2012, to the Space Research Centre and Space Commission;
- Nashville (Tennessee, USA), 17 to 21 September 2012, for the ION meeting and for the meeting of the Civil GPS Interface Committee (CGSIC) acting as chair of the Timing Sub-committee;
- Beijing (China), 5-9 November 2012, for the 7th Meeting of the ICG and to chair the meeting of the WG D Task Force on Timing References;
- Warsaw (Poland), 19 to 21 November, for meetings on the Galileo system, organized by the European Commission;
- Reston (USA), 26-29 November 2012, for the 44th PTTI, invited presentations and for a meeting of TW participant laboratories;

G. Panfilo to:

- Gothenburg (Sweden), 24 to 27 April 2012, for the European Frequency and Time Forum (EFTF) to give an invited lecture and for the meetings of the CCTF WGs on Coordination of the Development of Advanced Time and Frequency Transfer Techniques and on TWSTFT;
- Turin (Italy), 11-12 October 2012, invited to give a lecture on "Algorithms for the International Atomic Time" at the University of Turin;

G. Petit to:

- Bern (Switzerland), 18-19 January 2012, to attend the IGS workshop on GNSS biases, with oral presentations;
- Paris (France), 1 February 2012, for the meeting of the *Groupe de travail de Physique Fondamentale* of the CNES;
- Paris (France), 8 February 2012, to attend the kick-off meeting of the Labex First-TF network;
- Paris (France), 23 March and 4 October 2012, to attend meetings of the GNSS Science Advisory Committee;
- Vienna (Austria), 20-22 April 2012, to attend a workshop of the IERS Global Geophysical Fluid Center and a meeting of the Directing board of the IERS;
- Gothenburg (Sweden), 24-27 April 2012, to attend the European Frequency and Time Forum, and presentation;
- Alicante (Spain), 11-12 May 2012, to chair a PhD jury;
- Washington DC (USA), 1-5 July 2012, to attend the Conference on Precision Electromagnetic Measurements, to give a presentation, and to visit the USNO and NRL time laboratories;
- Paris (France), 14 November 2012, to attend the *Rencontres de l'Observatoire de Paris*;
- Hoofddorp (The Netherlands), 20-21 November 2012, to attend the workshop 'Optical networks for accurate time and frequency transfer'.

L. Robertsson to:

- Gothenburg (Sweden), 24 to 27 April 2012, for the European Frequency and Time Forum (EFTF) and for the meeting of the CCTF WG on Coordination of the Development of Advanced Time and Frequency Transfer Techniques and on TWSTFT (as the secretary);
- Hoofddorp (The Netherlands), 20-21 November 2012, for the Workshop on Optical Networks for Accurate Time and Frequency Transfer.

2.15 Visitors

- P. Lejba, Space Research Centre of the Polish Academy of Sciences (Poland), for activities on GNSS time transfer and calibration, under the supervision of W. Lewandowski, 6 to 9 February 2012;
- L.S. Ma, State Key Laboratory of Precision Spectroscopy East China Normal University (China), for discussions on optical frequency activities, 9 to 18 September 2012;
- M. Zucco, INRIM (Italy), for discussions on optical frequency activities, 9 to 18 September 2012;
- Y. Almleaky, Makkah Time Centre (Saudi Arabia), for discussions about the contribution to UTC;
- J. Davis, NPL (UK), for an external audit of the Time Department, 6 December 2012.

BIPM Electricity Department

Director: M. Stock

(1 January 2012 to 31 December 2012)

3.1 Electrical potential difference (voltage) (R. Chayramy, S. Solve)

During the first part of 2012, work in the laboratory focused on the maintenance of the primary and secondary voltage standards, the test of the bias current source for the Josephson voltage standard for the BIPM watt balance, and on the comparison programme.

The BIPM's new automated measurement set-up for the calibration of Zener secondary voltage standards is now operated on a regular basis. Details of the switching system (scanner) developed at the BIPM to connect up to 12 Zener secondary standards with the primary Josephson voltage standard by introducing only very low thermal electromotive forces (EMFs), were presented at the Conference on Precision Electromagnetic Measurements (CPEM) held in Washington DC, USA, in July 2012. After more than one hundred thousand connection operations degradation of the quality of the silver contacts was experienced. The corresponding error on the calibrations has been evaluated and is below the noise floor of the Zener standards and thus does not affect the quality of the calibration results. However, the voltage offsets which comprise residual thermal EMFs and contact EMFs at each input of the scanner need to be monitored to guarantee the metrological performance of the scanner. At present the automatic calibration set-up allows calibrations of the 1.018 V Zener voltage output. A study was carried out to evaluate a possible extension of the present system to the level of 10 V using a 10 V programmable array.

The Electricity Department is developing a Josephson array voltage standard for the watt balance experiment. It will be used to measure the induced voltage of the induction coil, when it moves through the magnetic field. The programmable quantized Josephson array voltage will be opposed to the voltage over the coil and the small difference will be measured with a nanovoltmeter. The programmable Josephson SNS array consists of 13 segments, which are independently biased by a 13-channel bias current source. The first tests of the bias current source together with the SNS array showed that some adjustments were required to prevent the superconducting structures of the array from trapping magnetic flux during changes in the bias currents when the array voltage changed. The bias current source is now operational. Details of the apparatus were presented on a poster and in a paper at the CPEM conference held in Washington DC, USA, in July 2012. Furthermore the problems with the array identified in 2011 were confirmed by our colleagues at the NIST. The failures are related to the partial destruction of the on-chip leads for three least-significant-bit sub-arrays and damaged radiofrequency (RF) splitters for the most-significant-bit arrays. The RF splitters can be repaired whereas the damage to the LSB sub-arrays is irreversible. These failures relate to the large number of temperature cycles performed on the array since its delivery to the BIPM in 2006. The array can no longer be operated for the measurement of the induced voltage produced across the moving coil of the watt balance because all the sub-arrays are required for successful operation of this procedure. The NIST has offered to replace the array.

The Electricity Department carries out an ongoing on-site comparison of Josephson voltage standards, BIPM.EM-K10, at 1.018 V and 10 V. For this purpose a new transportable Josephson voltage standard (JVS) is being developed. A problem with the propagation of the RF signal along the oversized circular waveguide and the antenna on the chip was encountered in 2011 and no solution has been found so far. The reason why the array does not seem to receive the RF power which is available at the end of the waveguide remains difficult to understand. However, ideas for new experiments to determine the origin of this major problem exist. In the meantime, the new BIPM transportable JVS is operational with the previous measurement probe.

A peer review of the voltage metrology laboratory was conducted by an external expert in May 2012 which demonstrated the very satisfactory application of the BIPM Quality Management System in the laboratory.

Dr S. Solve has been sent on secondment to the NIST (Boulder, USA) for one year from July 2012 to learn about the use of NIST SNS arrays in the field of synthesis of AC voltages and corresponding applications in AC voltage metrology. The BIPM plans to propose a comparison in the field of AC voltages in the future. The work has initially focused on the determination of the metrological characteristics of a 20-bit digitizer operated to sample the voltage difference between the synthesized stepwise waveform produced by the programmable Josephson voltage standard (PJVS) and the sine-wave produced by a calibrator. The sampler measurements allow reconstruction of the sine-wave and therefore calibration of the calibrator signal with respect to the calculable quantum voltage levels of the PJVS.

In parallel, investigations have been carried out on 13 SNS 1 V arrays to determine their individual characteristics such as the number of junctions per sub-array (compared to the theoretical numbers) and the determination of the operating margins of the bias currents as a function of frequency and power level. The objective is to select a new array for the BIPM watt balance experiment, to replace the defective array.

A large number of experiments have been carried out to determine a typical value for the leakage resistance to ground (LRG) of a NIST 10 V programmable Josephson voltage standard. Two different measurement techniques have been implemented and software developed to conduct the measurements and to analyse the data. Two different types of digital nanovoltmeters have been used to cross-check the results. It is important to apply different measurement techniques and corresponding instrumentation as the experiments are sensitive to the level of electromagnetic noise and in particular to the electrical field distribution around the set-up. All results were found to converge towards the same conclusions:

1. The leakage resistance of the complete PJVS system is limited by that of the power supply which provides electrical energy to the PJVS bias source;
2. The leakage resistance to ground decreases proportionally with the number of DAC cards inserted in the PXI chassis. The LRG of a single card varies from 60 GΩ to 110 GΩ depending on the individual characteristics of the components of the DAC card;
3. The LRG value depends on the Josephson array chip and the cryo-probe;
4. While the low potential side of the PJVS remains floating from the ground, its leakage resistance to ground does not introduce an error on the PJVS voltage output. However, if the low potential side of the array needs to be grounded, for instance, in a direct comparison measurement with another JVS, the leakage resistance to ground will introduce a diminution of the PJVS output voltage, and will thus introduce a systematic voltage error.

The process to determine the leakage resistance to ground has been integrated into the NIST PJVS core software as a basic, but necessary check of the quantum standard. A measurement procedure has also been written and integrated into the user manual.

3.2 Electrical impedance (resistance and capacitance)

3.2.1 DC resistance and quantum Hall effect (N. Fletcher, R. Goebel, B. Rolland)

This activity is mainly dedicated to resistance comparisons and calibrations for NMIs and includes the maintenance of the primary references and facilities, which are used both for measurement services and research. The BIPM derives the resistance unit, the ohm, from the quantized Hall resistance. The operation of the quantum Hall effect is also required for the BIPM watt balance and for the derivation of the capacitance unit, the farad.
Calibration activities were interrupted for several months in 2012 as a result of repeated failures of the air conditioning systems in some of the electricity laboratories. Sudden temperature changes can lead to changes of the resistance values of the reference standards, which then have to be recalibrated against the quantized Hall resistance. The problem has been fixed, and replacement of the old air conditioning systems will take place in

the near future. Meanwhile, it has been decided to place the main conventional working standard in a new commercial large-size thermo-controlled enclosure, to protect it from possible thermal shocks. The power electronics of that enclosure had to be modified to avoid generation of high-frequency noise. During the interruption to the service, requests for calibrations and comparisons were postponed; these are now being carried out between September 2012 and early 2013.

The BIPM is preparing to restart the scheme of ongoing on-site comparisons of quantum Hall resistance standards and the associated measurement bridges (BIPM.EM-K12), upon request from member laboratories of the Consultative Committee for Electricity and Magnetism (CCEM). Five such comparisons were carried out between 1993 and 1999, with relative uncertainties of several parts in 10^9. Recently 15 NMIs have declared an interest in such a comparison with the BIPM, because this is the only method to verify the very low intrinsic uncertainty of quantum Hall resistance standards. Comparisons using wire-wound transfer resistors are limited by stability of the resistors during travel to a few parts in 10^8. The Electricity Department is therefore preparing the equipment needed for a transportable quantum Hall resistance standard. Three thermally stabilized enclosures for transportable resistance standards were constructed in 2012, on the basis of a prototype successfully developed in 2011. These enclosures achieve a temperature stability of better than 1 mK. The resistance elements which had been developed to be installed in the enclosure have been characterized with respect to their temporal stability and temperature coefficient. The temperature coefficients were found to be significantly larger than those specified by the manufacturer and investigations have been initiated by the supplier. A transportable 1 Hz bridge based on a BIPM design, optimized for the future on-site QHR comparisons, and a new cryostat have already been prepared. It is planned to carry out the first comparison in 2013.

The traceability chain from quantized Hall resistance to different resistance values critically relies on cryogenic current comparators (CCCs). The systems operated by the BIPM currently rely on analogue electronics concepts developed in the 1980s. The Electricity Department plans to investigate a CCC based on modern commercially available numerically controlled current sources as a more convenient and potentially more accurate alternative. Progress was made in the construction of a new CCC-based comparison bridge using such current sources: a high-accuracy resistance divider for the compensation current was built, and the software is under development.

A triple-shielded 40 m cable was installed in 2011 to link the electricity laboratory and the watt balance facility to enable the periodic calibration of the reference resistor used in the watt balance current source in-situ. It has been demonstrated that the long cable length does not significantly degrade the measurement results. The reference resistor initially chosen for the current source was found to be insufficiently stable and was therefore replaced by another type of high-quality standard resistor.

An external quality audit of the resistance measurement service took place in December 2012.

3.2.2 Maintenance of a reference of capacitance (R. Chayramy, N. Fletcher, R. Goebel)

The capacitance reference at 10 pF has been maintained by regular links to the quantum Hall resistance using a quadrature bridge (twice over the reporting period). The working standards were used to disseminate the farad via comparisons and calibrations as listed in sections 4 and 5 of this report. During 2012, 30 capacitance calibration certificates were issued for eight different NMIs.

An external quality audit of the capacitance measurement service took place in December 2012.

The BIPM participated in the EURAMET comparison EM-S31 of 10 pF and 100 pF standards traceable to the QHR during 2011. This comparison was started within the framework of the EMRP Joint Research Project "*Quantum Ampere: Realisation of the new SI ampere*" (REUNIAM). The draft A comparison report has shown some unexplained discrepancies, particularly in the frequency dependence of standards. A discussion meeting was held at the CPEM conference in July 2012 to look for ways forward to explain these results. It was decided to carry out supplementary measurements of the frequency dependence of resistors, which is a key step in the chain from the dc QHR to ac capacitance. Two 12.906 kΩ (= $R_K/2$) secondary ac-dc reference standards, one provided by the BIPM and one by the LNE are being circulated between four laboratories. The quantity to be compared is the variation in the resistance (using a four terminal-pair definition) between dc and frequencies up to a few kHz.

The reference at the BIPM for such measurements is a pair of coaxial resistors (Haddad type) with a simple geometry and calculable frequency dependence. The secondary standards used in the quadrature bridge have a stable frequency dependence, and only the absolute dc value is calibrated regularly. Full characterization of these secondary standards against the coaxial reference has not been performed for several years, and is now being studied as part of this comparison. One of the injection transformers in the bridge used for these measurements was found to be defective, and a replacement has been developed. New software for running the bridge has also been written.

The results of the supplementary measurements will be available in the first half of 2013, and will provide an important test between the BIPM and other laboratories with top-level capacitance traceability and coaxial bridge systems. The knowledge of the frequency dependence of the standards used in the quadrature bridge is one of the critical items in the uncertainty budget for the link between R_K and capacitance. The characterization of this component and the opportunity to compare directly with other laboratories at this time fits in very well with the ongoing work on the calculable capacitor (see section 3).

3.3 **Calculable capacitor** (N. Fletcher, R. Goebel, L. Robertsson, M. Stock)

Construction of the BIPM calculable capacitor started in early 2010 and is now essentially complete. Several major modifications were implemented after the testing of the main elements of the mechanical design during the secondment of John Fiander from the NMIA in 2010. After the construction of two sets of modified parts by the BIPM workshop, the formal collaboration with the NMIA was completed at the end of 2011.

The remaining mechanical problem with the moveable lower guard was resolved in 2012. The sensitivity to external vibration has been reduced by changes to the spring mounting, to the level that the interferometer can now be reliably operated. A system of servo electronics has been developed that uses the frequency modulation already present in the laser system to lock the cavity length to a maximum of the interferometer fringe pattern. The cavity is sufficiently stable to allow the servo to maintain a fringe lock for several hours, which is longer than the time required for capacitance measurements.

The improved stability of the interferometer signal has also allowed the development of an alignment technique to ensure the parallelism of the main electrodes, the laser beam and the movement of the upper guard electrode. An external autocollimator is used to monitor any rotation of the upper end of the moveable guard; the lower end is mechanically guided by a tight fitting PTFE ring. After iteration of the various mirror and beam alignments, the amplitude of the fringe signal produced by the interferometer remains unchanged along the entire travel of the guard, meaning that the angle between the upper mirror and the laser beam does not change. These conditions taken together mean that the optical length measured with the interferometer is the same as the electrical length that defines the capacitance change measured in the experiment. Further optimization of the interferometer is planned with the aid of a CCD camera installed to monitor the beam shape. This will allow, for example, a study of any undesirable higher order modes excited in the cavity.

The vacuum enclosure for the capacitor has been installed and several minor leaks detected and eliminated. A particular problem was found with the adhesive used in the custom coaxial connectors designed for the main electrode connections. The alignments of the upper guard electrode are driven by rotary piezo-motors to allow adjustment from outside the vacuum enclosure. These have proven to give sufficient control and sensitivity of the interferometer alignments once the instrument is sealed and under vacuum.

With the optical system aligned and the interferometer servo working, and the capacitor under vacuum, it was possible to proceed to some first measurements with the complete system. The first step is to scale the capacitance change given by the calculable capacitor (a 0.4 pF change from 0.2 pF to 0.6 pF, corresponding to a length change of 0.2 m) to a fixed 1 pF standard. A bridge for this purpose was constructed at the start of the project, but had not been fully used until now. It performed as expected when used with the real calculable capacitor. The noise level of the servo-locked capacitor combined with the bridge gives a resolution of around 1 part in 10^8 within a 5-10 minute measurement. A measurement algorithm for calibrating the 1 pF standard has been developed. The value measured in this way can be compared with the value of the same standard calibrated by the BIPM's existing chain of bridges linking to the quantum Hall effect (and the value of R_K). This is the same chain that is regularly operated to provide traceability for the BIPM's capacitance calibration services. The status of this work was presented at the CPEM conference held in Washington DC, USA, in July 2012.

The comparison of these two routes to 1 pF agreed to better than 2 parts in 10^8, which was very encouraging. This cannot be considered as a full measurement of R_K using the calculable capacitor, and a proper uncertainty budget has not yet been established. It is however an indication that there is no major problem with the new instrument. At present, the limiting factor is the main electrode alignment in the capacitor. The techniques for adjusting and verifying all the necessary alignments were demonstrated during the construction, but after the installation of all the modified parts, the capacitor was not rigorously set up. After these first check measurements, the capacitor will be fully stripped down, cleaned and carefully assembled and aligned as we progress towards a complete R_K measurement in 2013.

3.4 BIPM ongoing comparisons in electricity (R. Chayramy, N. Fletcher, R. Goebel, S. Solve, M. Stock)

One on-site comparison of Josephson voltage standards with the METAS (Switzerland) has been successfully completed both at 1 V and 10 V. The reports on 3 previous comparisons with the INTI (Argentina), the MSL (New Zealand) and the CENAM (Mexico) have been published and very good agreement was observed between the results obtained by the BIPM and the participants:

at 1.018 V:	$(U_{INTI} - U_{BIPM}) = -1.1$ nV	$u = 2.0$ nV
at 1.018 V:	$(U_{METAS} - U_{BIPM}) = +0.3$ nV	$u = 0.4$ nV
at 10 V:	$(U_{MSL} - U_{BIPM}) = +2.5$ nV	$u = 4.0$ nV
at 10 V:	$(U_{CENAM} - U_{BIPM}) = -0.6$ nV	$u = 0.7$ nV
at 10 V:	$(U_{METAS} - U_{BIPM}) = 0.3$ nV	$u = 1.0$ nV

The comparison with the CENAM indicated some limitations of their Josephson voltage standard and led to an improvement after the comparison.

One bilateral voltage comparison using Zener voltage standards as transfer standards was carried out at the level of 10 V with the NSAI (Ireland) in March 2012. The comparison results have been published and very good agreement was observed between the results obtained by the BIPM and the NSAI.

at 10 V: $(U_{NSAI} - U_{BIPM}) = 0.83$ μV $u = 1.35$ μV

A resistance comparison with the NPLI (India), at 1 Ω and 10 kΩ, was planned for early 2012 but was postponed due to the air conditioning problems mentioned earlier. After a first series of measurements at the BIPM, the transfer standards were shipped to India in November 2012. Comparisons with the BIM (Bulgaria) and the NSAI (Ireland) were also postponed and are expected to start in the near future.

The final report of a comparison at 10 kΩ with the KRISS (Republic of Korea) has been published with the result:

$$(R_{KRISS} - R_{BIPM}) / (10\ \text{k}\Omega) = -0.001 \times 10^{-6} \qquad u = 0.016 \times 10^{-6}$$

A capacitance comparison at the level of 10 pF and 100 pF was carried out with the BIM (Bulgaria). The draft A report is being prepared.

3.5 Calibrations (R. Chayramy, N. Fletcher, R. Goebel, B. Rolland, S. Solve, M. Stock)

Due to problems with the air conditioning of the electricity laboratories, the resistance calibration service had to be interrupted for several months in 2012. A number of planned calibrations had to be postponed and are now scheduled to be carried out at the end of 2012. During the period from January 2012 to December 2012 the Electricity Department issued certificates for the following standards:

Resistance standards at 1 Ω, 100 Ω or 10 k Ω were calibrated for: UTE (Uruguay), CMI (Czech Republic), INMETRO (Brazil), NMIA (Australia), BIM (Bulgaria), corresponding to 15 certificates. Calibrations for NIMT (Thailand) are under way. Requests are pending from NIS (Egypt), BEV (Austria), A*STAR (Singapore) and SMD (Belgium).

Capacitance standards at 1 pF, 10 pF and 100 pF were calibrated for: UME (Turkey), A*STAR (Singapore), MKEH (Hungary), NIMT (Thailand), VSL (The Netherlands), NRC (Canada), CEM (Spain), NMISA (South Africa), corresponding to 30 certificates. Requests are pending from BEV (Austria) and INM (Romania).

One Zener voltage standard was calibrated for the BIPM Ionizing Radiation Department.

3.6 Publications

1. Janssen T.J.B.M., Williams J.M., Fletcher N.E., Goebel R., Tzalenchuk A., Yakimova R., Lara-Avila S., Kubatkin S., Falko V.I., Precision comparison of the quantum Hall effect in graphene and gallium arsenide, *Metrologia*, 2012, **49**(3), 294-306.
2. Solve S., Stock M., BIPM direct on-site Josephson voltage standard comparisons: 20 years of results, *Meas. Sci. Techno*. **23** (2012) 124001 (10 pp).
3. Solve S., Chayramy R., Picard A., Kiss A., Fang H., Robertsson L., de Mirandés E., Stock M., A bias source for the voltage reference of the BIPM watt balance, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM)*, 2012, 52-53.
4. Aviles D., Navarrete E., Hernández D., Solve S., Chayramy R., Direct comparison of Josephson Voltage Standards at 10 V between BIPM and CENAM, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM)*, 2012, 68-69.
5. Chayramy R., Solve S., A very low thermal EMFs computer-controlled scanner, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM)*, 2012, 548-549.
6. Fletcher N., Goebel R., Robertsson L., Stock M., Progress towards a determination of R_K at the BIPM, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM)*, 2012, 252-253.
7. Rolland B., Goebel R., Fletcher N., A transportable thermoregulated enclosure for standard resistors, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM)*, 2012, 378-379.
8. Power O., Solve S., Chayramy R. and Stock M., Bilateral comparison of 10 V standards between the NSAI-NML (Ireland) and the BIPM, March to April 2011 (part of the ongoing BIPM key comparison BIPM.EM-K11.b), *Metrologia*, 2012, **49**, *Tech. Suppl.*, 01014.
9. Solve S., Chayramy R., Stock M., Jeanneret B., Comparison of the Josephson voltage standards of the METAS and the BIPM (part of the ongoing BIPM key comparisons BIPM.EM-K10.a and BIPM.EM-K10.b), *Metrologia*, 2012, **49**, *Tech. Suppl.*, 01010.

10. Solve S., Chayramy R., Stock M., Avilés D., Navarrete E., Hernández D., Comparison of the Josephson voltage standards of the CENAM and the BIPM (part of the ongoing BIPM key comparisons BIPM.EM-K10.b), *Metrologia*, 2012, **49**, *Tech. Suppl.*, 01011.

11. Solve S., Chayramy R., Stock M., Iuzzolino R., Tonina A., Comparison of the Josephson voltage standards of the INTI and the BIPM (part of the ongoing BIPM key comparisons BIPM.EM-K10.a), *Metrologia*, 2012, **49**, *Tech. Suppl.*, 010109.

12. Solve S., Chayramy R., Stock M., Christian L., Comparison of the Josephson voltage standards of the MSL and the BIPM (part of the ongoing BIPM key comparisons BIPM.EM-K10.b), *Metrologia*, 2012, **49**, *Tech. Suppl.*, 01015.

3.7 Activities related to the work of Consultative Committees

M. Stock is the Executive Secretary of the Consultative Committee for Electricity and Magnetism (CCEM) and the Consultative Committee for Photometry and Radiometry (CCPR) and a member of several of their working groups. The 21st CCPR meeting was held on 23 to 24 February 2012. At present the next CCEM meeting is being organized for March 2013 and CCPR working group meetings are planned for April 2013.

R. Goebel organizes the review of comparisons reports and protocols within the CCPR Working Group on Key Comparisons (WG-KC).

N. Fletcher acted as the scientific secretary of the BIPM Workshop on Challenges in Metrology for Dynamic Measurement, held at the BIPM headquarters on 15 to 16 November 2012.

3.8 Activities related to external organizations

M. Stock is a member of the Conference on Precision Electromagnetic Measurements (CPEM) Executive Committee. N. Fletcher and M. Stock are members of the Technical Committee of CPEM 2012. M. Stock is a member of the Scientific Committee for the 4th International Conference on Quantum Metrology (QM2013), to be held in Poznan, Poland, on 15 to 17 May 2013.

M. Stock is the contact person for the BIPM liaison with the International Commission on Illumination (CIE). A joint CCPR-CIE workshop was held on 22 February 2012 to coordinate the work between both organizations on the future *mise en pratique* for the definition of the candela.

N. Fletcher represents the BIPM on the 'Stakeholder Committee' for the EMRP Project GraphOhm (funded to start in July 2013).

3.9 Travel (conferences, lectures and presentations, visits)

S. Solve and R. Chayramy to:

- METAS, Bern (Switzerland), 24 January to 1 February 2012, to carry out a direct on-site BIPM Josephson voltage standard comparison.

M. Stock to:

- EURAMET TCPR, Helsinki (Finland), 7-8 March 2012;
- International School of Physics "Enrico Fermi", Varenna (Italy), 25-26 July 2012, to give a lecture on "Watt balance experiments for the determination of the Planck constant and the redefinition of the kilogram";
- LNE (France), 26-27 September 2012, to attend the kick-off meeting of the EMRP JRP kNOW (Realisation of the awaited definition of the kilogram).

M. Stock, N. Fletcher and S. Solve to:

- Boulder (United States), 30 June - 6 July 2012, CPEM 2012 and satellite meetings:
  - M. Stock attended the CPEM conference and meetings of CCEM working groups: WGLF (low frequency), GT-RF (radiofrequency), WGRMO and WGkg (monitoring the kilogram) and the CPEM Executive Committee meeting;
  - N. Fletcher gave a presentation on the BIPM calculable capacitor and presented a poster. He attended the discussion meeting on EURAMET.EM-S.31;
  - S. Solve presented two posters and attended the CCEM WGkg and WGLF, and a EURAMET workshop.

S. Solve to:

- NIST (United States), 1 July 2012 - 31 July 2013, as a guest researcher.

N. Fletcher to:

- SMU, Bratislava (Slovakia), 11-12 October 2012, EURAMET TCEM;
- NPL, Teddington (UK), 15-16 October 2012, to attend the conference 'Graphene: from Research to Applications'.

Presentations

- N. Fletcher gave a presentation at the BIPM headquarters, 9 February 2012, '*From wire wound resistors to epitaxial graphene: 30 years of progress in resistance measurements*' as part of the '*Hommage à Pierre Giacomo*'.

3.10 Visitors

- F. Piquemal, O. Thévenot, P. Gournay, G. Thillier, LNE (France), to discuss on the calculable capacitor, 10 January 2012;
- C. Williams, NIST (USA), to visit the laboratories, 24 January 2012;
- I.Budovsky, NMIA (Australia), to visit the laboratories, 4 April 2012;
- G. Small, NMIA (Australia), to discuss progress on the calculable capacitor project, 27 April 2012;
- R. Behr for a peer-review of the voltage metrology, 21-22 May 2012;
- M. Homklintian, NIMT (Thailand), to discuss calculable capacitor measurements, 20 August 2012;
- F. Torres, H.D. Alzate, A. Giraldo, A.P. Gomez (Delegation from Columbia), 24 August 2012;
- Speakers and steering committee members of the Workshop in Metrology for Dynamic Measurements (10 persons), 14 November 2012;
- C.S. Wey, A*STAR (Singapore), for a peer-review of the resistance activity, 29 November 2012;
- O. Thévenot, LNE (France), for a peer-review of the resistance activity, 4 December 2012;
- J.H. Ullrich (President, PTB), to visit the laboratories, 4 December 2012;
- R. Sayed, NIS (Egypt), to deliver resistors for calibration and to visit the electricity laboratories, 11 December 2012;
- P. Scheibenreiter, BEV (Austria), to deliver resistance and capacitance standards, 11 December 2012.

BIPM watt balance

(1 January 2012 to 31 December 2012)

4.1 **Watt balance** (R. Chayramy, A. Dupire, H. Fang, A. Kiss, J. Lan[1], E. de Mirandés, A. Picard, L. Robertsson, S. Solve, M. Stock, A. Zeggagh[2])

The BIPM watt balance is designed to operate in a conventional two-phase mode, which is used by other watt balances, as well as in an original single phase mode, in which the usual two phases are carried out simultaneously, thus eliminating the drifts that occur between both phases. Studies have been conducted to overcome the main difficulty in the novel BIPM simultaneous measurement approach: separating the resistive voltage drop over the coil (due to the current flowing through the coil) from the induced voltage (due to its movement through a magnetic field). In particular, a new technique using a bifilar coil has been intensively investigated. In this technique the current flows through one of the coils, while a voltage is induced – and can be measured – in both coils. In the current-carrying coil the induced voltage is superimposed on the resistive voltage drop, in the other coil only the induced voltage is present. This offers the interesting possibility to determine the Planck constant h in three different ways from one single measurement series: using only one (or the other) coil for the current flow and the voltage measurement, or combining the use of both coils, by measuring the voltage in the coil which does not carry the current. Moreover, the results of the approaches using only one coil or combining both coils can be compared with a lower uncertainty than that of individual determinations of h since most measurement quantities are common for both methods (and therefore their uncertainties are completely correlated). A set of 14 series of measurements was carried out without any perturbation of the experiment in January and February 2012. For each series of measurements, one value of the Planck constant was calculated using each of the three methods described above with a type A uncertainty of a few parts of 10^{-6}. The relative repeatability among the 14 series of measurements was in the order of 10^{-6} for all h determinations. The relative difference between the approaches using only one coil or combining both coils was 2×10^{-7} with a type A uncertainty of 3×10^{-7}, so that in this particular study both approaches agreed at the 10^{-7} level. The bifilar coil technique appears to be a promising approach because it allows a watt balance to be operated in a simultaneous measurement scheme without the superimposition of the two types of voltage.

The bifilar coil was also used to assess the performance of the method used at the BIPM to eliminate the unwanted resistive voltage drop on the normal mono-filar induction coil. To eliminate the resistive voltage drop the method essentially combines a pair of data taken at the same vertical position inside the magnetic field during up and down movements of the coil. The effect of any residual resistive voltage, after application of this technique, is evaluated by observing the ratio of the induced voltages across two windings when a current is driven successively through one winding and then through the other. Test measurements were carried out under conditions of poor temperature stability and the results showed that the accuracy of the data combination method is particularly affected by changes in the resistance of the coil due to temperature drift. Two additional methods have been developed to refine the data combination technique. The effect due to imperfect removal of the resistive voltage was largely reduced but further improvement is necessary.

The BIPM has successfully validated the dynamic coil alignment mechanism based on piezoelectric actuators. When measuring the Planck constant the system allows for compensation of undesirable deviations of the moving coil from a purely vertical movement during its travel in the working mode. The system also allows tilts and translations of the coil during alignment due to torques and forces to be distinguished. The performance of the system was evaluated on an independent set-up. Servo-control of the horizontal position and inclination was achieved either independently or simultaneously. The position stability over 1 minute (duration of travel of the coil over its total path) was better than 0.5 µm and the angular stability was within 0.5 µrad. An improved

[1] PhD student from Tsinghua University (China), January to June 2012
[2] Student from Ecole des Mines, Douai (France), May 2011 to September 2012

version of the system is being fabricated in the BIPM workshop before its integration into the watt balance experiment in 2013.

A new current source, fully automated and capable of delivering currents of 1 mA, 2 mA, 5 mA and 10 mA, has been developed. This new current source enables the Planck constant to be determined at different mass values up to 1 kg. Special efforts were made to select high quality components and to minimize electro-magnetic coupling in order to achieve a low noise and low drift current source. The new source is fully operational and has been integrated into the watt balance experiment. Extensive measurements were carried out to evaluate its performance under real working conditions. The relative standard deviation of the current was measured as less than 2×10^{-7} (integration time of 80 ms), which was mainly limited by voltmeter noise. The relative drift of the current was less than 1×10^{-9} min^{-1}.

A commercial vacuum compatible weighing cell of a capacity of 12 kg with a resolution of 10 µg has been procured and fully characterized. Characterization was performed in air with the weighing cell mounted on a small concrete block which was vibration-isolated from the floor. The conversion function between the mass and the raw voltage output and the temperature coefficient have been determined. The sensitivity to tilts was evaluated and coefficients of 0.8 µg µrad^{-1} and 7.0 µg µrad^{-1} were obtained on the longitudinal and transverse axes, respectively. The dynamic response of the weighing cell has also been estimated by means of a piezoelectric actuator placed on the loading pan of the balance. Results showed that the weighing cell would be able to follow changes in the electro-magnetic force on the coil during its travel through a non-uniform magnetic field with relative variations in the order of 10^{-4} (as expected for the new magnet), with a velocity larger than 80 mm s^{-1}, which is significantly larger than the actual velocity of 0.2 mm s^{-1}. The weighing cell was successfully tested in vacuum.

The current experimental apparatus is being transferred to inside a vacuum chamber located in a new laboratory with better thermal and vibrational isolation than the previous location. The transfer was delayed by several months due to a leaking element at the bottom of the vacuum chamber. The vacuum chamber is composed of four mobile parts which had to be completely dismantled for the repair. After the repair, the air-tightness of the chamber was tested and confirmed by evacuating it to a vacuum level of 10^{-2} Pa, using a pumping system composed of an oil-free roots pump and a turbo-molecular pump. The chamber is installed on a concrete block of 64 t. A polished granite block provides a plane surface to an air-cushion system which allows the magnetic circuit to be slightly lifted, allowing it to be centred and tilted during the alignment procedure. Vibration investigations were carried out on the concrete block and on the granite block as well as inside the vacuum chamber after its installation on the granite block. All measurements gave a similar vertical root-mean-square velocity due to vibration of about 0.8 µm s^{-1}, significantly less than those recorded in its previous location. In the horizontal and vertical directions, vibration peaks were observed close to 0.2 Hz, 30 Hz and 48 Hz. The 0.2 Hz peak is likely to be due to the ground floor motion generated by ocean waves, which is difficult to suppress. The higher frequency vibrations are expected to be generated by air-conditioning and other equipment and transmitted by the floor. This was confirmed by measurements made during a short-duration electricity outage in the building. The origin of the vibration will be investigated and if possible eliminated.

Relative gravity measurements were carried out in the watt balance room after the installation of the vacuum chamber with a CG5 relative gravimeter. The first gravity test measurements were made to verify that they complied with the 2008 gravity map of the whole laboratory. The gravity gradient was measured inside the vacuum chamber at the expected position of the test mass and was compared to the same gravity gradient measured in 2008 in the absence of the vacuum chamber. The difference of gravity between the test mass location inside the vacuum chamber and one of our gravimetric reference points far from the chamber was also measured. A difference of about 5 µGal was found with respect to the to the 2008 result, when the vacuum chamber was absent. A difference of about 1 µGal was observed between the vacuum chamber opened and closed at the test mass location.

The fabrication of the final magnetic circuit by a precision machining company is well advanced. The first parts are complete and the remainder are expected to be ready towards the end of 2012. On delivery of the parts the system will be assembled, initially without the magnets to verify the critical dimensions, in particular the parallelism of the pole faces. The final assembly – with the SmCo magnets in place and generating large magnetic forces – will be carried out at the BIPM, using an assembly device which is nearly complete.

The bias current electronics for the Josephson voltage standard, which will be used for the measurement of the induced voltage, has been completed and successfully tested. Problems were encountered with the SNS Josephson array which had been re-bonded at the NIST in April 2012. After its return it was found that the radiofrequency distribution did not work properly and that some sub-arrays were not functioning. These are considered to be the consequence of the large number of thermal cycles the array had undergone. A new array is being selected by a BIPM staff member who is currently working as guest researcher at the NIST. The fabrication of a second, simpler, Josephson voltage standard for the current measurement has started.

During 2012, the BIPM investigated the differences in the behaviour of a superconducting and a normal conducting coil when subjected to the same external magnetic field. The scope of this comparison was to try to verify experimentally if the principle of a watt balance, which is based on classical physics, could hold at the 10^{-8} level if the watt balance coil was superconducting. The problem of superposition of the resistive voltage and the induced voltage, discussed above, would be non-existent for a superconducting coil.

A bifilar coil was made with one NbTi wire (critical temperature about 9.3 K) and one Cu wire juxtaposed to the NbTi wire. This bifilar coil (~500 turns each winding, 230 µm wire diameter and ~17 cm coil diameter) was used in a configuration which closely resembles the typical moving phase of a watt balance. The bifilar coil was placed inside a cryostat. An external magnetic field, approximately radial and of value 3 mT at the position of the bifilar coil, was established by two field coils outside the cryostat.

In a typical moving phase of a watt balance, the relative motion between the balance coil and the magnetic field source induces an electromotive force in the balance coil which is proportional to the velocity of the relative motion, the strength of the magnetic induction and the wire length of the coil. In the experiment, the relative motion was achieved by displacing the external field coils vertically. For both the NbTi and the Cu wires, the velocity of the external coils is the same and the wire lengths are nearly identical. Therefore, *a priori*, if there is a difference in their induced voltages, it is expected to be the signature of a difference in the magnetic field "as seen" by each of the wires and which may be a demonstration of the quantum behaviour of the NbTi coil.

The experiment was carried out with the bifilar coils at 4 K (cryostat filled with liquid helium), at 10 K (just after evaporation of the liquid helium), at 77 K (cryostat filled with liquid nitrogen), and at 300 K (cryostat empty). Under these four conditions, the ratio of the voltages induced in the two wires of the bifilar coil was measured. The results showed evidence of a significantly different (about 1 %) voltage ratio between a superconducting NbTi wire and when the NbTi wire was conducting normally (the Cu wire acted as a normal conductor in all cases). This type of behaviour is thought to be a consequence of the Meissner effect experienced by the NbTi wire below the transition temperature.

To corroborate this hypothesis, a second bifilar coil was built. This second coil was composed of two NbTi juxtaposed wires and had the same dimensions as the NbTi–Cu coil. The two bifilar coils were placed together inside the cryostat with a vertical separation of 12 mm. The experiment was repeated and the four induced voltages were compared. Again, a significant difference in the voltage profiles was observed when comparing measurements above and below the critical temperature. By modelling all the experimental results together a preliminary conclusion was reached which assumes the following: superconducting coils experience a Meissner effect which is manifested by the expulsion of the external magnetic field. This is achieved through the generation of surface currents in the superconducting wire. Considering that a non-uniform external magnetic field is in motion with respect to the superconducting wires, the amplitude of the surface currents must change over time. The change of the related magnetic field induces an additional voltage in the adjacent wires (superconducting or not). This extra voltage is dependent on the particular configuration and on the distance between the source wire and the receptive wire.

As a preliminary conclusion, we consider that a superconducting wire under a changing magnetic field will induce an extra voltage in the neighbouring wires due to the Meissner effect. The question still pending is how symmetric this effect will be among the different turns of a single superconducting coil placed under the same conditions. Answering this question is crucial to assess the performance of a superconducting watt balance at the 10^{-8} level. These investigations are also of interest with respect to the use of superconducting solenoids to generate the magnetic field, in other existing or planned experiments. Future investigations of this phenomenon are planned.

4.2 Publications

1. Stock M., Watt balance experiments for the determination of the Planck constant and the redefinition of the kilogram, *Metrologia*, 2013, **50**, R1-R16.

2. Fang H. *et al*., Status of the BIPM watt balance, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM),* 2012, 424-425.

3. de Mirandés E. *et al*., Superconducting coil system to study the behavior of superconducting coils for a cryogenic watt balance, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM), 2012,* 470-471.

4. Solve S. *et al*., A bias source for the voltage reference of the BIPM watt balance, *Proc. 2012 Conference on Precision Electromagnetic Measurements (CPEM),* 2012, 52-53.

4.3 Travel

H. Fang, E. de Mirandés, S. Solve and M. Stock to:

- CPEM 2012 and satellite meetings, Boulder (USA), 30 June to 6 July 2012:
  - M. Stock attended the CPEM, meetings of CCEM working groups: WGLF (low-frequency quantities), GT-RF (radiofrequency quantities), WGRMO (RMO coordination) and WGKG (electrical methods to monitor the stability of the kilogram) and the CPEM Executive Committee meeting;
  - H. Fang gave two presentations on the BIPM watt balance at the WGKG meeting and at the CPEM;
  - E. de Mirandés attended the WGKG meeting and gave two presentations at the CPEM, a survey of measurements of the Planck constant (invited) and a talk on the superconducting moving coil experiment;
  - S. Solve presented two posters on the bias current source for the Josephson voltage standard and on a low thermal EMF switching system;
  - E. de Mirandés and M. Stock visited the NIST on 6 July 2012.

M. Stock to:

- International School of Physics "Enrico Fermi", Varenna (Italy), 25 to 26 July 2012 to give a lecture on "Watt balance experiments for the determination of the Planck constant and the redefinition of the kilogram".

4.4 Visitors

- C. Williams (NIST), 24 January 2012;
- S. Lee (KRISS), 5 March 2012;
- Film crew from France 2, 9 May 2012;
- B. Knapp (Professional Instruments Company) and E. Marsh (Pennsylvania State University), to discuss the fabrication of the magnet, 17 June 2012;
- L. Pitre (LNE), 23 July 2012;
- M. Gläser (retired from the PTB), 30 July 2012;
- T. Whipple (The Times), 2 August 2012;
- E. Massa (INRIM), 28 September 2012;
- M. Schreiber (Sartorius Company), 2 October 2012;
- S. Tu (CMS), 12 November 2012;
- Speakers and steering committee members of the Workshop on Metrology for Dynamic Measurements (10 persons), 14 November 2012;
- J. Cáceres (LATU), 20 November 2012;
- Attendees of the CCM workshop on the *mise en pratique* of the new definition of the kilogram (20 persons), 22 November 2012;
- J.H. Ullrich (President of the PTB), 4 December 2012.

4.5 Guest workers

- J. Lan, PhD student from Tsinghua University and NIM, China, 2 January to 27 June 2012;
- A. Zeggagh, student from Ecole des Mines, Douai (France), 16 May 2011 to 28 September 2012.

BIPM Ionizing Radiation Department

Director: J.M. Los Arcos (from 1 July 2012)

(1 January 2012 to 31 December 2012)

5.1 Staff Changes

P.J. Allisy-Roberts, former Director of the Ionizing Radiation Department retired on 31 May 2012; D.T. Burns acted as *ad interim* Director until J.M. Los Arcos took over on 1 July 2012.

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

5.2.1 Dosimetry standards and equipment

Work on the development of cavity ionization chambers continues. Chambers were constructed for the new Theratron ^{60}Co beam, including the development of a more robust waterproof sleeve arrangement, and for the transportable calorimeter system. Each of these chambers is mechanically measured using the 3D coordinate-measuring machine and characterized in the reference ^{60}Co beam, thus building up confidence in the determination of air volumes. A thin-walled chamber with a waterproof exterior was developed for the medium-energy absorbed-dose project and remains to be tested. The supply of a primary standard to the ININ was completed in 2012 with measurements in the BIPM reference beam. A secondee from the ININ was trained at the BIPM in the use of the Monte Carlo code PENELOPE to evaluate correction factors for this standard.

During 2012, the BIPM carried out two calorimeter comparisons in accelerator beams. The fourth comparison in the series BIPM.RI(I)-K6 was carried out at the LNE-LNHB (France) from 5 to 23 March 2012 in the 6 MV, 12 MV and 20 MV beams of their Saturne 43 accelerator. Preliminary results have been evaluated and a Draft A report prepared. The fifth K6 comparison was carried out at the ARPANSA (Australia) from 20 September to 9 October 2012 in the 6 MV, 10 MV and 18 MV beams of their Elekta Synergy accelerator. The BIPM equipment was shipped in advance, with the exception of the calorimeter core and ionization chambers which were hand carried. Preliminary results have been evaluated and a Draft A report is in progress. These comparisons serve to validate the NMI dose reference used in the calibration and audit services offered to hospitals in each country. The Draft B report for the comparison previously carried out at the NIST (USA) is close to publication.

The calorimeter is used at regular intervals in the BIPM reference ^{60}Co beam; the statistical standard uncertainty in the determination of the reference absorbed-dose rate from these accumulated measurements is around 4 parts in 10^4. The calorimetric absorbed-dose determination is around 1 part in 10^3 higher than the reference ionometric determination, which is well within the combined standard uncertainty.

As well as the additional transfer chambers noted above, a second calorimeter core and jacket were fabricated, to serve both as independent verifications and as replacements in the event of failure. The second core was tested in a 12 MV accelerator beam and was shown to agree with the first core at the level of the statistical standard uncertainty of the measurements of 1.7 parts in 10^3. However, a mechanical failure was identified on the second core and jacket ensemble on return from the comparison at the ARPANSA. The unusual calorimeter response noted in the BIPM ^{60}Co beam towards the end of 2011 was revealed to be a one-off event; the origin of this behaviour could not be established.

Monte Carlo calculations for the absorbed-dose conversion from graphite to water have continued, with calculations being made for the comparison with the LNE-LNHB (France) using photon spectra supplied by the LNE-LNHB. The results using these spectra show a curious behaviour that is thought to originate from the use of variance reduction at the LNE-LNHB. However, the final results are consistent with those of the other NMI participants, which demonstrates the robustness of the BIPM dose-conversion procedure. Measurements and calculations were made of the depth-dose distribution in water (in the BIPM ^{60}Co reference beam) for a fixed detector distance. These complement corresponding work carried out in graphite in 2011. The level of agreement between the measurements and calculations was used to derive a standard uncertainty for the ratio of photon attenuation coefficients in graphite and water of less than 1 part in 10^3. This completes the evaluation of the dose-conversion uncertainties, which will now be published.

Following the installation of the new Theratron irradiator in November 2011, the ^{60}Co source was finally installed in October 2012. The delay resulted from problems related to authorizations. Characterization of the new reference beam continues.

The project to develop an absorbed-dose standard for medium-energy x-rays has progressed and will continue into the Programme of Work 2013 to 2015. By simulating the x-ray tube and filter arrangement, phase-space spectra were generated for the four radiation qualities; the energy spectra observed were found to be in close agreement with those obtained using the Spekcalc software. The BIPM method for measuring the mean air-attenuation coefficient, μ_a, for each quality, using an evacuated tube, was simulated, allowing the photon interaction cross-section data for air used by PENELOPE to be tested. Using a 3 mm slab of graphite, and simulating this arrangement, a similar test was made of the cross-section data for graphite (measurements were also made using a water 'slab' for use later). Interestingly, while the calculated μ-values for air and graphite are each lower than the corresponding measured values by around 3 parts in 10^2, the calculated and measured ratios $\mu_{a,c}$ agree within the statistical standard uncertainty of 1 part in 10^2, except at 100 kV where a difference of 4 parts in 10^2 is observed. While this is a positive result, there is at present no explanation for differences exceeding 5 parts in 10^2 between the preliminary air-kerma rates determined using a thin-walled graphite cavity chamber and the reference values determined using the free-air chamber. Similar differences are observed using a chamber with air-equivalent walls.

The work on evaluating best estimates for the W_a-value for air and the I_c-value for graphite was completed and published in *Metrologia*. The conclusion is to change the recommended value for I_c from 78 eV to 81 eV (with standard uncertainty 2 eV) with no evidence for a need to change the present recommendation for W_a of 33.97 eV. However, the uncertainty of W_a should be increased from 1.5 parts in 10^3 to 3.2 parts in 10^3, which will result in a significant increase in the air-kerma determination using free-air chambers. The recommended value for the product $W_a\, s_{c,a}$ entering in the air-kerma determination for ^{60}Co using graphite-walled cavity standards is 33.72 eV with a standard uncertainty of 8 parts in 10^4. This work will be incorporated into the International Commission on Radiation Units and Measurements (ICRU) Report on Key Data.

The ICRU Report Committee on Key Data must also make a recommendation on the I-value for water. This is a complex data set including measurements made over the past 40 years and values derived from theory, with uncertainty estimates which are sometimes unreliable or non-existent. An analysis was made and presented to the Report Committee, the recommendation being the value I_w = 78 eV with standard uncertainty 2 eV. The aim is to finalize the draft report during 2013.

Primary measurements and reference chamber calibrations have continued in all of the reference x- and γ-ray beams. Comparisons and calibrations are underpinned by a significant effort in equipment calibration and maintenance, as required by the BIPM Quality Management System, and which were successfully audited in April 2012. Procedures, technical instructions, forms and laboratory records were subsequently modified in response to the audit (and to the change in Director of Department).

5.2.2 Dosimetry comparisons

Two comparisons in air kerma in gamma radiation were carried out, one in the ^{60}Co beam and the other in the ^{137}Cs beam, both with the NMIJ (Japan). Four air-kerma comparisons were carried out in x-ray beams, with the VSL (Netherlands) at low energies and with the VSL, the IAEA and the VNIIM (Russia) in the mammography beams. Two high-energy absorbed-dose comparisons were carried out in the accelerator beams of the LNE-LNHB (France) and the ARPANSA (Australia), as described earlier.

Seven comparison reports were published in the *Metrologia Technical Supplement*, two reports for the ARPANSA (Australia) and one each for the GUM (Poland), the MKEH (Hungary), the NIST (USA), the NPL (UK) and the VNIIM (Russia).

The report "*A blind test of the alanine dosimetry secondary standard of the PTB conducted by the BIPM*" of the irradiations made in the BIPM reference ^{60}Co beam at the end of 2011 has been submitted to *Metrologia*. This comparison was made at the request of the PTB to validate changes made to its alanine system.

5.2.3 Characterizations of national standards for dosimetry

Twenty-four characterizations of national standards were carried out; four in low-energy x-rays for the NIS (Egypt), ININ (Mexico) and the CMI (Czech Rep), three in mammography x-rays for the ININ and the CMI, three in medium-energy x-rays for the NIS, the ININ and the CMI, eleven in ^{60}Co for the NIS, the METAS (Switzerland), the CMI, the BIM (Bulgaria) and the KRISS (Republic of Korea) and three in ^{137}Cs for the NIS, the CMI and the BIM.

The IAEA/WHO dosimetry assurance programme continues to be supported by biannual reference irradiations, which in 2012 involved one series of irradiations for the radiotherapy level in the ^{60}Co beam and one for the radiation-protection level in the ^{137}Cs beam.

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

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

5.3.1.1 SIR submissions in 2012

During 2012, the BIPM received nine ampoules filled with six different radionuclides from six laboratories (i.e. one ampoule each containing ^{59}Fe (PTB), ^{60}Co (BARC and NRC), ^{109}Cd (LNE-LNHB), ^{131}I (LNE-LNHB and NIST), ^{133}Ba (BEV and LNE-LNHB) and ^{222}Rn (LNE-LNHB). All the submissions had been made to generate equivalence values in the associated ongoing BIPM key comparisons BIPM.RI(II)-K1.

Two radionuclides with short half lives, a gas, ^{222}Rn ($T_{1/2}$ = 3.8235 d, u = 0.0003 d) to supersede a previous entry which was questionable, and ^{131}I ($T_{1/2}$ = 8.021 d, u = 0.001 d) were measured in 2012.

Reporting forms for four previous submissions which were pending were received in 2012; in consequence the corresponding SIR results have been evaluated; this concerns ^{60}Co, ^{152}Eu and ^{241}Am from the CNEA (Argentina) and ^{64}Cu from the ENEA (Italy).

5.3.1.2 SIR Reports and quality assurance

Updated reports of three comparisons were published in the *Metrologia Technical Supplements* covering ^{57}Co and ^{222}Rn. Each result prior to 2008 has now been published in the *Metrologia Technical Supplements* except for two which are now in preparation. There are three outstanding results from 2008 pending publication, one of which is actually in circulation. To date, all the Draft A reports have been submitted except for 3 results that are still to be received from the NMIs concerned.

There are 47 results awaiting publication in the KCDB and every effort will be made to ensure that reports are published as quickly as possible, particularly when NMIs make submissions that are to replace out-dated results already removed from the KCDB.

All the SIR measurements are covered by the BIPM Quality Management System and a successful external audit, including for the first time the extension to short-lived radionuclides, was carried out on 21 September 2012 by Prof Dr F.O. Bochud from the IRA-METAS (Switzerland).

5.3.2 Gamma spectrometry

Routine measurements of potential impurities in SIR ampoules are made using the Ge(Li) and HPGe spectrometers. No impurity was detected in the ^{131}I and ^{109}Cd solutions submitted to the SIR by the LNE-LNHB and the NIST. Measurements of several swabs for BIPM sealed source leakage tests were carried out and a certificate issued.

No time was available to analyze the calibration measurements of the high-purity germanium spectrometer (HPGe).

5.3.3 Extension of the SIR to short-lived radionuclides

The BIPM.RI(II)-K4.Tc-99m key comparison using the SIR Transfer Instrument (SIRTI) is now running at a rate of two comparisons per year: the NIM (China) and the CNEA (Argentina) participated in 2012 and the publication of the results is in preparation. Although the measurements suffered some technical problems at the NMIJ in 2011, the result of this third ^{99m}Tc comparison, show agreement with the KCRV within one standard uncertainty and has been published. The LNMRI (Brazil), the IFIN-HH (Romania), the VNIIM (Russian Federation) and the NMISA (South Africa) are the next planned participants. A copy of the SIRTI electronics was updated for running in connection with a new laptop with Windows 7. A backup copy of the SIRTI detector has been purchased and is being characterized before calibration against the SIR.

The extension of the SIRTI for measuring ^{18}F is in development. Test measurements are in progress before calibration against the SIR. The NIST already volunteered to participate together with the ENEA which will participate immediately in the ^{99m}Tc and ^{18}F comparisons.

5.3.4 Extension of the SIR to pure beta emitters

Within the framework of the comparison initiated by the Working Group for the extension of the SIR to the measurement of pure beta emitters, seven laboratories (ENEA (Italy), IRMM (EU), LNE-LNHB (France), NIST (USA), NMISA (South Africa), PTB (Germany) and POLATOM RC (Poland)) have sent an ampoule filled with a solution of ^{63}Ni previously standardized with their own appropriate methods as already reported in the BIPM Director's Report 2011. The radionuclide ^{63}Ni decays with a 100 % probability by an allowed transition of energy 65.87 keV, u = 0.20 keV, to the ground state of ^{63}Cu.

The BIPM received a further ampoule of this radionuclide prepared by the NPL (United Kingdom) during 2012. Three sets of five samples were prepared from the ^{63}Ni solution with the three commercial scintillators, Ultima Gold, Hionic Fluor and Bio Fluor + already used in the first part of this exercise. In parallel three sets of ten quenched standards were prepared by addition of increasing number of drops of nitromethane from the same solution of ^{3}H provided by the LNE-LNHB and used in 2011.

All these samples were measured with the BIPM Beckman LS spectrometer, for which the method based on the use of universal efficiency curves (UEC) were used, and the BIPM TDCR spectrometer.

The analyses of the parallel TDCR measurements are ongoing.

5.3.5 CCRI activity comparison of ^{99}Tc

The CCRI(II)-K2.Tc-99 activity comparison was organized and piloted by the NPL in 2012. The deadline for the results obtained by the laboratories was the end of July 2012; this date was set after a first postponement of the original deadline. A further extension to the end of February 2013 has been accepted by the participants. The BIPM participated in the activity comparison of this long-lived ($T_{1/2}$ = 211.5 ×10^3 a, u = 1.1 × 10^3 a) almost pure beta emitter. Only the second order transition to the ground state with an end-point energy of 293.6 keV, u = 1.8 keV was considered in this study, since the emission probability of the other existing (second forbidden) beta transition is 1000 times smaller. Further, the gamma emission with an emission probability amounting to 1.6 × 10^{-3} was also neglected. At the same time three sets of quenched standards of ^{3}H from a solution of tritiated water provided by the LNE-LNHB have been prepared, using nitromethane as a quenching agent, to enable the use of the CIEMAT/NIST method. The measurements were carried out with the commercial liquid-scintillation Beckman spectrometer and the BIPM developed TDCR instrument. Three commercial scintillators were used to prepare the samples (Ultima Gold, Hionic Fluor and Bio-Fluor+) by the pycnometer technique. The deposited masses of radioactive solution in the form of 0.1 M ammonium hydroxide ranged from 60 mg to 84 mg. Results obtained for the three scintillators were in good agreement but only the value obtained at reference date with Ultima Gold A = 56.09 kBq/g, u = 0.20 was kept as the BIPM result. The BIPM will take advantage of the postponement of the deadline to evaluate the data obtained with the TDCR spectrometer.

5.3.6 Other CCRI activity comparisons with BIPM involvement

The final report of the CCRI(II)-K2.Pu-241 comparison piloted by the NPL at the end of 2009 has been published. The comparison results were communicated by the participants using the Excel-based reporting forms produced by the BIPM and the corresponding BIPM software, KCsoft, helped to speed up the publication process.

5.4 **Thermometry** (S. Picard, M. Nonis)

The Ionizing Radiation Department provides internal calibration services for thermometry at the BIPM, under the BIPM Quality Management System. An external audit was carried out in May 2012 and the recommendation was made that the BIPM should participate in key comparisons of fixed points or standard platinum resistance thermometers (SPRTs). In June 2012, the BIPM was invited by the Consultative Committee for Thermometry (CCT) to take part in the CCT-K9 comparison which was already under way and piloted by the NIST (USA). For this purpose, comparison measurements were carried out at the BIPM during September 2012 and two BIPM SPRTs were hand-carried to the NIST at the end of September for subsequent measurement.

In 2012, 18 SPRTs and nine commercial laboratory thermometers belonging to the Chemistry, Electricity, Mass, Time and Ionizing Radiation Departments were calibrated.

5.5 Publications

External publications

1. Burns D.T., An analysis of existing data for W_{air}, I_{c} and the product $W_{\mathrm{air}}\, s_{\mathrm{c,air}}$, *Metrologia*, 2012, **49**, 507-512.
2. Andreo P., Burns D.T., Salvat F., On the uncertainties of photon mass energy-absorption coefficients and their ratios for radiation dosimetry, *Physics in Medicine and Biology*, 2012, **57**, 2117–2136.
3. Burns D.T., Kessler C., Roger P., Csete I., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the MKEH, Hungary and the BIPM in low-energy x-rays, *Metrologia*, 2012, **49** *Tech. Suppl*., 06010.
4. Burns D.T., Lye J.E., Roger P., Butler D.J., Key comparison BIPM.RI(I)-K3 of the air-kerma standards of the ARPANSA, Australia and the BIPM in medium-energy x-rays, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06007.
5. Burns D.T., Kessler C., O'Brien M., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the NIST, USA and the BIPM in low-energy x-rays, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06006.
6. Burns D.T., Roger P., Villevalde A.Y., Oborin A.V., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the VNIIM, Russian Federation and the BIPM in low-energy x-rays, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06003.
7. Burns D.T., Roger P., Knyziak A.B., Key comparison BIPM.RI(I)-K2 of the air-kerma standards of the GUM, Poland and the BIPM in low-energy x-rays, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06002.
8. Kessler C., Burns D.T., Allisy P.J., Butler D., Lye J., Webb D., Comparison of the standards for absorbed dose to water of the ARPANSA and the BIPM for ^{60}Co gamma-radiation, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06009.
9. Kessler C., Allisy P.J., Burns D.T., Duane S., Manning J., Comparison of the standards for absorbed dose to water of the NPL, United Kingdom and the BIPM for ^{60}Co gamma-rays, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06008.
10. Bé M.-M., Cassette P., Lépy M.C., Amiot M.-M, Kossert K., Nähle O.J., Ott O., Wanke C., Dryák P., Ratel G., Sahagia M., Luca A., Antohe A., Johannsson L., Keightley J., Pearce A., Standardization, decay data measurements and evaluation of ^{64}Cu, *Appl. Radiat. Isot*., 2012, **70**(9), 1894-1899.
11. Michotte C., Ratel G., Cassette P., Update of the BIPM.RI(II)-K1.Rn-222 comparison of activity measurements for the radionuclide ^{222}Rn to include the LNE-LNHB, France, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06001.
12. Michotte C., Ratel G., Courte S., Fitzgerald R., Sahagia M., Activity measurements of the radionuclide ^{57}Co for the NIST, USA and the IFIN-HH, Romania in the ongoing comparison BIPM.RI(II)-K1.Co-57, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06005.

13. Michotte C., Johansson L., CCRI(II) activity comparison of ^{241}Pu: CCRI(II)-K2.Pu-241, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06012.

14. Michotte C., Sato Y., Unno Y., Yunoki A., Activity measurements of the radionuclide ^{99m}Tc for the NMIJ, Japan, in the ongoing comparison BIPM.RI(II)-K4.Tc-99m, *Metrologia*, 2012, **49**, *Tech. Suppl*., 06013.

BIPM publications

15. Michotte C., 2012, Verification of the linearity of the new SIR using sources of ^{64}Cu and ^{99m}Tc *Rapport BIPM*-2012/02, 12 pp.

16. Roger P., 2012, High-voltage measurement for the BIPM x-ray generators *Rapport BIPM*-2012/04, 8 pp.

5.6 Activities related to the work of Consultative Committees

P.J. Allisy-Roberts was Executive Secretary of the CCRI until she retired on 31 May 2012. D.T. Burns acted as *ad interim* Executive Secretary until J.M. Los Arcos took over as the new Executive Secretary on 1 July 2012. There was one CCRI meeting in May and six Working Group meetings during 2012.

J.M. Los Arcos is the coordinator of the CCRI(II) Working Group on the Extension of the SIR to beta-emitters using liquid scintillation (ESWG(II)), which met in May 2012.

C. Michotte is the coordinator of the CCRI(II) Transfer Instrument Working Group (TIWG(II)), which met in May 2012, and a member of the Key Comparisons Working Group (KCWG(II)) which met in May and December 2012.

G. Ratel is a member of the CCRI(II) Working Group on the Extension of the SIR to beta-emitters using liquid scintillation (ESWG(II)), which met on 9 May 2012 and of which he was the *rapporteur*, the KCWG(II) which met on 10 May and 3-4 December 2012, the Transfer Instrument Working Group (TIWG(II)) which met on 10 May 2012 and the Working Group on Realization of the becquerel (BqWG(II)) which met on 9 May 2012.

D.T. Burns is a member of the CCRI(I) Key Comparisons Working Group (KCWG(I)), Accelerator Dosimetry Working Group (ADWG(I)) and Brachytherapy Standards Working Group (BSWG(I)) and the *ad hoc* group evaluating the effect of excess charge on the value for W_{air}. Since 2009 he has been *rapporteur* at annual meetings of the CCRI.

S. Picard is Executive Secretary of the Consultative Committee for Acoustics, Ultrasound and Vibration (CCAUV), which held its 8th meeting on 13 to 14 June 2012. She is a member of the CCAUV Working Group for RMO Coordination (CCAUV-RMOWG), the CCAUV Working Group on Strategic Planning (CCAUV-SPWG) and the newly constituted CCAUV Working Group for Key Comparisons (CCAUV-KCWG) which met on 11 to 12 June 2012. She was appointed Interim Acting Executive Secretary of the Consultative Committee for Thermometry (CCT) in August 2012.

5.7 Activities related to external organizations

D.T. Burns is a Fellow of the Institute of Physics (FInstP) in the UK and in 2012 was elected as a Commissioner of the ICRU. He is a member of the ICRU Committee on Fundamental Quantities and Units and of two ICRU Report Committees (on Key Data for Dosimetry and on Operational Quantities for Radiation Protection). He is also Commission Sponsor for two reports (Key Data for Dosimetry and

Small and Non-Standard Fields) and is the BIPM contact person for the EURAMET-TC for ionizing radiation (replaced for the 2012 meeting by S. Picard).

C. Michotte is the contact person at the BIPM and *rapporteur* for the JCGM/WG1 meetings in June and November 2012.

G. Ratel is the BIPM representative on the International Committee for Radionuclide Metrology (ICRM), is President of the ICRM Nominating Committee, and a member of the Scientific Committee for the 19th International Conference on Radionuclide Metrology and its Applications (ICRM 2013) which will be held in Antwerp (Belgium) on 17 to 21 June 2013.

P.J. Allisy-Roberts has been the BIPM representative on the IAEA SSDL Scientific Committee which she currently chairs. She is a member of the Working Group for the UK NMS programme for ionizing radiation and acoustics and of the *Comité scientifique rayonnements ionisants* (LNE, France). She is a member of the editorial board of the Journal of Radiological Protection and of the *Revue Française de Métrologie*. She was elected to the Board of the European Federation of Medical Physicists where she currently serves as the European Matters Committee Chairman.

5.8 Travel (conferences, lectures and presentations, visits)

D.T. Burns to:

- Saclay (France), 5 to 9 March 2012, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the LNE-LNHB;
- Saclay (France), 10 September 2012, to participate in the jury for the defence of the thesis by N. Perichon (LNE-LNHB) entitled "*Etablissement des références nationales en termes de dose absorbée, par calorimétrie dans l'eau, pour les faisceaux de rayons X de moyenne énergie, applicables en radiothérapie*";
- Yallambie (Australia), 20 to 28 September 2012, to participate in the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the ARPANSA and to give a joint talk with S. Picard entitled "The BIPM Graphite Calorimeter Standard for Absorbed Dose to Water".

S. Picard to:

- Saclay (France), 17 February 2012, to prepare the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the LNE-LNHB;
- Saclay (France), 5 to 23 March 2012, to carry out the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the LNE-LNHB;
- Beijing (China), 25 to 26 May 2012, to visit the dosimetry department of the NIM and to give a talk entitled "The BIPM Graphite Calorimeter Standard for Absorbed Dose to Water";
- Beijing (China), 26 to 31 May 2012, to participate in the 2012 World Congress on Medical Physics and Biomedical Engineering and to chair a session and give an oral presentation entitled "The BIPM Calorimetric Standard for Accelerator Dosimetry";
- Yallambie (Australia), 20 September to 9 October 2012, to carry out the BIPM.RI(I)-K6 comparison of absorbed dose to water in accelerator beams with the ARPANSA; she also gave a joint talk with D.T. Burns entitled "The BIPM Graphite Calorimeter Standard for Absorbed Dose to Water";
- Melbourne (Australia), 8 October 2012, to attend a seminar given by T. Knöös at the Peter MacCallum Cancer Centre entitled "Flattening Filter Free Linacs";

- Bucharest (Romania) 25 to 26 October 2012 to attend the EURAMET TC-IR meeting and to give a talk entitled "The BIPM Graphite Calorimeter Standard for Absorbed Dose to Water";
- Sèvres (BIPM) 15 to 16 November 2012 to attend the BIPM Workshop on Challenges in Metrology for Dynamic Measurements.

G. Ratel to:

- LNE-LNHB, Saclay (France) 11 September 2012, to attend the defence of the thesis of Florestan Ogheard for which he was the rapporteur;
- Roissy (France), 17 to 20 September 2012, to attend an APAVE course to obtain the aptitude certificate for the transport of dangerous goods;
- LNE, Paris (France) 8 and 9 October a.m. 2012, to attend the DDEP Workshop and to give a talk entitled "The BIPM and the International Reference System (SIR)";
- PTB, Braunschweig (Germany), 26 to 28 November 2012, to attend the ICRM LSCWG and to give a talk entitled "^{63}Ni pilot comparison";
- Issy-les-Moulineaux (France), 29 to 30 November 2012, to attend the "*les 8^èmes^ rencontres des personnes compétentes en radioprotection*";
- IRMM, Geel, 11 to 13 December 2012, ICRM 2013 Scientific Committee;
- IRMM, Geel, 14 December 2012, ICRM 2013 Executive Board.

C. Michotte to:

- Saclay (France), 15 March 2012, to give a seminar on the SIRTI and the ^{99m}Tc comparison;
- Saclay (France), 7 September 2012, as member of jury for a PhD thesis at the LNE-LNHB.

C. Michotte and M. Nonis to:

- Beijing (China), 22 to 29 March 2012, to carry out an activity comparison of ^{99m}Tc (BIPM.RI(II)-K4.Tc-99m) at the NIM using the SIR Transfer Instrument;
- Buenos Aires (Argentina), 8 to 16 November 2012, to carry out an activity comparison of ^{99m}Tc (BIPM.RI(II)-K4.Tc-99m) at the CNEA using the SIR Transfer Instrument.

Internal seminars

- The SIR transfer instrument, a tool for international comparisons of ^{99m}Tc activity measurements, C. Michotte, October 2012.

5.9 Visitors

- F.L. Prez and L. Joulaeizadeh (VSL), 23 January to 3 February 2012
- L. Joulaeizadeh (VSL), 8 February 2012
- G. Hassan (NIS), 7 to 18 May 2012
- N. Saito and T. Kurosawa (NMIJ/AIST), 6 June 2012
- L. Czap (IAEA), 18 to 22 June 2012
- M. Marobela (BOBS), 26 July 2012

- Delegation from the NMI of Colombia, 24 August 2012
- Delegation from the NIM (China), 16 to 17 October 2012
- D. Mohamad (Deputy Director General IAEA) and A. Fajgelj (IAEA) 12 November 2012
- Steering committee of the BIPM Workshop on Dynamic Measurements, 14 November 2012
- A. Villevalde (VNIIM), 23 to 30 November 2012
- J. Ullrich, (President of the PTB) 4 December 2012.

5.10 Secondees and guest workers

- C. Carmeli (University of Birmingham), 18 June to 18 July 2012
- J. Alvarez (ININ), 20 March to 26 June 2012.

BIPM Chemistry Department

Director: R.I. Wielgosz

(1 January 2012 to 31 December 2012)

6.1 **Gas metrology programme** (J. Viallon, E. Flores, P. Moussay, F. Idrees, R.I. Wielgosz)

6.1.1 Ozone photometer comparison and calibration programme

In 2012, seven laboratories brought or sent their ozone national standard to the BIPM for a comparison with the BIPM-SRP27 reference standard as part of the key comparison BIPM.QM-K1: the FMI (Finland) in February; the KRISS (Republic of Korea) and the UBA (Germany) in March; the LNE (France) in May; the EMPA (WMO, Switzerland) in June; the NMISA (South Africa) in July; and the ISCIII (Spain) in December. Five of the comparison reports have been reviewed and published in *Metrologia Technical Supplement*. In addition, a calibration of the ozone analyser of the NMISA was performed in July 2012.

6.1.2 Maintenance of the NO_2 facility and CCQM-K74, CCQM-P110 coordination

The CCQM-K74 and the pilot study P110 B1 final reports were published as Technical Supplements in *Metrologia*. The validation of the method and facility developed to run this comparison was the subject of a paper, "*Highly accurate nitrogen dioxide (NO_2) in nitrogen standards based on permeation*", which was submitted and published in the peer-review journal *Analytical Chemistry*. A second paper, which focuses on the uncertainty achievable with FTIR measurements with traceability to line strength data, is currently being finalized and will be submitted to a peer-reviewed journal. The report of the pilot study CCQM-P110-B2 has been updated to incorporate decisions taken during the 28th meeting of the Consultative Committee for Amount of Substance: Metrology in Chemistry (CCQM) Working Group on Gas Analysis (GAWG) held in St Petersburg, Russian Federation, on 23 to 24 October 2012. It is expected that this report will be ready for publication in January 2013.

The BIPM-NO_2 primary gas facility and procedures are currently being updated in preparation for new measurements for gas phase titration, future comparisons, and to underpin the BIPM's ozone standard facility.

6.1.3 Key comparison on methane standards

The protocol of CCQM-K82 (Methane in air at ambient level) was updated following the decisions taken during the 27th Meeting of the CCQM GAWG held at the BIPM headquarters on 17 to 18 April 2012 and redistributed to all participants in May 2012. The comparison is being coordinated by the BIPM, and requires all participating NMIs to send two standards to the BIPM, which will be analysed under repeatability conditions. A number of participants have been unable to meet the deadline for delivery of gas standards to the BIPM, and this has now been extended from November 2012 to January 2013.

The analytical uncertainties for CH_4/Air mole fraction measurements by the GC-FID with the BIPM measurement facility were significantly improved in 2012. Standard relative uncertainties ranging from 0.01 % to 0.04 % can now be achieved and have been described and validated in internal reports. A procedure on "Measurement steps for the value assignment of CH_4/Air cylinders by the BIPM-CH_4 facility using CRDS and GC-FID" has been written and reviewed. The facility is now fully validated and ready for use in CCQM-K82.

6.1.4 Formaldehyde

Further studies have been performed in preparation for the key comparison CCQM-K90, which is planned to start at the end of 2013 with the BIPM as pilot laboratory.

The first series of potential transfer standards were tested in 2011 and found to be insufficiently stable for use in a key comparison, and as a result new analyses were started on nine new cylinders of formaldehyde in nitrogen produced by a company and certified by the VSL in June 2012. Analysis of the stability of the formaldehyde mole fraction, performed over six months, showed promising results, and the analysis will continue for four more months in 2013. A purity analysis of the gas mixtures was performed by FTIR spectroscopy, and will be repeated at the end of the stability study.

FTIR analysis was performed on the first series of cylinders. It showed that the mixtures contain different impurities depending on the source of formaldehyde used in their production. Formaldehyde generated from paraformaldehyde is contaminated with water and mixtures made from trioxane are contaminated with unreacted trioxane. This was concluded after a trioxane in nitrogen source was setup and used to acquire FTIR spectra. No trioxane could be detected in the gas mixtures produced by the BIPM facility, in which water is considered to be the only impurity. A CRDS instrument to quantify the water mole fractions in the mixtures generated by the BIPM facility was acquired in September 2012 and is currently being tested.

6.1.5 Development of a laser-based SRP and ozone absorption cross-section measurements

A paper on "*Relative measurements of ozone absorption cross-sections at three wavelengths in the Hartley band using a well-defined UV laser beam*" was published in the *Journal of Geophysical Research* in early 2012. Activities then focused on developing a method to produce pure ozone with the generator that was assembled in 2011. This generator is based on the dissociation of pure molecular oxygen by applying high voltage discharges, leading to the formation of ozone by further recombination of atomic oxygen. The reaction cell, made entirely of quartz, is kept inside a temperature controlled cryostat to instantaneously trap the produced ozone in the liquid phase. The first sample of liquid ozone was obtained in May 2012, and the improvement of the production method was the subject of a study by K. Tworek, GUM (Poland), during a six month secondment at the BIPM. This resulted in the method being successfully applied to routinely generate ozone, trap it in the liquid phase and maintain the obtained sample at cryogenic temperatures. Two measurement methods described in this project were also tested. The first allowed the total pressure of the mixture of ozone and oxygen to be monitored while ozone slowly converted back to oxygen. The second involved trapping the ozone and pumping away any non-converted oxygen. The second method resulted in ozone mole fractions as high as 90 % to be obtained. Further improvements in the methodology are expected to result in even purer ozone.

In parallel, measurement of oxygen, ozone and impurities in the sample by mass spectrometry was also improved. The best conditions within the mass spectrometer to measure ozone were determined. The effect of heated surfaces inside the spectrometer on the dissociation of ozone was identified and solved. The expected fragmentation of the ozone molecule in molecular and atomic oxygen due to electron impact in the instrument was observed, and will be used to accurately quantify traces of molecular oxygen in the pure ozone sample.

6.2 **Organic analysis programme** (S. Westwood, R. Josephs, A. Daireaux, T. Choteau, N. Stoppacher, R.I. Wielgosz)

6.2.1 Purity methodology and small molecule purity analysis

A paper outlining the BIPM implementation of the mass balance method for determining the mass fraction of the main component of a high purity organic material has been drafted and submitted to *Analytical Chemistry*.

An overview of the CCQM Working Group on Organic Analysis (OAWG) purity comparison results and the preliminary approach for their use to link comparison results to CMC claims initially discussed in 2011 has been revised and an updated version of the proposal was discussed further at the OAWG meeting held in Hong Kong (China) on 6 to 9 November 2012.

A "White Paper" describing an objective approach to the use of this data to demonstrate a general capability for purity assignment of organic primary calibrators and for linking the results of key comparisons to the reporting and assessment of CMC claims is being prepared by the BIPM for distribution and discussion within the OAWG.

Feasibility studies for the implementation of a BIPM capability for quantitative nuclear magnetic resonance (qNMR) assignments of the purity of organic calibrators was undertaken in 2012.

These studies established:

- preparation and ampouling of gravimetrically assigned standard solutions of selected organic materials in deuterated NMR solvents;
- methods for the verification of gravimetrically assigned analyte concentration in solution by an independent quantitative chromatographic analysis;
- identification of a number of external NMR laboratories to obtain ^{1}H qNMR spectra for BIPM assigned-value analyte solutions;
- capability at the BIPM for the interpretation of qNMR data and its use in the assignment of purity.

6.2.2 Organic programme quality system

A Quality System has been implemented that will ensure all aspects of the technical work within the BIPM Organic Analysis work programme, related to key comparisons, complies with relevant BIPM technical and quality system requirements.

The corrective actions arising from external peer review of the Organic Programme Quality System in 2011 have been implemented. The key action was the installation of an online, remote temperature monitoring system for the equipment used for the low-temperature storage of BIPM comparison materials.

The Department is already preparing for the next BIPM Internal Audit of the Organic Programme Quality System which is scheduled for 2013.

6.2.3 Purity comparison CCQM-K55.a (Estradiol)

The Final Report for the CCQM-K55.a (Estradiol) comparison was completed and the results were approved for equivalence and accepted for publication by the OAWG members and by the CCQM Working Group Chairs. The report was published in the BIPM KCDB Appendix B in August 2012. A report for the parallel pilot study, CCQM-P117.a (Estradiol), has been finalized.

6.2.4 Purity comparison CCQM-K55.b (Aldrin)

The Draft B report for the comparison had been circulated for discussion by the CCQM OAWG in November 2011. The Final Report for the CCQM-K55.b (Aldrin) key comparison, which incorporated feedback obtained from review of the Draft B report, was completed and distributed to the OAWG members prior to the April 2012 CCQM OAWG meeting. After minor changes, it was accepted by the OAWG and the results were approved for equivalence and for publication in the BIPM KCDB following a final review by the CCQM Working

Group Chairs. The report was published in the BIPM KCDB Appendix B in October 2012. A draft report for the parallel pilot study, CCQM-P117.b (Valine) has been circulated for discussion.

6.2.5 Purity comparison CCQM-K55.c [(L)-Valine]

The CCQM-K55.c [(L)-Valine] key comparison and the parallel CCQM-P117.c pilot study was coordinated by the BIPM. The call for participation and a comparison protocol was circulated in March 2012. Twenty five institutes participated in the key comparison and ten institutes in the parallel pilot study. The comparison samples were distributed to participants without major incident in May 2012 and the results were received in October 2012. A preliminary result summary was distributed to participants in November 2012 and the comparison results were discussed at the CCQM OAWG meeting held in Hong Kong (China) on 6 to 9 November 2012. Generally, good agreement was observed between participants using a mass balance or titration approach to assign the valine content of the comparison material. A wider range of values for valine content were obtained using quantitative NMR (qNMR) methods. While the majority of reported qNMR results were consistent within their associated uncertainties with the assignments made by mass balance approaches, some qNMR estimates were significantly lower. Possible reasons for these discrepancies will be investigated prior to the CCQM OAWG meeting to be held at the BIPM headquarters in April 2013 and in parallel with the preparation of the Draft A report for the comparison.

6.2.6 Organic large molecule purity – angiotensin I

A range of analytical methods for the purity determination of the intact decapeptide angiotensin I (ANG I) was developed and validated by the BIPM. These activities have received additional support through a collaborative agreement with the NIST. ANG I is a hypertension marker that is frequently monitored and crucial for clinical chemistry, and provides a model system for developing future large organic molecule purity comparisons for the CCQM. Procedures developed and validated for the identification and quantification of structurally related peptide impurities for use in the characterization of the model peptide ANG I include:

- LC-hrMS/MS (Orbitrap);
- LC-MS/MS (Qtrap);
- LC-UV-CAD.

External calibration has been used for all three methods for the quantification of six commercially available related original peptide fragment standards.

Procedures developed for the identification and quantification and other non-related peptide impurities include:

- GC-MS method for determination of volatile organic impurities;
- Karl Fischer titration and elemental analysis methods for the determination of water content;
- Thermogravimetric analysis as a supporting method for the estimation of the total volatiles, water content and residues;
- Ion chromatography for the determination of peptide counter ions;
- NMR spectroscopy and elemental microanalysis to provide confirmatory data for the characterization of the ANG I (currently ongoing).

The purity mass fraction value of a pure ANG I material provided by the NIST and the corresponding measurement uncertainty has been evaluated using the mass balance approach.

In addition, a set of analytical methods for purity determination of amino acids (AAs) has been developed and validated. Pure AAs are used for peptide value assignment based on AA analysis. Identification and quantification of the major impurities present in a set of six selected stable amino acids of high importance for the characterization of ANG I was undertaken. The six selected pure AAs were isoleucine, leucine, phenylalanine, proline, tyrosine and valine, and the methods that were developed and validated for the determination of structurally related and other impurities were:

- LC-hrMS/MS (Orbitrap);
- LC-MS/MS (Qtrap);
- LC-UV-CAD.

All three methods employ reverse-phase liquid-chromatography analytical columns with embedded acidic ion-pairing groups for the identification and quantification of the selected AAs and related compounds.

Procedures developed for the determination of other non-structurally related impurities in the set of the six selected pure AA materials included:

- GC-MS methods for determination of volatile organic impurities;
- Karl Fischer titration and elemental analysis methods for the determination of the water content;
- Thermogravimetric analysis as a supporting method for the estimation of the total volatiles, water content and residues;
- NMR spectroscopy and elemental microanalysis to provide confirmatory data.

The purity mass fraction values of the six selected AAs and the corresponding measurement uncertainties have been evaluated using the mass balance approach.

The value assigned AAs are currently being used to develop and validate a quantitative AA analysis method for the mass fraction value assignment of ANG I subsequent to complete microwave-assisted vapour-phase hydrolysis. The LC-MS/MS (Qtrap) method for AAs has been further developed to serve as an LC-ID-MS/MS method for the AA analysis of the hydrolyzed peptides.

6.2.7 Organic large molecule purity – insulin

Insulin (INS) plays an important role in the treatment and monitoring of diabetes. Pure primary calibrator materials are a fundamental requirement for pharmaceuticals, laboratory medicine and clinical chemistry, and the molecule provides a model system for developing future large organic molecule purity comparisons for the CCQM. Method development studies for the characterization and mass balance purity value assignment of an intact INS material provided by the United States Pharmacopoeia (USP) are under way. Procedures developed or investigated to date for use in the characterization of model organic large molecule include:

- GC-MS methods for determination of volatile organic impurities;
- Karl Fischer titration and elemental analysis methods for the determination of the water content;
- Thermo gravimetric analysis as a supporting method for the estimation of the total volatiles, water content and residues;
- Ion chromatography for the determination of peptide counter ions;
- ICP-MS for the determination of Zn (INS complex anion).

In addition, different methods for the identification and quantification of structurally related impurities are currently in the development and validation stage, including:

- LC-hrMS/MS (Orbitrap);
- LC-MS/MS (Qtrap);

- LC-UV-CAD;
- NMR spectroscopy to provide confirmatory data for the characterization of the INS.

The same set of AAs that has been value assigned for ANG I will also be used to perform quantitative AA analysis for the mass fraction value assignment of INS subsequent to complete microwave-assisted vapour-phase hydrolysis. The LC-MS/MS (Qtrap) method for AAs has been further developed to serve as an LC-ID-MS/MS method for the AA analysis of hydrolyzed INS. The method optimization and validation is in progress.

6.3 Activities related to the JCTLM (S. Maniguet and R.I. Wielgosz)

R.I. Wielgosz is Executive Secretary of the Joint Committee for Traceability in Laboratory Medicine (JCTLM) and a member of its review team on Quality Systems and Implementation. S. Maniguet is coordinating the development of the JCTLM Database.

The annual joint meeting of the JCTLM Working Groups 1 and 2, and *Ad Hoc* Working Group 3 was held in conjunction with the American Association for Clinical Chemistry (AACC) meetings in Los Angeles, USA, in July 2012. This was followed by a workshop on 'JCTLM reference measurement systems for HbA1c', and a joint JCTLM and *In Vitro* Diagnostic (IVD) industry workshop on 'Meeting traceability requirements for the IVD industry'. The 11th meeting of the Executive Committee of the JCTLM was held at the BIPM headquarters on 6 to 7 December 2012.

The list of JCTLM review teams in Working Group 1 and 2 was updated to include the review team member appointed for the review of nominations for Drugs. The document for the terms of reference for each review team was revised for harmonization by the Quality Review team leader and finalized after review by WG1 review teams. The Executive Committee procedure for the selection and appointment of Working Group review team leaders and members was updated to include the term of membership which is five years renewable.

WG2 procedures have been updated to include a requirement for consistency of the expanded uncertainty ranges in the database in line with those stated in the reference measurement service providers' scope of accreditation.

The JCTLM Database was updated in January 2012 to remove temporary reference measurement services from laboratories undergoing the accreditation process after the accreditation deadline for compliance with ISO 15195 and ISO/IEC 17025 as Calibration laboratory. In March 2012, the WG1 Cycle 8 reference materials, and measurement methods, and WG2 Cycle 7 reference measurement laboratory services approved by the Executive Committee during its 10th annual meeting in December 2011 were published in the database.

As of December 2012 the JCTLM Database contained:

- 262 available certified reference materials covering 11 categories of analytes. Of these reference materials, 33 are in List II, which includes reference materials value-assigned using internationally agreed protocols, and three are in List III, which covers reference materials with nominal properties;
- 157 reference measurement methods or procedures that represent about 80 different analytes in eight categories of analytes;
- 74 reference measurement services, delivered by eight reference laboratories and one national metrology institute in five countries and which cover six categories of analytes.

The WG1 Cycle 9 call for nominations of higher order reference materials and reference measurement methods or procedures, and the WG2 Cycle 7 call for nominations of reference measurement laboratory services were announced on the JCTLM website in January 2012, and email notifications were sent to about 300 JCTLM potential contributors. As of July 2012, 42 nominations for materials, two nominations for methods, and six nominations for services had been received and sent to Review Teams for evaluation.

6.4 Publications

External publications

1. Viallon J., Moussay P., Idrees F., Wielgosz R., Zellweger C., Final report on the ongoing key comparison BIPM.QM-K1: Ozone at ambient level, comparison with EMPA (June 2012), *Metrologia,* 2012, **49***, Tech. Suppl.,* 08011.

2. Viallon J., Moussay P., Idrees F., Wielgosz R., Walden J., Kuronen P., Final report on the ongoing key comparison BIPM.QM-K1: Ozone at ambient level, comparison with FMI (February 2012), *Metrologia,* 2012, **49***, Tech. Suppl.,* 08013.

3. Viallon J., Moussay P., Idrees F., Wielgosz R., Stummer V., Schinz V., Final report on the ongoing key comparison BIPM.QM-K1: Ozone at ambient level, comparison with UBA (March 2012), *Metrologia,* 2012, **49***, Tech. Suppl.,* 08008.

4. Viallon J., Moussay P., Idrees F., Wielgosz R., Macé T., Couette J., Final report on the ongoing key comparison BIPM.QM-K1: Ozone at ambient level, comparison with LNE (May 2012), *Metrologia,* 2012, **49***, Tech. Suppl.,* 08012.

5. Viallon J., Moussay P., Idrees F., Wielgosz R., Lee S., Lee J.Y., Woo J.C., Kim B.M., Final report on the ongoing key comparison BIPM.QM-K1: Ozone at ambient level, comparison with KRISS (March 2012), *Metrologia,* 2012, **49***, Tech. Suppl.,* 08007.

6. Petersen M., Viallon J., Moussay P., Wielgosz R.I., Relative measurements of ozone absorption cross-sections at three wavelengths in the Hartley band using a well-defined UV laser beam, *J. Geophys. Res.,* 2012, **117***,* D05301.

7. Flores E., Idrees F., Moussay P., Viallon J., Wielgosz R., Highly accurate nitrogen dioxide (NO_2) in nitrogen standards based on permeation. *Anal. Chem*., 2012, **84** (23), 10283–10290.

8. Flores E., Idrees F., Moussay P., Viallon J., Wielgosz R., Fernandez T., Ramirez S., Rojo A., Shinji U., Walden J., Sega M., Sang-Hyub O., Mace T., Couret C., Qiao H., Smeulders D., Guenther F.R., Thorn III W.J., Tshilongo J., Ntsasa N.G., Stovcik V., Valkova M., Konopelko L., Gromova E., Nieuwenkamp G., Wessel R.M., Milton M., Harling A., Vargha G., Tuma D., Kohl A., Schulz G. International comparison CCQM-K74: Nitrogen dioxide, 10 µmol/mol. *Metrologia* 2012, **49**, *Tech. Suppl*., 08005.

9. Flores E., Idrees F., Moussay P., Viallon J., Wielgosz R., Fernández T., Rojo A., Ramírez S., Aoki N., Kato K., Jeongsoon L., Moon D., Kim J., Harling A., Milton M., Smeulders D., Guenther F.R., Gameson L., Botha A., Tshilongo J., Ntsasa N.G., Valková M., Konopelko L., Kustikov Y., Ballandovich V., Gromova E., Tuma D., Kohl A., Schulz G. Final report of the pilot study CCQM-P110-B1: A comparison of nitrogen dioxide (NO_2) in nitrogen standards at 10 µmol/mol by Fourier transform infrared spectroscopy (FT-IR). *Metrologia,* 2012, **49**, *Tech. Suppl*., 08006.

10. Westwood S., Josephs R., Daireaux A., Wielgosz R., Davies S., Wang H., Rodrigues J., Wollinger W., Windust A., Kang M., Fuhai S., Philipp R., Kuhlich P., Wong S., Shimizu Y., Pérez M., Avila M., Fernandes-Whaley M., Prevoo D., de Vos J., Visser R., Archer M., LeGoff T., Wood S., Bearden D., Bedner M., Boroujerdi A., Duewer D., Hancock D., Lang B., Porter B., Schantz M., Sieber J., White E., Wise S.A. Final report on key comparison CCQM-K55.a (Estradiol): An international comparison of mass fraction purity of estradiol, *Metrologia,* 2012, **49**, *Tech. Suppl*.,08009.

11. Westwood S., Josephs R., Choteau T., Daireaux A., Mesquida C., Wielgosz R., Rosso A., Ruiz de Arechavaleta M., Davies S., Wang H., Pires do Rego E.C., Marques Rodrigues J.,

de Freitas Guimarães E., Barreto Sousa M.V., Monteiro T.M., Alves das Neves Valente L., Marques Violante F.G., Ribeiro Almeida R.R., Baptista Quaresma M.C., Nogueira R., Windust A., Dai X., Li X., Zhang W, Li M., Shao M., Wei C., Wong S., Cabillic J., Gantois F., Philipp R., Pfeifer D., Hein S., Klyk-Seitz U.-A., Ishikawa K., Castro E., Gonzalez N., Krylov A., Lin T.T., Kooi L.T., Fernandes-Whaley M., Prevoo D., Archer M., Visser R., Nlhapo N., de Vos B., Ahn S., Pookrod P., Wiangnon K., Sudsiri N., Muaksang K., Cherdchu C., Gören A.C., Bilsel M., LeGoff T., Bearden D., Bedner M., Duewer D., Hancock D., Lang B., Lippa K., Schantz M., Sieber J. Final report on key comparison CCQM-K55.b (aldrin): An international comparison of mass fraction purity assignment of aldrin, *Metrologia* 2012, **49** ,*Tech. Suppl*., 08014.

12. Westwood S., Josephs R., Choteau T., Daireaux A., Wielgosz R. Mass Balance Method for the SI Value Assignment of the Purity of Organic Compounds, *Anal. Chem*., 2013, submitted for publication.

13. Josephs R.D., Daireaux A., Westwood S., Wielgosz R.I. Simultaneous determination of various estrogens by normal phase - liquid chromatography – tandem mass spectrometry with atmospheric pressure photoionization for the purity assessment of the monitored drug ß-estradiol, submitted for publication.

14. Stoppacher N., Daireaux A., Josephs R. D., Wielgosz R.I. Impurity identification and determination for the peptide hormone angiotensin I by liquid chromatography – high resolution tandem mass spectrometry and the metrological impact on value assignments by amino acid analysis, *Anal. Bioanal. Chem.* 2013 submitted for publication.

BIPM publications

15. Davis R., How the non-ideality of real gases affects results of the ozone Standard Reference Photometer (SRP), *Rapport BIPM*-2012/03, 10 pp.

6.5 Activities related to the work of Consultative Committees

The CCQM held its 18th meeting at the BIPM headquarters on 19 to 20 April 2012, and was preceded by meetings of its working groups.

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

S. Westwood is a member of the CCQM Working Group on Organic Analysis (OAWG) and the CCQM Working Group on Organic Analysis Taskforce on Core Key Competencies.

R. Josephs is a member of the CCQM Working Group on Bioanalysis (BAWG) and the CCQM Working Group on Organic Analysis (OAWG).

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

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

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

6.6 Activities related to external organizations

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

S. Westwood is the BIPM and CCQM liaison to the ISO-REMCO and is a member of the World Anti-Doping Agency (WADA) Laboratory Committee.

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.

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

6.7 Travel

R.I. Wielgosz to:

- Teddington (UK), 10-11 January 2012, to participate in the NMS Chem/Bio-metrology advisory working group;
- Biarritz (France), 2-3, February 2012, to attend the EURAMET Metchem Plenary Session;
- WMO Headquarters, Geneva (Switzerland) 14 February 2012, to attend the Joint Liaison Group with the WMO;
- IRMM, Geel, 13-14 March 2012, to participate in meetings of ISO TC 212 WG2;
- Lisbon (Portugal), 5-6 May 2012, to attend the IUPAC-ICTNS meeting;
- Washington DC (USA), 15-16 May 2012, to represent the JCTLM at the AACC Harmonization meeting;
- London (UK), 18-19 June 2012, to attend a BSI training course on the implementation of BS OHSAS 18001:2007;
- Los Angeles (USA), 14-15 July 2012, to attend the JCTLM Working Groups meeting and workshops;
- DIN, Berlin (Germany), 22 August 2012, to participate in meetings of ISO TC 212 WG2;
- Istanbul (Turkey) 9-11 October 2012, for the CCQM Working Group on Inorganic Analysis meeting;
- St Petersburg (Russian Federation), 24-26 October 2012, to attend the 28th CCQM Working Group on Gas Analysis meeting;
- Hong Kong (China), 6-9 November 2012, for the CCQM Working Group on Organic Analysis meeting.

J. Viallon to:

- York (UK), 11-12 September 2012, to attend the 4th Expert Workshop on Volatile Organic Compounds;
- St Petersburg (Russian Federation), 24-26 October 2012, to attend the 28th CCQM Working Group on Gas Analysis meeting.

E. Flores to:

- PTB, Braunschweig (Germany), 12-13 July 2012, to give an invited lecture on Gas Metrology by Fourier-Transform Infrared Spectroscopy;
- PTB, Braunschweig (Germany), 14-16 November 2012, to attend the EUMETRISPEC workshop on "Traceable spectral reference line data for atmospheric monitoring".

S. Westwood to:

- Harlow (UK), 3-6 March 2012, for the WADA Laboratory Directors Meeting and a WADA Laboratory Expert Group meeting;
- Vienna (Austria), 18-22 June 2012, for the 35th ISO-REMCO meeting;

- Vienna (Austria), 25-28 June 2012, for the 13th Biological and Environmental Reference Materials Symposium where he presented a keynote lecture entitled "Mass Balance Method for the SI Value Assignment of the Mass Fraction Content (Purity) of Organic Calibrators";
- Strasbourg (France), 1-2 October 2012, for the 11th International Symposium on Pharmaceutical Reference Standards;
- Hong Kong (China), 6-9 November 2012, for the CCQM Working Group on Organic Analysis meeting.

R. Josephs to:

- IAM, Budapest (Hungary), 1 March 2012, to represent the BIPM at the Inter-Agency Meeting of the Codex Alimentarius Commission;
- IAEA, Vienna (Austria), 25-29 June 2012, to present a poster International Symposium on Biological and Environmental Reference Materials (BERM-13);
- Adebiotech, Romainville (France), 2-3 October 2012, to attend the symposium: *Peptides issus des procédés d'hydrolyse*;
- HKGL, Hong Kong (China), 6-9 November 2012, to give presentations at the meeting of the CCQM Working Group on Bioanalysis;
- NIM, Beijing (China), 14-17 November 2012, to give a presentation and to discuss the future collaboration on the C-peptide project;
- World Bank, Paris (France), 11-12 December 2012, to attend the 1st Global Food Safety Partnership Conference.

N. Stoppacher to:

- Thermo Fisher Scientific, Courtabœuf (France), 16 February 2012, to attend the "Proteome Discoverer" software user meeting;
- IAEA, Vienna (Austria), 25-29 June 2012, to present a poster at the International Symposium on Biological and Environmental Reference Materials (BERM-13).

S. Maniguet to:

- Los Angeles (USA), 14-15 July 2012, to attend the JCTLM Working Groups meeting and workshops;
- Nantes (France), 15-18 October 2012, for a training course in the theory and application of qNMR for organic purity assessment.

6.8 Visitors

- S. Lee and J.Y. Lee (KRISS), 5-8 March 2012
- V. Stummer and V. Schinz (UBA), 26-30 March 2012
- J. Couette (LNE), 21 and 25 May 2012
- C. Zellweger (EMPA), 4-6 June 2012.

6.9 Guest workers

- K. Tworek (GUM), 1 June to 30 November 2012.