SIR Transfer Instrument

Protocol for the ongoing activity comparisons on site at the NMIs

BIPM.RI(II)-K4

Pilot laboratory: Bureau International des Poids et Mesures

CONTENT

1. Objective of the comparison

Short-lived radionuclides are essential for nuclear medicine where radionuclides with half-lives much shorter than one day are used as radiotracers. The use of nuclear medicine is increasing as these radionuclides become more accessible and this imposes new requirements on the National Metrology Institutes (NMIs). However, as a consequence of the short half-lives, sending ampoules that contain such a radioactive material to the Bureau International des Poids et Mesures (BIPM) for activity measurement of γ-ray-emitting radionuclides in the International Reference System (SIR) is only practical for NMIs that are based in Europe. In order to extend the use of the SIR and to enable other NMIs to participate, a transfer instrument (SIRTI) was developed at the BIPM following a request by the CCRI(II) in 2005 and with the support of the Consultative Committee for Ionizing Radiation CCRI(II) Transfer Instrument Working Group [1].

The SIRTI was calibrated against the SIR for the radionuclides of interest using ampoules sent to the SIR by the NMIs geographically close to the BIPM, and consecutively measured in the SIRTI after waiting until the activity had decayed sufficiently. These measurements allowed a linking factor to be deduced between the BIPM.RI(II)-K4 and the corresponding BIPM.RI(II)-K1 comparisons [2].

2. Radionuclides considered

The present protocol is currently applicable to ^{99m}Tc ($T_{1/2}$ = 6.0067(10) h), ^{18}F ($T_{1/2}$ = 1.8288(3) h), ^{64}Cu ($T_{1/2}$ = 12.7003(20) h) and ^{11}C ($T_{1/2}$ = 0.33935(38) h) which are called RN hereafter.

3. Realization

The SIR transfer instrument [3] is based on a well-type NaI(Tl) crystal the stability of which is monitored using a ^{94}Nb reference source ($T_{1/2}$ = 20 300(1 600) a) from the IRMM. The measurand is the RN count rate above a low-energy threshold defined by the ^{93m}Nb x-ray peak at ~ 16.6 keV, relative to the ^{94}Nb counting rate above the same threshold.

The reference source, which is an irradiated wire in a Plexiglass holder, contains both the ^{94}Nb and the ^{93m}Nb ($T_{1/2}$ = 16.12 (0.15) a) isotopes. Once the threshold is set, a brass liner is placed in the well to suppress the ^{93m}Nb contribution to the ^{94}Nb stability measurements.

The SIR ampoule to be compared is placed in the detector well with either a brass liner to suppress from the counts possible x-ray peaks close to the threshold, or a PVC liner to minimize the production of bremsstrahlung in the case of high-energy beta emitters. No extrapolation to zero energy is carried out as all the measurements are made with the same threshold setting.

The electronic system is described in [3]. The live-time technique (the MTR2 module from the LNE-LNHB [4]) is used to correct for dead-time losses. Each individual counting interval is limited in duration to minimize decay-related effects of the live-time correction [5-7]. The count rate effect of the SIRTI was measured using strong sources of ^{99m}Tc and ^{18}F showing that the data deviate from the decay curve only for count rates higher than 20 000 s^{-1}.

A SIRTI equivalent activity A_E is deduced from the RN and ^{94}Nb counting results and the RN activity measured by the NMI. The presence of a radioactive impurity in the solution should normally be accounted for using γ-ray spectrometry measurements carried out by the NMI.

$$A_{\mathrm{E}} = A\frac{c}{\lambda_{\mathrm{RN}}}\frac{\rho_{\mathrm{Nb}}}{N}\left[1 + \sum_i R_i \frac{\varepsilon_i}{\varepsilon_{\mathrm{RN}}}\frac{\lambda_{\mathrm{RN}}}{\lambda_i}\frac{c_i}{c} + BR_{99}\frac{\lambda_{\mathrm{Tc}}}{c_{\mathrm{Tc}}}\left(\frac{\lambda_{\mathrm{Tc}}c_{\mathrm{Mo}} - \lambda_{\mathrm{Mo}}c_{\mathrm{Tc}}}{\lambda_{\mathrm{Mo}}(\lambda_{\mathrm{Tc}} - \lambda_{\mathrm{Mo}})}\right)\right]$$

A	RN activity
N	RN counting corrected for live time and background
ρ_{Nb}	count rate of the ^{94}Nb measurement corrected for live time and background and calculated at t = 1 March 2007
$\varepsilon_{\mathrm{RN}}$ and ε_i	SIRTI detection efficiency for RN and for the impurity i, respectively
c and c_{i}	decay correction for RN and for the impurity i, respectively
c_{Tc} and c_{Mo}	decay correction for ^{99m}Tc and for ^{99}Mo, respectively

λ_{RN} and λ_i	decay constant of RN and of the impurity *i*, respectively
λ_{Tc} and λ_{Mo}	decay constant of ^{99m}Tc and ^{99}Mo, respectively
R_i	impurity to RN activity ratio
R_{99}	^{99}Mo to ^{99m}Tc activity ratio
B = 0.876 (19)	branching ratio of production of ^{99m}Tc in the ^{99}Mo decay.

The second term in the square brackets corresponds to the contribution of any possible impurity while the third term only applies to the case of ^{99m}Tc with a ^{99}Mo impurity and corresponds to the ^{99m}Tc growth from the ^{99}Mo decay. The detection efficiency of the SIRTI for the impurities are taken from earlier measurements in the SIRTI of a solution of the impurity nuclide or from the efficiency curve of the SIRTI evaluated by Monte-Carlo simulations with PENELOPE 2008 [13].

The influence of the radioactive solution density and volume has been investigated using a Monte-Carlo method with PENELOPE 2008 [3]. These simulations enabled to define the constraints on the RN solutions to be used (see section 9). The simulation was also used to deduce the uncertainty components related to the glass ampoule geometry. A typical uncertainty budget for a ^{99m}Tc measurement in the SIRTI (to which the uncertainty of the NMI activity measurement should be combined) is shown in Table 1.

For radionuclides emitting low-energy γ-rays like ^{99m}Tc, in which case interaction in the detector is dominated by the photo-electric effect, fluctuations of the threshold setting have negligible influence on the SIRTI measurement result. For higher energy γ-ray emitters like ^{18}F, the relative sensitivity of the response of the SIRTI versus the threshold position was evaluated to be about 2×10^{-4} per channel. This means that a change of threshold position by a few channels would have a non-negligible effect on the results. Consequently, when measuring such radionuclides, the room temperature should fluctuate by less than 2°C during the measurements, and readjustments of the threshold position may be necessary if exceeded.

For ^{11}C, an additional relative standard uncertainty of 6 parts in 10^5 related to the measurement time should be taken into account.

The calibration of the SIRTI against the SIR for RN[1] was carried out using RN solutions sent to the SIR by the NMIs geographically close to the BIPM, and consecutively measured in the SIRTI after waiting until the activity had decayed sufficiently to produce an acceptable count rate in the SIRTI [2]. This enabled linking factors $L = A_e/A_E$ to be deduced between the BIPM.RI(II)-K4 and BIPM.RI(II)-K1 comparisons (see Table 2), which are then used to calculate the SIR equivalent activity values for the SIRTI measurement results and consequently obtain degrees of equivalence with the Key Comparison Reference Value (KCRV) defined in the frame of the K1 comparison. The decision to include or not SIRTI comparison results in the KCRV is taken by the CCRI(II) (see section 8.2).

[1] In the case of ^{11}C, linking to the SIR has not been considered to date.

Table 1: typical uncertainty budget for a ^{99m}Tc measurement in the SIRTI

Uncertainty contributions due to	**Comments**	**Evaluation method**	**Relative standard uncertainties $\times 10^4$**
^{94}Nb measurement including threshold setting [a]	Weighted standard deviation of 6 series, each series consisting of 10 measurements	A	0.8
Long-term stability of the SIRTI	Weighted standard deviation of Nb reference source measurements since 2007	A	0.3
^{99m}Tc measurement including live-time, background and decay corrections [b]	Standard uncertainty of the weighted mean, taking into account the correlation due to the ^{99m}Tc half-life.	A	2
SIRTI drift at high count rate	Mean drift over all ^{99m}Tc measurements at the NMI.	B	3
Ampoule dimensions	From IRMM report [8] and sensitivity coefficients from Monte-Carlo simulations	B	7
Ampoule filling height	Solution volume requested in the protocol is 3.6(1) cm^3; sensitivity coefficients from Monte-Carlo simulations	B	3
Solution density	Between 1 g/cm^3 and 1.01 g/cm^3 as requested in the protocol; sensitivity coefficients from Monte-Carlo simulations	B	0.8
Unseen solution droplet on the walls of the ampoule head	From Monte-Carlo simulations; not relevant if ampoule centrifuged	B	1.6
Relative combined standard uncertainty			**8.7**

a. The uncertainty associated with the decay correction using $T_{1/2}$ = 20 300 (1600) a, is negligible. The standard uncertainty associated with the live-time correction (effect of finite frequency) and the background is also negligible.

b. The measurement duration is limited so that the effect of decay on the live-time correction is less than 10^{-4}.

Table 2: Linking factors between the BIPM.RI(II)-K4 and BIPM.RI(II)-K1 comparisons

Radionuclide RN	**Linking factor *L***	**Date of evaluation**
^{18}F	1495.1(18)	March 2015
^{64}Cu	1479.9(17)*	April 2015
^{99m}Tc	12 165(23)	March 2015

* preliminary value; further measurements are planned

4. Participants

Participants can be any NMI or designated institute of a BIPM Member State, situated far from the BIPM (more than 1500 km or outside the European Union). The NMIs having developed primary measurement methods or with CMCs in the KCDB will be given first priority. The CCRI(II) members will then have second priority. Participants ready to compare several RN in a single campaign will also be given priority, in order to minimize costs. The pilot laboratory is the BIPM. Laboratories who would like to participate should contact C. Michotte (cmichotte@bipm.org).

5. Preparation of the comparison

5.1. Coordination with the NMI

The NMI needs to be prepared to make the shipping arrangements and pay for the return of the equipment from their site back to the BIPM. The participant is informed about the case dimensions and its weight for reception at the laboratory. The Nb reference source forms part of the equipment, but its activity is below the exemption level so that the package is not radioactive material under the transport regulations of the IATA, the ICAO, the IAEA, or the U.S. DOT, and the package is exempt from all radioactivity labeling requirements.

The BIPM will pay the travel costs of its staff members and the subsistence indemnity. It is requested that the NMI pays the local hotel accommodation for the BIPM staff members.

The NMI needs to make available some equipment necessary for the comparison (see list in section 10). The synchronization of the clock of the SIRTI laptop to UTC within about 2 s (0.4 s for ^{11}C) is necessary. If the use of the software http://www.timesynctool.com/ on the SIRTI laptop connecting to an NTP time server is not possible, the NMI should contact the BIPM to discuss the issue. The NMI informs the BIPM whether empty NBS/SIR ampoules are needed as these are necessary for the comparison (see section 10).

About six months in advance, the NMI is requested to fill-in, sign and send the registration form (annex 1) to the BIPM. Grouping of the SIRTI comparisons for different radionuclides should be organized when possible. The NMI informs the BIPM as soon as possible about:

- additional information needed by the NMI to allow the BIPM staff to work in their laboratory;
- expected duration of the process of importation and re-exportation of the equipment by the NMI;
- availability of a centrifuge for SIR ampoules at the NMI.

5.2. Registration of the participant in the Key Comparison Database (KCDB)

At least four months in advance, the date for the comparison is decided in agreement with the NMI (comparison duration is about one week for a single RN). The participation is registered in the KCDB.

5.3. Preparation of the importation/exportation of the equipment

Four months in advance to the comparison, or more depending on the destination, the BIPM checks whether the participating country accepts an ATA carnet[2] for professional equipment. In the affirmative, the carnet is prepared by the BIPM for the equipment exportation and the same carnet is also used by the participant for re-exportation of the equipment back to the BIPM (see section 7). In the negative, temporary importation and documentation is organized in collaboration with the NMI.

The NMI and the BIPM exchange the name and full contact details of the person responsible for the reception of the equipment and eventually the contact details of a shipping agent at the airport, as well as the list of all necessary documents that the BIPM and the NMI need to prepare for the importation and re-exportation of the equipment. The NMI and the BIPM should agree on whether a door-to-door delivery or a delivery at the airport should be organized.

5.4. Test of the SIRTI

Three months in advance to the comparison, the SIRTI is tested using the two Nb reference sources at the BIPM [9-10] before shipping the electronic equipment to the NMI.

5.5. Shipping to the NMI

Two months in advance (or more depending on the destination), the equipment is shipped to the NMI by cargo except for the detector and liners which are transported by the BIPM staff as hand luggage. The NMI is reminded of the list of equipment they need to provide for the comparison (see section 10).

The NMI informs the BIPM immediately on the arrival of the equipment. If the case is visibly damaged, the NMI informs the BIPM and opens the case to check whether some equipment has been damaged and needs to be replaced. If the case is intact, the case is not opened until the arrival of the BIPM staff (except for security reasons). The ATA carnet should be kept safely until re-exportation (see section 7).

[2] The ATA carnet is an international customs document used for temporary importation /exportation (within one year) without having to pay the normal customs duties and taxes. The ATA carnet is not needed within E.U.

6. Comparison at the NMI [9-10]

During the comparison, the NMI should have, for each RN, one solution available (if possible, two solutions delivered at different dates) from which sources with possibly different dilution factors are made for the NMI activity measurements, γ-ray spectrometry impurity check and for the SIRTI measurements. See section 9 for more details about the RN solution.

6.1. Measurement method selected by the NMI

The NMI measures the activity concentration of the solution by the method of its choice. For practical reasons, the NMI may decide not to repeat a primary measurement during the comparison but use e.g. an ionization chamber calibrated by the NMI beforehand. The traceability of any secondary measurement method should be clearly stated (see also section 8.1). It is recommended that the NMI prepares the possibility of standardizing the solution by a second method in case of failure of their main measurement system. It is important also to remember that, as for any other international comparison, the method selected by the participant for the calculation of the degrees of equivalence in the KCDB should correspond to the reference primary method disseminated in the country (or be traceable to it).

Activity measurements and impurity checks are carried out by the NMI using sources made from the same solution as the one measured in the SIRTI. The impurity check should preferably be carried out at low count rate to limit pile-up, and, if possible, during the last hours of the SIRTI measurements.

6.2. The SIRTI measurements

The detector case which was carried by hand should be opened only after some temperature stabilization in the laboratory.

The stability of the system is checked by repeating the ^{94}Nb, background and clock measurements. Standalone background measurements are carried out overnight.

The comparison is carried out by measuring in the SIRTI a SIR ampoule with the RN solution produced locally (see section 9) and standardized by the NMI, alternating with background measurements. It is recommended that the laboratory prepares two ampoules at the same time so that a quick check of consistency between the two ampoules can be carried out at the start of the SIRTI measurements. If a discrepancy is noted between the equivalent activities of the two ampoules, this information will be used as an additional uncertainty contribution to the comparison result uncertainty. Measurements of a single ampoule are carried out until the count rate in the SIRTI is about 1000 s^{-1} (after about 24 h at maximum). On the last day, a final series of ^{94}Nb measurements is carried out.

If the activity in the ampoule is too low for a reliable SIRTI result, the NMI is informed immediately so that another ampoule can be prepared.

If the presence of impurities is suspected from the SIRTI measurements (decreasing trend in the results or peak in the energy spectrum), the NMI is informed.

The participant should give his preliminary results as early as possible during the comparison. The BIPM staff evaluates a preliminary equivalent activity value and informs the participant whether the result is an outlier using the normalized error test with a test value of four:

$$(A_{\mathrm{e,NMI}} - \mathrm{KCRV}) / \sqrt{u(A_{\mathrm{e,NMI}})^2 + u(\mathrm{KCRV})^2} \; > \; 4 .$$

In case of an outlier, investigations are carried out to identify the reason for the discrepancy. If the discrepancy remains unsolved, a further RN ampoule can be produced and measured immediately, or the next day if there is time and activity available. For ^{18}F, it is not advisable to measure the same solution more than 24 hours after production because of possible impurities which can start to have a significant influence on the measurement result. If the discrepancy still remains unsolved, a second RN solution, if available, can be measured later during the comparison.

If the result for a first RN solution is not identified as an outlier, then a second solution should not be measured, unless the first measurements were carried out under bad measurement conditions (e.g. failure of some equipment, problem in the solution delivery, unstable SIRTI measurements, large drop in the ampoule top, or large impurity).

At the end of the comparison, the SIRTI is dismantled and packed in the cases by the BIPM staff.

7. Shipping the SIRTI back to the BIPM

The NMI will ship back the equipment to the BIPM within 3 weeks of the end of the comparison (see in Annex 2 the BIPM procedure ADM-DOU-T-02.pdf and the form BIPM/ADM-DOU/F-12 to be completed – except section 4 – and sent to the BIPM) and will pay the shipping costs, including insurance. Upon reception of the form F-12, the BIPM sends a letter to the NMI authorising the NMI or its shipping agent to use the ATA carnet, if relevant. The ATA carnet should be submitted to customs on leaving the country and on arrival in France.

The BIPM acknowledges receipt of the equipment and within one month the SIRTI is set up again and checked with the Nb source at the BIPM.

8. Publication of the comparison result

8.1. Analysis of the comparison data [11]

The analysis is made once the RN activity value (and impurity content) together with an uncertainty budget is obtained from the NMI using the reporting form in annex 3.

This form should be returned to the BIPM within 6 months after the comparison. The acronyms for describing the measurement methods are also listed in annex 3.

If the participant submits several results corresponding to different standardization methods, a single value and uncertainty (e.g. one of the results or a weighted mean of some or all results) that represents its national reference should also be provided as this will be used to calculate the degrees of equivalence for the KCDB.

If the result appears to be anomalous (i.e. one that could be identified as an "outlier" as defined by the KCWG), the participant is contacted but without being informed as to the magnitude or sign of the apparent anomaly. A two-month deadline is given to check their results for numerical errors (typographical or transcription errors, units used) according to [14]. If the anomaly is not solved, the result is published or could be withdrawn under conditions described in [14]. If two RN solutions were measured and both results are anomalous, a mean result is used as final result.

8.2. Comparison report

Within 3 months after reception of the final NMI results, the draft A is prepared at the BIPM including the link to the BIPM.RI(II)-K1 key comparison. The degrees of equivalence are calculated in the frame of the K1 comparison (SIR), using the linked SIRTI results.

For ^{99m}Tc and ^{18}F, the linked result of the K4 comparison is eligible to be included in the KCRV of the K1 comparison if the K4 result is based on a primary method or on an ionization chamber measurement calibrated within the NMI by primary measurements of ^{99m}Tc or ^{18}F within one year of the comparison date. For the other RN considered in this protocol, the K4 comparison results are presently not included in the KCRV but could be included at a later stage.

After agreement on the draft A, the draft B is prepared and the NMI is invited to communicate some detail of their measurements or to refer to a publication. The amended draft B is then circulated through the CCRI(II) for approval to become the final report and be published normally in the *Metrologia* technical supplement.

9. Source characteristics

The participant is requested to prepare 3.6(1) cm^3 of RN solution in a NBS/SIR ampoule of 8 cm height maximum. The density of the solution should be lower than 1.01 g/cm^3 (RN and NaCl dissolved in water or low molarity HCl or HNO_3). For ^{18}F, it is advised to use a ^{18}FDG[3] solution as it is known to contain less impurities than other types of ^{18}F solutions. The solution may be the same as that standardized by the NMI or be a dilution of this solution. The latter enables a measurement sooner in the SIRTI and so with less effect on the results from the possible impurities. This also reduces the uncertainty related to the half-life but adds

[3] fluoro-deoxy-glucose

an uncertainty related to the dilution. The total activity in the SIR ampoule needed for the SIRTI measurements is given in the following table:

Radionuclide RN	**Activity / kBq**
^{11}C	20 – 30
^{18}F	10 – 25
^{64}Cu	60 – 120
^{99m}Tc	15 – 30

Empty NBS/SIR ampoules can be provided in advance if necessary (as for the SIR). They should be carefully cleaned, and eventually treated using a hydrophobic agent like dichlorodimethylsilane, in order to avoid the formation of droplets on the walls of the ampoule. Indeed the presence of 5 medium-size droplets on the walls of the top of the ampoule may be sufficient to produce a relative change of about 2×10^{-3} in the SIRTI measurement result. At the KRISS, a siliconizing solution is used to treat ampoules. Another procedure used at the ENEA (Italy) can be obtained at request. See also Appendix 1 and 2 of the *Monographie* BIPM-1 [12]. If droplets occur despite careful cleaning, centrifuging the ampoule (3000 r/min for 5 min) has shown to be effective in draining the solution into the bottom of the ampoule. The NMI should inform the BIPM about 6 months prior to the comparison whether a centrifuge for SIR ampoules is available in their laboratories.

No paper label should be glued on the ampoule.

The RN activity and impurity ratio should be measured by the NMI.

10. Equipment to be prepared by the NMI

In an air-conditioned room (temperature fluctuation less than 2 °C in 24 hours):
- NIM crate (5 units wide) with +/- 24 V (min. 500 mA), +/- 12 V (min. 500 mA) and +/- 6 V power supply (min. 500 mA) (free of pick-up noise);
- voltmeter (range 0 V to 10 V at 1 %);
- oscilloscope (digital preferably);
- table 40 cm × 40 cm min, preferably not higher than 70 cm, for the detector stand;
- table/desk for the laptop, with internet access by cable (to synchronize the computer clock to a time server);
- uninterruptible power supply because the measurements should continue over night;
- centrifuge for SIR ampoules (3000 r/min) if available at the NMI.

The distance between the detector and the crate should be less than 6 m and between the crate and the laptop less than 4 m.

The measurement laboratory and neighbouring rooms should be free of sources that could affect the stability of the background.

References

[1] Remit of the CCRI(II) Transfer Instrument Working Group, 2009, CCRI(II)/09-15.
[2] Michotte C. *et al*., Calibration of the SIRTI against the SIR for Tc-99m and F-18 and trial comparisons at the NPL. Paper in preparation.
[3] Michotte C. *et al*., The SIRTI, a new tool developed at the BIPM for comparing activity measurements of short-lived radionuclides world-wide. *Rapport BIPM*-2013/02.
[4] J. Bouchard, 2000, *Appl. Radiat. Isot*. **52**, 441-446.
[5] B. Chauvenet, private communication.
[6] Baerg A.P. et al., 1976, Live-timed anti-coincidence counting with extending dead-time circuitry, *Metrologia* **12**, 77-80.
[7] R. Fitzgerald, The combined dead-time and decay effect on live-timed counting systems with fixed, extending dead times, TIWG(II)/09-10, BIPM, June 2009.
[8] Sibbens G., 1991, A comparison of NIST/SIR-, NPL-, and CBNM 5 ml ampoules, GE/R/RN/14/91, CEC-JRC Central Bureau for Nuclear Measurements, Belgium.
[9] C. Michotte, Setting up the SIR Transfer Instrument, Technical Instructions BIPM/RI-SIR-T-15, BIPM QMS.
[10] C. Michotte, Measurements using the SIR Transfer Instrument, Technical Instructions, BIPM/RI-SIR-T-16, BIPM QMS.
[11] C. Michotte, Analysis of the measurements data of the ^{99m}Tc K4 comparisons, Technical Instructions, BIPM/RI-SIR-T-18, BIPM QMS.
[12] *Monographie BIPM*-1, 1975, Procedures for accurately diluting and dispensing radioactive solutions, BIPM.
[13] Michotte C. *et al*., Monte-Carlo simulations of the SIRTI using PENELOPE. Paper in preparation.
[14] CIPM MRA, Measurement comparisons in the CIPM MRA, CIPM MRA-D-05 Version 1.5.

Annex 1

Registration form for BIPM.RI(II)-K4 (SIRTI) comparisons

Laboratory:

Proposed period for the comparison[§]:
Proposed period for SIRTI exportation from BIPM[§]:

Radionuclide	Measurement methods planned by the NMI	Protocol agreed

- Contact person at the NMI (name, phone, e-mail):

- Shipping agent to be contacted (name of company, contact name, address, phone, fax and e-mail):

- Addressee[**] for the equipment (name, address, phone, fax and e-mail):

- Expected duration of the import process of the SIRTI by the NMI:

- Expected duration of the re-export process of the SIRTI by the NMI:

- Authorization or special Visa needed to work at the NMI:
  (If relevant, please send details to the BIPM as soon as possible)

[§] To be filled-in by the BIPM
[**] The addressee should be present at the NMI for reception of the equipment

INSTRUCTIONS FOR METROLOGY INSTITUTES SHIPPING EQUIPMENT TO THE BIPM FOR COMPARISONS

1 General Information

- Equipment shipped to the BIPM for comparisons is subject to Customs' formalities, which vary according to the country of origin.
- Before shipping any material to the BIPM, the metrology institute shall complete the relevant parts of the form **BIPM/ADM-DOU/F-12**, and return it duly signed to the BIPM (fax: **+33 1 45 07 70 99** or e-mail at **ldelloro@bipm.org**. The form should be received by the BIPM at least 2 weeks before shipment is planned.
- Parcels from countries other than the E.U. must be labelled as follows:

BIPM - REGLEMENTATION SPECIALE - NE PAS DEDOUANER D'OFFICE

and the metrology institute from which the equipment originates should give specific instructions to their carrier to contact the BIPM
[Contact: Administration, tel.: +33 1 45 07 70 29 fax: +33 1 45 07 70 99]
prior to clearing the instrument through Customs. The BIPM will then take the appropriate action to clear the equipment through French Customs.

- No Customs' operations are carried out on Saturdays or Sundays. The metrology institute should ensure that if their equipment is subject to Customs' formalities, it should arrive in France on a working day of the week preceding that planned for the comparison.
- Customs' operations for hand carried equipment may require processing by the BIPM. In this case, relevant costs will be charged to the metrology institute.

2 Customs' formalities

2.1 Equipment arriving from a country within the E.U.:

- There are no Customs' formalities. The metrology institute does not need to take further action.

2.2 Equipment arriving from a country outside the E.U.:

- There are Customs' formalities. In order for the equipment to pass through Customs, the metrology institute is required to undertake one of the following procedures:

i. ship the equipment with an ATA carnet. This carnet is available through the Chamber of Commerce and Industry (or equivalent within your country, provided your country

recognises this system) and is issued with one year validity. It simplifies the Customs' operations and avoids duties and taxes;

ii. ship the equipment by diplomatic bag to the relevant Embassy in Paris (although this has the advantage of by-passing all Customs' formalities, it is unlikely that this process is available to all metrology institutes);

iii. if neither of these procedures can be adopted, a temporary importation will be arranged by the forwarding agent of the BIPM (all sections of the form **BIPM/ADM-DOU/F-12** must then be completed) and the relevant costs will be charged to the metrology institute. For hand carried equipment this will include an appointment on arrival at the airport with the forwarding agent of the BIPM, on a working day..

3 Transport of equipment between Paris Airports and the BIPM

3.1 Equipment arriving from a country within the E.U.:

- For equipment originating from a metrology institute within the E.U., it is expected that the metrology institute will arrange a door-to-door delivery.
- In the case of air transport, it is expected that the metrology institute will arrange for their carrier to transport the equipment to and from Paris airports and the BIPM.

3.2 Equipment arriving from a country outside the E.U.:

- For those countries employing the ATA carnet system, it is expected that the metrology institute will arrange a door-to-door delivery. In the case of air transport, it is expected that the metrology institute will arrange for their carrier to transport the equipment to and from Paris airports and the BIPM. The relevant costs will be charged to the metrology institute.
- For hand carried equipment, the metrology institute will arrange its transport between Paris airports and the BIPM.
- Where a temporary importation has to be arranged, the BIPM via its forwarding agent will arrange and meet the transport of the equipment to and from Paris airports and the BIPM.

4 Insurance of equipment

- In all cases, organisation and payment of insurance for a visiting metrology institute's instrument remain the responsibility of the visiting metrology institute.

5 Return of equipment

- It is the responsibility of the metrology institute to make prior arrangements for the return of their equipment after the comparison. The BIPM should be informed of these arrangements using form **BIPM/ADM-DOU/F-12.**
- No shipment back to the metrology institute will be arranged by the BIPM in the absence of this form duly completed and signed.
- Part "4. Instructions for return" of the form BIPM/ADM-DOU/F-12 is not applicable for BIPM equipment.

Version number	Date of Issue/Review	Author	Modifications / comments
2.1	10-12-2013	LD	Updated contact names

Procédures Administration / Shipping instructions for comparisons				QUALITY MANAGEMENT SYSTEM
Authors : Isabelle Andernack Brigitte Perent	Date : 2012/09/12 Version : 1.1	Authorized : Brigitte PERENT	BIPM/ADM-DOU-F-12	

1. SHIPPING INSTRUCTIONS FOR COMPARISONS

- Name of the metrology institute:
- Person to be contacted:
- Address:
- Tel.: • Fax: • e-mail:

2. ATA carnet: ☐ Diplomatic bag: ☐ Other case: ☐

3. SHIPPING INFORMATION

- Description of the equipment (copy of proforma invoice required):
- Value of the equipment: • Number of packages:
- Gross weight: • Net weight:
- N° AWB (when available): • Date AWB:
- Name of the carrier:
- Hand carried by air (if necessary): • flight number*: • Date:
  * A copy of the flight ticket and passport is required for travellers coming from non European countries
- Hand carried by other means of transportation (to specify): • Date:

4. INSTRUCTIONS FOR RETURN

- Insurance: ☐ Yes ☐ No
- Name of the carrier:
- Tel.: • Fax: • e-mail:
- Your client number with the carrier:

5. I agree to pay for all the costs related to Customs' formalities and transport of equipment.

Date **Name and title** **Signature**

- SIRTI box dimensions, weight and UN number[§]:

- SIRTI reference source characteristics[§]:

  # Please note that the NaI(Tl) detector and accessories will be transported by the BIPM staff as hand luggage.

- Number of empty SIR ampoules needed: ☐

- Are there constraints for the weekday of delivery of the radionuclides (yes/no)?
  (If relevant, please send details to the BIPM as soon as possible)

- Is it possible to access the laboratory

  over the week-end (yes/no)?
  early in the morning (yes/no)? late in the evening(yes/no)?
  with a laptop, camera or other electronic equipment (yes/no)?

  # Please note that standalone measurements with the SIRTI will run over night.

- Is it possible to centrifuge the SIR ampoules (~3000 t/min) at your laboratory (yes/no)?

- Is a connection to a NTP time server possible from your laboratory (e.g. 193.104.127.9), to synchronize the laptop time to UTC (yes/no)?

- The NMI is kindly requested to pay the shipping back of the SIRTI to the BIPM and the hotel accommodation of the BIPM staff (X persons)
- The NMI should make available some equipment necessary for the comparison as described in the protocols
- The NMI agrees with the protocol(s) mentioned above

Date Name Signature

BUREAU INTERNATIONAL DES POIDS ET MESURES

International Reference System
for activity measurements of gamma-ray emitting nuclides (SIR/SIRTI) (in solution)

Participating laboratory: _

Radionuclide (main contribution) [ ] $T_{1/2}$: _ _ _ _ _ _ u: _ _ _ _ _

Chemical composition of the solution:

Solvent: _ _ _ _ _ _ _ and its concentration: _ _ _ _ _ _ _ mol per dm^3 of solution

Carrier: _ _ _ _ _ _ _ _ and its concentration: _ _ _ _ _ _ _ µg per g of solution

Density of the solution: _ _ _ _ _ _ _ g cm^{-3}

Ampoule number: _ _ _ _ _ _ _ Mass of solution (corrected for buoyancy): _ _ _ _ _ _ _ g

Activity per gram of solution (main radionuclide): _ _ _ _ _ _ _ Bq g^{-1}

Reference date: _ _ _ _ year _ _ _ _ month _ _ _ _ day _ _ _ _ h UTC*

Measurement date: _

Uncertainties (in the form of standard uncertainties). **Please attach also a detailed uncertainty budget** :

Type A (evaluated applying statistical methods): _ _ _ _ Bq g^{-1}; _ _ _ _%

Type B (evaluated by other means): _ _ _ _ Bq g^{-1}; _ _ _ _%

Method(s) of measurement: _

Acronym: (see attached page)

For relative methods, please indicate the primary methods and the standards used to calibrate your experimental setup : _
_ _
the date of calibration :_ _
and also the date of the primary measurement :_ _.

Radionuclide impurities :	Nuclide	Ratio of activity of impurity to activity of main radionuclide at reference date
	_ _ _ _ _	_ _ _ _ _; u = _ _ _ _ _
	_ _ _ _ _	_ _ _ _ _; u = _ _ _ _ _

Date of gamma spectrometer measurements: _

Remarks: _

This sample has been submitted in the frame of a pilot study _ _ or to generate an equivalence value X.

Date: _ _ _ _ _ _ _ _

Name of person responsible: _ _ _ _ _ _ _ _ _ _ _ _ _ _ _

Name of person(s) who carried out the measurements: _ _ _ _ _ _ _ _ _ _ _ _ _ _ _

*UTC = coordinated universal time

Annex 3

Detailed Uncertainty Budget

Laboratory: _ _ _ _ _ _ _ _ _; Radionuclide: _ ^{99m}Tc_ _ _ _ _ _ ; Ampoule number: _ _ _ _ _ _ _ .

Uncertainty components[*], in % of the activity concentration, due to

	Remarks	Evaluation type (A or B)	Relative sensitivity factor
counting statistics			
weighing			
dead time			
background			
pile-up			
counting time			
adsorption			
impurities			
tracer			
input parameters and statistical model			
quenching			
interpolation from calibration curve			
decay-scheme parameters			
half life ($T_{1/2}$ = _ _ _ _ _ u = _ _ _ _ _)			
self absorption			
extrapolation of efficiency curve			
other effects (if relevant) (explain)			
combined uncertainty (as quadratic sum of all uncertainty components)			

[*] The uncertainty components are to be considered as approximations of the corresponding standard deviations (see also *Metrologia*, 1981, **17**, 73 and *Guide to expression of uncertainty in measurement*, ISO, corrected and reprinted 1995).

List of acronyms proposed to be used to identify different measurement methods

Each acronym has six components, geometry-detector (1)-radiation (1)-detector (2)-radiation (2)-mode. When a component is unknown, ?? is used and when it is not applicable 00 is used.

Geometry	acronym	Detector	acronym
4π	4P	proportional counter	PC
defined solid angle	SA	press. prop. counter	PP
2π	2P	liquid scintillation counting	LS
undefined solid angle	UA	NaI(Tl)	NA
		Ge(HP)	GH
		Ge(Li)	GL
		Si(Li)	SL
		CsI(Tl)	CS
		ionization chamber	IC
		grid ionization chamber	GC
		Cerenkov detector	CD
		calorimeter	CA
		plastic scintillator	SP
		PIPS detector	PS
Radiation	**acronym**	**Mode**	**acronym**
positron	PO	efficiency tracing	ET
beta particle	BP	internal gas counting	IG
Auger electron	AE	CIEMAT/NIST	CN
conversion electron	CE	sum counting	SC
mixed electrons	ME	coincidence	CO
bremsstrahlung	BS	anti-coincidence	AC
gamma rays	GR	coincidence counting with efficiency tracing	CT
X - rays	XR	anti-coincidence counting with efficiency tracing	AT
photons (x + γ)	PH	triple-to-double coincidence ratio counting	TD
alpha - particle	AP	selective sampling	SS
mixture of various radiations	MX	high efficiency	HE

Examples	
method	**acronym**
4π(PC)β–γ-coincidence counting	4P-PC-BP-NA-GR-CO
4π(PPC)β–γ-coincidence counting eff. trac.	4P-PP-MX-NA-GR-CT
defined solid angle α-particle counting with a PIPS detector	SA-PS-AP-00-00-00
4π(PPC)AX-γ(GeHP)-anticoincidence counting	4P-PP-MX-GH-GR-AC
4π CsI-β,AX,γ counting	4P-CS-MX-00-00-HE
calibrated IC	4P-IC-GR-00-00-00
internal gas counting	4P-PC-BP-00-00-IG