Criteria for a RMO SIRTI

In order to extend the use of the SIR to short-lived radionuclides, especially for NMIs and DIs located farther from the BIPM, a transfer instrument (SIRTI) based on a transportable well-type 3" × 3" NaI(TI) scintillator, has been developed at the BIPM in 2009. When appropriate, the BIPM.RI(II)-K4 comparisons carried out with the SIRTI are linked for each radionuclide of interest to the corresponding BIPM.RI(II)-K1 comparisons through the measurement of the same (diluted) radioactive solution in both the SIRTI and the SIR.

To date (March 2024), the SIRTI has been linked to the SIR for ¹⁸F, ^{99m}Tc and ⁶⁴Cu and the estimation of the link for ¹²³I and ¹⁵³Sm is in progress. The SIRTI comparison service also includes ¹¹C and ¹³N (without link to the SIR) and would be enlarged to ⁵⁶Mn, ⁶⁸Ga and ¹⁶⁶Ho in the next few years. Needs for further radionuclides have been identified.

A need has been expressed by the RMOs (APMP, SIM and possibly EURAMET) to develop regional SIRTIs, under the responsibility and budget of each RMO.

The interest of RMO SIRTIs in radionuclide metrology is on several levels:

- to increase the frequency of world-wide SIRTI comparisons for radionuclides critical to nuclear medicine as the BIPM can hardly conduct more than one K4 comparison per year,
- to increase the number of participants to institutes that are not members of the CCRI(II) and to institutes that could not directly benefit from current BIPM services (associates or non NMI/DIs) but can participate in RMO comparisons.

The BIPM workplan includes support to future RMOs pilot laboratories in constructing and validating RMO SIRTIS. In addition, the BIPM and the RMOs could collaborate in developing digital electronics for the SIRTI.

In 2023, the CCRI agreed that RMO SIRTI comparisons would be linked to the SIR through the BIPM SIRTI. Therefore, the linking measurements between the BIPM and the RMO SIRTIs would be carried out in the RMOs. The CCRI also decided that a RMO instrument can be named "RMO SIRTI" and run RMO.RI(II)-K4 comparisons if the instrument is sufficiently similar to the BIPM SIRTI so that the same measurement procedure can be used (see annex 3).

The criteria are the following:

1. Detector type and size:

A NaI(Tl) scintillator substantially similar to the BIPM SIRTI detector (see annex 1) should be used. Note that

- smaller detectors would have larger Compton-to-photoelectric ratio at medium /high γ -ray energy, and so larger uncertainty from SIRTI threshold setting;

- larger detectors would need larger dilution factors of the mother solutions and thicker shielding to keep acceptable source to background ratio.

- the well depth of 5 cm for the BIPM SIRTI enables to have a negligible effect of the solution drops often appearing on the cylindrical wall of the ampoule on the

SIRTI response. Drops in the ampoule's head need to be removed by centrifuging.

2. Detector stand and shielding (see annex 2):

The BIPM Nal(Tl) detector is fixed in an aluminium ring which remains screwed permanently for better reproducibility of the detector position. It is connected to a tripod enabling the adjustment of verticality of the detector as well as its centering in the lead shielding to better than 0.5 mm.

The BIPM SIRTI has a one-piece cylindrical graded shielding made of 2 mm Cu / 3 mm Sn / 5 mm Pb. A thicker lead layer would be useful but becomes too heavy for a single piece so that a different design would be needed. The BIPM lead shielding can be fixed to the tripod which is mandatory for seismic countries.

3. The **minimum count rate** to be used in comparisons should be higher than 20 times the background rate.

Note that the background count rate measured with the BIPM SIRTI can vary by a factor up to five depending on the natural radioactivity in the country, limiting in some cases the dynamic range of the measurements.

4. Threshold energy:

The BIPM SIRTI is based on integral counting above a low energy threshold defined by the energy of the ^{93m}Nb x-ray peak and showing a relative FWHM of about 30 %. Other x-ray peak energy between 15 keV and 20 keV could be used if needed.

For the BIPM SIRTI, the threshold position is at least 5 times higher than the noise floor:



A threshold position close to the noise floor may have an impact on the linearity of the SIRTI at high rate because of pileup between under-threshold events with noise.

Note that for comparison measurements a brass or low-Z **liner** should be used to attenuate the x-rays with an energy around the threshold or minimize bremsstrahlung from beta emission, respectively.

5. Threshold setting repeatability:

Monte-Carlo simulations of the BIPM SIRTI showed that the under threshold to total energy spectrum ratio is the largest for γ -rays between 300 keV and 800 keV, in correspondence with a large Compton continuum. Consequently, this γ -ray energy range is where the threshold setting repeatability is expected to have the largest impact on *R*, the measured count rate above threshold. The sensitivity of *R* on the threshold position was measured as 9×10^{-5} per channel for ⁹⁴Nb (see [1] page 10). If it is assumed that the threshold energy is set within an uncertainty interval of 4 channels (with squared probability distribution), the corresponding u(R)/R is calculated as 0.01 %* for the BIPM SIRTI. Note that for ⁵⁷Co the sensitivity of *R* on the threshold position was found to be negligible. Using digital electronics may reduce this uncertainty component. Nevertheless a relative uncertainty component of maximum 0.03 % would still be acceptable to run RMO SIRTI comparisons.

6. Reference source

The decay correction of the reference source should have a relative uncertainty less than 0.05 % over min. 20 years.

Reproducibility of the reference source count rate above threshold (dismount the SIRTI and re-setup; different environmental conditions) should be maximum 0.05 %. For the BIPM SIRTI, the ⁹⁴Nb count rate measured over 15 years of measurements at the BIPM and many other places world-wide show a relative standard deviation of 0.025 %.

- 7. Non-linearity versus integral count rate above threshold, corrected for dead time, should be 0.08 % maximum, in a count rate dynamic range of at least a factor of 10. For the BIPM SIRTI, the non-linearity reaches 0.05 % at 20 000 s⁻¹ ***. Note that during a SIRTI comparison the radionuclide solution is measured over at least two half lives so that only a small part of the measurements suffers a significant non-linearity.
- 8. A **validated Monte-Carlo model** should be available in view to evaluate uncertainty components from the source geometry (ampoule glass wall thickness, solution volume, etc.) and to calculate detection efficiencies for impurities. Validation of the detection efficiency against measurement within

better than 1 % should be obtained for Tc-99m and F-18. For the BIPM SIRTI, Monte-Carlo to measurement ratio is 1.0047(41) and 1.0074(33) for Tc-99m and F-18, respectively**.

9. The target combined standard uncertainty for RMO SIRTI comparison measurements is 0.15 %, excluding the uncertainty of the link to the SIR and uncertainties not related to the SIRTI instrument (e.g. the activity standardization of the solution and impurities by the participant or the unexpected spread in the results for the different ampoules measured). What is included, is the uncertainty from ampoule/solution geometry (ampoule glass walls, solution volume etc) which has been evaluated to 0.09 % at 140 keV. For BIPM SIRTI comparison measurements, the combined standard uncertainty is usually lower than 0.1 % but would be larger for ¹⁵³Sm due to the ampoule's wall effect at 100 keV.

If the uncertainty for one of the items above is larger than the recommended limit it can be compensated by a smaller uncertainty for another item. It is worth noting that a larger uncertainty for the threshold setting would impact the reproducibility of the system.

If for some reason, the proposed RMO SIRTI does not fully comply with these criteria, a full study of linearity versus count rate, measurement uncertainty, reproducibility, Monte-Carlo model, and justification for considering the system equivalent to the BIPM SIRTI should be carried out and made available to the CCRI(II).

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Footnotes:

* All uncertainties in the document are given at k = 1

** The uncertainty of the Monte-Carlo simulation includes the components from statistics and the decay data.

*** A way to check linearity is to measure a ¹⁸F source over minimum three half lives and look whether the count rate corrected for dead time (e.g. by the live-time method), for background and decay is constant. In the figure below, the SIRTI equivalent A_E which is inversely proportional to the count rate corrected for dead time, background and decay is plotted versus the input count rate. Several data sets obtained using the BIPM SIRTI show a decreasing trend at high count rate. The maximum rate to be used in BIPM SIRTI comparisons corresponds to the rate where the trend observed reaches a bias of 0.05 %, indicated by the vertical arrow. A maximum count rate for the BIPM SIRTI of 20 000 s⁻¹ has been deduced. Note: ^{99m}Tc can also be used if negligible ⁹⁹Mo breakthrough.



Reference:

[1] 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.

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ANNEXE 2



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Measurement with the BIPM SIRTI

Introduction

This document is intended for the measurement at the BIPM or at a NMI in the framework of the BIPM.RI(II)-K4 comparisons. Unless mentioned explicitly, the following guidance is applicable to the radionuclides (RN) ^{99m}Tc, ¹⁸F, ⁶⁴Cu, ¹¹C, ¹²³I, ¹⁵³Sm. For other radionuclides, some parts of this instruction might need modification.

The objective is to measure the count rate above a given threshold, produced by a standardized RN solution in a SIR ampoule, relative to the count rate produced by the reference Nb source (see the BIPM.RI(II)-K4 protocol on the <u>KCDB</u>). The Nb reference source contains ⁹⁴Nb for monitoring the SIRTI long-term stability and ^{93m}Nb for setting the SIRTI low-energy threshold.

At reception of the equipment, the SIRTI should be stored at a temperature similar to the one of the laboratory where the comparison will take place. After setting up or switching on the detector, the user should wait for a stabilization period of minimum 1.5 day. The measurements are carried out with the Labview interface "SIRTI NI USB SCALERS".

All the measurement conditions and modules/detector used in the setup are recorded in the logbook where the name of the data files is also noted. Any further changes will also be recorded.

1. Check of the stability

Before the start of the comparison measurements, the stability of the system should be verified in the environmental conditions at the NMI, by checking the threshold position and repeating ⁹⁴Nb, background, temperature and clock measurements for one day:

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- changes in the **threshold** position up to 20 channels (over 4096) in 24 h are acceptable¹. If the situation is worse, the threshold will need to be adjusted more frequently during the RN measurements. In the case of large and fast changes of the threshold, the integrity of the detector or the analogue electronics should be questioned;

- the spread of the ⁹⁴**Nb measurement results** (preceded each time by a threshold adjustment) should be less than 1×10^{-3} in relative terms. If the situation is worse, the source of instability should be sought. If the problem is not solved, the comparison uncertainty should be increased accordingly;

- fluctuations in the **background** count rate of up to 5 s⁻¹ in 24 h are acceptable. If the situation is worse, the source of instability should be sought. If the problem is not solved, the background count rate should be measured with an increasing frequency as the RN count rate decreases, but not more often than every 4 h.

- fluctuations in the measured 1 MHz **live-time clock** frequency due to temperature changes are generally observed (1 to 3 Hz per °C). Larger fluctuations up to 50 Hz in 24 h can be due to the clock or the scalers but would not affect the measurement results. If the situation is worse, the integrity of the clock or the scalers should be questioned.

If some component of the electronics fails, a backup component is available in the equipment sent to the NMI or could be dispatched from the BIPM to the NMI by express delivery.

2. Measurement of the clock frequency

The 1 MHz clock frequency should be measured **once per day**.

Typical results are between 0.999 97 MHz and 1.000 03 MHz, with standard deviations lower than 0.2 Hz.

¹ 3 channels in 6 h for the ¹⁸F, ⁶⁴Cu and ¹¹C comparisons

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3. Setting of the positive threshold of the MTR2

It is essential to check the threshold position <u>before each ⁹⁴Nb measurement and</u> regularly during the RN measurements.

- Put the Nb source in the detector well in a plastic holder in order to measure the ^{93m}Nb x-ray peak;

- set the positive threshold of the MTR2 at the minimum and visualize the x-ray peak at the MCA (minimum 10 000 counts at the peak). Note the channel number C_P of the position of the peak (around channel 100 typically).

- check that the vertical scale is linear; raise the positive MTR2 threshold until the MCA spectrum is cut at the peak position C_P , i.e. the position of the half height of the cut spectrum is at channel C_P (see figure 1).





Figure 1: the position of the x-ray peak (dotted line) had been identified at channel C_p = 105. The spectrum shows the remaining x-ray peak when the threshold has been raised to channel 105.

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4. ⁹⁴Nb measurement (integral counting above the threshold)

Each ⁹⁴Nb measurement should be preceded by an adjustment of the threshold position (see section 3). During a comparison, the ⁹⁴Nb measurements are carried out with the brass absorber **once per day**. They consist of a series of 10 measurements of 700 s using LabView. The brass absorber is used to remove the ^{93m}Nb x-rays from the stability measurements.

The mean ⁹⁴Nb count rate (corrected for live-time and background) since the setup of the SIRTI is 8492.6(21) s⁻¹ for Nb source number 1 and 7632.2(19) s⁻¹ for Nb source number 3, at reference date 1 March 2007. The half-life value used for the decay correction is $20.3(1.6) \times 10^3$ a [Nudat2.5].

5. Background measurement

At the beginning of a comparison, the background should be measured for several hours to identify possible fluctuations or trends.

Note: changes in the threshold position of a few channels has a negligible effect on the background count rate

Background measurements should be carried out before and after each Nb measurement and twice per day during the RN measurements, or more often if instabilities are identified (see section 1).

A background measurement consists of a series of ten measurements of 200 s each, using LabView. The background count rate observed at the BIPM is about 80 s⁻¹.

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6. RN measurement

The RN series of measurements should be preceded by clock, ⁹⁴Nb and background measurements.

It is **recommended**:

- not to put sources with activity higher than 300 kBq in the detector.
- to keep the ampoule always vertical to avoid the formation of drops and therefore use tongs to remove the ampoule from the well or from its lead pot.
- to check regularly the connection with the time server (e.g. NetTime software).

Check whether **drops of solution** remain in the neck or the top of the ampoule. If relevant try to remove them manually or, if necessary, by centrifuging 5 min. at 3000 r/min. If the drops cannot be removed, this may affect the comparison result depending of the size and position of the drops. Describe the drops in the logbook and take a picture of the ampoule.

At the start of the count-rate measurements, RN energy spectra are measured using the MCA and saved for the record. Typically, the relative FWHM of the ^{99m}Tc peak is \leq 9.0(1) % at high count rate (\leq 6.9(1) % at 511 keV). The ^{99m}Tc peak position should be monitored regularly. If it changes by more than 20 channels the measurement should be interrupted¹ and the threshold reset using the Nb source. For the β^+ emitters measurements, monitoring the 511 keV peak position is not helpful because it suffers a high drift versus the count rate. The threshold position should be checked each 5 hours if possible: if it changes by more than 3 channels, this should be taken into account in the uncertainty of the comparison result.

Due to the decay during measurement and its effect on the live-time correction, the **measurement duration** should not exceed given values dependent on the count rate in order to limit the relative effect to 1×10^{-4} (3×10^{-4} in the case of ¹¹C), as given in the Table below [Chauvenet, 1990]:

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Count rate corrected	Maximum measurement duration / s				
for life-time / s⁻¹	Tc-99m	F-18	Cu-64	C-11	
100 000	600	150	1300	60	
75 000	700	200	1500	70	
50 000	850	250	1800	80	
25 000	1250	350	2600	120	
20 000	1400	400	2900	130	
15 000	1600	500	3400	150	
10 000	2000	600	4200	180	
7 500	2300	650	4900	220	
5 000	2800	850	6000	280	
2 500	4000	1200	8500	400	

These values are compatible with the document of R. Fitzgerald, 2009 and were deduced using half lives from *Monographie* 5, and an extended dead time of 30 μ s.

To start a comparison measurement, the SIR-type ampoule is placed in the brass or PVC absorber. The PVC absorber has a cap in order to better define the position of the β^+ annihilation. It is thus used for β^+ emitters as well as for high-energy β^- emitters to minimize bremsstrhalung. The initial RN activity should be such that the observed count rate is not higher than ~20 000 s⁻¹ nor lower than ~10 000 s⁻¹ (see comparison protocol).

The measurements are carried out using LabView. Consecutive measurements are repeated in order to follow the RN decay for three half lives if possible. Measurements should be stopped and data saved at least each four hours (except during the night). The RN ampoule should be taken out from the well at least once during the whole series of measurements. In case of instabilities, background measurements are repeated more often (see section 1) and the threshold is re-adjusted during the ¹⁸F, ⁶⁴Cu and ¹¹C measurements.

When the contribution of an impurity is significant (i.e. significant decreasing trend in the comparison results) or when the count rate becomes lower than ca 2000 s⁻¹, the RN measurements are stopped. Background measurements are carried out, the threshold position is adjusted and a ⁹⁴Nb measurement is carried out followed by final clock frequency and background measurements.

Make a backup copy of the measurement files on a external drive or USB stick, and also on a server either by VPN or by e-mail.

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7. Typical comparison plan (for stable conditions)

Task	Duration	Output	Time
Setup of the system	half a day		day 1
Check of the stability of	one day	Nb energy spectrum	day 2
the SIRTI		series of ⁹⁴ Nb counts and threshold	
		channel numbers	
		series of background counts	
		series of clock counts	
Clock measurement	1000 s	clock counts	day 3
Background	2000 s	1 series of background counts	
measurement			
Set the MTR2 threshold	10 min	threshold channel number	
⁹⁴ Nb measurement	7000 s	⁹⁴ Nb counts	
Background	4000 s	2 series of background counts	
measurements			
^{99m} Tc measurements	until the	^{99m} Tc energy spectra, 140 keV peak	
	evening	channel numbers	
		contiguous series of ^{99m} Tc counts	
Clock and background	3000 s	clock counts	
measurement with		1 series of background counts	
ampoule			
^{99m} Tc measurements	over night	^{99m} Tc energy spectrum, 140 keV peak	
		channel number	
		contiguous series of ^{99m} Tc counts	
		preliminary comparison result	
Clock measurement	1000 s	clock counts	day 4
Background	2000 s	1 series of background counts	
measurement *			
Set the MTR2 threshold	10 min	threshold channel number	
^{99m} Tc measurements	until ⁹⁹ Mo is	^{99m} Tc energy spectrum, 140 keV peak	
	significant or	channel numbers	
	low count rate	contiguous series of ^{99m} Tc counts	
Background	4000 s	2 series of background counts	
measurements			
Set the MTR2 threshold	10 min	threshold channel number	
⁹⁴ Nb measurement	7000 s	⁹⁴ Nb counts	
Background	2000 s	1 series of background counts	
measurement with Nb			
holder			
Clock measurement	1000 s	clock counts	

* before or after the ^{99m}Tc measurements depending on the remaining ^{99m}Tc activity

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8. Stability review

Measurements of the Nb reference sources number 1 and 3 are carried out during the comparisons and at the BIPM before and after each comparison in order to monitor the stability of the SIRTI.

Annual review of long-term trends

- Plot all the measured ⁹⁴Nb count rates corrected for decay and background as a function of time, since the start of the SIRTI comparison program.
- If recent results show discrepancy from the mean of the previous results by more than 3 standard deviations, repeat the measurement and look for a step in the SIRTI response or a long-term trend. If a significant step or long-term trend is identified, the SIRTI link to the SIR should be re-measured.

9. Traceability to SI

Once every two years or when the live-time clock frequency shows a significant change, the time base of the NI scalers should be verified by measuring a frequency standard. This is carried out at the BIPM using the 10 MHz frequency signal coming from the Time department. The frequency of the signal is first divided to 100 kHz using a frequency shaping module. The frequency is then counted in both channels A and B of the scalers for 20 measurements of 100 seconds. The number of counts should be between 9 999 500 and 10 000 500. If not, a correction factor to be applied on all countings is evaluated.

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Chauvenet B., 1990, Utilisation du temps actif pour la mesure d'un radionucléide de courte période avec temps mort cumulative, *Rapport interne LPRI/90/101/Novembre*, CEA Saclay.

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