March 12, 2007

**Reference BIPM/RI-SIR-F-01** 

# International Reference System for activity measurements of gamma-ray emitting nuclides (SIR) (35th circular letter)

Dear Colleague,

During 2006, the BIPM received 30 ampoules from 13 laboratories (i.e. one ampoule containing <sup>65</sup>Zn from the BARC, one ampoule containing <sup>22</sup>Na from the CIEMAT, two ampoules from the ČMI-IIR (one containing <sup>56</sup>Co and one <sup>131</sup>I), two ampoules from the IFIN-HH (one containing <sup>60</sup>Co and one <sup>134</sup>Cs), one ampoule containing <sup>166</sup>Ho<sup>m</sup> from the IRA-Metas, four ampoules from the IRMM all containing <sup>237</sup>Np (for a link to a EUROMET comparison and to obtain a KCRV value for this radionuclide), nine ampoules from the LNE-LNHB (one ampoule containing <sup>51</sup>Cr, two <sup>54</sup>Mn, one <sup>75</sup>Se, four <sup>85</sup>Kr and one <sup>111</sup>In), one ampoule containing <sup>51</sup>Cr from the LNMRI/IRD, two ampoules from the NMIJ (one containing <sup>57</sup>Co and one <sup>133</sup>Ba), three ampoules from the NPL (one containing <sup>54</sup>Mn and two <sup>201</sup>Tl), one ampoule containing <sup>22</sup>Na and one <sup>186</sup>Re) and one ampoule containing <sup>241</sup>Am from the VNIIM (for a link to a COOMET comparison).

All the submissions had been made to generate equivalence values in the key comparisons. Counting only the newly registered measurements for 2006, the cumulative number of ampoules measured since the beginning of the SIR, in 1976, is now 894, corresponding to a total of 655 independent results for 63 different radionuclides.

The low activity in the ampoule of <sup>241</sup>Am produced a very small ionization current in the SIR chambers so it was decided to measure it several times under different experimental conditions, both with the original SIR electronic chain and the newly developed chain; the final result has been taken as the weighted mean of the individual measurements. In addition a correction factor, deduced from the work of A. Rytz, has been applied to take into account the high density of the solution of <sup>241</sup>Am. The definitive result is now available and shows agreement with the other entries for this radionuclide. This submission was made to link the results of a recent COOMET comparison.

The results for <sup>237</sup>Np, which will constitute a new SIR entry, are pending the formal submission of the activity determination. Once this is received, the Draft A report can be issued which will also contain the links for the EUROMET comparison.

In the frame of the preliminary study of the appropriate characteristics of the krypton to be distributed to the participants in the planned CCRI(II) comparison, measurements of additional ampoules of <sup>85</sup>Kr prepared by the LNE-LNHB have been carried out successfully. The definitive



value for the activity of the gas is not yet available and may not be ready before the comparison of activity measurements is finished sometime towards the end of 2007.

The NPL identified a preparation problem with the first ampoule of <sup>201</sup>Tl they submitted and this was withdrawn. We have just received their activity determination for the second submission that has been measured in the SIR, so the Draft A will be submitted shortly.

Also during 2006, the BIPM Quality System has been extended to include the SIR. This has involved writing the procedures and twenty-three technical instructions describing the operation of the SIR in detail. The first external audit was carried out by an expert from the NIST at the end of 2006 following an internal audit by the BIPM Quality Manager. We have responded to all the comments made to ensure a successful outcome in conformity with the ISO 17025 criteria.

The year 2006 has also seen the first full twelve months of running the new data acquisition chain for the SIR in parallel with the present system. The results show agreement to about  $10^{-4}$ . The systems will continue in parallel until the end of 2007 at which time the new system will be used exclusively as long as the testing continues to indicate the required stability. The new system has been fully documented ready for this implementation.

The present SIR has been carefully maintained during the year but has required a replacement component. The fault was identified during measurements of very low activities, the cause was traced, the component replaced and tests were made to ensure the integrity of the system.

All of these additional actions have inevitably led to some delays in issuing reports of the SIR results. As a consequence the results for only 4 new submissions have been registered in the SIR Master file, for 4 different radionuclides <sup>22</sup>Na (CIEMAT), <sup>131</sup>I, <sup>133</sup>Ba, <sup>241</sup>Am. However, full comparison reports were published for eight earlier SIR submissions made in 2005.

In a few weeks you will receive, under separate cover, a set of updated SIR registration tables for the above listed radionuclides. Each result prior to 2005 has been published in the *Metrologia Technical Supplements* and 60 % of those submitted in 2005 have also been published. The eight outstanding submissions of 2005 are awaiting the evaluations of the 2006 submissions for the same radionuclides. We are presently awaiting activity results for 30 % of recent submissions. The other reports are in progress and we plan to resume regular publication activities shortly. Please note that the SIR results that are more than 20 years old are coloured black in the KCDB (a total of 134 results) and that at the end of 2007, twenty-four of these results that are more than 30 years old will be deleted from the KCDB.

Enclosed herewith are the forms relating to the 2007 programme. Would you be kind enough to fill in the **"2007 Questionnaire"** and return it to the BIPM preferably by 31 March 2007. At your convenience you may answer by fax or e-mail. The NBS-type ampoules or special NIST-type gas ampoules will then be supplied to you on request according to the number of samples you plan to submit; otherwise we shall assume you already have sufficient ampoules.

For ease of communication, it is now possible to download the reporting forms with the uncertainty budget forms from the BIPM web site at the address

<u>http://www.bipm.org/en/scientific/ionizing/radionuclides/sir/</u> where links to the SIR reporting forms for liquid samples and for gas samples are highlighted in blue. Please feel free to alter the form at your convenience. Each sample sent to the BIPM should be accompanied by a form completed with the required information. Please use one form per ampoule. We would also like to remind you that **no samples should be sent during the month of August**. In the case of radionuclides with short half-lives, please send the samples to arrive at the beginning of the week. It would also be preferable if you could contact the BIPM well in advance to enable the best possible synchronization to minimize the half-life uncertainty.

As usual, every new result will first be communicated to the supplier of the sample. According to the policy for the acceptance of results, resulting from the application of the CIPM Mutual Recognition Arrangement and from the use of the SIR for determining the equivalence between

laboratories, a **detailed uncertainty budget** is **required** with the submitted results. A proposed reporting form is included in the material attached to this letter. Please use it but feel free to add any other items you may find appropriate. Do not forget to indicate the evaluation type A or B of the different components of the uncertainty. It would be most helpful when describing your measurement methods, to use the acronyms listed in the attached sheet in addition to the usual wording. Finally, if you use several measurement methods, please use a separate form for each method and indicate the value you want to be used for the KCDB. This may be the value from one method, or some combination. A withdrawal of a result during a period of one month following the receipt of the result is now only possible if the measurements have been made in the frame of a pilot study. Results from pilot studies will not be used for equivalence purposes nor for deriving the key comparison reference value except with the express approval of the CCRI(II). Do not forget to state your choice between a pilot study or an equivalence value before sending your results.

Please note that following a decision at the CCRI(II) meeting in May 2005, only primary standardized solutions will be accepted for the KCRV in future, with the exception of radioactive gases. Of course, ionization chamber measurements traceable to primary measurements will still be accepted in the SIR and published in the KCDB but the result will not be used anymore for the calculation of the KCRV.

According to a previous decision of the CCRI Section II, the number of withdrawn results is indicated for each radionuclide without mentioning the value obtained or the laboratory involved. Since the beginning of the SIR a total of 40 results have been withdrawn, which represents 6.1 % of the number of registered results.

May I remind you that as part of our newly implemented Quality System and to facilitate Customs procedures, a requirement has been put in place by the BIPM administration concerning the shipment from third parties to the BIPM of items which are submitted for international comparisons? With regard to sending ampoules of radioactive material to be measured in the SIR, the procedure to be followed by each laboratory is described in the document labelled BIPM/ADM-DOU-P-03 and attached to this mail. Please follow the instructions given and, for each radionuclide to be sent to the BIPM, fill in the document BIPM/ADM-DOU/F-12 also attached to this mail, **at least two weeks in advance** of the planned transport and fax it to **Mrs Daniela Etter** (fax number : +33 1 45 07 70 99). The ampoules sent for the SIR will not normally be sent back by the BIPM to the participating laboratory so that the paragraph "Instructions for return" is usually not relevant and can be neglected. This slight increase in your administrative workload should smooth the passage of the samples through Customs and enable us to collect the samples from the airport without undue delay. I thank you in advance for your comprehension.

Finally, I would like to thank all the laboratories who have contributed in keeping the system alive last year by submitting interesting nuclides to the SIR. I look forward to receiving your replies concerning the samples you intend to send during 2007 and I hope to have in 2007, as in previous years, a fruitful and interactive cooperation with all of you.

Yours sincerely,

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G. Ratel

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## **International Reference System**

### for activity measurements of gamma-ray emitting nuclides (SIR)

## 2007 QUESTIONNAIRE BIPM/SIR/F-02

Our laboratory intends to take part in the International Reference System for activity measurements of gamma-ray emitting nuclides. Therefore, we shall send to the Bureau International des Poids et Mesures samples of solutions prepared and standardized at our laboratory.

For 2007 we plan to submit samples of the following radionuclides:

Number of samples		Radionuclide		Approximate date
	-			
	-			
	-			
I have sufficient amp	poules for th	ese submissions, or (delete as nece	essary)	
Please supply (for gas).	NBS-ty	pe ampoules (for solutions) and/or	[	NIST-type ampoules
Name and address o	f laboratory	Name o	of person re	sponsible

Date

Signature

Please return the completed form to Guy Ratel, BIPM by March 31, 2007

February 27, 2007

**International Reference System** 

## for activity measurements of gamma-ray emitting nuclides (SIR) (in solution)

Participating laboratory :	
Radionuclide (main contribution)	$T_{1/2}$ :
Chemical composition of the solution :	
Solvent :	and its concentration : mol per dm <sup>3</sup> of solution
Carrier :	and its concentration : $\mu$ g per g of solution
Density of the solution	: g cm <sup>-3</sup>
Ampoule number : Mass of	of solution (corrected for buoyancy) :g
Activity per gram of solution (main radi	ionuclide) :Bq g <sup>-1</sup>
Reference date :year	monthdayh UTC*
Measurement date :	
	ncertainties). Please attach also a detailed uncertainty budget : blying statistical methods) :Bq $g^{-1}$ ;%
Number of deg	grees of freedom :
Category B (evaluated by c	other means) :Bq $g^{-1}$ ;%
Method(s) of measurement :	
For relative methods, please indicate the experimental setup :	e primary methods and the standards used to calibrate your
the date of calibration :	ement :
Nuclide	Ratio of activity of impurity to activity
	of main radionuclide at reference date
Radionuclide impurities :	$(\_\_\_\_] \%; u = (\_\_\_] \%$
	$(\_\_\_\_] \%; u = (\_\_\_] \%$
Remarks :	
	of a pilot study or to generate an equivalence value
Date :	Name of person responsible :
	Name of person(s) who carried out the measurements :

Internationa	l Reference System
for activity measurements of ga	mma-ray emitting nuclides (SIR) (gas)
Participating laboratory :	
Radionuclide (main contribution)	$T_{1/2}$ :
Chemical composition of the gas :	
Amount of radioactive gas : mol.	Amount of inactive gas (if any) : mol
Gas pressure at 20 °C in the ampoule : H	Pa. Pressure value was measured or calculated
Volume of gas transferred in the	e ampoule at 20 °C : cm <sup>3</sup>
Ampoule nu	mber :
Activity of the gas (main radionuclide) :	Bq
Reference date :year month _	dayh UTC*
Measurement date :	
	). Please attach also a detailed uncertainty budget : tical methods) :Bq ;% edom :
Category B (evaluated by other means	s):Bq;%
Method(s) of measurement :	
	nd the standards used to calibrate your experimental
Nuclide	Ratio of activity of impurity to activity of main radionuclide at reference date
Radionuclide impurities :	$(\_\_\_\_)$ %; $u = (\_\_\_]$ %
	$(\_\_\_\_)$ %; $u = (\_\_\_]$ %
Remarks :	
This sample has been sent in the frame of a pilot st	udy or to generate an equivalence value
Date : Name of	f person responsible :
	f person(s) who carried out surements :

\*UTC = coordinated universal time February 27, 2007

## **Detailed Uncertainty Budget**

Laboratory :;	Radionuclide :		; Ampoule number	:	•
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# Uncertainty components\*, in % of the activity concentration, due to

	Remarks	Evaluation
		type (A or B)
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 $(T_{1/2} = \_\_\_];$		
<i>u</i> =)	 	
self absorption	 	
extrapolation of efficiency curve	 	
other effects (if relevant) (explain)	 	
<b>combined uncertainty</b> (as quadratic sum of all uncertainty components)	 	

<sup>\*</sup> 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).

### **IMPORTANT ADVICE FOR PARTICIPANTS IN THE SIR**

### **1. Filling of ampoules**

For solutions only:

Use exclusively NBS-type ampoules supplied by the BIPM.

Filling: mass = 3.6 g;  $u_m = 0.2 \text{ g}$ , i.e. height = 20 mm;  $u_h = 1 \text{ mm}$ , for  $\rho = 1 \text{ g/cm}^3$ .

For gas only:

Use exclusively NIST-type ampoules supplied by the BIPM.

#### 2. Labelling of ampoules

It is preferable for the measurements if identification of ampoules is only by permanent marker directly onto the glass. If a label is used, please ensure that it is placed above the filling height or does not completely encircle the ampoule. <u>Please ensure that the activity (even if approximate) of each radionuclide is clearly indicated on the transport label.</u>

#### 3. Importation requirements (for states outside the European Union)

Please send a pro forma invoice for the indicative sum of  $160 \in$ , **4 weeks** in advance so that we can obtain the import licence, and indicate, if possible at the same time, the airport or railway station of arrival, or that delivery is direct to the BIPM.

#### 4. Transportation requirements

It is essential that the customs procedures are followed correctly for the safe and timely arrival of your ampoules. Please complete and fax back the form ADM-DOU-F12 attached to this mail **at least 2 weeks before** you actually send the ampoule (fax: + 33 1 45 07 70 99 Mrs D. Etter).

Please state the following in the section of the Air waybill called Handling Information: "Please contact consignee on arrival for Customs clearance

and shipping instructions (+33 1 45 07 70 86 - Mr G. RATEL)".

Please fully address the package to:

BIPM (à l'attention de Mr G. Ratel; tel 01 45 07 70 86) Pavillon de Breteuil 12, bis Grande Rue 92310 SEVRES France

Thank you.

February 27, 2007 Reference BIPM/RI-SIR-F-04 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	Nal(TI)	NA
		Ge(HP)	GH
		Ge(Li)	GL
		Si(Li)	SL
		CsI(TI)	CS
		ionization chamber	IC
		grid ionization chamber	GC
		bolometer	BO
		calorimeter	CA
		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	СО
bremsstrahlung	BS	anti-coincidence	AC
gamma rays	GR	coincidence counting with efficiency tracing	СТ
X - rays	XR	anti-coincidence counting with efficiency tracing	AT
photons (x + γ)	PH	triple-to-double coincidence ratio counting	TD
photons + electrons	PE	selective sampling	SS
alpha - particle	AP	high efficiency	HE
mixture of various radiations	MX	digital coincidence counting	DC

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