

**PROTOCOL FOR A SUBSEQUENT COMPARISON OF MEASUREMENTS OF  
NEUTRON SOURCE EMISSION RATE CCRI(III)-K9.AmBe.1**

**INTRODUCTION**

It is the aim of Section III to compare the realisation of standards of all relevant neutron quantities over a ten year cycle. Each comparison exercise should take less than four years from initial planning to publication of report in open literature. The previous comparison of measurements of source emission rate started in 1999 and took six years to complete. Difficulties in transporting radionuclide sources between countries and some participants requiring the source for a second measurement were the main factors in the time taken for the previous exercise.

**The aim of this comparison is to compare primary measurements of the neutron emission rate of an  $^{241}\text{Am-Be}(\alpha,n)$  radionuclide source for laboratories who were outliers in the previous comparison (K9.AmBe) or who were unable to participate.**

The anisotropy factor may also be determined if a laboratory wishes to do so. It is particularly important if the laboratory requires support for a CMC entry concerning measurements of the anisotropy of neutron sources.

The source will be circulated to participating laboratories for measurement, with the NPL as the pilot institute. The results obtained by each will be sent to an independent Coordinator (José Maria Los Arcos [jm.losarcos@bipm.org](mailto:jm.losarcos@bipm.org)) for initial analysis. Once all the participants have submitted their results, the Coordinator will pass on the results to the pilot institute. The pilot institute will calculate reference values to be used for the assessment of equivalence.

The pilot institute will be responsible for writing the report of the comparison exercise for circulation to all participants and presentation to Section III. The report will pass through the Draft A, Draft B and Final Report stages as defined by [CIPM MRA-D-05](#). Following approval by members of Section III, a paper will be published in *Metrologia*.

**PARTICIPATING LABORATORIES**

The laboratories who have so far indicated an interest in participating in the CCRI comparison are listed in Appendix 1.

**TIMESCALE**

Participants must plan to take no more than two months to complete their part of the exercise (including measurements, receipt and despatch of source). Allowing additional time for problems with transportation means that no more than four laboratories will participate per year. The aim is to complete the measurement phase within two years, but this will depend on the number of participating laboratories. At the time of writing only five laboratories have expressed an active interest in participating in this exercise, including the NPL (UK) as a link to the K9.AmBe key comparison.

## **TIMETABLE FOR MEASUREMENTS**

The comparison started in 2012 with NIM who provided the source. At the time of writing, the source is at NPL, following long delays in getting approval for the exchange of the source between NPL and LNHB. The source needs to return to China for the special form certificate to be amended before it can be received by LNHB.

A provisional timetable is given in Appendix 1.

## **NEUTRON SOURCE**

Please refer to Appendix 2 for details of the  $^{241}\text{Am-Be}(\alpha,n)$  radionuclide source and transportation container.

## **TRANSPORTATION ARRANGEMENTS**

Please refer to Appendix 3 for details of the transportation arrangements that must be made. Participants are strongly advised to contact the laboratories from whom they will receive the source and also the laboratories who are to receive the source next as soon as possible. Otherwise there could be delays in the transportation of the source between countries.

## **MEASUREMENTS**

Please refer to Appendix 4 for details to be supplied to the Coordinator.

## **UNCERTAINTIES**

Please refer to Appendix 5 for details to be supplied to the Coordinator. Those shown are specific to the manganese bath technique.

If other techniques are used, the values of the appropriate uncertainties must be given.

The following laboratories have agreed to take part

**China Institute of Atomic Energy (CIAE)**

P O Box 275 (20)  
102 413 Beijing  
People's Republic of China  
Contact: Zhiqiang Wang ([wangzq@iris.ciae.ac.cn](mailto:wangzq@iris.ciae.ac.cn))

**ENEA**

Istituto Nazionale di Metrologia delle Radiazioni Ionizzanti  
ENEA Centro Ricerche Casaccia  
Via Anguillarese, 301  
00123 Santa Maria di Galeria (Roma)  
Italy  
Contact: Lina Quintieri ([lina.quintieri@enea.it](mailto:lina.quintieri@enea.it))

**Laboratoire National Henri Becquerel (LNHB)**

F-91191 Gif sur Yvette cedex  
France  
Contact: Philippe Cassette ([philippe.cassette@cea.fr](mailto:philippe.cassette@cea.fr))

**National Institute of Metrology (NIM)**

No.18, Bei-San-Huan Dong Str.,  
Beijing 100013  
China  
Contact: Zhang Hui ([zhanghui@nim.ac.cn](mailto:zhanghui@nim.ac.cn))

**National Physical Laboratory (NPL)**

Teddington  
Middlesex TW11 0LW  
United Kingdom  
Contact: Neil Roberts ([neil.roberts@npl.co.uk](mailto:neil.roberts@npl.co.uk))

## COMPARISON of NEUTRON SOURCE EMISSION RATE MEASUREMENTS

Participants	Preferred date	Received source	To receive source
NIM			
NPL		July 2012	
LNE-LNHB	September 2014?		
ENEA	November 2014?		
CIAE	February 2015?		

## APPENDIX 2 NEUTRON SOURCE

A2.1  $^{241}\text{Am}$ -Be( $\alpha$ ,n) radionuclide source

### HTA Co., Ltd Americium - Beryllium Neutron Source Specification

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**Product Code:** AB-3001

**Nuclide:** Am-241

**Half-life:** 433 years

**Target:** BeO

**Ingredients:** AmO<sub>2</sub> 1.3g

BeO 6.1g

**Neutron emission rate:**  $6.1 \times 10^6 \text{ n/s}$

**Measuring date:** 01/01/2011

**Source structure:**

Shell material: 304 stainless steel

Shell size:  $\Phi 24 \times 24 \text{ mm}$

Shell thickness: 1.25mm

Shell weight: 22.98g

Inner shell material: 304 stainless steel

Inner shell size:  $\Phi 21.5 \times 21.5 \text{ mm}$

Inner shell thickness: 1mm

Inner shell weight: 14g

Source core (active element) size:  $\Phi 19 \times 18 \text{ mm}$

Source core preparation: ceramic technology preparation. Mixing AmO<sub>2</sub> with BeO, and after 6 hours of 1600°C~1700°C high temperature burning, the ceramic source core with high density, high hardness, high stability of is made.

Source core weight: 7.4g

Aluminum pad size:  $\Phi 19 \times 1 \text{ mm}$

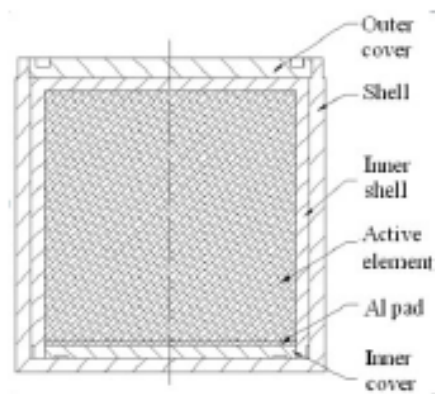
Aluminum pad weight: 0.62g

Sealed: Double stainless steel with TIG weld

**Surface contamination detection:** GB15849-1995 smear test, less than 185Bq.

**Leakage detection:** GB15849-1995 hot liquid immersion test, no leakage.

**Terms of use:** Open the radioactive source during the use is strictly prohibited, In case of accident, please contact with the production units, 010-69357819



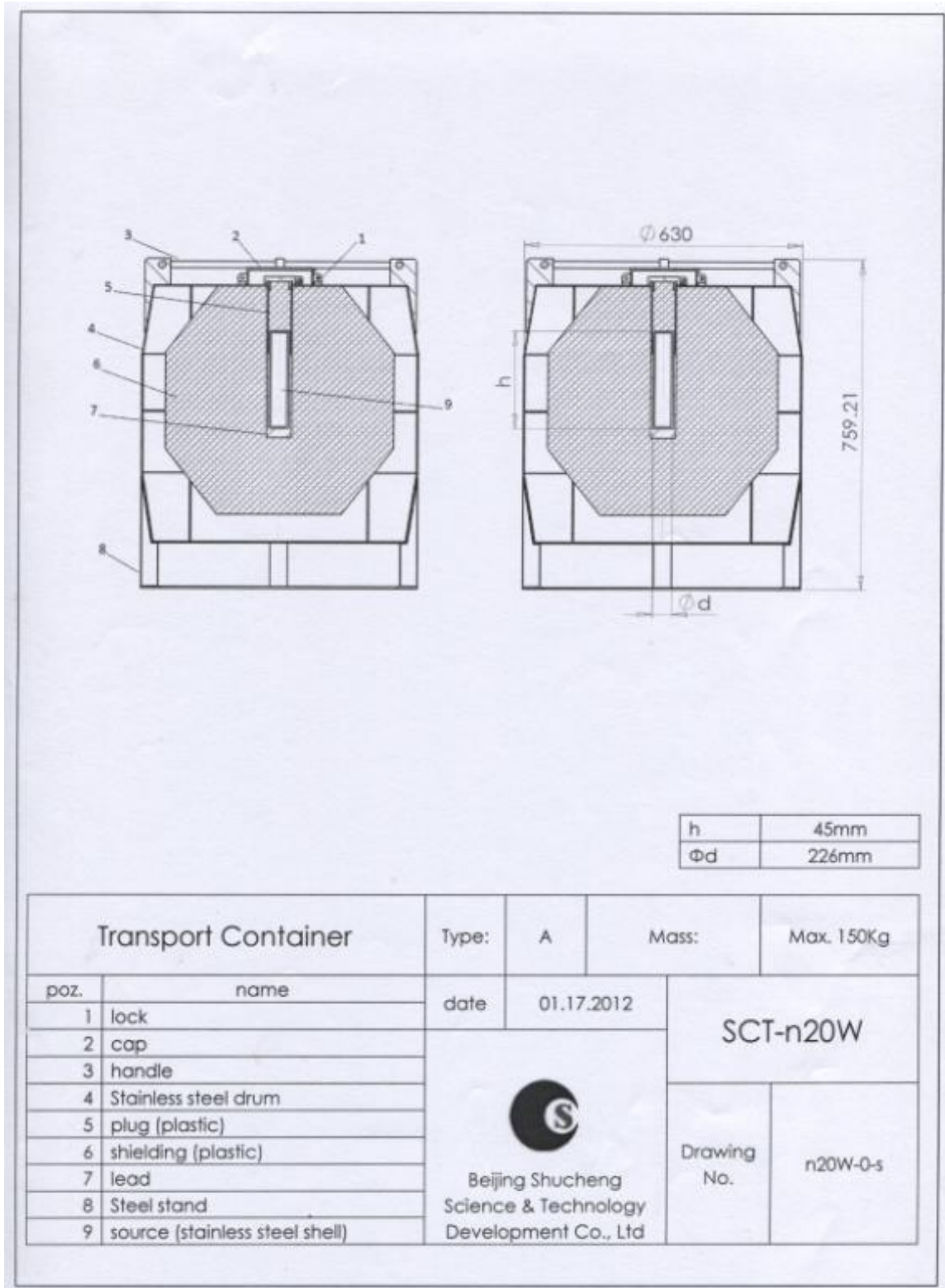
HTA Co. Ltd  
Quality Department  
01/01/2011

Manufacturer: HTA Co. Ltd.  
Serial number AB-3001  
Special form cert: CIAE/0629/S-96 Am-241/Be

The radioactive material is a compacted mixture of americium oxide with beryllium oxide doubly-encapsulated in welded stainless steel. The density of the mixture can be variable.

Length of capsule: 24 mm  
Diameter of capsule: 24 mm  
Nominal <sup>241</sup>Am activity: 142.9 GBq  
Approximate emission rate  $6.1 \times 10^6 \text{ s}^{-1}$  (neutron)  
Half life:  $1.5785 \times 10^5$  day  
Owner: NIM, No.18, Bei-San-Huan Dong Str., Beijing 100013, China

## A2.2 TRANSPORTATION CONTAINER



Model number: SCT-n20W

Type A, TI 0.9, II Yellow, UN3332

National Nuclear Safety Administration of PRC Registration number: CN/003/A-06(NNSA)

Manufacturer: Beijing Shucheng Science & Technology Development Co. Ltd.

Outer container

Height: 76 cm

Diameter: 63 cm

Overall Weight: 144 kg

Owner: NIM, No.18, Bei-San-Huan Dong Str., Beijing 100013, China



## APPENDIX 3                      TRANSPORTATION ARRANGEMENTS

A3.1. Each participant will be informed of the next laboratory that is to receive the source by the organiser.

A3.2 Each participant shall be responsible for arranging for transportation and insurance of the source and container to the next participant in agreement with the next participant.

A3.3 Each participant shall pay for -

- transport within own country
- customs charges within own country
- insurance charges within own country
- costs of any damage occurring within own country
- transport to the laboratory of the next participant.
- a share of the transport and customs charges incurred by NPL as the link laboratory

A3.4 Receipt

The previous participant (i.e. the sender) shall be informed of the safe arrival of the source.

The National Physical Laboratory shall also be informed.

A3.5 Handling

Care must be taken to protect the source from mechanical shock, corrosive chemicals, etc., at all times while it is out of the transport container and during measurements.

A3.5 Despatch

Arrangements shall be made with the next participant for the receipt of the source. The necessary authorities of the country and laboratory of the next participant shall be informed of the expected time of arrival of the source and given all relevant information.

The source (in its container) shall be sent by air (unless road or rail transport can be used without significantly delaying the exercise), using an appropriate courier company authorised for the transport of radioactive sources.

The shipment shall be made under the international regulations governing the transport of radioactive sources.

## **APPENDIX 4 EXPERIMENTAL DETAILS AND DATA**

The following information should be included in the report to the Coordinator.

### **A4.1 Neutron source emission rate measurement system**

- a) Data relating to stability (e.g. repeat measurements of the same source over many years, pre-measurement checks of detectors)
- b) Details of the system (e.g. bath size, circulating or static solution, solution concentration and impurity levels, source mounting arrangements, gamma detectors used)

### **A4.2 Neutron emission rate measurement**

- a) Complete account of neutron emission rate determination (e.g. number of measurement runs, length of solution irradiations, counting period)
- b) Traceability to absolute standards (i.e. how is the Mn bath, or other system, calibrated?)

### **A4.3 Analysis of data**

- a) Formulae used
- b) Application of corrections (e.g. deadtime, background, decay of solution in circulating system)

### **A4.4 Numerical values and uncertainties of corrections**

The following apply to the manganese bath technique.

- a) Correction applied for neutron escape (with associated uncertainty)
- b) Correction for neutrons captured by the source materials and its mounting assembly
- c) Correction for neutrons that undergo reactions with hydrogen, oxygen and sulphur in the solution of the detector.

Full details should be given of the methods used to determine these corrections including any computational codes and cross-section libraries used.

### **A4.5 Measured values and uncertainties**

- a) Emission rate at local reference time and associated uncertainty at 95% confidence level  
Reference time used locally.
- b) Emission rate corrected to the reference time for the comparison: 1 January 2012

If an anisotropy measurement is made then the following information should also be provided:

**A4.6 Anisotropy measurement**

- a) Complete account of anisotropy determination

**A4.7 Analysis of data**

- a) Formulae used
- b) Application of corrections

**A4.5 Measured values and uncertainties**

- a) Anisotropy factors in 10 degree steps and associated uncertainties at 95% confidence level

## APPENDIX 5 UNCERTAINTIES FOR MANGANESE SULPHATE BATH METHOD

- 5.1 The estimation, treatment and expression of uncertainties must be based on the *Guide to the Expression of Uncertainty in Measurement*. See JCGM 100:2008 and related documents at: <http://www.bipm.org/en/publications/guides/gum.html>
- 5.2 A complete statement of uncertainties is required for the reported values. This shall include:
- Values of Type A (or random) uncertainties
  - Values of Type B uncertainties
  - Values of combined Types A and B uncertainties
  - Method used to combine components
  - Correlation coefficients
  - Confidence level (normally 95 %)
- 5.3 Type A uncertainties are assumed to have a normal distribution.
- 5.4 Type B uncertainties are assumed to have a rectangular distribution unless it is known that they have a normal distribution.
- 5.5 Details of the principal uncertainty components below must be given, along with those of any other components considered to be significant: -
- 5.5.1 Statistical and random uncertainties associated with the measurement of radioactivity. These are usually quoted at 67 % confidence level with effectively an infinite number of degrees of freedom.
- 5.5.2 Cross section ratios used to determine the fraction of neutrons captured by the  $^{55}\text{Mn}$  in the solution. This is dominated by the uncertainty in the H : Mn capture cross section ratio.
- 5.5.3 Gamma-ray counter efficiency
- 5.5.4 Fast neutron losses due to oxygen and sulphur reactions
- 5.5.5 Neutron capture in the source and source container
- 5.5.6 Leakage of neutrons from the boundary walls of the bath
- 5.5.7 Solution mixing effects
- 5.5.8 Source transfer timing
- 5.5.9 Dead time effects
- 5.5.10 Half-life of source

5.6 Values used for the uncertainty components must be entered in an uncertainty budget similar to that shown below for the manganese bath technique -

Source of uncertainty	Value (±)	Probability distribution	Divisor	$c_i$	$u_i$ (± %)	$v_i$ OR $v_{eff}$
Counting		normal	1			
Cross section ratio		normal	2			
Counter efficiency		normal	1			
Oxygen and sulphur losses		rectangular	$\sqrt{3}$			
Source capture		rectangular	$\sqrt{3}$			
Leakage		rectangular	$\sqrt{3}$			
Mixing		rectangular	$\sqrt{3}$			
Timing		rectangular	$\sqrt{3}$			
Dead-time effects		rectangular	$\sqrt{3}$			
Half life of source		normal	1			
Combined uncertainty		normal				
Expanded uncertainty		normal ( $k=2$ )				

$c_i$  coefficient to convert value into percentage component of emission rate

$u_i$  percentage standard uncertainty

$v_i$  number of degrees of freedom

$v_{eff}$  effective number of degrees of freedom

Components for which  $u_i$  is likely to be less than 0.05% have been omitted.

The probability distributions are suggested - participants may wish to use other types.

## APPENDIX 6 UNCERTAINTIES FOR ANISOTROPY MEASUREMENT

- 6.1 The estimation, treatment and expression of uncertainties must be based on the *Guide to the Expression of Uncertainty in Measurement*. See JCGM 100:2008 and related documents at: <http://www.bipm.org/en/publications/guides/gum.html>
- 6.2 A complete statement of uncertainties is required for the reported values. This shall include:
- Values of Type A (or random) uncertainties
  - Values of Type B uncertainties
  - Values of combined Types A and B uncertainties
  - Method used to combine components
  - Correlation coefficients
  - Confidence level (normally 95 %)
- 6.3 Type A uncertainties are assumed to have a normal distribution.
- 6.4 Type B uncertainties are assumed to have a rectangular distribution unless it is known that they have a normal distribution.
- 6.5 Details of the principal uncertainty components below must be given, along with those of any other components considered to be significant: -
- 6.5.1 Statistical and random uncertainties associated with the measurement of neutron fluence. These are usually quoted at 67 % confidence level with effectively an infinite number of degrees of freedom.
- 6.5.2 Uncertainty in the positioning of the source in whatever holder or mounting assembly is used.
- 6.5.3 Uncertainty in the position of the centre of the active source material.
- 6.5.4 Uncertainty in the position of the detector used to measure the fluence.
- 6.5.5 Uncertainty in the neutron in-scatter correction.
- 6.5.6 Dead time effects

6.6 Values used for the uncertainty components must be entered in an uncertainty budget similar to that shown below -

Source of uncertainty	Value (±)	Probability distribution	Divisor	$c_i$	$u_i$ (± %)	$v_i$ OR $v_{eff}$
Counting		normal	1			
Source position		rectangular	$\sqrt{3}$			
Effective centre of source		rectangular	$\sqrt{3}$			
Detector position		rectangular	$\sqrt{3}$			
In-scatter correction		rectangular	$\sqrt{3}$			
Dead-time effects		rectangular	$\sqrt{3}$			
Combined uncertainty		normal				
Expanded uncertainty		normal ( $k=2$ )				

$c_i$  coefficient to convert value into percentage component of emission rate

$u_i$  percentage standard uncertainty

$v_i$  number of degrees of freedom

$v_{eff}$  effective number of degrees of freedom

Components for which  $u_i$  is likely to be less than 0.05% have been omitted.

The probability distributions are suggested - participants may wish to use other types.