

## International Key Comparison of Neutron Fluence Measurements in Monoenergetic Neutron Fields - CCRI(III)-K11

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### Abstract

To ensure the validity of their national standards, National Metrology Institutes, NMIs, participate regularly in international comparisons. In the area of neutron metrology, Section III of the Consultative Committee for Ionizing Radiation is in charge of the organization of these comparisons. From September 2011 to October 2012, the eleventh key comparison, named CCRI(III)-K11, took place at the AMANDE facility of the LNE-IRSN, in France. Participants from nine NMIs came with their own primary reference instruments, or instruments traceable to primary standards, with the aim of determining the neutron fluence, at 1 m distance from the target in vacuum, per monitor count at four monoenergetic neutron fields: 27 keV, 565 keV, 2.5 MeV and 17 MeV.

The key comparison reference values (KCRV) were evaluated as the weighted mean values of the results provided by seven participants. The uncertainties of each KCRV are between 0.9 % and 1.7 %. The degree of equivalence (DoE), defined as the deviation of the result reported by the laboratories for each energy from the corresponding KCRV, and the associated expanded uncertainty are also reported and discussed.

### 1. Introduction

Monoenergetic neutron fields are amongst the reference neutron fields recommended in the ISO 8529-1 Standard [1]. These fields allow the determination, at certain neutron energies, of instrument response variations with energy (i.e. the response function). Response functions are generally calculated using simulation codes, and a validation at several monoenergetic fields covering the energy range between a few tens of keV up to about 15 MeV is sufficient for most purposes. Monoenergetic fields are produced by bombarding thin targets with ion beams from accelerators and can be produced between a few keV and 20 MeV. A review of their production methods has been published recently [2]. The commonly used reactions are  $^{45}\text{Sc}(p,n)$ ,  $^7\text{Li}(p,n)$ ,  $^3\text{H}(p,n)$ ,  $^2\text{H}(d,n)$  and  $^3\text{H}(d,n)$ , and the ISO 8529-1 Standard recommends a set of “standardised” neutron energies: 2 keV, 24 keV, 144 keV, 250 keV, 565 keV, 1.2 MeV, 2.5 MeV, 2.8 MeV, 5 MeV, 14.8 MeV and 19 MeV.

In order to ensure the validity of their own national standards, National Metrology Institutes, NMIs, regularly participate in international comparisons. In the field of neutron

metrology, Section III of the Consultative Committee for Ionizing Radiation, CCRI, is in charge of the organization of these comparisons. In the past, Section III of the CCRI regularly organised comparisons of neutron fluence measurements in monoenergetic neutron fields recommended by the ISO 8529-1 Standard [3]. The last comparison of neutron fluence measurements in monoenergetic neutron fields (CCRI(III)-K10) was performed at the 144 keV, 1.2 MeV, 5 MeV and 14.8 MeV fields of the PTB in 2001 and was published in 2007 [4]. In 1993-96, the CCRI(III)-K1 comparison for fluence measurements of 24.5 keV neutrons was conducted with 3 Bonner Spheres circulating among several participants. A publication of the agreed results appeared in Metrologia in 2010 [5]. Since a CCRI key comparison needs to be repeated at least every ten years, a comparison of fluence measurements of neutrons at about 24 keV and in the energy range from a few hundred keV up to about 20 MeV was urgently required. The CCRI-K11 comparison was therefore organised with energies completing the energy set of CCRI-K10: 27.4 keV (preferred to 24 keV since it allows the use of the  $^{45}\text{Sc}(p,n)$  reaction at  $0^\circ$ ), 565 keV, 2.5 MeV and 17 MeV. Despite not being an ISO recommended neutron energy, 17 MeV appeared to be a better and easier choice than 19 MeV for reference fluence measurements following the results of the EUROMET.RI(III)-S2 comparison [6].

The protocol is similar to the CCRI(III)-K10 comparison. All measurements were performed in the same neutron fields produced, this time, at the AMANDE facility [7] of the LNE-IRSN<sup>1</sup>, the pilot laboratory.

The protocol states: participants shall determine  $\Phi/M$ , the fluence per monitor  $M$  count of the desired monoenergetic neutrons at 1 m distance to the target in vacuum. The participating laboratories employed their primary (or calibrated secondary transfer) instruments, which are specified for the determination of neutron fluences and which are (or are at least traceable to) their national standards. Measurements were performed over several months from September 2011 to October 2012. Nine participants took part in this comparison although only seven provided results in the requisite time scale.

Since the key comparisons K10 and K11 can be considered as complementary, it was decided that this report should follow the same structure as that for the CCRI(III)-K10 final report. This report will therefore detail the neutron fields and their specifications, the fluence measurement reference devices employed by the participants, the general method to derive the fluence from the measurements together with the evaluation of the results with their uncertainty budgets and, finally, the determination of the key comparison reference values and the degrees of equivalence.

## 2. Neutron fields: Production, Specification and Monitoring

### 2.1. Neutron fields

The monoenergetic neutron fields were produced at the AMANDE facility, which is based on a HVEE 2 MV Tandetron accelerator system delivering proton or deuteron beams, in the energy range 100 keV - 4 MeV, in DC mode or in pulsed mode [8]. This latter mode [9] was

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<sup>1</sup> IRSN is a Designated Institute of LNE, the French National Metrological Institute, for neutron fluence and related dosimetric quantities. As participant of the comparison, its full name is LNE-IRSN. For convenience, it will however appear mostly as IRSN in the rest of the document.

used for time-of-flight experiments to verify the neutron energy distribution of the 2.5 and 17 MeV neutron fields.

As this facility is at the centre of a 300 m radius exclusion area, experiments take place in a 20 m x 20 m x 16 m experimental hall surrounded mainly by metal walls. The neutron-producing target is placed at the centre of this hall, 7.2 m above the concrete floor.

Measurements were all performed in the forward direction of the neutron emission ( $0^\circ$  relative to the proton or deuteron beam direction). The target and field specifications are detailed in Table 2.1. Although these conditions reduce the contribution of scattered neutrons to the fluence at the measuring points, a correction of the instrument readings was still required for the room-return and air-inscattered neutrons. This contribution was subtracted, if necessary and possible, by an additional measurement with an appropriate shadow cone inserted between the target and the fluence measuring device.

**Table 2.1.** Properties of the neutron fields (projectile energy  $\langle E_p \rangle$  with an uncertainty of 2 keV; neutron energy  $\langle E_n \rangle_{\text{calc}}$  and width  $\Delta E_n$  calculated with the TARGET code [10]).

Reaction	$\langle E_p \rangle$ / MeV	Target / $\mu\text{g}\cdot\text{cm}^{-2}$	Backing /mm	$\langle E_n \rangle_{\text{calc}}$ / MeV	$\Delta E_n$ / MeV	Participants
$^{45}\text{Sc}(p,n)^{45}\text{Ti}$	2.929*	Sc - 19.7	Ta - 0.3	0.0274	< 0.001**	All (except IRMM)
$^7\text{Li}(p,n)^7\text{Be}$	2.302	LiF - 184.5	Ta - 0.3	0.5635	0.016	All
$\text{T}(p,n)^3\text{He}$	3.356	Ti(T) – 1960 <sup>#</sup>	Au - 0.5	2.502	0.099	All
$\text{T}(d,n)^4\text{He}$	1.381	Ti(T) – 1960 <sup>#</sup>	Au - 0.5	17.000	0.484	NPL, CIAE, PTB, NIST, IRSN
	1.242	Ti(T) – 811 <sup>#</sup>	Au - 0.5	16.995	0.202	PTB, LNMRI, IRSN
	1.381	Ti(T) - 811 <sup>#</sup>	Au - 0.5	17.227	0.182	AIST, IRSN

\*This energy was reduced by a tuneable positive 10-kV high voltage connected to the end of the beamline in order to reach the energy of the 27.4 keV resonance by applying a high voltage of about 3 kV.

\*\*The neutron monoenergetic peak width can be assumed to be that of the  $^{45}\text{Sc}(p,n)$  resonance, i.e. lower than a few hundred eVs.

<sup>#</sup>Thicknesses quoted are for the titanium layer.

## 2.2. Monitors

Three monitors were used to track the relative variation with time of the neutron fluence. A current integrator related to the charge of the ion beam on the target and two De Pangher long counters, designated M1 and M2, located at approximately 6 m from the target but at two different angles ( $20^\circ$  and  $100^\circ$  respectively). Ratios between the different monitors were used to control the stability of the neutron field. Since the fluence per integrated charge strongly depends on the ion beam position and since the monitor M2 is very sensitive to backscattered neutrons (e.g. by the instrument itself), the monitor M1 was selected to determine the reference value for the fluence per monitor count.

The long term stability of the monitor M1 was checked using a small AmBe neutron source introduced inside the polyethylene shell of the long counter and the measurements did not reveal any changes with time for the monitor.

### 2.3. Beam energy

The AMANDE accelerator ion beam energy was calibrated in 2007, 2009 and in October 2011 (i.e. during the K11 comparison). This calibration was performed using (p, $\gamma$ ) resonances as well as (p,n) thresholds in order to establish the magnetic factor  $k_p$  of the 90° analysing magnet [11]. This factor links the proton beam energy  $E$  with the magnetic field  $B$  in the magnet as determined by a NMR (nuclear magnetic resonance) teslameter:  $E = k_p \cdot B^2$ . Due to relativistic corrections,  $k_p$  depends on  $E$ .

From these calibrations, it has been established that the proton beam energy is determined with a relative uncertainty of  $5.8 \times 10^{-4}$  and that can also be applied to the deuteron beam. The stability of the ion beam energy during the whole CCRI-K11 comparison was checked by comparing the high voltage required at the target in order to reach the 27.4 keV resonance of the  $^{45}\text{Sc}(p,n)$  reaction for all participants, since the ion beam was always tuned to the same energy. The maximum deviation is 1 kV, i.e. 1 keV, that corresponds to a relative ion beam energy deviation of  $3.4 \times 10^{-4}$ , which is in good agreement with the default uncertainty of  $5.8 \times 10^{-4}$  considered for all AMANDE ion beam energies.

### 2.4. Targets and backings

Targets and backing details are given in Table 2.1.

Several LiF or Sc targets, with the same characteristics, were used. Limiting their use to one or two participants maximum reduced the risk of any degradation of the target throughout the comparison.

The same TiT target (IRSN23) and corresponding blank target (i.e. same titanium layer thickness but without tritium) was used for the 2.5 and 17 MeV fields. However, from June to October 2012, a second and thinner target (IRSN29, with corresponding blank target) was required for the 17 MeV field due to considerable deuterium contamination of the first TiT target.

To experimentally verify the theoretical energy distribution, several measurements were performed by time-of-flight [12] and by proton recoil spectrometry with a 2"x2" BC501A liquid scintillator [13].

## 3. Neutron fluence measurement devices used by the participating laboratories

The participants in this comparison were asked to determine neutron fluence in the forward direction (0 degrees) at one or several distances up to 5 m well suited for the particular measuring device employed in the fields with different neutron energies. From

these measurements the fluence at 1 m distance in vacuum, i.e. corrected for air-outscattering, per monitor M1 count had to be derived. Tables with all relevant monitor data and carefully evaluated correction factors were provided to all participants by the pilot laboratory.

The instruments used by the participants are listed in Table 3.1. In the following discussion, only the major characteristics will be presented with a view to identifying the most important contributions to the uncertainty budgets. Details may be found in the reports of the participants and related papers cited therein.

**Table 3.1.** Transfer instruments used by participant laboratories and traceability to their primary national standards

Laboratory (country)	Neutron energy / MeV	Transfer monitor used by the laboratories	Traceability to primary standards	Remarks
NPL (GB)	0.027 0.565 2.5 17	De Pangher long counter with BF <sub>3</sub> PC as thermal neutron detector	NPL Mn-bath calibrated radionuclide neutron sources	MCNP-calculated response
	17	<sup>27</sup> Al activation foil	T(d,n) neutron field and AP method	Linked to primary standards at 14.8 MeV via the energy dependence of the cross-section
VNIM (RU)	0.027 0.565 2.5	MAP-150: 15 cm diameter PE moderator sphere, 1 mm Cd shield, cylindrical <sup>3</sup> He PC	VNIM Mn- and water-bath calibrated neutron sources	Calculated response function
CIAE (CN)	0.027 0.565 2.5 17	De Pangher long counter with <sup>3</sup> He PC as thermal neutron detector	No information	No results provided
IRMM (EU)	0.565 2.5	Recoil Proton Telescope	Response based on $\sigma_{np}$ cross-section from ENDF/B-V,	H-content of radiators from manufacturer
PTB (DE)	0.027	De Pangher long counter with BF <sub>3</sub> PC as thermal neutron detector	NPL Mn-bath calibrated radionuclide neutron sources	MCNP-calculated response
	0.565	P2: RPPC (600 hPa propane)	Hydrogen content from pressure gauge and active volume related to length gauge	Response based on $\sigma_{np}$ cross-section from ENDF/B V
	2.5 17	T1: RPT (two PC + Si-det.) tristearin radiator and 30 kPa CO <sub>2</sub>	H content of radiator by weighing at IRMM	
NIST (US)	0.027 0.565 2.5 17	<sup>235</sup> U fission chamber, 93.18 % enr., Cd shielded	Response based on $\sigma_{nf}$ cross-section from ENDF/B VI and calibration with <sup>252</sup> Cf source (traceable to Mn-bath and NBS-1 of NIST)	Abundances of other isotopes well specified for corrections
AIST /NMIJ (JP)	0.027	5'' radius PE-moderator sphere (BS) with 0.275 atm <sup>3</sup> He PC (SP9)	24 keV and 144 keV NMIJ fields with respectively <sup>3</sup> He and <sup>1</sup> H proportional counter	MCNP 4B-calculated response
	0.565 2.5 17	8'' radius PE-moderator sphere (BS) with 0.2 atm <sup>3</sup> He PC (SP9)	0.565, 2.5 and 17 MeV NMIJ fields with respectively <sup>1</sup> H PC, thick radiator detector and RPT	
LNMRI (BR)	0.027 0.565 2.5 17	De Pangher long counter with BF <sub>3</sub> PC as thermal neutron detector	No information	No results provided
LNE-IRSN (FR)	0.027 0.565 2.5 17	PLC: homemade long counter with <sup>3</sup> He PC as thermal neutron detector	Calibration with IRSN <sup>252</sup> Cf source (traceable to Mn bath of LNE-LNHB)	MCNP-calculated response

### 3.1. National Physical Laboratory (NPL)

Two different long counters are available at NPL to determine the fluence of neutrons with energies up to 5 MeV. Owing to its smaller size and weight, as well as a more stable moderator, the De Pangher long counter [14] was taken to IRSN.

There exist two sets of neutron responses and effective centres for the long counter:

- Old data based on analysis of measurements made over a range of source to detector distances, with both radionuclide source neutrons and monoenergetic neutrons [15], have been in use at NPL since the mid-1970s.
- New data were calculated using the MCNP code and re-calibrated using all radionuclide sources available at NPL [16-17].

The present measurements at IRSN Cadarache were analysed using both the old set of efficiencies and effective centres, which were used successfully in the past in international neutron fluence comparisons, and also the new efficiency values together with the new calculated effective centres. Although the new long counter parameters should be more accurate than the old ones, they have not yet been fully verified. The optimum parameter values have thus not yet been decided upon and, for the present, a mean is taken of the fluences determined using the new and old parameters. These usually agree to better than 2 %, which is the uncertainty normally assigned to the combined effect of efficiency and effective centre uncertainties. In cases where the difference is greater than 2 %, allowance is made by increasing the uncertainty on the mean to cover the two values.

For 565 keV, 2.5 MeV and 17 MeV measurements of the fluence were made with the long counter at three different distances from the target. These were 250 cm, 300 cm and 350 cm for 565 keV, and 300 cm, 350 cm and 400 cm for 2.5 MeV and 17 MeV. For 27 keV, only a single distance of 190 cm was used.

Activation foils were also used for the fluence measurement at 17 MeV. Four foils, two of 0.30 mm and two of 0.60 mm thickness, both of 25 mm diameter, were placed together in the order thick-thin-thick-thin with the thick foil facing the target. The distance of the front foil from the target face was set to be  $90.0 \pm 0.2$  mm.

Foil activities can be measured using  $4\pi\beta\text{-}\gamma$  coincidence counting, or if the foil  $\beta$ -counting efficiency is known, by  $\beta$ -counting alone. The foils used at NPL have had their  $\beta$ -counting efficiency measured by  $4\pi\beta\text{-}\gamma$  counting following activation in a high neutron fluence. As the foil activation after irradiation at the AMANDE facility was low, simple  $\beta$ -counting was the preferred option since this allows the use of an anti-coincidence shielded low-background counter, thus reducing background effects. The reference values for the  $\beta$ -counting efficiencies of the foils were determined at NPL in a 14.7 MeV neutron field with the fluence standardised using proton recoil telescopes and the associated alpha particle technique.

### 3.2. China Institute of Atomic Energy (CIAE)

CIAE brought their reference De Pangher long counter to IRSN. However, the central counter, a  $\text{BF}_3$  tube, was not allowed to exit China. CIAE then decided to buy a new  $^3\text{He}$  counter from Centronic and to perform the measurements with this new configuration. Once the counter was returned to China after the comparison, a cross calibration in CIAE monoenergetic fields with both the new  $^3\text{He}$  and reference  $\text{BF}_3$  tube was planned.

CIAE performed measurements at the AMANDE facility at several distances: 4 at 27 keV (1, 1.5, 1.7 and 2 m), 6 at 565 keV (1.5, 2, 2.5, 3, 3.5 and 4 m) and 5 at 2.5 and 17 MeV (same as at 565 keV but without 1.5 m).

However, CIAE encountered a serious problem with its accelerator facility, compromising the continuation of its participation in the CCRI(III)-K11 comparison.

### 3.3. D.I. Mendeleev Institute for Metrology (VNIIM)

The VNIIM laboratory used an in-house designed neutron monitor designated MAP-150. The response of the polyethylene moderator, 150 mm in diameter and covered by 1 mm Cd, is rather high, in particular for neutron energies around 500 keV, because the central thermal neutron detector is a voluminous cylindrical proportional counter filled with 710 kPa  $^3\text{He}$  gas. Unfortunately, as this instrument is not suitable for measurements of neutron fields with energies higher than 5 MeV, VNIIM did not participate in the comparison for the 17 MeV field. The MAP-150 is a transfer neutron monitor traceable to the primary standards of the laboratory. The neutron fluence response of the MAP-150 has been determined by means of Monte Carlo calculations normalised by calibrations with two monoenergetic (2.5 and 14.8 MeV using associated particle method as primary standard) and various radionuclide source neutrons (calibrated by VNIIM Mn-Bath).

Stability problems were observed with the electronics of the acquisition system based on a SCA for pulse counting. An additional set of measurements was therefore performed using a MCA (IRSN Fast Comtec MPA-3 system) with a predefined threshold.

The neutron fluence rates of the 565 keV and 2.5 MeV fields were measured at several distances: 0.805, 1.0 and 1.5 m at 27 keV, only with the MCA; 1.0, 1.5, 2.0, 3.0 and 4.0 m with the SCA and 1.5, 2 and 3 m with the MCA.

The final results were derived from the mean of the values obtained with the MCA (when available) and the SCA.

### 3.4. Institute of Reference Materials and Measurements (IRMM)

IRMM participated in the CCRI(III)-K11 comparison with the same recoil proton telescope as used in CCRI(III)-K10. This detector consists of two pill-box proportional counters filled with  $\text{CO}_2$  and Ar (or Kr) in front of a Si surface barrier detector thick enough to stop 2.5 MeV recoil protons, but not 17 MeV protons. The material and thickness of the radiator in front of this detector telescope were selected to optimise the efficiency for the

neutrons to be measured and the energy distribution of recoil protons to be analysed. The efficiency, first calculated by the designer of the instrument about 40 years ago [18-19], has been recalculated with a formalism published 20 years later [20]. It was uncertain that this RPT could provide confident results at 565 keV and it was impossible to be used at 27 keV. Measurements were therefore performed only at 565 keV (two distances from the target: 10 cm and 15 cm) and 2.5 MeV (15 cm, 20 cm and 25 cm). Finally, IRMM provided results only for the 2.5 MeV field considering the mean value between measurements at 20 cm and 25 cm.

### 3.5. Physikalisch-Technische Bundesanstalt (PTB)

The PTB intended to use its primary reference, i.e. the recoil proton proportional counter (RPPC) P2 for the determination of the fluence for neutrons with energies between 24 keV and 1.2 MeV [21] and the recoil proton telescope T1 [22] above 1.2 MeV. These instruments are directly based on the differential np scattering cross-section. In addition, measurements with the PTB long counter LC1 were carried out in all neutron fields to establish this instrument as a new secondary reference instrument that is more efficient to use than P2 and T1.

In the course of these measurements it turned out that the use of the proportional counter P2 was severely affected by electromagnetic interference and by microphonic disturbances induced by the strong air jet used to cool the target. These problems impaired use of the instrument in the 27 keV field, although it had been demonstrated at the PTB that a recoil proton proportional counter is suitable for measurements at 27 keV [23], even if the neutron field is produced using the  $^{45}\text{Sc}(p,n)$  reaction with its intense photon component. As a consequence, the long counter LC1 had to be used in the 27 keV field instead of P2. Because of these technical difficulties, the pilot laboratory IRSN granted the PTB a second opportunity for measurements, which were carried out in June 2012. However, despite improvement of the target cooling system by IRSN, P2 could still not be used at 27 keV but all other measurements could be repeated. The quantity to be reported for the present comparison is therefore calculated from the mean of the two results obtained with LC1 (27 keV field determined at 215 cm from the target), P2 (565 keV field at 116 cm) and T1 (2.5 MeV and 17 MeV fields at 22.6 cm).

### 3.6. National Institute for Standards and Technology (NIST)

NIST employed a  $^{235}\text{U}$  fission chamber at all neutron fields. The rather large and massive construction is completely surrounded by 1 mm of Cd in order to reduce the reading due to thermalized room-return neutrons. The U layer is enriched to 93.15 % only, but the abundances of other isotopes are specified such that corrections could be reliably calculated on the basis of evaluated cross-sections.

The relative energy dependent neutron detection efficiency is simply determined by the fission cross-section, which can be taken from evaluated data files with uncertainties ranging from 2 % - 4 %. The efficiency of the fission chamber was determined by means of a  $^{252}\text{Cf}$  neutron source, which was calibrated in the NIST Mn-bath and is in this way

traceable to the emission rate of the NIST primary standard Ra/Be( $\gamma$ ,n) neutron source NBS-1.

Measurements were performed with alternatively both sides of the fission chamber (back and front) facing the target. Distances from the front face of the fission chamber to the target were 28 cm at 27 keV, 70 cm at 565 keV and 160 cm at 2.5 and 17 MeV. Additional measurements without a shadow cone were performed at 2.5 MeV and 17 MeV at 50 cm and 80 cm.

Two methods were therefore employed at 2.5 MeV and 17 MeV to determine the scattered neutron contribution to the total fluence: the shadow cone method and an estimation based on the three distances measurements and the  $1/d^2$  law. A discrepancy, up to 1 % - 2 %, was observed between the two methods, *a priori* due to the high sensitivity of the fission chamber to the thermal/epithermal neutrons created by the shadow cone. Numerical simulations using the well-established Monte Carlo transport code MCNP at NIST and at Cadarache were employed to make adjustments for the energy dependence of the fission chamber, for the differing geometry of the calibration at NIST and the SC-AC (without cone - with cone) runs at Cadarache, and for the epithermal neutrons emitted by the shadow cone. MCNP was also employed for numerous corrections supplied by the host laboratory for scattering in the backing of the accelerator target and from hardware in the experimental hall.

NIST then provided final results applying the average of the two methods at 2.5 MeV and 17 MeV and the shadow cone method at 27.4 keV and 565 keV. An additional uncertainty was considered to take into account the method used to determine the scattered neutron contribution: 2 % at 27.4 keV and 565 keV, 1 % at 2.5 MeV and 17 MeV.

### 3.7. National Metrology Institute of Japan (NMIJ/AIST)

NMIJ used two moderator detectors (Bonner Spheres) as transfer instruments. The response of two polyethylene spheres, 20.32 cm (8") and 12.70 cm (5") in diameter with spherical  $^3\text{He}$  proportional counters in the centre (Centronic model SP9) were simulated with the MCNP-4B code.

Traceability to national standards was established through calibration measurements in monoenergetic neutron fields at NMIJ. The response of the 5" sphere for 27.4 keV neutrons was derived from 24 keV and 144 keV monoenergetic neutron fields. Neutrons for 24 keV were obtained with iron filtering of a neutron beam produced by the  $^7\text{Li}(p,n)^7\text{Be}$  reaction, this latter reaction also being used for the 144 keV field. The neutron fields at 565 keV and 17000 keV were produced using the same reaction and nearly same field characteristics as at IRSN. For the 2.5 MeV field, the D(d,n) reaction was used with low deuteron energy (230 keV).

The neutron fluence energy distributions of NMIJ fields were calculated in a very similar way as at IRSN, i.e. by Monte Carlo simulation but using the MCNP-ANT code [24].

The neutron fluence reference at the NMIJ 24 keV field was determined using a  $^3\text{He}$  cylindrical proportional counter with a 0.5 mm thick Cd cover, which was calibrated with

the thermal neutron fluence standard [25]. In the case of 0.144 MeV neutrons, the fluence was determined with a proportional counter filled with 955.5 kPa hydrogen and an admixture of 34.7 hPa methane. At 2.5 MeV, the fluence was determined using a proton recoil neutron detector, called thick radiator (TR) detector, consisting of a 0.5 mm thick polyethylene radiator disk mounted in front of a silicon surface barrier detector [26]. At 17 MeV, a  $\Delta E-E$  type proton recoil telescope (RPT), consisting of a polyethylene radiator (1 mm thickness and 30 mm diameter) and two silicon surface barrier detectors, was used. For the CCRI-K11 comparison at the AMANDE facility, the 5" sphere was placed at 60 cm from the target in the 27.4 keV field. In the other fields, the 8" sphere was used at distances of 97 cm (565 keV) and 211 cm (2.5 MeV and 17 MeV). Measurements with shadow cone were performed in all cases.

### 3.8. Laboratorio Nacional de Metrologia das Radiações Ionizantes (LNMRI)

LNMRI participated in the measurements with a De Pangher long counter. The measurements were performed in all the neutron fields at the following distances: 2.5 m at 27 keV, 3.8 m at 565 keV and 2.5 MeV, and 4 distances at 17 MeV (2.5 m, 2.8 m, 3.15 m and 3.8 m). LNMRI was unfortunately not able to provide results within the comparison deadlines.

### 3.9. Institute for Radiological Protection and Nuclear Safety (LNE-IRSN)

Throughout the comparison, IRSN used its home-made long counter known as PLC, corresponding to an optimised design of the De Pangher type [27].

The PLC measurements were performed just before each participant to check the repeatability of the neutron fields. The PLC was placed at 3 m distance from the target in the 27 keV field and 5 m for the other fields. At the end of the comparison, the fluence per monitor count was measured 9 times at 27 keV, 13 times at 565 keV, 16 times at 2.5 MeV and 12 times at 17 MeV. The final results provided by IRSN correspond to the mean value of all measurements performed at each energy level.

Fluence traceability to primary standards is established through the calibration of the PLC at the IRSN  $^{252}\text{Cf}$  neutron reference field [28], previously calibrated by the LNE-LNHB Mn-bath [29]. The IRSN PLC also participated in the EURAMET 936 comparison in 2008 [30], whereas several parameters such as the dead time, effective centre and simulated response have since been modified. All these parameters, as well as the method for establishing the fluence reference per monitor count at the AMANDE facility, are detailed in reference [31].

As the pilot laboratory, IRSN also performed measurements of the neutron energy distributions at 2.5 and 17 MeV using the time-of-flight method and a BC501A liquid scintillator, as other contributions than the reaction within the reactive layer may occur in the target:

- The BC501A liquid scintillator (2" x 2") allowed, after (n, $\gamma$ ) discrimination, measurement of the energy distribution of the energy deposited by recoil protons scattered by neutrons between 1 MeV and 20 MeV. The energy distribution of the

neutron fluence is obtained by unfolding the proton spectrum using a response matrix validated at reference neutron fields (monoenergetic and/or radionuclide sources) [13]. An energy calibration was performed with photon sources before each measurement. Measurements were repeated several times at 17 MeV to monitor any variation in the neutron energy distribution due to target modification with time (increase of the deuterium content and of the tritium implanted in the backing for example). Unfortunately, all the data in the hard disk containing these measurements were lost with the exception of the measurement performed in June 2011 for the first use of the thicker TiT target (IRSN23).

- Using the pulsed mode of the AMANDE accelerator, time-of-flight measurements were performed in February 2012 at 2.5 MeV and 17 MeV with the BC501A liquid scintillator at two distances: 1.788 m and 10.749 m. The target was IRSN23 for both energies and the beam pulse widths were  $(3.0 \pm 0.1)$  ns at 17 MeV and  $(2.1 \pm 0.1)$  ns at 2.5 MeV. Time-of-flight measurements at 17 MeV were also performed in July 2012 at 2.088 m and 10.743 m with both IRSN23 and IRSN29 targets. The pulse widths were between 3.8 ns and 5.1 ns.

#### 4. Measurements

The experiments were carried out within eleven weeks split between June 2011 and October 2012, nine weeks being dedicated to the participants and two weeks for spectrometry and several reproducibility checks by IRSN. One dedicated week was allowed per participant, with all the required energies realized within this week. IRSN measured the neutron fluence at each energy, and sometimes several times at the same energy when this energy was used for more than a day, to determine the reproducibility of the direct fluence per monitor M1 count over the whole comparison.

##### 4.1. Mean Neutron Energy and Fluence Energy Distributions

The ion beam energies from the Tandetron accelerator were selected to obtain the desired mean neutron energy for given target parameters, with the exception of AIST at 17 MeV, where the deuteron energy was not modified after the change of target thickness (see table 2.1).

The mean neutron energy  $\langle E_n \rangle$  was calculated by the TARGET code from the fluence energy distribution of uncollided neutrons in the beam axis (i.e. calculation scored in an infinitesimal volume placed at  $0^\circ$ ). The 27 keV field produced by the  $^{45}\text{Sc}(p,n)$  reaction cannot be modelled using TARGET. Therefore, the spectral distribution was assumed to have a Gaussian shape with mean energy and standard deviation depending on the kinematics and target thickness. Since this assumption does not take into account the peak width reduction due to the resonant structure of the  $^{45}\text{Sc}(p,n)$  reaction, the true width could be far lower than the assumed value.

For all the neutron fields, the fluence energy distribution is determined by simulation coupling, within a dedicated interface, the TARGET code (or the kinematics for the 27.4 keV field) for the neutron source and MCNPX 2.5.0 [32] for neutron transportation and

scattering in a fully detailed geometry of the target holder and the AMANDE experimental hall. If the shadow cone technique was used, the simulation result with the shadow cone was subtracted from that obtained without the shadow cone.

For transportation, the Monte Carlo code MCNPX 2.5.0 was used and the neutron energy distribution at  $0^\circ$  was obtained using (except for proton recoil telescopes) a point detector (i.e. a F5 tally) in order to acquire sufficient statistics in about 200 channels. The energy distribution was calculated for all the configurations (distance, type of detector, type of shadow cone). In the case of the RPTs, the energy distribution was calculated on the whole surface of the radiator (i.e. tally F2 on a 1.5 cm radius disk) taking into account neutrons having an incidence angle only between  $-15^\circ$  and  $+15^\circ$  with respect to the normal of the disk.

At 17 MeV, the spectrometry measurements revealed several secondary peaks that are explained by [33]:

- An intermediate layer (made of titanium and gold with about 10 % titanium) between the Ti layer and the gold backing, as revealed by the morphologic analysis of a blank target, and tritium implanted in this intermediate layer, leading to a secondary peak in the range 14 to 16 MeV by the T(d,n) reaction.
- Deuterium contamination in both the Ti layer and the intermediate Ti/Au layer, generating a secondary peak respectively at about 4.3 MeV and between 3 and 4 MeV, all by the D(d,n) reaction.
- Deuteron implantation in the backing, at the end of their range producing a secondary peak between 2.5 and 3 MeV by D(d,n) reaction.

All these additional neutron sources were included in the TARGET code calculation with individual weight matching two extreme cases, for both the TiT targets IRSN23 and 29, corresponding to the minimal and maximal contribution of these secondary peaks in the neutron energy distribution as observed by spectrometry at different times during the comparison. The two calculated energy distributions were considered as providing the uncertainty (rectangular distribution) on the response or fluence induced by the limited knowledge of the real energy distribution during the measurements of each participant. It was also agreed by all participants that this comparison would only compare results for neutrons above 13 MeV energy, i.e. produced by the T(d,n) reaction.

For 2.5 MeV, an additional contribution also had to be taken into account in the simulation due to the tritium present in the Ti/Au intermediate layer. The tritium content in this layer was the same as that obtained for the 17 MeV neutron field with the IRSN23 target (since it is the same target used for both energies), i.e. between 1.8 % and 5 %, assumed to be uniformly deposited in the layer. As at 17 MeV, two calculated energy distributions were considered, with a low energy tail going from the monoenergetic peak down to 2.2 MeV to 2 MeV.

All relevant spectra were provided to the participants for correction of their fluence measurements. One example for each neutron energy is shown in Fig. 4.1.

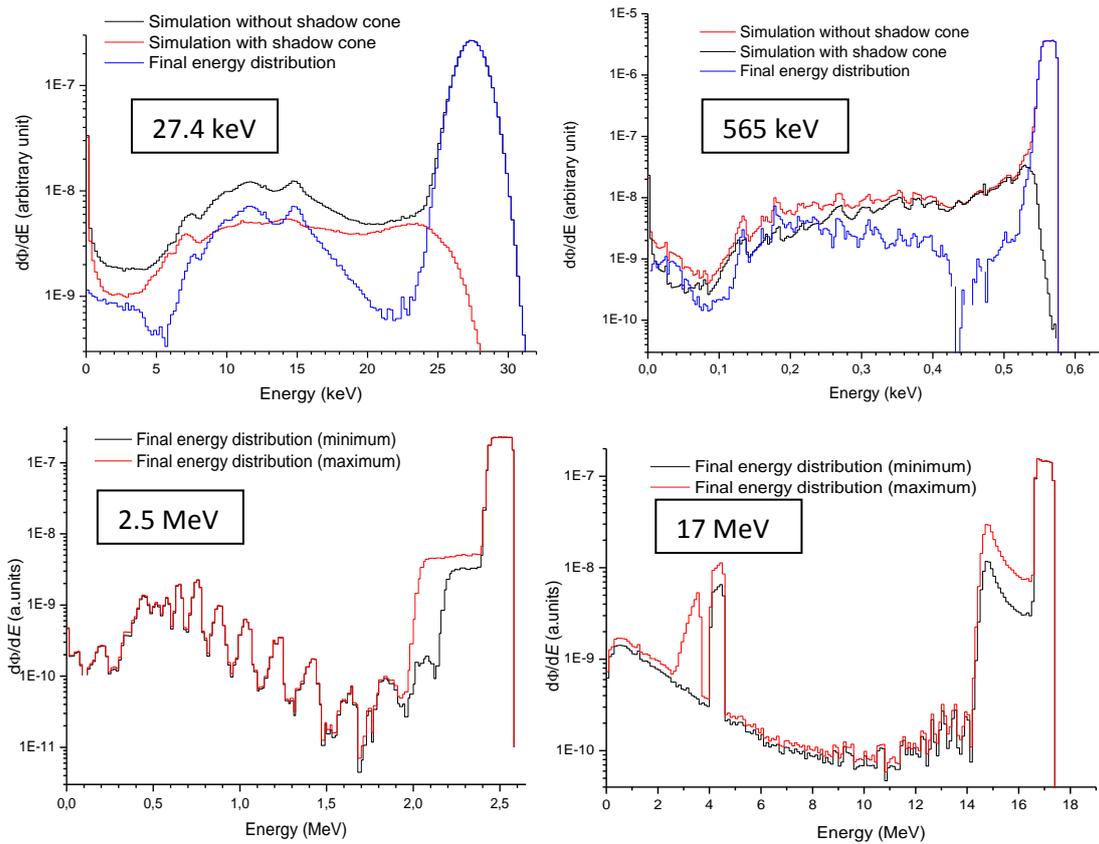


Figure 4.1. Simulation of the neutron energy distribution with the TARGET code output coupled to MCNP. At 27.4 and 565 keV, simulations with a shadow cone, without a shadow cone and their difference are shown. At 2.5 and 17 MeV, the two energy distributions with minimal and maximal contribution of the secondary peaks are shown. The structures below 2 MeV in the 2.5 MeV field come from a problem, still under investigation, in interfacing TARGET with MCNP.

#### 4.2. Neutron Monitoring

To normalise the different measurements made at each energy level by each participant, IRSN had available three devices for monitoring the fluence:

1. A current integrator, CI, provided pulses corresponding to a fixed amount of charge collected at the target and hence to a fixed number of protons or deuterons incident on the target. The beam current is derived from this monitor with traceability to LNE, the French National Metrological Institute.
2. Monitor M1, a long counter located at  $20^\circ$  to the beamline and  $\sim 6$  m from the target, gave a direct measure of the neutrons produced.
3. Monitor M2, also a long counter, located at a backward angle relative to the beamline and  $\sim 6$  m from the target provided a back-up for M1 but was eventually not used.

For each measurement, a set of monitor data was recorded in a multi-scaler (MS) file, namely the total measurement time, the total count in the IRSN PLC, the total counts of

the neutron monitors M1 and M2, the digitised beam charge (CI), and the digitized voltage on target. These data were used to calculate the count rates (for dead time corrections), the mean beam current at the end of the beamline and the ratios of the monitor rates.

The three monitors made available to normalise the fluence measurements of the various participants have various advantages and disadvantages. The CI, measuring the current on the neutron producing target, provides a good measure of neutron production, provided the beam position on the target does not change and especially does not reach part of the target without reactive layer. If this was observed for a given beam setup, study of the reproducibility of the fluence per CI count has shown that the position of the beam on the target was clearly different from one participant to another. That is why this monitor was not considered for the comparison reference value.

The other two monitors, M1 and M2, measure neutrons directly. Their counts have to be corrected for dead time and for background due to neutrons not produced in the reactive layer of the target. This background is determined by measurements without beam on target (natural background) and with a backing or blank target.

These monitors suffer, however, from the fact that their readings are affected by the presence of whatever devices are located in the neutron field in the vicinity of the target. These can scatter neutrons into, or away from, the monitors. Since the quantity required for the present comparison exercise is the fluence in vacuum at 1 m from the target, in the absence of the fluence measuring equipment, corrections needed to be applied to allow for the presence of the detecting devices and of a shadow cone in the measurements where one was present, by correcting the M1 and M2 readings to what they would have been in the absence of these objects. Data were taken for M2 as well as M1 but the M2 monitor was, in the end, not used.

Monitor corrected counts and correction factors for M1 were provided by IRSN for all participants. These were obtained from CI and M1 readings taken during the measurements by the participants, and also for so-called 'free field' arrangements. For the free field measurements, all fluence measuring equipment was moved as far away as possible from the neutron producing target to reduce scatter into the monitors to a negligibly low level. The ratios of the monitor readings for the two different arrangements provided the correction factors.

The measurements of the monitor corrections depend on a stable measure of the neutron output to relate the free field measurements to those with the fluence measuring equipment in place. The device used to measure the output was the CI. The measurement of the M1 scatter correction factors thus depends on the CI being a stable monitor, at least for the period between the free field measurement and the measurement with the fluence measuring equipment in position. In some measurements, this was not the case and reference values for the corrections were then proposed by IRSN.

All the monitor correction factors are less than or equal to unity with the exception of NIST and IRMM measurements with their devices partly shadowing the monitor M1 from the target. Values less than one reflect the fact that the devices and the shadow cone scatter neutrons into the monitor. Values larger than one indicate that the device absorbs some of the neutrons. For the measurements with shadow cone, the corrections might be expected to be larger than for the case without a cone. In general, this was true but not in all cases and the correction with a cone was not significantly larger. The largest correction for

values less than one was about 4 %, and reached 36 % in the case of the NIST large fission chamber placed at 28 cm from the target and completely masking the monitor M1.

### 4.3. Fluence Measurements of the Participants

The participants were requested to determine the neutron fluence of the unscattered neutrons in vacuum at 0 degrees and 1 m distance.

The detection system, installed on one of the automated transport systems, could be moved and aligned in any desired position at 0 degrees. Any distance to the target between 9 cm for the Al-activation foils, up to 500 cm for the IRSN long counter and 10 m for time-of-flight measurements were realised.

All the instruments, with the exception of the PTB and IRMM recoil proton telescopes (RPT), were used with and without a shadow cone to remove most of the contribution of the scattered neutrons to the fluence measurements. For the RPT, a background measurement with the radiator in the reverse position was performed instead.

In all cases, except for proton recoil telescopes, additional measurements were carried out with appropriate IRSN shadow cones between the target and the detector. Only PTB used their own shadow cones for the measurements with their proportional counter P2.

Corrections for neutrons not produced in the reactive layer of the target were estimated by measurements replacing the target by the backing only (27.4 keV) or a blank target as defined in 2.4 (2.5 MeV and 17 MeV) and completed by natural background measurements in the experimental hall. Measurements were performed with and without a cone and with a backing or blank target, but only with the radiator for the RPT because the number of detected events with the blank target and the radiator in reverse position was negligible. These corrections appeared particularly difficult to estimate at 17 MeV due to deuterium implantation in both the TiT target and the blank target with time differing in homogeneity, implantation level and speed. In addition, the order of measurements with the TiT target rarely corresponded to the order of measurements with the blank target. IRSN provided values to the participants based on the slope of the increase of the ratio between M1 and CI (assumed linear with time) for a given ion beam setup and for both the TiT and blank targets.

Once all these corrections based on measurements were applied, there were still some unwanted neutrons in the energy distribution, mainly coming from scattering in the target backing or just surrounding materials. To determine the unscattered neutron fluence, an additional correction, designated  $C_s$ , was therefore required. IRSN provided these  $C_s$  corrections calculated by TARGET and MCNP as explained in 4.1. It corresponds to the ratio between the calculated total neutron fluence (the difference between the total fluences calculated without and with shadow cone or, for the RPT case, the total fluence within a 15 % incidence angle) and the corresponding direct fluence (i.e. of unscattered neutrons). Values were calculated using both the F2 (surface tally) and F5 (point detector) options in MCNP. The choice between these two tallies, leading to up to 3 % variation, was to be made by the participants in light of their knowledge of the detector used since it depends on the detector sensitivity to the energy and fluence variations over its detection surface

(especially for large detectors at small distances) but also on the method used to determine the instrument response and/or effective centre.  $C_s$  with F2 tally are between 0.912 and 1.006, and between 0.905 and 0.990 for F5 tally.

Measurements followed a general scheme:

1. Fluence measurement by the IRSN PLC at 5 m (or 3 m at 27.4 keV).
2. Determination of background in the PLC due to in-scattered neutrons with shadow cone.
3. Monitor readings in 'free field' arrangement (see 4.2).
4. Fluence measurement by the participant's device at optimal positions.
5. Determination of background due to in-scattered neutrons with the recommended shadow cone or in the case of RPT an additional measurement without the radiator in order to correct for reactions in the radiator backing.
6. Background measurements with tantalum backing (27.4 keV) or blank target (2.5 MeV and 17 MeV), with and without a shadow cone (except for RPT) for the participant's device and the IRSN PLC.
7. Background measurements without any beam at target.

The general scheme for data analysis was:

1. Correction for dead time losses.
2. Evaluation of the net count rates, spectra or activations.
3. Interpretation of these results in terms of direct neutron fluence at the point of measurement, corrected for the contribution due to target scattered neutrons, folding the spectral fluence provided by the pilot laboratory with the energy-dependent detector response (mean energy distribution for the 2.5 and 17 MeV fields).
4. Calculation of the fluence of neutrons with the desired energy for a distance of 100 cm from the target in vacuum, i.e. corrected for air-outscattering if necessary.
5. Relating this fluence to the corresponding M1 counts corrected for dead time losses and inscattering/shadowing.
6. At 17 MeV, calculating the fluence per monitor count only for neutron energies above 13 MeV using the simulated energy distribution of the unscattered neutrons.
7. Evaluation of the uncertainty budget according to the ISO GUM recommendations [34] and documentation as detailed as necessary for the evaluation of the comparison exercise.

## 5. Discussion of the Results

### 5.1. Reproducibility of the Fields

To determine the reproducibility of the unscattered neutron fluence per monitor M1 count during the whole comparison, the ratio between this fluence measured with the PLC for a specific participant (or IRSN test) and the mean direct fluence for all performed measurements can be used. These ratios are presented for each energy in Figure 5.1, all the corrections detailed in the previous paragraphs having been applied. Since only reproducibility is considered, the uncertainty reported in the graphs (with a coverage factor of  $k=1$ ) takes into account only statistics (with dead time and background) as well as the monitor M1 and PLC stabilities over one day.

The standard deviation of the PLC measurements is each time lower than or equal to the mean uncertainty of these measurements, giving a clear indication of the reproducibility of the fluence per monitor count in all CCRI(III)-K11 neutron fields.

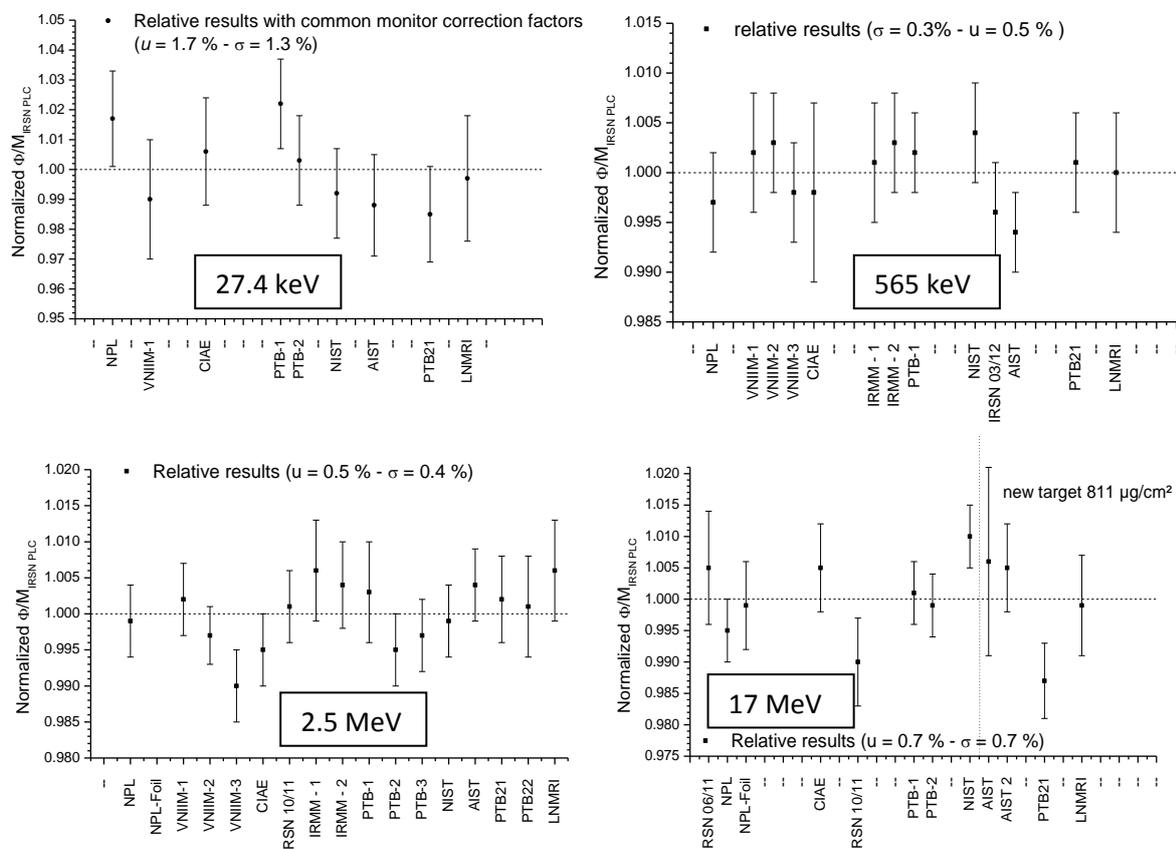


Figure 5.1. Ratios between the unscattered neutron fluence at 1 m per monitor M1 corrected count measured with the PLC for each participant and the mean direct fluence measured by the PLC throughout the comparison, at each energy level.  $u$  and  $\sigma$  are respectively the mean uncertainty and the standard deviation of the set of measurements at a given energy.

## 5.2. Evaluation of the Results of all Participants

The participants were requested to report only one result for each energy even if the fluence was determined with different methods.

For all the data, the weighted mean value (w.m.) was calculated using the reported uncertainties for weighting. In this procedure we neglected any correlation of the uncertainties although some uncertainty budgets obviously include contributions of the same origin. Even in the case of the uncertainties considered for the (differential)  $n, p$  scattering cross-sections this assumption seems, however, to be justified because the laboratories partially refer to different evaluated data sets. Concerning the specification of the hydrogen content of tristearin radiators used in the RPT of IRMM and PTB, the correlation cannot clearly be deduced from the IRMM certificates. In general, however, the measurement and analysis methods are sufficiently different to support the assumption.

Following the method described in reference [35], the consistency of the results was verified with a  $\chi^2$  test, where  $P(\chi^2(\nu) > \chi^2_{\text{exp}})$ , the probability that the theoretical  $\chi^2$  for

the degrees of freedom of the evaluation is greater than the experimentally observed value has to be higher than 5 %. Since this test was successful at all energies, the weighted mean was considered as the KCRV, i.e. the key comparison reference value.

### 5.2.1. Fluence of 27.4 keV Neutrons

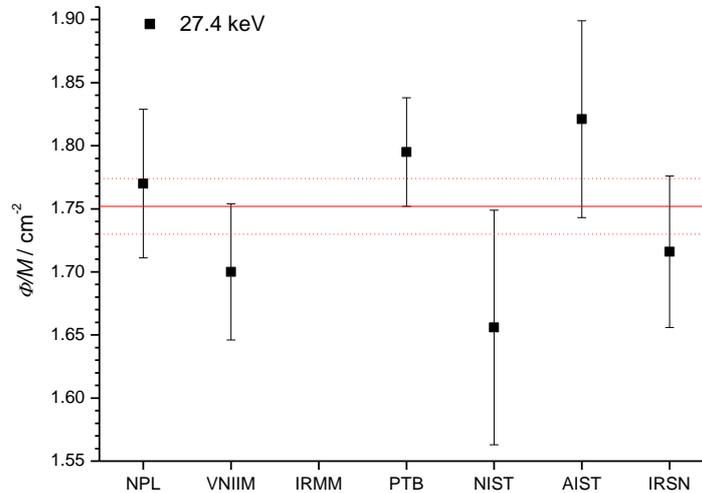


Figure 5.2. Neutron fluence in vacuum at 0 degrees and 1 m distance from the target per count of the neutron monitor M1, for the 27.4 keV field. The weighted mean is shown as the key comparison reference value KCRV. All the uncertainties are shown with a coverage factor  $k=1$ .

Fig. 5.2 shows that all data are rather close to the mean value. The experimentally observed  $\chi^2$  value is 4.228 with 5 degrees of freedom and  $P(\chi^2(\nu) > \chi^2_{\text{exp}}) = 0.52$ .

### 5.2.2. Fluence of 565 keV Neutrons

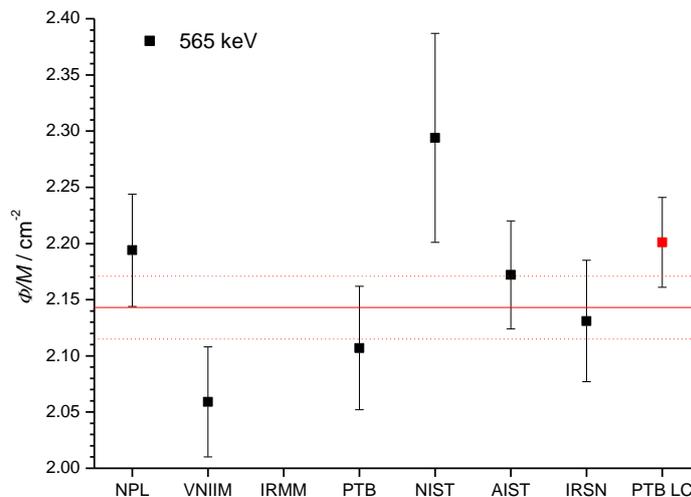


Figure 5.3. Neutron fluence in vacuum at 0 degrees and 1 m distance from the target per count of the neutron monitor M1, for the 565 keV field. The weighted mean is shown as the key comparison reference value KCRV. All the uncertainties are shown with a coverage factor  $k=1$ .

The scatter of the data at 565 keV is larger than at the other energies, mainly due to VNIIM and NIST results. The experimentally observed  $\chi^2$  value is 8.032 with 5 degrees of freedom and  $P(\chi^2(\nu) > \chi^2_{\text{exp}}) = 0.15$ . The KCRV is however still considered as the weighted mean value. The PTB long counter was not considered in the KCRV calculation and is shown only for information purposes.

Due to its lower uncertainty, the VNIIM value exhibits the largest deviation to the mean value. If the VNIIM value is ignored, the  $\chi^2$  value is reduced to 4.174 and the weighted mean value becomes  $(2.164 \pm 0.023) \text{ cm}^{-2}$ .

Nevertheless, the VNIIM and NIST results are both included in the final KCRV calculation.

### 5.2.3. Fluence of 2.5 MeV Neutrons

IRMM provided a reference value that was  $(3.28 \pm 0.12) \text{ cm}^{-2}$  whereas the weighted mean value based on the other participants was  $(2.235 \pm 0.020) \text{ cm}^{-2}$ . IRMM was informed that this result had to be checked but was not able to correct the value within the deadline. Once the comparison result had been initially provided to all the participants, IRMM noticed that, due to a misprint, an incorrect distance from the target was entered in the Excel file used to calculate the fluence with their instruments: the front face of the telescope instead of the converter distance. Since the distances were 15 cm to 25 cm, such a misprint generates large differences in the fluence value.

A new value was therefore sent to the evaluator with full details and proof that the only difference between the old and new values stemmed from the distance. The new IRMM value is in good agreement with the results of the other participants at 2.5 MeV.

In view of the nature of the error detected (misprint regarding the distance used), of the explanation given by the IRMM and of the difficulty in organising a new bilateral comparison at AMANDE over coming years, all the participants agreed to accept the new result of the IRMM as prescribed in the document "Measurement comparisons in the CIPM-MRA" (CIPM MRA-D-05). Further agreement from the KCWG(III), the entire CCRI(III) and then from the CCRI was also requested and was unanimously given.

Fig. 5.4 shows that, with the exception of the old IRMM value, and to a lesser extent of the VNIIM value, all data are rather close to the KCRV. With the new IRMM result, the experimentally observed  $\chi^2$  value is 6.836 and  $P(\chi^2(\nu) > \chi^2_{\text{exp}}) = 0.34$ . The values in red (former IRMM and PTB long counter) were not considered in the KCRV calculation, the PTB long counter being shown only for information purposes.

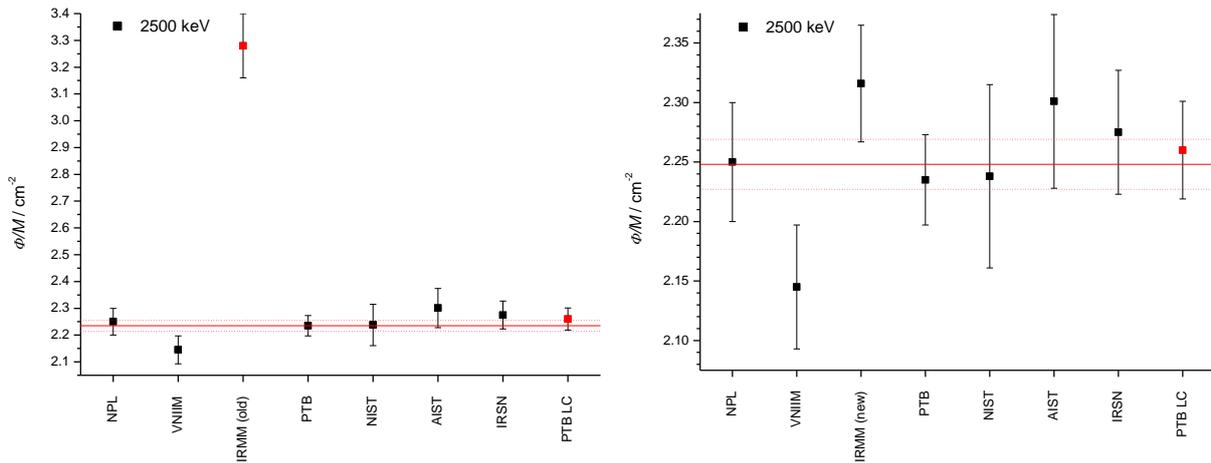


Figure 5.4. Neutron fluence in vacuum at 0 degrees and 1 m distance from the target per count of the neutron monitor M1, for the 2500 keV field with the old (left) and new (right) IRMM values. The weighted mean (calculated with the new IRMM value, i.e. on the right) is considered as the key comparison reference value KCRV. All the uncertainties are shown with a coverage factor of  $k=1$ .

As at 27 keV and 565 keV, the VNIIM value is lower than the KCRV by 3 % to 4 % and could reveal an underestimated calibration factor of the device. If the VNIIM value that exhibits the largest deviation to the mean value is ignored, the  $\chi^2$  value is reduced to 2.249 and the weighted mean value is modified from  $(2.248 \pm 0.021) \text{ cm}^{-2}$  to  $(2.265 \pm 0.014) \text{ cm}^{-2}$ . Nevertheless, the weighted mean taken as the KCRV includes the VNIIM data.

#### 5.2.4. Fluence of 17 MeV Neutrons

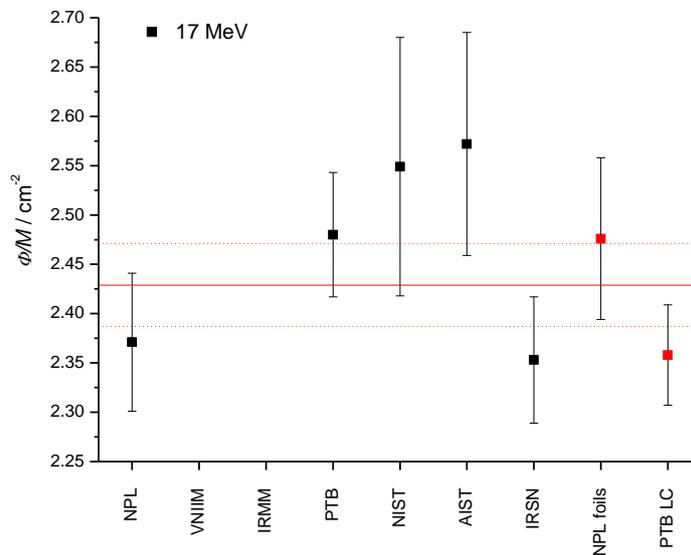


Figure 5.5. Neutron fluence in vacuum at 0 degrees and 1 m distance from the target per count of the neutron monitor M1, for the 17 MeV field. The weighted mean is shown as the key comparison reference value KCRV. All the uncertainties are shown with a coverage factor  $k=1$ .

Fig. 5.5 shows that all data are rather close to the KCRV. The values in red (NPL foils and PTB long counter) were not considered in the KCRV calculation and are shown only for information purposes.

Two groups can be distinguished: a “long counter” group comprising NPL, IRSN and PTB LC, and a group comprising other kinds of detectors (RPT for PTB, fission chamber for NIST, Bonner Sphere calibrated on RPT reference for AIST, activation foil for NPL). The first and second groups have a weighted mean of respectively  $(2.358 \pm 0.004) \text{ cm}^{-2}$  and  $(2.500 \pm 0.023) \text{ cm}^{-2}$ , i.e. they exhibit a large discrepancy between them. The comparison tends therefore to indicate that there could be a device dependent bias that needs more investigation to be confirmed.

This deviation between the values obtained by long counters (NPL and IRSN) and the three other participants (PTB, NIST and AIST) explains why the experimentally observed  $\chi^2$  value is relatively high (6.043) and why the  $\chi^2$  consistency test is low as  $P(\chi^2(\nu) > \chi^2_{\text{exp}}) = 0.20$ . The KCRV is still however considered as the weighted mean value.

### **Summary for all neutron fields:**

After a critical review of the evaluation of all data sets, we came to the conclusion that the weighted mean values of the data sets should be reported as key comparison reference values in all the monoenergetic neutron fields of CCRI(III)-K11 (see Appendix A and Table 7.1).

## **6. Degree of Equivalence**

According to the CIPM MRA, the degree of equivalence (DoE) must be established for each comparison, which can provide traceability for a calibration measurement capability announced in the Key Comparison Data Base, KCDB, Appendix C, by the laboratory. The DoE must be calculated as the deviation of the value reported by the laboratory from the key comparison reference value evaluated in the framework of an internationally accepted key comparison. When calculating the expanded uncertainty ( $k = 2$ ) of each deviation, it must be taken into account that the uncertainty reported by the laboratory is also considered in the evaluation of the weighted mean value.

The DoE  $d_i$  for each participant  $i$  is then  $d_i = x_i - x_{ref}$  with  $x_i$  the participant value and  $x_{ref}$  the KCRV. The variance of DoE is calculated as  $u^2(d_i) = u^2(x_i) - u^2(x_{ref})$  and the expanded uncertainty with  $k = 2$  is  $U(d_i) = 2 \times u(d_i)$ .

The DoE  $D_{ij}$  between two participants  $i$  and  $j$  is  $D_{ij} = x_i - x_j$ . The corresponding variance is  $u^2(D_{ij}) = u^2(x_i) + u^2(x_j)$  and the expanded uncertainty is  $U(D_{ij}) = 2 \times u(D_{ij})$ .

The deviations and the associated uncertainties, calculated according to reference [35], are listed in the Tables of Appendix A.

The degree of equivalence  $d_i$  is shown in Figure 6.1 for the four neutron energies for which the fluence measurements have been compared.

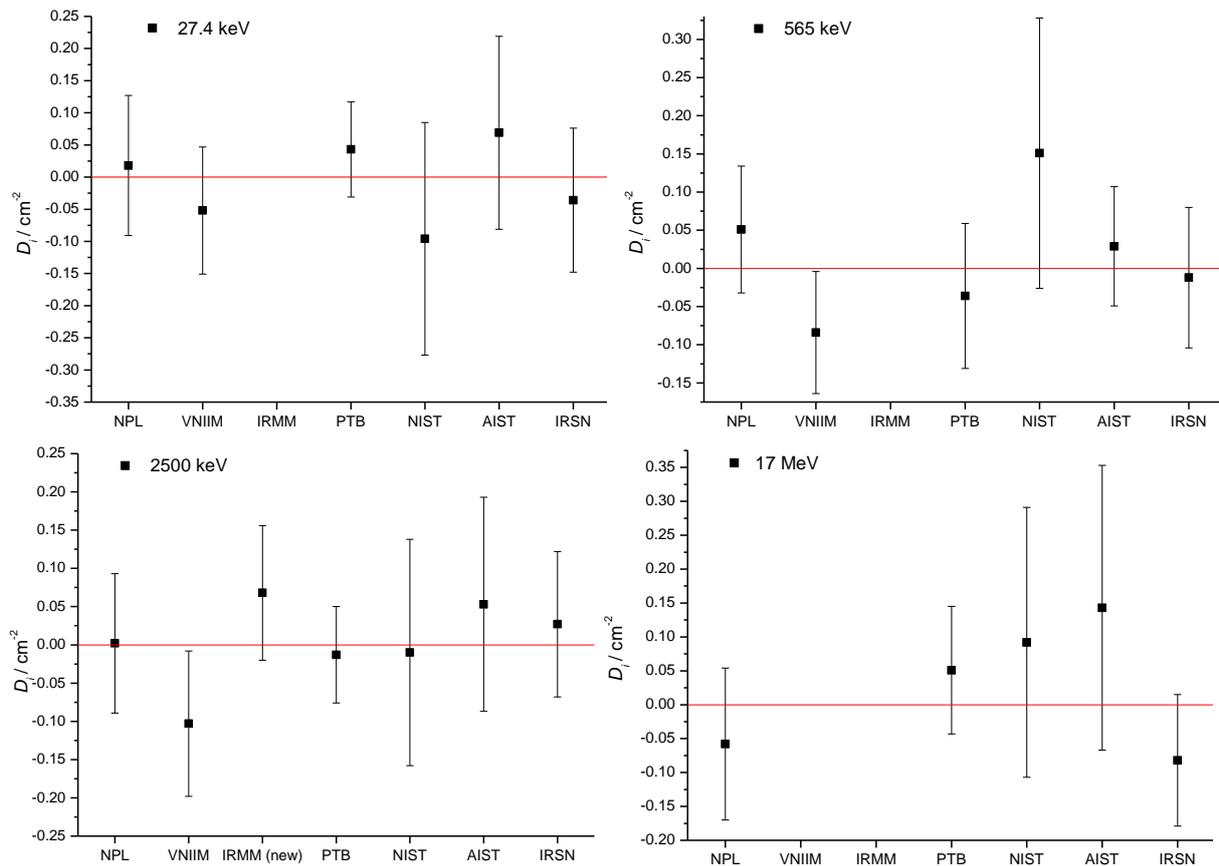


Figure 6.1. Degree of equivalence (DoE) defined as the deviation of the result reported by the laboratory from the KCRV and the expanded uncertainty

## 7. Summary and Conclusions

The key comparison of the determination of the fluence of monoenergetic neutrons was successfully conducted between June 2011 and October 2012 at the AMANDE accelerator facility of IRSN at Cadarache (France). Nine laboratories, authorised by their national metrology organizations, employed either their primary standard fluence measuring devices, or appropriate transfer instruments traceable to their primary standards, in order to determine the fluence of 0.0274 MeV, 0.565 MeV, 2.5 MeV and 17 MeV neutrons. These four neutron energies were selected to complete and/or update the K1 and K10 comparisons.

Stable monitoring and control measurements demonstrated the reproducibility and the stability of the neutron fields over the duration of the comparison of more than one year, even with a change of target thickness at 17 MeV. Irradiations were performed in open geometry and with a rather low room-return neutron background such that no significant uncertainty resulted from the experimental conditions and procedures. The pilot laboratory provided the spectral fluence including the uncollided, and therefore almost monoenergetic, neutrons and the neutrons scattered in the target assembly. The monitor data recorded during the measurement campaigns in which the laboratories participated, corrected for background and dead time, were also provided. The participants determined the fluence in vacuum at a common point of measurement, at 0 degrees and a distance of

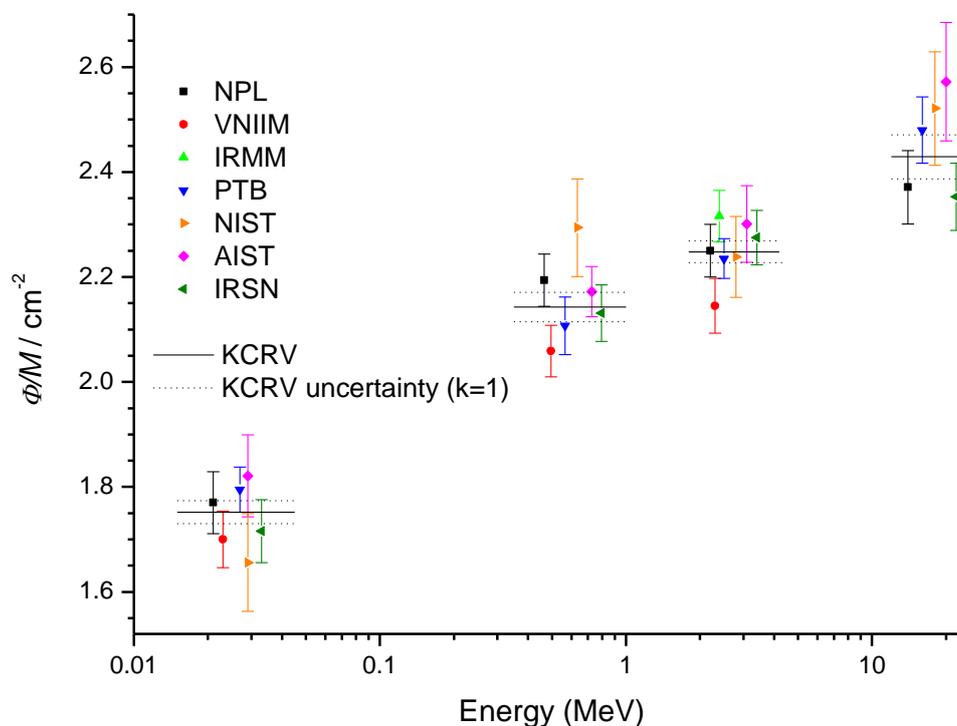
one metre, related this fluence to the properly corrected reading of a selected neutron monitor and evaluated a detailed uncertainty budget.

The reported data were then evaluated. The weighted means of all data obtained for one neutron energy served as the key comparison reference value. The results are summarized in Table 7.1.

**Table 7.1.** Key comparison reference values KCRV, determined as the weighted mean of fluence per corrected monitor M1 count at 1 m in vacuum reported by the laboratories (uncertainties for  $k = 1$ )

Neutron energy / MeV	KCRV / $\text{cm}^{-2}$	Rel. uncertainty of the KCRV / %	$\chi^2/\nu$
0.0274	1.752	1.26	0.846
0.565	2.143	1.31	1.606
2.5	2.248	0.93	1.139
17	2.429	1.72	1.511

All results are shown in Fig. 7.1 in absolute scale together with the weighted mean value.



**Figure 7.1.** Neutron fluence in vacuum at 0 degrees and 1 m distance from the target per count of the neutron monitor M1, for the reactions  $^{45}\text{Sc}(p,n)^{45}\text{Ti}$  (0.0274 MeV),  $^7\text{Li}(p,n)^7\text{Be}$  (0.565 MeV),  $\text{T}(p,n)^3\text{He}$  (2.5 MeV) and  $\text{T}(d,n)^4\text{He}$  (17.0 MeV). The weighted means of the agreed data sets are shown as key comparison reference values KCRV.

The scatter of the data results in reduced  $\chi^2$ -values that are sufficiently close to 1 to indicate that the values and their uncertainties have been correctly estimated. The VNIIM values are, however, systematically lower than the KCRV and a larger scatter of the data is observed at 17 MeV. This suggests that the calibration of the VNIIM instrument needs to be checked and that the fluence measurement methods and/or the evaluation of the uncertainty budget at 17 MeV needs to be re-investigated.

The key comparison has been evaluated with the results submitted by the participants up to December 2013, with the exception of new IRMM data provided in January 2014. The objective of any key comparison is to determine the capabilities of the participating NMIs at the date of measurement and evaluation. The main result of the key comparison K-11 is therefore that the degree of equivalence is satisfactory for all energies and participants.

### **Acknowledgements**

The participants in this comparison exercise gratefully acknowledge the help of the technical staff of the AMANDE accelerator, in particular A. Martin and M. Pepino. The continuous support in setting up and aligning the detector systems and stable beam conditions were important preconditions for the success of the comparison exercise.

Many thanks are also addressed to staff members of participants who contributed to the success of the experiments at AMANDE facility. In particular, the participants from PTB and NPL are grateful to respectively S. Löb for his support and P. Kolkowski who performed the counting of the activation foils.

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## 8. Appendices

### 8.1. Appendix A: Data Reported by the Participants and Results Evaluated for all Energies

Measurand: fluence per M1-count at 1 m from the target in vacuum in  $\text{cm}^{-2}$

$x_i$ : result of measurement carried out by laboratory  $i$

$u_i$ : combined standard uncertainty of  $x_i$

Table A.1.1: Results for 27.4 keV neutron fluence measurements

Lab $i$	$x_i$ / ( $\text{cm}^{-2}$ )	$u_i$ / ( $\text{cm}^{-2}$ )	$u_i/x_i$ / %
NPL	1.770	0.059	3.33
VNIIM	1.700	0.054	3.18
IRMM	-	-	-
PTB	1.795	0.043	2.40
NIST	1.656	0.093	5.62
AIST	1.821	0.078	4.28
IRSN	1.716	0.060	3.50

Table A.1.2: Results for 565 keV neutron fluence measurements

Lab $i$	$x_i$ / ( $\text{cm}^{-2}$ )	$u_i$ / ( $\text{cm}^{-2}$ )	$u_i/x_i$ / %
NPL	2.194	0.050	2.28
VNIIM	2.059	0.049	2.38
IRMM	-	-	-
PTB	2.107	0.055	2.61
NIST	2.294	0.093	4.05
AIST	2.172	0.048	2.21
IRSN	2.131	0.054	2.53

Additional measurements

PTB LC	2.201	0.040	1.82
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Table A.1.3: Results for 2500 keV neutron fluence measurements

Lab $i$	$x_i$ / ( $\text{cm}^{-2}$ )	$u_i$ / ( $\text{cm}^{-2}$ )	$u_i/x_i$ / %
NPL	2.250	0.050	2.22
VNIIM	2.145	0.052	2.42
IRMM	2.316	0.049	2.10
PTB	2.235	0.038	1.70
NIST	2.238	0.077	3.44
AIST	2.301	0.073	3.17
IRSN	2.275	0.052	2.29

Additional measurements

PTB LC	2.260	0.041	1.81
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Table A.1.4: Results for 17000 keV neutron fluence measurements

Lab <i>i</i>	$x_i$ / (cm <sup>-2</sup> )	$u_i$ / (cm <sup>-2</sup> )	$u/x_i$ / %
NPL	2.371	0.070	2.95
VNIIM	-	-	-
IRMM	-	-	-
PTB	2.480	0.063	2.54
NIST	2.521	0.108	4.28
AIST	2.572	0.113	4.39
IRSN	2.347	0.064	2.73

Additional measurements

NPL foils	2.476	0.082	3.31
PTB LC	2.358	0.051	2.16

Table A.2: Degrees of equivalence with respect to the key comparison reference value

Energy Lab <i>i</i>	27.4 keV		565 keV		2.5 MeV		17 MeV	
	$d_i$ / (cm <sup>-2</sup> )	$U(d_i)$ / (cm <sup>-2</sup> )	$d_i$ / (cm <sup>-2</sup> )	$U(d_i)$ / (cm <sup>-2</sup> )	$d_i$ / (cm <sup>-2</sup> )	$U(d_i)$ / (cm <sup>-2</sup> )	$d_i$ / (cm <sup>-2</sup> )	$U(d_i)$ / (cm <sup>-2</sup> )
NPL	0.018	0.109	0.051	0.083	0.002	0.091	-0.058	0.112
VNIIM	-0.052	0.099	-0.084	0.080	-0.103	0.095	-	-
IRMM	-	-	-	-	0.068	0.088	-	-
PTB	0.043	0.074	-0.036	0.095	-0.013	0.063	0.051	0.094
NIST	-0.096	0.181	0.151	0.177	0.010	0.148	0.092	0.199
AIST	0.069	0.150	0.029	0.078	0.053	0.140	0.143	0.210
IRSN	-0.036	0.112	-0.012	0.092	0.027	0.095	-0.082	0.097

Table A.3.1: Degrees of equivalence between laboratories at 27.4 keV

lab <i>i</i>	NPL		VNIIM		PTB		NIST		AIST		IRSN	
	$D_{ij}$	$U(D_{ij})$										
NPL	-	-	0.070	0.160	-0.025	0.146	0.114	0.220	-0.051	0.196	0.054	0.168
VNIIM	-0.070	0.160	-	-	-0.095	0.138	0.044	0.215	-0.121	0.190	-0.016	0.161
PTB	0.025	0.146	0.095	0.138	-	-	0.139	0.205	-0.026	0.178	0.079	0.148
NIST	-0.114	0.220	-0.044	0.215	-0.139	0.205	-	-	-0.165	0.243	-0.060	0.221
AIST	0.051	0.196	0.121	0.190	0.026	0.178	0.165	0.243	-	-	0.105	0.197
IRSN	-0.054	0.168	0.016	0.161	-0.079	0.148	0.060	0.221	-0.105	0.197	-	-

Table A.3.2: Degrees of equivalence between laboratories at 565 keV

lab <i>i</i>	NPL		VNIIM		PTB		NIST		AIST		IRSN	
	$D_{ij}$	$U(D_{ij})$										
NPL	-	-	0.135	0.140	0.087	0.149	-0.100	0.211	0.022	0.139	0.063	0.147
VNIIM	-0.135	0.140	-	-	-0.048	0.147	-0.235	0.210	-0.113	0.137	-0.072	0.146
PTB	-0.087	0.149	0.048	0.147	-	-	-0.187	0.216	-0.065	0.146	-0.024	0.154
NIST	0.100	0.211	0.235	0.210	0.187	0.216	-	-	0.122	0.209	0.163	0.215
AIST	-0.022	0.139	0.113	0.137	0.065	0.146	-0.122	0.209	-	-	0.041	0.144
IRSN	-0.063	0.147	0.072	0.146	0.024	0.154	-0.163	0.215	-0.041	0.144	-	-

Table A.3.3: Degrees of equivalence between laboratories at 2.5 MeV

lab <i>i</i>	NPL		VNIIM		IRMM		PTB		NIST		AIST		IRSN	
	$D_{ij}$	$U(D_{ij})$												
<b>NPL</b>			0.105	0.144	-0.066	0.142	0.015	0.126	0.012	0.184	-0.051	0.177	-0.025	0.144
<b>VNIIM</b>	-0.105	0.144			-0.171	0.142	-0.090	0.129	-0.093	0.186	-0.156	0.179	-0.130	0.147
<b>IRMM</b>	0.066	0.142	0.171	0.142			0.081	0.123	0.078	0.182	0.015	0.175	0.041	0.147
<b>PTB</b>	-0.015	0.126	0.090	0.129	-0.081	0.123			-0.003	0.172	-0.066	0.165	-0.040	0.129
<b>NIST</b>	-0.012	0.184	0.093	0.186	-0.078	0.182	0.003	0.172			-0.063	0.212	-0.037	0.179
<b>AIST</b>	0.051	0.177	0.156	0.179	-0.015	0.175	0.066	0.165	0.063	0.212			0.026	0.179
<b>IRSN</b>	0.025	0.144	0.130	0.147	-0.041	0.147	0.040	0.129	0.037	0.186	-0.026	0.179		

Table A.3.4: Degrees of equivalence between laboratories at 17 MeV

lab <i>i</i>	NPL		VNIIM		NIST		AIST		IRSN	
	$D_{ij}$	$U(D_{ij})$								
<b>NPL</b>			-0.109	0.188	-0.150	0.257	-0.201	0.266	0.024	0.190
<b>PTB</b>	0.109	0.188			-0.041	0.250	-0.092	0.259	0.133	0.180
<b>NIST</b>	0.150	0.257	0.041	0.250			-0.051	0.313	0.174	0.251
<b>AIST</b>	0.201	0.266	0.092	0.259	0.051	0.313			0.225	0.260
<b>IRSN</b>	-0.024	0.190	-0.133	0.180	-0.174	0.251	-0.225	0.260		

## 8.2. Appendix B: Uncertainty Budgets Reported by the Participants

In this appendix, the uncertainty budget has been limited to the most significant contributions to the total uncertainty. Parameters with a negligible contribution have not been reported here. All relative uncertainties are given with a coverage factor of  $k = 1$ .

### 8.2.1 Uncertainties reported by NPL

Instrument: De Pangher long counter

Parameters	27 keV	565 keV	2.5 MeV	17 MeV
Statistics, monitors, distances, etc.	2.2 %	0.8 %	0.7 %	0.8 %
LC response	2.0 %	2.0 %	2.0 %	2.3 %
LC stability	0.5 %	0.5 %	0.5 %	0.5 %
Monitor stability	0.3 %	0.3 %	0.3 %	0.3 %
Scattered neutron correction	1.3 %	0.2 %	0.4 %	0.7 %
Energy distribution				1.4%
<b>Total uncertainty</b>	<b>3.3 %</b>	<b>2.3 %</b>	<b>2.2 %</b>	<b>3.0 %</b>

### 8.2.2 Uncertainties reported by VNIIM

Instrument: MAP-150 (polyethylene sphere with cylindrical  $^3\text{He}$  central detector)

Parameters	27 keV	565 keV	2.5 MeV
Detector counting	1.0 %	0.4 %	0.3 %
Detector stability	0.4 %	0.4 %	0.4 %
Monitor	1.0 %	0.2 %	0.2 %
Correction for monitor in-scattering	0.9 %	0.3 %	0.3 %
Monitor stability	0.3 %	0.3 %	0.3 %
Detector response	2.0 %	2.0 %	2.0 %
Scattered neutron correction	1.4 %	0.4 %	0.6 %
Covariances	1.0 %	1.0 %	1.0 %
<b>Total uncertainty</b>	<b>3.2 %</b>	<b>2.4 %</b>	<b>2.4 %</b>

### 8.2.3 Uncertainties reported by IRMM

Instrument: Proton recoil telescope

Parameters	2.5 MeV
Statistical uncertainty	0.8 %
Converter hydrogen content	1.0 %
H(n,p) cross-section	1.0 %
Internal geometry	1.3 %
<b>Total uncertainty</b>	<b>2.1 %</b>

## 8.2.4 Uncertainties reported by PTB

- At 27.4 keV with De Pangher long counter

Parameters	27 keV
Statistical uncertainty	0.6 %
Correction for monitor in-scattering	1.0 %
Scattered neutron correction	1.4 %
Long counter response	1.6 %
Distance + Effective centre	0.3 %
<b>Total uncertainty</b>	<b>2.4 %</b>

- At 565 keV with proton recoil proportional counter P2

Parameters	565 keV
Correction of events outside fit region	1.7 %
Scattered neutron correction	1.1 %
Wall effect correction	1.7 %
<b>Total uncertainty</b>	<b>2.6 %</b>

- At 2.5 and 17 MeV with proton recoil telescope T1

Parameters	2.5 MeV	17 MeV
Statistical uncertainty	0.8 %	0.8 %
Converter mass	0.6 %	-
Cross-section and geometry	1.2 %	2.3 %
Distance	0.4 %	0.4 %
Monitor stability	0.3 %	0.3 %
<b>Total uncertainty</b>	<b>1.7%</b>	<b>2.5 %</b>

## 8.2.5 Uncertainties reported by NIST

Instrument: Fission chamber (FC)

Parameters	27 keV	565 keV	2.5 MeV	17 MeV
Calibration with $^{252}\text{Cf}$ source	1.9 %	1.9 %	1.9 %	1.9 %
Fission rate	2.6 %	1.9 %	1.5 %	1.6 %
Monitor rate	0.4 %	0.1 %	0.6 %	0.3 %
Correction for monitor in-scattering	0.4 %	0.2 %	0.4 %	0.2 %
Spectrum averaged cross-section variation / $^{252}\text{Cf}$	4.0 %	2.0 %	2.0 %	3.0 %
Scattering in FC correction	0.4 %	0.8 %	0.4 %	0.8 %
Scattered neutron suppression method	2.0 %	2.0 %	1.0 %	1.0 %
Scattered neutron correction	0.9 %	0.3 %	-	-
Air transmission	0.5%	0.5 %	0.5 %	0.5 %
Neutron energies > 13 MeV	-	-	-	1.0 %
<b>Total uncertainty</b>	<b>5.6 %</b>	<b>4.0 %</b>	<b>3.5 %</b>	<b>4.3 %</b>

8.2.6 Uncertainties reported by AIST

Instrument: Two Bonner Spheres

Parameters	27 keV	565 keV	2.5 MeV	17 MeV
Corrected number of counts	1.5 %	0.3 %	0.4 %	1.1 %
Sphere response	3.9 %	2.1 %	3.1 %	3.9 %
Sphere stability	0.2 %	0.2 %	0.2 %	0.2 %
Monitor stability	0.3 %	0.3 %	0.3 %	0.3 %
Scattered neutron correction	1.1 %	0.4 %	0.5 %	1.2 %
Energy distribution				1.0 %
<b>Total uncertainty</b>	<b>4.3 %</b>	<b>2.2 %</b>	<b>3.2 %</b>	<b>4.4 %</b>

8.2.7 Uncertainties reported by IRSN

Instrument: PLC long counter

Parameters	27 keV	565 keV	2.5 MeV	17 MeV
Standard deviation of measurements (stats + stabilities)	1.4 %	0.3 %	0.4 %	0.6 %
PLC response variation / <sup>252</sup> Cf	1.9 %	1.1 %	0.4 %	1.2 %
PLC calibration at <sup>252</sup> Cf	2.2 %	2.2 %	2.1 %	2.2 %
Effective centre	0.7 %	0.5 %	0.3 %	0.5 %
Scattered neutron correction	1.3 %	0.3 %	0.4 %	0.7 %
Energy distribution	-	-	-	0.6 %
<b>Total uncertainty</b>	<b>3.5 %</b>	<b>2.5 %</b>	<b>2.3 %</b>	<b>2.7 %</b>

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