Pilot study for a CCRI comparison of thermal neutron fluence rate measurements using $^{10}$B ionisation chambers

P Kolkowski

July 2005
NATIONAL PHYSICAL LABORATORY

Pilot study for a CCRI comparison of thermal neutron fluence rate measurements using $^{10}$B ionisation chambers

P Kolkowski

Neutron Metrology Group, DQL
National Physical Laboratory, Teddington, Middlesex, TW11 0LW

ABSTRACT

This report contains details of the experimental procedures used by NPL during a trial run for an international comparison of thermal neutron fluence rate measurement. A description is given of the measurements which were performed at NPL using the thermal column facility. Also described are the analysis technique and the various corrections applied. The characteristics of the transfer instrumentation proposed for this comparison are discussed. The results for the ratio of the measured fluence rate to the transfer detector count rate are given together with a detailed breakdown of the uncertainties.
Crown copyright 2005
Reproduced by permission of the Controller of HMSO

ISSN 1744-0629

National Physical Laboratory
Teddington, Middlesex, United Kingdom, TW11 0LW

Extracts from this report may be reproduced provided that the source is acknowledged and the extract is not taken out of context.

We gratefully acknowledge the financial support of the UK Department of Trade and Industry (National Measurement System Policy Unit)

Approved on behalf of Managing Director, NPL, by Dr T D MacMahon, authorised by the Director, Quality of Life Division
## CONTENTS

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>INTRODUCTION .......................................................... 1</td>
</tr>
<tr>
<td>2</td>
<td>NPL THERMAL NEUTRON FACILITY ........................................ 1</td>
</tr>
<tr>
<td>3</td>
<td>THE TRANSFER INSTRUMENTS AND ELECTRONICS ......................... 3</td>
</tr>
<tr>
<td>4</td>
<td>THERMAL NEUTRON IRRADIATIONS ........................................ 6</td>
</tr>
<tr>
<td>5</td>
<td>ANALYSIS OF TRANSFER INSTRUMENT RESPONSES ...................... 8</td>
</tr>
<tr>
<td>6</td>
<td>RESULTS ........................................................................ 12</td>
</tr>
<tr>
<td>7</td>
<td>CONCLUSIONS AND SUMMARY ............................................. 15</td>
</tr>
<tr>
<td>8</td>
<td>ADDENDUM – COMPARISON PROTOCOL .................................... 17</td>
</tr>
</tbody>
</table>
1. INTRODUCTION

The previous international comparison of thermal neutron fluence [1,2] was carried out more than thirty years ago and most of the laboratories involved used thermal neutron fluence assemblies that employed ($\alpha$, n) neutron sources distributed throughout a suitable moderator. The participants all based their measurements on the activation of thin gold foils and $\beta$-counting to assess the induced activity. Eleven laboratories took part either as $\beta$-counting laboratories, as irradiating laboratories, or as both, and sets of irradiated gold foils were exchanged between them.

The thermal neutron standard fields compared in the last comparison have mostly been replaced by more intense fields and beams, and techniques to measure these fields have greatly improved since 1966 when the last comparison was started. A new comparison was recently proposed under the auspices of Section III of the Comité Consultatif des Rayonnements Ionisants, CCRI, -with the aim of evaluating the degree of equivalence between participating laboratories. It is hoped that, in future, more frequent comparisons can be organised for the benefit of physicists and technicians that make measurements using these relatively new beams and fields employing new techniques of measurement.

The aim of the proposed comparison was to concentrate on thermal neutron fluence rate measurement in thermal neutron beams or beam like geometry and to compare results obtained using transfer instruments supplied by the American national standards laboratory, National Institute of Standards and Technology, NIST. Also supplied by NIST would be the associated electronics and gas flow system. As a trial of the proposed procedures and instrumentation NPL performed a series of measurements in June 2001. This report presents the details and discusses the results of these measurements.

2. NPL THERMAL NEUTRON FACILITY

The NPL Thermal Neutron Facility was designed primarily for producing a precisely-controlled, well-characterised thermal neutron field in a cavity near the centre of a large graphite moderator block, or ‘pile’. Neutrons are produced by bombarding beryllium targets with deuterons. The facility has been described in detail by Ryves and Paul, in 1968 [3]. Another arrangement was added later to the pile which enabled larger devices to be irradiated in a thermal neutron beam and this facility is known as the ‘thermal column’. The thermal column is incorporated above one of the beryllium targets in the graphite moderator block. The column consists of a stainless steel cylinder with cadmium lined sides and a cross sectional area of 1000 cm$^2$. Its length can be adjusted from 1 m to 1.5 m

Fast neutrons are produced by bombarding two beryllium targets with a 2.8 MeV deuteron beam from the NPL 3.5 MV Van de Graaff accelerator. The semi-circular beryllium targets are fixed 160 cm apart in a horizontal beam-line which passes through the entire length of the moderator block. The targets are mounted equi-distant from the centre of the graphite block and a servo control system equalises the fraction of the deuteron beam incident on each target. At the entrance to the moderator block, a tantalum semi-circular plate is fixed vertically in the beam-line, see Figures 1 and 1a. This acts as a “beam-dump” and forms part of the pile control mechanism. The primary neutrons are moderated by the graphite producing a thermal neutron field within the block at a sufficient distance from the targets.
The neutron production in the pile and the fraction of the deuteron beam striking the beryllium targets and tantalum plate is controlled by signals from three ionisation chambers. Two of these chambers are positioned below the beryllium targets underneath the beam-line and the other ionisation chamber is positioned below the central cavity. The ionisation chambers monitor the neutron output from the targets and their response is used to control a servo-system which acts upon vertical and horizontal steering plates in the beam-line. The central ionisation chamber controls the horizontal deflection of the beam and thus the distribution of beam between the targets and tantalum plate. The ionisation chambers below the targets control the vertical deflection of the beam to give approximately equal thermal neutron outputs from the two targets. The servo-system can be preset to give the desired thermal neutron flux output which is controlled by means of the horizontal beam deflection.

In addition to the thermal neutron field produced there is an epithermal component consisting of primary neutrons which have not been moderated to thermal energies. The primary neutrons are produced within the pile from the d-Be reaction, and to a lesser extent, from the d-D reaction and from the action of the deuteron beam on contaminants deposited on the beryllium targets and the tantalum plate. All these reactions contribute to the neutron field, but the ratio of the thermal component to the fast and epithermal component will vary depending on the deuteron beam conditions and the cleanliness of the vacuum system, targets and tantalum plate.

![Fig. 1. Schematic diagram of the NPL Thermal Neutron Standards Facility](image-url)
3. THE TRANSFER INSTRUMENTS AND ELECTRONICS

The transfer instruments used for the pilot exercise were gas-flow type $^{10}$B ionisation chambers. They were chosen for their near perfect $1/v$ cross section shape below 30 keV and the fact that present technology enables extremely well characterised $^{10}$B deposits to be produced [4,5].

Two transfer instruments were supplied with boron coatings of 5 and 50 µg cm$^{-2}$, both 1 cm in diameter, which were deposited on single-crystal silicon wafers about 0.3 mm thick. These silicon wafers formed the cathodes of the two chambers. See Figures 2 and 2a. The blank face of the cathode, i.e. the face without the boron coating, forms one external face of the ionisation chamber. The opposite external face, which forms an earth to the body of the instrument, is also a single-crystal silicon wafer, and this is mounted in parallel with a third single-crystal wafer which forms the anode. The internal side walls are also part of the anode. The external surfaces normal to the thermal neutron beam are constructed of single crystal silicon because of its very low scattering and absorption properties, and the body of the instrument is made of aluminium.

Other items of equipment sent with the ionisation chambers included the electronics, an aperture arrangement, a well-fitting Cd box, and a ground-insulating block. The aperture was made of $^{10}$B-Al and was designed to allow the $^{10}$B layer of the ionisation chambers to be irradiated while shielding the body of the ionisation chamber. A Cd plate was also provided which fitted into this aperture arrangement so that the $^{10}$B layer could also be shielded from the thermal neutron beam. All measurements with this aperture were performed with the Cd plate.
in place. The well-fitting Cd box was supplied with flex-boron pieces to block any holes remaining when the ionisation chambers were inserted. Finally the ground-insulating block was essentially a piece of polyethylene which allowed improved insulation of the chambers.

![Diagram of ionisation chamber](image1)

**Fig. 2. Cross section view of the NIST ionisation chamber used as the transfer instrument**

![Diagram of ionisation chamber](image2)

**Fig. 2a. Plan view of the NIST ionisation chamber used as the transfer instrument**
The transfer instruments were removed from their desiccator cabinet, connected to a gas flow system and a supply of high quality counting gas (90% argon and 10% methane) was continuously passed through the instrument at the same time as the electronics were connected and the bias applied, about two hours prior to irradiation.

The electronics, shown schematically in Figure 3, were connected so that a pulse height distribution could be acquired at the same time as a scalers recorded the output pulses from the single channel analyser (TSCA) an Ortec model 551. Pulses from the TSCA were input to NPL Width/Delay units and then to the NPL scalers.
The high voltage and amplifier settings were adjusted according to the values given in the protocol. The high voltage was set to +100V and the amplifier gain settings to ‘maximum’ because of the small amplitude pulses from the ionisation chamber.

A PC based, Ortec MCA card was used to acquire the pulse height distribution from the ionisation chambers. The upper level discriminator (ULD) was set at the minimum \( V_U \) between the two major peaks of the pulse height distribution and the lower level discriminator (LLD) set at \( V_L = V_U /3 \). The peaks in the pulse height distribution from the 5\( \mu \)g ionisation chamber were well defined and the procedure for discriminator setting described in the protocol was followed. However, for the heavier boron deposit, 50\( \mu \)g, the LLD could not be set at the positioned dictated by the protocol as the cut-off would have been into the noise region of the pulse height distribution. The LLD was therefore set to the minimum in the valley between the noise region and the \(^7\text{Li}\) peak, and the ULD was set to the mid-point of the trough between the \(^7\text{Li}\) peak and the alpha peak.

4. THERMAL NEUTRON IRRADIATIONS

The transfer instruments were irradiated in the ‘thermal column’ while the thermal neutron fluence was simultaneously monitored by a \(^{235}\text{U}\) fission chamber positioned in the graphite between the bottom of the thermal column and the beryllium target. This monitor was calibrated in terms of the fluence at two reference positions (1 and 1.5 m) in the thermal column, using the gold activation technique \([6,7]\). Gold foil calibrations of the monitor are performed on a regular basis at, or close to, the position at which artefacts are to be calibrated in order to reduce the uncertainty in the cadmium ratio when measured at a significantly different height in the thermal neutron beam. The sensitive areas of the transfer instruments were positioned close to these two reference positions.

The 5\( \mu \)g and 50\( \mu \)g transfer instruments were supported in the thermal column by a thin, insulated aluminium plate which enabled the transfer instruments to be positioned centrally in the thermal neutron beam with their major axis perpendicular to the beam. The 50\( \mu \)g detector was irradiated at both positions in the thermal neutron beam and the 5\( \mu \)g instrument at the 1.5 m position only. The 5\( \mu \)g instrument stopped working before measurements could be carried out at the second position. Figures 4a, 4b and 4c show details of the positioning of the transfer instruments for the ‘free-in-air’ irradiation, with the aperture arrangement, and enclosed in the cadmium box. Irradiations were performed for a combination of these configurations.

Prior to the irradiations the column was evacuated to a pressure of 3 x 10\(^{-2}\) mbar. The active cathode face was positioned within 1 mm of the centre of the beam in the horizontal plane at the 1.5 m and 1.0 m reference positions. The vertical position of the sensitive surface of the transfer instruments when irradiated ‘free-in-air’ was 5.5 mm above the 1.5 m and 1.0 m reference positions. When the cadmium box and cadmium aperture were used to shield the transfer instruments the distance above the two reference positions was increased to 6.5 mm and 15.0 mm respectively.

The 50\( \mu \)g instrument was irradiated, both bare and cadmium-covered, on a number of separate days to check consistency of positioning and the reproducibility of the Van de Graaff deuteron beam conditions.
Fig. 4a. Schematic diagram of the ionisation chamber positioned for 'free-in-air' irradiation

Fig. 4b. Schematic diagram of the ionisation chamber with 30 mm, $^{10}\text{B} - \text{A aperture plate and Cd foil in the irradiating position}$

Fig. 4c. Schematic diagram of the ionisation chamber enclosed in cadmium box and positioned for irradiation
The response of a cylindrical BF₃ proportional counter tube, normalised to the fission chamber monitor, was used to measure the change in thermal neutron fluence with height at points just above the 1.5 m and 1.0 m positions so that a small correction to the transfer instrument responses could be made for the slightly different heights of the boron coated surface above the 1.5 m and 1.0 m positions. It was assumed that the thermal neutron spectrum does not change significantly when the largest correction is for a height difference of 15 mm.

The fluence received during each irradiation was determined by the fixed ²³⁵U fission counter monitor whose response was calibrated in terms of fluence rate measured free-in-air at the 1.5 m and 1.0 m positions using the gold foil activation technique. For the fluence rate measurement, a pair of gold foils were irradiated at each reference position before and after the transfer instrument irradiations, with one foil enclosed in a 1 mm thick cadmium metal box to enable a correction for the epi-cadmium neutron component to be made.

5. ANALYSIS OF TRANSFER INSTRUMENT RESPONSES

The quantity to be compared for this comparison, as in the earlier comparison, is the conventional thermal neutron fluence rate

\[ \phi_0 = n_{th}v_0 \]

where
- \( \phi_0 \) is the thermal neutron fluence rate
- \( n_{th} \) is the neutron density below the cadmium cut-off energy
- \( v_0 \) is the reference thermal neutron velocity of 2200 ms\(^{-1} \)

The protocol requires that the quantity to be reported is the instrument calibration coefficient given by:

\[ \frac{\phi_0}{CR} \]

where
- CR is the count rate of the transfer instrument corrected for the small epithermal response

All the present measurements were performed at almost the same two heights (1.5 m and 1.0 m) in the thermal column beam and close to the height at which the fluence measurements with the gold foils were performed to calibrate the fission chamber monitor in terms of \( n_{th}v_0 \) (as in the Westcott convention) per unit fission chamber count.

Data from gold foil measurements at different heights have shown that the cadmium ratio, and hence the neutron spectrum, and thus the temperature of the thermal peak, varies with height. For these measurements the assumption is made that the effects of changes in the thermal spectrum on the quantity measured were negligible for the range of heights involved. The largest correction was for a height difference of 15 mm above the reference positions.

Timed irradiations were carried out whilst simultaneously recording the fission counter monitor events and the pulse height distribution from the ionisation chamber. The fluence at the reference position was calculated from the dead time corrected monitor counts using the
calibration factor obtained from the gold activation. A small correction was then applied for the height of the boron coated surface above the reference position.

The counts in the pulse height distribution were integrated between the upper and lower level discriminators (Peak1), above the upper level discriminator (Peak 2) and also all the counts above the lower level discriminator. The integrated counts obtained for each of these regions was then corrected for the dead time of the MCA and recorded. This procedure was repeated for each irradiation of the ionisation chambers. Examples of the spectra obtained are shown in Figures 5 a to 5e.

![Figure 5a. Pulse height spectrum from 5 µg ionisation chamber ‘free in air’ showing the upper and lower discriminator positions.](image)

The response of the ionisation chambers in each arrangement was calculated in terms of count rate per unit fluence rate.
Figure 5b. Pulse height spectrum of 5 µg ionisation chamber with $^{10}\text{B/Al + Cd}$ aperture.

Figure 5c. Pulse height spectrum from 50 µg ionisation chamber ‘free in air’
Figure 5d. Pulse height spectrum from 50 µg ionisation chamber with $^{10}$B/Al + Cd aperture.

Figure 5e. Pulse height spectrum from 50 µg ionisation chamber enclosed in Cd box.
6. RESULTS

A summary of the irradiations performed is given in Table 1. Three sets of measurements were made: for the 5 µg ionisation chamber at the 1.5 m reference position, and for the 50 µg ionisation chamber at both the 1.5 m and the 1 m reference positions.

Table 1. Details of the irradiations performed

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Ionisation chamber configuration</th>
<th>Irrad. time (s)</th>
<th>Fission chamber counts x 10^7</th>
<th>Total Fluence x 10^7 cm^-2</th>
<th>Fluence rate (φ₀) x 10^4 cm^-2 s^-1</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Free-in-air</td>
<td>1900</td>
<td>1.284</td>
<td>4.713</td>
<td>2.480</td>
</tr>
<tr>
<td>2</td>
<td>Free-in-air</td>
<td>3100</td>
<td>2.109</td>
<td>7.737</td>
<td>2.496</td>
</tr>
<tr>
<td>3</td>
<td>On ^10^B Al, Cd aperture</td>
<td>4100</td>
<td>2.812</td>
<td>10.18</td>
<td>2.483</td>
</tr>
<tr>
<td>4</td>
<td>Free-in-air+Al &amp; poly mounting block</td>
<td>900</td>
<td>0.611</td>
<td>2.242</td>
<td>2.491</td>
</tr>
<tr>
<td>5</td>
<td>Free-in-air+Al &amp; poly mounting block</td>
<td>1600</td>
<td>1.088</td>
<td>3.992</td>
<td>2.495</td>
</tr>
<tr>
<td>6</td>
<td>On ^10^B Al, Cd aperture</td>
<td>2200</td>
<td>1.492</td>
<td>5.404</td>
<td>2.456</td>
</tr>
<tr>
<td>7</td>
<td>Free-in-air+Al &amp; poly mounting block</td>
<td>2400</td>
<td>1.620</td>
<td>5.946</td>
<td>2.478</td>
</tr>
<tr>
<td>8</td>
<td>On ^10^B Al, Cd aperture</td>
<td>2300</td>
<td>1.566</td>
<td>5.672</td>
<td>2.466</td>
</tr>
<tr>
<td>9</td>
<td>In Cd box with Boroflex plugs</td>
<td>2700</td>
<td>1.826</td>
<td>6.690</td>
<td>2.478</td>
</tr>
<tr>
<td>10</td>
<td>Free-in-air</td>
<td>1500</td>
<td>1.081</td>
<td>8.148</td>
<td>5.432</td>
</tr>
<tr>
<td>11</td>
<td>Free-in-air+Al &amp; poly mounting block</td>
<td>1400</td>
<td>1.004</td>
<td>7.569</td>
<td>5.407</td>
</tr>
<tr>
<td>12</td>
<td>On ^10^B Al, Cd aperture</td>
<td>1900</td>
<td>1.365</td>
<td>10.10</td>
<td>5.315</td>
</tr>
<tr>
<td>13</td>
<td>In Cd box with Boroflex plugs</td>
<td>1900</td>
<td>1.367</td>
<td>10.28</td>
<td>5.412</td>
</tr>
</tbody>
</table>

During the three sets of measurements the thermal fluence rates were quite consistent, at a value of about 2.48 x 10^4 cm^-2 s^-1 at the 1.5 m reference position, and 5.4 x 10^4 cm^-2 s^-1 at the 1 m reference position. The largest range of the values for any set was about 2% for the fluence rates at 1 m. (These variations do not affect the eventual results since the ionisation chamber count rates are normalised to the fluence value received.) Measurements were performed for four different configurations: free-in-air, free-in-air with aluminium and polyethylene mounting block, screened by the ^10^B Al Cd aperture, and fully enclosed in the Cd box with boroflex plugs to fill any apertures, although not all configurations were used for each of the three sets of measurements.

For all the irradiation conditions listed in Table 1 the counts in the three regions of interest in the ionisation chamber spectra are given in Table 2 together with values for the fluence rate, φ₀, divided by the ionisation chamber count rates, CR, for each of these three regions.
Table 2. Total counts taken from MCA spectra, count rates corrected for MCA live time, and corrected count rate divided by fluence rate. The $^7$Li recoils are designated as peak 1 and the alpha particles as peak 2.

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Counts above LL</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 1</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 2</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
</tr>
</thead>
<tbody>
<tr>
<td>1§</td>
<td>39331</td>
<td>20.70</td>
<td>1.198</td>
<td>19960</td>
<td>10.50</td>
<td>2.361</td>
<td>19376</td>
<td>10.20</td>
<td>2.433</td>
</tr>
<tr>
<td>2</td>
<td>62325</td>
<td>20.73</td>
<td>1.204</td>
<td>31603</td>
<td>10.51</td>
<td>2.374</td>
<td>30725</td>
<td>10.22</td>
<td>2.442</td>
</tr>
<tr>
<td>3</td>
<td>1613</td>
<td>0.4167</td>
<td>59.52</td>
<td>938</td>
<td>0.2423</td>
<td>102.0</td>
<td>677</td>
<td>0.1748</td>
<td>142.9</td>
</tr>
</tbody>
</table>

5 µg ionisation chamber @1.5 m reference position

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Counts above LL</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 1</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 2</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
</tr>
</thead>
<tbody>
<tr>
<td>4§</td>
<td>178088</td>
<td>197.9</td>
<td>0.1259</td>
<td>159928</td>
<td>103.5</td>
<td>0.2411</td>
<td>156133</td>
<td>101.1</td>
<td>0.2469</td>
</tr>
<tr>
<td>5</td>
<td>316061</td>
<td>204.6</td>
<td>0.1220</td>
<td>3638</td>
<td>1.711</td>
<td>14.35</td>
<td>3601</td>
<td>1.694</td>
<td>14.49</td>
</tr>
<tr>
<td>6</td>
<td>7231</td>
<td>3.401</td>
<td>7.220</td>
<td>223708</td>
<td>95.60</td>
<td>0.2591</td>
<td>231384</td>
<td>98.88</td>
<td>0.2506</td>
</tr>
<tr>
<td>7</td>
<td>455092</td>
<td>194.5</td>
<td>0.1274</td>
<td>3668</td>
<td>1.637</td>
<td>15.06</td>
<td>3947</td>
<td>1.761</td>
<td>14.01</td>
</tr>
</tbody>
</table>

50 µg ionisation chamber @1.5 m reference position

<table>
<thead>
<tr>
<th>Run No.</th>
<th>Counts above LL</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 1</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
<th>Counts in peak 2</th>
<th>Corrected rate* (CR) x 10³</th>
<th>$\phi_0$/CR x 10³</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>619964</td>
<td>431.1</td>
<td>0.1260</td>
<td>296872</td>
<td>206.4</td>
<td>0.2631</td>
<td>323096</td>
<td>224.7</td>
<td>0.2418</td>
</tr>
<tr>
<td>11</td>
<td>585031</td>
<td>433.7</td>
<td>0.1243</td>
<td>281758</td>
<td>208.9</td>
<td>0.2572</td>
<td>303270</td>
<td>224.8</td>
<td>0.2405</td>
</tr>
<tr>
<td>12</td>
<td>14103</td>
<td>7.690</td>
<td>6.911</td>
<td>6760</td>
<td>3.686</td>
<td>14.41</td>
<td>7344</td>
<td>4.004</td>
<td>13.28</td>
</tr>
<tr>
<td>13</td>
<td>11655</td>
<td>6.341</td>
<td>8.532</td>
<td>5720</td>
<td>3.112</td>
<td>17.39</td>
<td>5935</td>
<td>3.229</td>
<td>16.75</td>
</tr>
</tbody>
</table>

* Corrected for the dead-time of the MCA or the scalers.
§ MCA misbehaving, used data from scalers.
¶ MCA seized up, used data from scalers.

Several repeat measurements were made for the same configuration, e.g. runs 1 and 2, 5 and 7, 6 and 8, and in general these showed good reproducibility. For runs 1 and 2, and for runs 6 and 8 the agreement is within the statistical uncertainties. For runs 7 and 8 the differences range from 1.5% to 7.2%, depending on the region of the spectrum. These differences are way outside expected statistical variations, and suggest possible problems with defining the counting regions, e.g. variations of the gain or of the discriminator settings.

For the 50 µg chamber a change of the LLD by one channel would result in a change of 4 to 9% in the number of events recorded for peak 1 and a 0.4% change in the total number of events recorded above the LLD. A change in the ULD of one channel results in a change of approximately 0.1% in the number of events recorded in peak 2.

For the 5 µg chamber which has superior resolution the effect of a one channel change in the discriminator positions is 0.1% or less on the number of events recorded for both peaks.

Where more than one measurement was carried out with the aluminium and polyethylene mounting block the weighted mean value of the count rate (CR) was calculated. The 5 µg ionisation chamber failed before measurements could be made with the aluminium and
polyethylene mounting block attached and in this case the weighted mean of two free-in-air measurements were used.

The count rates of the chambers were normalised to \( \phi_0 \) so that the epithermal component could be subtracted and the value obtained expressed as \( \phi_0/CR \).

Table 3. Ionisation chamber calibration coefficients corrected for the epithermal component

<table>
<thead>
<tr>
<th></th>
<th>All Counts above LLD</th>
<th>Counts in Peak 1 ((^7)Li)</th>
<th>Counts in Peak 2 ((\alpha))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \phi_0/CR \times 10^2 \text{ cm}^{-2} )</td>
<td>( \phi_0/CR \times 10^2 \text{ cm}^{-2} )</td>
<td>( \phi_0/CR \times 10^2 \text{ cm}^{-2} )</td>
</tr>
<tr>
<td>50 ( \mu g ) @ 1.5 m Cd aperture only</td>
<td>1.255</td>
<td>2.544</td>
<td>2.532</td>
</tr>
<tr>
<td>50 ( \mu g ) @ 1.5 m ‘fully enclosed in Cd’</td>
<td>1.248</td>
<td>2.529</td>
<td>2.517</td>
</tr>
<tr>
<td>50 ( \mu g ) @ 1.0 m Cd aperture only</td>
<td>1.275</td>
<td>2.650</td>
<td>2.457</td>
</tr>
<tr>
<td>50 ( \mu g ) @ 1.0 m ‘fully enclosed in Cd’</td>
<td>1.274</td>
<td>2.642</td>
<td>2.447</td>
</tr>
<tr>
<td>5 ( \mu g ) @ 1.5 m Cd aperture only</td>
<td>12.26</td>
<td>24.24</td>
<td>24.80</td>
</tr>
<tr>
<td>Ratio of 50 ( \mu g ) to 5 ( \mu g ) chamber responses @ 1.5 m with Cd aperture</td>
<td>0.102</td>
<td>0.105</td>
<td>0.102</td>
</tr>
</tbody>
</table>

The first measurements were carried out with the 5 \( \mu g \), all on the same day, and followed by the 50 \( \mu g \) at the 1.5 m reference position. The latter measurements took place over three days. The final measurements were carried out with the 50\( \mu g \) positioned at the 1.0 m reference position and completed on the same day.

Better statistics were attained for the measurements at 1.0 m, which were all carried out on the same day. It would seem that any inconsistencies in the results above are most likely to be due to gain changes or shifts in the discriminator positions.

Although no other sets of measurements were available to compare with the present results, a preliminary estimate of the expected \(^{10}\)B(\(n, \alpha\))\(^7\)Li rate was provided by NIST [8] in order to determine whether adequate count rates would be achieved at NPL. Table 4 provides a comparison of the present measurements with calibration coefficients derived from those predictions.
Table 4. Comparison of calibration coefficients measured at NPL with those predicted by NIST for these instruments.

<table>
<thead>
<tr>
<th>Measurement configuration</th>
<th>Calibration coefficient (cm(^{-2}))</th>
<th>Ratio Measured Predicted</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Measured at NPL</td>
<td>NIST prediction</td>
</tr>
<tr>
<td>50 µg chamber at 1.5 m</td>
<td>252.9</td>
<td>249.4</td>
</tr>
<tr>
<td>50 µg chamber at 1.0 m</td>
<td>264.2</td>
<td>249.4</td>
</tr>
<tr>
<td>5 µg chamber at 1.5 m</td>
<td>2454</td>
<td>2508</td>
</tr>
</tbody>
</table>

One of the major uncertainty in measuring the calibration coefficient is that associated with the fluence measurement using gold foils at the reference position, normalised to the fission chamber monitor, is 1.4% at the 95% confidence level. This value applies only to the fluence as determined according to the Westcott convention.

The uncertainty in the ion chamber readings has a number of components. The statistical uncertainties are relatively small; the values for ‘without-Cd’ measurements being less than 1% even in the two worst cases (the measurements for the 5 µg chamber), and being considerably less in most other cases. Dead-time corrections and hence the uncertainties in these corrections were small. There must inevitably be uncertainties associated with the discriminator settings, but in view of the other problems these were not investigated. It is possible that there is some evidence of variations in discriminator settings from a comparison of the data given in Table 3 for the 1.5 m and the 1.0 m positions for the 50 µg chamber. For the sum of both peaks the calibration coefficient is, on average, 1.8% higher at the 1.0 m position. For peak 1 the 1.0 m results are, on average, 4.3% higher than the 1.5 m position, but the results for peak 2 are on average 2.9% lower for the 1.0 m position than the 1.5 m position.

From Table 3 it can be seen that the chamber calibration coefficients are on average slightly higher for the Cd aperture measurements, by on average 0.4%. This is the opposite to what would be expected if thermal neutrons were incident on the chamber from any direction other than from below – see Figures 4(a) – (c).

All the results are quoted for the sub-cadmium-cut-off fluence in the Westcott convention. If the ‘true’ thermal fluence value had been used the calibration factors would have been larger by a factor of 1.193, and the fluence uncertainty would have an additional component of ± 2% [9].

7. CONCLUSIONS AND SUMMARY

The comparison of the chamber responses do not agree with the NIST calculations and the calibration coefficient was not consistent at different heights in the thermal column

Repeat measurements carried out on the same day showed reasonable reproducibility however gain changes were noticed and the multi channel analyser data was used in preference to that from the scalars. The reason for the gain changes was not ascertained.
It seems most likely that there was an intermittent fault that caused some inconsistencies in the measurements but these could not be investigated because of time constraints as the ionisation chambers were required for tests in the USA.

It is not known why the 5 µg ionisation chamber failed nor if problems were subsequently found to exist in the 50 µg ionisation chamber after returning to the USA but the indications are that the ionisation chambers are too fragile to survive many long journeys.

**Advantages of the ionisation chambers**

- Well defined geometry and characterisation of the boron deposits.
- Good resolution for the 5 µg ionisation chamber: Setting the discriminators is relatively easy as the peaks are well defined which is not the case with the 50 µg ionisation chamber.
- Accuracy and ease of positioning: Precise information about the dimensions and a circular shape with parallel sides enable relatively accurate positioning.
- Relatively simple electronics.
- Relatively simple data processing.

**Disadvantages**

- Fragile detector walls: Only 0.3 mm thick silicon
- Stability of the boron deposits: The boron coatings are very susceptible to moisture
- The 5 µg ionisation chamber has relatively low sensitivity
- The 50 µg ionisation chamber has relatively poor resolution
- Relatively low amplitude pulse produced. Attendant problems of electronic noise and earthing: All amplifier gain settings were at maximum.

**Recommend**

- Response much easier to use as a quantity in the analysis and then to convert to conversion coefficient at the end.

**ACKNOWLEDGEMENTS**

The author wishes to thank A Bennett for his operation of the NPL Van de Graaff and his assistance in operating the servo-control system of the NPL Thermal Neutron Facility. The work was funded by the National Measurement System Policy Unit of the UK Department of Trade and Industry.
8. ADDENDUM – COMPARISON PROTOCOL

The Consultative Committee for Ionizing Radiation, Section III (Neutron Measurements)

Protocol for Comparison of Fluence Rate Measurements in Thermal Neutron Beams

INTRODUCTION

One of the earliest inter-laboratory comparisons carried out under the auspices of what is now Section III was a comparison of thermal neutron fluence rate measurements. The results of the comparison were published [1,2] in 1970, more than 30 years prior to the beginning of this comparison. The current interest in demonstrating the extent of equivalence of measurements of Section III participants requires that these comparisons be repeated more frequently.

The present comparison differs from the previous one in two ways: the present comparison is focused on fluence rate measurements in thermal neutron beams or beam-like thermal neutron fields, such as the neutron field emanating into the void surrounding a moderated isotopic source, rather than at a location inside a moderating medium or inside a cavity within a moderating medium; and the measurements will be compared relative to a set of transfer detectors, rather than by exchange of activated foils. As in the earlier comparison, the quantity to be compared is the conventional thermal neutron fluence rate \( N_0 = n_{th} v_0 \), where \( n_{th} \) is the neutron density below the cadmium cut-off energy, and \( v_0 \) is the reference thermal neutron speed of 2200 m/s. The quantity \( N_0 \) was called the “flux density” in the publications of the previous comparison, but the International Commission on Radiation Units has since then recommended the term “fluence rate” for this quantity. The quantity \( N_0 \) is also called the “2200 m/s fluence rate” and is often referred to as the “capture flux” in the published specifications of research reactors. Appendix 1 gives further details about the quantity to be reported.

Transfer Detectors

As transfer instruments, NIST will prepare and maintain a set of three flow-type \(^{10}\text{B}\) ionization chambers; and NIST will also supply a basic electronic system and some components of the argon(90%)-methane(10%) gas flow system. The three ionization chambers will each have a 1 cm diameter deposit of \(^{10}\text{B}\), with nominal areal density of 0.5, 5.0, or 50 \(\mu\)g cm\(^{-2}\), respectively. Any problem with the stability of the boron deposits will be addressed by comparison with a \(^{235}\text{U}\) deposit in a monochromatic beam at NIST, before and after the use by each participant. The statistical precision (one standard deviation) of these comparisons at NIST will be better than 0.1%. The stability of the \(^{235}\text{U}\) deposit is assured by alpha counting.

The choice of \(^{10}\text{B}\) as the active nuclide of the transfer detector is based on the nearly perfect \(1/v\) cross section shape below 30 keV, the availability of very high quality deposits, and the relative ease of shipment to all participating countries.

Further details concerning the transfer instrument set are given in Appendix 2.
Thermal Neutron Beams and Beam-Like Thermal Neutron Fields

At most neutron research facilities around the world, the great majority of users employ neutron beams for materials science experiments. Typical 2200 m/s fluence rates for primary thermal or cold neutron beams at these facilities are of the order of $10^8$ to $10^9$ neutrons cm$^{-2}$ s$^{-1}$, while monochromatic beams from crystal monochromators have fluence rates typically in the range $10^5$ to $10^6$ neutrons cm$^{-2}$ s$^{-1}$. Measurements of these fluence rates are frequently made to evaluate the performance of cold sources, filters, collimators, guides, monochromators, and detector arrays. Analytical chemistry users still employ in-pile irradiations for activation analysis, but most of these are done at fluence rates which are 3 to 7 orders of magnitude higher than those of the standard fields which were compared in the earlier Section III comparison. The comparison in beam or beam-like geometry can be applied with little change to the foil activation techniques which are frequently employed for in-pile measurements as well as for beam measurements.

Since thermal neutrons are strongly absorbed and only weakly scattered by several conveniently available materials, it is possible to make shields and apertures which collimate the field to be measured without significantly perturbing it. This ability to collimate the beam (or other divergent field) makes it possible to shield the more massive components of an active detector to prevent scattering perturbation of the beam to be measured. It is similarly possible to shield the detector from thermal neutrons scattered by laboratory walls and structures.

PARTICIPATING LABORATORIES AND TIMETABLE FOR MEASUREMENTS

Tentative expressions of interest in participation were given by seven laboratories: CIAE, ETL, IRMM, NIST, NPL, PTB, and VNIIM. Both the NPL and the CIAE asked to be included as early as possible in the schedule.

It should be possible to begin the series of measurements by December, 2000. The transfer detector system could be kept by each participant for two to four months, depending on the number of different beams to be measured by the participant. The three different transfer detectors should cover at least the range $10^3$ – $10^8$ cm$^{-2}$ s$^{-1}$, and perhaps a bit more, depending on local background rates and dead time correction techniques. Each participant is encouraged to include as many points within this 5 orders of magnitude as his or her time allows.

A detailed timetable and additional information about the participants are given in appendix 3. (Not included here.) This Appendix will be updated throughout the course of the comparison, as needed. All participants will be notified if unexpected delays are encountered which will require changes in the comparison schedule.

REPORTING RESULTS AND UNCERTAINTY ANALYSIS

All results will be sent to the Coordinator, David M. Gilliam, at NIST, who will compile a report. Since the Coordinator represents a participating laboratory, all NIST results will be filed with the BIPM Ionizing Radiations Section before NIST accepts results from any other participants.
The report of the results should include a brief description of the facility, including beam temperature or mean energy, if known, and any sort of beam filtration. These beam energy details are most needed for cold neutron beams, for which absorption within the boron deposit is most significant. NIST will correct the transfer detector results for this factor.

The report of results should include a listing of all significant uncertainty components. Participants are urged to follow the *Guide to The Expression of Uncertainty in Measurement* [3] in estimating and reporting their uncertainty, using $k = 1$ (corresponding to one standard deviation).

**PUBLICATION OF THE RESULTS**

The results will be submitted for publication in *Metrologia* after circulation to all participants and approval by members of Section III. The separate results will be ordered by $N_{th}$, labelled by laboratory acronyms, and shown as a scatter plot in which the weighted mean inter-laboratory value of $N_{th}/CR$ (see Appendix 1) is normalized to zero with individual results and their uncertainties plotted as percentage deviations from this axis. The weighting will be done on the basis of inverse squared uncertainties as reported by the participants and/or as accepted by Section III.

The results from the three separate ranges covered by the three different transfer chambers will be plotted separately first. Then the $N_0/CR$ values for all three detectors over the 5 orders of magnitude in fluence rate will be displayed on a single plot based on normalization of the three detectors to a common scale. This normalization will be based on characterization measurements at NIST and any at other interested laboratories.

Interim reports may also be sent to Section III members showing relative agreement of the results so far on hand. So long as the current mean of $N_0/CR$ is normalized to zero in the interim reports, no loss of independence or “blindness” in the comparisons would occur.

**TRANSPORTATION ARRANGEMENTS**

NIST will pay for shipping to the participating laboratory, and the participating laboratory will pay for the return shipment to NIST. More detailed specifications for the shipping arrangements are given in Appendix 4. *(Not included here.)*

**REFERENCES**


APPENDIX 1. The Quantity to be Reported

As noted in the Introduction, the quantity whose measurement is to be compared is $N_0 = n_0 v_0$, where $n_0$ is the neutron density (neutrons/cm$^3$) with energies below the cadmium cut-off energy, and $v_0$ is the reference thermal neutron speed of 2200 m s$^{-1}$, exactly the same as in the previous comparison. The cadmium cutoff energy is taken to be 0.4 eV. For well-thermalized beams, the precise value of the Cd cutoff energy is not important; for less well-thermalized beams, a careful correction to a specific cutoff energy is required and larger uncertainties and/or more extensive work may be entailed in making this correction.

The quantity to be reported by each participant is $N_0 / CR$, the ratio of the measured fluence rate $N_0$ to the count rate CR of the transfer detector (with CR also corrected for its small epi-cadmium response). The ratio $N_0 / CR$ and an estimate of its uncertainty is to be reported for each beam or beam-like field measured. The measured fluence rate $N_0$, as defined above, may be determined by whatever means the participant chooses. Some laboratories will use foil activation and others may use active detectors of some kind. The reported value of CR must be from exposure of the transfer detector such that the boron deposit is positioned in “effectively” the same position where $N_0$ was measured. If $N_0$ and CR are not measured simultaneously, and if the neutron source is significantly time-varying, then some sort of additional run-to-run monitor must be employed to normalize the $N_0$ and CR data. The word “effectively” is inserted above to account for cases in which the volume of space over which $N_0$ is measured cannot be made to coincide with the position of the boron deposit in the transfer detector and for cases in which it may be more accurate not to try to make those measurement positions coincide exactly. For example, if the volume in space over which $N_0$ is measured is larger than the boron deposit, then the value of CR must be determined by experimentally averaging the response of the transfer detector over the larger volume or by making an equivalent analytic correction to the boron response over some part of that volume.

Another very important case in which it is usually more accurate not to try to measure $N_0$ and CR in exactly the same volume in space is the case in which $N_0$ is determined by the foil activation method. In this case, it would usually better to employ the special recessed cap which permits positioning a 1 cm diameter activation foil very near the boron deposit without touching the fragile silicon backing. Then two foil irradiations are done, one with the activation foil nearer to the neutron source, and one with the boron deposit nearer to the neutron source, i.e. with the assembly rotated 180$^\circ$. The (geometric) average of the two measured values of $N_0 / CR$ will be “automatically” corrected almost perfectly for beam divergence and mutual shielding of the activation foil by the boron layer and vice versa. [Cadmium blocked runs must also be done to subtract any epithermal neutron contributions.] If CR is measured over the entire foil irradiation period and the half-life of the induced activity is very long compared to the irradiation time, then no run-to-run monitor is needed for this method, even for a time-dependent source.

It is requested that both bare and Cd covered results (as well as the Cd-corrected results) be reported explicitly for both the transfer detectors and the participant's detectors.
APPENDIX 2. Transfer Detector Details

The $^{10}$B deposits were all prepared by vacuum deposition onto single-crystal silicon disks (wafers), 49.9 mm in diameter and about 0.3 mm thick. The preparation was done by the Institute for Reference Materials and Measurements in Geel, using a special planetary rotation system during the deposition to produce very uniform deposits with very nearly perfect edges.

The detector is constructed such that the position of the $^{10}$B deposit can easily be determined accurately, $\pm 0.1$ mm along the beam axis and $\pm 0.3$ mm perpendicular to that axis. The blank side of the silicon disk comprises one of the external faces of the detector. This face is recessed about 3.7 mm and must not be touched by measuring instruments. What one needs to measure is the distance to the inside face of this disk, where the thin $^{10}$B deposit is located. That face is optically flat and is precisely 4 mm inside the rim of the ionization chamber. A very flat 1 mm thick cap is provided to mount on the chamber rim so that mechanical measuring instruments can touch this face and determine the $^{10}$B position by adding 5 mm. A cross scratched on the center of the cap face gives the other coordinates of the deposit.

Problems of stability of the $^{10}$B deposits in humid conditions have been reported by to Section III by NIST, but these problems were small, especially for the heavier deposits. The boron stability will be carefully monitored by NIST before and after each use of the ionization chambers, and it is believed that the problem can be reduced to an easily managed level by storing the chambers in desiccated cases when not in use, by asking all participants to use high quality argon(90%)-methane(10%) “P-10” gas, and by using a drying chamber for the P-10 gas line.

NIST will supply the following equipment and references:

- three $^{10}$B ionization chambers,
- a small NIM crate with high voltage supply, amplifier, dual integral discriminators / single channel analyzer (SCA), and a dual channel pulse counter-timer
- a preamplifier and cables
- a ground-insulating mounting block and an aluminum mounting plate
- gas tubing and desiccator chamber for assuring the dryness of the P-10 gas
- a dry-storage case for the ionization chambers
- a 30 mm aperture of $^{10}$B-Al and Cd to shield the body of the ionization chamber
- a 1 mm thick Cd foil for determining the correction for epithermal reaction rates
- caps for mechanical measurements, alignment, and protection of the Si wafers,
- a recessed, thin aluminum cap for holding activation foils near the deposit backing (silicon wafer)
- a well-fitting Cd box, and
- copies of relevant sections of the electronics manuals.

The 30 mm aperture has a mounting ring attached that may be used to mount the aperture directly onto the cathode face (smaller diameter face) of the detector. A holder for a 1 mm Cd foil is attached to the opposite side of the 30 mm aperture plate for determining the Cd ratio of the beam. (If the foil holder is used, the aperture must be supported in some other way.) Any shielding of the detector from ambient neutrons outside of the beam must be provided by the participating laboratory.
(Although there is no foreseen need for mounting the 30 mm aperture on the anode face, an adapter ring is provided to permit this if it should be needed for some reason. The thin aluminum mounting plate may be used to support the chamber if the 30 mm aperture is mounted on the anode face of the detector. An insulating mount must still be used in addition.)

A close-fitting Cd box is provided for background checking and for determination of the Cd ratio when using the activation foil method. The aluminum mounting plate can be used when the chamber is operated within the Cd box. An insulating mount must still be used in addition. The open end of the Cd box should be packed with borated rubber or screened in some other manner.

Discriminator Settings

The electronics setup for the transfer detector is shown in Figure 1. Please note that a bias of +100 V should be applied.

Participants will need some means of assuring the proper placement of the SCA discriminators, preferably a multichannel pulse height analyzer and pulse generator. If necessary, a scan with a narrow window on the SCA itself could be used to verify the discriminator settings. The upper level discriminator is set at the minimum in the pulse height distribution between the two major peaks, shown as $V_U$ in Figure 2. The lower level discriminator is set at $V_L = V_U/3$. The various peaks and minima of the pulse height distribution are slightly less well resolved for the heaviest boron deposit, but the minimum for setting $V_U$ is still unambiguous.

Noise and Grounding (Earthing) Considerations

Participants might prefer to use some of their own electronics in place of that supplied by NIST, but the comparisons are probably more accurate if all participants use exactly the same system. In particular, the amplifier-preamplifier combination has been arranged to have very low noise and to be free of ground loops. Since the ionization chamber signal is not nearly as robust as that from a fission chamber, the participants will need to be on guard against electronic noise. In particular, THE DETECTOR SHOULD BE SUPPORTED IN AN ELECTRICALLY INSULATED MANOR (using the ground-insulating block supplied or some other means), with its only connection to ground (earth) through its preamplifier cable.

Precautions for Handling the Transfer Detectors

When shipped, both faces of each ionization chamber will be covered by protective caps. These caps should be left in place except when the detector is in use, in order to protect the fragile single-crystal silicon cathode and anode-cover pieces.

Please do not attempt to clean the exposed silicon surfaces. A finger print on one of these surfaces will do no harm, but attempting to clean the surface by wiping or with solvents could very easily break one of the silicon disks or get solvent onto the internal boron surface, invalidating the detector calibration. The detectors are not tightly sealed; solvents could penetrate into the interior.
Please inform NIST immediately of any problems with operating the transfer detectors and return them to NIST for any repairs. **Please do not open the ionization chambers for any reason.** The closures are marked for security purposes; and not all of the security marks are visible. **If the chambers are found to have been opened by a participant, the data from that participant may be excluded from the comparison.**
**Electronics Hardware**

1) Tennelec Portable NIM Bin  
2) Tennelec TC 909 NIM Power Supply (+100 V)  
3) Detectors: NIST Ion Chambers  
4) Bias Supply: Tennelec 953A Dual HVPS  
5) Preamp: Tennelec TC 170 Low Noise Preamp  
6) Pulser: not supplied  
7) Amplifier: Tennelec TC 241 Amplifier  
8) Single Channel Analyzer: Ortec 551 Timing SCA  
9) Counter/Timer: Ortec 994 Dual Counter/Timer  
   with null modem cable  
10) Computer: not supplied

**FIGURE 1**
REFERENCES


