# Radioactive Neutron Sources Emission Rates

## Purpose

The purpose of this procedure is to describe the setup, measurement, and reporting procedures for radioisotope neutron source calibrations with neutron emission rates ranging from $5 \times 10^5$ n/s to $1 \times 10^{10}$ n/s.

## Scope

This procedure covers the calibration of radioisotope sources of neutrons via the manganese sulfate ($MnSO_4$) bath method, in which the emission rate of the source to be calibrated is compared to the emission rate of NBS-1, the national standard Ra-Be photoneutron source. Additional details and references can be found in Refs. [1, 2, 3].

## Definitions

**NBS-1** The national standard Ra-Be photoneutron ($\gamma$,n) source. Its emission rate has been absolutely determined with an estimated uncertainty of ± 0.85 % (see Ref. [1]). Its emission rate in October 2003 was $1.234 \times 10^6$ n/s, and it has a half-life of 1600 years.

**BIPM** In addition to NBS-1, we have three calibrated neutron sources from the Bureau International des Poids et Mesures (BIPM). Their accuracy is about 1 %. They are an AmBe source ($2.285 \times 10^6$ n/s in 12/1986, half life 432 years), a Ra-Be ($\alpha$,n) source ($3.360 \times 10^6$ n/s in 12/1986, half life 1600 years), and a Ra-Be photoneutron source ($6.404 \times 10^5$ n/s in 12/1969, half life 1600 years).

## Equipment

Figures 1 and 2 show the manganese sulfate bath and control rooms, respectively. The equipment located in the manganese sulfate bath room is:

- 1.27 m diameter bath containing $MnSO_4$ solution with a density of 1.25 kg/L
- Marathon Electric pump for circulating the manganese sulfate solution
- Teflon source holder
- stepping motor, several pulleys, and string
- Barnstead B-pure water filter system
- Anton Paar DMA 35N density meter

The equipment located in the control room is:

- Central Research Laboratories remote manipulator
- StepPak MCU-2 stepping motor controller

- two Bicron detectors ("main" and "remote") utilizing sodium iodide crystals and photomultipliers
- stainless steel Marinelli beaker housing the "main" detector
- two Ortec 556 high voltage (HV) power supplies for the photomultiplier tubes
- two Ortec preamps for the photomultiplier outputs
- two Ortec linear amplifiers

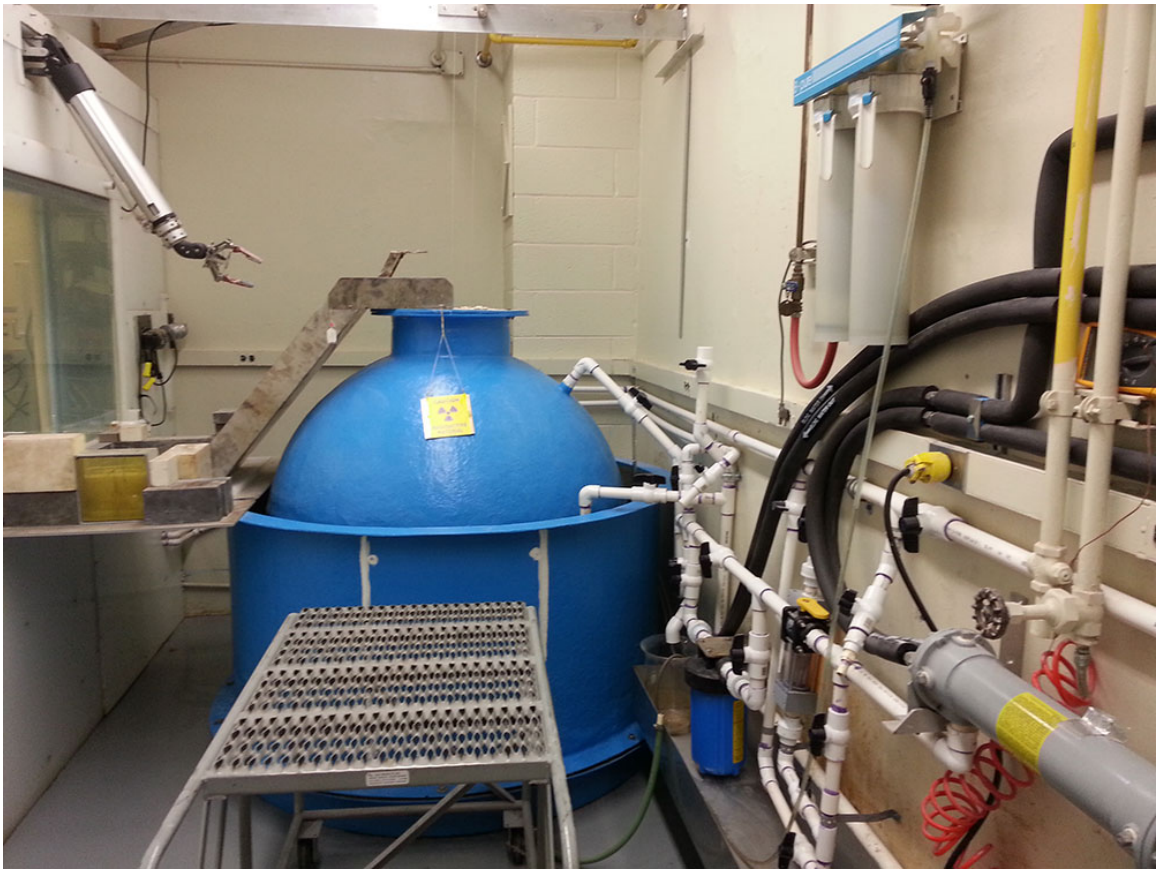

Figure 1: The manganese sulfate bath room.

Figure 2: The manganese sulfate bath control room.

- two NIST-built constant deadtime discriminators
- three Ortec 772 counters
- Ortec 776 counter timer
- Tennelec TC 566A time of day, year clock
- Ortec 779 interface controller
- computer running IGOR and in-house analysis software
- generic precision pulse generator
- Tracor Northern TN-7200 multichannel analyzer (MCA)

Because this measurement is a relative measurement (vendor's source versus NBS-1), it is sensitive only to the stability of the employed electronics over the measurement period (a few days). All other effects, such as the specific choice of electronic modules, are common mode and do not enter into the final result.

# Health & Safety

The calibration of neutron sources involves several different safety aspects.

1. The radiation safety aspect emphasizes minimization of personnel exposure. The magnitude of neutron source strengths that can be safely handled at NIST is limited to $10^{10}$ n/s, or less, so several hours of close proximity exposure would be necessary to produce a lethal dose of radiation. Our emphasis is upon obtaining

as small an exposure as practical in consideration of As Low As Reasonably Achievable (ALARA) practices.
2. The other aspects are safe handling of neutron sources to prevent any damage and industrial safety practices when handling heavy shielding materials, shipping casks, and the removable parts of shipping containers.

With regard to radiation safety, Nuclear Regulatory Commission (NRC) regulations and NIST policy require that all personnel handling neutron sources be given a radiation safety and awareness training course, with a refresher course to be given every two years. Radiation safety and training services are provided by the NIST Gaithersburg Radiation Safety Division (GRSD). Safe handling of neutron sources and the industrial safety aspects are taught through an apprentice-type relationship with each new handler.

All neutron-source transfer operations at NIST are accomplished by trained professionals with the cognizance of, and frequently with the help of, GRSD personnel. Temporary, in-transit, source storage is accomplished with movable water tanks and shielding barrels. Procedures are reviewed prior to each handling operation. Whole body dosimetry (TLD) and extremity dosimetry (finger rings) are worn during all source transfers and a minimum of two authorized source users are present for a source transfer. When $10^9$-$10^{10}$ n/s sources are calibrated, supplemental gamma/neutron sensitive dosimeters are worn in order to document the radiation exposure received during the calibration; these transfers are carried out in close consultation with and in the presence of GRSD.

# Procedures

## Summary

Based upon email/telephone discussions with the customer, a calibration schedule is fixed and a NIST 364 (“Radioactive Material Request”) form is filled out. When each source arrives, it is carefully inspected by GSRD for damage or leaks. As these sources tend to be securely encapsulated, passing examination by GRSD personnel means they are very likely to work in the $MnSO_4$ bath without a problem. The sources are stored in a secure area, which is monitored carefully by GRSD. Any given source is likely to be present in storage for no more than three months.

Four separate measurements are needed to calibrate a source and establish the reliability of the results. They are:

1. measurement of the background
2. measurement of NBS-1
3. measurement of the customer's source
4. remeasurement of NBS-1

Ideally the background measurement should be done when the bath has been empty for at least a month. These four measurements are subsequently analyzed to obtain customer

source strength and uncertainty. Occasionally this order is changed if, for example, there is a backlog of sources to calibrate. In this case, several sources may be measured between NBS-1 measurements.

## Procedure for each measurement

1. If the source strength is greater than $10^8$ n/s, turn off the HV on the "main" detector and tally results from the "remote" channel; otherwise, tally results from the "main" channel. For the purposes of these instructions, the tallied channel will be called the *active* channel.
2. Remove the neutron source from its container following the customer's instructions.
3. Quickly transport the source to the loading area adjacent to the bath using an appropriate grappling pole. Return to the control room.
4. Using the remote manipulator, load the Teflon source container with the source, screw the top onto the container rendering it watertight, put the source container in the bottom part of the Teflon carriage, position the carriage on the stand sitting atop the bath, grab and attach the dangling carriage top to the carriage bottom. Finally, release the carriage from the stand allowing it to dangle freely over the bath.
5. Using the motor controller's up and down controls, lower the source until the red mark on the string attached to the carriage is centered on the black arrow located on the far wall. At this point the source is centered in the bath.
6. Once the decay activity has built up sufficiently (~30 hours), use an oscilloscope to verify that the pulse width coming out of the *active* discriminator is 3.4 μs. This measurement does not have to be done every time. It suffices to do this a couple of times per year as this quantity has been observed to be very stable.
7. Verify that the "time of day, year clock" unit is reading the proper time. If it is not, adjust it.
8. Over the course of the next few days, periodically adjust the gain of the *active* amplifier until the 846 keV $^{56}$Fe peak occurs in channel 316 on the MCA (the MCA should be set for 512 channels full scale).
9. Name and activate (open) a log file in IGOR on the acquisition PC. Accumulate data for a couple of days. Every 50 minutes, the accumulated counts are logged.
10. Periodically type the peak channel number observed on the MCA into IGOR. This information will be added to the log file. If the peak has drifted away from channel 316, bring it back by adjusting the gain.
11. Once sufficient data have been obtained (typically after 2 days), the source can be removed from the bath and returned to its proper storage container. Reverse the steps followed above for transporting the source.

For a background measurement, only steps 7 and 9 are followed. Since a background measurement provides results relevant for both detectors, both amplifier gains should be set near their normal operating points during this measurement.

In terms of maintenance, the water level in the bath should be checked periodically. The level will drop due to evaporation. The level should be kept near the lower rim of the entrance tube on top of the bath by the addition of deionized water. It is also possible to add manganese sulfate if the specific gravity of the bath liquid becomes too low.

This measurement is not significantly sensitive to the room or bath temperature over the range within which they normally vary.

## Procedure for analysis

1. From each of the four file sets that have been written, select usable rows. For the background run this should be everything; for the other runs, it will be those rows where the peak was in channel 316.
2. Run **mnbath.exe** specifying the background file to be analyzed. Answer the prompts. Now background rates will be available for both channels.
3. Run **mnbath.exe** on the other three files. In each case, it will be necessary to specify the background for both channels.
4. From each of the three non-background output files, gather the number of counts per second ($R_{\text{customer}}$ and $R_{\text{NBS-1}}$) and their uncertainties.
5. Extract source strengths for the customer's source. The source strength $S$ is derived from $R$ through the equation

   $$S = S_{NBS-1} F \frac{R}{R_{NBS-1}} \frac{c}{c_{NBS-1}}$$, <-changed “NBS-i” to “NBS-1” in first fraction

   where $R$ is $R_{\text{customer}}$, $S_{\text{NBS-1}}$ is the known source strength of NBS-1 on the calibration date, $F = 1$ if the *active* channel was "main", $F = 72.25$ if the *active* channel was “remote” (the remote detector is 72 times less efficient than the main detector), and both $c$'s are obtained by summing the source-appropriate column in Table 1.
6. An additional code has been developed in the programming language S to analyze the data (S is similar to R). This code carries out the same calculations as does the program mnbath.exe, however it has the advantage that it auto-archives all data.

# Acceptance Criteria

Agreement between new values of $R_{\text{NBS-1}}$ and previous values at the 1 % or better level provides great confidence in this process. In particular there is always a long history of $R_{\text{NBS-1}}$ values available. New values should be within 2 % of the trend suggested by previous values unless the reason for the difference is understood (for example additional $MnSO_4$ has been added to the bath). When this is not the case, it usually means that some part of the apparatus is not functioning properly (for example neutron absorbing material may have been inadvertently introduced into the bath). In this case, calibrations must stop and the cause of the deviation must be determined and corrected. A thorough

investigation shall accompany any deviation that cannot be corrected to within the expected value range. These actions shall be documented and submitted for technical review.

In addition, it is important to look at all the individual counts to see whether or not there are any trends or outliers. Finally, when a previously calibrated source is recalibrated, the new number should agree with the previous number. When the calibration is finished, the source is returned to the customer.

| **Source of correction** | **$^{252}$Cf** | **$^{239}$Pu-Be** | **Am-Be** | **Ra-Be ($\alpha$,n)** | **AmB** | **AmF** | **Am-Li** | **Ra-Be NBS-1** | **SbBe** |
|---|---|---|---|---|---|---|---|---|---|
| Fast leakage | 0.030 | 0.333 | 0.227 | 0.193 | 0.013 | 0 | 0 | 0 | 0 |
| Thermal leakage | 0.015 | 0.050 | 0.030 | 0.026 | 0.007 | 0.003 | 0 | 0 | 0 |
| Oxygen capture | 0.344 | 2.092 | 2.031 | 1.528 | 0.254 | 0 | 0 | 0 | 0 |
| Sulfur capture | 0.280 | 0.913 | 0.848 | 0.627 | 0.274 | 0.06 | 0 | 0 | 0 |
| Teflon capture | 0.170 | 0.700 | 0.680 | 0.530 | 0.160 | 0.095 | 0.095 | 0.002 | 0 |
| Cavity flux/Q | 0.186 | 0.141 | 0.143 | 0.190 | 0.156 | 0.197 | 0.350 | 0.43 | 0.52 |

Table 1: The magnitude of correction or effect in % for the indicated types of neutron source. Calculations performed by E. J. Axton, NPL, for a $MnSO_4$ bath with dimension and manganese concentration same as that at NIST. We multiply the Cavity flux/Q correction by a factor that ranges from 1—3 depending on the size of the source encapsulation. Larger Sources correspond to larger factors.

# Determination of uncertainties

The basis for the determination of uncertainties associated with these measurements is the *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* [4]. The expanded uncertainty consists of components evaluated by statistical means (the so-called "Type A" uncertainties) and components determined on the basis of alternative techniques (the so-called "Type B" uncertainties). Type A uncertainties are the uncertainties on the *R*'s, typically less than 0.5 %. Type B uncertainties include the following:

- NBS-1 emission-rate (± 0.85 %)
- Detector calibration (± 1 %); this is present only when the "remote" detector is used (customer source strength in the range $10^8$ n/s to $10^{10}$ n/s)
- Uncertainty associated with the applied corrections for both NBS-1 and the calibrated source (± 0.3 %, and typically 1 to 2 %, respectively)

The final report includes the expanded uncertainty. The expanded uncertainty corresponds to the quadrature-sum of the stated uncertainty components multiplied by a coverage factor equal to two (2). The expanded uncertainty, therefore, represents an approximate level of confidence of 95 %.

# Documentation

All of the data and analysis files are stored in customer-specific folders. In addition, a global log book of bath-related activity is maintained.

For customer calibration, prepare calibration report and obtained required signatures. Make copy for customer file and send original.

# References

[1] E. Dale McGarry and Edward W. Boswell. Neutron source strength calibrations. Technical report, National Institute of Standards and Technology, 1988. NBS Special Publication 250-18.
[2] J. M. Adams. Present and future trends for neutron source calibrations at the National Institute of Standards and Technology. *Nucl. Instrum. Meth. B*, 213:218—222, 2004.
[3] Craig R. Heimbach. The neutron spectrum of NBS-1. J. ASTM International, Volume 3, Issue 4 (available online at www.astm.org).
[4] Barry N. Taylor and Chris Kuyatt. Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, NIST Technical Note 1297, September 1994.

# Appendix A Calibration Report

# REPORT OF CALIBRATION

**Neutron Source Strength Calibration Report**

NIST Test Folder Number: XXXXXX
Service ID: 44020C

Calibration Performed for: Si Grande Corp
Quelque part, USA

Neutron Source Description: Source Type: $^{252}$Cf
Serial Number XXX-XX-XXXX

Calibration Results:

| | |
|---|---|
| Calibrated neutron emission-rate: | $1.68 \times 10^{8}$ neutrons per second |
| Expanded uncertainty: | ± 4.30 % (95 % level of confidence) |
| Calibration date: | August 11, 2003 |
| NBS-1 emission-rate on date of calibration: | $1.234 \times 10^{6}$ neutrons per second |

This calibration was performed by:

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Maynard S. Dewey, Physicist
Neutron Physics Group

Reviewed by

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Muhammad Arif, Leader
Neutron Physics Group

For the Director of the National Institute of
Standards and Technology:

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Lisa Karam, Chief
Radiation Physics Division

National Institute of Standards and Technology

**Appendix for $^{252}$Cf Neutron Source Strength Calibration Reports**

Calibration Method

Neutron source strength measurements performed at NIST are accomplished by comparing the emission rate of the source being calibrated to that of the national primary standard neutron source, NBS-1, whose emission rate has been determined absolutely. The measurements of source emission rate are made by activating a circulating, aqueous solution of manganese sulfate, and continuously counting the induced $^{56}$Mn activity with a scintillation counter. During calibration, the neutron source is placed within a small Teflon cavity that is positioned at the center of the 1.3 m diameter spherical bath; activity measurements are taken once the bath has reached saturation. The purpose of the cavity is to reduce the absorption of thermal neutrons by the source. Corrections to the measured source strength have been applied in order to account for the following effects: capture of fast neutrons by oxygen and sulfur in the bath, capture of fast and thermal neutrons by fluorine in the Teflon source holder, neutron escape from the bath, and thermal neutron absorption in the source. Typical values for these corrections are:

| | |
|---|---|
| Fast neutron capture by oxygen and sulfur: | 0.624 % |
| Fast and thermal neutron capture by fluorine: | 0.170 % |
| Neutron escape from the bath: | 0.045 % |
| Thermal neutron absorption in the source: | 0.186 % |

Uncertainties

The expanded uncertainty consists of components evaluated by statistical means (the so-called “Type A” uncertainties) and components determined on the basis of alternative techniques (the so-called “Type B” uncertainties). The “Type A” and “Type B” uncertainty components relevant to this calibration are identified below.

Type A uncertainties: Count-rate associated with NBS-1 (typically < 0.5 %)
Count-rate associated with the calibrated source (typically < 0.5 %)

Type B uncertainties: Uncertainty associated with the applied corrections for both NBS-1 and the calibrated source (± 0.3 %, and typically 1 to 2 %, respectively)
NBS-1 emission-rate (± 0.85 %)
Detector calibration (± 1.0 %)

The expanded uncertainty reported corresponds to the quadrature-sum of the stated uncertainty components multiplied by a coverage factor, *k*, equal to two (2). The expanded uncertainty, therefore, represents an approximate level of confidence of 95 %.

National Institute of Standards and Technology