

## RADIOACTIVE NEUTRON SOURCES EMISSION RATES

# 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 neutron sources via the manganous sulfate 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  $\pm 0.85$  % (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 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 bath and control rooms, respectively. The equipment located in the manganese bath room is:

- 1.27 m diameter bath containing  $\text{MnSO}_4$  solution with a density of 1.25 kg/L of solution
- Marathon Electric pump for circulating the manganous 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

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- 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 bath room.

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Figure 2: The manganese 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 Procomm 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 many 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.

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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, NRC and NIST regulations require that all personnel handling neutron sources be given a radiation safety and awareness training course. Radiation safety and training services are provided by the NIST Health Physics Group. This training is essentially the same as that required by the NIST reactor. Therefore, the training requirement for neutron source handling is satisfied every two years by attendance at the reactor health physics course. 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, health physics personnel. Temporary, in-transit, source storage is accomplished with movable water tanks and shielding barrels. Procedures are reviewed prior to each handling operation. Each participant must wear an albedo-type neutron dosimetry badge, a gamma badge, and pocket dosimeter. The latter serves as a device for periodically estimating doses during an operation. Frequently, in those situations where sources are to be removed from an unfamiliar surrounding, or for in-air source transfers, audible-signal dosimeters are also used.

## **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 health physics professionals for damage or leaks. As these sources tend to be securely encapsulated, passing examination by the NIST Health Physics personnel means they are very likely to work in the Mn bath without a problem. The sources are stored in Building 245, Room B143, a secure area that is monitored carefully by the NIST Health Physics Group. Any given source is likely to be present there 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 (occasionally a "BIPM" source is substituted for this)

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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.

**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  $\mu$ s.
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  $^{56}\text{Fe}$  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 Procomm 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 Procomm. 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.

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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.

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

The specific gravity of the bath has declined over a period of years due to loss of  $\text{MnSO}_4$ . Because this calibration relies upon a ratio, it is not sensitive to its precise value. Nevertheless, the signal to noise ratio is adversely affected when there is a loss of  $\text{MnSO}_4$ . This value is now monitored regularly, and additional  $\text{MnSO}_4$  may be added to the bath.

### Procedure for analysis

1. From each of the four files 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 **mn bath.exe** specifying the background file to be analyzed. Answer the prompts. Now background rates will be available for both channels.
3. Run **mn bath.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 ( $R_{\text{customer}}$ ,  $R_{\text{NBS-1}}$ , and  $R_{\text{BIPM}}$ ) and their uncertainties.
5. Extract source strengths for the customer's source and those from the BIPM. The source strength  $S$  is derived from  $R$  through the equation

$$S = S_{\text{NBS-1}} F \frac{R}{R_{\text{NBS-1}}} \frac{c}{c_{\text{NBS-1}}},$$

where  $R$  is  $R_{\text{customer}}$  or  $R_{\text{BIPM}}$ ,  $S_{\text{NBS-1}}$  is the known source strength of NBS-1 on the calibration date,  $F = 1$  if the *active* channel was "main",  $F = 74$  if the *active* channel was "remote" (the remote detector is 74 times less efficient than the main detector), and both  $c$ 's are obtained by summing the source-appropriate column in Table 1.

### Acceptance Criteria

The agreement between the new value of  $S_{\text{BIPM}}$  and its known value to better than 1 % provides great confidence in this process. 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.

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Source of correction	<sup>252</sup> Cf	<sup>239</sup> Pu-Be	Am-Be	Ra-Be (α,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<sub>4</sub> 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, *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. These are typically less than 0.5 %. Type B uncertainties include the following:

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- NBS-1 emission-rate ( $\pm 0.85$  %)
- Detector calibration ( $\pm 1$  %); 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 ( $\pm 0.3$  %, and typically 1 to 2 %, respectively)

The final report is 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](http://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.

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## 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:  $^{252}\text{Cf}$   
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 Interactions & Dosimetry Group

Reviewed by

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Muhammad Arif, Leader  
Neutron Interactions & Dosimetry Group

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

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Lisa Karam, Chief  
Ionizing Radiation Division



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