

Ionizing Radiation Division	43030C, 43040C, 43050C	IRD-P-02
RADIATION DETECTORS CALIBRATION		

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Alpha Particle Radioactivity Measurements

Purpose

The purpose of these procedures is to describe the setup, measurement, and reporting procedures for alpha particle radioactivity measurements. Measurements using the NIST $2\pi\alpha/\beta$ proportional (small-area counter), NIST large-area $2\pi\alpha/\beta$ proportional (large-area counter), and the NIST " 0.8π " α defined-geometry scintillation (0.8π counter) counters are described.

Scope

These procedures cover the alpha-particle-emission-rate measurements for "thin" solid alpha-particle sources by means of the small- and large-area proportional counter detectors, and the activity measurements of similar sources using the " 0.8π " counter. Test number 43030C and 43040C correspond to, respectively, emission rate and activity calibrations of alpha-particle-emitting radio-nuclides as solids. Test number 43050C refers to the performance of both previously mentioned tests. Circular sources up to 10 cm can be measured in the 2π counter. The large-area counter can measure a rectangular source with dimensions of 18 cm by 30 cm. Sources with diameters up to 2.5 cm can be measured in the " 0.8π " counter. Sources inserted in the proportional counters should have an electrically conducting surface layer so that no accumulated charge is developed that can cause field distortion.

Definitions

Alpha-particle-emission-rate: the number of alpha particles emitted into 2π geometry per unit time. The measurement unit is s^{-1} .

Activity: the number of nuclei which disintegrate per unit time. The measurement unit is Becquerel (Bq), also in s^{-1} .

Equipment

- small-area proportional counter
- large-area 2π proportional counter
- " 0.8π " defined solid angle alpha counter

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- computer with data collection and reduction capabilities
- P-10 counting gas and hydrogen gas bottles and attachments and controls
- associated electronic equipment including amplifiers and preamplifiers

Equipment Quality Control

The functioning of the instrumentation is checked by comparing the measurement results for a standard, corrected for decay and background, with previous results for the standard. These measurements on the standards are recorded in the alpha-calibration log book.

Validation of software

Validation of processing software is performed by manual calculation of experimental results and comparison of this value to that obtained from the program. This is performed upon the initial version and subsequent to any changes in the program. Results of validations are recorded in the alpha-calibration log book. This software is stored and used on only one computer exclusively used for these procedures and by authorized personnel only.

Health and Safety Precautions

Radiation Safety

Rooms containing radioactive sources commonly used are kept locked at all times and are accessible only to designated members of the Radioactivity Group. Sources are handled by operators using gloves. Radioactivity danger signs are posted in the relevant areas.

Electrical Safety

All high voltages are encased in protective boxes and cannot be touched without opening these boxes with screwdrivers, etc.

Procedures

Preliminary

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- Customer contact - give specifications for physical dimensions and activity limits, emphasize that source container must protect active area from contact with anything within the container, and send calibration booklet to the customer.
- Inspection for damage - if damage such as broken seals, dented packages etc. has occurred, the customer will be notified before proceeding with the calibration
- Record identification information in log book.
- Test check measurements - perform measurements of background and a calibrated test source to make sure the system is operating correctly.
- NIST paperwork and acceptance procedure- submit completed NIST 364, "Proposal to Acquire a Radiation Source," for approval before arrival of materials, notify the Chief of Health Physics of the arrival and departure of all plutonium and uranium, complete other necessary forms (NIST 64,77,796A).
- Test folders - request test folder from OPMS on receipt of material to be calibrated. Note dates of material received and returned.

General Operational Procedures

The measurements are taken in the following order: background, standard reference source, submitted source, background. The counting times are adjusted so that 10^6 counts from the source are collected. The functioning of the instrument is checked by comparing the measurement results for the standard, corrected for decay and background, with previous results. The data is reduced as described in the following sections and certificates are prepared and submitted to the customer. Traceability is accomplished by measuring a national alpha-particle standard at the same time as the source submitted for measurement (Hutchinson, 1987). For customer calibration, prepare calibration report and obtain required signatures. The computer calculations, to be described in the next section, are cross-checked by manual calculations. Make copy for alpha-calibration record book and send original to the customer. For proficiency testing, follow the same procedure, but make an additional copy to be sent to the accrediting body. Calibration results are stored in both folders and in computer; the folder storage and computer access are both securely maintained by the calibration staff. The date that the calibration is performed and submitted to the customer is recorded with other information pertaining to the calibration is recorded.

Step-by Step Calibration Procedures

Procedures for calibrations using three instruments are described: (1) small-area counter, (2) large-area counter, and (3) the "0.8 π " counter.

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Small Area 2π Proportional Counter

General Set-up

- Introduce source into the counter
- Turn on the high voltage and other electronics
- Flush P-10 gas through the system until the pulses recorded on an oscilloscope reach a maximum value

General Setup

To Start

- Turn on the electronics.
- Turn on the air-pressure valve.
- Raise top of counter by pulling handle up slowly.
- Place source in the center of the counter, handling the source with gloves to prevent possible contamination.
- Lower top of counter by pulling handle down slowly.
- Turn on P-10 gas flow.
- Open black knob on regulator and flush rapidly for 10 minutes at maximum bubble rate on the bubbler.
- Turn on high voltage (1450 volts) at the start of the flushing.
- Reduce flow to low bubble rate (approximately 60 bubbles per minute on the bubbler).
- Use the following settings for the Tenelec TC 247 Dual Amplifier –coarse gain of 200 and fine gain of 10.0.

Data Collection

- The output of the amplifier is input to the multichannel analyzer card in the computer which uses a MAESTRO program services.
- Initiate job control.
- Use file named: prop1.job.
- Edit file to establish set-up parameters including:
 - number of sets of measurements(spectra)
 - elapsed live times
 - sample description
 - file name
 - counter description
- Collect spectral data (hit OPEN).
- Hand record history of measurements on the standards in the alpha-calibration log book.

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Data Reduction

When the data collection is complete, bring up file called SPECTINC, which sets 6 regions to be used in an extrapolation procedure which gives the integrated total count-rate.

Typically the regions would be channel integrals from 0-20, 21-40, 41-60, 61-80, 81-100, 101-1000.

Then, on EDIT file, type number of files to be reduced.

Run program called NLEVCON.EXE, which executes the data reduction into counts per second for each integral region specified above.

As a check on the results arrived at above, run the data called NLEVEL.EXE which performs the extrapolation automatically.

To Change Source

- Cut off High Voltage.
- Turn off gas flow at black knob.
- Raise top of counter by pulling handle up slowly.
- Remove source carefully.
- Place next source in center of counter.
- Lower counter top slowly.
- Turn on gas with black knob.
- Continue as above.

To Shut Down

- Turn off electronics (high voltage and NIM BIN).
- Turn of gas (close black knob, main valve, and release the diaphragm control).
- Turn off air-pressure valve.
- Take smears of counter and immediate area.

Large Area 2π Proportional Counter

To Start

- Turn on the electronics.
- Turn on the air pressure valve.
- Lower base-plate with switch in down position.
- Place source in the center of the counter, handling the source with gloves to prevent possible contamination.
- Raise base-plate with switch in up position.

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- Turn on P-10 gas flow.
- Open black knob on regulator and flush rapidly for 10 minutes at flow rate of 3 bubbles per minute on the bubbler.
- Turn on high voltage (1150 volts) at the start of the flushing.
- Reduce flow to approximately 1.5 bubbles per minute on the bubbler.
- Use the following settings for the Tenelec TC 247 Dual Amplifier –coarse gain of 200 and fine gain of 10.0.

Data Collection

- The output of the amplifier is input to the multichannel analyzer card in the computer which uses a MAESTRO program services.
- Initiate job control.
- Use file named: prop1.job.
- Edit file to establish set-up parameters including:
 - number of sets of measurements(spectra)
 - elapsed live times
 - sample description
 - file name
 - counter description
- Collect spectral data (hit OPEN).
- Hand record history of measurements on the standards in the alpha-calibration log book.

Data Reduction

When the data collection is complete, bring up file called SPECTINC, which sets 6 regions to be used in an extrapolation procedure, which gives the integrated total count-rate.

Typically the regions would be channel integrals from 0-20, 21-40, 41-60, 61-80, 81-100, 101-1000.

Then, on EDIT file, type number of files to be reduced.

Run program called NLEVCON.EXE, which executes the data reduction into counts per second for each integral region specified above.

As a check on the results arrived at above, run the data called NLEVEL.EXE which performs the extrapolation automatically.

To Change Source

- Cut off high voltage.
- Turn off gas flow at black knob.
- Lower base-plate.
- Remove source carefully.
- Place next source in center of counter.

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- Raise base-plate.
- Lower counter top slowly.
- Turn on gas with black knob.
- Continue as above.

To Shut Down

- Turn off electronics (high voltage and NIM BIN).
- Turn of gas (close black knob, main valve, and release the diaphragm control).
- Turn off air-pressure valve.
- Take smears of counter and immediate area.

0.8 π Defined Solid-Angle-Alpha-Particle Counter

To Start

- Turn on electronics (NIM BIN) and high voltage interlock box.
- High voltage power supply must be in the off position.
- Remove electrical connection from interlock to breech-block.
- Carefully unscrew breech-block which is also the source mount.
- Be ready to support the weight (approximately 2000 grams) of the breech-block when it comes free.
- Place source in center of breech-block (spacers for sources of various diameters in drawer under counter).
- Replace block and turn to stop position carefully. (Care should be taken not to advance source to the point of contact with the central baffle).
- Replace electrical connection from the interlock.
- Turn on gas (hydrogen) at main valve and adjust diaphragm control to about 4 bubbles per minute on the bubbler. (The gas is exhausted directly to the outside).
- Open black knob between regulator and flow meter and flush for about 10 minutes, at 3 L min⁻¹ as indicated on the flow meter.
- Turn on high voltage, (+) 1500, at start of flushing.
- Reduce flow rate to about 0.5 L min⁻¹ (observe flow rate on flow meter).
- Connect amplifier output to analyzer at attenuator input; coarse gain + 4, fine gain = 3.20.
- Counting may begin.

Data Collection

- The output of the amplifier is input to the multichannel analyzer card in the computer which uses a MAESTRO program services.
- Initiate job control.
- Use file named: prop1.job.
- Edit file to establish set-up parameters including:

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Data Reduction

When the data collection is complete, bring up file called SPECTINC, which sets 6 regions to be used in an extrapolation procedure, which gives the integrated total count-rate.

Typically the regions would be channel integrals from 0-20, 21-40, 41-60, 61-80, 81-100,101-1000.

Then, on EDIT file, type number of files to be reduced.

Run program called NLEVCON.EXE, which executes the data reduction into counts per second for each integral region specified above.

As a check on the results arrived at above, run the data called NLEVEL.EXE which performs the extrapolation automatically.

To Change Source

- Turn off high voltage.
- Turn off gas flow by closing black knob.
- Remove interlock lead.
- Remove source carefully.
- Take smears of block and counter area and count at Health Physics.
- Replace block and turn to stop position.
- Turn off interlock control.

To Shut Down

- Turn off electronics (high voltage at switch and NIM).
- Turn off gas (close black knob, main valve, and release diaphragm).
- Remove interlock lead.
- Remove source carefully.
- Take smears of block and counter area and count at Health Physics.
- Replace block and turn to stop position.
- Turn off interlock control.

Determination of Uncertainties

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The basis for the determination of uncertainties associated with alpha-particle calibrations is: Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results by Barry N. Taylor and C. E. Kuyatt, NIST Technical Note 1297, September 1994.

Uncertainty components are given below. All uncertainties are type B except for counting statistics and background, which are type A.

Significant Uncertainties

Counting statistics - this value was obtained from the standard deviation of replicate measurements

Background – based on statistical estimate

Live-time – obtained by measurements with sources of varying and known activities

Extrapolation to zero keV – estimated from largest possible variability in assumed extrapolation functionality

Self-absorption and scattering from source and support - estimated from customer stated source thickness and inaccuracies in back-scattering factors

The following uncertainties are recognized but are not significant

Counter geometry - the values were obtained from estimated mechanical accuracies measurements made on the systems

Extension and non-uniformity of the sources- this was derived from known limitations in the accuracies of measurements because of source size

Scattering off counter wall (0.8 π only)– calculated from known energy absorption values

Transmission through collimator edge (0.8 π only) – calculated from known energy absorption coefficients.

Scattering in/on detector – estimated from known parameters

Transmission through detector (no count) – based on comparisons with standards using other direct measurement methods

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1. The Standardization of Alpha-Particle Sources, L.L. Lucas. Proceedings of the ASTM Conference of Effluent and Environmental Surveillance, July 9-14, 1978, Johnson, Vermont, in ASTM Spec. Tech. Publ. 698, pp. 342-354 (1980).
2. Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, Barry N. Taylor and Chris Kuyatt. NIST Technical Note 1297, September 1994.
3. Alpha-Particle Calibrations, J. M. Robin Hutchinson. N.B.S. Special Publication 250-5, U.S. Government Printing Office, Washington D.C. 20402-9325 (1987).

Records

Customer log sheet includes customer name, date received, kind of source, source number and identification including RS number and test number, date calibrated, date returned to customer, copies of the purchase order, shipping documentation, and forms 364(proposal to acquire radiation source) and NIST 64 ("test record" - or invoice).

Alpha calibration record book includes hard copy of the data, data reduction, calibration results, and copy of certificate. Computer records include information contained in the customer log book and the alpha calibration record book.

Filing and Retention

All paper copies of customer files are stored in the test folder for that service; test folders are stored in a file cabinet maintained by the calibration staff. All customer-related electronic files are stored either on password-protected calibration-staff desktops or in the "High Dose" folder on the shared network drive.

The IRD Quality Manager shall maintain the original and all past versions of this IRD Procedure. Copies of the current revision of this Procedure shall be placed in controlled Quality Manuals. Electronic copies of this Procedure are uncontrolled versions.

All deleted Procedures (including old revisions) shall be maintained by the IRD Quality Manager. All old revisions shall be maintained until such time as it is decided to delete the Procedure. Once the decision has been made to delete the Procedure, only the last revision shall be maintained by the IRD Quality Manager.

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APPENDIX A

Table of Uncertainties

Factor	Type A Uncertainty (1 σ) percent	Type B Uncertainty (1 σ) percent
Counting statistics	0.1	
Background	0.1	
Live time		0.1
Extrapolation		0.7
Source self absorption		0.1

Combined Uncertainties Added In Quadrature	0.14	0.7
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Total Combined Uncertainty (2 times the sum of the quadratic sum of the two combined uncertainties)	1.4
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APPENDIX B



U.S. DEPARTMENT OF COMMERCE
National Institute of Standards & Technology
Gaithersburg, MD 20899

REPORT OF CALIBRATION

for

**COMPANY, INCORPORATED
TOWNESVILLE, STATE**

Radionuclide	Linolium-239
Source identification	4321
Source description	funny looking
2π alpha-particle counting rate	2.383 x 10 ³ s ⁻¹ ^{(1)*}
Relative expanded uncertainty	1.4 percent ⁽²⁾
Reference time	30 February 2004
Measuring instrument	NIST 2π α/β proportional counter

Measurements Performed by *Signature here*
Lynne Nason, Technician

<p><i>Signature here</i></p> <p>Lisa R. Karam, Acting Division Chief Ionizing Radiation Division Physics Laboratory</p>	<p>For the Director, National Institute of Standards and Technology by</p> <p><i>Signature here</i></p> <p>Michael P. Unterweger, Acting Group Leader Radioactivity Group Physics Laboratory</p>
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Gaithersburg, MD 20899
Report Issued: 1 March 2004
NIST Folder No.: xxxxxx-xx

*Notes on back

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NOTES

- (1) The total number of alpha particles counted per second emitted into a 2π -steradian geometry (including those scattered).
- (2) The uncertainty analysis methodology and nomenclature used for the reported uncertainties are based on uniform NIST guidelines and are compatible with those adopted by the principal international metrology standardization bodies [cf., B.N. Taylor and C.E. Kuyatt, *NIST Technical Note 1297* (1994)].

The relative combined standard uncertainties, $u_c = 0.72$ percent, is the quadratic combination of the standard deviations (or standard deviations of the mean where appropriate), or approximations thereof, for the following component uncertainties:

- | | | |
|----|---|--------------|
| a) | 5 proportional measurements on this sample | 0.12 percent |
| b) | pulse-height extrapolation
Percent difference in the certified value between the estimate based on horizontal extrapolation from the minimum point on the spectrum to that from the same spectral point to zero count rate at zero energy. | 0.72 percent |
| c) | system live time
Using the two-source method, the live time was verified to within 0.1 percent. | 0.1 percent |

The relative expanded uncertainty, $U = 1.4$ percent, is obtained by multiplying u_c by a coverage factor of $k = 2$ and is assumed to provide an uncertainty interval of approximately 95 percent confidence.

For further information, please contact Michael P. Unterweger at (301) 975-5536 or Lynne Nason at (301) 975-5544.

Source identification: 4321
NIST Folder No.: xxxxxx-xx

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