ALPHA- AND BETA-PARTICLE-EMITTING SOLID SOURCE CALIBRATIONS

Purpose

The purpose of these procedures is to describe the setup, measurement, and reporting procedures for alpha- and beta-particle radioactivity-measurements. Included, are descriptions of measurements using the NIST $2\pi\alpha$ proportional (small-area), NIST $2\pi\alpha/\beta$ proportional (large-area), passive implanted planar silicon (PIPS), and NIST table (external counters).

Scope

These procedures cover the alpha- and beta-particle-emission-rate measurements for "thin" conductive solid alpha- and beta-particle emitting sources by means of the small- and large-area proportional counters, mixed source spectroscopy using the PIPS, and the emission-rate measurements of higher activity sources using the external counter. Test number 43030C corresponds to emission rate calibrations of alpha- and beta-particle-emitting radionuclides and activity calibrations of alpha-particle-emitting radionuclides. Test number 43040C is for activity calibrations of beta-particle-emitting radionuclides. Test number 43050C refers to calibrations of mixed-alpha-particle-emitting sources. Circular sources with a diameter of up to 10 cm can be measured in the small-area $2\pi\alpha$ counter. Sources with dimensions of 18 cm by 30 cm or smaller can be measured in the large-area $2\pi\alpha/\beta$. Small-circular sources with a diameter of up to 5 cm can be measured in the PIPS. Sources with dimensions of 18 cm by 30 cm can be measured on the NIST table. All sources measured in the counters must 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} (counts per second or cps).

Activity: the number of nuclei that disintegrate per unit time. The measurement unit is the becquerel (Bq).

Equipment

- Small-area $2\pi\alpha$ proportional counter
- Large-area $2\pi\alpha/\beta$ proportional counter
- PIPS counter
- NIST table external counter
- Computers and MCA with data collection and reduction capabilities
- P-10 counting gas tanks, attachments, and controls
- Associated electronic equipment including voltage supply and amplifiers

Equipment Quality Control

The functioning of the instrumentation is checked by comparing the measurement results for a standard reference source, corrected for decay and background, with previous results for the same standard. The measurements on the standards are recorded in the Excel spreadsheet file “CheckSA” for the small-area counter and the spreadsheet file “CheckLA” for the large-area counter, found in the “Excel Files” folder in the current “old data C folder” accessed on the desktop of the investigator conducting the calibrations and backed-up on an external drive.

Validation of Software

Validation of manual calculation of experimental results is performed by comparing values found using data processing software. This is performed upon the initial version and subsequent to any changes in the program. Results of validations are recorded in the current alpha- and beta-test binder. This software is stored on computers used exclusively for these procedures and by authorized personnel.

Health and Safety Precautions

Radiation Safety

Radiation safety training and assessment services are provided by the NIST Gaithersburg Radiation Safety Division (GRSD). Rooms containing radioactive sources are kept locked when not occupied and are accessible only to designated members of the Radiation Physics Division and emergency response personnel. Sources are handled by operators using gloves. Radiation signage is posted in the relevant areas. Basic radiation monitoring and smear counting are handled in accordance with standard GRSD procedures.

Electrical Safety

All high voltages are encased in protective boxes and cannot be easily opened.

Procedures

Preliminary

• Customer contact: when customers request information prior to placing orders they are given specifications for physical dimensions and activity limits, emphasizing that submitted source must be electrically conductive. Customers are directed to place their order on the E-commerce storefront, or to contact calibration service staff for assistance in setting up a profile in the system. When possible a copy of the purchasing documentation is obtained for the test binder. The customer is provided the NIST shipping address, indicating GSRD Building 245, Room B131 to ensure

that GRSD receives the package directly. They are also instructed to wait until safety paperwork is approved before shipping.
• NIST paperwork and acceptance procedure- submit completed NIST 364, "Radioactive Material Request," for approval before notifying the customer to ship the source(s). Upon clearance of the source(s) by GRSD after receipt(?), the order is updated in e-commerce to reflect the source(s) have arrived and measurements will be started soon.

Source Receipt from GRSD

• Review 364 to determine if any contamination was found on source and packaging materials during GRSD check-in.
• Inspection for damage - if damage such as broken seals has occurred, the customer will be notified before proceeding with the calibration.
• Record identification information (including GRSD-assigned radioactive source RS number) on the log sheet.
• Test check measurements - perform measurement of the alpha- or beta-particle emitting standard to ensure the system is operating correctly.
• E-commerce orders – record the order number assigned to the calibration. Note dates of all steps completed including material received and returned on the log sheet.

General Operational Procedures

The measurements are taken in the following order: standard reference source, submitted source(s), and finally, background. Counting times are adjusted so that 10^6 counts are collected from each source, whenever possible. The functioning of the instrument is checked by comparing the measurement results for the standard, corrected for decay and background, with previous results. The manual calculations, to be described in the Alpha and Beta Laboratory Procedures document (ABLP), are cross-checked with computer software calculations. The data is reduced and corrected, as described in the ABLP. The results are reviewed and used to create calibration reports. The calibration report is checked, proof-read, signed, and sealed. Copies of the report are made for the current alpha and beta test record binder and sent to calibration services before the original is sent to the customer. Calibration results are stored both in binders and in the computer; the binder storage and computer access are both securely maintained. The dates that the calibration is performed and the report is submitted to the customer are recorded in the test record binder with other pertinent information.

Calibration Procedures

Procedures for calibrations using four instruments are described in the ABLP: (1) small-area, (2) large-area, (3) PIPS and (4) external counters.

At the completion of measurements, calculations, and corrections, results are entered into a spreadsheet. Results for sources that have been calibrated in the past are compared to previous results, accounting for decay. The difference should be less than 2 % or further investigation is necessary. Results for sources that have not been previously calibrated are compared to manufacturers' certified, or customer provided, values.

Determination of Uncertainties

The basis for the determination of uncertainties associated with alpha- and beta-particle calibrations is *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* (see References).

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 – determined from systematic tests using a NIST live-time module (see References)

Extrapolation– estimated from largest possible variability in assumed extrapolation functionality, geometry of the source will impact the method of extrapolation (see references)

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

Recognized Uncertainties

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 in/on detector – estimated from known parameters

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

References

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, (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).

4. Counting Yields for Beta and Alpha Particle Sources, Martin Berger. NISTIR 6464, (2000).

5. Large area alpha sources with a lip: Integral counting and spectral distortions, King, L; Fitzgerald, R ; Tosh, RE. Applied Radiation and Isotopes. V134, April 2018, pp.376-379 (2018).

Preliminary Reference

1. Accurate Integral Counting with Multi-Channel Analyzers (MCAs), submitted to Applied Radiation and Isotopes, 2019

Records

Customer log sheets include customer name and contact information, date received, kind of source, source number and identification including RS and order numbers, date calibrated, and date returned to customer. Copies of the shipping documentation, and 364 forms are kept with the log sheet.

Alpha and beta test record book includes hard copy of the data, data reduction and related calculations, calibration results, customer calibration spreadsheets, copy of certificate, and the documents described above. Computer records include unsigned copies of certificates and spreadsheets used to check standards and customer calibration sources.

Alpha and Beta Laboratory procedures include the detailed step-by-step instructions for the use of each of the four counters, the data collection and reduction file names and file locations for these calibrations.

Filing and Retention

All paper copies of customer files are stored in the alpha- and beta-test binders kept in building 245 room E103) or the investigators office (building 456 room B109-A). All customer-related electronic files are stored on password-protected systems in the laboratory (building 245 room E107), the investigator's desktop computer (building 456 room B109-A), or protected shared network drive.
Copies of the alpha and beta laboratory procedures are kept in the laboratory where the equipment is used (building 245, room E107) and in the investigator's office (building 456, room B109-A).

The RPD Quality Manager shall maintain the original and past versions of this RPD Procedure. See Guide RPD-G-01 for additional policies on Procedure maintenance.

APPENDIX A

Table of Uncertainties

Factor	Type A Uncertainty (1σ) percent	Type B Uncertainty (1σ) percent
Counting statistics	0.04-2.10	
Background	0.1	
Live time		0.20-2.25
Extrapolation		0.20-5.20
Uncertainties Combined in Quadrature	0.11-2.10	0.28-5.70
Expanded Uncertainty (k = 2, an approximate level of confidence of 95 %.)	0.60-12.20	

APPENDIX B

National Institute of Standards & Technology

REPORT OF CALIBRATION

for

Company, Incorporated
123 Standards Drive
Towntown, State 03691

Radionuclide	Lynolium-238
Source identification	FJ538
2π alpha-particle counting rate	1.455×10^5 s^{-1} (1)*
Expanded uncertainty ($k = 2$)	4.3 percent (2)
Measurement date FK514 (Reference time)	30 July 2019
Measuring instrument	NIST 2πα/β proportional counter (3) and "external" Si counter
Measurements Performed by	Lynne King, Physical Scientist

For the Director,
National Institute of Standards and Technology by

Brian E. Zimmerman, Leader
Radioactivity Group
Physical Measurement Laboratory

James M. Adams, Chief
Radiation Physics Division
Physical Measurement Laboratory

Gaithersburg, MD 20899
Report Issued: August 2019
Service ID No.: 43030C
Order No.: O-123457890

*Notes on back

Page 1 of 2

NOTES

(1) The 2π alpha-particle counting rate is the total number of alpha particles counted (including those scattered) per second emitted into a 2π-steradian geometry and is traceable to the NIST standard for the second. The 2π alpha-particle counting spectra are extrapolated to zero energy, integrated, and corrected for dead-time and background.

Due to the high activity of FK538, the 2π alpha-particle counting rate, was determined by using an "external" counter to measure the ratio of the count rate for FK538 to that of a lower-activity source (FK514), then scaling that ratio by the measured 2π alpha-particle counting rate of FK514. The "external" measurements were averaged over the same 5 positions on each source.

(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 combined standard uncertainty, $u_c = 2.13$ 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) measurement variability — 1.80 percent

Combinations in quadrature of the standard deviations of the mean for: 2π alpha-particle counting rate of FK514 (0.04 %, N=5), "external" counting rate averaged over 5 positions (each an average of 3 repeat measurements) for FK538 (1.19 %, N=5) and FK514 (1.35 %, N=5)

b) pulse-height extrapolation — 1.08 percent

Difference in the extrapolated 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

c) live-time correction of value for FJ514 — 0.38 percent

Estimate of uncertainty in the live-time correction determined from systematic tests using a NIST live-time module

The expanded uncertainty, $U = 4.3$ 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.

(3) The functioning of the instrument is checked by comparing measurement results corrected for decay and background, of the plutonium standard AC-8171.

For further information, contact Brian Zimmerman at (301) 975-4338 or Lynne King at (301) 975-5544.

Order No.: O-123457890
Source Identification: FJ538