Purpose

The purpose of this procedure is to describe the setup, measurement, and reporting procedures for the absorbed-dose certification of customer-irradiated NIST transfer dosimeters.

Scope

NIST provides transfer standards in the form of sets of calibrated alanine pellets packaged in polystyrene. The sealed, packaged dosimeters are sent to the customer for irradiation to nominal, agreed–upon absorbed dose levels in a prescribed geometrical arrangement. The unopened packaged dosimeters are then returned to NIST to be measured and evaluated and the results reported in the form of an absorbed-dose certificate. The absorbed dose range that is suitable for use with these transfer dosimeters is 20 Gy to 100 kGy.

Definitions

Absorbed dose to water: the energy absorbed from ionizing radiation per unit mass of water: 1 J/kg = 1 Gy.

Dosimeter batch: quantity of dosimeters made from a specific mass of material with uniform composition fabricated in a single production run under controlled conditions, and having a unique identification code.

Electron Paramagnetic Resonance (EPR): the process of resonant absorption of microwave radiation by paramagnetic ions or molecules in the presence of a static magnetic field.

Equipment

Essential Equipment	Calibration Method	Calibration Frequency
^{60}Co Gamma-Ray Source	Comparison to Vertical Beam Source	Determined by control charts
EPR Spectrometer	Dosimeter Check Standard Measurement	As needed
Microbalance	External Service	Annual

Health & Safety Precautions

Radiation safety

The high-activity ^{60}Co sources used for system calibration are housed in secure areas. Radiation safety and training services are provided by the NIST

Gaithersburg Radiation Safety Division.

Magnetic field safety

A Gaithersburg Office of Safety, Health and Environment survey of the vicinity of the EPR spectrometers determined that the magnetic field strengths are below actionable limits. However, as an extra precaution the room entrance has magnetic field warning signs; individuals with pacemakers should avoid rooms containing electromagnets associated with EPR spectrometers.

Procedures

1. *Dosimeter Batch evaluation and testing*

1.1 The procedure that describes the requirements for performance characterization of transfer dosimeters used in NIST high-dose dosimetry certification services are documented in the Dosimetry System Databook. The evaluation testing provides data to assess influence quantities that may have significant effects on the performance of a dosimetry system. Data from all tests shall be recorded in the Dosimetry System Databook.

2. *Instrument maintenance*

2.1 EPR Spectrometers are operated and maintained according to the manufacturer's guidelines. The specific system operating protocols are documented in the Dosimetry System Databook. Significant maintenance activities are recorded in the EPR spectrometer log book.

3. *Dosimeter system calibration*

Once a batch of pellets has been characterized and passed all acceptance criteria, a calibration curve shall be established.

3.1 Dosimeter Irradiation

3.1.1 Irradiate dosimeters to specified doses as detailed in RPD Procedure

3.2 Measure pellets

3.2.1 After all irradiations are completed, measure the pellets as described in Section 4.

3.3 Analyze the measurement data according to Dosimetry System Databook protocols.

3.4 Record the calibration curve and any supporting information in the Dosimetry System Databook

4. *Customer-irradiated absorbed-dose certification for alanine pellets*

4.1 Customer contact

4.1.1 Receive Purchase Order via postal mail, email or fax.

4.1.2 Input customer information into calibration log book, and assign a Division calibration number (HDxxxx). Start a new paper file (HD folder) to hold all printed documents.

4.1.3 Request and obtain a test folder number from the Calibration Office.

4.1.4 Mail dosimeters for requested test (service) numbers with instruction letter to the customer (Appendix A).

4.1.4.1 Each dosimeter consists of four alanine pellets in a polystyrene vial [2].

4.1.4.2 Step 4.1.4 is not applicable to service code 49030C.

.

4.2 Receiving customer dosimeters

4.2.1 Verify that irradiator and irradiation information (target dose, temperature, etc.) on instruction letter has been provided by customer and reconcile the dosimeters returned with the list cited in the accompanying documents.

4.2.1.1 Customer letter for service code 49030C must contain the lot/batch number for the dosimeters provided.

4.2.2 Note pellet numbering scheme correspondence to the dosimeter vial numbers.

4.2.3 Opening only one vial at a time, remove pellets from vials, numbering consecutively (top to bottom) as removed from the vial. An additional sequential mark is placed on the service code 49030C pellets as a visual aid.

4.2.4 NOTE: Empty vials are cleaned by immersing in ethanol with agitation, allowing them to remain immersed overnight, then dried in a fume hood overnight. Service code 49030C vials are not cleaned by NIST; they are returned to the customer.

4.2.5 Weigh pellets and input masses into Excel spreadsheet.

4.2.6 Create Excel spreadsheet (see Dosimetry System Databook) to record data from measurements.

4.3 EPR Spectrometer setup (see Dosimetry System Databook)

4.4 Spectrometer stability check (see Dosimetry System Databook)

4.5 Excel spreadsheet setup (see Dosimetry System Databook)

4.6 ECS106 measurement (see Dosimetry System Databook)

4.7 Analyze measurement results (see Dosimetry System Databook)

4.8 Report and test closure

4.8.1 Complete the QM Checklist (Appendix B)

4.8.2 Create an “Absorbed Dose Measurement Certificate” report by inserting the company information, folder number and Transfer Dosimetry Databook references (footnote), and the final dose values into the report template (see Appendix C)

4.8.3 The fully-signed certificate is mailed to the customer and copies are retained for the test folder and HD folder.

5. Quality control

5.1 Absorbed-dose check standards

Approximately every month, or as needed, dosimeter pellets are irradiated to the following doses: 0.025 kGy, 0.20 kGy, 1.0 kGy, 10 kGy, and 40 kGy. These check standards are routinely measured ~24 hours to 72 hours after irradiation, as well as prior to service measurements. Data from these check standards are archived in working spreadsheet files for reference availability. Check dose measurements that measure outside of set limits must be resolved through re-measurement, repetition of the check standard process, or a complete recalibration of the dosimetry system.

5.2 Transfer dosimetry controls

Each set of transfer dosimeters shipped to a customer is paired with a control vial that is packaged separately and marked “Do Not Irradiate”. Control vials contain pellets of the same type that have been previously irradiated to a calibrated dose. These dosimeters are typically check standards that have exhibited good stability after repeated measurements. A continuous history of these data is recorded. Any nonconformance shall be reported; action to be taken is at the discretion of the calibration staff.

5.3 International comparisons

Upon mutual agreement, dosimetry comparisons are performed with the high-dose calibration facility of the National Physical Laboratory of the United Kingdom. Dosimeters from each facility are exchanged, measured, and the results compared. The RPD participates in larger international comparisons, such as those organized by the BIPM, when available (approximately every ten years) [3]. These data are summarized in the High-Dose International Comparisons Databook.

6. Traceability

6.1 Dose rate transfer

The SI unit of absorbed dose is the Gray (Gy). For this service, the Gy is realized through water calorimetry measurements in the Vertical Beam ^{60}Co Source. These measurements are transferred to the pool source and subsequently to the three Gammacell calibration sources by source-rate ratio measurements using alanine dosimetry. These transfer measurement protocols are described in NIST SP250-44 [4]; the traceability scheme was later modified [5].

Determination of Uncertainties

The basis for the determination of uncertainties associated with High-Dose calibrations is the *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* [6]. The purpose of this section is to explain the derivation of the various components of uncertainty for absorbed-dose certification. Examples of the values for the uncertainty components are listed in Appendix D.

Water Calorimetry: uncertainty from realization of the Gy [7].

Source Ratio Data: uncertainty from source dose-rate transfer (water calorimetry rate to high-dose calibration source rate) through ratio measurements.

Field Uniformity: radiation field uniformity within a dosimeter volume.

Environmental Effects: temperature control during irradiation.

Timer: uncertainty of timer readout relative to shortest irradiation time interval.

Decay Correction: half-life correction factor uncertainty.

Mass: uncertainty of microbalance relative to pellet mass.

Repeatability and Reproducibility: standard deviation of replicate pellet measurements.

Interspecimen Contamination: cross contamination of pellets during the measurement process.

Ruby Correction: uncertainty resulting from EPR spectrometer fluctuations during the time interval between the alanine pellet measurement and the reference (ruby) measurement.

System Drift: uncertainty arising from temporal EPR spectrometer response fluctuations.

Temperature Correction: uncertainty from alanine dosimeter temperature coefficient measurement.

Calibration Curve: fit uncertainty from alanine dosimeter calibration curve.

Additional uncertainties are applied to irradiations in electron beams and ^{137}Cs, or for absorbed dose to silicon conversions.

References

1. Sleptchonok, O.F., Nagy, V., Desrosiers, M.F., 2000 Advancements in accuracy of the alanine dosimetry system. Part 1. The effects of environmental humidity, Radiat. Phys. Chem. **57**, 115-133.
2. Radiation Processing Dosimetry Calibration Services: Manual of Calibration Procedures, Humphreys, J.C., Puhl, J.M., Seltzer, S.M., McLaughlin, W.L., Desrosiers, M.F., Bensen, D.L., Walker, M.L. 1998 NIST Special Publication 250-45.
3. Burns, D.T., Allisy-Roberts, P.J., Desrosiers, M.F., Sharpe, P.H.G., Pimpinella, M., Lourenço, V., Zhang, Y.L., Miller, A., Generalova, V., Sochor, V. 2011 Supplementary comparison CCRI(I)-S2 of standards for absorbed dose to water in Co-60 gamma radiation at radiation processing dose levels, Metrologia, 48, Tech. Suppl. 06009 1-18.
4. Radiation Processing Dosimetry Calibration Services and Measurement Assurance Program, Humphreys, J.C., Puhl, J.M., Seltzer, S.M., McLaughlin, W.L., Desrosiers, M.F., Bensen, D.L., Walker, M.L. 1998 NIST Special Publication 250-44.
5. Desrosiers, M.F., Puhl, J.M., Cooper, S.L. 2008 Discovery of an Absorbed-Dose / Dose-Rate Dependence for the Alanine-EPR Dosimetry Systems and Its Implications in High-Dose Ionizing Radiation Metrology, NIST J. of Res., 113, pp. 79-95.
6. NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, 1994.
7. Domen, S.R., A sealed water calorimeter for measuring absorbed dose, NIST J. of Res., 99, pp. 121 – 141, 1994.

Records

Record	Contents/Purpose	Location
Calibration Log Book	Login all tests to obtain test folder number	245/C217
Dosimetry System Databook	Records dosimetry system calibrations and dosimeter batch characterization	245/C207
Internal Calibrations	Source ratio measurements and data analysis	245/C217
EPR Spectrometer User Log Book	Usage and maintenance records	245/C207
Transfer Dosimetry Databook	Records all transfer dosimeter certification data	245/C207
High-Dose International Comparisons Databook	Interlaboratory measurement comparison data summaries	245/C217

Filing and Retention

All paper copies of customer files are stored in the test folder for that service. 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 RPD Quality Manager shall maintain the original and past versions of this RPD Procedure.

Appendix A – Sample Transfer Letter

September 3, 2014

HD0000

Cobalt Jones
Slamma Gamma Irradiators

Return Shipments to:
NIST High-Dose Service
Building 245, Room C229
100 Bureau Drive, Stop 8460
Gaithersburg , MD 20899-8460
USA

Dear Mr. Jones,

Enclosed are the alanine transfer dosimeters that you requested for irradiation in your facility. There are six vials for irradiation (7901-7906), each filled with four alanine pellets from the batch T030901. The other vial (#44) is a control and should not be irradiated. Do not open the vials. The useful life of the dosimeters is approximately 30 days from the date of receipt at your facility. If dosimeters are not used within this time frame, please contact NIST for further instruction. Please complete the table on the following page and return it with the dosimeters.

Dosimeter	**Date(s) of irradiation**	**Target Dose, kGy (approximate)**	**Average Irradiation Temperature (°C)**
7901			
7902			
7903			
7904			
7905			
7906			

How would you like for us to identify your irradiator on the certificate?

Any other information you wish to be noted on the certificate?

Sincerely,

Calibrations Technician
Dosimetry Group
Physical Measurement Laboratory
PHONE: 301-975-xxxx FAX: 301-869-7682 E-MAIL: caltech@nist.gov
Enclosures

Appendix B – QM Checklist for 49020C and 49030C

NIST ID:
Date:

Checklist for 49020C and 49030C:

____ The ECS106 Spectrometer was set up with the spectral parameters that were used for the corresponding calibration curve.
____ The spectral parameters were noted in the Excel spreadsheet.
____ The pellet masses are paired correctly with the corresponding pellet number.
____ The Excel spreadsheet reflects the appropriate NIST ID, company name, irradiation temperatures, etc.
____ The Excel spreadsheet reflects the appropriate file names and dates for both the data file and calibration file.
____ The correct calibration curve coefficients were used in the dose calculation and noted on the spreadsheet.
____ All mathematical calculations embedded in cells have been checked for accuracy and correct cell linkage.
____ The appropriate correction factors were applied (i.e., temperature, dose-to-water, dose-to-silicon, ^{137}Cs, etc.) to the calculated dose.

Signed by: ________________________________ Date: _____________

Appendix C – Example Certificate

National Institute of Standards and Technology

Absorbed-Dose Measurement Certificate

NIST Service Identification Numbers 49020C and 49030C

IRRADIATOR
MDS Nordion Gammacell XXX

CUSTOMER
Slamma Gamma Irradiators
7 Electron Avenue
Mega Rad, LA 99817

ATTN: Cobalt Jones
Reference: PO # 5678

Measurements made by Lonnie Cumberland

Report reviewed by Marc F. Desrosiers

Report approved by

Michael G. Mitch, Leader
Dosimetry Group

For the Director
National Institute of Standards and Technology
by

Lisa R. Karam, Chief
Radiation Physics Division
Physical Measurement Laboratory

Information on technical aspects of this report may be obtained from Lonnie Cumberland, NIST, 100 Bureau Drive Stop 8460, Gaithersburg, MD 20899, 301-975-6869

REFERENCE HD0000
TEST FOLDER 123456
NIST DB 2000/000
October 8, 2014
Page 1 of 3 Pages

Transfer dosimeters were sent to Slamma Gamma Irradiators for irradiation in their facility. The dosimeters were NIST alanine pellets of FWT batch T030901; four each in a polystyrene vial. The dosimeters were analyzed on September 23, 2014, using a Bruker ECS106 spectrometer. Dose interpolations are based on a NIST calibration of batch T030901 alanine dosimeters performed February 25, 2014. The results are summarized in the following table.

Dosimeter Identification	Absorbed Dose kGy(H_2O)
9901	4.99
9902	4.98
9903	6.03
9904	6.11
9905	8.08
9906	7.99

UNCERTAINTIES AND RELATED FACTORS IN HIGH-DOSE MEASUREMENTS

Absorbed Dose Evaluations Based on Use of Mailed Alanine Pellet Transfer Standard Dosimeters Irradiated Using ^{60}Co

(Expanded uncertainty: ± 1.9 % at a 95 % confidence level)

The customer's use of NIST-certified transfer standard dosimeter measurements to determine their radiation-source dose rate is subject to limitations and precautions described in the letter accompanying the dosimeters. The customer must follow the prescribed procedures carefully in order to ensure that the results obtained from the transfer dosimeters are valid.

The absorbed dose in water evaluation is based on NIST alanine pellet dosimeters that are traceable to standard water calorimeter measurements at NIST. The uncertainty value cited above may be assumed as long as suitable care is exercised. That value does not include uncertainty in the customer-reported irradiation temperature or non-uniformity in the customer's irradiation field.

A detailed list of the various sources of uncertainty and estimates of the magnitude of those uncertainties that make up the overall uncertainty given above may be obtained through the Internet (http://www.nist.gov/pml/div682/qualitysystem.cfm) or by requesting this information from NIST. The uncertainties are divided into two types: A and B. Type A uncertainties are those evaluated by statistical methods, often associated with random effects. Type B uncertainties are those evaluated by other means, often associated with systematic effects.

Type A Uncertainties
The combined standard uncertainty evaluated by statistical methods is ±0.72 % at an approximate level of confidence of 68 %.

Type B Uncertainties
The combined standard uncertainty based on scientific judgment is estimated to be ±0.60 % at an approximate level of confidence of 68 %.

Expanded Uncertainty
The Type A and Type B uncertainties have been combined in quadrature (the square root of the sum of the squares) and multiplied by a coverage t-factor of 2.05 to yield an expanded uncertainty of ±1.9 % at an approximate level of confidence of 95 %.

NIST
National Institute of
Standards and Technology
U.S. Department of Commerce

REFERENCE HD0000
TEST FOLDER 123456
NIST DB 2000/155
October 8, 2014
Page 3 of 3 Pages

Appendix D- Example Uncertainties Table for Transfer Dosimetry Service

High-Dose Alanine Response, Far West / Gamma Service Alanine Pellets

Uncertainty Source		**Type A (%)**	**Type B (%)**
Repeatability and Reproducibility		0.30	
Mass Determination		0.20	
Interspecimen Contamination			0.10
Ruby Correction			0.05
System Drift			0.10
	sqrt(sum)	0.36	0.15

Alanine Pellet Dosimeter Transfer Dose(water), Gamma/X-Ray, >100 Gy

Uncertainty Source		**Type A (%)**	**Type B (%)**
Alanine Dose Rate (GC207 Center)		0.25	0.55
Alanine Response		0.36	0.15
Temperature Correction			0.10
Dose Rate Effect			0.10
Calibration Curve		0.50	0.10
	sqrt(sum)	0.66	0.59
	combined in quadrature		0.89
	t-factor for 45 d.f at 95.45 %		2.06
	Expanded Uncertainty at 95.45 % conf.		**1.8**