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.

Single-Hole vial geometry (SH): irradiation geometry with pellets stacked vertically in a polystyrene vial that is placed in the absolute center of the isodose irradiator-source profile. This geometry is used as the calibration point to which all other geometries are referenced.

Equipment

Essential Equipment	Calibration Method	Calibration Frequency
^{60}Co Pool Source	Comparison to Vertical Beam Source	Determined by control charts
MDS Nordion Gammacell 45	Comparison to Pool Source	Determined by control charts
MDS Nordion Gammacell 232	Comparison to Pool Source	Determined by control charts

Essential Equipment	Calibration Method	Calibration Frequency
MDS Nordion Gammacell 207	Comparison to Pool Source	Determined by control charts
Bruker ECS 106 EPR Spectrometer	Dosimeter Check Dose Measurement	As needed
Microbalance	External Service	Annual
Platinum Thermometer	External Service	As needed
Type-T Thermocouple	Comparison to Platinum Thermometer	As needed

Health & Safety Precautions

Radiation safety
Rooms containing large ^{60}Co sources have been designated as High Radiation Areas. Radiation safety and training services are provided by the NIST Health Physics Group.

Magnetic field safety
Individuals with pacemakers should avoid rooms containing electromagnets associated with EPR spectrometers.

Procedures

1. *Dosimeter Batch evaluation and testing*

1.1 General

1.1.1 This procedure describes the requirements for performance characterization of transfer dosimeters used in NIST high-dose dosimetry certification services. It is intended to provide data that identifies influence quantities that may have significant effects on the performance of a dosimetry system.

1.1.2 This procedure is intended for routine evaluation of a commercially available dosimetry system that is currently in use. Evaluation of a new dosimetry system may require additional tests or more extensive testing not described here.

1.1.3 Irradiation and measurement procedures are described in Sections 3 and 4, respectively. Irradiations should be done at ambient temperature, unless stated otherwise.

1.1.4 Data from all tests described in this section shall be recorded in the Dosimetry System Databook.

1.1.5 Acceptance criteria for the tests described in this section are listed in Appendix A. A failure to meet these criteria shall result in a repeat of the specific test(s) that failed. Continued failures must be resolved by either correcting or controlling the influence

quantity. If corrective action is required, see IRD Guide IRD-G-08.

1.1.6 Reasonable efforts should be made to minimize the exposure of stored pellets to environmental influences despite data that indicate exposure of commercial alanine pellets to moderate variations in ambient temperature, relative humidity (RH), and light pose no long-term problem.

1.2 Selection

1.2.1 Determine the total number of pellets needed for the performance characterization.

1.2.2 The selection of test pellets should attempt to be random, both in creating the working subset of pellets (1.2.3 below) and in selecting from this subset of pellets to perform specific tests.

1.2.3 If the dosimeter batch is distributed over several containers, the selection process should include pellets from each container to the extent practical. Containers should possess or be marked with unique identifiers.

1.2.4 Records should include a correlation between the pellet identifier, its batch, and the container from which it was removed.

1.3 Mass test

1.3.1 This test is required for alanine pellet dosimeters only.

1.3.2 Select and weigh pellets to the nearest 0.0001 g.

1.3.3 Plot a histogram of the mass distribution.

1.3.4 Determine the mean, standard deviation, and relative standard deviation (RSD) of the pellets measured.

1.4 Height test

1.4.1 This test is required for alanine pellet dosimeters only.

1.4.2 Select and measure the pellet heights to the nearest 0.01 mm.

1.4.3 Plot a histogram of the height distribution.

1.4.4 Determine the mean, standard deviation, and relative standard deviation of the pellets measured.

1.5 Response variation

1.5.1 Irradiate 4 pellets to each of the prescribed doses: 0.10 kGy, 1.0 kGy, 10 kGy, and 100 kGy.

1.5.2 Measure the EPR response (see Section 4) of the individual pellets.

1.5.3 Determine the mean, standard deviation, and relative standard deviation of the measured pellets grouped by prescribed absorbed dose.

1.6 Dose fractionation
- 1.6.1 Irradiate pellets to prescribed doses.
  - 1.6.1.1 Irradiate six pellets (groups 3A & 3B) to 1.0 kGy, wait 1 hour then irradiate the six pellets to an additional 1.0 kGy, remove three pellets (group 3B), wait 1 hour then irradiate the group 3A pellets to an additional 1 kGy, wait overnight (~20 hours), then irradiate group 3B to an additional 1.0 kGy.
  - 1.6.1.2 Irradiate three pellets (group 3C) to 3.0 kGy.
  - 1.6.1.3 Irradiate six pellets (groups 30A & 30B) to 10 kGy, wait 1 hour then irradiate the six pellets to an additional 10 kGy, remove three pellets (group 30B), wait 1 hour then irradiate the group 30A pellets to an additional 10 kGy, wait overnight (~20 hours), then irradiate group 30B to an additional 10 kGy.
  - 1.6.1.4 Irradiate three pellets (group 30C) to 30 kGy.
- 1.6.2 After a 24 h wait period from the last irradiation, measure the absorbed dose for all pellets.
- 1.6.3 Determine the mean, standard deviation, and relative standard deviation of the pellet groups measured, and compare fractionated doses to continuously applied doses.

1.7 Relative humidity (RH)
- 1.7.1 Store 12 pellets in controlled 0 % RH and 55 % RH environments [1].
- 1.7.2 After ~4 days, remove pellets and quickly seal pellets in groups of three in a sealed vial
- 1.7.3 Irradiate a pair of 0 % RH and 55 % RH pellets to 1.0 kGy.
- 1.7.4 Irradiate a pair of 0 % RH and 55 % RH pellets to 10 kGy.
- 1.7.5 Allow pellets to equilibrate with the laboratory ambient RH for 1 to 2 hours before measuring the response for each pellet.
- 1.7.6 Determine the mean, standard deviation, and relative standard deviation of the pellet groups measured and compare by absorbed dose delivered and RH.

1.8 Post-irradiation time dependence
- 1.8.1 Irradiate six pellets, three to 1.0 kGy and three to 10 kGy.
- 1.8.2 Measure the absorbed dose approximately every other day for 2 weeks.
- 1.8.3 Determine the mean, standard deviation, and relative standard deviation of the measured pellets according to absorbed dose delivered.

1.9 Irradiation temperature coefficient

1.9.1 In the aluminum irradiation geometry, irradiate pellets to 10 kGy at each of the three irradiation temperatures: ambient; ambient + 20 °C; and ambient - 20 °C.

1.9.2 Measure the EPR response of the individual pellets.

1.9.3 Compute the resultant temperature coefficient (slope of the percent change, relative to the predicted value at the ambient temperature, in response versus the irradiation temperature) and compare this value to the accepted value.

1.10 Batch mean

1.10.1 Irradiate simultaneously (co-located) two pellets from previous batch with two pellets from the batch undergoing evaluation to each of the following doses: 1.0 kGy and 10 kGy.

1.10.2 Measure the EPR response (not absorbed dose) of the individual pellets.

1.10.3 Determine the mean of the pellet groups and compute the percent difference in response between the batches.

2. *Instrument maintenance*

2.1 ECS106 EPR Spectrometer

2.1.1 The in-line filter for the cooling water should be maintained according to the manufacturer's guidelines.

2.1.2 As necessary, or before each new calibration curve is measured, the quartz sample tube should be cleaned.

2.1.2.1 Note the depth of the dip in the tune mode pattern on the spectrometer display.

2.1.2.2 Dampen a long stick cotton swab with methanol and insert into the sample tube to clean the pellet resting area.

2.1.2.3 Draw a vacuum until sample tube is dry (as evidenced by the depth of the dip in the tune mode pattern on the spectrometer display).

2.1.3 Record major maintenance activities in the ECS106 maintenance log book.

3. *Dosimeter batch calibration*

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

3.1 Initiate irradiation data record

3.1.1 Refer to this section heading in IRD Procedure 11.

3.2 Prepare pellets for irradiation
3.2.1 Refer to this section heading in IRD Procedure 11.

3.3 Control pellet temperature
3.3.1 Refer to this section heading in IRD Procedure 11.

3.4 Operate Gammacell
3.4.1 Refer to this section heading in IRD Procedure 11.

3.5 Record irradiation data
3.5.1 Refer to this section heading in IRD Procedure 11.

3.6 Measure pellets
3.6.1 After all irradiations are completed, measure the pellets as described in Section 4.

3.7 Analyze data
3.7.1 Enter dose and measured response values into the curve fitting software, TableCurve2D (made by Systat Software Inc.). Fit the data to one or more equations, examples include: linear; 3rd order polynomial; exponential saturation.
3.7.2 Choose the best fit, based on experience, residuals analysis, and F-statistic.

3.8 Record calibration curve
3.8.1 Print the graph of the fitted equation and the graph of the residuals (in percent).
3.8.2 Paste the two graphs into the Dosimetry System Databook.
3.8.3 Type the new curve coefficients into the appropriate formulas of the Excel spreadsheets that calculate dose from pellet response.

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) in which to keep all printed documents.
4.1.3 Mail dosimeters (see Section 6.2) for requested test (service) numbers with instruction letter to the customer (Appendix B)
4.1.3.1 Each dosimeter consists of four alanine pellets in a polystyrene vial [2].
4.1.4 Input PO and customer information into Information System to Support Calibrations (ISSC) database to obtain a folder number.

4.1.5 After folder number is obtained, print fee sheet and division record.
4.1.6 Fax PO and folder number assignment to Measurement Services Division.
4.1.7 Receive calibration test folder via NIST internal mail and add it to the HD folder along with PO, fee sheet, and division record.

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
4.2.2 Turn on the ECS106 Spectrometer (see Section 4.3)
4.2.3 Remove pellets from vials, numbering consecutively (top to bottom) as removed from the vial
4.2.4 Empty vials are cleaned by immersing in ethanol with agitation, allowing them to remain immersed overnight, then dried in a fume hood overnight
4.2.5 Weigh pellets (see Section 4.4) and input masses into Excel spreadsheet
4.2.6 Create Excel spreadsheet (see Section 4.5) to record data from measurements

4.3 ECS106 setup
4.3.1 Turn on magnet cooling water chiller
4.3.2 Turn on magnet
4.3.3 Turn on the ECS106 computer
4.3.4 Logon to the ECS106, using the appropriate login name
4.3.5 Once the standby light changes from red to green, use the right arrow key to change to tune mode
4.3.6 With a pellet in the EPR sample tube, use the up-arrow key to perform the auto-tune procedure at least twice, and allow approximately one hour for the spectrometer to warm up, then run the auto-tune procedure once more
4.3.7 Load the spectral parameters that were used for the calibration curve (refer to the corresponding spreadsheet file)
4.3.8 Load the ruby EPR reference parameters
4.3.9 Load the applicable measurement routine command file (see Appendix C)
4.3.10 Set the parameters to the instrument by scrolling to the Parameters list and clicking the ‘greater than’ (>) sign

4.4 Mass determination
4.4.1 Measure the pellet mass and record the data by utilizing the computer local to the analytical microbalance

4.5 Excel spreadsheet setup

4.5.1 Verify that the Parameter worksheet contains the ECS106 parameters matching those of the corresponding calibration file

4.5.2 The measurement worksheet should include the date, vial number, target dose, pellet mass, alanine signal max/min peak height and ruby signal max/min peak height, peak-to-peak amplitude, and mass-ruby normalized signal amplitudes (response)

4.5.3 The summary worksheet should include the company name, date, calibration function and the corresponding solution formula, coefficients of the calibration curve being used, the calculated mass-ruby normalized signal (from the measurement worksheet), the calibration temperature and temperature coefficient, and the irradiation temperature (from the customer instruction letter)

4.5.4 The summary worksheet should calculate the temperature corrected response and absorbed dose

4.6 ECS106 measurement

4.6.1 For each pellet to be measured, use the in-house vacuum line with suction attachment and insert pellet into the instrument, ensuring that the instrument remains tuned (meters in the spectrometer display should be at the midpoint, else the spectrometer should be retuned).

4.6.2 Run the measurement procedure by clicking A (Acquisition), then E (Execute) on the drop down menu.

4.6.3 When all pellets have been measured, record the maximum and minimum peak-to-peak values for the alanine and ruby measurements according to the procedure found in the Dosimetry System Databook.

4.7 Analysis

4.7.1 The mean response and RSD is calculated for each group of four pellets within a vial. Check the RSD for each grouping, perform an outlier test and/or repeat measurements as needed. The mean response is the value used to calculate the absorbed dose. However, an increasing pellet response trend that exceeds a 1 % RSD across the stack of four alanine pellets in the vial is considered to be a significant absorbed dose gradient. In this case, the absorbed dose shall be calculated and reported for each pellet.

4.7.2 Calculate dose by using the coefficients from the applicable calibration curve (refer to Dosimetry System Databook)

4.7.3 Check the calculations and data for accuracy.

4.7.4 Print copies of data, and insert in Transfer Dosimetry Databook

4.7.5 Copy Transfer Dosimetry Databook pages and insert into HD folder

4.8 Report and test closure

4.8.1 Complete the QM Checklist (Appendix D)

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 E)

4.8.3 Log in to the ISSC database and close out the test and folder (see ISSC quality manual)

4.8.4 Sign Fee sheet and Division Record (will be dated by Administrative Officer)

4.8.5 Make two copies of the Fee sheet, and one copy of the Division Record

4.8.6 Staple together the original Fee sheet, one copy of the Fee sheet, and a copy of the customer purchase order

4.8.7 Paperclip original Division Record to stapled Fee sheets

4.8.8 Sign blue test folder, and check the three year box

4.8.9 Staple the contents of the test folder, with a copy of the calibration report on top, to the inside back cover of the test folder

4.8.10 Staple copy of the Fee sheet to the inside front cover of the test folder

4.8.11 Staple copy of the Division Record to the inside front cover of the HD folder

4.8.12 Paperclip copies to the front of the test folder and place folder in Administrative Officer's inbox

4.8.13 Write "Mailed on" date on copy of calibration report and place in HD folder

5. Quality control

5.1 Absorbed-dose check standards

Approximately every two months, or as needed, dosimeter pellets are irradiated to the following doses: 0.025 kGy, 1.0 kGy, 10 kGy, and 50 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 compiled into a control chart for tracking and comparison in the Quality Control Databook. Check dose measurements that measure outside of set limits must be resolved through re-measurement, repetition of the check standard process, or reconfiguration of spectrometer settings (the latter may require a total 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 IRD participates in larger international comparisons organized by the BIPM when offered (approximately every ten years). 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 (See: http://ts.nist.gov/ts/htdocs/230/233/calibrations/Publications/series-pdf/SP250-44.pdf) [3].

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 [4]. 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 F.

Water Calorimetry: uncertainty from realization of the Gy [5].

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 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 (See Appendix G).

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. 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.
4. NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, 1994.
5. Domen, S.R., A sealed water calorimeter for measuring absorbed dose, NIST J. of Res., 99, pp. 121 – 141, 1994.
6. M.F. Desrosiers, J.M. Puhl, S.L. Cooper, 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.

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/C217
Internal Calibrations	Source ratio measurements and data analysis	245/C217
Quality Control	Check dose measurements and other routine quality control	245/C217
ECS106 User Log Book	User records	245/C207
ECS106 Maintenance Log Book	Maintenance records	245/C207
Transfer Dosimetry Databook	Records all transfer dosimeter certification data	245/C217
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 IRD Quality Manager shall maintain the original and past versions of this IRD Procedure.

Appendix A – Dosimeter Batch Acceptance Criteria

Dosimeter Test	Acceptance Level
Mass	≤ 10 %
Height	≤ 10 %
Dosimeter Response	≤ 1 %
Fractionation	≤ 2 %
Relative Humidity	≤ 3 %
Time	≤ 2 %
Temperature Coefficient	≤ 30 %
Batch Response	≤ 20 %

Appendix B – Sample Transfer Letter

September 3, 2003

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 three 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
Radiation Interactions & Dosimetry Group
Physics Laboratory
PHONE: 301-975-xxxx FAX: 301-869-7682 E-MAIL: caltech@nist.gov
Enclosures

Appendix C – Example EPR Measurement Routine Program

ECS106 Acquisition routine, Alaplusruby:

```
1> setp
2> wait 5 s
3> mtu
4> racq
5> tp inc 2
6> pg inc 1
7> setp
8> wait 5 s
9> mtu
10> racq
11> tp inc 2
12> pg inc 1
13> setp
```

Appendix D – 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 E – 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 James Puhl

Report reviewed by Marc F. Desrosiers

Report approved by

Michael G. Mitch, Leader
Radiation Interactions and Dosimetry Group

For the Director
National Institute of Standards and Technology
By

Lisa R. Karam, Chief
Ionizing Radiation Division
Physics Laboratory

Information on technical aspects of this report may be obtained from James Puhl, NIST, 100 Bureau Drive Stop 8460, Gaithersburg, MD 20899, 301-975-5581

REFERENCE HD0000
TEST FOLDER 123456
NIST DB 2000/000
October 8, 2003
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, 2003, using a Bruker ECS106 spectrometer. Dose interpolations are based on a NIST calibration of batch T030901 alanine dosimeters performed February 25, 2003. 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://physics.nist.gov/Divisions/Div846/QualMan/index.html) 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 %.

Appendix F- 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**

Appendix G- Calibration Curve Fit Uncertainty Estimation

The following protocol is used to estimate uncertainties associated with the curve fit to calibration data. These protocols were developed in consultation with the NIST Statistical Engineering Division (SED). Our results were validated by comparing our computations to the NIST SED computations from identical data sets. All files cited here are located in the "High Dose" folder on the shared network drive.

For a linear function:

1. Open linear_fit_uncertainty.xls.
2. Follow instructions located there.

For non-linear functions:

1. Save x-y data as a text file.
2. Open appropriate ".boot" macro with NotePad (for macro details see below).
3. Edit read line to reflect text file name.
4. Save file.
5. Run DataPlot.
6. In DataPlot text window type "call abcd.boot" (where abcd is the selected macro name) and press enter.
7. DataPlot will write results as "abcd.out".
8. Open abcd.out in NotePad and select an appropriate uncertainty.

Code for cubic.boot:

```
dimension 40 columns
reset data
. ..device 1 x11
. ..
skip 1
read kodak307data.txt x y
. ..
let n = number y
let minx = minimum x
let maxx = maximum x
let minx = minx - 2
let maxx = maxx + 2
. ..
cubic fit y x
let ycalib = distinct pred
let ncalib = size ycalib
.
.  Check fit first
.
```

```
line bl so
char x bl
plot y pred vs x
.
let x1 = x
let x2 = x*x
let x3 = x*x*x
bootstrap samples 200
bootstrap fit y x1 x2 x3
skip 0
read dpst1f.dat a0coef a1coef a2coef a3coef
system cp dpst1f.dat bootstrap_coef.txt
let nboot = size a0coef
. ..
. .. Now loop to perform the calibrations
. ..
feedback off
printing off
.
loop for k = 1 1 ncalib
  let y0 = ycalib(k)
  loop for l = 1 1 nboot
    let a0 = a0coef(l)
    let a1 = a1coef(l)
    let a2 = a2coef(l)
    let a3 = a3coef(l)
    let function f1 = a3*x**3 + a2*x**2 + a1*x + a0 - y0
    let r = roots f1 wrt x for x = minx maxx
    let rr = r(1)
    let xtemp(l) = rr
    let xtag(l) = k
  end of loop
  if k = 1
    let x0 = xtemp
    let tag = xtag
  else
    extend x0 xtemp
    extend tag xtag
  end of if
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
```

```
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write kodak307cubic.out ycalib mb sb rb
. ..
```

Code for katz2term.boot:

```
. ..
. ..  sample bootstrap code for
. ..  calibration interval estimate
. ..  on EXP model inversion
. ..
dimension 20 columns
. ..
skip 1
read marc.nonlinear x y
let n = number y
. ..
fit y = a*(1-exp((-x)/b))
let astart = a
let bstart = b
. ..
let pred2 = pred
let y0 = distinct pred2
let ndist = size y0
let xtag = sequence 1 1 ndist
let res2 = res
let numboot = 200
. ..
let a = astart
let b = bstart
let ind = bootstrap index for i = 1 1 n
let res3 = bootstrap sample res2 ind
let y3 = pred2+res3
fit y3 = a*(1-exp((-x)/b))
let x0 = (-b)*log(1-(y0/a))
let tag = xtag
. ..
loop for k = 2 1 numboot
   let a = astart
   let b = bstart
   let ind = bootstrap index for i = 1 1 n
```

```
   let res3 = bootstrap sample res2 ind
   let y3 = pred2+res3
   fit y3 = a*(1-exp((-x)/b))
   let xtemp = (-b)*log(1-(y0/a))
   extend tag xtag
   extend x0  xtemp
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write katz2term.out y0 mb sb rb
. ..
. .. exit
```

Code for katz3term.boot:

```
. ..
. ..  sample bootstrap code for
. ..  calibration interval estimate
. ..  on EXP model inversion
. ..
dimension 20 columns
. ..
reset data
skip 1
read gs604datald.txt x y
let n = number y
. ..
let function f = a*(1-exp(-(x+b)/c))
pre-fit y = f for a = 50 25 500 for b = .01 0.05 .1 for c = 25 25 150
fit y = f
let astart = a
let bstart = b
let cstart = c
. ..
let pred2 = pred
let y0 = distinct pred2
let ndist = size y0
let xtag = sequence 1 1 ndist
```

```
let res2 = res
let numboot = 200
. ..
let a = astart
let b = bstart
let c = cstart
let ind = bootstrap index for i = 1 1 n
let res3 = bootstrap sample res2 ind
let y3 = pred2+res3
fit y3 = a*(1-exp(-(x+b)/c))
let x0 = -b-(c*log(-(y0-a)/a))
let tag = xtag
. ..
loop for k = 2 1 numboot
   let a = astart
   let b = bstart
   let c = cstart
   let ind = bootstrap index for i = 1 1 n
   let res3 = bootstrap sample res2 ind
   let y3 = pred2+res3
   fit y3 = a*(1-exp(-(x+b)/c))
   let xtemp = -b-(c*log(-(y0-a)/a))
   extend tag xtag
   extend x0  xtemp
end of loop
. ..
skip 1
tabulate standard deviation x0 tag
read dpst1f.dat junk sb
tabulate mean x0 tag
read dpst1f.dat junk mb
let rb = (sb/mb)*100
. ..
write gs604ld.out y0 mb sb rb
. ..
line bl so
char x bl
plot y0 vs mb
. ..
```