CALIBRATION IRRADIATIONS OF CUSTOMER SUPPLIED DOSIMETERS WITH ^{60}Co GAMMA RAYS

Purpose

The purpose of this procedure is to describe the setup, measurement, and reporting procedures for irradiation of customer supplied dosimeters.

Scope

This procedure covers the irradiation of passive dosimeters in the NIST ^{60}Co Gammacell irradiators. The irradiator dose rates are comparable to those used in the sterilization and radiation processing industries. Customer-supplied dosimeters are irradiated at prescribed temperatures and returned to the customer with an absorbed-dose irradiation certificate.

Definitions

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

5-hole cup – A polystyrene cylinder with five equidistant holes. Used for electronic equilibrium build-up material when irradiating perspex or ampoule dosimeters.

Equipment

Essential Equipment	Calibration Method	Calibration Frequency
^{60}Co Vertical Beam Source	Water Calorimeter	See RPD Procedure 6
^{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
MDS Nordion Gammacell 207	Comparison to Pool Source	Determined By Control Charts
Platinum Thermometer	External Service	As Needed
Type-T Thermocouple	Comparison to Platinum Thermometer	As Needed

Health & Safety Precautions

Radiation safety

Rooms containing ^{60}Co sources have been designated as High Radiation Areas. Radiation safety training and assessment services are provided by the NIST Gaithersburg Radiation Safety Division (GRSD).

Procedures

These are the general customer dosimeter calibration procedures with specific details updated and outlined in the Irradiation Facilities Record Book (IFRB).

Receive Customer Irradiations Inquiry

Upon receipt of customer inquiry for irradiation services, provide customer with quotation(s) for the services based upon the customer's type of dosimeters and job temperature specifications as per current Ionizing Radiation Measurements fee schedule; await receipt of customer supplied Purchase Order (PO).

Receipt of Customer Purchase Order (PO)

Upon receipt of customer purchase order (PO) log service request into the Calibration Log Book (CLB) and submit a Test Folder Request for administrative processing within the NIST Calibration Support System (CSS). After notification from the CSS, the customer can be authorized to ship the dosimeters.

Receive/Store Dosimeters

Upon receipt of dosimeter(s) shipment from the customer, inspect for any damage. The shipping package should be examined as well as the contents. Dosimeter packaging should be checked for damage to seals (if applicable). If the dosimeters are unsealed, inspect them for any damage (i.e., cuts, breaks, scratches). If damage has occurred, follow Guide RPD-G07. In all instances, if there are any signs of damage, notify the customer.

Establish a Dosimeter Calibration Service Plan

Prior to dosimeter(s) calibration, establish a Dosimeter Calibration Service Plan (DCSP) that will outline the specific actions and details for the completion of the calibration service. The DCSP is reviewed by the Dosimetry Group Leader or staff member to ensure that that the dosimeters are irradiated according to customer's specifications. The DSCP serves as a guide during the calibration service and is retained in the customer's test file upon completion of the needed services.

Initiate Irradiation Data Record

Set up the appropriate Excel spreadsheet and Timing Worksheet form as required in preparation for dosimeter irradiation services.

By using an Excel workbook (databook) from the customer's previous irradiation services, or another similar irradiation service Excel workbook (databook), configure a workbook and Timing Worksheet form specific for this particular customer's dosimeter irradiation service(s) to be performed.

Prepare Dosimeters for Irradiation

Prepare the dosimeters (Perspex dosimeter, Alanine dosimeters, Film dosimeters, or other previously calibrated customer supplied or NIST calibration geometry) and Quality Control (QC) pellets for irradiation service

Control Sample Temperature

Samples are normally irradiated at ambient temperature, 22 ^{o}C to 25 ^{o}C. The GC45 does not require cooling to maintain ambient temperature, but blowing compressed air into the sample chamber is necessary to maintain that temperature in the GC232 or GC207. This is done by opening the valve of the flow meter mounted on the side of the appropriate Gammacell. Temperature is monitored with a type-T thermocouple placed inside the sample chamber. The type-T thermocouple is calibrated against the high-precision platinum thermometer in the Gammacell sample chamber (in the “up” position) over a temperature range that corresponds to service irradiations. The operational status of the thermocouple is monitored by periodic checks and control charts. Thermocouples that do not perform within the control limits are replaced. The thermocouple calibration protocol can be found in the Irradiation Facilities Record Book.

Start the temperature-recording computer program.

Active cooling with the ThermoJet, or Vortex, air chiller is available for the GC232 and GC207 to maintain sample temperatures up to about +75 °C or down to -75 ^{o}C.

Operate Gammacell

Operate the Gammacell(s) as standardly prescribed for the safe operation of GC45, GC232, and/or GC207 to ensure proper laboratory safety and associated functionality for the Gammacell(s) being used during the irradiation service.

Record Irradiation Data

Record all appropriate irradiation data before, during, and after each irradiation as needed to ensure accurate completion of a particular dosimeter irradiation on the customer’s Timing Worksheet form and/or associated job Excel workbook (databook).

Analyze Quality Control

The QC pellet doses are measured as described in Procedure RPD-P-12. If the measured dose is within control limits (±5 %), the quality check is successful. If the measured dose is beyond the control limits, halt all irradiations and investigate to determine the source of the discrepancy. If the discrepancy cannot be resolved, the irradiations must be repeated.

Return Dosimeters

After all irradiations have been completed and QC pellets have been validated to be within control limits, unless otherwise requested by the customer, ship the dosimeters back to the customer. Package the dosimeters appropriately to avoid any damage during transit. Include a copy of the worksheet databook record.

Issue certificate and input ISSC to close folder

Once the quality check is successful, write a report entitled “Absorbed-Dose Irradiation Certificate.” An example is shown in Appendix A. By using a Certificate report from the customer’s previous irradiation services, or another similar irradiation service certificate report, configure a new certificate report that is specific for this particular customer’s dosimeter irradiation service(s) that was performed. Route the report for the required signatures, which will result in the mailing of the original to the customer. A CSS database notification will be generated which will signify the closing out the test and folder (reference CSS quality manual).

Determination of Uncertainties

The basis for the determination of uncertainties associated with high-dose irradiations is the *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results* [1]. The purpose of this section is to explain the derivation of the various components of uncertainty for absorbed-dose certification. The values for the uncertainty components are listed in Appendix B.

Water Calorimetry: This is the published uncertainty from realization of the gray (Gy) [2].

Source Ratio Data: This is the statistical determination of uncertainty from the measurement of ratios of electron paramagnetic resonance (EPR) signal response for both water calorimetry geometry vs. pool source, and pool source vs. Gammacell geometry.

Geometry Correction Factor: This is the statistical determination of uncertainty from the measurement of ratios of EPR signal response for single-hole vial geometry vs. film block, Perspex, or ampoule geometry.

Field Uniformity: This is a Type-B estimate of uncertainty for radiation field uniformity within a dosimeter volume.

Timer: This is a Type-B estimate of uncertainty for the timer readout relative to the shortest irradiation time interval.

Decay Correction: This is a Type-B estimate of uncertainty for the ^{60}Co half-life value.

Traceability

The SI unit of absorbed dose is the Gy. For this service, the Gy is realized through water calorimetry measurements in the Vertical Beam ^{60}Co Gamma-Ray Source [2]. These measurements are transferred to the three Gammacell calibration sources by source-rate ratio measurements using alanine dosimetry. These transfer measurement protocols were first described in the NIST SP250-44 and later partially revised as described in a NIST Journal of Research manuscript [3, 4].

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. Larger (i.e., among more laboratories) international comparisons occur approximately every 10 years [5]. These data are summarized in the High-Dose International Comparisons Databook.

References

1. NIST Technical Note 1297, Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results, 1994.
2. Domen, S.R., A sealed water calorimeter for measuring absorbed dose, J. Res. Natl. Inst. Stand. Technol., 99, pp. 121 – 141, 1994.
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. CCRI supplementary comparison of standards for absorbed dose to water in ^{60}Co gamma radiation at radiation processing dose levels, D.T. Burns, P.J. Allisy-Roberts, M.F. Desrosiers, V. Yu. Nagy, P.H.G. Sharpe, R.F. Laitano, K. Mehta, M.K.H. Schneider, Y. Zhang, Radiat. Phys. Chem. 75 (2006) 1087-1092.
5. Discovery of an Absorbed-Dose / Dose-Rate Dependence for the Alanine-EPR Dosimetry Systems and Its Implications in High-Dose Ionizing Radiation Metrology, M.F. Desrosiers, J.M. Puhl, S.L. Cooper, NIST J. of Res., 113 (2008) 79-95.

Records

Record	Contents/Purpose	Location
Calibration Log Book	Login all tests to obtain test folder number	245/C217
High-Dose Irradiations Databook	Records all dosimeter calibrations	245/C217
Irradiation Facilities Record Book	Records dose rates for irradiation geometries and instructions	245 facility

Record	Contents/Purpose	Location
High-Dose International Comparisons Databook	Interlaboratory measurement comparisons data summaries	245/C217
Internal Calibrations	Source ratio measurements and data analysis	245/C217
Gammacell User Log Books	^{60}Co irradiator logs	245 facility

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 in the "High Dose" folder on the shared network drive. The Radiation Physics Division (RPD) Quality Manager shall maintain the original and all past versions of this RPD Procedure.

Appendix A – Example Certificate

National Institute of Standards and Technology

ABSORBED-DOSE IRRADIATION CERTIFICATE

NIST Service Identification Number 49010C

Dosimeter
Film Dosimeters Type 92 Batch 17

Customer
Steel Curtain Irradiators, Inc.
00 Points Road
Pittsburg, PA 18888

ATTN: T. Bradshaw
Reference: PO# 2009-1234

Irradiation performed by Lonnie T. Cumberland

Reviewed by Marc F. Desrosiers

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
Radiation Physics Division
Physical Measurement Laboratory

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

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

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Steel Curtain Irradiators supplied radiochromic film dosimeters in pre-sealed foil pouches. The dosimeters were irradiated using gamma radiation from a calibrated ^{60}Co irradiator, the NIST Gammacell 220-207. During irradiation, the dosimeter pouches were held in a polystyrene block assembly with a wall thickness of 5 mm. The dates of irradiation, values of dose rate, absorbed dose, and mean irradiation temperature were as follows:

Dosimeter Identification	Date of Irradiation	Dose Rate (kGy/h)	Irradiation Temp. °C	Absorbed Dose kGy(H_2O)
5A-5E	June 14, 2015	10.2	25	5.0
10A-10E	June 13, 2015	10.2	25	10.0
15A-15E	June 13, 2015	10.2	25	15.0
20A-20E	June 14, 2015	10.2	25	20.0
25A-25E	June 13, 2015	10.2	25	25.0
30A-30E	June 12, 2015	10.2	25	30.0
35A-35E	June 12, 2015	10.2	25	35.0
40A-40E	June 12, 2015	10.2	25	40.0
45A-45E	June 14, 2015	10.2	25	45.0
50A-50E	June 14, 2015	10.2	25	50.0
55A-55E	June 13, 2015	10.2	25	55.0

UNCERTAINTIES AND RELATED FACTORS IN HIGH-DOSE IRRADIATIONS

High-Dose Irradiations in Standard Geometries using the NIST ^{60}Co Gammacell 220-207

(Expanded uncertainty: ±1.3 % at a 95 % confidence level)

The high-dose irradiations at NIST involve the administration of ^{60}Co gamma radiation under environmentally controlled conditions. The dose values are based on standard water calorimeter measurements and EPR/Alanine dosimetry, which are corrected by certain modifying factors (such as the geometry attenuation factor and source decay factor).

The uncertainty cited above is pertinent to absorbed dose in water in calibrated geometries. 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.physics.nist.gov/Divisions/Div846/QualMan/index.html) or by requesting such 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 ±0.25 % at an approximate level of confidence of 68 %.

Type B Uncertainties

The combined standard uncertainty based on scientific judgment is estimated to be ±0.55 % 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 2.16 to yield an expanded uncertainty of ±1.3 % at an approximate level of confidence of 95 %.

Appendix B - Sample Table of Uncertainty

d.f.	Gammacell 207 Calibration Geometry Dose Rate Uncertainty Source	Type A (%)	Type B (%)
6	Water Calorimetry in Vertical Beam	0.16	0.51
7	GC207/Pool Source Ratio Data	0.08	
7	Pool/B036 Source Ratio Data	0.17	
3	Geometry Correction Factor (Perspex)	0.10	
	Field uniformity		0.01
	Timer Error (irradiation time > 8min)		0.20
	^{60}Co Decay Correction		0.02
	sqrt(sum)	0.27	0.55
effective d.f.	combined in quadrature		0.61
19	t-factor for 19 d.f at 95.45 %		2.14
	Expanded Uncertainty at 95.45 % conf.		**1.3**

Other standard irradiation geometries used in Gammacell 207, as well as other geometries in Gammacells 45 and 232, will have slightly different values for source ratio uncertainty and geometry correction factor uncertainty. In those cases, the expanded uncertainty remains unchanged at 1.3 %. For more specialized geometries, such as low-temperature irradiations held inside a Dewar, additional components of uncertainty must be included and the uncertainty recomputed.

Appendix C - **QM Checklist for High-Dose Irradiation Services**

NIST ID:

Preliminary Analysis

_ An irradiation geometry matching this type of dosimeter/sample has been previously calibrated.

_ The correct stem height for the samples has been identified. Or, if a stem is not used, the geometry is confirmed as correct according to the dose-rate calibration.

_ The requested irradiation temperature has been confirmed.

Within the Databook Record Spreadsheet

_ The header fields are all checked for accuracy: contact, company, date, HD#, etc.

_ The written description correctly identifies the dosimeters and the irradiation geometry including the stem height and any surrounding materials.

_ The base dose rate used is a number from the current year's dose-rate sheet, and both the reference date and the transit dose time are correct.

_ The cells on the row which calculate elapsed time, decay correction factor, current dose rate, total hours, irradiation time, stop date, and stop time have all been checked for having the proper formulas.

_ The values have been entered correctly for start date, start time, total dose and they match with the handwritten record of the irradiation worksheet.

_ After each irradiation, the independent backup timer was noted and confirmed the time as recorded in the spreadsheet.

Signed by:______________________________________ Date: _______________