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 curing 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 IRD 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 Health Physics Group.

Procedures

Receive/Store Dosimeters

1. Upon receipt of shipment 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 IRD-G07. In all instances, if there are any signs of damage, notify the customer.
2. In the Calibration Log Book, assign the next available HD number. Next to that, record the customer name, city, state, contact person, purchase order number, and date received.
3. Record the HD number on the Purchase Order, then enter the data into the ISSC database to generate the official test folder number.
4. Store the dosimeters in room B140 while waiting for irradiation. If the dosimeters are unpackaged radiochromic films, protect them from ambient light, but leave them open to the 50 % ± 5 % relative humidity atmosphere.

Initiate Irradiation Data Record

1. Choose the appropriate geometry for the given dosimeters.
   a. Perspex dosimeters fit in the 5-hole cup
   b. Alanine pellet dosimeters fit in the single-hole vial
   c. Pre-packaged films fit in the film-block geometry with the 37 mm square x 5 mm polystyrene slabs
   d. Unpackaged films fit into the polystyrene film blocks with the machined center cavity
   e. Liquid ampoules fit in the 5-hole cup
2. Print a blank copy of the form “QM Checklist for High-Dose Irradiation Services.” An example is shown in Appendix C. All items in the list must be noted and the page is to be signed and retained in the records.
3. Refer to the Irradiation Facilities Record Book to get the dose rate for the given irradiator and geometry.
4. Open the Excel spreadsheet databook template and enter the appropriate header information, including the above mentioned annual dose rate.
5. Before each irradiation, enter an approximate value into the “start time” cell so the embedded function can calculate the decay, dose rate, and irradiation time.
6. Print a blank copy of the form “Timing Worksheet,” which should be a separate sheet within the Excel file. This will be used to keep a handwritten record of the irradiation times.

Prepare Dosimeters for Irradiation

Perspex dosimeters:

1. Roll each dosimeter lengthwise and slip it into one of the five holes of the cup.
2. Choose an appropriate length polystyrene stem according to the printed table in the Irradiation Facilities Record Book.

3. Screw an aluminum base onto the stem.
4. Place two alanine pellets of the current quality control (QC) batch into the top of the stem.
5. Place the 5-hole cup over the stem.

Alanine dosimeters:

1. Fill a single-hole vial with pellets. Depending on the pellet height, it will accept three or four pellets.
2. Choose an appropriate length polystyrene stem according to the printed table in the Irradiation Facilities Record Book.
3. Screw an aluminum base onto the stem.
4. Place two alanine pellets of the current QC batch into the top of the stem.
5. With a foam collar on the stem, place the single-hole vial into the collar to hold it atop the stem.

Film dosimeters:

1. Choose an appropriate length polystyrene stem according to the printed table in the Irradiation Facilities Record Book.
2. Screw an aluminum base onto the stem.
3. Place two alanine pellets of the current QC batch into the top of the stem.
4. Using electrical tape, secure the film block into an upright position atop the stem.

Control Sample Temperature

Samples are normally irradiated at room 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 turbojet air chiller is available for the GC232 and GC207 to maintain sample temperatures down to -77 ^{o}C. To operate the turbojet air chiller:

1. Turn the system on. Allow 10 minutes for system to get fully operational.
2. Switch turbojet from idle to manual mode and change setpoint to adjust sample temperature.
3. Monitor fluctuations in sample temperature and adjust turbojet setpoint when necessary to maintain the desired sample temperature.

Operate Gammacell

1. Place dosimeter assembly into the sample chamber.

2. Set timer to desired irradiation time as calculated by the spreadsheet.
3. Press the "DOWN" (GC45) or "CYCLE START" (GC232) or "START" (GC207) button to begin the irradiation.
4. When the chamber arrives at the down position, note the actual clock time, write it on the timing worksheet, and enter it into the databook spreadsheet.
5. When the timer finishes and sample chamber comes up, remove the dosimeters.

Record Irradiation Data

1. After each irradiation, use the computer program to calculate the average temperature during irradiation. Enter the value into the databook spreadsheet.
2. When all irradiations are finished, save the file, print the spreadsheet and paste it into the High-Dose Irradiations databook. Store the timing worksheet with the other test folder documents.

Return Dosimeters

After all irradiations are completed, ship the dosimeters back to the customer. Package the dosimeters appropriately to avoid any damage during transit. Include a copy of the databook record. Also include a letter explaining that this copy is unofficial and that the official record will be the formal certificate, which will be mailed later.

Analyze Quality Control

Measure the QC pellets as described in Procedure IRD-P-12. Prior to dose assessment, apply the correction factor to the response of the QC pellets to adjust for the different location within the geometry compared to the customer samples. Control charts of QC measurements are maintained in the Quality Control Databook. If the measured dose is within control limits (typically 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.

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. Certificates are generated by retrieving the current format that is stored as a continuously updated template in the internal network folder "\CALIBS\490Xtemplates\". The template file includes multiple uncertainty pages to account for the most common variations of the high-dose calibration services. Generate the certificate by extracting the appropriate pages for the needed certification. Route the report for the required signatures. Make two copies of the completed report before mailing the original to the customer. Note the mailed date on one of the copies. Log into the ISSC database and close out the test and folder (reference ISSC 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 Gy [2].

Source Ratio Data: This is the statistical determination of uncertainty from the measurement of ratios of 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 Gray (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
Quality Control	Check dose measurements and other routine quality control	245/C217
Irradiation Facilities Record Book	Records dose rates for irradiation geometries and instructions	245/B140
High-Dose International Comparisons Databook	Interlaboratory measurement comparisons data summaries	245/C217
Internal Calibrations	Source ratio measurements and data analysis	245/C209
GammaCell User Log Books	^{60}Co irradiator logs	245/B140

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 Ionizing Radiation Division (IRD) Quality Manager shall maintain the original and all past versions of this IRD 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 James M. Puhl

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
Ionizing Radiation Division
Physics Laboratory

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

REFERENCE HD00
TEST FOLDER 00000000000
NIST DB 0000000
June 24, 2009
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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, 2009	10.2	25	5.0
10A-10E	June 13, 2009	10.2	25	10.0
15A-15E	June 13, 2009	10.2	25	15.0
20A-20E	June 14, 2009	10.2	25	20.0
25A-25E	June 13, 2009	10.2	25	25.0
30A-30E	June 12, 2009	10.2	25	30.0
35A-35E	June 12, 2009	10.2	25	35.0
40A-40E	June 12, 2009	10.2	25	40.0
45A-45E	June 14, 2009	10.2	25	45.0
50A-50E	June 14, 2009	10.2	25	50.0
55A-55E	June 13, 2009	10.2	25	55.0

REFERENCE HD00
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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 %.

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Appendix B - Sample Table of Uncertainty

	Gammacell 207 Calibration Geometry Dose Rate		
d.f.	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: _______________