



NIST Special Publication 250-44

Radiation Processing Dosimetry Calibration Services and Measurement Assurance Program

Jimmy C. Humphreys
James M. Puhl
Stephen M. Seltzer
William L. McLaughlin
Marc F. Desrosiers
Debra L. Bensen
Marlon L. Walker

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

IC
00
57
0.250-44
998

The National Institute of Standards and Technology was established in 1988 by Congress to “assist industry in the development of technology . . . needed to improve product quality, to modernize manufacturing processes, to ensure product reliability . . . and to facilitate rapid commercialization . . . of products based on new scientific discoveries.”

NIST, originally founded as the National Bureau of Standards in 1901, works to strengthen U.S. industry’s competitiveness; advance science and engineering; and improve public health, safety, and the environment. One of the agency’s basic functions is to develop, maintain, and retain custody of the national standards of measurement, and provide the means and methods for comparing standards used in science, engineering, manufacturing, commerce, industry, and education with the standards adopted or recognized by the Federal Government.

As an agency of the U.S. Commerce Department’s Technology Administration, NIST conducts basic and applied research in the physical sciences and engineering, and develops measurement techniques, test methods, standards, and related services. The Institute does generic and precompetitive work on new and advanced technologies. NIST’s research facilities are located at Gaithersburg, MD 20899, and at Boulder, CO 80303. Major technical operating units and their principal activities are listed below. For more information contact the Publications and Program Inquiries Desk, 301-975-3058.

Office of the Director

- National Quality Program
- International and Academic Affairs

Technology Services

- Standards Services
- Technology Partnerships
- Measurement Services
- Technology Innovation
- Information Services

Advanced Technology Program

- Economic Assessment
- Information Technology and Applications
- Chemical and Biomedical Technology
- Materials and Manufacturing Technology
- Electronics and Photonics Technology

Manufacturing Extension Partnership Program

- Regional Programs
- National Programs
- Program Development

Electronics and Electrical Engineering Laboratory

- Microelectronics
- Law Enforcement Standards
- Electricity
- Semiconductor Electronics
- Electromagnetic Fields¹
- Electromagnetic Technology¹
- Optoelectronics¹

Chemical Science and Technology Laboratory

- Biotechnology
- Physical and Chemical Properties²
- Analytical Chemistry
- Process Measurements
- Surface and Microanalysis Science

Physics Laboratory

- Electron and Optical Physics
- Atomic Physics
- Optical Technology
- Ionizing Radiation
- Time and Frequency¹
- Quantum Physics¹

Materials Science and Engineering Laboratory

- Intelligent Processing of Materials
- Ceramics
- Materials Reliability¹
- Polymers
- Metallurgy
- NIST Center for Neutron Research

Manufacturing Engineering Laboratory

- Precision Engineering
- Automated Production Technology
- Intelligent Systems
- Fabrication Technology
- Manufacturing Systems Integration

Building and Fire Research Laboratory

- Structures
- Building Materials
- Building Environment
- Fire Safety Engineering
- Fire Science

Information Technology Laboratory

- Mathematical and Computational Sciences²
- Advanced Network Technologies
- Computer Security
- Information Access and User Interfaces
- High Performance Systems and Services
- Distributed Computing and Information Services
- Software Diagnostics and Conformance Testing

¹At Boulder, CO 80303.

²Some elements at Boulder, CO.

NIST Special Publication 250-44

NIST MEASUREMENT SERVICES: Radiation Processing Dosimetry Calibration Services and Measurement Assurance Program

Jimmy C. Humphreys, James M. Puhl, Stephen M. Seltzer,
William L. McLaughlin, Marc F. Desrosiers, Debra L. Bensen,
and Marlon L. Walker

Ionizing Radiation Division
Physics Laboratory
National Institute of Standards and Technology
Gaithersburg, MD 20899-0001

March 1998



U.S. Department of Commerce
William M. Daley, Secretary

Technology Administration
Gary R. Bachula, Acting Under Secretary for Technology

National Institute of Standards and Technology
Raymond G. Kammer, Director

National Institute of Standards and Technology Special Publication 250-44
Natl. Inst. Stand. Technol. Spec. Publ. 250-44, 40 pages (Mar. 1998)
CODEN: NSPUE2

U.S. GOVERNMENT PRINTING OFFICE
WASHINGTON: 1998

TABLE OF CONTENTS

	Page
PREFACE	iii
SCOPE	1
REFERENCED DOCUMENTS	1
TERMINOLOGY	1
SIGNIFICANCE AND USE	3
GENERAL CRITERIA	4
SPECIFIC CRITERIA FOR IONIZING RADIATION.....	14
SPECIFIC CRITERIA FOR CALIBRATIONS USING PHOTONS AND ELECTRONS	21
KEYWORDS	36
REFERENCES	36

PREFACE

The basis for this document was developed within the American Society for Testing and Materials (ASTM) subcommittee E10.01 on Dosimetry for Radiation Processing. The version that is published as an ASTM standard is designated as E1400 entitled “Standard Practice for Characterization and Performance of a High-Dose Radiation Dosimetry Calibration Laboratory,” with the latest revision dated 1995. This document differs substantially from the ASTM standard in that it addresses calibration services operated under the direction of NIST staff, whereas the ASTM standard deals with the accreditation of secondary calibration laboratories.

1. Scope

1.1 This document contains descriptions of the characteristics of the high-dose radiation dosimetry calibration services at the National Institute of Standards and Technology (NIST).

2. Referenced Documents

2.1 *ASTM Standards:*

E 170 Terminology Relating to Radiation Measurements and Dosimetry

E 1249 Practice for Minimizing Dosimetry Errors in Radiation Hardness Testing of Silicon Electronic Devices using Co-60 Sources

E 1250 Method for Application of Ionization Chambers to Assess the Low Energy Gamma Component of Cobalt-60 Irradiators Used in Radiation-Hardness Testing of Silicon Electronic Devices

E 1261 Guide for Selection and Calibration of Dosimetry Systems for Radiation Processing

E 1607 Practice for Use of the Alanine-EPR Dosimetry System

E 1707 Guide for Estimating Uncertainties in Dosimetry for Radiation Processing

2.2 *International Organization for Standards:*

ISO/IEC Guide 25 (1990) General requirements for the competence of calibration and testing laboratories

2.3 *American National Standards Institute:*

ANSI/NCSL Z540-1-1994 Calibration Laboratories and Measuring and Test Equipment - General Requirements

2.4 *National Institute of Standards and Technology:*

NIST Special Publication 250-45 High-Dose Radiation Dosimetry Calibration Services: Manual of Calibration Procedures

3. Terminology

3.1 *Descriptions of Terms Specific to This Document:*

3.1.1 calibration - the process whereby the response of a dosimeter or measuring instrument is characterized through comparison with an appropriate standard that is traceable to, and consistent with, a national standard.

3.1.2 dosimetry system - a system used for determining absorbed dose, consisting of

dosimeters, measurement instruments and their associated reference standards, and procedures for the system's use.

NOTE 1 - The types of dosimeters include reference standard dosimeters, transfer standard dosimeters, and routine dosimeters. See Guide E 1261 for guidance on the selection and calibration of the various dosimetry systems.

3.1.3 measurement assurance program - a documented program for the measurement process that quantifies on a continuing basis the overall uncertainty of the measurements. This program requires traceability to and consistency with national or international standards, and shall ensure that the overall uncertainty meets the requirements of the specific application.

3.1.4 measurement traceability - the ability to demonstrate and document periodically that the measurement results from a particular measurement system are in agreement with comparable measurement results obtained with a national standard (or some identifiable and accepted standard) to a specified uncertainty.

3.1.5 primary standard dosimeter - dosimeter, of the highest metrological quality, established and maintained as an absorbed dose standard by a national or international standards organization.

3.1.6 quality assurance - all systematic actions necessary to provide adequate confidence that a calibration or measurement is performed to a predefined level of quality.

3.1.7 quality control - the operational techniques and procedures that are employed routinely to achieve and sustain a predefined level of quality.

3.1.8 quality manual - document stating the quality policy, quality system, and quality practices of an organization.

3.1.9 quality system - organizational structure, responsibilities, procedures, processes, and resources for implementing quality management.

3.1.10 radiation processing - the intentional irradiation of products or materials to preserve, modify, or improve their characteristics.

3.1.11 reference standard dosimeter - a dosimeter, of high metrological quality, used as a standard to provide measurements traceable to, and consistent with, measurements made using primary standard dosimeters.

3.1.12 routine dosimeter - dosimeter calibrated against a primary-, reference-, or transfer-standard dosimeter and used for routine absorbed dose measurement.

3.1.13 transfer standard dosimeter - a dosimeter, often a reference standard dosimeter, suitable

for transport between different locations, used to compare absorbed dose measurements.

3.1.14 verification - confirmation by examination of objective evidence that specified requirements have been met.

NOTE 2 - In the case of measuring equipment, the result of verification leads to a decision either to restore to service or to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases it is required that a written trace of the verification performed be kept on the instrument's individual record.

3.1.15 working standard - a standard, usually calibrated against a reference standard, used routinely to calibrate or check measuring instruments or devices.

3.2 Also see Terminology E 170.

4. Significance and Use

4.1 The radiation industry needs a source of reliable, prompt, dosimeter calibration services to support accurate measurements of absorbed dose during radiation processing. Those measurements, made routinely in industrial facilities, should be consistent with and traceable to the physical measurement standards maintained by an appropriate national or international standards laboratory.

4.2 To ensure the provision of adequate services, a calibration laboratory should be operating with a full measurement assurance program (MAP). The fundamental requirements for such a program include: (1) compliance with operational requirements of this document; (2) documented procedures and in-house quality assurance (QA) program specific to the calibration services provided; and (3) periodic performance evaluations, such as internal review by NIST staff and measurement intercomparisons between NIST and other national laboratories **(1,2)**¹.

4.3 In addition to standard calibration services such as irradiation of dosimeters and supplying transfer dosimeters for client in-house source calibration, the NIST calibration laboratory MAP can provide technical information, troubleshooting (at the client's facility if necessary), and serve as an independent group for resolution of disputes. NIST can also conduct long term studies of dosimetry system characteristics, such as environmental effects, and evaluate new, emerging dosimetry systems to assist in technology transfer to industry.

4.4 Section 5 sets forth general criteria that shall be satisfied by the NIST calibration laboratory. These general criteria are completely consistent with ISO/IEC Guide 25 and its U.S. equivalent ANSI/NCSL Z540-1. Laboratories that meet these general requirements comply, for calibration activities, with Guide 25/Z540 and the relevant requirements of the ISO 9000 series of standards, including those of the model described in ISO 9002 when they are acting as suppliers producing

¹The bold numbers in parenthesis indicate references at the end of the text.

calibration results.

4.5 For laboratories engaged in specific fields of calibration, the general requirements of ISO/IEC Guide 25/ANSI Z540 need amplification and interpretation. Section 6 of this document contains specific criteria which provide that amplification and interpretation for ionizing radiation. Section 7 contain specific criteria for particular types of ionizing radiation, that is, gamma rays, electron beams and x-ray (bremsstrahlung) beams. It contains the theoretical basis for the traceability of dosimetry measurements used in the calibration services. The detailed procedures used in the handling of customer's calibration requests, for the generation of calibration reports, and in the determination of the uncertainties for the various calibration services are contained in the companion document NIST Special Publication 250-45 *Manual of Calibration Procedures*.

4.6 For ease of use, all sections of this document after Section 5 employ the format established in Section 5. It is therefore readily apparent how the subsequent Sections amplify and interpret the general requirements contained in Section 5.

5. General Criteria

5.1 This section sets forth the general requirements that shall be satisfied by a laboratory. In addition to satisfying the general criteria of this section, a laboratory shall also satisfy the specific criteria contained in Section 6 and in those parts of Section 7 relevant to each calibration service offered (see 4.4 and 4.5).

5.2 This section may also be used by calibration laboratories in the development and implementation of their quality systems, and by others concerned with evaluating the competence of laboratories.

5.3 Organization and Management

5.3.1 The laboratory shall be organized and shall operate in such a way that its facilities meet the requirements of this section.

5.3.2 The laboratory shall:

- a) have managerial staff with the authority and resources needed to discharge their duties;
- b) have arrangements to ensure that its personnel are free from any commercial, financial, and other conflicts which might adversely affect the quality of their work;
- c) be organized in such a way that confidence in its independence of judgement and integrity is maintained at all times;

- d) specify and document the responsibility, authority, and interrelation of all personnel who manage, perform, or verify work affecting the quality of calibrations;
- e) provide adequate supervision by persons familiar with the calibration methods and procedures, the objective of the calibration and the assessment of the results;
- f) have a technical manager (however named) who has overall responsibility for the technical operations;
- g) have a quality manager (however named) who has responsibility for the quality system and its implementation. The quality manager shall have direct access to the technical manager and to the highest level of management at which decisions are made on calibration laboratory policy or resources. In some laboratories, the quality manager may also be the technical manager or deputy technical manager of the calibration laboratory;
- h) nominate deputies in case of absence of the technical or quality manager;
- i) where relevant, have documented policy and procedures to ensure the protection of clients' confidential information and proprietary rights;
- j) where appropriate, participate in interlaboratory comparisons.

5.4 *Quality System and Review of Activities*

5.4.1 The laboratory shall establish and maintain a quality system appropriate to the type, range, and volume of calibration activities it undertakes. The elements of this system shall be documented. The quality documentation shall be available for use by the laboratory personnel. The laboratory shall define and document its policies and objectives for, and its commitment to, good laboratory practice and quality of calibration services. The laboratory management shall ensure that these policies and objectives are documented in a quality manual and communicated to, understood, and implemented by all laboratory personnel concerned. The quality manual shall be maintained current under the responsibility of the quality manager.

5.4.2 The quality manual, and related quality documentation, shall state the laboratory's policies and operational procedures established in order to meet the requirements of this section. The quality manual and related quality documentation shall also contain:

- a) a quality policy statement, including objectives and commitments, by top management;
- b) the organization and management structure of the laboratory, its place in any parent organization and relevant organizational charts;
- c) the relations between management, technical operations, support services, and the quality

system;

- d) procedures for control and maintenance of documentation;
- e) job descriptions of key staff and reference to the job descriptions of other staff;
- f) identification of the laboratory's approved signatories;
- g) the laboratory's procedures for achieving traceability of measurements;
- h) the laboratory's scope of calibrations;
- i) arrangements for ensuring that the laboratory reviews all new work to ensure that it has the appropriate facilities and resources before commencing such work;
- j) reference to the calibration and verification procedures used;
- k) procedures for handling calibration and test items;
- l) reference to the major equipment and reference measurement standards used;
- m) reference to procedures for calibration, verification, and maintenance of equipment;
- n) reference to verification practices including interlaboratory comparisons, use of reference materials, and internal quality control schemes;
- o) procedures to be followed for consultation and corrective action whenever discrepancies in interlaboratory comparisons are detected, or departures from documented policies and procedures occur;
- p) the laboratory management arrangements for exceptionally permitted departures from documented policies and procedures or from standard specifications;
- q) procedures for dealing with complaints;
- r) procedures for protecting confidentiality and proprietary rights;
- s) procedures for review of calibration program and measurements; and
- t) statements of uncertainties of all offered services, including detailed breakdown of all components contributing to each uncertainty.

5.4.3 The laboratory shall arrange for review of its activities at appropriate intervals to verify

that its operations continue to comply with the requirements of the quality system. Such reviews shall be carried out by trained and qualified NIST staff. Where the review findings cast doubt on the correctness or validity of the laboratory's calibration results, the laboratory shall take corrective action and shall notify, in writing, as soon as practically possible, any client whose work may have been affected.

5.4.4 The quality system adopted to satisfy the requirements of this section shall be reviewed at least once a year by the management to ensure its continuing suitability and effectiveness and to introduce any necessary changes or improvements. Any changes in the quality system shall be approved by NIST oversight staff prior to implementation.

5.4.5 All review findings and any corrective actions that arise from them shall be documented. The person responsible for quality shall ensure that these actions are discharged within the agreed time scale.

5.4.6 In addition to periodic reviews, the laboratory shall ensure the quality of results provided to clients by implementing and documenting checks. These checks shall be reviewed by management and shall include, as appropriate, but not be limited to:

- a) internal quality control schemes using, whenever practical, statistical techniques;
- b) participation in interlaboratory comparisons;
- c) replicate calibrations using the same or different methods; and
- d) re-calibration of retained instruments and dosimeters.

5.5 *Personnel*

5.5.1 The laboratory shall have sufficient personnel with the necessary education, training, technical knowledge, and experience to carry out their assigned functions.

5.5.2 The laboratory shall ensure that the training of its personnel is kept up-to-date (see Section 6).

5.5.3 Records on the relevant qualifications, training, skills, and experience of the technical personnel shall be maintained by the laboratory.

5.6 *Facilities and Environment*

5.6.1 Laboratory facilities including calibration areas, electrical power sources, lighting, heating, and ventilation shall be adequate to facilitate proper performance of calibrations.

5.6.2 The environment in which calibrations and related activities are undertaken shall not invalidate the results or compromise the specified uncertainty of measurement.

5.6.3 The laboratory shall provide facilities for the effective monitoring, control, and recording of environmental conditions as appropriate. Due attention shall be paid, for example, to dust, electromagnetic interference, humidity, electrical power stability, temperature, and sound and vibration levels, as appropriate to the calibrations performed.

5.6.4 The laboratory design shall provide adequate protection between areas where the activities are incompatible.

5.6.5 Access to and use of all areas affecting the quality of calibration and related activities shall be defined and controlled.

5.6.6 Adequate procedures shall be taken to ensure good housekeeping.

5.6.7 The laboratory shall comply with all relevant health and safety requirements.

5.7 *Equipment*

5.7.1 The laboratory shall be furnished with all items of equipment required for the correct performance of calibrations. In those cases where the laboratory needs to use equipment outside its permanent control it shall ensure that the relevant requirements of this section are met.

5.7.2 All equipment relevant to calibration processes shall be properly maintained. Maintenance procedures shall be documented. Any item of equipment which has been subjected to overloading or mishandling, or which gives suspect results, or has been shown by verification or during calibration or use to be defective, shall be taken out of service, clearly identified, and wherever possible stored at a specified place until it has been repaired and shown by calibration, verification, or test to perform satisfactorily. The laboratory shall examine the effect of this defect on previous calibrations.

5.7.3 Each item of equipment shall, when appropriate, be labeled, marked, or otherwise identified to indicate its calibration status.

5.7.4 Records shall be maintained for each item of equipment significant to the calibrations performed. The records shall include:

- a) the name of the item of equipment;
- b) the manufacturer's name, type identification, and serial number or other unique identification;
- c) the date received and the date placed in service;
- d) the current location, where appropriate;
- e) the condition when received (for example, new, used, reconditioned);
- f) a copy of the manufacturer's instructions, where available;
- g) the dates and results of calibrations or verifications, or both, and the date of the next calibration or verification or both;
- h) the name and signature of the person who performed the calibrations or verifications, or both;
- I) the details of maintenance carried out to date and planned for the future; and
- j) the history of any damage, malfunction, modification, or repair.

5.8 *Measurement Traceability and Calibration*

5.8.1 All measuring equipment having an effect on the accuracy or validity of calibrations shall be calibrated or verified, or both, before being put into service. The laboratory shall have an established program for the calibration and verification of its measuring equipment (3).

5.8.2 The overall program of calibration or verification, or both, and validation of equipment shall be designed and operated so as to ensure that, wherever applicable, measurements made by the laboratory are traceable to national or international standards of measurement where available. Calibration certificates shall, wherever applicable, indicate the traceability to national standards of measurement, and shall provide the measurement results and associated uncertainty of measurement or a statement of compliance, or both, with an identified metrological specification.

5.8.3 Where traceability to national or international standards of measurement is either not available or not applicable, the laboratory shall provide satisfactory evidence of correlation of results, for example by participation in a suitable program of interlaboratory comparisons.

5.8.4 Reference standards of measurement held by the laboratory shall be used for calibration only and for no other purpose, unless it can be demonstrated that use for other purposes will not

invalidate their performance as reference standards.

5.8.5 Reference standards of measurement shall be traceable to a national or international standard of measurement. There shall be a program of calibration and verification for reference standards.

5.8.6 Where relevant, reference standards and measuring equipment shall be subjected to in-service checks (constancy checks) between calibrations and verifications.

5.9 *Calibration Methods*

5.9.1 The laboratory shall have documented instructions for using and operating all relevant equipment, for handling and preparing dosimeters, and for performing calibrations, where the absence of such instructions could jeopardize the calibrations. All instructions, standards, manuals, and reference data relevant to the work of the laboratory shall be maintained up-to-date and be readily available to the staff.

5.9.2 The laboratory shall use appropriate methods and procedures for all calibrations and related activities within its responsibility (including handling, transport, storage, and preparation of dosimeters, estimation of uncertainty of measurement, and analysis of calibration data (4,5)). The methods and procedures shall be consistent with the accuracy required, and with any standard specifications relevant to the calibrations concerned.

5.9.3 Where methods and procedures are not specified, the laboratory shall, wherever possible, use those that have been published as international or national standards, published by reputable technical organizations, or published in relevant scientific texts or journals.

5.9.4 Where it is necessary to employ methods and procedures that have not been established as standard, these shall be subject to agreement with the client, be fully validated and documented, and be available to the client and other recipients of the relevant reports.

5.9.5 Calculations and data transfers shall be subject to appropriate checks.

5.9.6 Where computers or automated equipment are used to capture, process, manipulate, record, report, store, or retrieve calibration data, the laboratory shall ensure that:

- a) computer software is validated and documented where the software provides operational control or contributes to a quality management decision process;
- b) procedures are established and implemented for protecting the integrity of data; such procedures shall include, but not be limited to, integrity of data entry or capture, data storage, data transmission, and data processing;

- c) computer and automated equipment is maintained to ensure proper functioning and is provided with the environmental and operating conditions necessary to maintain the integrity of calibration data; and
- d) appropriate procedures for the maintenance of security of data including the prevention of unauthorized access to, and the unauthorized amendment of, computer records are established and implemented.

5.9.7 Documented procedures shall exist for the purchase, receipt, and storage of consumable materials used for the technical operations of the laboratory.

5.10. *Handling of Client's Dosimeters*

5.10.1 The laboratory shall have a documented system for uniquely identifying the dosimeters, to ensure that there can be no confusion regarding the identity of such dosimeters at any time.

5.10.2 Upon receipt, the condition of the dosimeters, including any abnormalities or departures from standard condition as prescribed in the relevant calibration method, shall be recorded. Where there is any doubt as to the dosimeter's suitability for calibration, where the dosimeter does not conform to the description provided, or where the calibration required is not fully specified, the laboratory shall consult the client for further instruction before proceeding. The laboratory shall establish whether the dosimeter has received all necessary preparation, or whether the client requires the laboratory to perform or arrange for such preparations.

5.10.3 The laboratory shall have documented procedures and appropriate facilities to avoid deterioration or damage to the dosimeters during storage, handling, preparation, and calibration; any relevant instructions provided with the dosimeter shall be followed. Where dosimeters have to be stored or conditioned under specific environmental conditions, these conditions shall be maintained, monitored, and recorded where necessary. Where dosimeters are to be held secure (for example, for reasons of record, safety, or value, or to enable check calibrations to be performed later), the laboratory shall have storage and security arrangements that protect the condition and integrity of the secured dosimeters.

5.10.4 The laboratory shall have documented procedures for the receipt, retention, or safe disposal of dosimeters, including all provisions necessary to protect the integrity of the laboratory.

5.11 *Records*

5.11.1 The laboratory shall maintain a record system to suit its particular circumstances and comply with any applicable regulations. It shall retain on record all original observations, calculations and derived data, calibration records, and a copy of the calibration certificate or report, for an appropriate period. The records for each calibration shall contain sufficient

information to permit their repetition. The records shall include the identity of personnel involved in calibration.

5.11.2 All records (including those listed in 5.7.4 pertaining to calibration equipment), certificates, and reports shall be safely stored, held secure, and in confidence to the client.

5.12 *Certificates and Reports*

5.12.1 The results of each calibration or series of calibrations carried out by the laboratory shall be reported accurately, clearly, unambiguously, and objectively in accordance with any instructions in the calibration methods or procedures. The results should normally be reported in a calibration certificate or report, and should include all the information necessary for the interpretation of the calibration results and all information required by the method used.

5.12.2 Each certificate or report shall include at least the following information:

- a) a title, for example, "Calibration Certificate", or "Calibration Report";
- b) name and address of the laboratory, and location where the calibration was carried out if different from the address of the laboratory;
- c) unique identification of the certificate or report (such as serial number) and of each page, and the total number of pages;
- d) name and address of client, where appropriate;
- e) description and unambiguous identification of the dosimeters calibrated (supplier, type, and batch number);
- f) characterization and condition of the dosimeters;
- g) date of receipt of dosimeters and date(s) of performance of calibration, where appropriate;
- h) identification of the calibration method used, or unambiguous description of any non-standard method used;
- i) any deviations from, additions to, or exclusions from the calibration method, and any other information relevant to a specific calibration, such as environmental conditions;
- j) measurements, examinations, and derived results, supported by tables, graphs, sketches, and photographs as appropriate, and any failures identified;
- k) a statement of the estimated overall uncertainty of the calibration result (where relevant);

- l) a signature and title, or an equivalent identification of the person(s) accepting responsibility for the content of the certificate or report (however produced), and date of issue;
- m) where relevant, a statement to the effect that the results relate only to the dosimeters calibrated; and
- n) a statement that the certificate or report shall not be reproduced except in full, without the written approval of the laboratory.

5.12.3 Where the certificate or report contains results of calibrations performed by sub-contractors, these results shall be clearly identified.

5.12.4 Particular care and attention shall be paid to the arrangement of the certificate or report, especially with regard to presentation of the calibration data and ease of assimilation by the reader. The format shall be carefully and specifically designed for each type of calibration carried out, but the headings shall be standardized as far as possible.

5.12.5 Material amendments to a calibration certificate or report after issue shall be made only in the form of a further document, or data transfer including the statement "Supplement to Calibration Certificate (or Calibration Report), serial number... (or as otherwise identified)", or equivalent form of wording. Such amendments shall meet all the relevant requirements of 5.12.

5.12.6 The laboratory shall notify clients immediately, orally and in writing, of any event such as the identification of defective measuring equipment that casts doubt on the validity of results given in any calibration certificate or report, or amendment to a report or certificate.

5.12.7 The laboratory shall ensure that, where clients require transmission of calibration results by telephone, telex, facsimile, or other electronic or electromagnetic means, staff will follow documented procedures that ensure the requirements of this Section are met and that confidentiality is preserved.

5.13 *Sub-contracting of Calibration*

5.13.1 Where a laboratory sub-contracts any part of the calibration, this work shall be placed with a laboratory complying with these requirements. The laboratory shall ensure and be able to demonstrate that its sub-contractor is competent to perform the activities in question and complies with the same criteria of competence as the laboratory in respect to the work being subcontracted. The laboratory shall advise the client in writing of its intention to sub-contract any portion of the calibration to another party.

5.13.2 The laboratory shall record and retain details of its investigation of the competence and compliance of its subcontractors and maintain a register of all sub-contracting.

5.14 *Outside Support Services and Supplies*

5.14.1 Where the laboratory procures outside services and supplies other than those referred to in this section, in support of calibrations, the laboratory shall use only those outside support services and supplies that are of adequate quality to sustain confidence in the laboratory's calibrations.

5.14.2 Where no independent assurance of the quality of outside support services or supplies is available, the laboratory shall have procedures to ensure that purchased equipment, materials, and services comply with specified requirements. The laboratory should, wherever possible, ensure that purchased equipment and consumable materials are not used until they have been inspected, calibrated, or otherwise verified as complying with any standard specifications relevant to the calibrations concerned.

5.14.3 The laboratory shall maintain records of all suppliers from whom it obtains support services or supplies required for calibrations.

5.15 *Complaints*

5.15.1 The laboratory shall have documented policy and procedures for the resolution of complaints received from clients or other parties about the laboratory's activities. A record shall be maintained of all complaints and of the actions taken by the laboratory.

5.15.2 When a complaint, or any other circumstance, raises doubt concerning the laboratory's compliance with the laboratory's policies or procedures, or with the requirements of this section or otherwise concerning the quality of the laboratory's calibrations, the laboratory shall ensure that those areas of activity and responsibility involved are promptly reviewed in accordance with 5.4.3.

6. Specific Criteria for Ionizing Radiation

6.1 This section sets specific requirements to which a laboratory shall adhere if it is to perform calibrations using ionizing radiation. This section amplifies and interprets the general requirements set forth in Section 5 (see 4.4 and 4.5).

6.2 This section may also be used as a guide by ionizing radiation calibration laboratories in the development and implementation of their quality systems.

6.3 *Organization and Management*: The relationship among the various technical groups, the quality manager, deputy quality manager, and the calibration services of the Ionizing Radiation Division are shown in Fig. 1. This chart includes all calibration activities within the Division, not just those relating to high-dose level dosimetry. However, only those subjects relating to high-dose calibration activities are specifically addressed in this document.

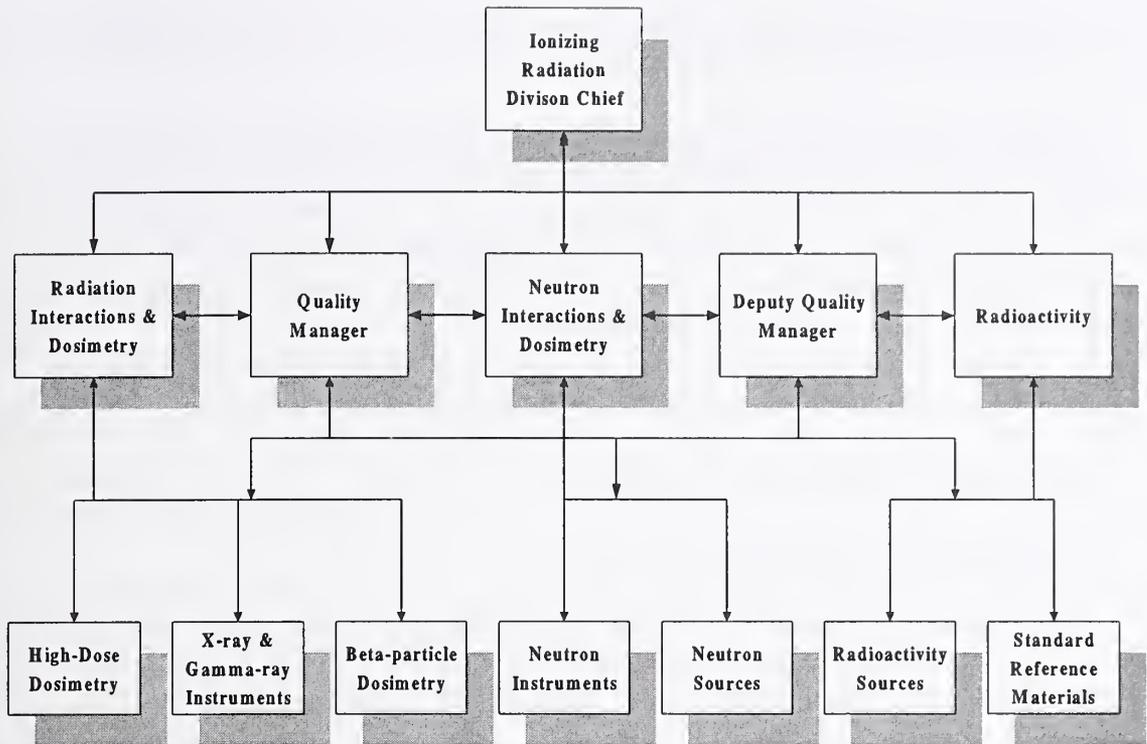


Figure 1 - Organizational Chart for Ionizing Radiation Division Calibration Services

6.4 *Quality System and Review of Activities*

6.4.1 The laboratory shall perform interlaboratory comparisons at least annually for those types of services offered. If the test results indicate that corrective action is required, the laboratory shall take action to achieve the overall uncertainty stated in the appropriate section of this document.

6.4.2 The interlaboratory comparisons shall be performed with a nationally or internationally recognized standards laboratory.

6.4.3 If necessary, the interval between interlaboratory comparisons may be increased. Under no circumstances shall more than 15 months pass between such comparisons.

6.4.4 The quality manual and related quality documentation shall contain:

- a) a statement of the scope of the calibration services to be performed by the laboratory, including the radiation types, energies, and dose rates;
- b) documentation of the model and serial numbers of each critical piece of equipment used in a particular calibration;
- c) a fully documented generic (or representative specific) procedure for each type of calibration performed (for example, passive radiochromic dosimeters calibrated with gamma radiation). The procedure shall provide the appropriate operational steps to permit a knowledgeable person to reproduce a particular calibration technique with a precision consistent with the specified uncertainty of the laboratory. Each calibration procedure shall give the following information where relevant:
- concise but complete account of the procedure;
 - range and limitations of the procedure;
 - equipment and standards to be used;
 - environmental constraints to be met in addition to those in 6.6;
 - sequence of the procedure, drawing attention to special precautions;
 - an example of a completed data sheet; and
 - an example of a calibration report or certificate.
- d) a tabulated assessment of the various components of uncertainty and their associated confidence levels, the method used in combining the components, and the calculated overall uncertainty associated with determination of the reference field for each generic calibration; and
- e) the procedure or reference for auditing calibration data and approving reports.

6.4.5 Each page of the quality manual shall indicate its date of initiation or revision.

6.4.6 A copy of the latest revision of the laboratory's quality manual shall be kept on file and be readily available to the staff.

6.5 *Personnel*

6.5.1 The technical manager (however named) shall understand the quality system and calibration procedures, and ensure that they are being followed.

6.5.2 The technical manager shall have a minimum of a bachelor's degree and should have at least three (3) years of experience in radiological physics or a closely related scientific field.

6.5.3 The supervisor of the calibration laboratory shall have at least three (3) years of experience in radiological physics or a closely related scientific field.

6.5.4 NIST oversight staff may consider an alternative organizational structure utilizing existing expertise, provided a clear line of responsibility exists and the organization clearly provides for continuing control.

6.6 *Facilities and Environment*

6.6.1 Suitable storage facilities shall be provided for reference standards, equipment, documented instructions, manuals, and calibration certificates and reports.

6.6.2 Environmental monitoring equipment shall be provided for recording temperature and relative humidity within the laboratory.

6.6.3 Although strict temperature control is not essential, it is desirable that the laboratory be kept at a reasonably uniform temperature so that the accuracy of equipment is not adversely affected, and so that an adequate stability is achieved before the start of calibration measurements. It is recommended that the laboratory temperature be maintained within the range of 20°C to 24°C.

6.6.4 It is recommended that the relative humidity should be maintained within the range of 15 to 65 percent for laboratory operation unless the calibration of specific dosimeter types require a different range.

6.6.7 A closely controlled environment is not normally necessary in a storage area, but wide temperature and humidity fluctuations should be avoided so as to protect instruments and standards temporarily held there, and to minimize the time required for an instrument to reach equilibrium when brought to the calibration laboratory from the storage area.

6.6.8 The electrical power shall be appropriate to the equipment used, suitably stable, and free of switching surges and significant line noise. When necessary, local auxiliary voltage stabilizers and filters shall be provided. For critical control and data acquisition equipment, battery backup uninterruptable power supplies may be required.

6.6.9 The laboratory shall be provided with an adequate grounding system. Where there is a possibility of interference arising from equipment connected to a single grounding system, separate grounding systems shall be provided and adequate precautions taken against any possible interconnection between systems.

6.6.10 The radiation room shall be of sufficient size and design such that scattered radiation at the positions where dosimeters or instruments are normally placed for calibration does not compromise the specified accuracy goals. If necessary, appropriate scatter corrections shall be applied. In uncollimated free-air calibration facilities, the radiation room should be used exclusively for calibrations to avoid variable scatter conditions. The contribution to absorbed dose by scattered radiation should be known.

6.6.11 If compressed air is used, a pressure regulator and means for removing moisture, dust, and oil from the compressed air shall be provided.

6.7 *Equipment* - The laboratory shall have reference standards and transfer standards that cover the range of calibrations performed.

6.8 *Measurement Traceability and Calibration*

6.8.1 The reference standards used by the laboratory shall be traceable to national or international standards.

6.8.2 The use of a working standard instead of a reference standard is acceptable for calibrations.

6.8.3 The standards or equipment originally calibrated by comparison with a higher-level standard shall be recalibrated when the need is demonstrated by the results of interlaboratory comparisons or routine quality control.

6.9 *Calibration Methods* - All new or amended calibration procedures that could have significant impact on the accuracy of a calibration shall receive approval from the NIST oversight staff before being adopted for routine use.

6.10 *Handling of Dosimeters and Associated Equipment* - All handling, unpacking, and packing of dosimeters, instruments, and reference standards, shall be done by trained staff who are familiar with the equipment.

6.11 *Records*

6.11.1 The laboratory's permanent records shall include:

- a) the date, client, description of the dosimeters calibrated, the batch number or serial number, details of the service provided, calibration report or certificate number, and invoice or other accounting number;
- b) documentation of routine quality control actions and any resultant control charts;

c) the results of all interlaboratory comparisons.

6.12 *Certificates and Reports*

6.12.1 Calibration certificates or reports shall include an appropriate statement clearly specifying the conditions under which the calibrations or measurements were performed. These conditions could include the type of radiation (gamma ray, x ray, or electron beam), the dose rate(s), temperature, and for electron beams, the electron energy, pulse width, dose rate within the pulse, and repetition rate.

6.12.2 Certificates or reports should state that application of the calibration results to an individual measurement is the responsibility of the user, and that care must be exercised in interpolation of the calibration results.

6.12.3 The laboratory shall indicate whether the calibration was performed using either an accredited or non-accredited procedure. The use of non-accredited procedures shall be justified and those procedures completely explained and documented.

6.12.4 If the calibration laboratory discovers a mistake in a calibration report, the person or institution that received the report shall be immediately notified. The mistake shall be corrected as soon as possible, either by sending a corrected report to the client or by recalibrating a new group of dosimeters, as applicable. The laboratory shall determine the reason for the mistake and take action necessary to prevent recurrences.

6.13 *Sub-contracting of Calibration* - There are no additional requirements beyond the general requirements set forth in Section 5.

6.14 *Outside Support Services and Supplies* - There are no additional requirements beyond the general requirements set forth in Section 5.

6.15 *Complaints*

6.15.1 The policy of the Ionizing Radiation Division on complaints is:

All complaints received from customers or other parties on calibration services are important and every effort shall be made to resolve the complaints in a prompt and professional manner. A record shall be maintained of all complaints and of all actions taken by the Division to resolve them.

6.15.2 A customer *complaint* is an expression of dissatisfaction with a requested calibration service. Complaints can be broken, damaged, or missing dosimeters or equipment, excessive delay from the estimated completion date without a reasonable explanation, or missing data in the report. A complaint can be caused by the action (or inaction) of Division personnel or

associated equipment involved with the calibration services.

NOTE 3 - A complaint is **not** that an estimated completion date does not meet the customer's schedule. The Division will make every effort to help a customer, but the Division's service schedule can not be dictated by a customer's needs. Also, complaints are **not** that the calibration fees are too costly, that the type of calibration services offered do not meet the customer's requirements, or that the reported uncertainties are too large.

6.15.3 A *discrepancy* is a customer expression of disagreement or divergence (an inconsistency) in measurement results from a calibration service. This is different than a complaint but should be handled in essentially the same manner, since this could indicate a problem with the calibration service's data handling or quality control.

NOTE 4 - Many times discrepancies are the result of the customer's inappropriate use of the calibrated dosimeters or misunderstanding how to apply the calibration data. Division personnel should determine if the calibration service is operating properly or if the customer is having a problem at their end. The latter situation is often impossible to determine definitively, but a reasonable effort should be made as resources allow (i.e., Division personnel can not do the measurements for the customer unless they are willing to pay for it).

6.15.4 *Complaint Process Procedures*

6.15.4.1 Any Division personnel receiving a complaint about calibration services shall follow the procedures detailed in this section.

6.15.4.2 The complaint (whether received by phone, fax, mail, or in person) shall be entered in a Complaint Log. This Log shall contain the customer's (company) name and address, name of the point-of-contact person, and telephone number, date received, date closed and the Group Leader for that service. The complaints shall be numbered in chronological order.

6.15.4.3 A Complaint Response Form shall be filled out for each complaint showing the person receiving the complaint, date received, calibration service complaint number (from the Complaint Log), the Group Leader for that service, the customer information from the Complaint Log, a description of the complaint or a copy of a written complaint attached to the Response Form, a description of the action taken to resolve the complaint, and the date of closure. Upon closure, the Response Form shall be initialed by the Group Leader, the Technical Manager, and the Quality Manager.

6.15.4.4 Any and all immediate and reasonable action shall be taken to resolve the complaint. Each action shall be recorded and dated in the Response Form.

6.15.4.5 If the complaint is received by a person other than the Group Leader, then the complaint shall be forwarded to the Group Leader who is responsible for resolving the complain.

6.15.4.6 Copies of the Complaint Response Form shall be distributed to Technical Manager,

the Quality Manager, and all Division personnel involved.

6.15.4.7 In most cases the complaint probably can not be resolved by the person receiving the complaint. In those cases the Group Leader shall be responsible for resolving the complaint. If necessary, the Group Leader and all Division personnel involved shall meet to decide on a plan of action to resolve the complaint. That plan and the date shall be noted on the Complaint Response Form and a copy of the plan attached.

6.15.4.8 The Quality Manager or Deputy shall follow up on all complaints to see that they are resolved in a timely manner. When the complaint is resolved, the date shall be entered on the Complaint Response Form and the Group Leader, Technical Manager, and Quality Manager shall initial the Form. The date shall also be entered in the Compliant Log. A copy of the closed (resolved) Complaint Response Form shall be filed with the Complaint Log.

7. Specific Criteria for Calibrations using Photons and Electrons

7.1 This section sets specific requirements to which the laboratory shall adhere if it is to provide calibrations using gamma rays, electron beams, or x-ray (bremsstrahlung) beams. This section amplifies and interprets the general requirements set forth in Section 5, and expands upon the specific requirements contained in Section 6 (see 4.4 and 4.5).

7.2 The criteria contained in this section apply to calibration of dosimeters at absorbed-dose levels appropriate for radiation processing.

7.3 *Organization and Management* - There are no additional requirements beyond those set forth in Sections 5 and 6.

7.4 *Quality System and Review of Activities* - There are no additional requirements beyond those set forth in Sections 5 and 6.

7.5 *Personnel* - There are no additional requirements beyond those set forth in Sections 5 and 6.

7.6 Facilities and Environment

7.6.1 If interpretation of the response of a particular type of dosimeter requires a history of the environmental conditions, the temperature and humidity shall be recorded.

7.6.2 Fluorescence lamps, sunlight, and other sources of ultraviolet light shall be filtered if the dosimeters are adversely affected by ultraviolet radiation.

7.6.3 Any area used for storage of dosimeters shall have its temperature and relative humidity controlled as required for the specific dosimetry system employed.

7.7 *Equipment*

7.7.1 *Radiation Source(s)*

7.7.1.1 The laboratory shall have access to a source of gamma radiation, either ^{60}Co or ^{137}Cs , with a fluence rate sufficient to deliver an absorbed dose within the range of 10 to 10^5 Gy (10^3 to 10^7 rad) within a reasonable time.

7.7.1.2 In addition, the laboratory may have an electron beam or x-ray beam (bremsstrahlung) radiation source, or both, that can provide dose rates appropriate to radiation processing conditions.

7.7.2 *Characterization of the Radiation Field*

7.7.2.1 Determine the absorbed-dose rate in each location in which dosimeters are irradiated using reference standard dosimetry systems. Ensure that dosimeters are irradiated in the locations where the dose rate is determined. At the time a given radiation facility is first calibrated and at intervals not to exceed three years thereafter, demonstrate that the dose rates are traceable to appropriate national standards by direct measurement intercomparisons.

7.7.2.2 Ensure that the absorbed-dose rate over the volume in which dosimeters are irradiated does not vary more than $\pm 1\%$ at a 95% confidence level from its average value.

7.7.2.3 If the dosimeters are irradiated in open air (for example, with a beam-port or panoramic irradiator), ensure that the room is of sufficient size and design such that scattered radiation at each position where dosimeters are placed for irradiation does not compromise the specified overall accuracy goals.

7.7.2.4 Monitor and control the temperature of the irradiation volume during irradiation to the degree required by the characteristics of the dosimeter. Measure this temperature during a simulated irradiation of dosimeters or in a manner that will not perturb the radiation field during the irradiation of dosimeters.

7.7.2.5 Maintain information related to the photon or electron energy spectrum at each dosimeter irradiation location.

7.7.2.6 Minimize low-energy components of the photon source spectrum through the use of a filter box when dosimeters used for radiation hardness testing are irradiated. (For additional information see Practice E 1249 and Method E 1250.)

7.7.3 Descriptions of NIST Irradiation Facilities

7.7.3.1 Vertical Beam ^{60}Co Source - This source consists of a single capsule containing about 7 kilocuries (kCi), housed in a shielding head with a shutter and collimator assembly, as shown in Figure 2. The dosimeters to be irradiated are placed at a measured distance from the source, the shutter assembly is opened, and the gamma rays emerge through the collimator unit into the shielded room. The irradiation time is controlled by an automatic preset timer on the shutter assembly. The absorbed dose rate available from this unit varies as the inverse square of the distance from the source.

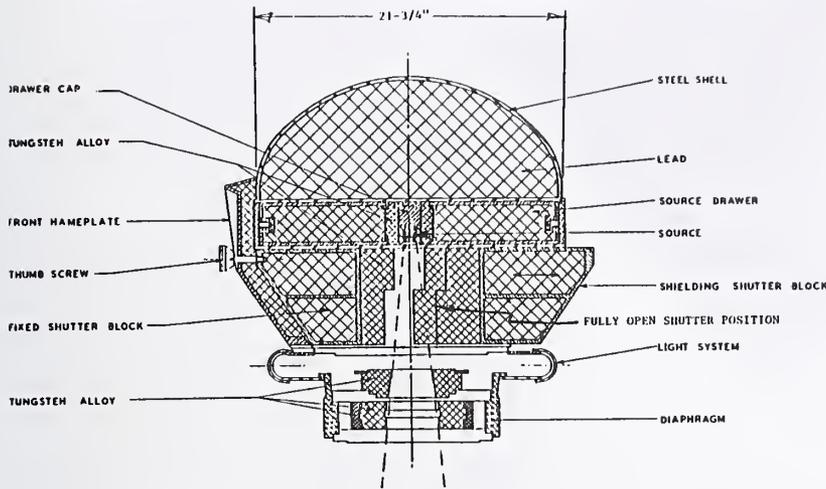


Figure 2 - Vertical Beam ^{60}Co Source

The dosimeters to be irradiated are placed at a measured distance from the source, the shutter assembly is opened, and the gamma rays emerge through the collimator unit into the shielded room. The irradiation time is controlled by an automatic preset timer on the shutter assembly. The absorbed dose rate available from this unit varies as the inverse square of the distance from the source.

At a distance of 146 cm (105 cm scale distance), for December 1995, the absorbed dose rate in water was 0.43 Gy/min. This absorbed dose rate has been determined with an NIST water calorimeter (6). The estimated overall uncertainty in this rate is about $\pm 0.8\%$ at a 95% confidence level. See the companion document SP250-45 *Manual of Calibration Procedures* for detailed discussion of uncertainties in absorbed dose values for dosimeters irradiated with this unit. This source is located in Room B036 of Building 245. There is a similar source with a much lower dose rate located in Room B034; it is generally used only for low dose applications and has not been calibrated with the NIST water calorimeter.

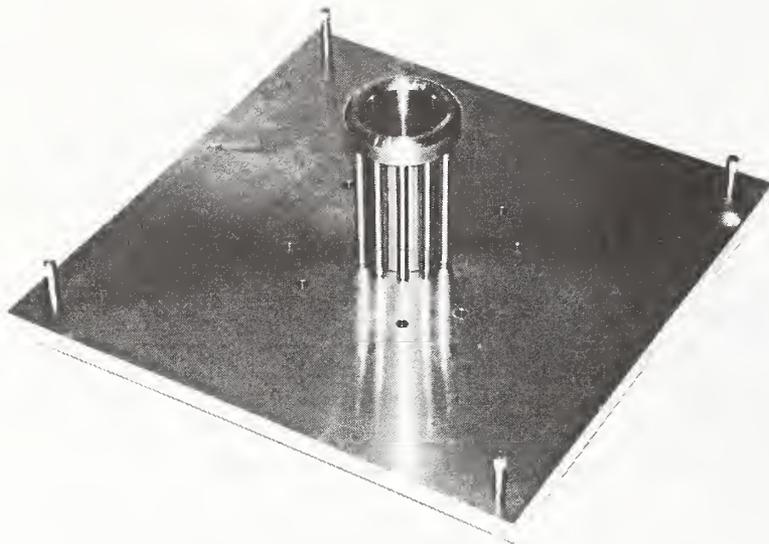


Figure 3 - Pool ^{60}Co Source

7.7.3.2 *Pool ^{60}Co Source* - The pool-type source consists of a stationary annular array of twelve source rods of about 1 kilocurie (kCi) total activity. It is situated at the bottom of a twelve-foot deep tank filled with water for shielding and personnel protection. The source array is shown in Figure 3. Irradiation of dosimeters is done manually by inserting into the source array a watertight stainless-steel can with an inside diameter of 8 cm. The volume of uniform absorbed dose rate is about 4 cm in diameter by 4 cm high, centered at 6.5 cm above the inside bottom of the can on the central axis. The absorbed dose rate of this source has been previously determined by an adiabatic graphite spherical calorimeter and a graphite ionization chamber of identical dimensions to an overall uncertainty estimated to be about $\pm 0.4\%$ at a 99% confidence level (7). Periodic verification checks of the absorbed dose rate is done annually by means of reference transfer dosimeters in collaboration with the National Physical Laboratory (NPL) of the U.K. The absorbed dose rate in water in December 1995 was 22 Gy/min. A complete discussion is given in reference (7) of the sources of the uncertainties in the absorbed dose rate of this ^{60}Co facility. The effect of these uncertainties on absorbed dose values assigned to irradiations performed in this facility is discussed in the SP250-45 *Manual of Calibration Procedures*.

7.7.3.3 *Gammacell 220 ^{60}Co Irradiators* - Two Gammacell 220 irradiators² are employed in the irradiation of dosimeters and other materials.

²The mention of commercial products throughout this document does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the products identified are necessarily the best available for the purpose.

These units, illustrated in Figure 4, are manufactured by Nordion, Intl. of Canada. Each unit contains 24 ^{60}Co source rods in a stationary annular array, one with a total activity of about 18 kCi (serial number GC-232) and the other with about 5 kCi (serial number GC-45). The source rods are located inside a massive lead-steel shielding structure that provides personnel protection.

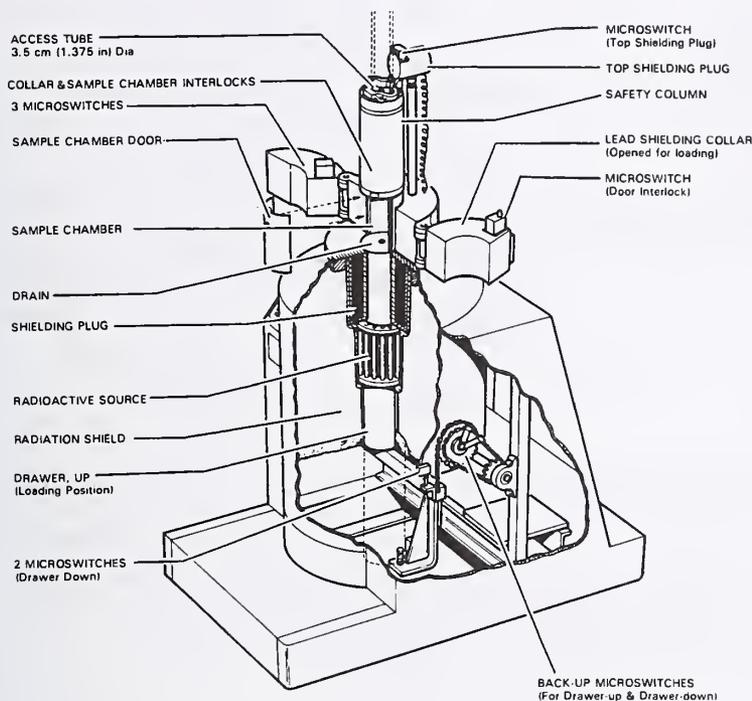


Figure 4 - Gammacell 220 ^{60}Co Irradiator

and 62 Gy/min for GC-45. These rates were determined by means of alanine reference transfer dosimeters referenced to the dose rate of the Vertical Beam source described in 7.7.3.1. These rates, in turn, are verified on an annual basis by means of interlaboratory comparisons using transfer dosimeters from the National Physical Laboratory (NPL) in the U.K. and with NIST transfer dosimeters. The estimated overall uncertainty in this rate is about $\pm 2\%$ at a 95% confidence level. Discussion of uncertainties associated with absorbed dose values for dosimeters irradiated with these units is given in the SP250-45 *Manual of Calibration Procedures*. Significant radiation heating of dosimeters can occur during irradiation in GC-232, so an active cooling system consisting of a flow of chilled dry compressed air is provided to maintain the sample temperature at 23°C.

A motorized "drawer" assembly, consisting of an open irradiation chamber and shielding plugs above and below, can be moved up and down through the cylindrical source array. Dosimeters are placed in the irradiation chamber (with the drawer up), then the drawer is lowered so that the chamber is positioned reproducibly in the geometrical center of the source array. Control of the drawer is by means of a preset timer so that irradiations may be performed automatically. The irradiation chamber has inside dimensions of 15 cm diameter by 20 cm high. The absorbed-dose rate in water in December 1995 was 199 Gy/min for GC-232

7.8 Measurement Traceability and Calibration

7.8.1 The Vertical Beam ^{60}Co source described in 7.7.3.1 is the reference source to which all the other source facilities are compared.

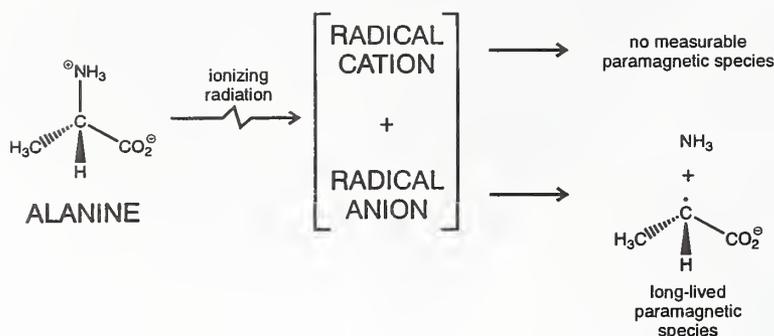


Figure 5 - Production Mechanism of Paramagnetic Species

dosimeter. Upon absorption of ionizing radiation by dry crystalline alanine, deamination of the radical anion (Fig. 5) produces a free-radical center that is exceptionally stable (9). The attributes of the alanine system have been summarized elsewhere (see ASTM Practice E 1607).

7.8.2 The alanine dosimeters used in this work were 90 percent L- α -alanine (Aldrich, 99.9 %) by weight and prepared as described below. The alanine was ground in a centrifugal mill (Brinkmann) fixed with a 0.5 mm ring sieve. The ground crystals of alanine were transferred to a vibrating sieve (Brinkmann) from which a particle size distribution in the range 53 μm to 125 μm was selected. Appropriate weights of sieved alanine and polyethylene (Polysciences, mw = 700, 60 μm) were blended in a powder mixer (Paterson-Kelly). The mixture was loaded into a Manesty hand-tableting press to produce alanine dosimeters 4.9 mm in diameter and approximately 2.7 mm thick. The dosimeters were then placed in a 130 °C oven for 30 min, followed by 5 min in an 85 °C oven. They were then stored in the dark at ambient temperature under controlled humidity conditions (45 \pm 5 % r.h.).

7.8.3 The NIST EPR dosimetry facility consists of a Bruker ESP 300E and an ECS 106 electron paramagnetic resonance spectrometers. The alanine dosimeters were measured in a Bruker TMH microwave resonator. A specially designed clear-fused quartz holder (Wilma Glass) held the dosimeter in the center of the resonator reproducibly. The spectrometer settings were: microwave frequency, 9.8 GHz; center magnetic field strength, 0.3485 T; magnetic field sweep width, 8.0 mT; microwave power, 10 mW; modulation frequency, 100 kHz; modulation amplitude, 1.0 mT; conversion time, 41 ms; sweep-time, 41 s; time constant, 655 ms. The EPR spectrum for an alanine dosimeter irradiated with gamma radiation to 100 Gy is shown in Fig. 6.

7.8.4 The EPR signal intensity of each alanine pellet was measured in two orientations, one at an initial orientation (0°) and then with the pellet rotated 90°. As can be seen from Fig. 6, the signal is the vertical distance between the minimum and maximum of the trace.

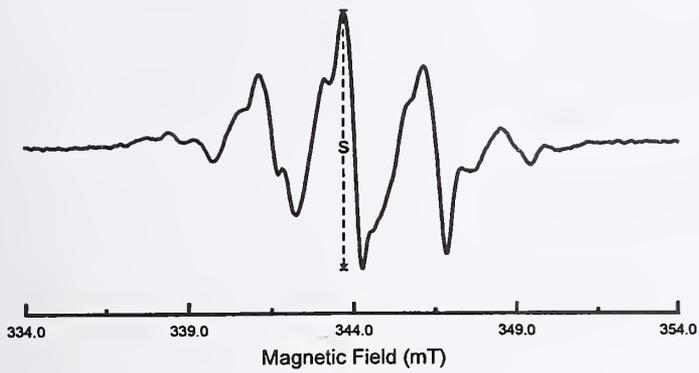


Figure 6 - Electron Paramagnetic Resonance Spectrum of Alanine

A normalized signal, S_N , is obtained by taking the average of the signals at the two orientations and dividing by the mass of the pellet. The mass of each pellet is measured with a Denver Instrument Company model 160 electronic balance to the nearest 0.1 mg before measuring its EPR signal. The masses of the pellets average about 55 mg. The EPR system sensitivity is checked at the

beginning and end of each measurement session by means of a stable paramagnetic species (a pitch sample). The response of alanine has a small temperature dependence during irradiation of about 0.18%/°C. The mass normalized signal, S_N , from above is normalized to a reference irradiation temperature by means of the following equation:

$$S_{NT} = S_N + [S_N (0.0018 (T_{REF} - T_{IRR}))] \quad (1)$$

where S_{NT} = signal normalized for mass and irradiation temperature;
 T_{REF} = reference temperature (usually 23°C); and
 T_{IRR} = irradiation temperature.

7.8.5 Alanine dosimeters were used to measure ratios of the dose rates of each ^{60}Co facility, i.e., the Pool source and the two Gammacells, in comparison to the vertical beam source. This was accomplished by irradiating several alanine pellets in a polystyrene holder to the same absorbed dose in each facility and calculating the ratios of responses.

7.8.6 The dose rate of the reference vertical beam source was measured with an NIST water calorimeter at a source-to-detector distance of 100 cm at a depth of 5 cm in a water phantom (6). To transfer those measurements to alanine dosimeters, it was decided that a polystyrene phantom was more practical than one of water. In order to determine the equivalent distance and thickness in polystyrene that corresponds to those for the water phantom, a *scaling theorem* was employed (10). This theorem has the following assumptions:

- Radiation interacts with the media only by pure Compton scattering;

- Two configurations, each consisting of regions of medium surrounded by vacuum:

System 1, medium has electron density of:

$$\epsilon_1 = \rho_1 \langle Z/A \rangle_1 N_A \quad (2)$$

System 2, medium has electron density of:

$$\epsilon_2 = \rho_2 \langle Z/A \rangle_2 N_A \quad (3)$$

where ρ = medium density;

$\langle Z/A \rangle$ = ratio of the number of electrons in a compound to the molecular weight;
and

N_A = Avogadro constant.

- All distances and dimensions are scaled according to

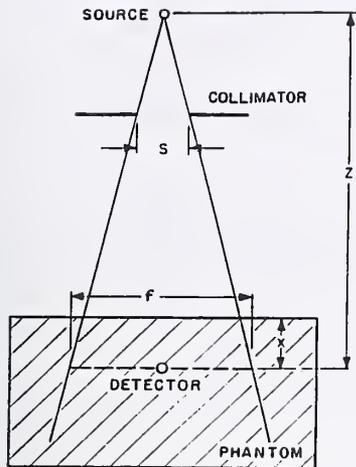
$$x_2 = \frac{\epsilon_1}{\epsilon_2} x_1 \quad (4)$$

where the quantity ϵ_1/ϵ_2 is the scaling factor.

As a result of using the scaling theorem, the fluence at points P_1 and P_2 at appropriately scaled positions has identical energy and angular distributions, differing only by a constant given by the square of the scaling factor, i.e.,

$$\phi_2 = \left(\frac{\epsilon_2}{\epsilon_1} \right)^2 \phi_1 \quad (5)$$

The relationship of the various elements of the vertical beam irradiation geometry is shown in Fig. 7. The calculation of x and z distances are shown in Table 1. With the collimator opening s held constant, the field size f scales automatically.



Monte Carlo calculations were performed to verify the validity of the scaling theorem. The measured incident fluence rate spectra from the ^{60}Co source, averaged over corresponding field sizes, were calculated for the corresponding detector planes, i.e., at depths of 5 cm in water, and 4.87 cm in polystyrene. Both fluence rate spectra were integrated over the mass energy-absorption coefficient for water to obtain the dose rate for a point water detector. For a point source, the square of the scaling factor corresponds exactly to the $1/R^2$ correction factor. Comparisons of the dose rates from the two methods indicate agreement within 0.1%. Independent experimental test measurements (10) indicate agreement with the scaling theorem within 0.5% or better.

Figure 7 - Vertical ^{60}Co Beam Geometry for Polystyrene Phantom

Table 1

Medium	ρ (g/cm ³)	$\langle Z/A \rangle$	ϵ/N_A	x (cm)	z (cm)
water	1.00	0.555087	0.555087	5.00	100.00
polystyrene	1.06	0.537680	0.569941	4.87	97.39

7.8.7 Take the normalized EPR signal, S_{NT} , from 7.8.4, and divide by the irradiation time, t_{IRR} , to get a response proportional to dose rate:

$$R = \frac{S_{NT}}{t_{IRR}} \quad (6)$$

For an irradiation in the Vertical Beam source in a polystyrene phantom, the scaling theorem gives:

$$R_0 = R \frac{(97.4)^2}{(100)^2} \quad (7)$$

For the Gammacells or Pool sources, the alanine is located inside a polystyrene cylinder with a wall thickness of 3.7 mm. It has been experimentally determined that this wall thickness causes an attenuation of 0.980 to the incident gamma radiation, requiring a correction to the dose rate given by:

$$R_0 = \frac{R}{0.980} \quad (8)$$

The ratio of the response, R, for a particular gamma source to another is equal to the ratio of the dose rates for those gamma sources:

$$\frac{R_1}{R_2} = \frac{\dot{D}_1}{\dot{D}_2} \quad (9)$$

For example, the ratio of responses of the Pool source, R_0^{POOL} , to that of the Vertical Beam source, R_0^{VB} , is given by:

$$\frac{R_0^{\text{POOL}}}{R_0^{\text{VB}}} = \frac{\dot{D}_0^{\text{POOL}}}{\dot{D}_0^{\text{VB}}} \quad (10)$$

Thus,

$$\dot{D}_0^{\text{POOL}} = \dot{D}_0^{\text{VB}} \cdot \frac{R_0^{\text{POOL}}}{R_0^{\text{VB}}} \quad (11)$$

Experimentally determined ratios of dose rates for the Pool source and Gammacells, with serial numbers GC-45 and GC-232, relative to the Vertical Beam source are shown in Table 2. The chain of traceability is shown in Fig. 8.

Table 2

$\frac{\dot{D}_{\text{POOL}}}{\dot{D}_{\text{VB}}}$	$\frac{\dot{D}_{\text{GC-45}}}{\dot{D}_{\text{VB}}}$	$\frac{\dot{D}_{\text{GC-232}}}{\dot{D}_{\text{VB}}}$
26.85	75.18	248.6

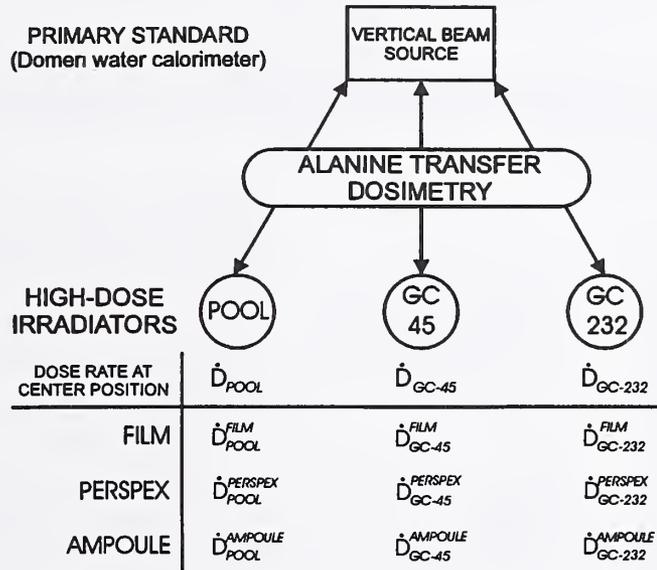


Figure 8 - Traceability Chain for Vertical Beam Source to High-Dose Rate Irradiators

Calculation of the dose rate for a given geometrical configuration, e.g., radiochromic film dosimeters in the Pool source, is given by:

$$\frac{R_{\text{FILM}}^{\text{POOL}}}{R_0^{\text{POOL}}} = \frac{\dot{D}_{\text{FILM}}^{\text{POOL}}}{\dot{D}_0^{\text{POOL}}} \quad (12)$$

and,

$$\dot{D}_{\text{FILM}}^{\text{POOL}} = \dot{D}_0^{\text{POOL}} \cdot \frac{R_{\text{FILM}}^{\text{POOL}}}{R_0^{\text{POOL}}} \quad (13)$$

Now, using equation (11), gives:

$$\dot{D}_{\text{FILM}}^{\text{POOL}} = \dot{D}_0^{\text{VB}} \cdot \frac{R_0^{\text{POOL}}}{R_0^{\text{VB}}} \cdot \frac{R_{\text{FILM}}^{\text{POOL}}}{R_0^{\text{POOL}}} \quad (14)$$

and,

$$\dot{D}_{\text{FILM}}^{\text{POOL}} = \dot{D}_0^{\text{VB}} \cdot \frac{R_{\text{FILM}}^{\text{POOL}}}{R_0^{\text{VB}}} \quad (15)$$

Equivalent equations have been developed for other configurations and dosimeter types; these are discussed in detail in the next section.

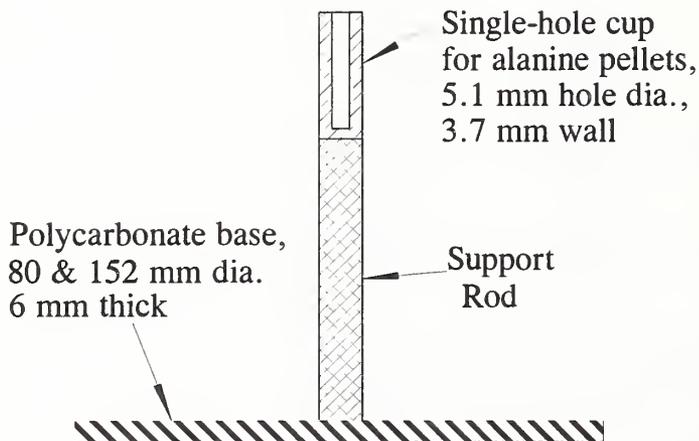


Figure 9 - Single-Hole Geometry for Alanine Pellets in High-Dose Rate Irradiators

7.8.8 The alanine ratio measurements of the various sources described thus far were for a single geometrical arrangement, namely, a polystyrene single-hole cylindrical cup with a hole diameter slightly larger than the alanine pellets (5 mm) and a wall thickness of 3.7 mm. This cup was mounted on a polystyrene rod which was, in turn, mounted to a 6 mm thick polycarbonate base disk to locate the alanine pellets in the geometrical center of the radiation field of each irradiator (see Fig. 9).

7.8.9 The Pool source and Gammacells are used to calibrate a variety of types of dosimeters including liquid solutions sealed in glass ampoules such as ceric cerous sulfate

and dichromate, thin radiochromic solid films, and thick solid slabs such as Red Perspex. Each type of dosimeter requires a unique holder and geometry during irradiation. For the liquid in ampoules type, a polystyrene holder was used as shown in Fig. 10. This holder is referred to as the "five-hole cup" since the five circumferential holes are normally used to hold the dosimeters being calibrated and the center hole is used for quality control dosimetry. The hole size was chosen so that a 2-mL ampoule would fit snugly. To determine the effects of scattering and attenuation of the holder, alanine pellets were placed in polystyrene cylinders that fit snugly in the holes in place of the ampoules. Red Perspex dosimeters are normally sealed in aluminum pouches by the manufacturer and are not opened until after irradiation. The usual method for

handling these during calibration is to fold the pouch tightly around the slab of each dosimeter and place it into the hole of the five-hole cup normally occupied by an ampoule. To determine the effects of this geometry, Red Perspex slabs were removed from their pouches, 5 mm holes were drilled in each in three places, and alanine pellets were inserted in these holes. The slabs were reinserted in the pouches, the pouches are refolded and inserted into the holes of the five-hole cup.

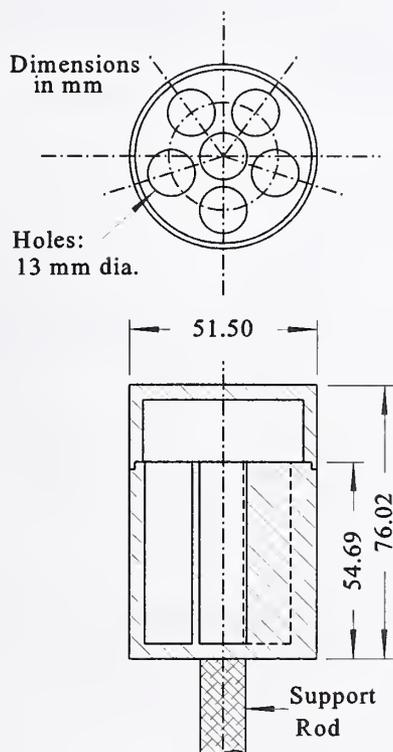


Figure 10 - Five-Hole Cup for Ampoule Irradiation

The radiochromic thin film dosimeters are normally irradiated in polystyrene two-piece square slab holders as shown in Fig. 11. The slab assembly, with several radiochromic films sealed in the interior cavity, is held vertically during irradiation. To simulate this geometry with alanine dosimeters, a 3 mm thick polystyrene slab was drilled through in five places as shown in Fig. 12. After alanine pellets were placed in the five holes, slabs of 3 mm thick polystyrene were sandwiched on each side of the alanine-loaded slab. The effects of the three main irradiation configurations on the dose rates in the three calibration sources were determined by the alanine measurements and compared to the single-hole cup alanine measurements and appropriate correction factors calculated. The dose rates for all sources and configurations are given in Table 3. The conversion of absorbed dose values in water to dose in silicon was done with the use of ratios of mass energy absorption coefficients.

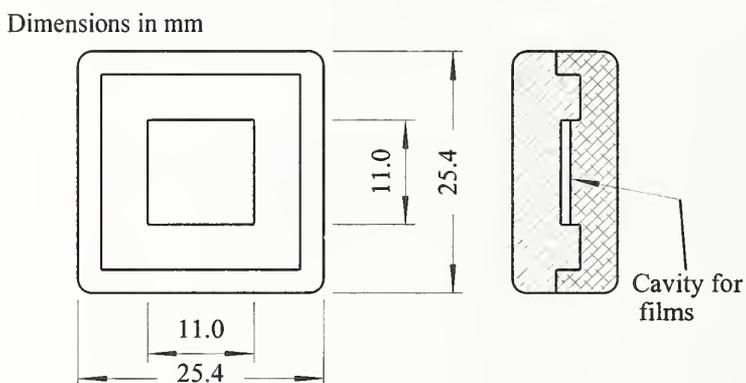


Figure 11 - Holder for Radiochromic Thin Film Irradiation

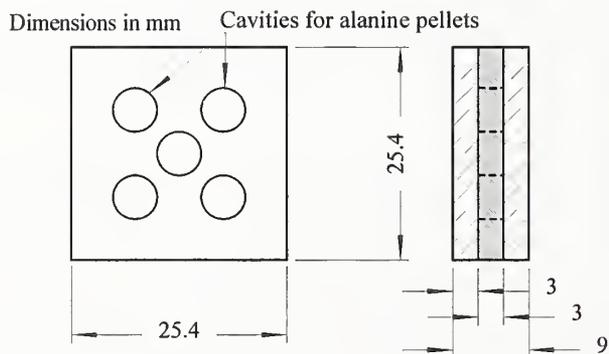


Figure 12 - Holder for Alanine Pellets to Simulate Thin Film Holder Geometry

Table 3
Dose Rates for ⁶⁰Co Sources for December 31, 1996

filename = B140 P5-90 c:\qpw\DR97c.WB1

Effective 3/1/97

Co-60 half-life value is adjusted from 1925.34 days to 1925.12 days

Domen Water Calorimeter Transfer Calibration	
Source in B036 under 5 cm water at 100 cm source	
for 14.5 cm by 14.5 cm field size	
For 1/11/90 Dose rate =	1.8144 Gy/min
For 12/31/96 Elapsed time =	2546 days
Co-60 daily decay constant (ln2 / 1925.12) =	3.60054e-04
Decay correction factor =	0.399837
Dose rate =	0.72546 Gy/min
or	0.012091 Gy/s

Absorbed Dose Rate in Water

GC232	Ratio	Gy/s	Gy/min	kGy/h
3.5 mm Single-hole vial geometry	248.6	3.005	180.3	10.82
5.0 mm Polystyrene Film Block Geometry	0.9870	2.966	178.0	10.68
4.2 mm polystyrene 5-hole cup Perspex Geometry	0.9710	2.918	175.1	10.51
4.2 mm polystyrene 5-hole cup Ampoule Geometry	0.9560	2.873	172.4	10.34
GC232 in Stainless Steel Dewar				
3.5 mm Single-hole vial geometry	239.9	2.901	174.0	10.44
5.0 mm Polystyrene Film Block Geometry	0.9913	2.875	172.5	10.35
4.2 mm polystyrene 5-hole cup Perspex Geometry	0.9692	2.811	168.7	10.12
4.2 mm polystyrene 5-hole cup Ampoule Geometry	0.9599	2.784	167.1	10.02
GC45				
3.5 mm Single-hole vial geometry	75.18	0.9090	54.54	3.272
5.0 mm Polystyrene Film Block Geometry	0.9889	0.8989	53.93	3.236
4.2 mm polystyrene 5-hole cup Perspex Geometry	0.9730	0.8844	53.07	3.184
4.2 mm polystyrene 5-hole cup Ampoule Geometry	0.9595	0.8722	52.33	3.140
F101 Pool Source				
3.5 mm Single-hole vial geometry	26.85	0.3246	19.48	1.169
5.0 mm Polystyrene Film Block Geometry	0.9941	0.3227	19.36	1.162
4.2 mm polystyrene 5-hole cup Perspex Geometry	0.9883	0.3208	19.25	1.155
4.2 mm polystyrene 5-hole cup Ampoule Geometry	0.9749	0.3164	18.99	1.139

for Gammacell and Pool, Combined Relative Expanded Uncertainty = 1.8%; dose rate (Silicon) = dose rate (Water) * 0.913

elapsed time = 366 days, decay correction factor = 0.876534

B036 Co-60 Vertical Beam	Geometry	Gy/s	Gy/min	Gy/h
B036 s.d.=30.0 cm, coll=20 x 20	6 mm ps plate	2.676e-02	1.605e+00	96.32
B036 s.d.=41.6 cm, coll=20 x 20	6 mm ps plate	1.944e-02	1.167e+00	70.00
B036 s.d.=105 cm, coll=20 x 20	6 mm ps plate	6.227e-03	3.736e-01	22.42
B034 Co-60 Vertical Beam				
B034 s.d.=15.0 cm, coll.= 25 x 25	6 mm ps plate	5.306e-03	3.184e-01	19.10
B034 s.d.=100 cm, coll.= 25 x 25	6 mm ps plate	9.476e-04	5.685e-02	3.411
B034 s.d.=130 cm, coll.= 25 x 25	6 mm ps plate	6.568e-04	3.941e-02	2.364

for vertical beams, Combined Relative Expanded Uncertainty = 2.2%; dose rate (Silicon) = dose rate (Water) * 0.896

8. **Keywords** - Absorbed dose; calibration laboratory; dosimeter; dosimetry system; electron beam; gamma ray; ionizing radiation; radiation processing; reference standard dosimeter.

9. References

1. Inn, K.G.W., Coursey, B.M., Eisenhower, E.H., Walker, M.D., Humphreys, J.C., Heaton, H.T., Duvall, K.C., "The role of the Office of Radiation Measurement in quality assurance," *The Science of the Total Environment*, **130/131**, Elsevier Science Publishers B.V., Amsterdam, 1993, pp. 497-507.
2. McLaughlin, W.L., "Reference dosimetry and measurement quality assurance," *Rad. Phys. Chem.* **40**, 1989, pp. 945-951.
3. Humphreys, J.C., Hocken, D., and McLaughlin, W.L., "Dosimetry for High Dose Applications," NBS Special Publication 250-11, Natl. Bur. Stand., Gaithersburg, MD, 1988.
4. McLaughlin, W.L., "Standardization of high-dose measurement of electron and gamma-ray absorbed doses and dose rates," *High-Dose Dosimetry*, Proceedings of International Symposium, Vienna, 1984, IAEA STI/PUB/671, International Atomic Energy Agency, Vienna, 1985, pp. 357-371.
5. McLaughlin, W.L., Boyd, A.W., Chadwick, K.H., McDonald, J.C., and Miller, A., *Dosimetry for Radiation Processing*, Taylor and Francis, London, 1989.
6. Domen, S.R., "A Sealed Water Calorimeter for Measuring Absorbed Dose," *J. Res. Natl. Inst. Stand. Tech.*, **99**, 1994, pp. 121-141.
7. Petree, B. and Lamperti, P., "A Comparison of Absorbed Dose Determinations in Graphite by Cavity Ionization Measurements and by Calorimetry," *J. Res. Natl. Bur. Stand.*, **71C**, 1967, pp. 19-27.
8. Bradshaw, W.W., Cadena, C.G., Crawford, E.W., and Spetzler, H.A.W., "The Use of Alanine as a Solid Dosimeter," *Radiat. Res.* **17**, 1962, pp. 11-21.
9. Sinclair, J.W. and Hanna, W., "Electron Paramagnetic Resonance Study of L-Alanine Irradiated at Low Temperatures," *J. Phys. Chem.*, **71(1)**, 1967, pp. 84-88.
10. Pruitt, J. S. and Loevinger, R., "The photon-fluence scaling theorem for Compton-scattered radiation," *Med. Phys.*, **9(2)**, 1982, pp. 176-179.

THE SP 250 SERIES ON NIST MEASUREMENT SERVICES*

SP 250-1	Spectral Radiance Calibrations PB87179883	SP 250-20	Spectral Irradiance Calibrations PB88123781
SP 250-2	Far Ultraviolet Detector Standards PB87227609	SP 250-21	Calibration of Beta-Particle Radiation Instrumentation PB88201579
SP 250-3	Radiometric Standards in the Vacuum Ultraviolet PB87227625	SP 250-22	Platinum Resistance Thermometer Calibrations PB88138367
SP 250-4	Fricke Dosimetry in High-Energy Electron Beams PB88110374	SP 250-23	Liquid-in-Glass Thermometer Calibration Service PB89128888
SP 250-5	Alpha-Particle Calibrations PB88168620	SP 250-24	Standard Cell Calibrations PB88123690
SP 250-6	Regular Spectral Transmittance PB88108550	SP 250-25	Calibration Service for Inductive Voltage Dividers
SP 250-7	Radiance Temperature Calibrations PB88123674	SP 250-26	NBS Phase Angle Calibration Services PB88225636
SP 250-8	Spectral Reflectance PB88109905	SP 250-27	AC-DC Difference Calibrations PB892222616
SP 250-9	Calibration of Beta-Particle-Emitting Ophthalmic Applicators PB88108535	SP 250-28	Solid-State DC Voltage Standard Calibrations PB88168703
SP 250-10	Radioactivity Calibrations with the "4 π " Gamma Ionization Chamber and Other Radioactivity Calibration Capabilities PB88123708	SP 250-29	Traceable Frequency Calibrations PB88168364
SP 250-11	Dosimetry for High Dose Applications PB88201587	SP 250-30	GOES Satellite Time Code Dissemination: Description and Operation PB88168760
SP 250-12	Neutron Personnel Dosimetry PB87227617	SP 250-31	Mass Calibrations PB89153894
SP 250-13	Activation Foil Irradiation with Californium Fission Sources PB88217443	SP 250-32	A Calibration Service for 30 MHz Attenuation and Phase Shift PB88238324
SP 250-14	Activation Foil Irradiation by Reactor Cavity Fission Sources PB88217435	SP 250-33	A Calibration Service for Voltage Transformers and High-Voltage Capacitors PB882252903
SP 250-15	Photometric Calibrations PB88153747	SP 250-34	High Vacuum Standard and Its Use PB89193841
SP 250-16	Calibration of X-Ray and Gamma-Ray Measuring Instruments PB88211826	SP 250-35	The Calibration of Thermocouples and Thermocouple Materials PB89209340
SP 250-17	The NBS Photodetector Spectral Response Calibration Transfer Program PB88201595	SP 250-36	A Calibration Service for Current Transformers
SP 250-18	Neutron Source Strength Calibrations PB88211818	SP 250-38	NIST Leak Calibration Service
SP 250-19	Calibration of Gamma-Ray-Emitting Brachytherapy Sources PB89193858	SP 250-40	Absorbed-Dose Calibration of Ionization Chambers in a ⁶⁰ Co Gamma-Ray Beam SN003-003-03034-1 \$2.00

* Entries containing a stock number (SN003-003-) and price can be purchased from the Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402. GPO will accept checks, money orders, VISA, and MasterCard. For more information, or to place an order, call (202) 783-3238. Be sure to cite the stock number on all orders.

Entries containing PB numbers can be purchased from the National Technical Information Service, Springfield, VA 22161. NTIS will accept American Express in addition to the payment methods listed for GPO. For more information call (703) 487-4650; to place an order call (800) 336-4700. Be sure to cite the PB number on all orders.

Entries without stock or PB numbers are in preparation.

U.S. Department of Commerce
National Institute of Standards
and Technology
Gaithersburg, MD 20899-0001

Official Business
Penalty for Private Use \$300