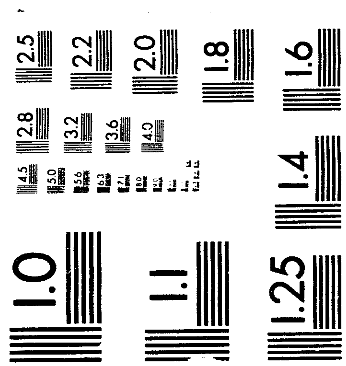
1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0

1 of 1

CONF-9303220--2

PNL-SA-22049

QUALITY ASSURANCE PROGRAMS AT THE PNL CALIBRATIONS LABORATORY

R. K. Piper
J. C. Mcdonald
R. A. Fox
F. N. Eichner

March 1993

Presented at the
Workshop on Measurement Quality
Assurance for Ionizing Radiation
March 16-18, 1993
Gaithersburg, Maryland

Work supported by
the U.S. Department of Energy
under Contract DE-AC06-76RLO 1830

MASTER

Pacific Northwest Laboratory
Richland, Washington 99352

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

DISCLAIMER

QUALITY ASSURANCE PROGRAMS AT THE PNL CALIBRATIONS LABORATORY

R. K. Piper[1]
J. C. McDonald
R. A. Fox
F. N. Eichner

INTRODUCTION

The calibrations laboratory at Pacific Northwest Laboratory (PNL) serves as a radiological standardization facility for personnel and environmental dosimetry and radiological survey instruments. As part of this function, the calibrations laboratory must maintain radiological reference fields with calibrations traceable to the National Institute of Standards and Technology (NIST). This task is accomplished by a combination of 1) sources or reference instruments calibrated at or by NIST, 2) measurement quality assurance (MQA) interactions with NIST, and 3) rigorous internal annual and quarterly calibration verifications. This paper describes a representative sample of the facilities, sources, and actions used to maintain accurate and traceable fields.

LABORATORY GOALS

The goal of the calibrations laboratory is to maintain an array of photon (high and low energy), beta, and neutron reference fields (see Table 1) traceable to NIST. These fields are used primarily to calibrate instruments and dosimeters used at the U.S. Department of Energy's (DOE's) Hanford Site. Traceability to NIST is required for instrument calibration per DOE Order 5480.6. Dosimeter standardization to NIST-traceable radiological reference fields is necessary to ensure accurate analysis and performance in periodic U.S. Department of Energy Laboratory Accreditation Program (DOELAP) proficiency testing for personnel dosimetry programs.

PNL also uses the NIST-traceable fields to support additional functions outside the Hanford Site. The calibrations laboratory provides traceable x-ray and neutron reference fields to support the DOELAP proficiency testing program of the Radiological Engineering and Standards Laboratory (RESL). The calibrations laboratory also serves as the sole proficiency testing laboratory for the external dosimetry National Voluntary Laboratory Accreditation Program (NVLAP). This program accredits external dosimetry programs for facilities licensed by individual states or by the U.S. Nuclear Regulatory

[1] Pacific Northwest Laboratory, Richland, Washington. Work supported by the U.S. Department of Energy under contract DE-AC06-76-RLO 1830.

Commission. PNL provides these radiological reference fields, as well as irradiations using these reference fields, for instrument and/or dosimeter calibrations. In addition, PNL provides characterizations for various nuclear utilities, universities, and dosimeter/instrument vendors and providers.

METHODS AND PRACTICES

To establish accurate radiological reference fields, PNL uses 1) calibration instrumentation or sources that have, themselves, been calibrated at NIST, or 2) instrumentation considered to be of "reference class." Instruments used for calibration are selected for their reliability and reproducibility. Following initial establishment of reference fields, periodic constancy verifications are performed to ensure that no unexpected changes have occurred. Traceability to NIST is reaffirmed, typically on a two-year basis, using MQA interactions. These interactions involve an exchange of instruments or other devices to perform equivalent calibrations of similar reference fields within each facility for comparison. In addition to routine calibrations and intercomparisons, process quality control activities are applied. To monitor the irradiation of dosimeters, PNL uses "quality control chambers," which are air ionization chambers mounted either within phantoms or in air at a nearby position to verify the delivered exposure.

Recognizing a need for more than technical verification, PNL also applies a philosophy of administrative quality assurance. Procedures govern the performance of routine practices, and personnel training is conducted to ensure the safe and effective operation of radiation-generating devices. In addition, technicians undergo apprenticeships in performing irradiation and calibration activities. Audits and assessments are performed periodically by various sponsors, including NVLAP and DOELAP, as well as by PNL's own internal quality assurance organization. Records are maintained in a well-organized format; a records inventory and disposition system (RIDS) is used for long-term retention in a safe environment. Current efforts within the calibrations laboratory are focussed on establishing a well-structured and comprehensive program for performing self-assessments. This program, when initiated, will be used to identify beneficial programmatic needs and improvements, to review proper performance of procedures, and to ensure that records and personnel training are current and complete.

RADIOLOGICAL REFERENCE FIELDS

This section contains examples of how each characteristic radiological reference field is maintained to be accurate and traceable to NIST.

Gamma (^{137}Cs) Reference Field

As its primary ^{137}Cs reference field, the calibrations laboratory maintains a Shepherd Model 81 irradiator equipped with a 100-Ci source. This device is situated near one end of an elongated facility that is 3.7-m wide, 6.4-m long, and 4-m high. Its placement facilitates a collimated field that is uninterrupted for approximately 5 m, producing a beam that is relatively low in scattered photons. A plastic filter, 1-cm thick, is placed at the collimator opening to further reduce low-energy photons and Compton electrons scattered by the collimator.

The Shepherd reference field was initially calibrated using a NIST-traceable Capintec PM-30 air ionization chamber. This chamber has demonstrated excellent stability and reproducibility over time, and its energy dependency is relatively minor, varying only about 5% in the energy range of 35 keV to 1350 keV. In addition, it is manufactured with accurate reproducibility, which is evidenced by the similarity in correction factors among the several PM-30 ionization chambers used at the calibrations laboratory.

Approximately every two years, MQA interactions are performed with NIST. These are typically performed by making calibration measurements of NIST ionization chambers using the PNL sources. Before and after the PNL calibrations, NIST calibrates the chambers using the National Standard ^{137}Cs source. Comparing the results of the NIST and PNL calibrations provides an indication of how accurately the PNL source is calibrated and used. PNL maintains an informal action level of $\pm 2\%$. When agreement is outside this informal action level, investigative measures are initiated to identify anomalies.

The history of these MQA intercomparisons is shown in Figure 1. The various intercomparisons performed since 1984 have used Keithley, Shonka, Exradin, and RADCAL detection systems from NIST, and have involved two different PNL "primary reference" ^{137}Cs gamma sources. It can be seen from this figure that these intercomparisons have verified traceability.

On a quarterly basis, the ^{137}Cs reference field is verified through internal constancy checks, again using the PM-30 ionization chamber. The results of these measurements are compared to the decayed exposure rates to ensure that no unexpected changes have occurred (see Figure 2).

X-Ray Reference Field

The calibrations laboratory uses two Philips Model 324 x-ray machines. One of these systems is configured to reproduce the NIST-referenced, filtered techniques (e.g., M30, S60, and H150), and the other is configured to produce K-fluorescence (characteristic) x-rays. These techniques are used to support algorithm development and testing for onsite dosimetry, in addition to providing all of the x-ray techniques within both the NVLAP and DOELAP proficiency testing programs. Future plans include developing specific International Standards Organization (ISO) techniques that are consistent with those developed by NIST.

The x-ray facility is adjacent to the Shepherd irradiation facility, beneath a former reactor-containment room that is heavily shielded. The facility is 4.7-m wide, 12.6-m long, and 4-m high. The x-ray systems are near one end of the long dimension, allowing an irradiation range of approximately 8 m. The K-fluorescence x-ray beam, which is produced at an angle of 90° to the primary beam, is surrounded by a lead shield to prevent scattered photons from the primary beam from contaminating the useful field. Because of this enclosure, the calibration/irradiation range is limited to approximately 1.5 m.

Each x-ray system is equipped with a transmission chamber. Calibration of each x-ray technique is accomplished by calibrating the transmission chamber for the applicable filter pack. The primary reference standard for filtered x-ray techniques with average energy greater than approximately 35 keV is, again, the NIST-traceable PM-30 ionization chamber. Filtered x-ray techniques with average energy less than 35 keV (with half-value layers ranging from about 0.07 to 3.5 mm of aluminum) are calibrated with a NIST-traceable Victoreen Model 415A air ionization chamber. This chamber, like the PM-30 chamber, has been shown to be extremely stable, with minimal (approximately 5%) variance over the applicable range of x-ray energies.

K-fluorescence techniques are not as directly traceable because NIST does not maintain these techniques. Original reference calibration factors for K-fluorescence were obtained for both the PM-30 and 415A ionization chambers through the National Radiological Protection Board in the United Kingdom. Long-term stability has been assured by constancy verifications using the same instruments that are used to calibrate the beams.

Internal calibrations for commonly used x-ray techniques (e.g., DOELAP and NVLAP techniques) are performed quarterly. The applicable reference chamber is used to establish a new correction factor for the bremsstrahlung and K-fluorescence transmission chambers. The new factors are compared to the old values to screen for any major changes or anomalies involving the system. Agreement of the values is consistently within $\pm 2\%$, and typically within $\pm 1\%$, as shown in Figure 3 for the M30 and M150 techniques.

As with ^{137}Cs and ^{60}Co gamma sources, MQA intercomparisons with NIST are performed for x-rays approximately every two years. Typical interactions involve a subset of several bremsstrahlung (filtered) x-ray techniques, which are performed identically to those for high-energy photons, as described above. Agreement with NIST has been maintained consistently within $\pm 5\%$, as shown in Figure 4 for the M30 and M150 techniques.

Beta Reference Field

Beta sources within the calibrations laboratory are used primarily for dosimetry testing and/or characterization. The isotopic point sources used most frequently include ^{90}Sr/^{90}Y (760-keV average energy) and ^{204}Tl (250-keV average energy), although planar (distributed) sources of these isotopes, depleted uranium, and a ^{147}Pm (75-keV average energy) source are available. Of highest order within the traceability hierarchy is an Amersham-Buechler beta set, including irradiation jig. This system is directly traceable to the Physikalisch Technische Bundesanstalt (PTB), Germany's national physical standards organization. The system includes two ^{90}Sr/^{90}Y sources with activities of 74 and 1850 MBq, an 18.5-MBq ^{204}Tl source, and a 518-MBq ^{147}Pm source. Other sources that will fit within the irradiation jig have been procured from U.S. manufacturers. One, a ^{90}Sr/^{90}Y source, has encapsulation modified from that of the PTB system to meet specifications of ANSI N13.11, *American National Standard for Dosimetry - Personnel Dosimetry Performance - Criteria for Testing*. Another source, ^{204}Tl, has a much higher activity than its PTB counterpart.

As stated above, traceability to the PTB has been supplied for the Amersham-Buechler sources. In turn, these sources have been used to establish a PTW extrapolation chamber as an internal reference standard. The extrapolation chamber is then used to calibrate other non-PTB beta sources within the calibrations laboratory. The two non-PTB sources procured to fit within the PTB jig have also been calibrated by NIST personnel within PNL's calibrations laboratory.

Approximately every two years, traceability to NIST is confirmed through an intercomparison using a NIST-owned transfer standard. In the past, NIST has used a PTW Model 2047, large-volume, thin-window transmission chamber, which is described in detail in NBS Special Publication 250-21, *Calibration of Beta-Particle Radiation Instrumentation and Sources*. The intercomparisons have focused on PTB sources to maintain as much continuity of encapsulation and activity parameters as possible. Agreement between PNL and NIST has typically been maintained within ±5%.

Established absorbed-dose rates from the various sources are decayed to each date of use; however, these values are verified periodically for constancy. Annually, the extrapolation chamber is verified to be in proper working order, using the PTB standards, and then used to reassess each of the routinely used non-PTB sources. On a quarterly basis, thermoluminescent dosimeter (TLD) measurements are performed which assist in verifying constancy and provide a parameter for trending. Figure 5 indicates the traceability and intercomparison pathway for the various commonly used PNL beta sources.

Neutron Reference Field

Neutron capabilities within the calibrations laboratory include two ^{252}Cf spontaneous-fission sources. These sources may be used either in unmoderated conditions, producing a spectrum of approximately 2.2-MeV average energy, or within a D_2O moderating sphere (30 cm in diameter and covered with 0.051 cm of cadmium), producing a softer spectrum of approximately 0.5 MeV. The sources are used near the center of a "low scatter facility" that is 10-m wide, 16.4-m long, and 9-m high. The sources are positioned for irradiation through the use of a pneumatic air-transfer system with minimal transit time from storage to exposure.

Each neutron source was submitted to NIST for initial calibration prior to receipt at PNL. Calibrations consist of determining the neutron emission rate using the Manganous Sulfate Bath Method, from which the bare and moderated free-field-dose-equivalent rates are determined using relationships defined in NBS Special Publication 633, *Calibration Techniques for Neutron Personnel Dosimetry*.

During the past several years, several joint efforts have been made by NIST and PNL to establish a suitable MQA method for neutron reference fields. These efforts have involved various measurement devices and outcomes, as indicated in Table 2. Although nominal agreement is somewhat poorer than is typically observed for beta and photon fields, the results are encouraging. With the experience gained from these prior attempts, a successful method of intercomparison may be forthcoming.

Inhouse constancy verifications have been performed periodically for the neutron sources, using a "SNOOPY" neutron detector. This device is a neutron survey instrument that uses a BF_3 tube to detect thermal neutrons. The detector is surrounded by a cylindrical moderator, as opposed to the spherical moderator used by several other systems. The detector, which has not been used outside the calibrations laboratory, has an established history of accurate performance. Consistent internal agreement within ±2% has been demonstrated against decayed dose-equivalent rates.

QUALITY CONTROL

In addition to the periodical measurements performed to demonstrate the traceability and constancy of radiological reference fields, systems are established to assess the quality of each dosimeter irradiation. These systems are composed of air ionization chambers placed at key locations within the phantoms, or near the source, to monitor delivered quantities. The use of such devices can provide several key benefits:

- ensuring that the exposure or dose equivalent quantity is correct
- verifying phantom positioning when the device is placed inside the phantom
- verifying the selection of the appropriate source or x-ray technique when there is a multitude of possibilities
- verifying the proper use of flattening filters in the case of beta source irradiations
- accessing exposure time if linked to a backup or recording system, when inadvertent source returns or irradiation-system shutdowns occur.

When not used to detect or recover from anomalies, devices for monitoring quality control provide a means to trend the performance of irradiation systems. Although the monitoring measurements are somewhat less accurate than measurements performed for annual or quarterly constancy verifications, the quantity of the monitoring measurements has the potential for detecting long-term, as well as short-term, biases that may not otherwise be readily observable.

Each type of monitored radiological reference field is configured with a chamber of appropriate sensitivity that is capable of measuring at least some component of the field. Photon irradiations are monitored with air ionization chambers similar to those used for annual calibrations and quarterly constancy checks. Chambers are placed in fixed positions either within the phantom (if used) or in another jig near irradiated dosimeters. Beta irradiations are monitored using a small extrapolation chamber with a thin entrance window suitable for beta particle use. This chamber, which is used in a fixed-volume mode, is mounted within the dosimeter irradiation phantom to ensure reproducible positioning. For neutron irradiations, a tissue-equivalent ionization chamber monitors the photon component yielded by ^{252}Cf sources. This particular chamber is mounted adjacent to the source, but away from the dosimeter or phantom positions. In most cases, these quality control monitors are capable of verifying each irradiation to within $\pm 3\%$. Although the precision level decreases slightly for low-activity sources, the monitors are still extremely useful in measuring irradiation quantity and quality.

SUMMARY

MQA interactions with NIST are now, and will continue to be, a vital element in maintaining high-quality radiological reference fields at PNL. The interaction process for photon fields has a well-established history. Although the interaction process for beta source measurements is effective, it is somewhat tedious and easily affected by slight differences in measurement techniques between the facilities. NIST is currently investigating methods to improve this interaction. The area currently in most need of development appears to be neutron measurement interaction. The calibration and periodic reassessment of neutron sources at NIST, using the Magnanous Sulfate Bath Method, is valid; however, this method is extremely time-consuming, it requires the ordeal of radioactive shipments, and it does not evaluate free-field dose equivalents within the field environment. In addition, this method cannot be used to assess the effects of moderator usage, which are of prime importance, particularly if subtle differences exist between NIST and field moderator assemblies, as is the case for the PNL D_2O-filled sphere. The most comprehensive assessment requires the simultaneous application of field measurements that, in theory, assess the source, its placement within the facility, the effects of moderation, and the procedure for use. Efforts to identify a suitable interaction method have been on-going but have left NIST and PNL in a quandary due to differences in outcome.

The use of constancy verifications lends confidence to each traceable radiological reference field and reduces the need for frequent NIST interactions. The use of quality control chambers or other suitable monitoring devices facilitates a high degree of confidence on a daily basis and provides valuable information for long-term trending of each reference field. These devices can also be used to detect potentially interfering radiations from adjoining facilities.

Implementing these practices, in addition to having a comprehensive procedural basis, adequate personnel training, periodic self-assessments, and an open policy for receiving external assessments, provides consistency in calibration techniques and results, and demonstrates a commitment to maintaining a high-quality program.

REFERENCES

American National Standards Institute (ANSI). 1983. *American National Standard for Dosimetry - Personnel Dosimetry Performance - Criteria for Testing*. ANSI N13.11-1983, ANSI, New York.

Eisenhauer, C. M., J. B. Hunt, and R. B. Schwartz. 1985. *Calibration Techniques for Neutron Personnel Dosimetry*. NBS Special Publication 633, National Bureau of Standards, Gaithersburg, Maryland.

Pruitt, J. S., C. G. Soares, and M. Ehrlich. 1988. *Calibration of Beta-Particle Radiation Instrumentation and Sources*. NBS Special Publication 250-21, National Bureau of Standards, Gaithersburg, Maryland.

U.S. Department of Energy (DOE). 1986. "Safety of Department of Energy-owned Nuclear Reactors." DOE Order 5480.6, Washington, D.C.

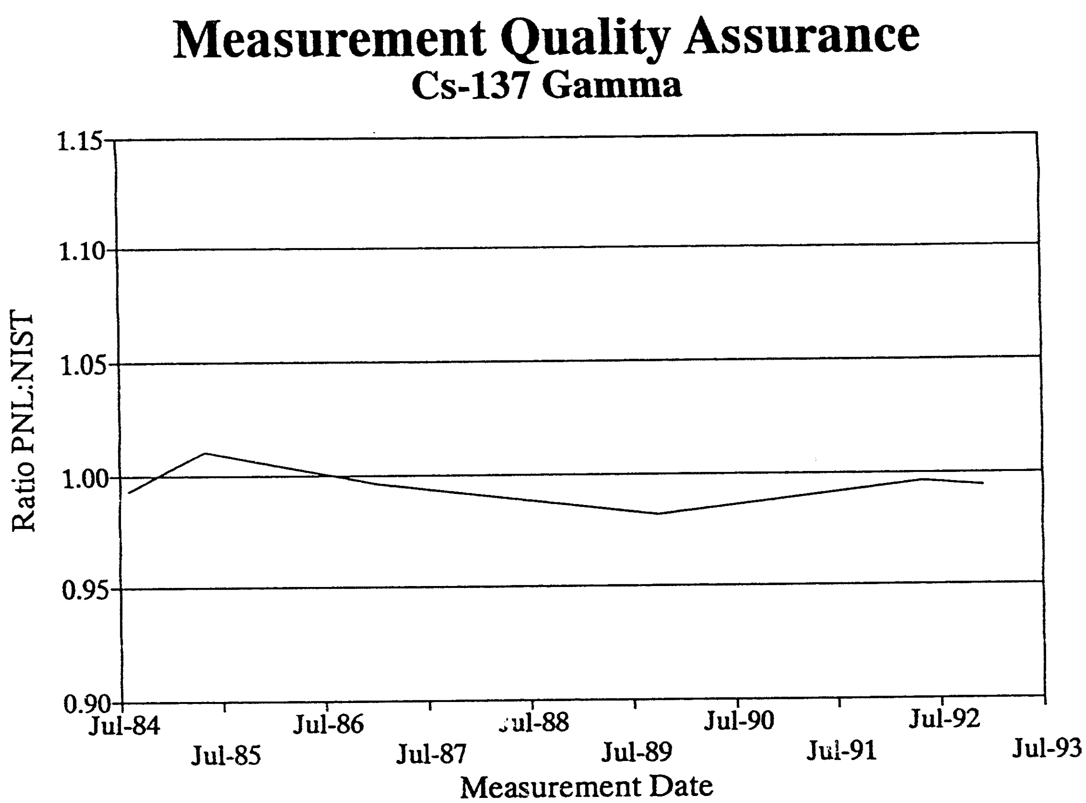

Figure 1 - Comparison of PNL and NIST MQA Results for ^{137}Cs Gamma Sources

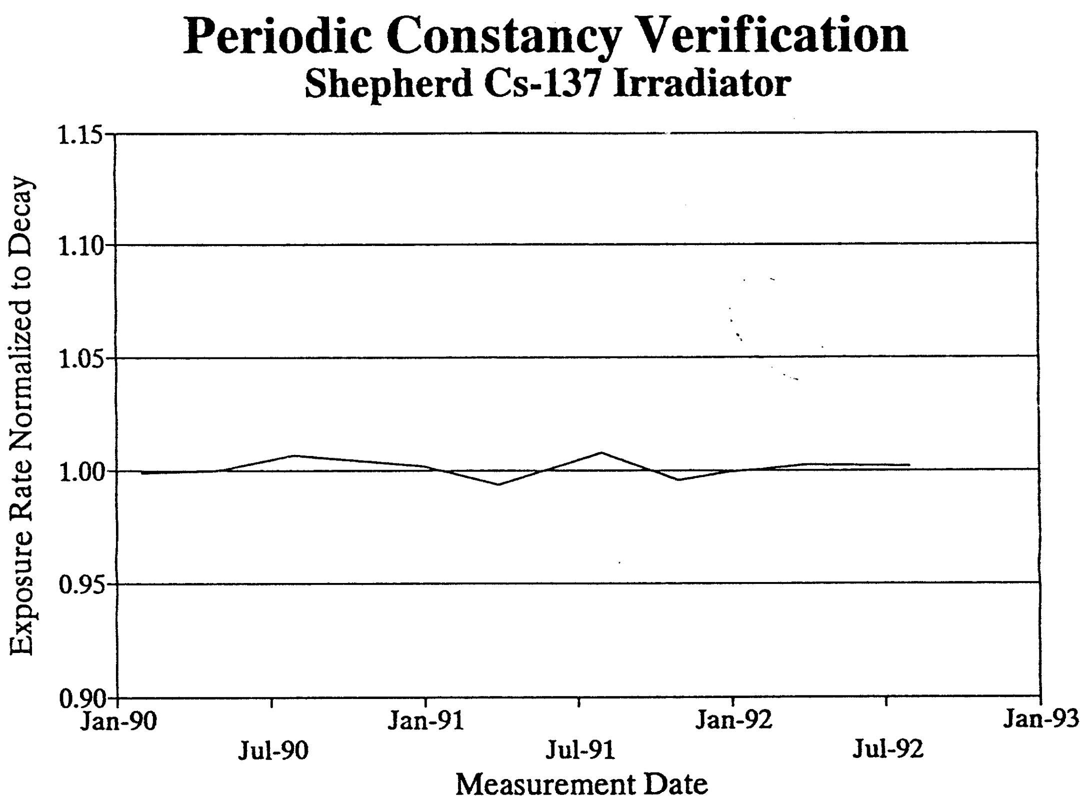

Figure 2 - Constancy Verification Results for Shepherd ^{137}Cs Gamma Irradiator

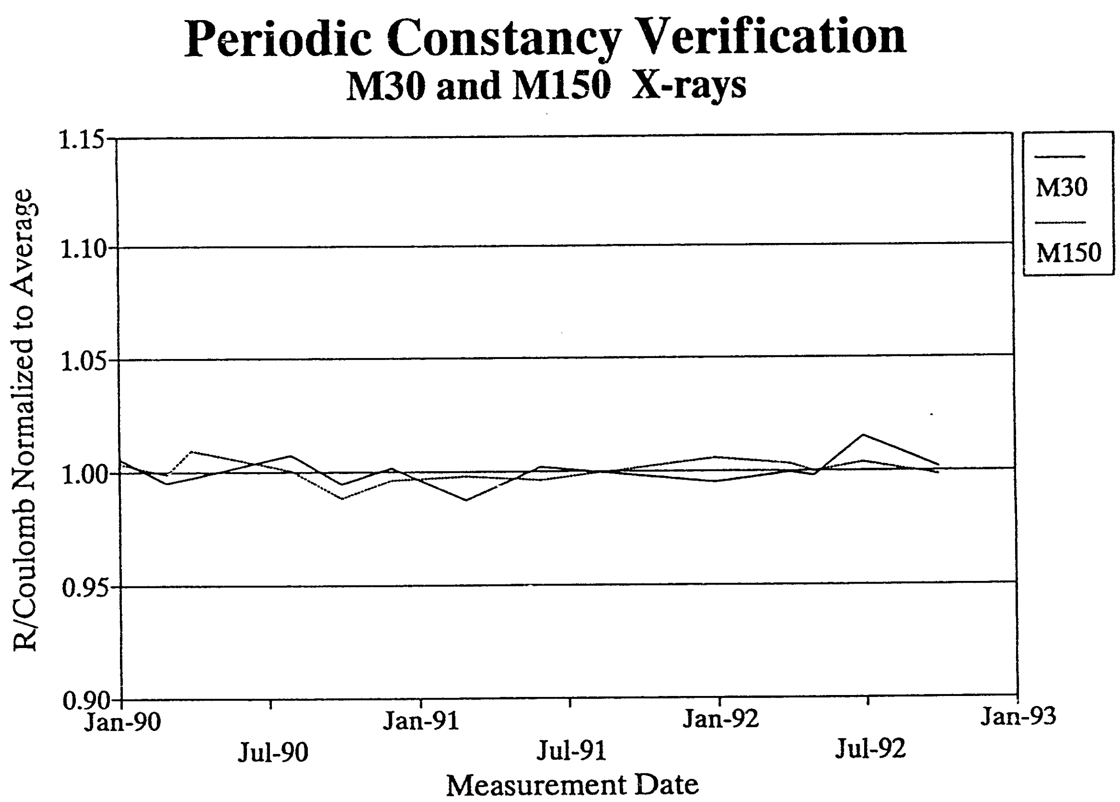

Figure 3 - Verification Results for M30 and M150 Bremsstrahlung X-Ray Techniques

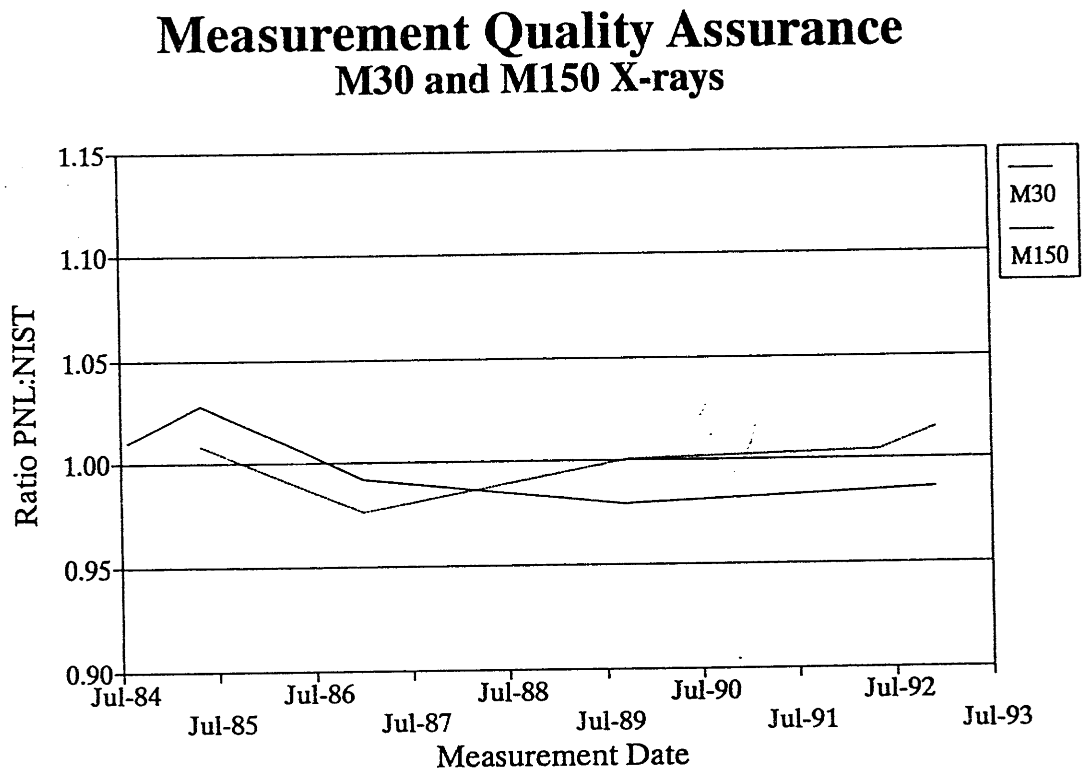

Figure 4 - Comparison of PNL and NIST MQA Results for M30 and M150 Bremsstrahlung X-Ray Techniques

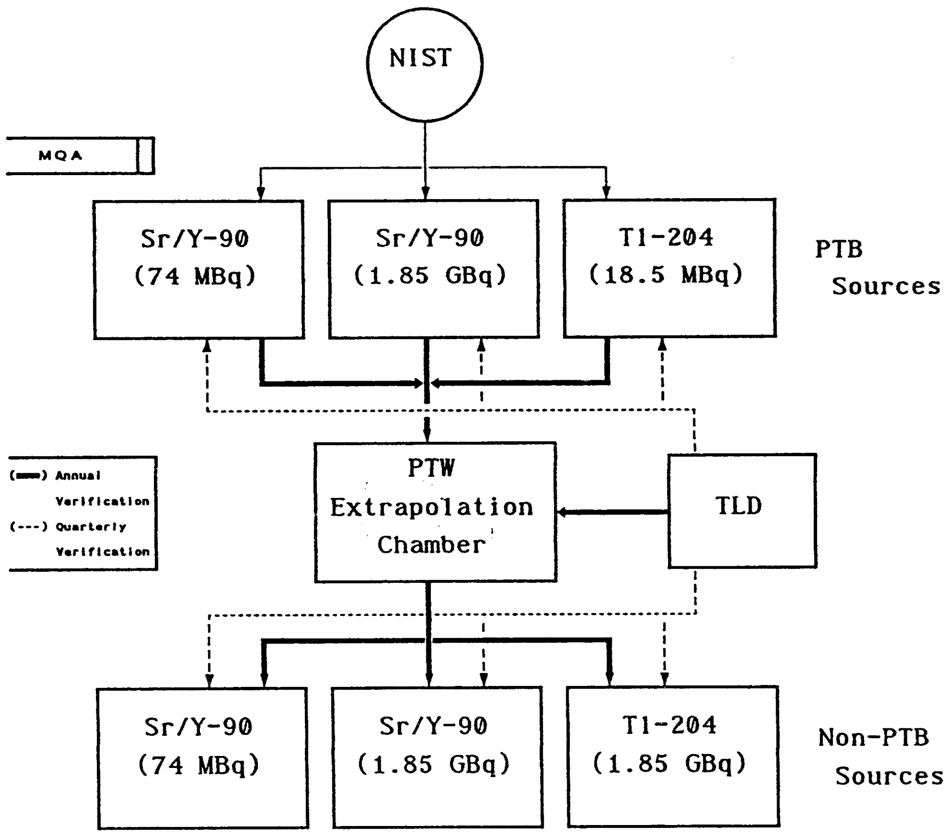

Figure 5 - Pathway for Maintaining and Verifying the Traceability of Beta Radiation Fields

Table 1 - Reference Fields Maintained at the PNL Calibrations Laboratory

Radiation Category	Capabilities Maintained
High-energy photons	^{137}Cs
	^{60}Co
Low-energy photons	Bremsstrahlung (filtered) x-rays
	K-fluorescence x-rays
	^{241}Am
Beta particles	$^{90}Sr/^{90}Y$
	^{204}Tl
	Depleted uranium
Neutrons	D_2O-moderated ^{252}Cf
	Unmoderated ^{252}Cf

Table 2 - PNL Neutron MQA History with NIST

^{252}Cf Neutron MQA		
Method	Ratio of PNL to NIST	
	Moderated	Unmoderated
TLD[(a)]	1.18	0.95
CR-39	1.01	1.03
TEPC[(b)]	1.13	1.09
TEIC/GM[(c)]	1.13	1.01

(a) Thermoluminescent dosimeter. TLD irradiations are performed using the phantom.
(b) Tissue equivalent proportional counter.
(c) Tissue equivalent ion chamber Geiger-Müller detector.

DATE FILMED

1 / 26 / 94

END