MEASUREMENT QUALITY ASSURANCE FOR BETA PARTICLE CALIBRATIONS AT NIST

C. G. Soares[1]
J. S. Pruitt[1]

Abstract - Standardized beta-particle fields have been established in an international standard and have been adopted for use in several U.S. dosimeter and instrument testing standards. Calibration methods and measurement quality assurance procedures employed at the National Institute of Standards and Technology (NIST) for beta-particle calibrations in these reference fields are discussed. The calibration facility including the NIST-automated extrapolation ionization chamber is described, and some sample results of calibrations are shown. Methods for establishing and maintaining traceability to NIST of secondary laboratories are discussed. Currently, there are problems in finding a good method for routine testing of traceability to NIST. Some examples of past testing methods are given and solutions to this problem are proposed.

INTRODUCTION

Because of their limited penetrating power, beta particles exhibit strong gradients of energy and angular distribution as functions of spacial position. This makes it necessary for reference fields to be rigorously defined so as to be reproducible between laboratories. Use of these reference fields by secondary laboratories performing accreditation testing necessitates traceability of beta-particle calibrations to national standards.

Reference beta-particle fields have been defined by the International Organization for Standardization (ISO 1984). The reference fields are divided into two types: Series 1 and Series 2. Series 1 reference fields are designed to be uniform over large areas and are suitable for instrument calibration and irradiation of arrays of passive dosimeters. Three sources are included in this series: $^{90}Sr+^{90}Y$ (E_{max}=2.274 Mev), ^{204}Tl (E_{max}=0.763 MeV), and ^{147}Pm (E_{max}=0.225 MeV). A uniformity of $\pm 5\%$ over a 15-cm diameter is specified for the two higher-energy sources; $\pm 10\%$ over the same area is specified for the low-energy source. To achieve the necessary uniformity, the sources are used with precisely-defined field flattening filters. Typically, absorbed-dose rates from Series 1

(1) National Institute of Standards and Technology, Gaithersburg, Maryland 20899.

sources are less than 10 μGy/s. When higher dose rates are needed, the Series 1 sources, as well as ^{14}C (E_{max}=0.156 MeV) and ^{106}Ru +^{106}Rh (E_{max}=3.54 MeV), may be used without flattening filters. There are requirements on all sources of the maximum energy of the beta-particle spectrum at the calibration distance (E_{res}). This requirement limits encapsulation thickness and distances at which the sources can be used. Specified values for E_{res} are given in Table 1; methods for determining this quantity are given in the ISO Standard 6980 (ISO 1984).

The ISO Series 1 reference fields were implemented at National Institute of Standards and Technology (NIST) in the early 1980's (Pruitt, Soares and Ehrlich 1988). Some of the ISO reference beta-particle fields have as well been adopted in the U.S. for accreditation testing. The ^{90}Sr+^{90}Y field was chosen for personnel dosimeter testing in the American National Standards Institute (ANSI) Standard N13.11, "Personnel Dosimetry Performance - Criteria for Testing" (ANSI 1983). This reference field, as well as the ^{204}Tl field was adopted for personnel dosimetry testing by the U.S. Department of Energy (DOE 1986). In addition to the ISO criteria, DOE added additional criteria concerning ratios of dose rates; these are shown in Table 1. The addition of the ^{204}Tl source as well as the DOE criteria have been proposed for inclusion in the revision of ANSI N13.11 (ANSI 1993).

Performance testing of personnel dosimetry processors with these standards is carried out by secondary testing laboratories. It is important that the irradiations performed in the fields employed by the secondary laboratories be traceable to NIST. In addition, the quality of measurements at NIST must be assured. The steps in this process are shown schematically in Figure 1. The horizontal line represents the NIST internal quality assurance procedures. The procedures for establishing traceability to NIST of secondary calibration laboratories is represented by the slanted line. Finally, the vertical line represents routine measurement quality assurance procedures necessary to maintain secondary laboratory traceability. Each of these is discussed in the following sections.

INTERNAL QUALITY CONTROL AT NIST

Measurement Procedure

A service (Simmons 1991) for the calibration of protection-level beta-particle sources and instrumentation was established at NIST in 1985. Sources accepted for calibration include ISO 6980 Series 1 sources, as well as suitably active small-area sources and large-area plaque sources including, but not restricted to, ISO 6980 Series 2 sources. Ionization chambers accepted for calibration are thin-windowed fixed-gap parallel-plate ionization chambers and thin-windowed extrapolation chambers. Beta-particle sources are calibrated in terms of absorbed-dose rate to tissue at either the surface, the chamber window-thickness depth (2.6 mg/cm^2), or 7 mg/cm^2. Absorbed-dose rate to tissue is determined from current measurements with the NIST-automated extrapolation chamber at a range of air gaps. The absorbed-dose rate to tissue in Gy/s is given by

$$\dot{D}(z)=\frac{(\bar{W}/e)S_{air}^{tissue}}{\rho_0 A}\left(\frac{\Delta I_c}{\Delta d}\right)$$

where $\dot{D}(z)$ is the absorbed-dose rate to tissue at depth z, $(\bar{W}/e)$ is the average energy required to produce an ion pair in dry air (33.97 J/C), S_{air}^{tissue} is the ratio of the average mass stopping power of tissue to air, ρ_0 is the density of dry air at the reference conditions of 22°C and 760 Torr (1.197 kg/m^3), A is the area of the collecting electrode (7.083 x 10^{-4} m^3), and $(\Delta I_c/\Delta d)$ is the fitted slope of the corrected current versus air gap function in A/m. For all sources calibrated, a stopping power

ratio of 1.12 is employed (ISO 1993). The effect of direct beam charge collection is removed by averaging measurements made at both positive and negative polarities. A constant potential gradient of 10 V/mm is employed for all air gaps to avoid deformation of the thin entrance window by attraction to the collecting electrode. For sources measured off contact, a range of air gaps from 0.5 to 2.5 mm will yield a linear function of corrected current versus air gap. For contact measurements, smaller air gaps are necessary to yield a linear function. For depleted uranium slab sources, for example, a range of 0.3 to 1.1 mm has been used successfully.

At NIST, signal current is measured by a negative-feedback amplifier with a capacitive feedback element (Lamperti, Loftus, and Loevinger 1988). This is accomplished by voltage measurements with an electrometer in the feedback mode for known periods of time. The corrected signal at each polarity, $I_c^{+,-}$, is given by

$$I_c^{+,-}=[(\frac{V^{+,-}C}{t})\Pi_c\Pi_k]-I_B^{+,-}$$

where $V^{+,-}$ is the voltage measured at each polarity, C is the external feedback capacitance (101.19 pF), t is the integration time, Π_c is the product of various chamber signal correction factors, Π_k is the product of various source output correction factors, and $I_B^{+,-}$ is the current measured in the absence of a source (background or leakage signal). For the NIST extrapolation chamber, the last term is less than ± 2 fA, so it can be neglected for signal currents above about 1 pA. The correction factors employed at NIST are described in Table 2, along with the sources that they should be employed with. Details on their determination and calculation are given elsewhere (Pruitt, Soares, and Ehrlich 1988).

When absorbed-dose rate at depths other than the extrapolation chamber window-thickness depth are desired, a transmission factor must be employed to account for the difference in depth. The transmission factor, $T(z)$, is defined as the ratio of the absorbed dose rate at depth z that at the surface. Values for some transmission factors are given in the latest revision of ISO 6980 (ISO 1993).

Thin-windowed, fixed-gap parallel plate ionization chambers are calibrated in terms of absorbed-dose rate to tissue at 7 mg/cm^2 per unit of corrected ionization current. Measurements are made in well-characterized NIST beams of known absorbed-dose rate. Ionization currents are corrected to reference temperature and pressure only.

Thin-windowed extrapolation chambers are also accepted for calibration. A calibration of an extrapolation chamber alone can really only tell two things. The first is the accuracy of the air gap setting as indicated on the micrometer barrel. The correction is a constant independent of air gap and is determined from the x-intercept of the fitted corrected current versus nominal air gap function; it is normally in the range of less than ± 70 μm. The offset value determined in this way is slightly dependent upon the beta-particle source used. The second thing that can be learned from a calibration is the area of the collecting electrode, which is inversely proportional to the absorbed-dose rate as determined from the slope of the fitted corrected current verses nominal air gap function. Thus, if the current induced in the air gap volume is measured carefully and accurately, and the proper corrections are applied to the measured currents, absorbed-dose rate can be accurately inferred.

The level of precision in an extrapolation chamber measurement is a function of source strength and the background or leakage signal in the extrapolation chamber. Typical values for leakage currents are $< \pm 2$ fA for high-quality instruments. Thus, with care, signals of a few fA can be measured with satisfactory precision (standard deviation less than 5%) at this leakage level. A signal level of 2 fA/mm corresponds to an absorbed-dose rate of about 0.09 μGy/s (33 mrad/hr). To achieve adequate precision at these levels requires fairly long signal integration times, interleaved background signal measurements for background subtraction, and a large number of replicates. In this laboratory, 3 min runs are used, with 30 measurements of both background and source-induced currents per polarity and air gap. With the NIST-automated extrapolation chamber, this procedure takes about 30 hours for 5 air gaps.

Results of some recent source and extrapolation chamber calibrations are shown in Figures 2 to 4. In Figure 2, corrected current versus nominal air gap curves are shown for the NIST extrapolation chamber and a customer extrapolation chamber. The source used is the NIST Series 2 ^{90}Sr+^{90}Y source measured at a 50-cm distance from the source to the chamber entrance window (SSD). The figure shows that the measured slopes, 453.1 $\pm$0.2 fA/mm for the NIST chamber and 452.6 $\pm$0.3 fA/mm for the customer chamber, are very similar, but that the intercepts are somewhat different, being -61.4 $\pm$0.7 μm for the NIST chamber and +13.5 $\pm$1.0 μm for the customer chamber. The uncertainties shown represent one standard deviation. To remove the effect of the different intercepts, the same data are plotted in Figure 3, but with the air gaps corrected for the x-axis intercept position. Also, to bring out more detail, current per unit corrected air gap rather than current is plotted on the y-axis; in this representation, the fitted slope is a constant. The error bars (too small to be seen in Figure 2) are $\pm$1 standard deviation of the 30 replicates performed at each air gap using a 20-s integration time. The difference between the two slopes is about 0.1%. In Figure 4, similar results are shown of measurements with a customer Series 1 ^{204}Tl source. Measurements with three extrapolation chambers are shown, again with standard deviations from 30 replicates, but this time with integration times of 3 min each due to the low signal levels. Agreement among the three measurements is about 0.5%.

Quality Assurance

Routine measurement quality is assured by repeated measurements of the NIST Series 1 sources. Each time a customer source is calibrated, at least one NIST source is calibrated as well, as a check on the measurement system performance. A similar procedure is followed when a customer ionization chamber is calibrated. The measurement system is considered in control if the NIST source calibration is within 1% of the average of previous measurements for ^{90}Sr+^{90}Y and ^{204}Tl or within 3% for ^{147}Pm and contact sources.

When the NIST facility was being developed, Series 1 sources calibrated by the Physikalisch-Technische Bundesanstalt (PTB) were obtained. The NIST calibration of these sources is within 2% of the PTB values for ^{90}Sr+^{90}Y and ^{204}Tl and within 5% for ^{147}Pm. This comparison established traceability of the NIST measurements to another national standards laboratory. In setting up the measurement system, accuracy of measurement was ascertained for 1) voltage measurement by the electrometer, 2) temperature, 3) pressure, 4) relative humidity, and 5) time. In addition, the value of the feedback capacitor was determined. Periodically, the accuracy of these parameters is reascertained.

A major factor in continued high-quality measurement has been the complete automation of the NIST extrapolation chamber current measuring system, completed in 1991 (Pruitt 1991). Under computer control is the reading of the temperature, pressure and relative humidity, reading of the electrometer voltage, grounding and ungrounding of the electrometer, opening and closing of the shutter, setting of the extrapolation chamber high voltage, and setting of the air gap. The control program also contains all the information necessary to calculate the corrections needed for the extrapolation chamber signal currents and the source output. With the controls and readout fully automated, it is possible to set up lengthy integration times with many replicates; this makes it feasible to measure very low signal levels with reasonable accuracy. Automation minimizes mistakes in reading and instrument settings that are possible with normal human control.

ESTABLISHING TRACEABILITY TO NIST

When a secondary calibration laboratory is accredited it is necessary that traceability of the laboratory's calibrations to national standards be established. One mechanism for this establishment is for the laboratory's sources and extrapolation chamber to be calibrated by NIST. For a laboratory located at relatively high altitude, where low air density effects low energy source (^{147}Pm) output, the best solution is for the source calibration to be performed at the laboratory site. For higher energy sources, calibrations at NIST are preferred, since direct comparisons with national standards are possible. The secondary laboratory's extrapolation chamber also needs to be calibrated at NIST, subject to the caveats discussed above. These calibrations need to be coupled with accuracy checks of the secondary laboratory's temperature, pressure, humidity and current measurement systems. It is also desirable for there to be an on-site laboratory assessment by NIST personnel as part of the accreditation procedure. These steps allow the establishment of traceability to NIST of beta-particle calibrations and irradiations performed by the secondary laboratory.

MAINTAINING TRACEABILITY TO NIST

Secondary laboratory measurement quality is maintained by good quality control procedures. In addition, periodic external checks are necessary. These usually take the form of irradiation or calibration of mailed test devices for comparison to values obtained at NIST. The most desirable form of such a test is the comparison of calibrations of an unknown source. Calibration of an unknown source tests the entire measurement system, and for this reason, passing such a test is considered to best demonstration of traceability. Criteria for passing such a test is a difference of less than 5% (Eisenhower 1991) from the source output specified by NIST. To date, there have been no such tests carried out; however, such tests are planned for the future.

A second, less desirable form of test is comparison of calibrations of a suitable transfer instrument. For this purpose, a thin-windowed parallel-plate ionization chamber was procured and calibrated by NIST. Several mailings of this device have occurred since 1985; the results have proven that this device is not suitable for this purpose. The main problems with the instrument have been a lack of reproducibility of the calibrations at NIST with the low-level Series 1 sources, mainly due to very high and unstable leakage currents which are not independent of polarity. For this reason, this device will no longer be employed for measurement assurance studies.

A third form of measurement assurance is irradiation of NIST-supplied calibrated passive devices by the secondary laboratory with return to NIST for evaluation. Such tests have proven useful in the past for photons, but up to now they have not been employed by NIST for beta particles. A

promising development has been the electret ionization chamber (Kotrappa, Soares, and Hobbs 1993) which is capable of measuring absorbed dose to beta particles at the 0.3 mSv level with uncertainties of less than 5% at the 95% confidence level. Problems with transit doses can be avoided since the device can be rendered inactive during shipment by removing the collecting volume from the electret. Readout is very simple and non-destructive; readout devices are sufficiently inexpensive so that one could be included with the dosimeters and used by the secondary laboratory to read devices before and after irradiation. Blindness would be maintained since the calibration factors for the particular devices employed would be known only by NIST. Such a system has been obtained by NIST and will be tested in the coming year as a possible alternative to shipment of active devices.

CONCLUSIONS

Internal measurement quality assurance at NIST is well established for beta-particle dosimetry calibrations through quality control procedures followed for every customer calibration. Traceability to another national standards laboratory (PTB) has been established. Procedures for establishing traceability to NIST for secondary laboratories doing beta-particle source and instrument calibrations and dosimeter irradiations, are well understood and are being followed. Where challenges presently lie are in procedures for maintaining traceability to NIST through routine measurement quality assurance tests. Several alternatives were discussed and possible solutions outlined. The challenge for the future will be the successful implementation of a good measurement quality assurance test with the required accuracy and sensitivity for establishing traceability of secondary laboratories to NIST at the necessary level of 5% for beta particle fields.

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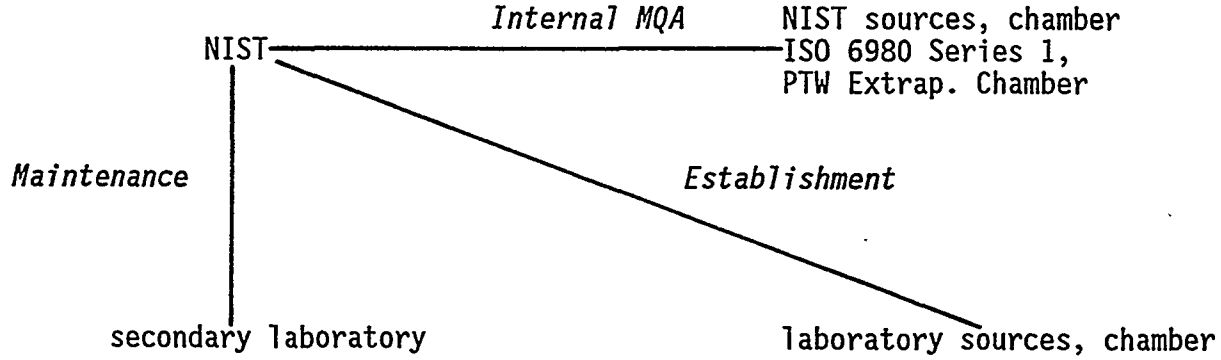

Figure 1 - Schematic Diagram of Measurement Quality Assurance Procedures for Beta-Particle Calibrations

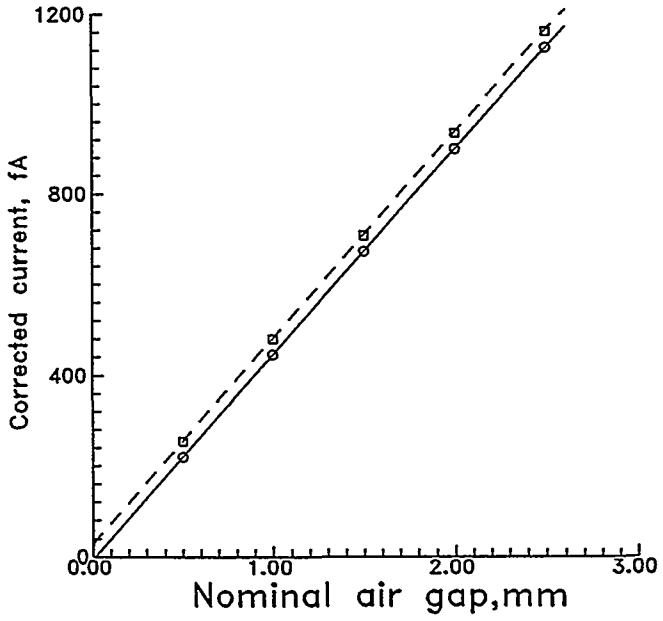

Figure 2 - Sample Extrapolation Chamber Calibration Data. Squares: NIST Chamber. Circles: Customer Chamber. Lines: Linear Least-Squares Fits to the Data.

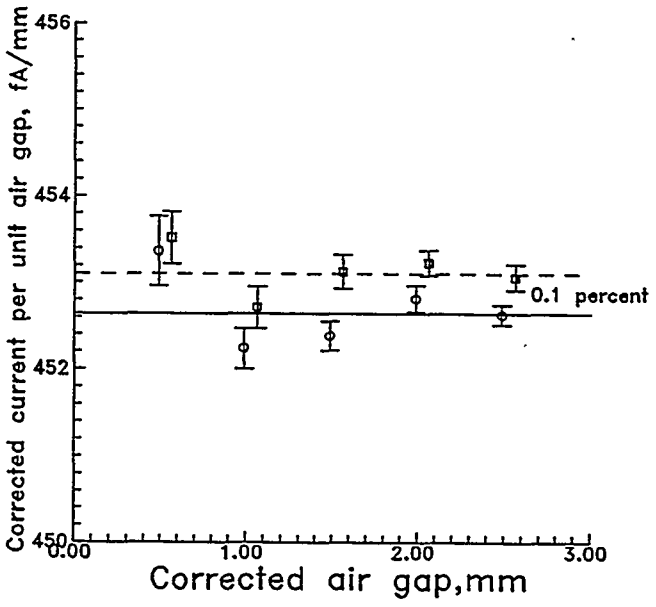

Figure 3 - The Same Data as in Figure 2, but Corrected for X-Intercepts and Expressed as Current Per Unit Corrected Air Gap

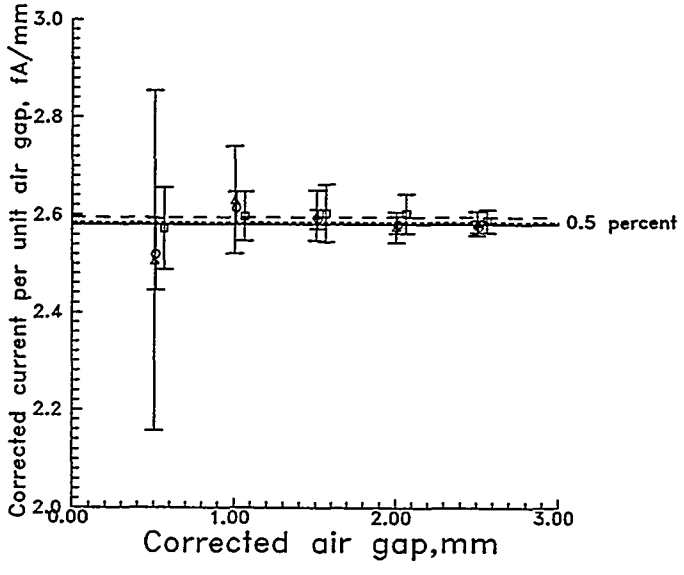

Figure 4 - Calibrations of a Customer ^{204}Tl Source With Three Different Extrapolation Chambers. Squares: NIST Chamber. Circles and Triangles: Customer Chambers

Table 1 - Beta-Particle Source Specifications

Source	ISO 6980	DOELAP (DOE/EH-0027)
^{14}C	$E_{res} > 0.09$ MeV	not used
^{147}Pm	$E_{res} > 0.13$ MeV $\pm 10\%$ uniform over 15 cm (Series 1 only)	not used
^{204}Tl	$E_{res} > 0.53$ MeV $\pm 5\%$ uniform over 15 cm (Series 1 only)	$\frac{D^a(100\ mg/cm^2)}{0.80 \pm 0.05\ D(7\ mg/cm^2)} =$
$^{90}Sr+^{90}Y$	$E_{res} > 1.80$ MeV $\pm 5\%$ uniform over 15 cm (Series 1 only)	$\frac{D(100\ mg/cm^2)}{1.01 \pm 0.03\ D(7\ mg/cm^2)} =$ $\frac{D(1000\ mg/cm^2)}{< 0.01\ D(7\ mg/cm^2)}$
$^{106}Ru+^{106}Rh$	$E_{res} > 2.80$ MeV	not used

[a]D refers to dose or dose rate at the depth indicated.

Table 2 - Correction Factors Applied to Measurements with the Extrapolation Chamber

		Applied to:
1. Extrapolation Chamber Signal		
$c_{T,p}$	ambient conditions different from STP	All sources
c_{recom}	incomplete ion collection in the collecting volume	All sources
c_{div}	decreasing electron flux with increasing air gap	Non contact
c_{atten}	attenuation in the chamber air gap	Series 1 only
c_{back}	difference in backscatter between tissue and PMMA	All sources
c_{side}	for scatter from the chamber sides	Series 1 only
c_{phot}	photon production in intervening material	Series 1 only
2. Source Output Corrections		
k_{dec}	source decay from reference date	All sources
k_{mass}	absorption in beam path different from that at STP	Series 1 only
k_{hum}	humidity	Series 1 only