CALIBRATION AND TRACEABILITY IN HIGH DOSE DOSIMETRY

Peter H G Sharpe
Centre for Ionising Radiation Metrology
National Physical Laboratory
Teddington TW11 0LW
United Kingdom

Abstract

Accurate dose measurements, traceable to recognized national standards, are an essential component of industrial irradiation processing. Applications such as the sterilization of medical devices and the irradiation of foodstuffs are both highly regulated and international in nature. In order to ensure both the safety of the process, and to facilitate international trade, it is essential that a widely accepted system of calibration and traceability is in place. Mutual equivalence and recognition of high dose dosimetry standards are achieved both by experimental intercomparisons, and by the adoption of standard procedures and methods, including formal accreditation of calibration laboratories by independent third parties. Dose measurements in industrial irradiation plants pose particular difficulties because the behaviour of many dosimetry systems is influenced by the environmental conditions in the plant. In particular, it is not possible to accurately mimic in a calibration laboratory the variable dose rates and temperatures characteristic of industrial plants. Calibration protocols have to be carefully designed to avoid the introduction of significant systematic errors. In this paper, the current status of industrial dosimetry standards and calibration methods are reviewed. Potential sources of error and uncertainty are discussed, and estimates made of the accuracy achievable in industrial dosimetry.

1. INTRODUCTION - NATIONAL AND INTERNATIONAL MEASUREMENT SYSTEMS

Coordination of measurements at an international level is undertaken under the framework of an international treaty, known as the “Convention of the Metre”. This treaty dates from 1875 and currently has 48 countries as signatories. The treaty establishes a number of bodies which are charged with various functions relating to the establishment and maintenance of a unified international system of measurement. Figure 1 shows the principal bodies established under the Treaty, the most important of which, from a practical point of view, are the International Committee for Weights and Measures (CIPM) and its associated laboratory the International Bureau of Weights and Measures (BIPM), located in Paris.

An important role of the BIPM is to act as a focal point for the intercomparison of standards held by individual countries, known as *national standards*. Ideally, each member state will possess only one designated *national standard* for each quantity of interest, and a system of calibrations will exist within that country to ensure that all measurements can be related to the *national standard* through an unbroken chain. Such a chain is known as a *traceability chain* and is discussed in more detail below. The relationships between BIPM, *national standards laboratories* and the “end users” of a measurement are shown schematically in Figure 2. In some instances BIPM holds physical standards with which individual *national laboratories* can compare, and in other instances BIPM acts as a coordinator of comparisons between one or more *national laboratories*. The “end users” of measurements within a country will either derive their calibrations direct from the *national standards laboratory* or from a *secondary calibration laboratory*. A significant development in recent years has been the development of formal third party accreditation schemes to ensure the competence of the *secondary calibration laboratories*. Increasingly, regulatory bodies demand documented *traceability* to *national standards* and this requirement is often most easily satisfied by obtaining calibrations from a laboratory having formal accreditation.

In this paper, the specific aspects of traceability and calibration applicable to high dose dosimetry are discussed. Significant sources of uncertainty are considered and estimates made of the accuracy achievable.

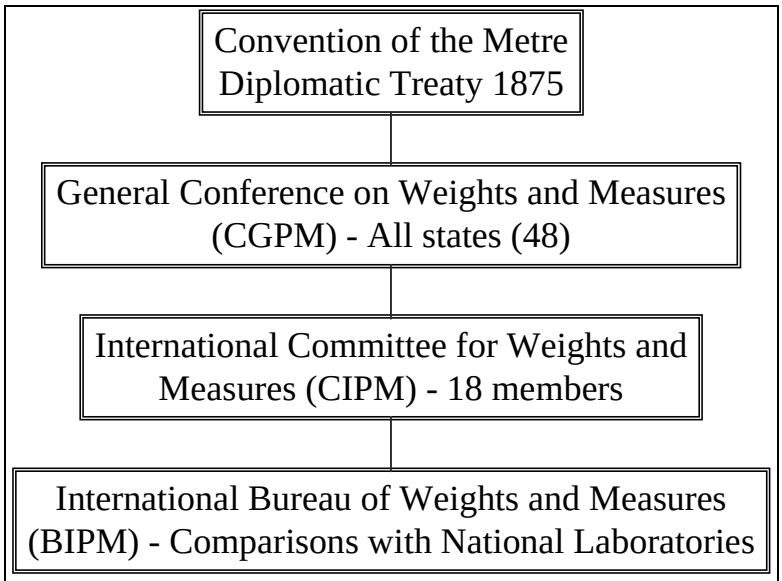

Figure 1 Structure of bodies set up under the Convention of the Metre.

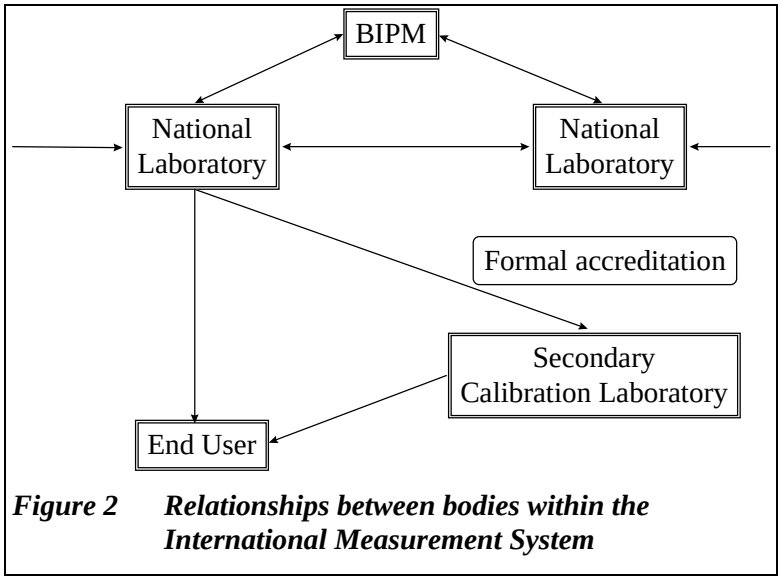

Figure 2 Relationships between bodies within the International Measurement System

2. NATIONAL TRACEABILITY CHAINS

It is useful to classify dosimeters into a hierarchy according to their intrinsic accuracy. Such a classification is shown in Figure 3 and includes dosimeters classified as *primary*, *reference* and *routine*. The figure also represents a *traceability chain* in that it outlines how *primary standard* dosimeters are used to calibrate *reference standard* dosimeters, which in turn are used to calibrate the

routine dosimeters, used for day-to-day measurement. Each class of dosimeter is described in more detail below.

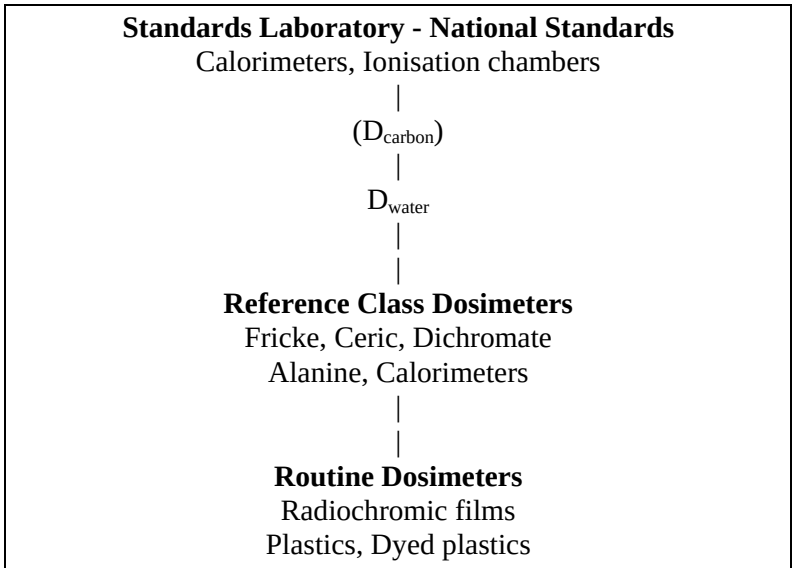

Figure 3 *A typical national traceability chain for high dose dosimetry.*

A *primary standard* dosimeter is one which enables an absolute measurement of absorbed dose to be made with reference only to the SI base units (mass, length, time, electric current etc) and fundamental physical constants. This type of dosimeter is generally operated by *national standards laboratories* and is used to provide the basic standard for use in a particular country. There are two main types of *primary standard* dosimeter, ionisation chambers and calorimeters. The quantity normally used in high dose dosimetry is *absorbed dose to water* (D_{water}), but many *primary standards* actually measure *absorbed dose to carbon* (D_{carbon}). This is shown for completeness in Figure 3, but in this paper the term *dose* is taken to mean *absorbed dose to water*.

A *reference dosimeter* is defined as a dosimeter of high metrological quality that can be used as a reference standard to calibrate other dosimeters. To be of use it must satisfy well-established criteria. It must have a radiation signal that is accurately measurable, and this signal must have a well-defined functional relationship with absorbed dose. The effect of parameters, such as irradiation temperature, post-irradiation stability, etc must be well characterised and capable of expression in terms of simple correction factors. Examples of commonly used *reference dosimeters* include chemical dosimeters, such as the Fricke, ceric-cerous, dichromate and alanine dosimeters.

Several calibration laboratories operate mailed dosimetry services in which dosimeters are shipped from the calibration laboratory to the industrial facility for irradiation and subsequently returned to the calibration laboratory for readout and certification of the dose received. These services are essential in enabling traceable calibrations of routine dosimeters, as described below. The dosimeters used in such services are known as *transfer dosimeters* and, in terms of the hierarchy in Figure 3, are usually *reference class dosimeters*. However, under certain well defined conditions, it may also be possible to use *routine class dosimeters* as *transfer dosimeters*.

A *routine dosimeter* is a dosimeter whose performance is not as good as that of a *reference dosimeter*, but whose cost and ease of use make it suitable for day-to-day measurements in a radiation processing facility. The effects of factors such as irradiation temperature and dose rate on a *routine dosimeter* tend to be complex, and it is often not possible to apply straightforward correction factors.

This means the calibration of a *routine dosimeter* is often specific to a particular environment (see below). Examples of commonly used routine dosimeters are systems based on polymethylmethacrylate (both dyed and un-dyed), cellulose tri-acetate and thin radiochromic films.

Examples of actual *traceability chains* for high dose dosimetry of Co-60 and high energy electron beams are given in Figures 4 & 5. In both cases the starting point is a *primary standard* graphite calorimeter, whose response has been compared with that of the standards maintained at BIPM. This calorimeter operates at therapy level dose rates of the order of 1 Gy/min, and a transfer dosimeter of some type is, therefore, required to step up to the high dose (kilogray) dose region. In the case of Co-60 radiation, this transfer is carried out using Fricke dosimeters, and in the case of electron beams by using an intermediate calorimeter, which is capable of operating over a wide range of dose rates. The overall uncertainty at each stage is also shown in the Figures.

System	Overall uncertainty in calibration (2σ)
Primary Standard Graphite Microcalorimeter (1 Gy / min)	1.3%
Fricke Transfer Dosimeter	1.4%
High Dose Co-60 Irradiator (100 Gy / min)	1.8%
Reference Dosimeters (Alanine & Dichromate)	2.1%

Figure 4 *UK National Traceability Chain Cobalt-60 Absorbed Dose to Water*

System	Overall uncertainty in calibration (2σ)
Primary Standard Graphite Microcalorimeter	0.6% (to graphite)
16 MeV	
High Dose Graphite Calorimeter	0.7% (to graphite)
10 MeV	
Water Phantom	2.3% (to water)

Figure 5 *UK National Traceability Chain Electron Beam Absorbed Dose to Water*

3. CALIBRATION OF HIGH DOSE DOSIMETERS

One of the major problems associated with high dose dosimeter calibration is the effect that environmental factors such as temperature (both before and after irradiation), dose rate, humidity etc

can have on the response of the dosimeter. In the case of the relatively "well behaved" *reference dosimeters*, it is necessary to state under what conditions a calibration was carried out in order that corrections can be made subsequently if the dosimeter is used to measure dose in different conditions. In the case of *routine dosimeters* it is necessary to calibrate the dosimeter under the conditions of final use, as post-calibration corrections are generally not possible. Before starting to calibrate a dosimeter it is necessary to have detailed knowledge of what environmental factors are likely to influence the particular system and to then devise a scheme which will allow any important factors to be taken into account.

For both *reference* and *routine* systems, it is generally necessary to generate a separate calibration for each identifiable “batch” of dosimeters. This is because small variations in component concentrations and production methods can alter both the response under reference conditions, and the effect of environmental factors.

Only a very few dosimeters exhibit a strict linear relationship between the readout signal and absorbed dose. This means that it is generally not possible to define a single *calibration factor* for a dosimeter, and a curved *calibration function* has to be used instead. The form of the function is somewhat arbitrary, and can be any mathematical expression that is capable of reproducing the observed response. In general, the best expression will be the simplest one that will adequately reproduce the observed response.

The curved nature of most dosimeter *calibration functions* has implications both for the dose range over which a system must be calibrated, and also for the number of discrete calibration dose points that must be used. A dosimeter must be calibrated over a dose range significantly wider than the dose range of subsequent use. A curved *calibration function* cannot be extrapolated outside the dose range over which it was derived; there is simply not the information available to do this. Polynomial based calibration functions, in particular, may exhibit totally unexpected behaviour outside the dose range of preparation. The number of discrete calibration dose points required will depend both on the dose range and also the degree of curvature of the calibration function. As a guide, at least five calibration dose points distributed arithmetically (i.e. spaced at a fixed dose apart) will generally be required for irradiations spanning less than one decade of dose. For irradiations spanning more than one decade of dose, at least five dose points will generally be required for each decade, and these should be distributed geometrically (i.e. spaced at fixed multiples of each other). There must always be more dose points than there are coefficients in the mathematical expression used to represent the observed response. It is also good practice to use several replicate dosimeters at each dose point, in order that a full statistical analysis can be carried out on the calibration data. In order not to bias the calibration line, the same number of replicates should be used at each dose point.

3.1 Calibration of reference dosimeters

The calibration of *reference dosimeters* is relatively straightforward in that the response of the dosimeter to environmental conditions is, by definition, well defined and corrections can therefore be applied to relate the response under one set of conditions to those under another. Nevertheless, such corrections are subject to uncertainty, and for the highest accuracy, the reference dosimetry system should be calibrated under conditions as close as possible to those of eventual use. The calibration process consists of measuring the response of the dosimetry system when irradiated to a series of known doses under well defined conditions. The conditions that need defining vary according to the properties of the dosimetry system, but are likely to include temperature, dose rate and radiation spectrum. Humidity during irradiation, and environmental storage conditions after irradiation, may also need to be either controlled, or monitored to ensure they are within acceptable limits.

3.2 Calibration of routine dosimeters

Because the response of *routine dosimeters* cannot, in general, be easily corrected for variations in environmental factors, the dosimeters must be calibrated in the same conditions as those of eventual use. There are two main ways of achieving this, both of which have advantages and disadvantages:

3.2.1 Calibration in the irradiation plant

The best method of ensuring all relevant environmental influence factors have been taken properly into account is to calibrate *routine dosimeters* in the plant in which they will used. The usual method of achieving this is to irradiate the dosimeters to be calibrated alongside *reference dosimeters* supplied and measured by an accredited calibration laboratory. To ensure both types of dosimeter receive the same dose, it is good practice to irradiate in a specially designed *calibration phantom*, in which the dosimeters are surrounded by material having approximately the same density and radiation absorption properties as themselves. In the case of electron beam irradiation, this phantom also ensures that all dosimeters are irradiated at the same point on the depth dose curve. The calibration phantom needs to be large enough to ensure that dosimeters do not shield each other, but not so large that errors are introduced because of non-uniformity of the radiation field.

The response of the *reference dosimeters* used in the calibration will need to be corrected for irradiation temperature. In an industrial irradiation plant, the temperature is unlikely to be constant, and some means has to be devised to determine an *effective temperature* for the *reference dosimeters*. For electron beam irradiations this is fairly straightforward as the dose will be delivered in a short time compared to the rate of heat loss, and the *effective temperature* can be taken as the average temperature during irradiation. The situation in an industrial gamma ray plant is far more complex as the irradiation is delivered over a relatively long time, and the dosimeter has time to exchange heat with the surroundings. It is reasonable to expect that the highest temperatures will be experienced close to the source, where the dose rate is highest, and the *effective irradiation temperature* would, therefore, be expected to be somewhere between the average and maximum temperatures experienced. In situations where the temperature range is large enough for uncertainties in *effective temperature* to make a significant contribution to the overall uncertainty, consideration should be given to using more than one type of *reference dosimeter*. If the chosen systems have different irradiation temperature coefficients, for example alanine and dichromate, it is possible to factor out the effect of irradiation temperature by analysing the difference in apparent doses measured by the two systems.

The principal disadvantage of the *in-plant* calibration method is that it may be difficult to achieve the required range of doses in some designs of industrial gamma irradiators. This can sometimes be overcome by irradiating calibration dosimeters to only part of the full irradiation cycle, but care needs to be taken to ensure that such dosimeters experience the same environmental conditions as dosimeters completing the full cycle.

3.2.2 Irradiation at a calibration facility followed by in-plant verification

It is possible to calibrate *routine dosimeters* by performing a set of irradiations at a remote calibration facility, provided steps are taken to verify the applicability of the calibration to the plant in which the routine dosimeters will be used. Without this verification step, systematic errors in the calibration may go undetected, and it is not possible to apply any meaningful uncertainty estimates to the calibration.

Calibration verification can be carried out by comparing the doses measured by *routine* and *reference* dosimeters irradiated alongside each other in the irradiation plant. In contrast to the full range of doses required for the *in-plant* calibration described above, the calibration verification need only be carried out at a small number of dose points (usually 3) spread along the dose range of the

routine dosimeters. If both *reference* and *routine* dosimeters give the same dose, within the uncertainty of the systems involved, then the calibration of the *routine dosimeters* is verified and can be used without further correction. If the verification shows a difference between the two systems that is essentially constant over the dose range, then a correction factor can be applied to bring the readings of the *routine dosimeters* into agreement with the readings of the *reference dosimeters*. Differences between *reference* and *routine* dosimeters that vary significantly across the dose range indicate a major discrepancy between environmental conditions in the plant and those used for the calibration irradiation. In such situations it is generally not possible to correct the calibration line, and a full *in-plant* calibration is required.

The purpose of the *calibration verification* is to detect small errors arising from differences between the conditions of calibration and final use of *routine dosimeters*. In order to minimise the size of potential errors, the calibration irradiation should be carried out using conditions of dose rate and temperature as close as possible to those which will be experienced in the industrial plant. Estimates of irradiation temperature for both the calibration irradiation, and the subsequent verification can be made using the same philosophy as outlined above. It may also be possible to use two types of reference dosimeter with differing temperature coefficients to correct for irradiation temperature.

4. UNCERTAINTY OF HIGH DOSE MEASUREMENTS

In order to establish the accuracy of a dose measurement, it is necessary to first identify and then quantify all possible sources of uncertainty. This is most easily done by considering in turn each step in the calibration and use of a dosimeter, and assessing what uncertainties are likely to be associated with each stage. The uncertainty associated with a dose measurement can then be calculated by combining the individual components. An example of this is given in Figure 4, where uncertainties for the calibration alanine and dichromate *reference dosimeters* in a Co-60 beam are given as 2.1% (2σ). Uncertainties in *routine dosimetry* measurements are more difficult to assess, as they are influenced significantly by environmental factors. A detailed discussion of uncertainties is outside the scope of this paper, but some of the principle components of uncertainty that affect high dose measurements are listed below.

4.1 Uncertainties in the Preparation of a Calibration Function

Uncertainty in calibration doses - The certificates supplied by calibration laboratories in connection with their *reference dosimeters* or calibration irradiations will contain statements about uncertainties and the *traceability* to *national standards*. Consideration also has to be given to dose variation arising from the relative positioning of dosimeters during calibration irradiations.

Uncertainty due to fit of calibration function - The process of fitting a *calibration function* to measured data will be subject to uncertainty. Statistical software packages may provide this information, but a detailed analysis is complex for anything except a straight line. Approximations of this uncertainty can be obtained from the differences between the original data and the calculated values.

Uncertainty due to environmental influence factors - Environmental factors such as temperature, humidity and dose rate can influence the response of high dose dosimeters. Calibration procedures should be designed to correct as much as possible for these effects, but uncertainties will still remain. Typical examples are uncertainties arising from the difficulties in assessing *effective temperatures and dose rates* in Co-60 plants.

4.2 Uncertainties in the Use of Dosimeters

Uncertainty due to dosimeter-to-dosimeter scatter - This is the statistical scatter observed between replicate dosimeters irradiated to the same dose under the same conditions.

Uncertainty due to variation in plant environmental conditions - Environmental conditions in industrial plants are often not well controlled. Variations, for example from summer to winter, may cause additional uncertainties in dose measurements.

Uncertainty due to instability of dosimeter reading - The readings of many dosimeters change with time after irradiation. Variations in readout times will, therefore, introduce uncertainties in dose measurements.

Uncertainty due to instability of instrumentation - Instrumental instabilities will translate directly into uncertainties in dose measurement. The effect of variations in, for example, the wavelength settings of spectrophotometers needs to be assessed.

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