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**Practice for calibration of routine  
dosimetry systems for radiation  
processing**

*Pratique d'étalonnage des appareils de mesure dosimétrique  
routinier pour le traitement par irradiation*



Reference number  
ISO/ASTM 51261:2013(E)

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## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75% of the member bodies casting a vote.

ASTM International is one of the world's largest voluntary standards development organizations with global participation from affected stakeholders. ASTM technical committees follow rigorous due process balloting procedures.

A pilot project between ISO and ASTM International has been formed to develop and maintain a group of ISO/ASTM radiation processing dosimetry standards. Under this pilot project, ASTM Committee E61, Radiation Processing, is responsible for the development and maintenance of these dosimetry standards with unrestricted participation and input from appropriate ISO member bodies.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. Neither ISO nor ASTM International shall be held responsible for identifying any or all such patent rights.

International Standard ISO/ASTM 51261 was developed by ASTM Committee E61, Radiation Processing, through Subcommittee E61.01, Dosimetry, and by Technical Committee ISO/TC 85, Nuclear energy, nuclear technologies and radiological protection.

A2.1 Curve fitting is the application of regression analysis techniques to a set of data where by the selected mathematical form (model) defines the dependent variable ( $Y$ ) in terms of the independent variable ( $X$ ). Regression analysis is used to fit data to a model and provide estimates of the fit parameters (coefficients) based on a minimization technique.

A2.2 Regression models are either an empirical or a mechanistic model. The empirical model describes the general shape of the data set. The parameters of the empirical model do not correspond to an underlying biological, chemical or physical process. The mechanistic model is formulated to provide insight or description of the process under study.

A2.3 The two basic types of regression analysis are linear regression and non-linear regression. Linear regression is where the unknown parameters (coefficients) appear linearly in the expression as in Eq A2.1. Non-linear regression is where the unknown parameters (coefficients) appear in a non-linear or nested fashion as in Eq A2.2.

$$y = a + bx + cx^2 + dx^3 \quad (\text{A2.1})$$

$$y = \frac{a}{1 + \left(\frac{x}{b}\right)^c} \quad (\text{A2.2})$$

NOTE A2.1—In the context of regression analysis, the terms linear and non-linear do not refer to the shape of the plotted curve, for example, both Eq A2.1 and Eq A2.2 represent curved plots.

A2.3.1 In both types of regression analysis (linear and nonlinear) several assumptions are made:

A2.3.1.1  $X$  is known precisely and all error is in  $Y$ . (It is sufficient that imprecision in measuring  $X$  is very small compared to the variability in  $Y$ . Error refers to deviation from the average.)

A2.3.1.2 Variability of  $Y$  at any  $X$  follows a known distribution, typically assumed to be Gaussian or near Gaussian.

A2.3.1.3 The standard deviation of the residuals is the same along the curve (homoscedasticity).

NOTE A2.2— In some dosimetric calibration data, homoscedasticity does not exist and is corrected with the use of a weighting factor, see Eq A2.3 and Eq A2.4.

A2.3.1.4 Observations ( $Y$ ) are independent (whether one point is above or below the regression analysis model curve is a matter of chance and does not influence whether another point is above or below the regression analysis model curve).

A2.4 A minimization technique is used to determine the coefficients of the regression model form that provides the best fit. The most common technique for linear fitting is a least squares algorithm which minimizes the sum of the squares of the residuals (SSE) where a residual is the vertical distance between the data point and regression model curve (reference Eq A2.3). The most common technique for non-linear fitting is the Levenberg-Marquardt algorithm. Most commercially available regression software will provide linear and non-linear regression and multiple minimization algorithms.

$$SSE = \sum_{i=1}^n w_i (y_i - \hat{y}_i)^2 \quad (\text{A2.3})$$

where:

$y_i$  = the observed dependent variable at an independent variable value,

$\hat{y}_i$  = the model predicted value of the dependent variable at the corresponding independent variable, and

$w_i$  = assigned weight which in most cases is assumed to be 1 unless a weighting is applied to compensate for a deviation of homoscedasticity (A2.3.1.3).

NOTE A2.3—When the Gaussian distribution of error assumption is invalid due to appreciable tails in the residuals distribution, the assumption that least squares provides the maximum likelihood fit is also invalid. In these instances a robust method of minimization may be used. The essence of robust fitting is to use a minimization technique that is less influenced by potential outliers and the range of the dependent variable. Several examples of nonlinear robust minimization are Least Absolute Deviation, Lorentzian, and Pearson.

A2.5 Goodness of fit describes how well the model fits a set of data. Measures of goodness of fit typically summarize the discrepancy between observed values ( $y_i$ ) and the values predicted by the model ( $\hat{y}$ ). A review of a plot of the residuals is critical when assessing goodness of fit. The most commonly used statistics for assessing goodness of fit are the coefficient of determination, lack of fit sum of squares ( $F$  statistic), confidence intervals of the fit coefficients, and the  $F$  test when comparing fits between different models. Another powerful non-statistical evaluation method is a review of the plot of the residuals.

A2.5.1 A plot of the residuals can reveal behaviour in the data that is otherwise difficult to see in the curve fit. A plot of the residuals should not demonstrate a form or trend. A residuals plot may also indicate potential or suspect outliers (see A2.6).

A2.5.2 The coefficient of determination ( $r^2$ ) has no units and ranges in value between 0 and 1 which is computed as shown in Eq A2.4. A value of 1.0 indicates the curve passes through all the data points. The coefficient of determination can be interpreted as the fraction of the total variance in  $y$  that is explained by the model. A common mistake is using the coefficient of determination solely as the gauge of goodness of fit; this may lead to the selection of a model that may fluctuate wildly with very large confidence intervals.

$$r^2 = 1 - \frac{SSE}{SSM} = 1 - \frac{\sum_{i=1}^n w_i (y_i - \hat{y}_i)^2}{\sum_{i=1}^n w_i (y_i - \bar{y})^2} \quad (\text{A2.4})$$

where:

$SSE$  = sum of the squares of the residuals,  
 $SSM$  = sum of the squares deviation about the mean,  
 $\bar{y}_i$  = average response at dose level  $i$ , and

$w_i$  = assigned weight which in most cases is assumed to be 1 unless a weighting is applied to compensate for a deviation of homoscedasticity (A2.3.1.3).

A2.5.3 The  $F$ -statistic is a measure of the extent to which the given model represents the data. The  $F$ -statistic is calculated as the ratio of the mean square error of the regression to the mean square error:

$$F = \frac{MSR}{MSE} = \frac{\left(\frac{SSM - SSE}{m - 1}\right)}{\left(\frac{SSE}{DF}\right)} \quad (A2.5)$$

where:

$$SSM = \sum_{i=1}^n w_i (y_i - \bar{y})^2$$

$$SSE = \sum_{i=1}^n w_i (y_i - \hat{y}_i)^2$$

$m$  = number of coefficients fitted,

$n$  = number of data points, and

$DF$  =  $n - m$

A larger  $F$  ratio indicates the model fits the data well.

A2.5.4 The regression analysis estimates coefficients of the model for the fit of the data. Most commercially available regression software provides an estimate of the standard error for each coefficient and the 95 % confidence interval about the coefficient estimate. The value of the standard error and the 95 % confidence interval provides a means to gauge how well the regression has determined the coefficients. If the assumptions of A2.3 are not significantly violated, the 95 % confidence interval is considered to be an interval that has a 95 % chance of containing the 'true' value of the coefficient. If the confidence intervals are wide, the coefficient has not been determined precisely. If the confidence intervals are narrow, the coefficients have been determined precisely.

A2.6 Suspect outlying observations can typically be identified from a review of the residuals plots (reference A2.5.1). Generally, a dosimetry system calibration consists of relatively few dependent replicate observations ( $y_i$ ) for any given independent value ( $x$ ). As a result of relatively few replicate observations, it is likely that variation in dependent response may express a value that appears to be significantly different than other observations even when the observation is from the same population with a Gaussian distribution of error. When a suspect outlier is proven to be an outlier it should be removed from the data set prior to regression analysis.

A2.6.1 Although not rigorously defined, an outlier is an observation from a population other than the population under study. Thus, a suspect outlier must be proven to come from a different population before it can be removed. An outlier then is the result of:

A2.6.1.1 An extreme observation that is part of the population under study (false discovery).

A2.6.1.2 An observation from a population other than the one under study (true discovery).

A2.6.1.3 An incorrect assumption of the population distribution of error (usually results in false discovery).

A2.6.2 Extreme observation values are probable in a Gaussian distribution of error although they are highly unlikely. Statistical outlier tests are the application of statistical infer-

ence which is based on an assumed probability distribution. Most statistical outlier tests are applied at a 95 % level of significance. This means that 5 % of the true population (either in a single-sided or double-sided test) will be identified by the statistical outlier test as significant. Unless it can be identified that the suspect observation is the result of an experimental error or the sample is in violation of criterion applied qualifying it as a viable sample, it can not be conclusively proven to come from a different population by a statistical outlier test. Although not conclusive in and of themselves, several methods are used to identify suspect outliers:

A2.6.2.1 Visual inspection of plot of the residuals (qualitative).

A2.6.2.2 Confidence Intervals (quantitative).

A2.6.2.3 Prediction Limits (quantitative).

A2.6.2.4 Statistical test such as a  $t$ -test (quantitative).

A2.6.3 A visual inspection of the residuals plot is a qualitative means of quickly identifying suspect outliers.

A2.6.4 Confidence intervals make use of the assumptions of linear and non-linear regression about the population distribution of the observations used to identify a measure estimate, specifically the assumption of a Gaussian distribution of error. The confidence interval is a range of values where at a specified confidence coefficient (95 or 99 %) the 'true' value exists. For regression analysis, this is an interval wherein the 'true' best fit curve lies for a specified level of confidence, for example, 95 % probability for the given model. This is not the same as inferring a 95 % confidence interval contains 95 % of the observations. Given this, a confidence interval is not a suitable measure for identifying suspect outliers.

A2.6.5 Prediction intervals, similarly to confidence intervals, assume a Gaussian distribution of error. The prediction interval describes error about the curve or scatter associated with the individual observations. In this case a 95 % prediction interval is expected to contain 95 % of the observations from the single experiment. Thus, prediction intervals are a useful tool in identifying suspect outliers. For example, a 99.9 % prediction interval would be expected to contain 99.9 % of the observations. Observations outside of this interval are then considered highly probable suspect outliers.

NOTE A2.4—A distinction between confidence intervals and prediction intervals is if the number of replicates is significantly increased, the confidence interval would become smaller while the prediction interval would not change appreciably provided the assumption of a Gaussian error distribution is valid.

A2.6.6 Statistical tests are routinely used to identify suspect outliers (see ASTM Practice E178). As identified, any statistical test in and of itself is not conclusive evidence of an outlier. The suspect outlier must be identified through investigation to be a sample from a population other than that population under study.

A2.6.7 The uncertainty of the regression curve describes the quality of the selected model and regression analysis in characterizing the relationship of the dependent and independent variables. The confidence interval about the regression curve is used to quantify the uncertainty of the curve fit. The confidence interval is not constant over the curve range and is

generally wider at the upper and lower extremes of the curve (see Fig. A3.6). The confidence interval represents an interval in which the true value of the curve exists at the identified confidence coefficient (95 or 99 %). The uncertainty of the dose estimate ( $\hat{x}$ ) can be estimated at any single dose as the ratio of the one half the dose range defined by the confidence interval to the dose estimate ( $\hat{x}$ ), reference Fig. A2.1 and Eq A2.6.

$$U_{\hat{x}}\% = \left( \frac{D_{UCL} - D_{LCL}}{\hat{x}} \right) \times 100 \quad (\text{A2.6})$$

$D_{UCL}$  = dose at the upper confidence level,  
 $D_{LCL}$  = dose at the lower confidence level.

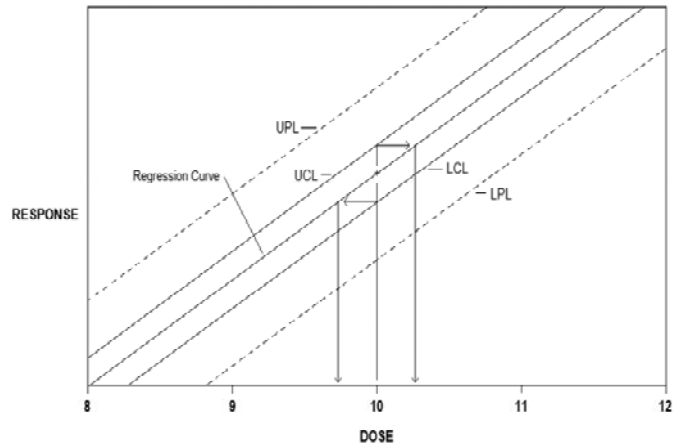


FIG. A2.1 Confidence and prediction intervals about the regression curve

### A3. CALIBRATION EXAMPLE

A3.1 The following is an example of a laboratory calibration of a routine dosimetry system based on a type II film dosimeter. This example is a simplified treatment of a calibration and focuses on the mechanics of computation, and does not address the measurement management system specifications and procedures or design of experiment that are required for both a laboratory calibration and in-situ calibration.

A3.2 Prior to the selection of calibration dosimeters, inspect the dosimeter stock for suitability in accordance with a measurement management system. Characteristics that are evaluated are those that impact the routine performance of the routine dosimetry system but also characteristics that affect the laboratory calibration method such as post-irradiation development.

A3.3 Upon dosimeter stock inspection and approval, calibration dosimeters are drawn from the stock. The number of dosimeters for each dose level and the number of dose levels are selected and the total number of dosimeters drawn.

A3.4 The calibration dosimeters are then irradiated.

A3.4.1 For a laboratory calibration method, calibration dosimeters are sent to an approved calibration laboratory for calibration irradiation. Specify and document the irradiation dose rate and irradiation temperature provided to the approved calibration laboratory. These parameters are critical for the success of the laboratory calibration method. Values for dose rate and irradiation temperature should be selected based on the knowledge of the routine measurement conditions and knowledge of the routine type II dosimeter response to the routine measurement influence quantity conditions. Calibration irradiation response data for a type II dosimeter are given in Table A3.1.

A3.5 Regression analysis is applied to the data set for the model below:

$$y = cx^2 + bx + a \quad (\text{A3.1})$$

A3.5.1 A review of the residuals of the fit model identifies a suspect outlier at the 40 kGy dose level (reference Fig. A3.1). The suspect outlier can be statistically tested, however, the statistical test alone should not be used as the sole basis for the datum omission.

A3.5.1.1 Removing the outlier, the data is re-fitted with the resulting residual plots; Fig. A3.2.

A3.5.1.2 An inspection of the residuals identifies an “oscillating” form. A more complex form should be evaluated for better fit. The more complex form is:

$$y = dx^3 + cx^2 + bx + a \quad (\text{A3.2})$$

A3.5.2 Using the  $F$  test to evaluate the more complex 3rd order polynomial model to the 2nd order polynomial gives:

$$F = \frac{(SS_{null} - SS_{alt})(DF_{null} - DF_{alt})}{SS_{alt}/DF_{alt}} = \frac{4.8714326 \times 10^{-3} - 6.2227065 \times 10^{-4}/(28 - 27)}{6.2227065 \times 10^{-4}/27} = 184.369 \quad (\text{A3.3})$$

where:

- $SS_{null}$  = sum of squares of the null hypothesis model (simple model),
- $SS_{alt}$  = sum of squares of the alternate hypothesis model (complex model),
- $DF_{null}$  = degrees of freedom of the null hypothesis model (simple model), and
- $DF_{alt}$  = degrees of freedom of the alternate hypothesis model (complex model).

A3.5.2.1 Solving the  $F$  distribution for an  $F$  value of 184.369 with 1 degree of freedom in the numerator and 27 degrees of freedom in the denominator) gives a  $p$  value of  $<<0.001$ .

NOTE A3.1—Microsoft Excel will calculate the  $p$  value with the

TABLE A3.1 Calibration sample response data

Dose Level	Replicate	Response	Thickness	$k$ , (response/ thick) x (Norm. Constant)
3 kGy	1	0.188	30.1	0.187
	2	0.188	29.9	0.189
	3	0.186	30.0	0.186
	4	0.186	30.2	0.185
5 kGy	1	0.318	30.1	0.317
	2	0.313	29.8	0.315
	3	0.314	30.0	0.314
	4	0.309	29.9	0.310
10 kGy	1	0.590	29.5	0.600
	2	0.605	30.3	0.599
	3	0.598	30.4	0.590
	4	0.593	30.0	0.593
15 kGy	1	0.842	30.0	0.842
	2	0.842	30.1	0.839
	3	0.829	29.7	0.837
	4	0.831	29.9	0.834
20 kGy	1	1.075	30.6	1.054
	2	1.063	30.3	1.052
	3	1.036	29.7	1.046
	4	1.041	29.8	1.048
30 kGy	1	1.373	29.9	1.378
	2	1.390	30.1	1.385
	3	1.401	30.2	1.392
	4	1.390	29.9	1.395
40 kGy	1	1.641	30.1	1.636
	2	1.705	30.5	1.677
	3	1.629	30.0	1.629
	4	1.600	29.6	1.622
50 kGy	1	1.764	29.4	1.800
	2	1.801	29.9	1.807
	3	1.798	29.8	1.810
	4	1.816	30.0	1.816

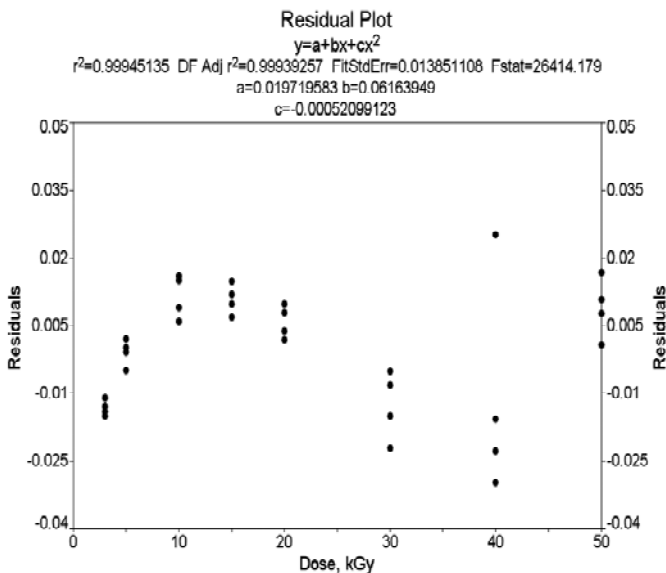


FIG. A3.1 Residual plots

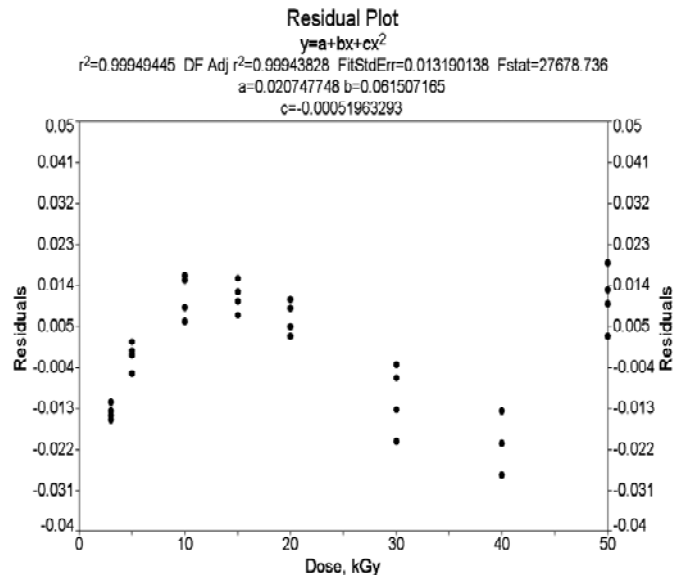


FIG. A3.2 Residuals plot

following formula syntax:  $Fdist(F, DF_n, DF_d)$ , where  $DF_n$  is the degrees of freedom in the numerator and  $DF_d$  is the degrees of freedom in the denominator.

A3.5.2.2 The extremely small  $p$  value warrants testing a more complex model, a 4th order against the 3rd order.

$$\begin{aligned}
 F &= \frac{(SS_{null} - SS_{alt}) / (DF_{null} - DF_{alt})}{SS_{alt} / DF_{alt}} \\
 &= \frac{6.2227065 \times 10^{-4} - (6.2069049 \times 10^{-4}) / (27 - 26)}{6.2069049 \times 10^{-4} / 26} \\
 &= 0.066191058 \tag{A3.4}
 \end{aligned}$$



A3.5.2.3 Solving the  $F$  distribution for an  $F$  value of 0.066191058 and degrees of freedom of 1 ( $DF_n$ ) and 26 ( $DF_d$ ) for a  $p$  value of  $p = 0.799$ , which indicates the less complex model is the better fit.

A3.5.2.4 A large  $p$  value means the relative increase in the sum of squares is approximately equal to the relative increase in degrees of freedom, i.e. nothing substantial is gained in the fit with the extra degree of freedom used to fit the additional fit coefficient and the less complex model is the better fit. Typically a  $p$  value above 0.05 indicates acceptance of the less complex model and a  $p$  value below 0.05 indicates acceptance of the more complex model. In the case of the 4th order polynomial and the 3rd order polynomial in the example, the 3rd order provides a better fit ( $p = 0.799$ ).

A3.5.3 The objective of regression analysis is to determine the best fit values of the parameters of the selected model. However, a statement must be made about the parameter estimate, specifically how precisely have the fit coefficients been determined. The standard error and confidence interval of the fit coefficient value is an estimate of how precisely the fit coefficient has been determined. The standard error of a fit coefficient is the expected value of the standard deviation of that coefficient. The construction of a confidence interval at a desired level of confidence about the parameter value is based on the standard error. The confidence interval identifies a range within which the 'true' value of the fit coefficient to be at a stated level of confidence. Thus, the smaller the confidence interval of a fit coefficient, the better the coefficient has been determined.

A3.5.3.1 Review of the parameter estimates of the 3rd order polynomial shown in Table A3.2, the values of standard error and confidence intervals for each parameter.

A3.5.3.2 The  $t$  value can also provide a degree of certainty with which the fit parameters are determined. The highest  $t$  value indicates the greatest contribution to the fit but is also determined to the greatest level of certainty. A positive  $t$  value indicates a direct relationship between the coefficient and the dependent variable ( $y$ ) where a negative value indicates an inverse relationship.

A3.5.3.3 As shown in Table A3.2 results, the 'b' coefficient is the best determined parameter and has a direct relationship with the dependent variable. The 'a' coefficient is the least well determined coefficient and has an inverse relationship with the dependent variable. The confidence intervals for each coefficient show relatively small intervals indicating the coefficients are well determined. Plots of the 3rd order regression curve and residuals plots are shown in Fig. A3.3, Fig. A3.4, and Fig. A3.5.

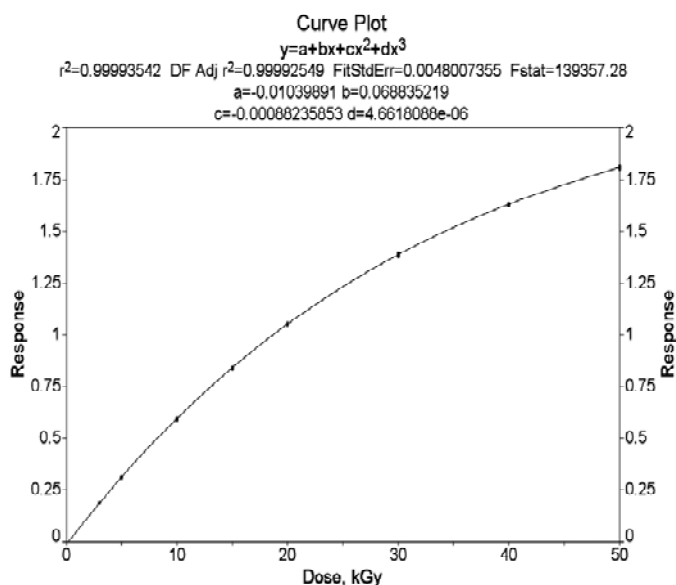


FIG. A3.3 Regression curve

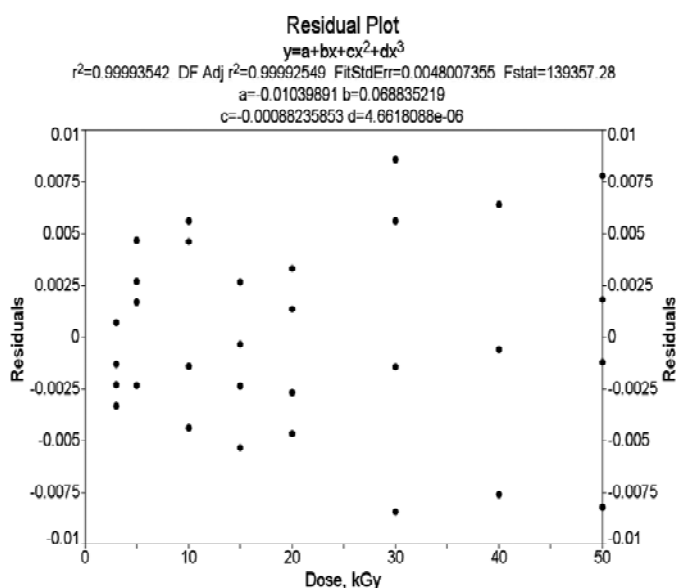


FIG. A3.4 Residuals plot

A3.6 The regression curve is fitted as  $y = f(x)$ , however the inverse  $x = f^{-1}(y)$  is used to estimate absorbed dose for a given dosimeter response value. Directly observable in Fig. A3.4 and Fig. A3.5, variation in the dosimeter response is expected. Well

TABLE A3.2 Third order polynomial coefficient standard error and confidence intervals

Coefficient	Estimate	Standard Error	$t$ value	Upper 95 % Confidence Interval	Lower 95 % Confidence Interval
a	-0.01039891	0.003164916	-3.28568331	-0.01689278	-0.00390504
b	0.068835219	0.000583230	118.0241762	0.067638530	0.070031908
c	-0.00088236	2.7033e-5	-32.6396921	-0.00093783	-0.00082689
d	4.66181e-6	3.43329e-7	13.57825192	3.95736e-6	5.36626e-6

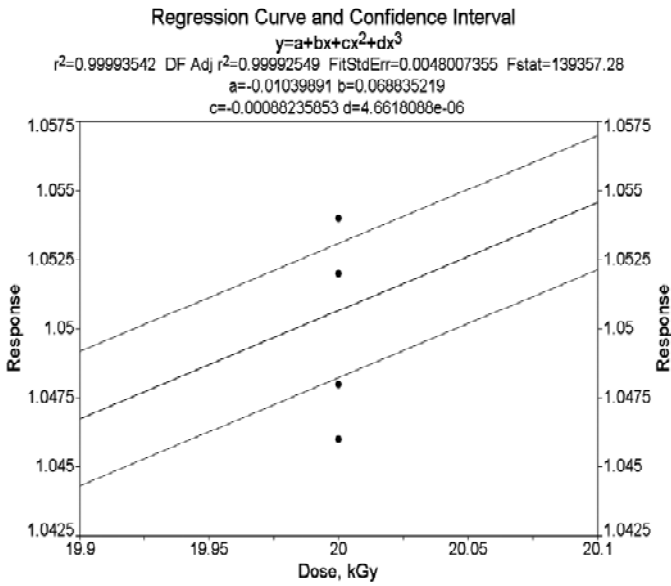


FIG. A3.5 20 kGy dose level regression curve and 95 % confidence intervals

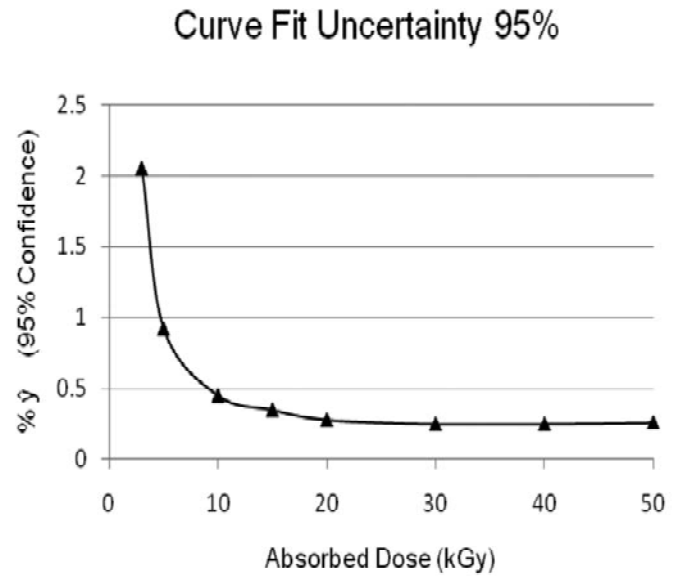


FIG. A3.6 95 % Confidence interval (5 of y)

controlled and monitored radiation processing requires knowledge and an accurate estimate of the repeatability of the routine dosimetry system absorbed dose measurement. Repeatability of the absorbed dose measurement is estimated using the inverse of the fit regression curve and the calibration sample response. The estimate of measurement repeatability is calculated as a pooled relative variance given by Eq A3.5. The 'k' values from Table A3.1 are used to calculate the dose for each calibration sample replicate. A summary of the components of Eq A3.5 are given in Table A3.3.

where:

- $s^2$  = the variance of the measurement estimate ( $x$ ) of the model inverse,  $x = f^{-1}(y)$ ,
- $\bar{d}^2$  = the square of the average replicate observation estimates ( $\hat{x}$ ) of the model inverse,  $x = f^{-1}(y)$ ,
- $n$  = the number of replicate estimates ( $\hat{x}$ ) at the dose level  $m$ ,
- $m$  = the number of dose levels, and
- $k$  = coverage factor ( $k=2$  approximates a 95 % confidence level, or  $2\sigma$ )

A3.6.1 The repeatability associated with absorbed dose measurements from the 3rd order polynomial is given by Eq A3.6:

$$Precision = k \left( \frac{\sum_{i=1}^m (n-1)_i \left( \frac{s^2}{\bar{d}^2} \right)}{\left( \sum_{i=1}^m n_i \right) - m} \right)^{\frac{1}{2}} \quad (A3.5)$$

$$\left[ k \left( \frac{\sum_{i=1}^m (n-1)_i \left( \frac{s^2}{\bar{d}^2} \right)_i}{\left( \sum_{i=1}^m n_i \right) - m} \right)^{\frac{1}{2}} \right] \quad (A3.6)$$

$$= \left[ 1 \left( \frac{3(8.1172 \times 10^{-5}) + 3(9.4169 \times 10^{-5}) + 3(8.2886 \times 10^{-5}) + 3(2.4372 \times 10^{-5}) + 3(2.1778 \times 10^{-5}) + 3(7.8801 \times 10^{-5}) + 2(7.1979 \times 10^{-5}) + 3(7.3000 \times 10^{-5})}{(31) - 8} \right)^{\frac{1}{2}} \right]$$

$$= \left[ 1 \left( \frac{1.1512492 \times 10^{-3}}{23} \right)^{\frac{1}{2}} \right] = \left[ 1 \left( \frac{1.151249 \times 10^{-3}}{23} \right)^{\frac{1}{2}} \right] = [1(8.109286 \times 10^{-3})] = 0.008109286$$

Which when reported as a percent is  $\pm 0.81$  % at  $1\sigma$ .

A3.7 Calibration verification for the conditions of use establishes the measurement traceability for the use of the routine dosimetry system within the routine measurement application. Testing consists of co-location of replicates of the routine dosimeters and transfer standard dosimeters at a minimum of three dose levels over the calibration curve range

(see 9.1.8). The specific irradiation pathways and parameters are part of the design of experiment. They should be selected so that the validity of the calibration curve near the extremes of expected routine use conditions is tested. For an in-situ/in-plant calibration, verification is only performed when an event such as those identified in 9.2.9.1 have occurred.

**TABLE A3.3 Component for the estimate of measurement repeatability**

Dose Level kGy	Dose Level Average (kGy)	Standard Deviation (s)	Variance ( $s^2$ )	Relative Variance ( $s^2/\bar{d}^2$ )	Number of Replicates (n)
3	2.975804	$2.6810721 \times 10^{-2}$	$7.18815 \times 10^{-4}$	$8.1172 \times 10^{-5}$	4
5	5.028182	$4.8793782 \times 10^{-2}$	$2.380833 \times 10^{-3}$	$9.4169 \times 10^{-5}$	4
10	10.021406	$9.1236514 \times 10^{-2}$	$8.324101 \times 10^{-3}$	$8.2886 \times 10^{-5}$	4
15	14.970798	$7.3907351 \times 10^{-2}$	$5.462297 \times 10^{-3}$	$2.4372 \times 10^{-5}$	4
20	19.983328	$9.3256357 \times 10^{-2}$	$8.696748 \times 10^{-3}$	$2.1778 \times 10^{-5}$	4
30	30.039379	$2.66658922 \times 10^{-1}$	$7.1106981 \times 10^{-2}$	$7.8801 \times 10^{-5}$	4
40	39.972510	$3.39129695 \times 10^{-1}$	$1.15008950 \times 10^{-1}$	$7.1979 \times 10^{-5}$	3
50	50.005350	$4.27245238 \times 10^{-1}$	$1.82538493 \times 10^{-1}$	$7.3000 \times 10^{-5}$	4

**TABLE A3.4 Calibration verification test results**

Dose Target	Routine Dosimeter Dose ( $d_R$ )	Transfer Standard Dose ( $\bar{d}_T$ )	$\bar{d}_T^2$	$s^2(\text{Var}_m)$	$s^2/\bar{d}_T^2$	Number of routine dosimeter replicates, n
15 kGy	13.9	14.2	201.64	$6.500 \times 10^{-2}$	$3.22357 \times 10^{-4}$	4
	13.9					
	14.0					
	14.0					
25 kGy	24.3	23.9	571.21	$4.9000 \times 10^{-1}$	$8.87279 \times 10^{-4}$	4
	24.3					
	23.7					
	23.7					
40 kGy	40.5	40.2	1616.04	$1.8500 \times 10^{-1}$	$1.14477 \times 10^{-4}$	4
	39.7					
	40.4					
	39.6					

A3.7.1 The absorbed dose results of the routine dosimeters and transfer standard dosimeters are evaluated as a pooled relative variance sum of squares (see Eq A3.7). For the

example, Table A3.4 shows absorbed dose results for the routine type II dosimeters and the co-located transfer standard dosimeters at three dose levels.

$$\begin{aligned}
 & \left[ k \left( \frac{\sum_{i=1}^m (n_i - 1) \left( \frac{\text{Var}_m}{(\bar{d}_{Tm})^2} \right)_i}{\left( \sum_{i=1}^m n_i \right) - m} \right)^{\frac{1}{2}} \right] \quad (\text{A3.7}) \\
 &= \left[ 1 \left( \frac{3(3.22357 \times 10^{-4}) + 3(8.87279 \times 10^{-4}) + 3(1.4477 \times 10^{-4})}{(12 - 3)} \right)^{\frac{1}{2}} \right] \\
 &= \left[ 1 \left( \frac{(3.972339 \times 10^{-3})}{(9)} \right)^{\frac{1}{2}} \right] = [1(0.021008831)] = 0.021008831
 \end{aligned}$$

When reported as a percent is  $\pm 2.10\%$  at  $1\sigma$ .

A3.7.2 Several components of the overall expanded estimate of uncertainty are also expressed in the calibration verification test result of  $\pm 2.10\%$ . In order to isolate the component of uncertainty of the absorbed dose measurement of routine dosimetry system for conditions of use, other components expressed in the calibration verification test result need to be 'backed out' of the verification result. Using assigned values for components which have not been directly solved in the example:

- $u_{CF} = 0.75$  (curve fit)
  - $u_{Lab} = 0.60$  (uncertainty of the transfer standard temperature correction)
  - $u_{GP} = 0.60$  (positioning or dose gradients)
  - $u_{IN} = \text{unknown}$
- and components that have been solved in the example:

- $u_{CV} = 2.10$  (calibration verification test result)
- $u_{Re} = 0.81$  (Repeatability – Precision)
- $u_{TM} = 0.5$  (Calibration verification thickness)

gives the following:

$$\begin{aligned}
 u_{CV}^2 &= (\sqrt{u_{Re}^2 + u_{CF}^2 + u_{TM}^2})^2 + (\sqrt{u_{IN}^2 + u_{Lab}^2 + u_{GP}^2})^2 \\
 (2.10)^2 &= (\sqrt{(0.81)^2 + (0.75)^2 + (0.25)^2})^2 \\
 &\quad + (\sqrt{u_{IN}^2 + (0.60)^2})^2 + (0.60)^2 \\
 4.41 &= (0.6561 + 0.5625 + 0.0625) + U_{IN}^2 + 0.36 + 0.36 \\
 u_{IN}^2 &= 4.41 - 0.6561 - 0.5625 - 0.0625 - 0.36 - 0.36 \\
 u_{IN}^2 - 2.4089 &= 0 \\
 (u_{IN} + 1.552)(u_{IN} - 1.552) &= 0 \\
 u_{IN} \pm 1.552 &\approx 1.55 \%
 \end{aligned}
 \tag{A3.8}$$

Generally, this is the value that is used to assess whether the calibration has been successful (see 9.1.9, 9.2.9.1, A3.10 and Note 10)

A3.8 The overall estimate of uncertainty for the routine dosimetry system absorbed dose measurements for the conditions of use is prepared by quadrature summation of the individual components of uncertainty. Generally, the components at  $1\sigma$  are used for quadrature summation and then a coverage factor 'k' = 2 is applied. For the components of the example the quadrature summation of the overall estimate is:

$$\begin{aligned}
 u_{ov} &= \sqrt{\sum_{i=1}^n u_i^2} \\
 &= (u_{Dose}^2 + u_{Re}^2 + u_{CF}^2 + u_{TR}^2 + u_{IN}^2)^{\frac{1}{2}} \\
 &= (1.05^2 + 0.81^2 + 0.75^2 + 1.45^2 + 1.55^2)^{\frac{1}{2}} \\
 &= (6.8261)^{\frac{1}{2}} \\
 &= 2.61268
 \end{aligned}
 \tag{A3.9}$$

NOTE A3.2—The component  $u_{TR}^2$  is the thickness uncertainty associated with routine use where the assumption of the dosimeter thickness of 30.0  $\mu\text{m}$  is used. This component is the parallel component for dosimetry systems where instead of a thickness correction, a mass correction is used. The component  $u_{Dose}^2$  is the uncertainty associated with the calibration curve independent variable. In the case of a laboratory calibration method (9.1) it is the uncertainty associated with the routine dosimeter calibration

irradiation by the approved laboratory, 2.1 %. In the case of the in-situ calibration method (see 9.2) it is the uncertainty associated with the transfer standard used as the calibration curve independent variable, 2.4 %.

A3.8.1 Applying a coverage factor of  $k = 2$  gives an overall expanded uncertainty of:

$$U_{ex} = 2(u_{ov}) = 2(2.61268) = 5.225 = \pm 5.2 \% \text{ at } 2\sigma \tag{A3.10}$$

A3.9 Review of the components of the overall expanded uncertainty shows three components constitute the major contribution to the uncertainty of the absorbed dose measurement for the conditions of use:

$$\begin{aligned}
 u_{IN} &= 1.55 \\
 u_{TR} &= 1.45 \\
 u_{Dose} &= 1.05
 \end{aligned}$$

A3.9.1 Of these only the value for influence quantities of the conditions of use,  $u_{IN}^2$  and the assumption of a 30.0  $\mu\text{m}$  dosimeter thickness,  $u_{TM}^2$  can be optimized by the user to reduce the overall expanded uncertainty.

A3.10 Stability Verification is an evaluation conducted between the dosimetry system calibrations (9.1.11 and 9.2.9) to determine if changes have occurred that affect the calibration (6.4.1).

A3.10.1 In the case of the laboratory calibration method, the calibration verification test of A3.7 is repeated and the results of the stability verification are compared to the results of the calibration verification of A3.7. Several methods of evaluation can be used. Examples of some of these methods are a  $t$ -test for a difference of means or a one-factor ANOVA (7).

A3.10.2 In the case of an in-situ calibration method, the stability verification consists of repeating several of the calibration dose level irradiations and comparing these results to the initial calibration dose level irradiation results. Several methods of evaluation can be used. Examples of some of these methods are; a  $t$ -test for a difference of means or a one-factor ANOVA (7).

#### A4. MEASUREMENT TRACEABILITY CHAIN

A4.1 The measurement traceability chain is an unbroken set of measurement comparisons (calibrations) each having stated uncertainties whereby traceability of the dose measurement of the routine dosimetry system is established to a stated reference, usually a national or international standard.

A4.2 The selected calibration method establishes the routine dosimetry system dose measurement traceability by a specific set of measurement comparisons to the stated reference.

A4.2.1 The measurement traceability chain of a laboratory calibration method (9.1) is shown in Fig. A4.1.

A4.2.1.1 The key feature of the laboratory calibration method measurement traceability chain is that the calibration

curve is derived from irradiation of calibration samples under a single set of influence quantity values (conditions). Therefore, a calibration verification (9.1.8) is conducted to establish traceability of the routine dosimetry system dose measurement for the conditions of use. The calibration verification evaluation represents a critical component of uncertainty of the traceable routine dosimetry system dose measurement for the conditions of use.

A4.2.2 The measurement traceability chain of an in-situ/in-plant calibration method (9.2) is shown in Fig. A4.2.

A4.2.2.1 The key feature of the in-situ/in-plant calibration method measurement traceability chain is that the calibration curve is derived from irradiation of the calibration samples under the influence quantity values of the conditions of use.

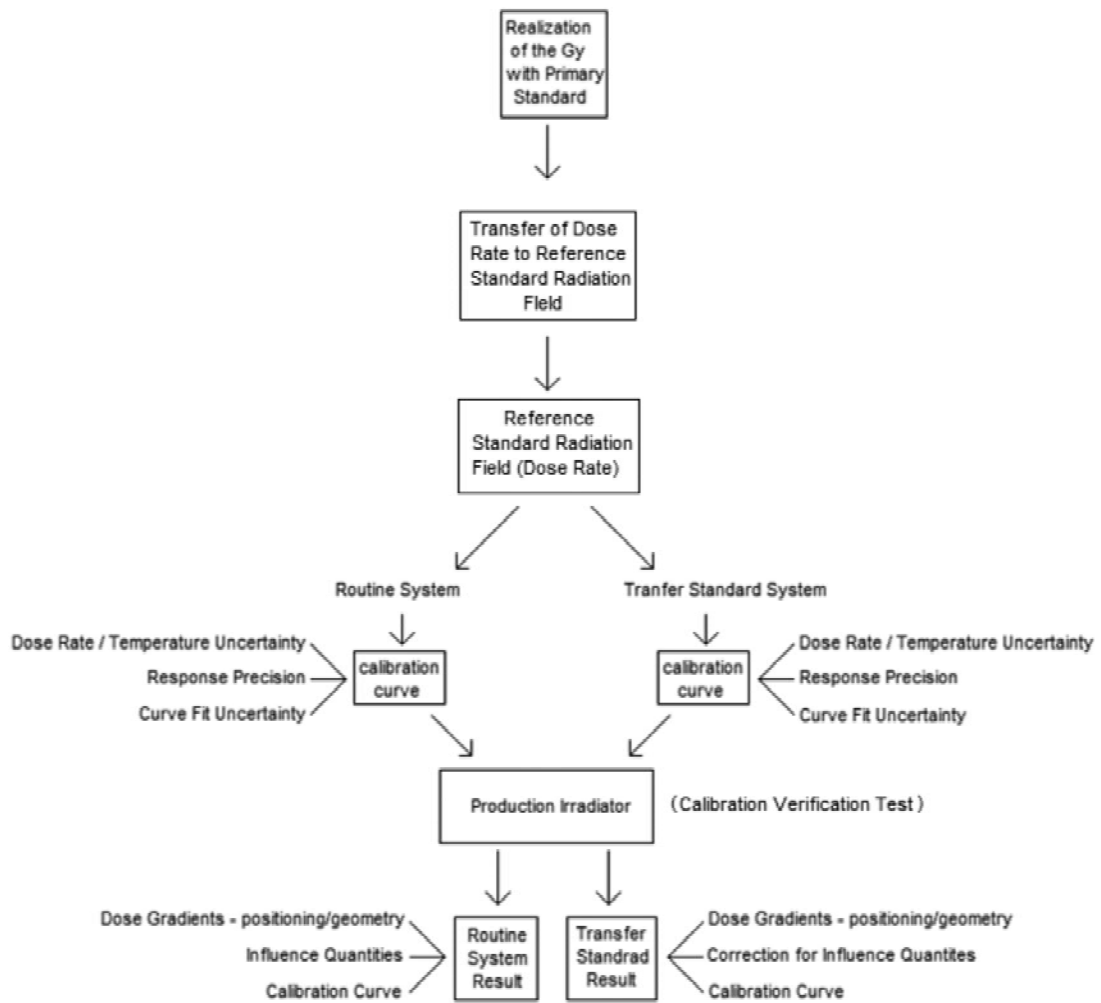


FIG. A4.1 Measurement traceability – laboratory calibration method

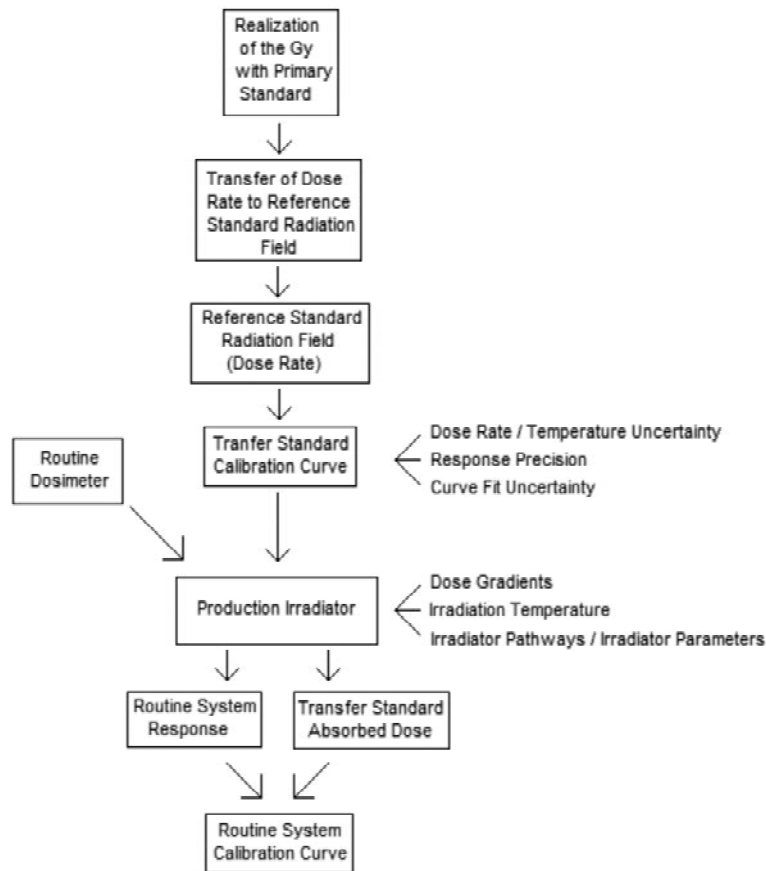


FIG. A4.2 Measurement traceability – in-situ calibration method

## Bibliography

- (1) United States Code of Federal Regulations, Title 21, Part 179.
- (2) "A Codex General Standard for Irradiated Foods and Recommended Code of Practice for the Operation of Radiation Facilities used for the Treatment of Foods" Joint FAO/WHO Food Standards Program, *Codex Alimentarius*, Vol XV, 1st ed., Food and Agriculture Organization; World Health Organization, Geneva, 1984.
- (3) United States Code of Federal Regulations, Title 21, 2, H, Part 820.
- (4) McLaughlin, W. L., Boyd, A. W., Chadwick, K. C., McDonald, P. C., and Miller, A., *Dosimetry for Radiation Processing*, Taylor and Francis Ltd., London, 1989.
- (5) Taylor, B.N. and Kuyatt, C.E., *Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results*, NIST Technical Note 1297, National Institute of Standards and Technology, Gaithersburg, MD, 1994.
- (6) Mandel, John, *The Statistical Analysis of Experimental Data*, Dover Publications, Inc., New York, 1964.
- (7) Crow, E., Davis, F., Maxfield, M., *Statistics Manual*, Dover Publications, inc., New York, 1960.

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