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## Standard Guide for Analysis of Calibration Data for Nuclear Instruments<sup>1</sup>

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### 1. Scope

1.1 This guide describes data analysis for efficiency calibrations of nuclear instruments using radioactive sources. It includes the calculation of the calibration parameters, evaluation and use of their uncertainties and covariances, and testing of the calibration data for outliers and overall lack of fit. It also provides guidelines for summarizing and reporting the results of a calibration.

1.2 The instrument counting efficiency is assumed to be independent of the radiation emission rate.

1.3 Guidance is provided for both single-point calibrations and calibration curves.

1.4 The guidance presumes the existence of measurement uncertainty models to provide statistical weighting factors for the calibration data.

1.5 This guide does not cover calibrations involving physically-based computer simulations.

1.6 The system of units for this guide is not specified. Dimensional quantities in the guide are presented only as illustrations of calculation methods. The examples are not binding on products or test methods treated.

1.7 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.8 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

### 2. Referenced Documents

#### 2.1 ASTM Standards:<sup>2</sup>

**D1129** Terminology Relating to Water  
**D7282** Practice for Setup, Calibration, and Quality Control of Instruments Used for Radioactivity Measurements  
**D7902** Terminology for Radiochemical Analyses  
**D8293** Guide for Evaluating and Expressing the Uncertainty of Radiochemical Measurements

#### 2.2 JCGM Documents:<sup>3</sup>

**GUM:JCGM 100:2008** Evaluation of measurement data—Guide to the expression of uncertainty in measurement  
**JCGM 102:2011** Evaluation of measurement data—Supplement 2 to the “Guide to the expression of uncertainty in measurement”—Extension to any number of quantities  
**VIM:JCGM 200:2008** International vocabulary of metrology—Basic and general concepts and associated terms (VIM)

### 3. Terminology

#### 3.1 Definitions:

3.1.1 For definitions of terms used in this practice, refer to Terminologies **D1129** and **D7902**, Practice **D7282**, **GUM:JCGM 100**, **JCGM 102**, and **VIM:JCGM 200**.

#### 3.2 Definitions of Terms Specific to This Standard:

3.2.1 *calibration curve, n*—functional model that calculates counting efficiency from the value of a predictor variable and one or more model parameters; also known as an *efficiency curve*.

3.2.1.1 *Discussion*—A calibration “curve” might be a linear or nonlinear function of the predictor variable.

3.2.2 *calibration parameter, n*—any of the parameters in a calibration model whose values are determined by a calibration

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard’s Document Summary page on the ASTM website.

<sup>3</sup> Available from [www.bipm.org](http://www.bipm.org).

and subsequently used together with observed values of the predictor variable to calculate counting efficiencies.

3.2.3 *calibration range*,  $n$ —interval between the least and greatest values of the predictor variable for which a calibration curve is considered valid.

3.2.4 *generalized weighting*,  $n$ —statistical weighting of data using both variances and covariances.

3.2.5 *relative residual* ( $\% \Delta_i$ ),  $n$ —quotient of a residual,  $e_i$ , and the corresponding predicted value,  $\hat{\epsilon}_i$ , typically expressed as a percentage.

3.2.6 *residual* ( $e_i$ ),  $n$ —difference,  $\epsilon_i - \hat{\epsilon}_i$ , between a measured value,  $\epsilon_i$ , and the corresponding predicted value,  $\hat{\epsilon}_i$ .

3.2.7 *simple weighting*,  $n$ —statistical weighting of data using variances but not covariances.

3.2.8 *single-point, adj*—relating to a calibration model in which the instrument counting efficiency is estimated by a single parameter with no predictor variable (a polynomial of degree zero).

3.2.9 *standardized residual* ( $\zeta_i$ ),  $n$ —quotient of a *residual*,  $e_i$ , and its combined standard uncertainty,  $u_c(e_i)$ .

### 3.3 Acronyms:

3.3.1 *GLS*—generalized least squares

3.3.2 *LLS*—linear least squares

3.3.3 *LOF*—lack of fit

3.3.4 *MQO*—measurement quality objective

3.3.5 *NLLS*—nonlinear least squares

3.3.6 *OLS*—ordinary least squares

3.3.7 *STS*—sample test source

3.3.8 *WCS*—working calibration source

3.3.9 *WLS*—weighted least squares

## 4. Summary of Guide

4.1 Calculation of an instrument counting efficiency requires a mathematical model described in terms of one or more *calibration parameters*. In a *single-point* efficiency model, there is one parameter, which equals the efficiency itself. In all other models there is a *calibration curve*, which calculates the efficiency from a *predictor variable*, such as gamma-ray energy, precipitate mass, or a quench indicator; and also one or more calibration parameters. An efficiency calibration measures the calibration parameter(s), providing their estimated values and uncertainties.

4.2 A calibration requires at least as many measurements as the number of model parameters. Whenever practical, the number of measurements should exceed the number of parameters, resulting in extra degrees of freedom that can be used to assess *lack of fit* (LOF). Additional measurements reduce the total uncertainty of the calibration, and the lack-of-fit test checks the consistency of the data with the efficiency and uncertainty models.

4.3 Three types of calibrations are considered:

4.3.1 A single-point calibration involving multiple measurements,

4.3.2 A calibration curve (or line), typically polynomial, determined by linear least squares (LLS), and

4.3.3 A calibration curve determined by nonlinear least squares (NLLS).

4.4 In each case, it is assumed that some number ( $n$ ) of efficiency measurements are made, producing measured efficiencies  $\epsilon_1, \epsilon_2, \dots, \epsilon_n$ . It is assumed that there is a valid uncertainty model for the measurements, providing an uncertainty for each  $\epsilon_i$  and an estimated covariance for each pair ( $\epsilon_i, \epsilon_j$ ). The parameter-fitting procedure statistically weights the measured efficiencies according to their estimated variances. Since using estimated variances instead of true variances for this purpose can bias the results, a two-stage procedure is employed, in which preliminary values for the calibration parameters are obtained first and used to refine the variance estimates and weights for the final fit.

4.5 Optimal weighting requires not only the variances,  $u_c^2(\epsilon_i)$ , but also the covariances,  $u(\epsilon_i, \epsilon_j)$ . Two options are discussed in each case for dealing with covariances. In each case, Option 1 (“simple weighting”) does not require explicit calculation of  $u(\epsilon_i, \epsilon_j)$ . Option 2 (“generalized weighting”) is the default option when Option 1 cannot be used.

4.6 Guidance is provided for calculating the calibration parameters, their uncertainties, and (when applicable) their correlation coefficients; for assessing LOF; and for calculating the counting efficiency and associated uncertainty for a subsequent sample test source (STS) measurement.

4.7 The emphasis of this guide is not on the details of the least-squares fitting algorithms. For more information, see Draper and Smith (1),<sup>4</sup> Marquardt (2), or Bevington and Robinson (3).

## 5. Significance and Use

5.1 The mathematical and statistical techniques described in this guide support implementation of the calibration requirements of Practice D7282 and the guidance for uncertainty analysis given in Guide D8293. The guidance is intended for use either by qualified specialists at a radioanalytical laboratory or by developers of software for calibration of nuclear instruments.

5.2 Applications for single-point calibrations might include:

5.2.1 Alpha-particle spectrometry,

5.2.2 Gas proportional counters used for thin sources with negligible attenuation, and

5.2.3 Gamma-ray spectrometers used for single nuclides.

5.3 Applications for calibration curves determined by LLS might include:

5.3.1 Mass attenuation curves for gas proportional counters (polynomial), and

5.3.2 Quench calibration curves for liquid scintillation counters (polynomial).

5.4 Applications for calibration curves determined by NLLS might include:

<sup>4</sup> The boldface numbers in parentheses refer to a list of references at the end of this standard.

5.4.1 Gamma-ray spectrometry across a range of gamma-ray energies,

5.4.2 Mass attenuation curves for gas proportional counters, and

5.4.3 Quench calibration curves for liquid scintillation counters.

5.5 Although this guide focuses on efficiency calibrations for nuclear instruments, the same general principles and paradigms should apply to other types of calibrations and to other instruments, as long as there are valid uncertainty models for the calibration data.

## 6. Overview

6.1 This section provides an overview of the recommended statistical procedures. Section 7 provides additional details for a single-point calibration. Section 8 describes procedures for fitting a calibration curve using linear least squares, and Section 9 describes procedures for fitting a calibration curve using nonlinear least squares. Section 10 provides instructions for assessing the fit in any of the three scenarios. Section 11 describes the content and organization of a calibration report.

| Topic                                     | Section     |
|-------------------------------------------|-------------|
| Single-point calibration                  | 7           |
| Calibration curve—Linear least squares    | 8           |
| Calibration curve—Nonlinear least squares | 9           |
| Assessing the fit                         | 10          |
| The calibration report                    | 11          |
| Estimating additional variability         | Appendix X1 |

6.2 A single-point calibration is used when the counting efficiency is modeled as a constant, with no predictor variable. In all other situations, a calibration curve is used, in which case the choice of least-squares fitting techniques (linear or nonlinear) is determined by whether the calibration model is linear or nonlinear in the calibration parameters.

6.3 For illustration, it is assumed that each measured efficiency  $\varepsilon_i$  is calculated with an equation such as:

$$\varepsilon_i = \frac{R_{Si} - R_{Bi}}{A_i \cdot I_i \cdot DF_i}, \quad i = 1, 2, \dots, n, \quad (1)$$

where:

- $i$  = index number of the calibration source,
- $R_{Si}$  = gross count rate,
- $R_{Bi}$  = background count rate,
- $A_i$  = activity of the calibration source at its reference date,
- $I_i$  = radiation emission probability, and
- $DF_i$  = decay factor.

NOTE 1—Eq 1 is only an example. Other equations are possible.

6.4 Given the efficiency equation, one uses uncertainty propagation to write equations for the combined variances  $u_c^2(\varepsilon_i)$  and covariances  $u(\varepsilon_i, \varepsilon_j)$ , as described in Guide D8293. For example, when using Eq 1,

$$u_c^2(\varepsilon_i) = \frac{R_{Si}^2/t_{Si} + R_{Bi}^2/t_{Bi}}{(A_i \cdot I_i \cdot DF_i)^2} + \varepsilon_i^2 \left( \frac{u^2(A_i)}{A_i^2} + \frac{u^2(I_i)}{I_i^2} + \varphi_{CS}^2 \right) \quad (2)$$

and:

$$u(\varepsilon_i, \varepsilon_j) = \frac{u(R_{Bi}, R_{Bj})}{(A_i \cdot I_i \cdot DF_i)(A_j \cdot I_j \cdot DF_j)} + \varepsilon_i \varepsilon_j \left( \frac{u(A_i, A_j)}{A_i \cdot A_j} + \frac{u(I_i, I_j)}{I_i \cdot I_j} \right) \quad (3)$$

where:

- $i, j$  = index numbers of two different calibration sources,
- $t_{Si}$  = source count time,
- $t_{Bi}$  = background count time, and
- $\varphi_{CS}$  = relative standard uncertainty due to variability of the calibration sources and possibly to model error.

6.4.1 This guide assumes a constant value for the factor  $\varphi_{CS}$ , which accounts for variations in counting geometry and imperfections of the mathematical model, although in principle its value might vary with that of the predictor variable.

6.5 In Eq 3,  $u(R_{Bi}, R_{Bj}) = u^2(R_B)$  if the same measured background  $R_B$  is used for both  $\varepsilon_i$  and  $\varepsilon_j$ . Otherwise,  $u(R_{Bi}, R_{Bj})$  may be 0.

6.6 If  $u(R_{Bi}, R_{Bj})$  is always zero or negligible, and if each of the relative covariances,  $u(A_i, A_j)/(A_i \cdot A_j)$  and  $u(I_i, I_j)/(I_i \cdot I_j)$ , is the same for all  $i \neq j$ , one may use Option 1, which requires only simple weights, not a full weighting matrix. For example, suppose  $I_i = I$  for each  $i$ , and each source activity  $A_i$  is calculated as:

$$A_i = AC \cdot V_i \cdot Y_i \quad (4)$$

where:

- $AC$  = activity concentration (or massic activity) of the calibration reference material,
- $V_i$  = volume (or mass) of the reference material used in the source, and
- $Y_i$  = chemical yield (for a working calibration source, WCS).

If all the yields,  $Y_i$ , are determined using the same yield tracer or carrier solution, with activity or mass concentration,  $c_T$ , and if  $u(V_i, V_j) \approx 0$  and  $u(R_{Bi}, R_{Bj}) \approx 0$ , then:

$$\frac{u(A_i, A_j)}{A_i \cdot A_j} = \frac{u^2(AC)}{AC^2} + \frac{u^2(c_T)}{c_T^2} \quad (5)$$

$$\frac{u(V_i, V_j)}{V_i \cdot V_j} \approx 0 \quad (6)$$

$$\frac{u(I_i, I_j)}{I_i \cdot I_j} = \frac{u^2(I)}{I^2} \quad (7)$$

Since all the relative covariance terms are equal, Option 1 may be used. In this case,  $\varphi_e^2$  will denote the sum of the shared relative variance/covariance components. For example,

$$\varphi_e^2 = \frac{u^2(AC)}{AC^2} + \frac{u^2(c_T)}{c_T^2} + \frac{u^2(I)}{I^2} \quad (8)$$

Note that  $u(\varepsilon_i, \varepsilon_j) = \varepsilon_i \varepsilon_j \varphi_e^2$  for  $i \neq j$ .

6.7 It might also be possible to decompose the variances  $u^2(V_i)$  into components due to random and fixed effects and to

include the latter component in  $\varphi_{\varepsilon}^2$ .

6.8 Option 2 should be used if the conditions required for Option 1 are not met. Option 2 statistically weights the data using the full covariance matrix of the measured efficiencies.

#### 6.9 Option 1—Simple Weighting without Covariances:

6.9.1 Option 1 uses partial variance estimates, denoted by  $u_{cp}^2(\varepsilon_i)$ , to calculate weighting factors. The relative variance terms that contribute to  $\varphi_{\varepsilon}^2$  above are omitted from  $u_{cp}^2(\varepsilon_i)$ . So,

$$u_{cp}^2(\varepsilon_i) = u^2(\varepsilon_i) - \varepsilon_i^2 \varphi_{\varepsilon}^2 \quad (9)$$

For example, if Eq 8 is used, then:

$$u_{cp}^2(\varepsilon_i) = \frac{R_{Si}/t_{Si} + R_{Bi}/t_{Bi}}{(A_i \cdot I \cdot DF_i)^2} + \varepsilon_i^2 \left( \frac{u_{cp}^2(A_i)}{A_i^2} + \varphi_{CS}^2 \right) \quad (10)$$

where  $u_{cp}^2(A_i)$  denotes a partial variance of  $A_i$  calculated without the relative variance terms  $u^2(AC)/AC^2$  and  $u^2(c_T)/c_T^2$  from Eq 5.

6.9.2 The preliminary weights  $\tilde{w}_1, \tilde{w}_2, \dots, \tilde{w}_n$  are given by:

$$\tilde{w}_i = \frac{1}{u_{cp}^2(\varepsilon_i)} \quad (11)$$

These weights are used for the preliminary fit as described below for each calibration scenario. For calculations with matrices, the weights are used to construct the preliminary weighting matrix,  $\tilde{\mathbf{W}}$ .

$$\tilde{\mathbf{W}} = \text{diag}(\tilde{w}_1, \tilde{w}_2, \dots, \tilde{w}_n) = \begin{pmatrix} \tilde{w}_1 & 0 & \dots & 0 \\ 0 & \tilde{w}_2 & \dots & 0 \\ \vdots & \vdots & \ddots & \vdots \\ 0 & 0 & \dots & \tilde{w}_n \end{pmatrix} \quad (12)$$

NOTE 2—Throughout this guide, a tilde (~) over a symbol denotes a preliminary estimate or value, which will be recalculated and replaced later.

6.9.3 Preliminary estimates of the calibration parameters,  $\tilde{\beta}_j$ , are found and used to calculate preliminary values for the predicted efficiencies,  $\tilde{\varepsilon}_i$ . These values are then used to refine the variance estimates,  $u_{*}^2(\varepsilon_i)$ . For example,

$$u_{*}^2(\varepsilon_i) = \frac{\tilde{\varepsilon}_i \cdot A_i \cdot I \cdot DF_i / t_{Si} + R_{Bi}(1/t_{Si} + 1/t_{Bi})}{(A_i \cdot I \cdot DF_i)^2} + \tilde{\varepsilon}_i^2 \left( \frac{u_{cp}^2(A_i)}{A_i^2} + \varphi_{CS}^2 \right) \quad (13)$$

6.9.4 The refined weights  $w_1, w_2, \dots, w_n$  are then given by:

$$w_i = \frac{1}{u_{*}^2(\varepsilon_i)} \quad (14)$$

These weights are used for the final fit as described below for each scenario. For calculations with matrices, the weights are used to construct the final weighting matrix,  $\mathbf{W}$ .

$$\mathbf{W} = \text{diag}(w_1, w_2, \dots, w_n) = \begin{pmatrix} w_1 & 0 & \dots & 0 \\ 0 & w_2 & \dots & 0 \\ \vdots & \vdots & \ddots & \vdots \\ 0 & 0 & \dots & w_n \end{pmatrix} \quad (15)$$

6.9.5 After the final fit is obtained, the predicted efficiencies,  $\hat{\varepsilon}_i$ , and their (partial) variances,  $u_{*}^2(\hat{\varepsilon}_i)$ , are calculated. The fit is assessed, and an outlier test might be performed. If the results are acceptable, the total combined standard uncertainties and

covariances of the calibration parameters are calculated.

#### 6.10 Option 2—Generalized Weighting with Covariance Matrix:

6.10.1 Uncertainty propagation is used to estimate the total combined variance,  $u_{\varepsilon}^2(\varepsilon_i)$ , and covariance  $u(\varepsilon_i, \varepsilon_j)$ , as described in 6.4. See Eq 2 and Eq 3 for examples.

6.10.2 The preliminary measurement covariance matrix  $\tilde{\mathbf{U}}_{\varepsilon}$  is constructed and inverted to obtain the preliminary weighting matrix,  $\tilde{\mathbf{W}}$ .

$$\tilde{\mathbf{W}} = \tilde{\mathbf{U}}_{\varepsilon}^{-1} \quad (16)$$

where:

$$\tilde{\mathbf{U}}_{\varepsilon} = \begin{pmatrix} u_{\varepsilon}^2(\varepsilon_1) & u(\varepsilon_1, \varepsilon_2) & \dots & u(\varepsilon_1, \varepsilon_n) \\ u(\varepsilon_2, \varepsilon_1) & u_{\varepsilon}^2(\varepsilon_2) & \dots & u(\varepsilon_2, \varepsilon_n) \\ \vdots & \vdots & \ddots & \vdots \\ u(\varepsilon_n, \varepsilon_1) & u(\varepsilon_n, \varepsilon_2) & \dots & u_{\varepsilon}^2(\varepsilon_n) \end{pmatrix} \quad (17)$$

6.10.3 Preliminary estimates of the calibration parameters,  $\tilde{\beta}_j$ , are found and used to calculate preliminary values for the predicted efficiencies,  $\tilde{\varepsilon}_i$ . These values are then used to refine the estimates of variance,  $u_{*}^2(\varepsilon_i)$ , and covariance,  $u_{*}(\varepsilon_i, \varepsilon_j)$ . For example,

$$u_{*}^2(\varepsilon_i) = \frac{\tilde{\varepsilon}_i \cdot A_i \cdot I_i \cdot DF_i / t_{Si} + R_{Bi}(1/t_{Si} + 1/t_{Bi})}{(A_i \cdot I_i \cdot DF_i)^2} + \tilde{\varepsilon}_i^2 \left( \frac{u^2(A_i)}{A_i^2} + \frac{u^2(I_i)}{I_i^2} + \varphi_{CS}^2 \right) \quad (18)$$

and:

$$u_{*}(\varepsilon_i, \varepsilon_j) = \frac{u(R_{Bi}, R_{Bj})}{(A_i \cdot I_i \cdot DF_i)(A_j \cdot I_j \cdot DF_j)} + \tilde{\varepsilon}_i \tilde{\varepsilon}_j \left( \frac{u(A_i, A_j)}{A_i A_j} + \frac{u(I_i, I_j)}{I_i I_j} \right) \quad (19)$$

6.10.4 The refined covariance matrix  $\mathbf{U}_{\varepsilon}^{*}$  is constructed and inverted to obtain the refined weighting matrix,  $\mathbf{W}$ .

$$\mathbf{W} = \mathbf{U}_{\varepsilon}^{*-1} \quad (20)$$

where:

$$\mathbf{U}_{\varepsilon}^{*} = \begin{pmatrix} u_{*}^2(\varepsilon_1) & u_{*}(\varepsilon_1, \varepsilon_2) & \dots & u_{*}(\varepsilon_1, \varepsilon_n) \\ u_{*}(\varepsilon_2, \varepsilon_1) & u_{*}^2(\varepsilon_2) & \dots & u_{*}(\varepsilon_2, \varepsilon_n) \\ \vdots & \vdots & \ddots & \vdots \\ u_{*}(\varepsilon_n, \varepsilon_1) & u_{*}(\varepsilon_n, \varepsilon_2) & \dots & u_{*}^2(\varepsilon_n) \end{pmatrix} \quad (21)$$

The weighting matrix  $\mathbf{W}$  is used for the final fit as described below for each scenario.

6.10.5 After the final fit is obtained, the predicted efficiencies,  $\hat{\varepsilon}_i$ , and their variances,  $u_{*}^2(\hat{\varepsilon}_i)$ , are calculated. The fit is assessed and an outlier test might be performed. If the results are acceptable, the total combined standard uncertainties and covariances of the calibration parameters are calculated.

#### 6.11 Both Options:

6.11.1 For a multi-parameter calibration curve, the correlation coefficient for each unordered pair of calibration parameters is calculated.

6.11.2 The efficiency,  $\varepsilon_{STS}$ , for a subsequent sample test source measurement and its combined standard uncertainty,  $u_c(\varepsilon_{STS})$ , are calculated. The uncertainty includes a component  $\varphi_{STS} \cdot \varepsilon_{STS}$  accounting for the variability of sample test sources, and in the case of a calibration curve,  $u_{*}^2(\hat{\varepsilon}_i)$ .

## 7. Single-Point Calibration—Average Value

7.1 Assume a single-point efficiency is measured  $n$  times ( $n > 1$ ). Let the measured efficiencies be denoted by  $\varepsilon_1, \varepsilon_2, \dots, \varepsilon_n$ . The calibration parameter will be calculated as a weighted or simple average of these values. See Section 6 to choose Option 1 or 2.

### 7.2 Option 1—Weighted Average without Covariances:

7.2.1 Use uncertainty propagation to estimate a partial variance  $u_{cp}^2(\varepsilon_i)$  for each measured efficiency, as described in 6.9.1. See Eq 10 for example.

7.2.2 Calculate the preliminary estimate of the efficiency,  $\bar{\varepsilon}$ , as a weighted average.

$$\bar{\varepsilon} = \frac{\sum_{i=1}^n \frac{\varepsilon_i}{u_{cp}^2(\varepsilon_i)}}{\sum_{i=1}^n \frac{1}{u_{cp}^2(\varepsilon_i)}} \quad (22)$$

Alternatively, and especially if all the partial variances are roughly equal, use the arithmetic mean,  $\bar{\varepsilon}$ , for the preliminary estimate.

$$\bar{\varepsilon} = \bar{\varepsilon} = \frac{1}{n} \sum_{i=1}^n \varepsilon_i \quad (23)$$

The predicted efficiency  $\bar{\varepsilon}_i$  has the same value,  $\bar{\varepsilon}$ , for each  $i$ .

7.2.3 Calculate a refined estimate of each variance,  $u_{*}^2(\varepsilon_i)$ , as described in 6.9.3. See Eq 13 for example.

7.2.4 Calculate the final efficiency estimate as a weighted average,  $\hat{\varepsilon}$ .

$$\hat{\varepsilon} = \frac{\sum_{i=1}^n \frac{\varepsilon_i}{u_{*}^2(\varepsilon_i)}}{\sum_{i=1}^n \frac{1}{u_{*}^2(\varepsilon_i)}} \quad (24)$$

The predicted efficiency  $\hat{\varepsilon}_i$  has the same value,  $\hat{\varepsilon}$ , for each  $i$ .

7.2.5 Calculate its variance.

$$u_{*}^2(\hat{\varepsilon}) = \left( \sum_{i=1}^n \frac{1}{u_{*}^2(\varepsilon_i)} \right)^{-1} \quad (25)$$

7.2.6 Assess the fit as described in Section 10 (with  $m = 1$  and with each  $\hat{\varepsilon}_i = \hat{\varepsilon}$ ).

7.2.7 Calculate the combined standard uncertainty of  $\hat{\varepsilon}$  as:

$$u_c(\hat{\varepsilon}) = \sqrt{u_{*}^2(\hat{\varepsilon}) + \hat{\varepsilon}^2 \phi_{\varepsilon}^2} \quad (26)$$

where  $\phi_{\varepsilon}^2$  is the sum of the previously omitted relative variance terms described in 6.6 and 6.9.1.

7.2.8 Continue at 7.4.

### 7.3 Option 2—Generalized Weighted Average with Covariance Matrix:

7.3.1 Use uncertainty propagation to estimate the total combined variances,  $u_{*}^2(\varepsilon_i)$ , and covariances,  $u(\varepsilon_i, \varepsilon_j)$ , as described in 6.4. See Eq 2 and Eq 3 for examples.

7.3.2 Construct the preliminary measurement covariance matrix,  $\tilde{U}_{\varepsilon}$ , and calculate the preliminary weighting matrix,  $\tilde{W}$ , as described in 6.10.2. See Eq 16 and Eq 17.

7.3.3 Calculate the preliminary efficiency estimate as a generalized weighted average,  $\bar{\varepsilon}$ .

$$\bar{\varepsilon} = \frac{\sum_{i=1}^n \varepsilon_i \sum_{j=1}^n \tilde{W}_{ij}}{\sum_{i=1}^n \sum_{j=1}^n \tilde{W}_{ij}} \quad (27)$$

where  $\tilde{W}_{ij}$  denotes the  $ij^{\text{th}}$  entry of  $\tilde{W}$ . Alternatively, use the arithmetic mean  $\bar{\varepsilon} = \bar{\varepsilon}$ . See Eq 23.

7.3.4 Calculate refined estimates of the variances,  $u_{*}^2(\varepsilon_i)$  and covariances,  $u_{*}(\varepsilon_i, \varepsilon_j)$ , as described in 6.10.3 (with each  $\varepsilon_i = \bar{\varepsilon}$ ). See Eq 18 and Eq 19 for examples.

7.3.5 Construct the refined covariance matrix,  $U_{*}^*$ , and weighting matrix,  $W$ , as described in 6.10.4. See Eq 20 and Eq 21.

7.3.6 Calculate the final efficiency estimate as a generalized weighted average,  $\hat{\varepsilon}$ :

$$\hat{\varepsilon} = \frac{\sum_{i=1}^n \varepsilon_i \sum_{j=1}^n W_{ij}}{\sum_{i=1}^n \sum_{j=1}^n W_{ij}} \quad (28)$$

where  $W_{ij}$  denotes the  $ij^{\text{th}}$  entry of  $W$ .

7.3.7 Calculate the estimated variance of the weighted average  $\hat{\varepsilon}$ .

$$u_{*}^2(\hat{\varepsilon}) = \left( \sum_{i=1}^n \sum_{j=1}^n W_{ij} \right)^{-1} \quad (29)$$

7.3.8 Assess the fit as described in Section 10 (with  $m = 1$  and with each  $\hat{\varepsilon}_i = \hat{\varepsilon}$ ).

7.3.9 Calculate the combined standard uncertainty of  $\hat{\varepsilon}$ .

$$u_c(\hat{\varepsilon}) = \sqrt{u_{*}^2(\hat{\varepsilon})} \quad (30)$$

### 7.4 Both Options—Single-Point Calibration:

7.4.1 The laboratory may establish an upper limit for the acceptable relative combined standard uncertainty,  $u_c(\hat{\varepsilon})/\hat{\varepsilon}$ , based on the uncertainty requirements of the measurement method or other measurement quality objectives (MQOs).

7.4.2 Record the estimated efficiency,  $\hat{\varepsilon}$ , and its combined standard uncertainty,  $u_c(\hat{\varepsilon})$ .

7.4.3 The efficiency for a subsequent measurement of a sample test source,  $\varepsilon_{\text{STS}}$ , is estimated by:

$$\varepsilon_{\text{STS}} = \hat{\varepsilon} \quad (31)$$

and its combined standard uncertainty is:

$$u_c(\varepsilon_{\text{STS}}) = \sqrt{u_c^2(\hat{\varepsilon}) + \hat{\varepsilon}^2 \phi_{\text{STS}}^2} \quad (32)$$

where:

$\phi_{\text{STS}}$  = relative standard uncertainty due to variations among individual sample test sources.

The relative uncertainty component  $\phi_{\text{STS}}$  may equal  $\phi_{\text{CS}}$ , especially when the calibration is performed with working calibration sources (WCSs) prepared in the laboratory.

## 8. Calibration Curve—Linear Least Squares

8.1 Assume the calibration model has the functional form:

$$\varepsilon = \beta_1 X_1 + \beta_2 X_2 + \dots + \beta_m X_m + \beta_{m+1}$$