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Standard Guide for Analysis of Calibration Data for Nuclear Instruments[1]

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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:*[2]

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:*[3]

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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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[3] Available from 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, $\hat{\varepsilon}_i$, typically expressed as a percentage.

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

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 (ζ_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 ε_1, ε_2, ..., ε_n. It is assumed that there is a valid uncertainty model for the measurements, providing an uncertainty for each ε_i and an estimated covariance for each pair (ε_i, ε_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, $u_c^2(\varepsilon_i)$, but also the covariances, $u(\varepsilon_i,\varepsilon_j)$. Two options are discussed in each case for dealing with covariances. In each case, Option 1 ("simple weighting") does not require explicit calculation of $u(\varepsilon_i,\varepsilon_j)$. 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**),[4] 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:

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[4] The boldface numbers in parentheses refer to a list of references at the end of this standard.