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Abstract. We determined uncertainties for WDS-EPMA (wavelength-dispersive X-ray spectroscopy - electron probe microanalysis) data using the globally accepted ISO/GUM (International Standards Organization/Guide to the Expression of Uncertainty in Measurement). For each calculation, such as the current drift correction and deadtime correction that precede the calculation of a k-value (net corrected X-ray counts of unknown/net corrected X-ray counts of standard), uncertainties were calculated from contributing factors and combined until a final combined standard uncertainty for the *k*-value was calculated. Our example used data from the analysis of the Ge Lα X-ray line in a SiGe alloy. Additional contributions to uncertainties in EPMA results, such as the matrix correction procedure and mass absorption coefficients (MACs) are considered. All statistical calculations used in the process of arriving at the combined uncertainty are included, and the basic steps of the ISO/GUM are described.

1. Introduction

In this report, the ISO/GUM is adapted to EPMA data. It provides the basic steps and statistics for determining uncertainties from the equations used in the EPMA community. Hopefully this is a first step in providing a universal system that can be used for reporting EPMA uncertainties.

Since the development of the electron microprobe was reported by Castaing in 1949 there have been few publications devoted specifically to uncertainties in EPMA. An early comparison by Poole of the calculated compositions from 229 binary alloys, many with well-known compositions, using early matrix correction procedures demonstrated that the deviation in the calculated composition from the accepted values could vary by as much as 18 % relative [1]. A derivation of the variance of the mean for *k*-values calculated from wavelength-dispersive X-ray spectrometer (WDS) data was reported by Ziebold [2]. This derivation applied only to the counting statistics in the analysis and it can also be used for energy-dispersive X-ray spectroscopy (EDS) data as demonstrated more recently Lifshin et al. [3, 4]. Both reports applied the results to the determination of the minimum detectable limit (MDL) for an element and to the determination of the optimum X-ray count acquisition time needed to achieve a desired precision for an element. A rigorous application of Poisson counting statistics was described and applied to microanalysis by Ancey [5]. Again this was applied to the MDL and optimum acquisition time for an element. The difficult task of sorting out uncertainties

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associated with the individual ZAF (atomic number-absorption-fluorescence) matrix correction factors has also been reported by Heinrich [6].

This report describes the evaluation of uncertainties in electron probe microanalysis according to currently acceptable practices in the scientific community. It includes guidelines and procedures from several references that are recommended reading for anyone involved in expressing uncertainties for EPMA data. The data and equations used here are from quantitative WDS-EPMA but can be used for quantitative EDS analysis from k-values extracted from multiple linear least squares fitting procedures.

2. Accuracy and precision

A distinction between the terms accuracy and precision must be made. These terms must not be used interchangeably. Accuracy is a measure of correctness or how close a result or measurand may be to the "true value." Precision is a measure of statistical repeatability or reproducibility. You cannot have good accuracy without good precision but you can have good precision without good accuracy. An enlightening and entertaining discussion by Eisenhart on accuracy and precision is available on the web in a National Bureau of Standards (NBS), former name of the National Institute of Standards and Technology (NIST), Special Publication [7].

3. Uncertainties

Before discussing how to evaluate uncertainties in EPMA, it is important to understand what uncertainty is and its importance in today's global economy. According to the ISO/GUM an uncertainty is a "parameter, associated with the results of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand" [8]. It can be reported in either absolute units or relative terms but should always be accompanied by a very clear explanation of how the uncertainty value was determined, including a description of all contributing factors. John Lyons, a former NIST director stated the need for clear uncertainty statements when he wrote the following in the Foreword to the NIST Technical Note, *Guidelines for evaluating and expressing the uncertainty of NIST measurement results* [9]:

"Results of measurements and conclusions derived from them constitute much of the technical information produced by NIST. It is generally agreed that the usefulness of measurement results, and thus much of the information that we provide as an institution, is to a large extent determined by the quality of the statements of uncertainty that accompany them. … Only if quantitative and thoroughly documented statements of uncertainty accompany the results of NIST calibrations can the users of our calibration services establish their level of traceability to the U.S. standards of measurement maintained at NIST."

The need for clear statements of the uncertainties assigned to reported values was also discussed in 1968, again by C. Eisenhart [10]. The statement above is not just applicable to NIST but also to any laboratory, especially to those issuing standards or experimental results that will be used by others. The ISO and NIST publications cited above were the result of a global perceived need to evaluate and express uncertainties assigned to standards using a single universally acceptable approach. The ISO publication was a cooperative effort of several international organisations, including the BIPM (International Bureau of Weights and Measures), IEC (International Electrotechnical Commission), IFCC (International Federations of Clinical Chemistry), ISO, IUPAC (International Union of Pure and Applied Chemistry), and OIML (International Organization of Legal Metrology). Jointly they adopted a provisional standard developed by CIPM (International Committee for Weights and Measures) that we refer to as ISO/GUM. The NIST Technical Note is in full agreement with the ISO Guide but is a more concise version. For any laboratory reporting data with uncertainties, these publications are a definite necessity. They provide a set of statistical terms and concepts with precise definitions and practical guidance. Another highly recommended publication because of its clarity and excellent examples is the EURACHEM/CITAC Guide [11] that is available on the web at no cost. A more recent publication on uncertainties is also recommended [12].

According to the ISO/GUM a measurement is the approximation of a measurand that is only complete with a quantitative statement of uncertainty. This uncertainty is a combination of several individual uncertainty contributions that are classified as Type A or Type B.

Type A includes statistical evaluations of a series of measurements, such as
- a series of independent measurements from which a standard deviation of the mean is calculated,
- parameters calculated for a curve from a set of data with a least squares fit from which a set of standard deviations are calculated, and
- analysis of variance (ANOVA) results to identify random effects in some types of measurements.

Type B includes other methods of evaluation, such as
- scientific judgment based on previous knowledge and understanding of relevant materials and instruments,
- previous measurement data,
- data provided in reports and calibrations,
- uncertainties assigned to reference data, and
- manufacturer's specifications.

Each Type A component of uncertainty that contributes to the uncertainty of a measurement result, or what is called a combined standard uncertainty, represented by σ_c, is referred to as a standard uncertainty. It is the same as an estimated standard deviation, σ_i, equal to the positive square root of the corresponding estimated variance, σ_j^2. Likewise, the standard uncertainty, or estimated standard deviation for a Type B component of uncertainty is referred to as σ_j, equal to the positive square root of the corresponding estimated variance, σ_j^2. Random and systematic effects can fall into either type of evaluation. The ISO/GUM discusses random and systematic effects and ways to reduce them to minimize uncertainties, and it assumes that every effort is made to identify and correct for all systematic effects in an analysis before calculating the combined standard uncertainty of a measurement.

The combined standard uncertainty, σ_C, is the positive square root of the combined variance, σ_C^2, determined from both Type A and Type B standard uncertainty contributions with propagation of error or RSS (root-sum-of-squares):

$$\sigma_c^2(y) \approx \sum_{i=1}^{N}\left(\frac{\partial f}{\partial x_i}\right)^2 \sigma^2(x_i) + 2\sum_{i=1}^{m-1}\sum_{j=i+1}^{m}\frac{\partial f}{\partial x_i}\frac{\partial f}{\partial x_j}\sigma(x_i, x_j). \qquad (1)$$

Here the measurand, y, is determined from N other quantities $x_1, x_2, ..., x_N$:

$$y = f(x_1, x_2, ..., x_N). \qquad (2)$$

Equation (1) above is based on a Taylor series approximation and is referred to as the law of propagation of uncertainty. The factors and terms that appear are

$\dfrac{\partial f}{\partial x_i}$, the sensitivity coefficients,

$\sigma(x_i)$, the standard uncertainty of x_i , and

$\sigma(x_i, x_j)$, the covariance of x_i and x_j .

The second term of equation (1) is the sum of the covariances between x_i and x_j and is sometimes small enough to be excluded, but should be determined when possible.

Explicit formulas for the first and second terms of equation (1) above for many specific functional forms of the equation used to determine y, can be readily found in some publications [11] and thus alleviate the need to calculate the partial derivatives. Today partial derivatives of complicated functional expressions can be readily calculated with the use of symbolic calculation facilities of software like MAPLE or MATHEMATICA [4].

For EPMA quantification we are first interested in the combined standard uncertainty for the *k*-value. This means that a combined standard uncertainty will have to be calculated for the deadtime correction, the current drift correction, and the *k*-value determination. The uncertainty associated with the matrix correction procedure will be dealt with later.

For a function that is determined from an addition (or subtraction) where variables x_1 and x_2 are independent and random, such as in equation (3) below,

$$y = a \cdot x_1 + b \cdot x_2 , \tag{3}$$

with sensitivity coefficients a and b ,

$$\frac{\partial y}{\partial x_1} = a \qquad \frac{\partial y}{\partial x_2} = b , \tag{4}$$

the standard uncertainties are summed in quadrature as in equation (5) below to obtain the combined standard variance for y,

$$\sigma_c^2(y) = a^2 \cdot \sigma^2(x_1) + b^2 \cdot \sigma^2(x_2) . \tag{5}$$

For a function determined by multiplication or division of independent or uncorrelated random variables, the relative standard uncertainties are used to obtain the combined standard uncertainty. So, for a function such as

$$y = a \cdot x_1 \cdot x_2 , \text{ and } \frac{\partial y}{\partial x_1} = a \cdot x_2 \quad , \frac{\partial y}{\partial x_2} = a \cdot x_1 \quad . \tag{6}$$

The combined relative variance will be the sum of the relative standard uncertainties added in quadrature according to equation (7) below.

$$\frac{\sigma_C^2(y)}{y^2} = \frac{\sigma^2(x_1)}{x_1^2} + \frac{\sigma^2(x_2)}{x_2^2}$$

(7)

For more complicated equations where y and x are not correlated, combinations of the two procedures can often be used to calculate the combined standard uncertainty.

4. Combined standard uncertainty for a *k*-value

We will proceed stepwise through the calculations used to obtain the *k*-value in a WDS-EPMA quantification to determine the combined standard uncertainty for the *k*-value of a single element. Equation (8) is used to correct the deadtime of the observed or measured count rate (counts/s), I', obtained with a WDS detector.

$$I = \frac{I'}{(1 - \tau I')},$$

(8)

where I is the deadtime corrected count rate in counts/s and τ is the deadtime constant in seconds. Note that this equation should be used with the count rate, not the raw counts. Since the count rate I is proportional to the beam current, i,

$$I = ri,$$

(9)

equation (8) can be written as

$$\frac{I'}{i} = r(1 - \tau I').$$

(10)

This equation is used to determine the deadtime by plotting $\frac{I'}{i}$ in counts/s/nA versus I' in counts/s resulting in a straight line plot with intercept r on the $\frac{I'}{i}$ axis and slope of $(-r\tau)$ so that τ is estimated as minus the ratio of slope to intercept. We use a linear least squares fit to such data to determine the deadtime and the variance. X-ray count data taken from a Cr Kα peak with a PET (pentaerythritol) crystal at currents ranging from 50 to 600 nA were fit to a straight line with a least squares linear fit. The deadtime, τ, was determined to be 2.098 μs with a single standard deviation of 0.087 μs. We will use this uncertainty for the deadtime when we later calculate a combined uncertainty for the current corrections. But there is the question of whether the component of uncertainty attributable to the covariance between the slope of the fitted line and intercept is large enough to merit incorporation in the variance determination. To do this we use the propagation of error formula [13], Table 1, page 337 for a function form $\frac{\overline{x}}{\overline{y}}$. The second order correction term can be estimated by

$$\left(\frac{\overline{x}}{\overline{y}}\right)^2 \left(-2\frac{s_{\overline{x}\overline{y}}}{xy}\right)$$

(11)

where $S_{\overline{xy}}$ is the covariance of x and y.   The correction is -0.0025, which is only about 0.1 % relative to the deadtime.

We will use the deadtime calculated above and its uncertainty in a propagation of uncertainties for Ge Lα in the WDS-EPMA quantification of a SiGe bulk specimen. The analysis was done at 15 kV, 30 nA beam current with a TAP (thallium acid phthalate) crystal, and a point beam using a Jeol 8600 electron microprobe. A polished slice taken from the SiGe boule was mounted on carbon tape. The expanded (k = 2, 95 % confidence)[5] combined heterogeneity uncertainty for Ge in the SiGe alloy was determined to be 0.92 % mass fraction [14]. Ten points were sampled for 40 s each, and beam current readings were acquired after sampling each point. Backgrounds for each analyzed point were taken from both sides of the Ge Lα peak. All count data reported here for the X-ray peak and background are the average of the 10 readings The X-ray counts acquired from the 10 points were first corrected for changes in the beam current during the analysis using the equation

$$ I_C = I' \times \frac{i_{Sc}}{i_{obs}} \tag{12} $$

where I_C is the average or raw X-ray counts corrected for beam current drift, I' is the measured X-ray counts at the observed current, i_{obs}, and i_{Sc} is the nominal value chosen to correct all X-ray data readings to in the data set. The nominal value can be a rounded number in the range of the observed current readings or it can be the first current reading in the data set. The calculated value for I_C is 138,927 cts in 40 s.

Since equation (12) uses only multiplication and division, the relative uncertainties of the mean are added in quadrature.

$$ \sigma_{I_C} = I_C \left(\frac{\sigma_{i_{obs}}^2}{(i_{obs})^2} + \frac{\sigma_{I'}^2}{(I')^2} \right)^{1/2} \tag{13} $$

where σ_{I_C} is the combined standard uncertainty of the mean for the X-ray counts corrected for beam drift, $\sigma_{i_{obs}}$ is the standard uncertainty of the mean for the observed current, and $\sigma_{I'}$ is the standard uncertainty of the mean for the observed X-ray counts. For the SiGe alloy the average for the 10 values of I' for Ge was 138953 cts in 40 s, with a standard uncertainty of the mean, $\sigma_{I'}$ equal to 210 cts in 40 s. The average observed current, i_{obs}, for the 10 readings was 30.535 nA with a standard uncertainty of the mean of 0.002 nA. Substitution into equation (12) and (13) results in the corrected counts of I_C = 138927 cts in 40s with a standard uncertainty of the mean of σ_{I_C} = 210 cts in 40s. This value is very close to the standard uncertainty of the mean for the observed current. The contribution from the current drift to the uncertainty is minimal and probably could have been ignored.

The evaluation of the combined standard uncertainty from the current drift and deadtime can be calculated with the equation

$$ \sigma_{CDT} = \left(\left[\frac{(I_C)^2}{(1 - \tau(I_C))^2} \right]^2 \sigma_\tau^2 + \left[\frac{1}{(1 - \tau(I_C))^2} \right]^2 \sigma_{I_C}^2 \right)^{1/2} , \tag{14} $$

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[5] The use of *k* here is an ISO/GUM coverage factor used to expand the bounds of the uncertainty.

where σ_{CDT} is the combined standard uncertainty of the mean for the current-corrected and deadtime-corrected X-ray counts. Using the values calculated above for the deadtime and the corresponding standard uncertainties of the means, the value of σ_{CDT} for Ge in the alloy was determined to be 211 cts in 40 s. For the Ge standard wafer that was used as the standard for Ge in the alloy, the current corrected counts, I_C, is 497168 cts in 40s with a standard uncertainty, σ_{IC}, of 657cts in 40s. The combined uncertainty for the Ge standard, σ_{CDT}, was calculated from equation (14) to be 675 cts in 40s.

Now we can calculate the uncertainty of the mean for the observed *k*-value for Ge in the SiGe alloy specimen. The equation for the determination of the *k*-value is

$$k = \frac{I_{U\,CDT} - BG_{U\,CDT}}{I_{STD\,CDT} - BG_{STD\,CDT}} \quad , \tag{15}$$

where $I_{U\,CDT}$ and $BG_{U\,CDT}$ are the beam current corrected and deadtime corrected X-ray counts for the peak and the background of the unknown, and $I_{STD\,CDT}$ and $BG_{STD\,CDT}$ are the beam current corrected and deadtime corrected X-ray counts for the peak and background of the standard. The background is determined from interpolation of readings taken from both sides of the X-ray peak where there were no interferences.

The experimental variance of the mean for the intensity of the unknown, σ^2_{IU}, in the numerator of equation (15) is

$$\sigma^2_{IU} = \sigma^2_{IU\,CDT} + \sigma^2_{BGU\,CDT} \;. \tag{16}$$

For the denominator, the experimental variance of the mean for the intensity of the standard, $\sigma^2_{I\,STD}$, is

$$\sigma^2_{I\,STD} = \sigma^2_{I\_STD\,CDT} + \sigma^2_{BG\_STD\,CDT} \;. \tag{17}$$

Then the combined standard uncertainty for the k-value, σ_k, is

$$\sigma_k = k \left(\frac{\sigma^2_{IU}}{\left(I_{U\,CDT} - BG_{U\,CDT}\right)^2} + \frac{\sigma^2_{I\,STD}}{\left(I_{STD\,CDT} - BG_{STD\,CDT}\right)^2} \right)^{1/2} \;. \tag{18}$$

This equation is the same as reported by Ziebold in 1967 and later used by Lifshin in 1999. In our case we have included the uncertainties from the deadtime and current corrections. From data in the Table 1, below, the *k*-value and the *k*-value combined standard uncertainty of the mean were calculated to be 0.27845 ± 0.00059 (0.21 %).

This is a small uncertainty, probably attributable to the good homogeneity and counting statistics for the Ge Lα X-ray peak from both the unknown and the standard as well as the low counting statistics for the background. It is not always possible in EPMA quantification to achieve these favourable conditions. When the element concentration is in minor or trace quantities or when the peak to background ratio is very low, then σ_k can be expected to be greater.

Table 1. Deadtime and current corrected X-ray counts ± standard uncertainties of the mean for the analysis of Ge Lα in a SiGe alloy with Ge wafer standard.

Counts/40 s ± Standard Uncertainty of the Mean		
Specimen	Ge Lα - on Peak	Background
SiGe Alloy	138,926 ± 210	841 ± 25
Ge Wafer (Standard)	497,167 ± 657	1,262 ± 23

The equations above are the foundation for evaluating Type A uncertainties for EPMA quantification. There may be additional contributing factors not considered here such as overlapping peak corrections or volatile elements that should be accounted for as needed. Spectrometer reproducibility, not specifically mentioned so far, is incorporated into the counting statistics of the original X-ray counts and does not have to be added as a separate component of uncertainty. It was reported in previous heterogeneity studies that spectrometer movement while reading the X-ray peak and two backgrounds during a data acquisition can result in a 30 % relative increase in the value of the standard uncertainty as compared to when the spectrometers are held stationary [14].

Type B uncertainties, if significant, should be included in the evaluation of the final uncertainties assigned to the *k*-values. The manufacturers' specifications such as the uncertainty of the current and the time readings and the impurity of standards are Type B uncertainties that could be included in the combined standard uncertainties determined above. But, according to ISO/GUM if the information is not available and there is no reliable evaluation of the uncertainty, it should be left out until such time as the information becomes available.

5. Combined standard uncertainty of the composition

From the measured *k*-value the composition in mass fraction percent is calculated using a specified ZAF or $\varphi(\rho z)$ matrix correction procedure. In spite of a" best-practices" analysis where standards of known, homogeneous compositions, good instrumental parameters (excitation potential, current, and beam size) and counting times resulting in statistically sound count rates, etc., have been used, the matrix correction procedure can contribute a significant Type B uncertainty to the calculated composition. From the present-day selection of correction procedures and MACs no single combination produces the best results for all combinations of elements and compositions. It is therefore valuable to be able to evaluate the results from several different combinations of correction procedures and MACs for any given EPMA quantification. If a multielement standard of known composition (determined from bulk quantitative analysis) that is similar to the unknown is available, EPMA quantification can be compared to the bulk certified or accepted composition to determine the best correction procedure/MAC combination. Another guideline in selecting the best correction combination is to select one that gives a mass fraction total of 100 % ± 1 % relative; but this is not a guarantee of the best choice (Note that it is absolutely not acceptable in EPMA quantification of flat polished samples to normalize data to a total of 100 % mass fraction. Normalisation is justified only in special situations such as particle analysis where geometry and size have a significant effect on quantification results). Another way to determine the best correction procedure/MAC combination is to conduct the analysis at two or three different voltages, assuming the specimen is homogeneous in depth. The best procedure will produce similar results at all voltages.

Since ISO/GUM recognizes the Type B uncertainties that are not derived from statistics obtained from experimental procedures, but rather, may result from the best judgment of the experienced scientist, an estimated uncertainty due to the correction procedure should be included in the total mass fraction uncertainty reported for each element in an EPMA quantification. This can be done with an equation proposed by Lifshin et al. [3] that determines the uncertainty of the mean for the mass

fraction by applying the equation proposed by Ziebold [2] for the empirical correction procedure. The Ziebold equation relates the *k*-value to composition, *M*, in mass fraction, with an *a*-factor

$$\frac{1-k}{k} = a\left(\frac{1-M}{M}\right).$$ (19)

From the determined *k*-value for each element the mass fraction can be calculated from a ZAF or $\varphi(\rho z)$ calculation. Then the *a*-factor can be calculated for each element from equation 18. Knowing the *a*-factor, the variance of the mean for the composition due only to counting statistics can be determined from the equation proposed by Lifshin et al.,

$$\sigma_M^2 = M^2\left[\frac{I_{UCDT}+BG_{UCDT}}{n\left(I_{UCDT}-BG_{UCDT}\right)^2} + \frac{I_{STDCDT}+BG_{STDCDT}}{n'\left(I_{STDCDT}-BG_{STDCDT}\right)^2}\right]\left[1-\frac{(a-1)M}{a}\right]^2$$ (20)

where *n* and *n'* are the number of measurements from the sample and standard respectively.

If the standard uncertainty of the mean for the matrix correction procedure/MAC combination, σ_{CP}, can be reasonably evaluated, it can be combined in quadrature with the combined uncertainty of the mean for the estimate of the composition, σ_M, to determine a combined standard uncertainty for the element quantification. This will give the final uncertainty of the mean for the element mass fraction including both Type A and Type B standard uncertainties.

Beyond determining the uncertainties, the ISO/GUM also provides guidelines for expanding uncertainties as well as how to report them. Details will not be included here as these are procedures that are already practiced in the electron probe community.

6. Conclusion

We have applied the ISO/GUM, the universally accepted guide for the evaluation of measurement uncertainties, to EPMA quantification. The ISO/GUM goes beyond the counting statistics uncertainties to include additional factors like manufacturers' specifications and scientific judgment in the evaluation of the uncertainty of a measurand. For basic quantitative EPMA the equations above can conveniently be used in a spreadsheet to facilitate repeated calculation of uncertainties. Not all details of the approach have been included here, so we recommend reading the original references.

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