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# Guidelines for estimating and reporting measurement uncertainty of chemical test results

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## Introduction

Those making decisions based on test results need to know if the results are sufficiently reliable for the intended purpose.

Every test or calibration result is subject to uncertainty. Estimates of measurement uncertainty (MU) provide information about the reliability of results. MU is an important part of a reported result and it may be argued that a result is incomplete unless accompanied by an estimate of MU.

Competent laboratories evaluate and monitor the performance of their test methods and are aware of the uncertainty associated with the results reported to customers.

ISO/IEC 17025[1], Section 5.4.6, requires calibration and testing laboratories to have and apply procedures to estimate the uncertainty of their measurements. Furthermore, Section 5.10.3 of the standard states that test reports shall include information regarding MU when a customer instructs the facility to provide the information, when it is relevant to the validity or application of test results, or when it affects compliance to a specification limit.

This Technical Note is directed primarily to chemical testing laboratories but the general principles also apply to other areas of testing. Drawing on authoritative references, it aims to define MU, explain why laboratories should estimate it, then provide general guidance on the estimation and reporting of MU. More detailed, specific information is provided in the cited references.

There are several approaches to estimates of uncertainty that are statistically sound. The methods described here are not the only ones that are acceptable. However uncertainty is determined, estimates must not give a wrong impression of MU.

This Technical Note provides guidance only.

## What is Measurement Uncertainty?

Measurement uncertainty has been defined as 'a parameter associated with the result of a measurement that characterises the dispersion of values that could reasonably be attributed to the measurand'[2] or more lately as a 'non-negative parameter characterising the dispersion of quantity values being attributed to a measurand, based on the information used'[3]

**Note 1:** The measurand is defined as 'the particular quantity subject to measurement'[3] For example, vapour pressure of a given sample of water at 20°C, mass percentage fat in a sample of cheese, or mass fraction (mg/kg) of a pesticide in a sample of apples.

ASTM[4] describes MU as 'an estimate of the magnitude of systematic and random measurement errors that may be reported along with the measurement result'. Whilst this definition perhaps does not properly embrace the concept of uncertainty it does provide some guidance regarding the factors contributing to MU and thereby some clues on how it may be estimated.

Both definitions make the important point that MU is associated with a test **result** not a test method. However, the MU associated with the results produced by application of a test method is a key performance characteristic of the method. Some regulators suggest the fitness for purpose of a test method to be judged solely on the MU associated with results[5].

The 'true value' is the result we would obtain if we made a perfect measurement. However no measurement can be perfect, so the true value can never be known. Considering the ISO definition of MU, the 'dispersion of values that could reasonably be attributed to the measurand' implies that the test result, effectively a range taking the estimated MU into account, will encompass the true value with a particular probability which depends on the level of confidence provided by the estimate of MU. By convention, in most instances an **expanded uncertainty** is estimated, applying a **coverage factor** to the **combined standard uncertainty** to provide a level of confidence of approximately 95%.

Laboratories estimating MU in this manner should be 95% confident that the reported result - with MU – will include the true value. MU may be estimated to provide different levels of confidence though the use of different coverage factors.

## ISO Guide to the Expression of Uncertainty of Measurement (GUM) definitions

**Standard uncertainty, $u(x_i)$**

Uncertainty of the result, $x_i$, of a measurement expressed as a standard deviation.

**Combined standard uncertainty, $u_c(y)$**

Standard uncertainty of the result, *y*, of a measurement when the result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances or covariances of these other quantities weighted according to how the measurement result varies with these quantities.

**Coverage factor, *k***

Numerical factor used as a multiplier of the combined standard uncertainty in order to obtain an expanded uncertainty.

**Expanded uncertainty, *U***

Quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand.

**Note 2**: The ISO GUM refers to the ISO/IEC *Guide to the Expression of Uncertainty of Measurement* (Reference 2)

**Note 3:** An expanded uncertainty is calculated from a combined standard uncertainty and a coverage factor, using $U = k \times u_c$

## Why estimate measurement uncertainty?

For facilities seeking accreditation to ISO/IEC 17025, the glib answer to this question is 'because the Standard requires you to do so!' However, there are good scientific reasons for the ISO Standard to stipulate this requirement. A good estimate of MU is necessary for facilities and their customers to:

- ensure results are fit for purpose,
- make informed decisions, and
- provide information that laboratories can use to improve their test methods.

Traceability is another important property of a test result, particularly if it is to be used for legal or regulatory purposes. Metrological traceability is defined as 'property of a measurement result whereby the result can be related to a reference through a documented unbroken chain of calibrations, **each contributing to the measurement uncertainty**'[3] The last phrase of this definition emphasises that link between MU and traceability. Further information about Metrological Traceability can be found in Policy Circular 11.

## How may facilities estimate MU?

There are a number of different approaches to estimating MU. In deciding upon the approach to use, laboratories should consider the nature of the test, the purpose of the test, the information available on which to base an estimate, how test results will be used and the risk associated with decisions based on test results.

If a well-recognised test method specifies limits to the values of the major sources of MU and the form of presentation of calculated results, a facility is considered to have satisfied the requirements of ISO/IEC 17025, with respect to the estimation of MU, by following the test method and reporting instructions (ISO/IEC 17025, Section 5.4.6.2, Note 2). Nevertheless, laboratories in this situation are advised to estimate MU since experience has shown that the limits imposed by standard methods are not always sufficient to ensure results are fit for purpose. If a facility's estimate of MU is based on the stated reproducibility for a standard method then the facility should ensure that the estimate covers all sources of uncertainty pertaining to their measurements.

The ISO/IEC 17025 Standard (Section 5.4.6.2) also recognises that the nature of some test methods may preclude vigorous, metrologically and statistically valid calculation of MU. In such cases, laboratories are still required to make a reasonable estimate of MU based on, for example, professional judgement and experience, knowledge of method performance and validation data. Such estimates must not give a wrong impression of MU.

The many different approaches for the estimation of MU may include components derived from what may be broadly categorised into either 'bottom-up' or 'top-down' calculations.

The fundamental metrological 'bottom-up' approach described in the ISO/IEC *Guide to the Expression of Uncertainty in Measurement*[2], commonly called the GUM, combines the uncertainties associated with all individual operations of an analytical procedure to calculate the combined standard uncertainty and, after multiplication by a coverage factor, the expanded uncertainty (usually the 95% confidence range). The GUM sets down general principles and guidance for estimating the uncertainty associated with single steps of analytical procedures (weighings, volumes etc.), calculated concentrations of standard solutions, spiked concentrations or relatively simple tests. For more complex test procedures it can be laborious, even if spreadsheets are used for calculations. Horwitz[6] considers the approach inappropriate for chemical tests because it ignores the fact that the uncertainties associated with some of the numerous factors influencing test results tend to cancel out. Furthermore, it is argued that chemical test results are often influenced by factors that overwhelm the uncertainties considered in the GUM approach.[6,7]

An advantage of the GUM approach is that it provides a clear understanding of the analytical operations that contribute significantly to MU, allowing the analyst to focus on improving these operations if required to reduce the MU associated with test results. However, experienced analysts are most likely able to identify the critical steps of analytical procedures independent of any estimates of MU.

Eurachem/CITAC[8] provides guidance on how the GUM approach may be applied to chemical measurements. It also includes examples using a 'top-down' approach. A 'top-down' approach utilises data from method validation, intra-laboratory QC and/or inter-laboratory studies. The use of such data, include all contributions to uncertainty. Numerous references, aimed at providing chemical testing facilities with practical guidance and examples of simple 'top-down' approaches to estimating MU, are available.[9-13]

A 'top-down' approach is generally more practical than the GUM approach for estimating the MU of chemical test results. However it is the facility's decision to use the method most appropriate for their circumstances and supported by the available data.

MU may not be the same over the range of concentrations tested. In this case, the laboratory must estimate the MU at the levels commonly tested, including levels close to the limit of detection, or regulatory limit, as appropriate.

The following sub-sections provide examples of how a top-down approach may be applied to estimate MU. A systematic procedure for obtaining fit-for-purpose estimates of MU is presented in Figure 1 and a practical example of how MU may be estimated utilising data on bias and precision is shown in Appendix 1.

## Estimating measurement uncertainty from reproducibility studies

*Note: A facility may estimate MU based on the between-laboratory reproducibility reported for a standard method, however the facility should first ensure that they are able to achieve the within-laboratory repeatability stated for the method.*

The standard deviation determined from inter-laboratory studies under reproducibility conditions (i.e. no variation to the method, including subsampling at the laboratory level), $s_R$, may be used as an approximation of the combined uncertainty associated with a result.[11] This estimate is then doubled to give the expanded uncertainty (95% Confidence Interval).

$u_c \approx s_R$, and

$U = 2\, s_R$

where: $u_c$ = combined standard uncertainty

$s_R$ = standard deviation under reproducibility conditions

$U$ = expanded uncertainty, 95% confidence interval

In the absence of data from inter-laboratory studies on a particular method, the reproducibility standard deviation may be estimated from an equation proposed by Horwitz. The Horwitz equation[14-16], $s_R = 0.02C^{0.85}$, was empirically derived by plotting reproducibility standard deviation vs concentration, *C* (in g/g) for more than 7000 inter-laboratory studies. This equation may be used to estimate $s_R$ and *U* at different concentrations. It may also be used to check the validity of estimates of $s_R$ from inter-laboratory studies. The equation predicts that lowering analyte concentration by two orders of magnitude will double the between-laboratory relative standard deviation, $RSD_R$, associated with a test result. However, advances in analytical chemistry with the introduction of techniques such as isotope-dilution mass spectrometry, have provided the capability to achieve very low limits of quantitation with less uncertainty than predicted by the Horwitz equation. Thompson and Lowthian[17] have reported that laboratories tend to out-perform the Horwitz function at concentrations below 10 µg/kg.

$$\sigma = \begin{cases} 0.22 * c & if\ c < 1.2 * 10^{-7} \\ 0.02 * c^{0.8495} & if\ 1.20 * 10^{-7} \le c \le 0.138 \\ 0.01 * c^{0.5} & if\ c < 0.138 \end{cases}$$

where c = concentration, (e.g. the assigned value $X$ expressed as a dimensionless mass ratio $1\text{ppm} \equiv 10^{-6}$ or $\% \equiv 10^{-2}$)

requires c to be dimensionless mass ratio, eg.$1\text{ppm} \equiv 10^{-6}$ or $1\% \equiv 10^{-2}$.

If results are corrected for bias, or the bias is small, estimates of MU based on $s_R$ calculated using the Horwitz equation may suffice where there is no requirement for a more rigorous estimate of the uncertainty associated with results.

## Estimation of MU from within-laboratory data of bias and precision

A reasonable estimate of MU may be gained from information on the bias and precision associated with a test result. This information may be gained from participation in suitable inter-laboratory and/or intra-laboratory studies and used to estimate MU.[11-13]

Suitable inter-laboratory studies are not always available and more often than not, laboratories are required to estimate MU from data generated 'in-house'.

Ideally, precision and bias should be determined within the same analytical run as the sample(s) analysed. This is generally impractical and cost-prohibitive for most chemical tests. However, if suitable QC samples (matrix-matched Certified Reference Materials [CRMs], other suitable Reference Materials [RMs] or spiked samples) are run within each sample batch to ensure the test method is consistently operating within acceptable control limits, then the data generated may be used to evaluate both intermediate reproducibility (within-laboratory precision under reproducibility conditions) and average bias over a given period of time or number of sample batches. Precision and bias should be evaluated at the concentration(s) most relevant to the purpose of the test.

**Note 4**: Estimates of MU may also be gained from the evaluation of precision and bias during initial validation of a method.

Considering a model for the result of a chemical test conducted under the above conditions:

$y = y_{true} + b + e$ (Equation 1)

where: $y$ = observed measurement, uncorrected for bias;
$y_{true}$ = true result;
$b$ = bias, defined as $\bar{y} - y_{true}$, the difference between the mean of a large number of observed results ($\bar{y}$) and the true result; and
$e$ = random error for within-laboratory reproducibility conditions, $s_L$.

**Expression for combined standard uncertainty**

The combined standard uncertainty of *y* may be estimated as:

$$u_c(y)^2 = s_L^2 + u_b^2 \quad \text{(Equation 2)}$$

where: $u_c(y)$ = combined standard uncertainty of *y*
$s_L$ = standard deviation of results obtained under within-laboratory reproducibility conditions
$u_b$ = standard uncertainty associated with bias.

**Estimating bias and the uncertainty associated with bias**

In practice, the true result is not known and it is necessary to base estimates of bias on the expected result for the analysis of a CRM, or other suitable sample.

$$b = \bar{y} - y_{exp} \quad \text{(Equation 3)}$$

where: $y_{exp}$ is the expected result

$$u_b^2 = u(\bar{y})^2 + u(y_{exp})^2 \quad \text{(Equation 4)}$$

where: $u(\bar{y})$ = standard uncertainty of observed result;
$u(y_{exp})$ = standard uncertainty of expected result.

If the observed result is taken to be the mean result from replicate analyses of a CRM or other suitable sample, performed over several months, the standard uncertainty may be taken as the standard deviation of the mean of the observed result.

i.e. $u(\bar{y}) = \frac{s_L}{\sqrt{n}}$ (Equation 5)

where *n* = number of replicates performed.

The standard uncertainty of the expected result may be known from the certified value of a CRM, the characterisation of a secondary reference material, estimated from the uncertainties associated with spiking, or estimated in some other way, as the particular situation demands.

Once *b* and $u_b$ are estimated according to Equations 3 and 4, it is important to test whether or not the bias, taking into account the uncertainty associated with its measurement, is significant. If $|b| > t(0.05, n\text{-}1)u_b$, where t is the Student-t value at n-1 degrees of freedom, then bias is significant and steps must be taken to account for bias when calculating results or estimating MU (see below). If the average bias is based on 20 or more measurements, |*b*| can be simply compared with $2\,u_b$ in order to check if the average bias is significant.

**Estimating combined standard uncertainty and expanded uncertainty**

If bias is not significant, the combined standard uncertainty may be calculated from Equation 2.

If $u_c(y)$ is based on sufficient data the expanded uncertainty, *U*, may be calculated using a coverage factor of 2 to give an approximate level of confidence of 95%.

$$U \text{ (95\% confidence interval)} = k\,u_c(y)$$
$$= 2\,u_c(y) \quad \text{(Equation 6)}$$

**Note 5**: The Eurachem/CITAC Guide[8], Section 8.3, recommends that for most purposes *k* is set at 2. The Guide states that this value may be insufficient where the combined uncertainty is based on statistical observations with relatively few degrees of freedom (less than about six) and further recommends that in such cases *k* be set equal to the two-tailed value of the Student's t for the number of degrees of freedom associated with the observations and the level of confidence required (normally 95%). It should be noted that about 19 degrees of freedom are required for *k* to be less than 2.1.

If bias is significant and based on reliable data such as the analysis of a CRM, then the measurement result should always be corrected for bias.[2] Where excessive bias is detected, action should be taken to investigate, and if possible, eliminate the cause of the bias.[11] Such actions should at least reduce bias to acceptable levels before a facility proceeds with analyses of test samples.

If results are not corrected for significant bias, the MU should be enlarged to ensure that the true result is encompassed by the reported confidence interval.

A number of approaches have been described for taking significant bias into account when estimating MU.

The best approach for handling significant bias is to eliminate or minimise the bias.

O'Donnell and Hibbert [18] critically evaluated different approaches and concluded the best estimate of the expanded uncertainty to be:

$U = k\,u_c(y) + |b_{run}|$ , where $b_{run}$ is the run bias

**Note 6:** The symbols used in the above equation are consistent with those used in this Technical Note, rather than those used by O'Donnell and Hibbert.

O'Donnell and Hibbert specify run bias in their recommended equation for enlarging the estimate of MU, and provide explanation for using this approach. A reasonable estimate of MU may be obtained if average total bias (when the bias is small), evaluated as described above, is used in place of run bias.

$U = k\,u_c(y) + |b|$ (Equation 7)

Failure to include the uncertainty of the estimation of bias in estimates of combined uncertainty or failure to correct for significant bias or enlarge the uncertainty of the result to account for it, invalidates both the test result and the estimated MU.

If bias can be quantified and is consistent, then it is preferable to correct the final result for bias, rather than expand the uncertainty to include bias. Presenting a result x $\pm$ the uncertainty assumes that x is in the middle of a normal distribution of results. Expanding the uncertainty to include a large, known bias changes the distribution, and gives a false impression of the result being in the middle of a wider range.

Laboratories should ensure that MU is estimated (at least) at the concentrations most relevant to the purpose of the test, for example regulatory, legal or specification levels.

**Reality checks for estimates of MU**

Having completed an uncertainty estimation, the estimate should be subject to a 'reality check'. A reality check can be as simple as considering whether the MU is consistent with the knowledge and experience of the facility. MU estimations reported by other facilities (using the same approach to estimate the MU, for the same analyses and using similar equipment) will not be exactly the same as the facility's own estimate, but they should not vary excessively. Data from collaborative trials, inter-laboratory reproducibility and proficiency program results will all provide useful information for comparison.[19]

# Reporting Measurement Uncertainty

The Eurachem/CITAC Guide[8] states that 'unless otherwise required, the result *x* should be stated together with the expanded uncertainty *U*, calculated using a coverage factor, *k = 2*.'

The recommended form for reporting a result is:

(Result): $x \pm U$ (units).

The coverage factor used to calculate *U* should be stated, for example;

'*DDT*: 3.52 ± 0.14 mg/kg
The reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2 which gives a level of confidence of approximately 95%'.

The value and its uncertainty should both be reported in the same units. Results reported as *x* ± (a percentage of *x*) are not recommended.

The value of a result and its uncertainty should not be reported with an excessive number of significant figures. It is rarely, if ever, necessary to report a chemical test result with the uncertainty stated to more than two significant figures. Results should be rounded to be consistent with their uncertainty.

It is recognised that some Laboratory Information Management Systems may not be able to readily report MU in the recommended format. Other presentations are acceptable providing the essential elements of the recommended format are clearly covered in the test report.

For further information regarding MU, contact your Client Coordinator or the Sector Manager, Life Sciences: Neil Shepherd, in NATA's Melbourne office on (03) 9274 8200 or email: Neil.Shepherd@nata.com.au

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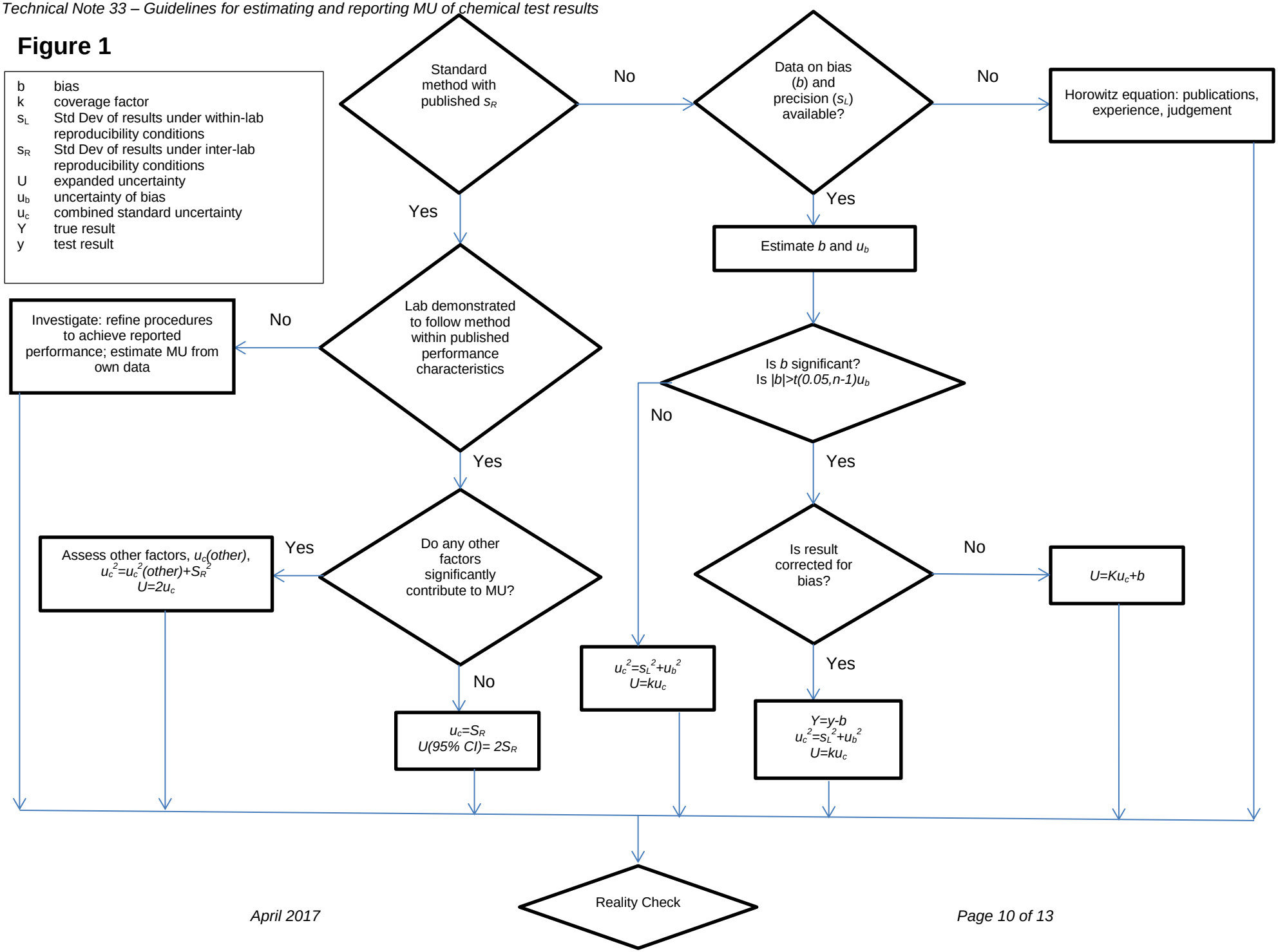
## Figure 1

| | |
|---|---|
| b | bias |
| k | coverage factor |
| $s_L$ | Std Dev of results under within-lab reproducibility conditions |
| $s_R$ | Std Dev of results under inter-lab reproducibility conditions |
| U | expanded uncertainty |
| $u_b$ | uncertainty of bias |
| $u_c$ | combined standard uncertainty |
| Y | true result |
| y | test result |



# Appendix 1:

# Estimation of MU from within-laboratory data on bias and precision

## Scenario

A facility has validated a method for the determination of residues of the pesticide, chlorpyrifos in tomatoes and has applied the method on customer samples submitted over a period of eighteen weeks. A matrix-matched certified reference material (MX 001) obtained from the NMI was used during the validation process and henceforth once a fortnight to supplement spiked samples routinely used for within-batch QC. The certified value for chlorpyrifos in the CRM is 0.489 ±0.031 mg/kg (95% confidence range).

The facility initially estimated the MU of chlorpyrifos results based solely on validation data but now wishes to update its estimate, taking into account the QC data generated during routine analysis of customer samples.

The facility analysed seven replicates of the CRM during method validation and a further nine replicates during routine analyses. The following data was generated via these processes:

Validation data: mean 0.391 mg/kg, standard deviation ( $s_r$ ) 0.051 mg/kg, CV 13.0%

QC data: mean 0.385 mg/kg, standard deviation ( $s_R$ ) 0.082 mg/kg, CV 21.3%

Control charts plotted for both the CRM and spiked samples run as QC samples demonstrate the test method to be under statistical control.

The average result was consistent for both validation and QC analyses, however, as expected, the results of the QC tests conducted under intra-laboratory reproducibility conditions were less precise than those for method validation conducted under near repeatability conditions. Both the % recovery (negative bias) and precision were considered acceptable for the test.

It was considered important to estimate the MU associated with results close to 0.5 mg/kg, the maximum residue limit (MRL) for chlorpyrifos in tomato. No doubt with the MRL in mind, the CRM produced by the NMI has a certified value for chlorpyrifos closely matching the MRL.

## Estimation of MU

**Data**

The mean of all results is the best estimate of the likely batch-to-batch result. This value (the average of all validation and QC replicate measurements) is the average observed result, $\bar{y}$ = 0.388 mg/kg; mean recovery = 79.3%

The standard deviation of the QC results best reflects the imprecision of results produced under normal test conditions; $s_L = s_R$ = 0.082 mg/kg

The expected result, $y_{\text{exp}}$, is the certified value of the CRM; 0.489 ± 0.031 mg/kg

The standard uncertainty of the expected result $u\left(y_{\text{exp}}\right)$ is obtained by halving the expanded uncertainty (95% confidence range) stated for the CRM.

$u\left(y_{\text{exp}}\right)$ = 0.031/2 = 0.0155 mg/kg

**Estimation of bias and the uncertainty of bias**

Bias ( $b$ ) = $\bar{y} - y_{\text{exp}}$ (Equation 3) = 0.388 – 0.489 = - 0.101 mg/kg

This is the average bias and the best available estimate of the bias that might apply to any individual test result.

Uncertainty of bias (from equation 4), $u_b = \sqrt{u\left(\bar{y}\right)^2 + u\left(y_{\text{exp}}\right)^2}$

Uncertainty of the observed result (Equation 5), $u(\bar{y}) = \frac{s_L}{\sqrt{n}}$ = 0.082/3 = 0.027 mg/kg

$u_b = \sqrt{u(\bar{y})^2 + u(y_{\exp})^2} = \sqrt{(0.027)^2 + (0.0155)^2}$ = 0.031 mg/kg

**Is the bias significant?**

Is $|b|$ > t (0.05, n-1) $u_b$ ?

Here, n = 9 since the uncertainty of the observed result is based on nine replicate tests
From t tables (critical values for two-tailed student t-tests), t (0.05, 8) = 2.306

Is 0.101 > 2.306 x 0.031? Is 0.101 > 0.071? Answer YES, so bias IS significant

It is necessary for the facility to correct results for bias (preferable alternative) or enlarge their estimate of MU to account for uncorrected significant bias.

**Estimating combined standard uncertainty and expanded uncertainty**

(i) <u>results corrected for bias</u>

If the facility decided to correct results at or about the MRL for the negative bias, the correction could be achieved by simply adding the bias to the measured value, assuming the bias to be constant over a narrow concentration range. There is uncertainty associated with this correction, and it is therefore necessary to include the uncertainty of bias in the combined standard uncertainty, irrespective of whether or not results are corrected for bias.

Assuming a raw test result = 0.35 mg/kg.
Corrected result = 0.45 mg/kg

Combined standard uncertainty, (from Equation 2)

$u_C(y) = \sqrt{(s_L)^2 + (u_b)^2} = \sqrt{(0.082)^2 + (0.031)^2}$ = 0.088 mg/kg

Expanded uncertainty (Equation 6);

$U$ (95% confidence interval) = 2 $u_C(y)$ = 0.18 mg/kg

The facility would report the result as 0.45 ± 0.18 mg/kg, noting that the reported uncertainty is an expanded uncertainty calculated using a coverage factor of 2 to give a level of confidence of approximately 95%.

(ii) <u>results not corrected for bias</u>

Expanded uncertainty (Equation 7): $U = 2u_c(y) + |b|$

= 0.18 + 0.10

= 0.28 mg/kg

The facility would report the result as 0.35 ± 0.28 mg/kg.

When the expanded uncertainty is enlarged in example (ii) to account for uncorrected significant bias, the reported result encompasses a 95% confidence range, which is wider than justified on the lower side of the measured value: the uncertainty allows for both negative and positive bias although only negative bias is present.

The result corrected for bias in example (i) provides the customer with a better estimate of the true result and a better basis for decision-making, although as expected when the 'true' value is close to a limit, the result is equivocal with respect to compliance with the MRL.

**AMENDMENT TABLE**

The table below provides a summary of changes made to the document with this issue.

| Section | Amendment |
|---|---|
| All | Editorial alterations to text to improve readability |
| | Corrections to statistical calculations and explanations; content significantly revised. |