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Quality of Test Results Expressed Through Measurement Uncertainty

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Abstract: *Measurement uncertainty can be quantified using calculation/estimation of single components of uncertainty. The application of such approach underestimates uncertainty in measurement partly because it is hardly possible to include all possible components of uncertainty. This paper presents methodology of calculation of measurement uncertainty based on existing and experimentally obtained data on internal quality control, method of data validation, data on inter-laboratory comparison as well as those obtained from reference materials, thus reaching maximum probability of comprising all components of uncertainty.*
The knowledge of uncertainty in measurement is of great importance for all users of laboratory services, laboratory itself and all interested parties that benefit from the results of research

Keywords: *measurement uncertainty, quality of results and research*

1. INTRODUCTION

When the first edition of ISO/IEC 17025 standards was presented, great importance was assigned to the measurement uncertainty concept. Since then, great many changes have occurred in the domain of measurement uncertainty calculations.

Within technical requirements of ISO/IEC standards 17025:2006 – General requirements for the competence of testing and calibration laboratories [7] in Section 5.4.6 there is a requirement that refers to evaluation of measurement uncertainty in testing laboratories:

„Testing laboratories shall have and shall apple procedures for estimating of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and shall ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. Reasonable estimation shall be based on knowledge of the performance of the method and on the measurement scope and shall make use of, for example, previous experience and validation data.

NOTE 1 The degree of rigor needed in an estimation of uncertainty of measurement depends on factors such as:

» the requirements of the test method;
» the requirements of the customer;
» the existence of narrow limits on which decisions on conformity to a specification are based.

NOTE 2 In those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions (see 5.10).

5.4.6.3 When estimating the uncertainty of measurement, all uncertainty components which are of importance in the given situation shall be taken into account using appropriate methods of analysis.

NOTE 1 Sources contributing to the uncertainty include, but are not necessarily limited to, reference standards and reference materials used, methods and equipment used, environmental conditions, properties and condition of the item being tested or calibrated, and operator.

NOTE 2 The predicted long-term behaviour of the tested and/or calibrated item is not normally taken into account when estimating the measurement uncertainty."

2. REQUIREMENTS OF CUSTOMERS FOR TEST QUALITY RESULTS

Analytical result can never absolutely be "correct", since at least two minimum different results are obtained in a repeated measurement. It is possible to deliver results with sufficiently small uncertainty i.e. to create a result that is fit for purpose.

Therefore, the analyst needs to know the intended use of the result before the requirement for analytical quality is defined.

On the other hand, the users of the results expect to be able to trust the data, but in most cases they do not have the expert knowledge necessary to explain what they need and they rely on the laboratory to supply the right answer to the problem – that is to deliver a result that is fit for the purpose

Fortunately the majority of users for a specific parameter in specific matrix, for example ammonium in drinking water will need the analyses for the same purpose and therefore have the same requirements for quality. The laboratory therefore does not need to think closely on the subject every day but can design its quality control programme so that the data delivered will have the correct quality for the purpose.

Still the correct quality needs to be defined.

In some cases, national or regional authorities have defined the required quality for regulatory analyses. For example, European Drinking Water Directive 98/83/EC contains requirements for quality. If no such national or regional requirements for quality exist, the laboratory must prepare its own requirements, preferably in cooperation with the customer.

The requirement on analytical quality can be given as:

» requirement for uncertainty. Uncertainty in measurement will show the customer possible maximum deviation for each result in comparison to reference value or arithmetic mean, which is obtained by testing of the same samples by large number of competent laboratories;

» requirement for dispersion of results (repeatability or reproducibility). These quality characteristics can be measured directly, for example by internal quality control. Internal quality control generally provides the customer with possible variance of results in the same characteristic given to laboratory for analysis (for example in January, July and December).

The main purpose of estimation of uncertainty in measurement is to detect whether a laboratory can fulfill the customer demands with the analytical method in question. However, in cases when requirements have not been established, expanded measurement uncertainty U should be equal to or less than two times the reproducibility, s_{Rw}.

3. MEASUREMENT UNCERTAINTY

There are several definitions of uncertainty in measurement: "uncertainty (of measurement) parameter, associated with the result of a measurement that characterizes the dispersion of the values that could reasonably be attributed to the measurand" [1] or "Uncertainty is a quantitative expression of quality results that provides comparison of results with other results, references, specifications and etalons" [9].

All measurements are affected by a certain error. The measurement uncertainty tells us what size the measurement error might be.

Therefore, the measurement uncertainty is an important part of the reported result.

Without reported uncertainty, a measurement result is not applicable, therefore not integral. Only an integral measurement result provides correct interpretation of a measurement result and making reliable decisions on the known scope of risk.

Overall measurement result is expressed in the following form [1]:

$Y = y \pm U$, where

Y is a measurand whose value is not precisely known, thus being considered a random variable with function of probability distribution, y is a measurement result as an estimation of the expected value and U denotes uncertainty. The uncertainty which is reported in the measurement result is usually expanded and obtained by multiplication of combined measurement uncertainty and numerical value of factor, most often k=2, that corresponds to the interval of approximately 9,5% of confidence level.

Standard measurement uncertainty $u(x)$ is measurement uncertainty expressed as a standard deviation and is equal to the square root of the estimated variance.

Combined measurement uncertainty u_c is determined when the obtained measurement result is based on measurements of several components (input measurements in measurement function) and is equal to the square root of the sum of the squares of standard components of measurement uncertainty.

Expanded measurement uncertainty U is half amplitude of symmetrical interval scope (confidence level), centered in relation to the estimation of a quantity with specific probability of coverage.

Measurement uncertainty, as well as accuracy, is a combination of random and systematic effects. This is illustrated in Figure 1 where different requirements on measurement uncertainty are illustrated with small and big circles.

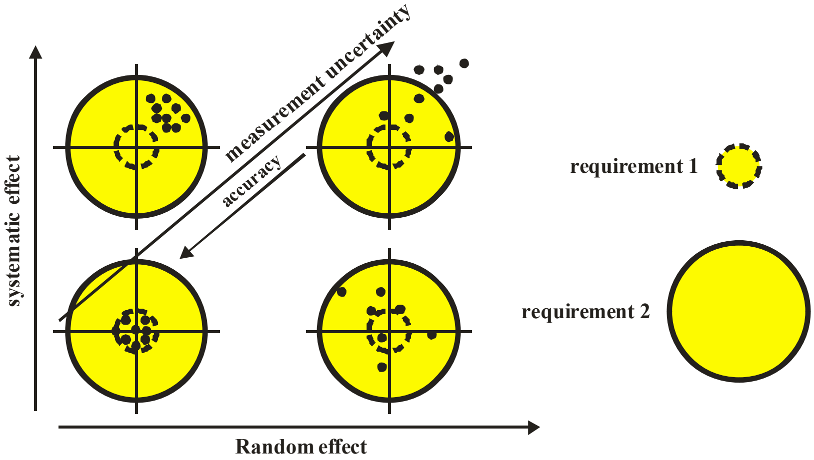


Figure 1 – Requirements on measurement uncertainty

Each point represents a reported analytical result. The two circles are illustrating different requirements on analytical quality. In the lower left circle the requirements on analytical quality are fulfilled, while those illustrated with circle 2 are fulfilled in all cases except the upper right. The upper left case represents a typical situation for most laboratories.

Figure 1 illustrates that the quality sufficient for one purpose is not necessarily sufficient for all other purposes. It is also extremely important to remember that it is always the intended use of the data, not the capability of the laboratory that defines the necessary quality. Data can be too bad to be useful; they can also be too good, as too good often means too expensive or to slow to obtain.

4. CALCULATION OF MEASUREMENT UNCERTAINTY

Measure uncertainty is obtained by measurements and statistical approach where different sources of uncertainty are estimated and combined into a single value.

The guidelines are given in GUM [1], EA Guidelines [6], Eurachem/CITAC guide [2], Technical Report by Eurolab [3] and ISO/DTS 21478 [5].

A common way of presenting the different contributions to the total measurement uncertainty is to use a so-called fish-bone (or cause-and-effect) diagram.

Constructing a detailed fish-bone diagram, the individual uncertainty components are calculated/estimated. This approach may prove very useful when quantifying individual uncertainty components.

It has been shown, though, that in some cases this methodology underestimates the measurement uncertainty [3], partly because it is hard to include all possible uncertainty contributions in such an approach. By using existing and experimentally determined data such as:

- » internal quality control,
- » method validation,
- » interlaboratory comparisons,
- » obtained by using reference material,

the probability of including all uncertainty contributions will be maximized.

Basis for the evaluation of measurement uncertainty is the existing knowledge (laboratories should not be required on specific scientific analysis). The existing experimental data should be used (diagrams of internal quality control, validation, interlaboratory comparisons, certified reference materials etc.) [6].

It is desirable to use the model presented in Figure 2, where within laboratory reproducibility R_w is combined with method bias and laboratory bias [8].

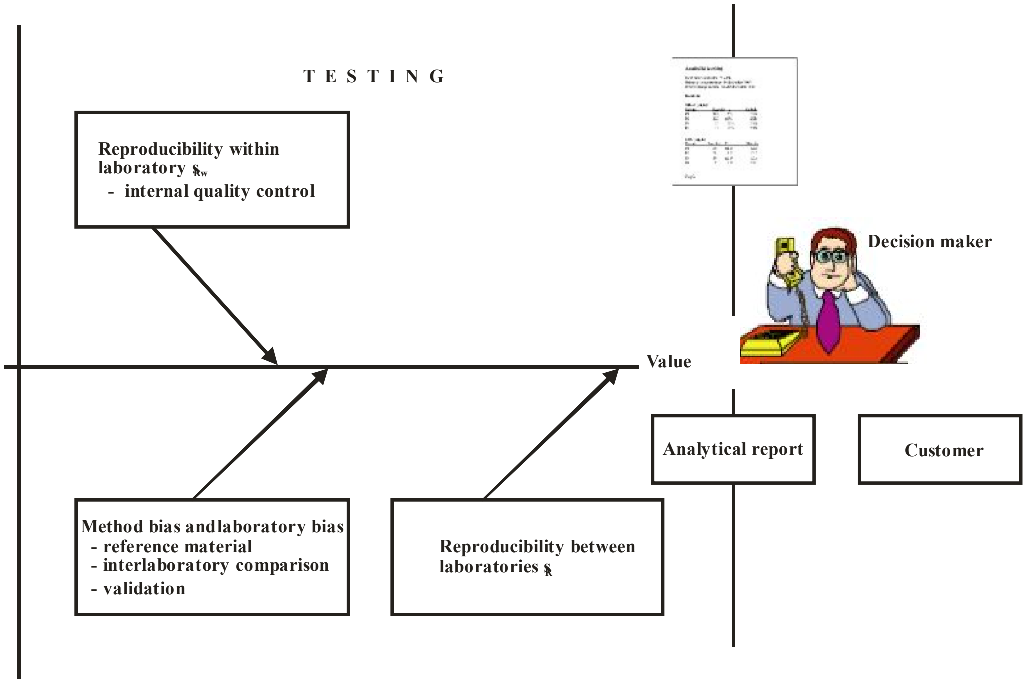


Figure 2 - Measurement uncertainty model – fish-bone diagram

Flow scheme of uncertainty calculation presented in Figure 2, involving 6 defined steps, should be followed in all cases (Figure 3).

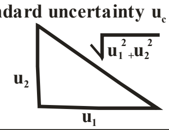
Step	Action	Measurand
1	Specify measurand	*Measurand in matrix by method ______. The customer demand on expanded uncertainty is ± __ % .*
2	Quantify *u* (R_w) A control sample B possible steps not covered by the control sample	A: B:
3	Quantify method and laboratory bias	
4	Convert components to standard uncertainty *u(x)*	
5	Calculate combined standard uncertainty u_c 	
6	Calculate expanded uncertainty $U=2*u_c$	

Figure 3 - Flow scheme for calculations measurement uncertainty

		Value	Relative u(x)	Comments
Reproducibility within laboratory s_{Rw}				
Control sample $\bar{X}$= (conc) (unit)	s_{Rw}			
Other components				
Method and laboratory bias				
Reference material	bias			
Interlaboratory comparison	bias			
Recovery test	bias			
Reproducibility between laboratories s_R				
Interlaboratory comparison	s_R			
Standard method	s_R			

Combined uncertainty u_c is calculated from ____ and bias from ____ .

Measurand	Combined uncertainty u_c	Expanded uncertainty U
		$2*u_c$=

Figure 4 – Summary table for uncertainty calculations

The results of the calculations done in the flow scheme are summarised in a summary table for uncertainty calculations (Figure 4).

5. REPORTING UNCERTAINTY

Figure 5 presents an example on what a data report could look like, when measurement uncertainty has been calculated and is reported together with the data. The laboratory and accreditation body logotypes are omitted, and the report does not contain all information normally required for an accredited laboratory.

Analytical Report

Sample identification: M1-M3
Samples received: 01. 04. 2009.
Completion of analyses: 05. 04. 2009.

Results:

NH_4-N (μg/l):

Sample	Result	U	Method
M1	103	6%	I34
M2	122	6%	I34
M3	12	10%	I33

TOC (μg/l):

Sample	Result	U	Method
M1	40	4%	I35
M2	35	3,5%	I35
M3	10	1%	I35

Signed:

____

Figure 5 – An example of report on measurement uncertainty

The laboratory should also prepare a note explaining how the measurement uncertainty has been calculated for the different parameters. Normally, such an explanatory note should be communicated to regular customers and other customers who ask for information. Example, where ± 7 is the measurement uncertainty: Ammonium (NH4-N) = 148 ± 7 μg/L. The measurement uncertainty, 7 μg/L (95 % confidence level, i.e. the coverage factor k=2) is estimated from control samples and from regular interlaboratory comparisons.

4. CONCLUSIONS

A laboratory should base its measurement uncertainty evaluation on existing knowledge and experimental data. Thus obtained measurement uncertainty is necessary for:

- » the user, together with results so that proper decision could be made for example when comparing results with acceptable values, tolerance limits or permissive (legal) values,
- » the laboratory, to be aware of quality of its own measurements (whether there is a difference among different laboratory results, or the results obtained at the same laboratory under different conditions), and thus improve it to necessary level.

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