



Nordic Committee on Food Analysis

www.nmkl.org

NMKL Protocol No. 5, 2011 (11. October)

Analytical Quality Control - Guidelines for the publication of analytical results of chemical analyses in foodstuffs

NMKL – Nordic Committee on Food Analysis

www.nmkl.org

Content

Foreword

1. Introduction
2. Analytical quality control requirements
3. General recommendations
 - 3.1 To authors
 - 3.2 To editors
 - 3.3 To reviewers/referees
 - 3.4 Check-list for the Analytical Quality Control criteria
4. Analytical quality control parameters
 - 4.1 Sampling
 - 4.1.1 Sample representativity
 - 4.1.2 Sampling procedure, transport and storage
 - 4.2 Measurement method and procedure
 - 4.2.1 Appropriate choice of method
 - 4.2.2 Clarity/transparency of method description
 - 4.2.3 Awareness of interferences
 - 4.2.4 Determination of recovery
 - 4.2.5 Handling of sample blanks
 - 4.2.6 Reporting limit
 - 4.2.7 Definition of the reporting limit
 - 4.2.8 Number of replicates
 - 4.3 Certified reference materials
 - 4.3.1 Relevant matrix
 - 4.3.2 Relevant concentration
 - 4.3.3 Reference to CRMs
 - 4.4 Proficiency testing
 - 4.4.1 Relevant matrix
 - 4.4.2 Relevant concentration
 - 4.5 Measurement uncertainty
 - 4.5.1 Random error (standard deviation)
 - 4.5.2 Systematic error (bias)
 - 4.6 Unexpected/extreme results
 - 4.6.1 Verification by another method

4.6.2 Verification by another laboratory

5. Organisation of the AQC in the manuscript
 - 5.1 Description of AQC procedures
 - 5.2 Presentation of AQC results
 - 5.3 Presentation of analytical results in tables (and text)
6. Terms and definitions
7. References

Foreword

The European Organisation of Standardisation for foods, CEN/TC 275, working group (WG) 10 – Elements and their chemical species - selects and elaborates validated methods of analysis in foodstuffs into standards. The working group has concluded that there is a need for guidance on what should be described regarding analytical quality control in publications containing analytical results of foodstuffs, in order to ensure a high degree of reliability. An ad-hoc group within WG 10 began the development of these guidelines in 2006. In order to gain greater transparency and easy distribution, their final development and publication was transferred to the NMKL in 2010, with the aim of having it published as a NMKL Protocol. Those primarily involved in the process were Lars Jorhem (SE, Project Leader), Jörg Oehlenschläger (GE), Kåre Julshamn (NO), Joakim Engman (SE) and Henk van der Schee (NL).

During the development process the journal editors Robert Rathbone (Journal of AOAC International), John Gilbert (Food Additives and Contaminants) and Rune Waagbø (Journal of Aquaculture Nutrition) were consulted regarding the content and format of these guidelines. Their suggestions and comments have been invaluable for its progress. This does not, however, mean that they endorse the guidelines in their entirety.

1. Introduction

There are many papers published annually on the content/occurrence of harmful and essential components in foodstuffs. Published data may be used for different purposes, such as legislation and intake recommendations. The reliability of (published) data depends to a large degree on the description of the analytical quality control procedures used prior to and during the analysis [1]. If such procedures are not reported, or poorly described, together with the results, the reliability of the results may be questioned. This in turn puts the potential user of the data at risk and can lead to severe unintended consequences in legislation, food composition tables, intake recommendations and problems in world trade (due to the WTOs TBT and SPS agreements).

Most laboratories have implemented a programme for analytical quality assurance (AQA), based on the requirements in ISO/IEC 17025:2005 [2]. Accreditation is given to a laboratory

after it has shown that it has fully implemented the AQA-programme, and it covers the use of one, or several, specific analytical methods. Accreditation is often seen as a guarantee for good analytical quality and reliable results, but this is not automatically true. Although analytical quality control (AQC) is an integrated part of AQA, the content of most AQC-procedures are not detailed by ISO/IEC 17025. Such procedures may include handling of contamination, loss of analytes, interference effects as well as other factors. Analytical quality control procedures must therefore be developed and integrated in the AQA-system in each laboratory. Published surveys/studies sometimes show results that are unusual, or extreme. Such results may be extremely important, but their verification is a prerequisite. It is also well known that the description of the quality control procedures and its outcome often is unstructured and follows no template [3]. This often makes it difficult to evaluate the analytical quality of publications.

These guidelines describe analytical quality control procedures that are intended to help ensuring the quality and reliability of published analytical data. They are based on quality criteria such as sampling procedures, method selection and description, use of certified reference materials and participation in proficiency testing. In addition they include some advice on how to organise the description of the AQC in the manuscript.

These guidelines are intended for use by all parties involved in the publishing process, i.e., manuscript authors, journal editors and manuscript reviewers/referees.

The AQC-criteria are summarised in a **Check-list** (under 3.4) that is intended to assist the author of the manuscript and to simplify/standardise the reviewing process.

2. Analytical quality control requirements

The goal of quality assurance and quality control (QA/QC) is to identify and implement sampling and analytical methodologies which limit the introduction of error into analytical data [4].

There are many quality parameters to consider in order ensuring the reliability of analytical data. Analytical problems in the form of interferences and contamination differ widely between techniques. The choice of method is sometimes not open for alternatives due to factors such as existing equipment and routines. Certified reference materials and proficiency tests are not available for every combination of analyte and matrix. It is therefore difficult to state absolute requirements for the analytical quality control. The AQC-parameters that make up the basis for these guidelines are described in the chapters 4.1 – 4.6 and summarised in the **Check-list** under 3.4. These AQC-parameters are commonly used in analytical chemistry laboratories. Other parameters may be applicable in certain areas or when specific requirements are encountered. Such parameters must be pointed out by the authors, but may also be identified by the editor or reviewer.

3. General recommendations

These guidelines are intended to be used by authors, (journal) editors and reviewers/referees. The evaluation process of a submitted manuscript involves the (journal) editor and, usually, one or several reviewers/referees. The manuscript's content and style is evaluated according to instructions that may vary somewhat between journals. The evaluation process should include a procedure for analytical quality control (AQC), which could consist of a reference to these guidelines.

The description of the AQC parameters and AQC data should be organised in a logical and transparent way in the manuscript. A description of how the manuscript could be organised is shown in section 5. The AQC-parameters (section 4) and structure recommendations (section

5) are summarised in the **Check-list** under 3.4. The **Check-list** can be used together with the Protocol, or separately (can be downloaded from the NMKL web site), in which case this Protocol should be consulted when clarification is needed.

These guidelines should serve as an aid to those involved in the publishing process. The author(s) should consult the **Check-list** in order to ensure that relevant criteria are complied with. The reviewer(s) should check if the AQC is sufficient to provide reliable results, and the editor decides if the paper should be published. Human errors, such as misplaced decimals, forgotten dilutions or transcription errors are not considered in these guidelines.

3.1 To authors

The analyst(s) doing the analytical work and the author(s) may be the same or different persons. It is the responsibility of the (corresponding/main) author to ensure that the analytical results are reliable and reflects the actual content of the analyte in that specific matrix/commodity. The author must be able to show awareness of the intrinsic problems or dangers with the technique used. The risk for uncompensated interferences, contamination or loss of analyte must be considered. If unusual/unexpected/extreme results are found it must be ensured that they are not due to analytical error. This can be done e.g., by analysis using another method or consulting another laboratory. If such results are not verified analytically, this should be stated in the manuscript.

The **check-list** (3.4) gives a comprehensive overlook of the most common AQC-parameters used in analytical control of foodstuffs, and provides a systematic approach to their control. The **check-list** should be consulted at the planning stage of a project to make sure that the relevant parts are complied with. When a project is finished it is often very costly and time-consuming to complement the results. Any claim of AQC-procedures (e.g., blanks, CRMs, PTs) carried out must be supported by analytical data. If AQC-data are not presented in the manuscript, they should be stored (by the author or at the editorial office) in order to be presented on request. If other AQC-parameters apply they should be presented, and explained, by the author.

3.2 To editors

The editors must make sure that their “Instructions for authors” also gives the author relevant and sufficient information on how to describe the AQC. This information could consist of a reference to these guidelines. The instructions for reviewer’s/referees should contain a request to check that the manuscript has sufficient information on the AQC to ensure the reliability of the published data, for example in relation to these guidelines.

Journals that do not publish descriptions of the analytical quality should nevertheless request such information from the author(s), and when the paper is accepted for publication archive the quality control data at the editorial office (or by the author). A note should be made in the publication with the information that AQC-data have been obtained and validated and how a reader can obtain this information. The editor shall require the AQC information from the author to follow a prescribed format, e.g. these guidelines.

3.3 To reviewers/referees

The reviewer should check that the analytical data presented in the manuscript are reliable according to a set of predetermined AQC criteria, e.g., those presented in the **Check-list** under 3.4 of these guidelines. A reviewer is usually expected to have a certain expertise in the subject and should therefore be able to give a rather detailed response to the author’s description of the AQC. For example, if an author has used an apparently unsuitable method, but motivate the choice, a reviewer may conclude that it nevertheless is acceptable for the analytical purpose.

The selection of certified reference materials (CRMs) and proficiency testing (PT) schemes should be given particular attention. The CRM should have been analysed in parallel to the sample, to check that the results are unbiased. The results of the PTs show the general competence of the analyst/laboratory at a specific time or over a period of time that should be near, or overlapping, the time when the survey was carried out.

Unexpected/extreme results must be verified and/or explained, even if the analyst has used CRMs and/or participated in PT-programmes.

3.4 Check-list for analytical quality control (AQC) criteria, to be considered in the process of publishing analytical data (also available in electronic format at www.nmkl.org under “Protocols”)

Paragraph in protocol	<u>Analytical parameter</u>	<u>Reviewer comments</u>	
		Sufficient	Insufficient
4.1	SAMPLING DESCRIPTION		
4.1.1	<i>Sampling representativity</i>		
4.1.2	<i>Sampling procedure, transport, storage</i>		
4.2	METHODOLOGY		
4.2.1	<i>Appropriate choice of method</i>		
4.2.2	<i>Clarity/transparency of description</i>		
4.2.3	<i>Awareness of interferences</i>		
4.2.4	<i>Determination of recovery</i>		
4.2.5	<i>Handling of sample blanks</i>		
4.2.6	<i>Reporting limit</i>		
4.2.7	<i>Definition of the reporting limit</i>		
4.2.8	<i>Number of replicates</i>		
4.3	CERTIFIED REFERENCE MATERIALS Available and used in study	Yes	No
4.3.1	<i>Relevant matrix</i>		
4.3.2	<i>Relevant concentration</i>		
4.3.3	<i>Clearly referenced</i>		
4.4	PROFICIENCY TESTING Available and used in study		
4.4.1	<i>Relevant matrix</i>		
4.4.2	<i>Relevant concentration</i>		
4.5	MEASUREMENT UNCERTAINTY Described/presented		
4.5.1	<i>Random error (standard deviation)</i>		
4.5.2	<i>Systematic error (bias)</i>		
4.6	UNEXPECTED RESULTS		
4.6.1	<i>If yes in 4.6, verified by other method</i>		
4.6.2	<i>If yes in 4.6, verified by other laboratory</i>		
5	ACCEPTABLE ORGANISATION OF AQC		
5.1	<i>Description of AQC procedures acceptable</i>		
5.2	<i>Presentation of AQC-results acceptable</i>		

4. Analytical quality control criteria/parameters and check-list

The criteria described in sections 4.1 – 4.6 refer to the same numbers in the **Check-list** (3.4)

4.1 Sampling

The sampling procedure is of vital importance for the interpretation of the result of the study/survey. It is important from a technical as well as statistical point of view. Often, however, the sampling process is separated from the analytical process, and the analyst has little or no information on the background of the samples. A publication including analytical results must, however, present information on the sampling process.

4.1.1 Sample representativity

The author must include information on what the samples represent and how they were selected¹. Depending on the nature of the publication, the quantity of information may differ considerably. Analytes are often more or less heterogeneously distributed in samples, e.g., cadmium in wheat grain. In cases where the sampling is a major issue, the following points may need to be described, but others may be equally important/ applicable:

Parts of sample analysed?

Pooled samples?

Weight of sub samples?

Number of sub samples?

Statistical considerations?

¹ : “Random sampling” in its strictest sense is almost never utilised. Usually the samples are stratified in some way, and in each stratum one or several samples can be randomly selected. For example, fish can be stratified according to number of samples, species, lakes, size etc.

4.1.2 Sampling procedure, transport and storage

Sampling procedures often involve a risk of either contaminating the sample, or of losing parts of the analyte. Transports involve risks of, e.g., overheating or freezing of the samples and cross contamination. Storage of samples involves risks of, e.g., loss of liquid (time/temperature, freezing out), contamination and chemical changes of matrix and/or analyte.

A description of these procedures is therefore necessary. Analytes that are susceptible to negative effects require more detailed descriptions than others.

Inexplicably high results may have been caused by contamination in a pre-analytical stage, which is often difficult to identify. A clear description of the sampling procedure may make it possible to conclude where or how the contamination occurred. Since contamination seldom is uniform it may be detected by large variations in replicated analysis (provided that it is larger than the normal heterogeneity). The risk of contamination varies with the type of sample, analyte and concentration level; e.g. in the case of certain trace elements the contamination risk is great all through the sampling and analytical process, whereas in sampling and analysis of pesticides or natural toxins the risk is very small. If different samples arrive to the lab in the same container cross contamination can be a problem.

It should be clearly described what has been analysed, e.g., homogenate of a whole fish, or a specific organ/tissue, and if the result is based on fresh or dry weight.

Loss of analyte

Just as for contamination, loss of analyte during sampling and shipping may be outside of the analyst's control. Loss of analyte can occur for several reasons and at different stages in the analytical process. Volatile analytes may be lost if the sample is exposed to high temperatures, e.g., during transport or storage. Other analytes, e.g., metals, may be lost if the sample "leaks" liquid. Loss of liquid will increase the dry matter and the concentration of the analyte (if not lost with the liquid), just as hygroscopic samples will decrease the dry matter and the analyte concentration.

4.2 Measurement method and procedure

4.2.1 *Appropriate choice of method*

The method must be fit for the analytical purpose. The choice of method is usually not decided only by the analytical need. Often factors such as tradition, availability of instruments and analytical cost influence the choice of method. It can therefore be of importance to briefly describe the rationale behind the choice of method.

4.2.2 Clarity/transparency of method description

The description of the method should be clear and unambiguous. The method principle and a reference should be given. A statement on the degree of method validation should be given, e.g., in-house or collaboratively validated. It should be clearly described if the result is based on fresh or dry weight.

Unit of measurement: Check with the journal which unit is preferred and do not mix units. The units ng/g, µg/g, µg/kg, mg/kg, ng/ml, µg/ml, µg/l and mg/l are by far the most used in international journals. Do not use, e.g. nmol/l or ppm, unless there is a specific reason for doing so. When comparing with legal requirements, e.g., maximum limits, use the same unit as in the legal text.

4.2.3 Awareness of interferences

The author must show awareness of the interferences present in the method/technique used. Common examples of interferences are background absorbance and mass overlap in AAS and ICP-MS, respectively, and ion suppression in LC/GC-MS. In other analytical techniques other types of interferences may be predominant.

4.2.4 Determination of recovery

This primarily concerns methods involving an extraction step or preconcentration of the analyte. In other types of methods, e.g., the determination of total levels of elements, recovery can even result in confusing/erroneous information. Recovery must not be confused

with bias and cannot replace the use of CRMs or results from PT. It must be clearly stated if reported results are adjusted for recovery or not.

4.2.5 Handling of sample blanks

In analysis where contamination during sample preparation and analysis is a potential problem the determination of analytical blanks is crucial. The blank may also provide the basis for the limit of detection. Chemical blanks should reflect the analytical process and will show if contamination has occurred during sample pretreatment or analyte detection. If the blank level is significant it must be clearly stated whether it has been deducted from the sample reading prior to calculation of the result, or not. Contamination that has occurred prior to sample pretreatment will go unnoticed through this procedure (see 4.1 Sampling).

4.2.6 Reporting limit

The reporting limit is usually given as the limit of detection (LOD) or the limit of quantification (LOQ). The reporting limit is particularly important when low concentrations are determined. In method validation the limit of detection (LOD) is one of the parameters that need to be established. If the measured levels are lower than 10 times the established LOD it is prudent to report the LOD. As the concentration increase the importance of stating the LOD decreases. Give the result as “< (numerical figure/unit)”, e.g. <0.01µg/L. Do not report results as, e.g., “Not detected” or “Below LOD or LOQ”.

Where appropriate, the Lowest Calibrated Level can be reported as the lowest level under AQC. The lowest point of the calibration curve, measured in each batch shows that response at that level is in accordance with QC.

4.2.7 Definition of the reporting limit

There are several ways to calculate/define the reporting limit (LOD or LOQ) and this must therefore be clearly described. A common method is based on the standard deviation (SD) for a signal at, or close to, the LOD. The LOD is then calculated as 3 x SD and the LOQ as 6 (or 10) x SD.

4.2.8 Number of replicates

State if the sample is replicated and how (e.g., all duplicates, or every 10th in duplicate/triplicate). Replication is a way to check for inhomogeneity or contamination. If the results are affected by (random) contamination or inhomogeneity, it may be seen as large variations in replicate analysis. Systematic contamination, however (e.g., by contaminated chemicals, or calibration errors), will not be detected. Replicates are used to calculate repeatability SD.

4.3 Certified Reference Materials

The use of Certified Reference Materials (CRMs, the US/NIST uses the trademark “Standard Reference Material”, i.e., SRM) is vital in the AQC system, and is primarily used to estimate bias (trueness or systematic errors), See 4.5.2. The CRM must be relevant in terms of matrix and analyte concentration. If a relevant CRM is available it shall be used and the result reported. If an available and relevant CRM is not used it should be stated why. Spike recovery may in some cases provide a (limited) complement.

4.3.1 Relevant matrix

The matrix of the CRM must be as similar as possible to the samples. There is no guide available to assist the analyst in the choice, so the analyst must make the best possible choice based on the information available. If a broad survey of many different types of foodstuffs is carried out it may be necessary to choose several CRMs, in order to have a representative selection.

4.3.2 Relevant concentration

The analyte level in the CRM must be relevant for the levels found in the samples, If the difference is too big the results of the samples cannot be said to be verified by the CRM. There is no absolute scale available for how big a difference that can be accepted, but as a rule of thumb the analyte concentration should not differ more than 10 times between the sample and the CRM.

4.3.3 Reference to CRMs

When CRMs have been used in a study its identity should be clearly stated, e.g., NIST SRM 1570a Spinach, or BCR CRM 185R Bovine liver.

4.4 Proficiency Testing

The analyte level in a Proficiency Test (PT) sample is unknown to the analyst at the time of measurement, and is the only independent way of demonstrating analytical proficiency. The individual results from PT are usually expressed as z-scores. Satisfactory results from repeated participation in relevant PTs makes strong evidence for analytical competence.

If a relevant (with respect to the matrix and analyte concentration) PT programme is available it should be used, and the result reported in the publication. If a suitable PT programme is not used, it should, if possible, be stated why. Reported PT results should be relevant in terms of time and instruments (i.e. having been analysed approximately at the time of the samples and using same method and instrumentation as for the samples). If results from several PTs are available, it is up to the analyst/author to decide if all the results should be reported. There must, however, not be a biased selection of results. State the name of the PT-provider.

4.4.1 Relevant matrix

The same requirements as for CRMs apply (see 4.3.1). However, the number of available PTs is limited so the analyst/laboratory may need to widen the search to find suitable programme.

4.4.2 Relevant concentration

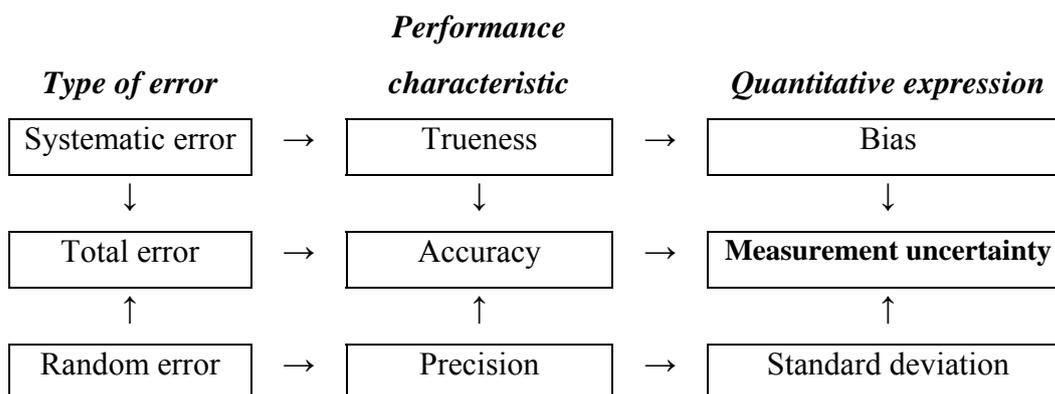
The same requirements as for CRMs apply (see 4.3.2).

4.5 Measurement uncertainty

An analytical value comprises two parts; the result and its uncertainty. Measurement uncertainty (MU) is important from many aspects: Interpretation of results near legal limits

require information about the MU. It is also a requirement for accreditation. Many users of published data need the MU in order to validate the analytical data. Figure 1 shows the components of MU and their relation [6]. Even if the author has not calculated the MU, information should nevertheless be provided on method bias and standard deviation.

Figure 1. The relationship between type of error, performance characteristics and their quantitative expression



4.5.1 Random error (standard deviation)

The random error can be estimated from the repeated measurements under laboratory internal reproducibility conditions, *e.g.* by repeated analysis of the same sample at different days

4.5.2 Systematic error (bias)

Bias, which is derived from the systematic error (see Figure 1), is primarily determined by way of:

- CRMs (SRMs) or
- Participation in PT.

Bias is determined for two purposes;

- For bias determination in the method validation process. These results are normally not presented in publications.
- For verification of results in a survey or study. Such results should always accompany the analytical results.

Bias is mainly applicable to methods not including an extraction or preconcentration step. The result of a CRM-analysis should be tested for bias using a valid statistical procedure. There are several ways to test for bias² [e.g. 7-9]. If the result from a CRM is unbiased it may be assumed that the results of the samples, together with which the CRM was analysed, also are unbiased. If significant bias is detected it should, in principle, not be corrected for, but the cause of the bias should be identified and eliminated, and the samples reanalysed. There may be isolated cases where correction for bias is justified, but these are exceptions. In certain applications (e.g., pesticide analysis) bias may be determined by spiked samples.

PT is usually not carried out in connection with specific samples or surveys, but shows the general competence of the analyst/laboratory, at a specific time or over a period of time. This state of the art information about the lab is important and PT-results should be described in the report/manuscript.

4.6 Unexpected/extreme results

An unexpected or extreme result deviates considerably from the expected or known level or range. For example: If the Pb-level in a “normal” milk sample is found to be 0.03 mg/kg it is higher than what is generally expected. Thus it is a level that would benefit from verification in order to maintain credibility. Even higher levels must definitely be verified. It is not possible to state a specific level at which a result goes from “normal” to “unexpected/unlikely”. It is up to the expertise and experience of the analyst, and the reviewer, to make that judgement.

4.6.1 Verification by another method

Ideally an unexpected/unlikely result should be verified using a different method, with an alternative detection technique. Such options are, however, not always available, in which case it is the responsibility of the analyst to find a way to ensure that the results are valid, or highlight that the result is unconfirmed.

² Results from CRMs are frequently published without any statistical test for bias. They are often just accompanied by a statement saying e.g., “the found mean result was close to the certified value” or “the found value was within the certified range”. Such statements are not statistically valid.

4.6.2 Verification by another laboratory

Having the sample analysed by another competent laboratory is usually a reliable way of verifying a result. It must be kept in mind, however, that if the other lab uses an identical analytical procedure there is a possibility that an analytical problem is duplicated. A different method is therefore preferable.

5. Organisation of the AQC in the manuscript

The criteria described in sections 5.1 – 5.2 refer to the same number in the **Check-list** in Annex. The information on AQC should be written in a consistent way that is easy to follow by the reader. Preferably, the journal shall give advice in the instructions to authors.

5.1 Description of AQC procedures

Analytical papers should be written in a consistent way for clarity. The structure is usually as described below, and the AQC activities should be mentioned or detailed where relevant:

- Abstract. Mention that a systematic AQC-procedure has been used, or that these guidelines has been used.
- Introduction. Strengthen the AQC-statement in the abstract, and with a reference. Do not present any analytical data here.
- Analytical procedure, and/or, Materials and methods. Follow the recommendations in these guidelines. Do not present analytical data from QC-analysis here.
- Results (and discussion). Present the AQC-results in a logical and consistent way. If discussion is included, make a statement on how the AQC -results have strengthened the analytical results.
- Discussion. If the discussion is separated from the results, make a statement on how the QC-results have strengthened the analytical results.
- Conclusion(s): If relevant for the text.
- References. Include a reference to these guidelines for clarity.

5.2 Presentation of AQC results

The QC results should be the first part under Results (and discussion). Whether data should be described in the text or in separate tables largely depends on the style of the journal and the quantity of data. Sometimes the number of results from e.g. CRMs (and PT) can be considerable. It must then be decided if the results should be presented individually or in a more generalised form. The results should be described together with their standard deviation and bias which, ideally, should be combined to show the total MU (4.5).

5.3 Presentation of analytical (CRM-) results in tables (and text)

- The unit of measurement should be clearly stated in all tables, and the text, and be of the same unit as the results of the study/survey.
- The number of significant figures in the found results should not exceed the number supported by the MU.

6. Terms and definitions

The terms described here are those used in these guidelines.

Analytical Quality Assurance (AQA)

All those planned or systematic actions necessary to provide enough confidence that a product or service will satisfy the given requirements for quality [10]

Analytical Quality Control (AQC)

The operational techniques and activities that are used to fulfil requirements of quality [10].

NOTE: 'Quality control' and 'quality assurance' are terms whose meanings are often varied according to the context. In practical terms quality assurance relates to the overall measures taken by the laboratory to ensure and regulate quality, whereas quality control describes the individual measures which relate to the monitoring and control of particular analytical operations [5].

]Bias

Estimate of a systematic measurement error [11].

Certified Reference Material (CRM)

Reference material accompanied by documentation issued by an authoritative body and providing one or more specified property values with associated uncertainties and traceabilities, using valid procedures [11].

Interlaboratory comparisons

Organisation, performance and evaluation of tests on the same or similar test items by two or more laboratories in accordance with predetermined conditions [12].

Limit of detection (LOD)

Measured quantity value, obtained by a given measurement procedure, for which the probability of falsely claiming the absence of a component in a material is β , given a probability α of falsely claiming its presence [11].

Smallest measured content, from which it is possible to deduce the presence of the analyte with reasonable statistical certainty. The limit of detection is numerically equal to three times the standard deviation of the mean of blank determinations ($n > 20$). [15].

NOTE: In e.g. elemental analysis the LOD is usually expressed as three times the standard deviation (SD) for the mean result of a large number of blanks ($LOD = 3 \times SD$). For methods where the blank is insignificant the SD is usually expressed as the mean blank plus three times the standard deviation ($LOD = \text{mean blank} + (3 \times SD)$). Other definitions may apply.

Limit of quantification (LOQ)

A method performance characteristic generally expressed in terms of the signal or measurement (true) value that will produce estimates having a specified relative standard deviation (RSD), commonly 10% (or 6%) [13].

Lowest content of the analyte which can be measured with reasonable statistical certainty. If both accuracy and precision are constant over a concentration range around the limit of detection, then the limit of quantification is numerically equal to six or 10 times the standard deviation of the mean of blank determinations ($n > 20$) [15].

Measurement uncertainty (MU)

Non-negative parameter characterizing the dispersion of the quantity values being attributed to a measurand, based on the information used [11].

Proficiency testing

Determination of laboratory testing performance by means of interlaboratory comparisons [12].

Recovery (recovery factor)

Yield of a preconcentration or extraction stage of an analytical process for an analyte divided by the amount of analyte in the original sample [14.]

Reference Material (RM)

Material, sufficiently homogeneous and stable with reference to specified properties, which has been established to be fit for its intended use in measurement or in examination of nominal properties [11].

NOTE; Reference materials with or without assigned quantity values can be used for measurement precision control whereas only reference materials with assigned quantity values can be used for calibration or measurement trueness control [11].

Standard deviation

Measures the spread of a distribution around the mean. It is often denoted as s and is the square root of the sample variance, denoted s^2 . A normal distribution is mainly defined by the mean and standard deviation.

Trueness

Closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value [11].

7. Bibliography (References)

1. Jorhem L, Engman J, Sundström B, Nilsson A. (2006). Evaluation of measurement data for Cd, Cr and Pb in certain uncontaminated foodstuffs published in surveys: Analytical quality vs. uncertainty of measurements. *Journal of Accreditation and Quality Assurance*. 10: 647-658.
2. ISO/IEC 17025: 2005. General requirements for the competence of testing and calibration laboratories. Case Postale 56, CH-1211, Genève 20. Switzerland
3. Jenks P, Stoepler M. (2001). The deplorable state of the description of the use of certified reference materials in the literature. *Fresenius Journal of Analytical Chemistry*. 370: 164-169.
4. The Multi-Agency Radiation Survey and Site Investigation Manual. Environmental Protection Agency. United States of America.
5. EURACHEM. Guide to the Fitness for Purpose of Analytical Methods. (1998). A Laboratory Guide to Method Validation and Related Topics
6. Menditto A, Patriarca M, Magnusson B (2006). Understanding the meaning of accuracy, trueness and precision. *Journal of Accreditation and Quality Assurance*. 12: 45-47
7. ISO-guide 33. Uses of certified reference materials. Case Postale 56, CH-1211, Genève 20. Switzerland
8. NMKL Procedure No 9 (2007). Evaluation of method bias using certified reference materials . Nordic Committee on Food Analysis, Norwegian Veterinary Institute, PB 750 Sentrum, 0106 Oslo, Norway.
9. [www.erm-crm.org/ERM products/application notes/application note 1/Pages/index.aspx](http://www.erm-crm.org/ERM_products/application_notes/application_note_1/Pages/index.aspx)

10. ISO 8402-1994. Quality – vocabulary. Case Postale 56, CH-1211, Genève 20. Switzerland.
11. ISO/IEC Guide 99:2007. International vocabulary of metrology — Basic and general concepts and associated terms (VIM). Case Postale 56, CH-1211, Genève 20. Switzerland
12. ISO 13528 (2005). Statistical methods for use in proficiency testing by interlaboratory comparisons. Case Postale 56, CH-1211, Genève 20. Switzerland
13. Guidelines on analytical terminology (CAC/GL 72-2009). Codex Alimentarius Commission.
14. Use of the terms “recovery” and “apparent recovery” in analytical procedures, Burns D.T, Danzer K, Townsend A. (IUPAC Recommendations 2002) Pure and Applied Chemistry. 2002. 74:2201-2205
15. COMMISSION REGULATION (EC) No 333/2007 of 28 March 2007 laying down the methods of sampling and analysis for the official control of the levels of lead, cadmium, mercury, inorganic tin, 3-MCPD and benzo(a)pyrene in foodstuffs