codex alimentarius commission

FOOD AND AGRICULTURE ORGANIZATION OF THE UNITED NATIONS

WORLD HEALTH ORGANIZATION

JOINT OFFICE: Viale delle Terme di Caracalla 00153 ROME Tel: 39 06 57051 www.codexalimentarius.net Email: codex@fao.org Facsimile: 39 06 5705 4593

Agenda Item 4 **CX/MAS 10/31/4**

JOINT FAO/WHO FOOD STANDARDS PROGRAMME

CODEX COMMITTEE ON METHODS OF ANALYSIS AND SAMPLING
Thirty-first Session
Budapest, Hungary, 8 - 12 March 2010

PROPOSED DRAFT REVISED GUIDELINES ON MEASUREMENT UNCERTAINTY
(At Step 3 of the Procedure)

(Prepared by the United Kingdom)

BACKGROUND

At the twenty-ninth Session of CCMAS there was discussion on the preparation of guidance on (analytical) measurement uncertainty and uncertainty of sampling. This arose because a number of delegations had previously requested of the Commission further guidance in order to address measurement uncertainty following the adoption of the text on "The Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards". The Commission had referred to the CCMAS this request.

The Delegation of the United Kingdom had prepared a paper to aid discussion. After extensive discussion the Committee agreed that, subject to the approval of the Commission, the Delegation of the United Kingdom, with the assistance of an electronic working group open to all members and observers and working in English, would prepare a Proposed Draft Revision of the Guidelines for comments at Step 3 and consideration by the next session.

There was general agreement that explanatory notes on the significance on the current Guidelines on Measurement Uncertainty (CAC/GL 54-2004) would be the appropriate way forward. These could then address of text as that have already been adopted by the Commission.

It was agreed that this activity would be a "New Work Item" for CCMAS. This was approved by the Codex Commission in 2008.

There was extensive discussion on the paper prepared by the Delegation of the United Kingdom at the thirtieth Session of CCMAS. In particular the following points were made and noted by delegates:

- The document was not intended for metrological experts but routine providers of analytical data, customers of laboratories reporting analytical data and Codex delegates. The document attempted to clarify the significance of measurement uncertainty and answer several specific questions in sections 1 to 9. Delegates recalled that measurement uncertainty has to be estimated under the requirements of ISO 17025:2005, already adopted in Codex by reference, and that it was anticipated that this request would be made for the purposes of international trade.

- It was noted that the document did not address sampling uncertainty; and that no specific procedures were recommended for estimating uncertainty, only that the procedure should be scientifically credible. The document highlighted the implications of the recommendations in the Procedural Manual on the *Use of Analytical Results* and in particular that Codex Commodity Committees should recognise the difference between the numeric value in the specification and the numeric value at which the specification would be enforced.

- There was concern that the sections referring to the need for accreditation should be clarified to avoid confusion as the Codex guidelines do not require accreditation status of laboratories but compliance with the international standard on accreditation.

It was agreed that the documents should be redrafted by an electronic working group led by the Delegation of the United Kingdom, open to all members and observers and working in English.

The document was circulated all participants at the thirtieth Session of CCMAS and a number of comments were received. In the light of the comments the current Guidelines have been revised by including explanatory notes. These are given in the attached draft revision of CAC/GL 54-2004.

Comments were prepared by delegates from Argentina, Australia, Chile, Cuba, the International Dairy Federation, Japan, The Netherlands, New Zealand, NMKL and the USA and sent to the UK for consideration since the thirtieth Session of CCMAS.

The comments received are often in conflict with each other but the paper was generally appreciated.

In particular it should be noted that:

The paper does **only** addresses uncertainty derived from the analytical process. At this time measurement uncertainty derived from analysis is long established though the consequences of its estimation are less so. Until Codex decides whether consideration of uncertainty derived from sampling should be formally addressed within the Codex system the Guidelines are written not to consider uncertainty derived from sampling. Whether uncertainty derived from sampling is to be considered within Codex is outside of the scope of this paper. They will be discussed in a separate document also to be considered at the thirty-first Session of CCMAS.

The explanatory notes are written to help non-specialists, and in particular to emphasise that as a result of the consequence of the adoption of the text "The Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards" in the Procedural Manual there are consequences for other Codex Committees, which was the original concern with the subject. The explanatory notes have therefore been written with this objective in mind and statistical considerations have been kept to a minimum.

No procedures are recommended for the estimation of uncertainty – that is not the objective of the draft revised guidelines.

Various definitions have been suggested for "measurement uncertainty" but since the last Session of CCMAS Codex has adopted formal analytical terminology and this has been used in the draft Guidelines.

Some concern was expressed by exporting countries that they would be disadvantaged by the procedures now given in the Procedural Manual. In fact because of the consequence of taking measurement uncertainty into account when establishing "beyond reasonable doubt" it is importing countries that have to be most concerned that the value of the specification in the Codex Standard is negotiated with the measurement uncertainty being appreciated.

The existing Guidelines on Measurement Uncertainty have been retained as far as possible – the objective of this paper is to help appreciate the consequences of their adoption by Codex.

RECOMMENDATIONS

It is recommended that:

- the Committee discusses whether the draft explanatory notes to the existing Codex Guidelines in Measurement Uncertainty meet the concerns of delegations following the adoption by the Commission of the text on "The Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, the Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards", and
- whether there are additional considerations that should be addressed.

The Proposed Draft Revised Guidelines and the accompanying explanatory notes are hereby circulated at Step 3 for comments and consideration by the 31st Session of the Committee on Methods of Analysis and Sampling. Governments and international organisations wishing to provide comments should do so in writing, preferably by email, to the Secretary, Codex Alimentarius Commission, Joint FAO/WHO Food Standards Programme, FAO, Viale delle Terme di Caracalla, 00153 Rome, Italy, Fax: +39 (06) 5705 4593, E-mail: codex@fao.org, with a copy to the Hungarian Codex Contact Point, Hungarian Food Safety Office, H- 1097 Gyáli út 2-6. Budapest Hungary, Fax:+36 13879400, e-mail: HU_CodexCP@mebih.gov.hu, **before 1 March 2010**.

ANNEX: PROPOSED DRAFT REVISED GUIDELINES ON MEASUREMENT UNCERTAINTY AND EXPLANATORY NOTES ON THE SIGNIFICANT OF THE GUIDDLINES (Revised CAC/GL 54-2004)

Introduction

It is important and required by ISO/IEC 17025:2005 that analysts are aware of the measurement uncertainty associated with each analytical result and estimates that uncertainty. The measurement uncertainty may be derived by a number of procedures. Food analysis laboratories are required, for Codex purposes, to be in control, use collaboratively tested or validated methods when available, and verify their application before taking them into routine use. Such laboratories therefore have available to them a range of analytical data which can be used to estimate their measurement uncertainty.

These guidelines only apply to quantitative analysis.

Most quantitative analytical results take the form of "a ± 2u or a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty and "U" (equal to 2u) is the expanded uncertainty. The range "a ± 2u" represents a 95% level of confidence (K=2) within which the true value would be found (in other cases can be increased as K=3 (99%)). The value of "U" or "2u" is the value which is normally used and reported by analysts and is hereafter referred to as "measurement uncertainty" and may be estimated in a number of different ways.

Terminology

Non-negative parameter characterising the dispersion of the values being attributed to a measurand, based on the information used.

Notes:

Measurement uncertainty includes components arising from systematic effects, such as components associated with corrections and the assigned values of measurement standards, as well as the definitional uncertainty. Sometimes estimated systematic effects are not corrected for but, instead associated measurement uncertainty components are incorporated.

The parameter may be, for example, a standard deviation called standard measurement uncertainty (or a given multiple of it), or the half-width of interval having a stated coverage probability.

Measurement uncertainty comprises, in general many components. Some of these components may be evaluated by Type A evaluation of measurement uncertainty from the statistical distribution of the values from a series of measurements and can be characterized by experimental standard deviations. The other components which may be evaluated by Type B evaluation of measurement uncertainty can also be characterized by standard deviations, evaluated from assumed probability distributions based on experience or other information.
In general, for a given set of information, it is understood that the measurement uncertainty is associated with a stated quality value attributed to the measurand. A modification of this value results in a modification of the associated uncertainty.

Reference:

VIM, International Vocabulary of Metrology – Basic and general concepts and associated terms, 3rd edition, JCGM 200: 2008

This definition for Measurement Uncertainty adopted by the Codex Alimentarius Commission.

Recommendations

1. The measurement uncertainty associated with all analytical results is to be estimated.

2. The measurement uncertainty of an analytical result may be estimated by a number of procedures, notably those described by ISO (1) and EURACHEM (2). These documents recommend procedures based on a component-by-component approach, method validation data, internal quality control data and proficiency test data. The need to undertake an estimation of the measurement uncertainty using the ISO component-by-component approach is not necessary if the other forms of data are available and used to estimate the uncertainty. In many cases the overall uncertainty may be determined by an inter-laboratory (collaborative) study by a number of laboratories and a number of matrices by the IUPAC/ISO/AOAC INTERNATIONAL (3) or by the ISO 5725 Protocols (4).

3 The measurement uncertainty and its level of confidence must, on request, be made available to the user (customer) of the results.

References

1. "Guide to the Expression of Uncertainty in Measurement", ISO, Geneva, 1993.
2. EURACHEM/CITAC Guide Quantifying Uncertainty In Analytical Measurement (Second Edition), EURACHEM Secretariat, BAM, Berlin, 2000. This is available as a free download from http://www.eurachem.ul.pt/
3. "Protocol for the Design, Conduct and Interpretation of Method Performance Studies", ed. W. Horwitz, *Pure Appl. Chem.*, 1995, 67, 33 1-343.
4. "Precision of Test Methods", Geneva, 1994, ISO 5725, Previous editions were issued in 1981 and 1986.

EXPLANATORY NOTES TO THE CODEX GUIDELINES ON MEASUREMENT UNCERTAINTY

These Explanatory Notes are written not for metrological experts but routine providers of analytical data, customers of laboratories reporting analytical data and delegates to Codex Commodity Committees.

INTRODUCTION

In the Procedural Manual it is stated in the section dealing with: "The Use of Analytical Results: Sampling Plans, Relationship between the Analytical Results, The Measurement Uncertainty, Recovery Factors and Provisions in Codex Standards":

Issues Involved

There are a number of analytical and sampling considerations which prevent the uniform implementation of legislative standards. In particular, different approaches may be taken regarding sampling procedures, the use of measurement uncertainty and recovery corrections.

At present there is no official guidance on how to interpret analytical results in the framework of Codex. Significantly different decisions may be taken after analysis of the "same sample". For example some countries use an "every-item-must-comply" sampling regime, others use an "average of a lot" regime, some deduct the measurement uncertainty associated with the result, others do not, some countries correct analytical results for recovery, others do not. This interpretation may also be affected by the number of significant figures included in any commodity specification.

It is essential that analytical results be interpreted in the same way if there is to be harmonization in the framework of Codex.

It is stressed that this is not an analysis or sampling problem as such but an administrative problem which has been highlighted as the result of recent activities in the analytical sector, most notably the development of International Guidelines on the Use of Recovery Factors when Reporting Analytical Results and various Guides prepared dealing with Measurement Uncertainty.

Recommendations

It is recommended that when a Codex Commodity Committee discusses and agrees on a commodity specification and the analytical methods concerned, it states the following information in the Codex Standard:

1. Sampling Plans

The appropriate sampling plan, as outlined in the Guidelines for Sampling (CAC/GL 50-2004), Section 2.1.2 Guidelines on Sampling to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, or to the average in a lot**,** or the proportion non-conforming;
- the appropriate acceptable quality level to be used;
- the acceptance conditions of a lot controlled, in relation to the qualitative/quantitative characteristic determined on the sample.

2 Measurement Uncertainty

An allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens.

3 Recovery

Analytical results are to be expressed on a recovery corrected basis where appropriate and relevant, and when corrected it has to be so stated.

If a result has been corrected for recovery, the method by which the recovery was taken into account should be stated. The recovery rate is to be quoted wherever possible.

When laying down provisions for standards, it will be necessary to state whether the result obtained by a method used for analysis within conformity checks shall be expressed on an recovery-corrected basis or not.

4 Significant Figures

The units in which the results are to be expressed and the number of significant figures to be included in the reported result.

The Explanatory Notes given below will help with the interpretation of the above, particularly with respect to section 2, measurement uncertainty.

EXPANATORY NOTES

1 What is Measurement Uncertainty?

It is not always appreciated that analytical results are variable, and just how large that variability may be, particularly when low concentrations of a measurand (i.e. ppb levels) are being determined. As stated in the Codex Guidelines, most quantitative analytical results take the form of "a ± 2u" or "a ± U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "u" is the standard uncertainty to 68% level of confidence and "U" (equal to 2u) is the expanded uncertainty to 95% level of confidence.. The range "a ± 2u" represents a 95% level of confidence in which the true value would be found. The value of "U" or "2u" is the value which is normally used and reported by analysts, normally referred to as "measurement uncertainty" and may be estimated in a number of different ways.

In food analysis it is the (approximately) 95% probability (i.e. 2u) which is used to calculate the expanded uncertainty. Other sectors may specify a different probability.

Thus measurement uncertainty may be regarded as the variability around the reported results which is quantified as the value "U" when considering the expanded uncertainty and within which the "true" result should lie.

2 Does the Measurement Uncertainty have to be Estimated in Codex?

Yes, one of the requirements of the Accreditation Standard, ISO 17025:2005 that Codex has adopted by reference is that the measurement uncertainty of a result must be estimated and then made available if requested or when the uncertainty affects compliance to a specification limit, for example a Codex Standard (the Codex Alimentarius Commission has developed Guidelines which require laboratories involved in the import/export of foods to be accredited). As Codex is concerned with goods moving in international trade it would be anticipated that the request will be made.

3 Does Measurement Uncertainty Apply to both Sampling and Analysis?

Measurement uncertainty applies to the whole measurement process. For analysts only "analytical" measurement uncertainty has been considered but it is now increasingly being recognised that the whole system must be considered, and so "sampling" measurement uncertainty is gaining an increasing importance. However, this guidance only considers "analysis" but may need to be revised as the discussions on measurement uncertainty from sampling are further discussed within Codex.

4 What is the Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result?

It is the estimation of the measurement uncertainty associated with an analytical result that is important. Measurement uncertainty is not associated with a method, but the values that are obtained in the validation of a method may be used to estimate the uncertainty of a result in some situations. This differentiation between "result" and "validated method" is frequently not appreciated and so causes some confusion. Different laboratories, even if using the same (validated) method on the "same" sample may report different measurement uncertainties. This is to be expected. As a consequence precision values for a validated method (the repeatability and reproducibility values) cannot to be taken to be the measurement uncertainty without qualification. In particular additional factors such as bias, matrix effect, and competence of laboratory must be considered.

5 Procedures for Estimating Measurement Uncertainty

There are many procedures available for estimating the measurement uncertainty of a result. The Codex guidelines do not recommend any particular approach, but it is important that whatever approach is used, the procedure is scientifically credible. No one approach may be said to be better than any other provided the procedure used is appropriate and credible - i.e. there is no "hierarchy" of the recognised procedures. All such procedures may be considered to be equally valid. However, the procedure that an individual laboratory uses will have to be considered appropriate by its Accreditation Agency as part of its 17025 accreditation

In general procedures are based on a component-by-component ("bottom-up") approach or on a "top-down" approach using data from collaborative trials, proficiency studies, validation studies or intra-laboratory quality control samples, or a combination of such data..

In Codex there is a requirement to use fully validated methods and so it is usually more cost-efficient to use data from the validation rather than using another approach (i.e. the component-by-component approach). The caveats to using such validation data are best described in the Eurachem Guide to quantifying uncertainty in analytical measurement, where in Section 7.6.1 of the Second Edition of the EURACHEM Guide it is stated:

"A collaborative study carried out to validate a published method, for example according to the AOAC/IUPAC protocol or ISO 5725 Standard, is a valuable source of data to support an uncertainty estimate. The data typically include estimates of reproducibility standard deviation, *sR*, for several levels of response, a linear estimate of the dependence of *sR* on level of response, and may include an estimate of bias based on CRM studies. How this data can be utilised depends on the factors taken into account when the study was carried out. During the 'reconciliation' stage indicated above, it is necessary to identify any sources of uncertainty that are not covered by the collaborative study data. The sources which may need particular consideration are:

- **Sampling.** Collaborative studies rarely include a sampling step. If the method used in-house involves sub-sampling, or the measurand (see Specification) is estimating a bulk property from a small sample, then the effects of sampling should be investigated and their effects included.
- **Pre-treatment**. In most studies, samples are homogenised, and may additionally be stabilised, before distribution. It may be necessary to investigate and add the effects of the particular pre-treatment procedures applied in-house.
- **Method bias.** Method bias is often examined prior to or during interlaboratory study, where possible by comparison with reference methods or materials. Where the bias itself, the uncertainty in the reference values used, and the precision associated with the bias check, are all small compared to SR, no additional allowance need be made for bias uncertainty. Otherwise, it will be necessary to make additional allowances.
- **Variation in conditions:** Laboratories participating in a study may tend towards the means of allowed

ranges of experimental conditions, resulting in an underestimate of the range of results possible within the method definition. Where such effects have been investigated and shown to be insignificant across their full permitted range, however, no further allowance is required.

- **Changes in sample matrix.** The uncertainty arising from matrix compositions or levels of interferents outside the range covered by the study will need to be considered.

Each significant source of uncertainty not covered by the collaborative study data should be evaluated in the form of a standard uncertainty and combined with the reproducibility standard deviation *SR* in the usual way.

For methods operating within their defined scope, when the reconciliation stage shows that all the identified sources have been included in the validation study or when the contributions from any remaining sources have been shown to be negligible, then the reproducibility standard deviation *SR*, adjusted for concentration if necessary, may be used as the combined standard uncertainty."

These Explanatory Notes are not intended to describe the available procedures for the estimation of measurement uncertainty, but procedures have been developed by:

- ISO, in the ISO Guide to the expression of uncertainty in measurement
- Eurachem through the Eurachem Guide to quantifying uncertainty in analytical measurement, where both the component-by-component approach and the use of collaborative trial data are described.
- ISO, in the ISO TS 21748 – Guide To The Use Of Repeatability, Reproducibility And Trueness Estimates In Measurement Uncertainty Estimation
- The concept set by (EU) Commission Decision 2002/657/EC Implementing Council Directive 96/23/EC Concerning The Performance Of Analytical Methods And The Interpretation Of Results
- Using results from internal quality control data, as developed by the Netherlands Food Inspection Service.
- The Nordic Committee on Food Analysis in the NMKL Procedure Estimation and Expression of Measu rement Uncertainty in Chemical Analysis and the NMKL Procedure Measurement of Uncertainty in Qu antitative Microbiological Examinations of Foods"

The use of collaborative trial data as first described in ISO 5725 critical differences approach is not endorsed as an approach as it concentrates only on the method validation study and not on how the method is subsequently used in the laboratory.

It is recognised that further procedures for the estimation of measurement uncertainty are being developed, and that, in this evolving situation, further recommendations will be made as to acceptable procedures. It is anticipated that procedures based on results obtained from participation in proficiency testing schemes, as an example, will be developed.

References for the procedures given above are listed in section 10.

6 Considerations when Estimating Measurement Uncertainty within the Context of Codex

It is important that the requirement to estimate measurement uncertainty does not place impose any unnecessary additional workloads on laboratories.

When deciding on which procedure is to be used when estimating measurement uncertainty within the Codex context it is important to recognise that Codex has adopted a number of formal quality assurance measures which have to be implemented by control laboratories. In particular, such laboratories have to be:

- accredited to an Internationally recognised Standard (now with ISO/IEC 17025 Standard); such accreditation is aided by the use of internal quality control procedures,
- participate in proficiency schemes, and
- use validated methods.

It is essential that the information provided as a result of these requirements being implemented is used by laboratories when estimating their measurement uncertainties in order to avoid unnecessary work being

carried out by laboratories. In Codex, where there is a high emphasis being placed on the use of "fully validated" methods of analysis, i.e. methods which have been validated through collaborative trials, information obtained from such trials can be used in many situations.

In addition information derived from internal quality control procedures may also be used to estimate uncertainties in some situations.

This section re-emphasises that for the analyst it is important that no unnecessary duplication of existing work is undertaken.

7 Values of Measurement Uncertainty Estimations

Stipulating information on the anticipated values of measurement uncertainty estimations is frequently not supported by analysts. The users of analytical data and the customers of the laboratories producing such data frequently ask for such information regarding the level of uncertainty that may be expected for test results. They have concerns that some laboratories underestimate the size of their uncertainties and so report unrealistically small uncertainties to their customers.

For chemical analyses, using the values of *SR* from collaborative trials, it would not be unreasonable to anticipate that the (expanded) uncertainties reported by laboratories would be of the following orders:

Concentration	Expanded Uncertainty	Range of Acceptable Concentrations*
100g/100g	4%	96 to 104g/100g
10g/100g	5%	9.5 to 10.5g/100g
1g/100g	8%	0.92 to 1.08g/100g
1g/kg	11%	0.89 to 1.11g/kg
100mg/kg	16%	84 to 116mg/kg
10mg/kg	22%	7.8 to 12.2mg/kg
1mg/kg	32%	0.68 to 1.32mg/kg
< 100µg/kg	44%	56 to 144µg/kg

* this effectively means that values falling within these ranges may be regarded as being of the same analytical population.

For microbiological analyses, where it is frequently stated that results within the range of +/- 0.5 log units are acceptable, then the range of actual counts that this equates to is frequently much larger than customers of analytical data appreciate (or require).

It would be expected that the reported measurement uncertainties by all laboratories would not significantly exceed the value estimated from the reproducibility standard deviation (*SR*) at the concentration of interest if the laboratory is in "analytical control". Very experienced laboratories carrying out any particular analysis on a regular basis would be expected to obtain values less that the values given above.

8. Significance of the Section in the Procedural Manual of the "use of analytical results: sampling plans, relationship between the analytical results, the measurement uncertainty, recovery factors and provisions in Codex Standards" (from Codex Procedural Manual, 18th Edition)

This section attempts to explain the significance of the adopted Codex text with respect to the measurement uncertainty and recovery in particular.

8.1 Measurement Uncertainty

It is stated that an allowance is to be made for the measurement uncertainty when deciding whether or not an analytical result falls within the specification. This requirement may not apply in situations when a direct health hazard is concerned, such as for food pathogens. This does mean that it is important for Codex Commodity Committees, when setting specifications, to recognise that there is a difference between the numeric value in the specification and numeric value at which the specification will be enforced. Put simply this difference equates to the measurement uncertainty of the result obtained by the "enforcing laboratory". Thus, when enforcing a maximum limit, the enforcement laboratory (normally the importer) will have to consider the value of the measurement uncertainty before deciding whether the sample meets the specification.

This is best illustrated diagrammatically, where the figure below illustrates four different situations:

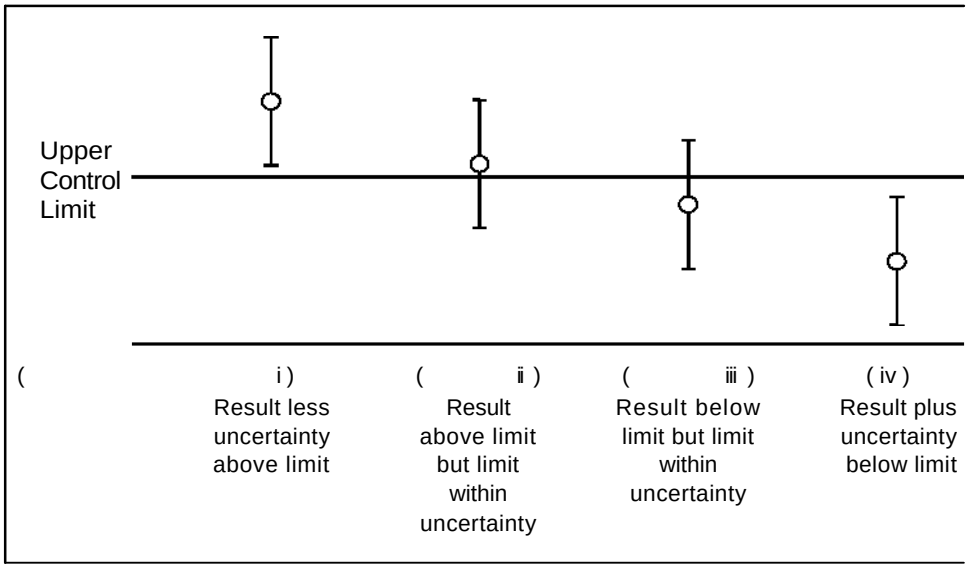

Figure: Uncertainty and compliance limits

Situation I

The analytical result together with the measurement uncertainty exceeds the maximum level. All authorities will consider the sample as being non-compliant with the specification.

Situation II

The analytical result exceeds the maximum level by less than the measurement uncertainty. Some authorities would have accepted the sample as being compliant with the specification, if they routinely take into account the measurement uncertainty. Others would have ignored the measurement uncertainty and so would not accept the sample. The effect of the accepted text is that all authorities will accept the result as being

compliant (i.e. the result is not non-compliant *"beyond reasonable doubt"*).

Situation III

The analytical result is below the maximum level by less than the measurement uncertainty. In general authorities will consider the sample to be compliant with the specification, but would probably be wary of future samples.

Situation IV

The analytical result is less than the maximum value by an amount greater than the measurement uncertainty. All authorities will consider the sample as being compliant without any hesitation.

It should be noted that the above situation will have to be interpreted with sensitivity in some instances. However, the risk of inadequate protection of the consumer may be reduced by a suitable selection of the specification – thus it is essential that the significance of measurement uncertainty deduction from the analytical result before assessing compliance is appreciated.

8.2 Recovery

It is stated that analytical results are to be expressed on a recovery corrected basis where appropriate and relevant, and when corrected it has to be stated.

If a result has been corrected for recovery, the method by which the recovery was taken into account should also be stated. The recovery rate is to be quoted wherever possible.

When laying down provisions for standards, it will be necessary to state whether the result obtained by a method used for analysis within conformity checks shall be expressed on an recovery-corrected basis or not.

The Codex Alimentarius Commission has adopted the IUPAC Guidelines on the use of recovery information by reference (see CAC/GL 37-2001).

9 Use of Measurement Uncertainty and Definition of a Dispute Situation

A dispute will arise when considering a Codex specification, which is a maximum value, if the exporter ascertains that the product meets the specification, normally taking measurement uncertainty into account, whereas the importer decides that it does not, even tasking the measurement uncertainty into account i.e.:

- the export certificate states that the analytical result to which its associated measurement uncertainty is then added is less than the Codex specification (i.e. "x + U" < L, where x is the reported analytical result, U is the expanded uncertainty and L is the Codex specification, which is a maximum limit) and so the sample meets the Codex specification, and

- the import certificate states that the analytical results to which its associated measurement uncertainty is then deducted is still greater than the Codex specification (i.e. "x – U" > L, where x is the reported analytical result, U is the expanded uncertainty and L is the Codex specification, which is a maximum limit) and so the sample does not meet the Codex specification.

This assumes that the laboratory at importation will deduct the measurement uncertainty, as implied in Section 4, above, of this guidance. If the value after deduction is still greater than the specification, then it may be stated, ***beyond reasonable doubt***, that the sample is not compliant with the specification.

It is important for the exporter to realise that in order to be sure that the exported product meets the specification the "certificated value" obtained by the producer/exported must have the uncertainty of the result added to it, and for that value to be below the specification.

10 Useful References

A number of references are given below. [NB: these are general references and do need up-dating.]

Guides for the Estimation of Measurement Uncertainty

Guide 98, Guide to the Expression of Uncertainty in Measurement (GUM) ISO, Geneva (1995).
EURACHEM/CITAC Guide Quantifying Uncertainty In Analytical Measurement (Second Edition), EURACHEM Secretariat, BAM, Berlin, 2000. This is available as a free download from http://www.eurachem.ul.pt/

Analytical Methods Committee of the Royal Society of Chemistry "Uncertainty of Measurement - Implications of its use in Analytical Science", Analyst, 1995, **120 (9)**, 2303-2308.

ISO/TS 2 1748:2004 Guidance for the Use of Repeatability, Reproducibility and Trueness estimates in Measurement Uncertainty Estimation, ISO, Geneva (2004).

NIST Technical note 1297 (1994 Edition): "Guidelines for Evaluating and Expressing the Uncertainty of NIST Measurement Results"

NMKL Procedure No. 5, 2nd edition (2003): "Estimation and Expression of Measurement Uncertainty in Chemical Analysis"
UKAS (United Kingdom Accreditation Service) 2000 The Expression of Uncertainty in Testing Edition 1, UKAS Publication ref: LAB 12

Eurolab technical Report No. 1/2007. Measurement Uncertainty Revisited: Alternative Approaches to Uncertainty Evaluation. Available as a free download from www.eurolab.org

Nordtest report TR 537. Handbook for Calculation of Measurement Uncertainty in Environmental Laboratories. Available as free downloads from www.nordtest.org (although this handbook is directed towards environmental analyses, the approaches and examples described are applicable to the results from tests on foods and feeds)

Procedures for the Validation of Analytical Methods and Method Performance

"Precision of Test Methods", Geneva, 1994, ISO 5725, Previous editions were issued in 1981 and 1986. (not adopted by Codex).

"Protocol for the Design, Conduct and Interpretation of Method Performance Studies", ed. W. Horwitz, *Pure Appl. Chem.*, 1995, 67, 33 1-343. (adopted by Codex).

European Commission Decision 2002/657/EC implementing directive 96/23/EC Concerning the Performance of Analytical Methods and the Interpretation of Results, Off J Eur Comm, L22 1 (2002) 8-36.

T.P.J. Linsinger, R.D. Josephs: Limitations of the application of the Horwitz

Validation of Chemical Analytical Methods. NMKL Procedure No 4, 3rd Version, 2009

Accreditation etc

ISO/IEC 17025:2005, General Requirements for the Competence of Testing and Calibration Laboratories, ISO, Geneva (2005).

EURACHEM Guidance Document No. 1/WELAC Guidance No. WGD 2: "Accreditation for Chemical

Laboratories: Guidance on the Interpretation of the EN 45000 series of Standards and ISO/IEC Guide 25"

Z., Ben-David, H., Mates, A. 2001 Proficiency testing as tool for ISO 17025 implementation in National Public Health Laboratory: a mean for improving efficiency. Accreditation & Quality Assurance, **6**: 190-194

NMKL Procedure no. 3 (1996) "Control charts and control samples in the internal quality control in chemical food laboratories"
Örnemark, U., Boley, N., Saeed, K., van Berkel, P.M., Schmidt, R., Noble, M., Mäkinen, I., Keinänen, M., Uldall, A., Steensland, H., Van der Veen, A., Tholen, D. W., Golze, M., Christensen, J.M., De Bièvre, P., De Leer, W. B (ed). 2001

Proficiency testing in analytical chemistry, microbiology, and laboratory medicine – working group discussions on current status, problems, and future directions. Accreditation & Quality Assurance, **6**: 140-146.

Compliance

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