EURACHEM SYMPOSIA: SAMPLING UNCERTAINTY AND UNCERTAINTY FOR COMPLIANCE ASSESSMENT

How legislators interpret specifications within the food sector with respect to measurement uncertainty

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MEASUREMENT UNCERTAINTY – ANALYSIS and now SAMPLING

Measurement uncertainty has been a contentious issue for analytical chemists for many years. It has been considered in a number of papers. In the majority of cases these papers have concentrated on how measurement uncertainty is to be estimated.

It is only recently that there have been discussions on how measurement uncertainty is to be used.

And looking forward, we now need to consider sampling uncertainty!

TO COVER

EU Report in the Food Sector

Codex Stipulations

Examples in Legislation

Problems when Not Specified

Precautionary approach - “wrong way round”?

EU REPORT:

THE RELATIONSHIP BETWEEN THE FINAL ANALYTICAL RESULT AND THE SAMPLING, THE MEASUREMENT UNCERTAINTY AND THE RECOVERY FACTOR USED TO OBTAIN THAT RESULT

These factors affect the relationship between the final analytical result and the provisions in legislation

Decisions taken by those responsible for the enforcement of legislation directly affect decisions as to whether a lot is in compliance with that legislation.

SCIENTIFIC CO-OPERATION TASK 9.1

"PREPARATION OF A WORKING DOCUMENT IN SUPPORT OF THE UNIFORM INTERPRETATION OF LEGISLATIVE STANDARDS AND THE LABORATORY QUALITY STANDARDS PRESCRIBED UNDER DIRECTIVE 93/99/EEC"

was initiated to identify differences amongst Member States.

14 participated. Final Report is now published.

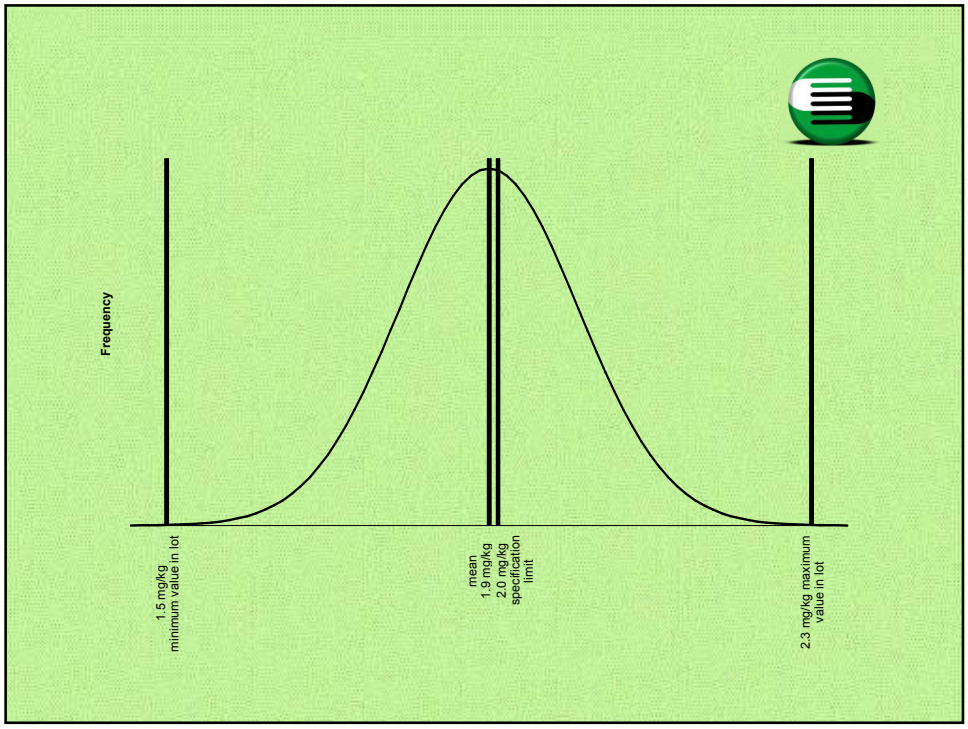
MAJOR ISSUES IDENTIFIED

The basic principles of the sampling procedures used by The Member States, the treatment of analytical variability (normally known as the measurement uncertainty) in the interpretation of an EU specification, and the use of recovery corrections when calculating and reporting analytical results were found to be different,

The effect of different countries taking different approaches for each of the issues identified are described. It must be appreciated that there may be other enforcement issues which have a similar effect.

There was no common interpretation of analytical results across the EU in the food sector so significantly different decisions may be taken after analysis of the "same sample". Material for which there is a statutory limit of, say, 4μg/kg for a contaminant (e.g. total aflatoxins) may be interpreted as containing 3μg/kg on analysis in one country but 8 μg/kg in another. This is because some countries correct analytical results for recovery, others do not; some countries use an "every-item-must-comply" sampling regime, others may use an "average of a lot" regime, some make an allowance for measurement uncertainty, others do not.

It is essential that interpretation of analytical results is similar if there is to be equivalence across the EU; without it there is no uniform interpretation of legislation.



Two countries may have different national rules for the interpretation of results from lots.

Country A requires that each and every item in the lot meets the specification. In this example it means that all 1,000 units, if analysed separately, would have to be less than 2.0 mg/kg. Here a significant number of units are greater than 2.0 mg/kg so the lot would be deemed to be in non-compliance with the legal specification and so would be rejected.

Country B requires that the mean value of the specification in the lot is to be less than the legal specification. In this case the mean value is 1.9 mg/kg so the lot would be deemed to be in compliance with the legal specification.

Consequence: the two countries A and B will make different judgements as to compliance with a legal specification on essentially the same lot. This is unacceptable and can only be avoided if the sampling procedures are elaborated at the same time as the commodity standard is elaborated. In addition it should also be noted that the number of units to be analysed also influences the decision on compliance.

REPORTING OF RESULTS WITH RESPECT TO THEIR MEASUREMENT UNCERTAINTY

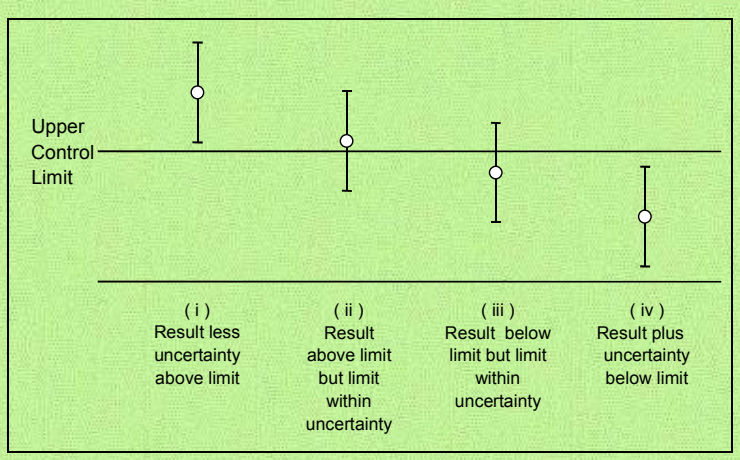
All analytical results should be reported in the form "a $\pm$ U" where "a" is the best estimate of the true value of the concentration of the measurand (the analytical result) and "a-U" to "a+U" is the range within which the true value is estimated, with a given probability, to fall. The value of "U", the (expanded) "measurement uncertainty", may be estimated by the analyst in a number of different ways.

REPORTING OF RESULTS BY FOOD CONTROL ANALYSTS

The procedure adopted by some food control analysts is to report samples as containing “not less than “a” – “U”” in situations where the statutory limit is a maximum permissible concentration. Thus, in any enforcement situation the maximum benefit is given to the food producer. This is consistent with the requirement to prove ***beyond reasonable doubt*** that a limit has been exceeded, if the case should come to Court. This does mean that the effective enforcement limit is, in such countries, not identical to the numerical value given in legislation.

Other food analysts may report the value “a” without taking into account any measurement uncertainty considerations when assessing compliance with a specification.

This was found to be the case in the SCOOP Task



This means that the legal specification and enforcement limit are different.

This should be appreciated when specification is being set.

REPORT TO THE STANDING COMMITTEE ON THE FOOD CHAIN AND ANIMAL HEALTH

ON THE RELATIONSHIP BETWEEN ANALYTICAL RESULTS, THE MEASUREMENT UNCERTAINTY, RECOVERY FACTORS AND THE PROVISIONS IN EU FOOD AND FEED LEGISLATION WITH PARTICULAR FOCUS ON THE COMMUNITY LEGISLATION CONCERNING:

- **CONTAMINANTS IN FOOD (COUNCIL REGULATION (EEC) No 315/93 OF 8 FEBRUARY 1993 LAYING DOWN COMMUNITY PROCEDURES FOR CONTAMINANTS IN FOOD[1])**

[1] Official Journal of the European Communities, L37, 13.2.1993, p. 1

- **UNDESIRABLE SUBSTANCES IN FEED (DIRECTIVE 2002/32/EC OF THE EUROPEAN PARLIAMENT AND OF THE COUNCIL OF 7 MAY 2002 ON UNDESIRABLE SUBSTANCES IN ANIMAL FEED[2])**

[2] Official Journal of the European Communities, L 140, 30.5.2002, p. 10

Website Address

http://europa.eu.int/comm/food/food/chemicalsafety/contaminants/report-sampling_analysis_2004_en.pdf

and

http://europa.eu.int/comm/food/food/animalnutrition/sampling/index_en.htm

Recommendations from EU Report

- The Working Group which discusses specifications in legislation and any associated method performance criteria, should also discuss the maximum measurement uncertainty which may be accepted as being fit-for-purpose.

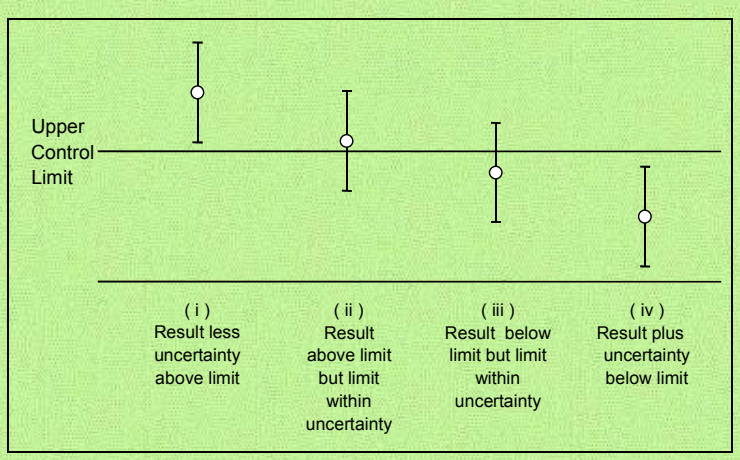
- Enforcement Authorities shall use the measurement uncertainty associated with an analytical result when deciding whether an analytical result falls within the specification or not for food and feed control purposes. The way that measurement uncertainty is to be used by Enforcement Authorities must be taken into account when analytical specifications are discussed. In practice, the analyst will determine the analytical level and estimate the measurement uncertainty at that level. The value obtained by subtracting the uncertainty from the reported concentration, is used to assess compliance. Only if that value is greater than the maximum level in legislation, it is sure "beyond reasonable doubt" that the sample concentration of the analyte is greater than that prescribed by legislation.

Values of Measurement Uncertainty Estimations

There is concern that some laboratories underestimate the size of their uncertainties and report unrealistically small uncertainties to their customers.

For chemical analyses, using the results 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 means that the legal specification and enforcement limit are different.

This should be appreciated when specification is being set.

Similar considerations identified in the Codex Alimentations Commission

Here a section on:

"The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards"

has been approved and is included in Procedural Manual

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 across the Codex Community. 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 analytical results are interpreted in the same way if there is to be equivalence across the Codex Community.

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 to control conformity of products with the specification. This should state:

- whether the specification applies to every item in a lot, to the average in a lot or the proportion nonconforming;
- 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

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.

3. Recovery

Where relevant and appropriate the analytical results are to be reported on a recovery corrected basis and that the recovery should be quoted in any analytical report. Analytical results are to be expressed on a recovery corrected basis where appropriate and relevant, **and when corrected it has to be so stated.**

In all cases it has to be stated when the result is corrected for recovery.

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.

CODEX GUIDELINES ON MEASUREMENT UNCERTAINTY

(CAC/GL 54-2004)

Introduction

It is important and required by ISO/IEC 17025:1999 that analysts are aware of the 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 where the true value would be found. 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.

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.

Use of Measurement Uncertainty and Definition of a Dispute Situation

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.

TYPICAL LEGISLATION IN EU FOOD SECTOR

COMMISSION REGULATION (EC) No 401/2006 of 23 February 2006 laying down the methods of sampling and analysis for official control of the levels of the mycotoxins in foodstuffs

Acceptance of a lot or sublot

Acceptance: if the laboratory sample conforms to the maximum limit, taking into account the correction for recovery and measurement uncertainty;

Rejection: if the laboratory sample exceeds the maximum limit beyond reasonable doubt taking into account the correction for recovery and measurement uncertainty.

This form of legislation is replicated in sector as it is reviewed, discussed in Brussels.

e.g. for metals, PAHs, patulin etc etc.

Ill-Defined Situations

UK Animal Feeding Stuffs Regulations 2000

For ash, ash insoluble in hydrochloric acid, calcium, cystine, fibre, lysine, magnesium, methionine, moisture, oils and fats, phosphorus, potassium, protein, protein equivalent of biuret, diureidoisobutane, urea or urea phosphate, sodium, starch and total sugar plus starch,threonine, total sugar, tryptophan

tolerances are given.

Limits of Variation

Ash:

If present in excess -

2% (absolute) for declarations of 10% or more

20% of the amount stated for declarations of 5% or more but less than 10%

1% (absolute) for declarations less than 5%

Limits of Variation

These are not defined – manufacturing and analytical?

Suspect both in one figure as how treated/regarded historically!

LACORS GUIDANCE ON TOLERANCES TO BE APPLIED TO NUTRITION LABELLING DECLARATIONS : NOVEMBER 2007

Sodium

Declared Value: greater than 0.5%

Recommended Maximum Variation: +/- 30% (of declared value)

Declared Value: less than or equal to 0.5%

Recommended Maximum Variation: +/- 0.15g

DESCRIPTION

The revised tolerances which are more accurately identified as “recommended maximum variation “ include analytical/measurement uncertainty in respect of the determination of the values to be declared (as distinct from measurement uncertainty of any method used for enforcement purposes, for which a result would need to be corrected before concluding that a sample is outside the tolerance).

Double counting likely?

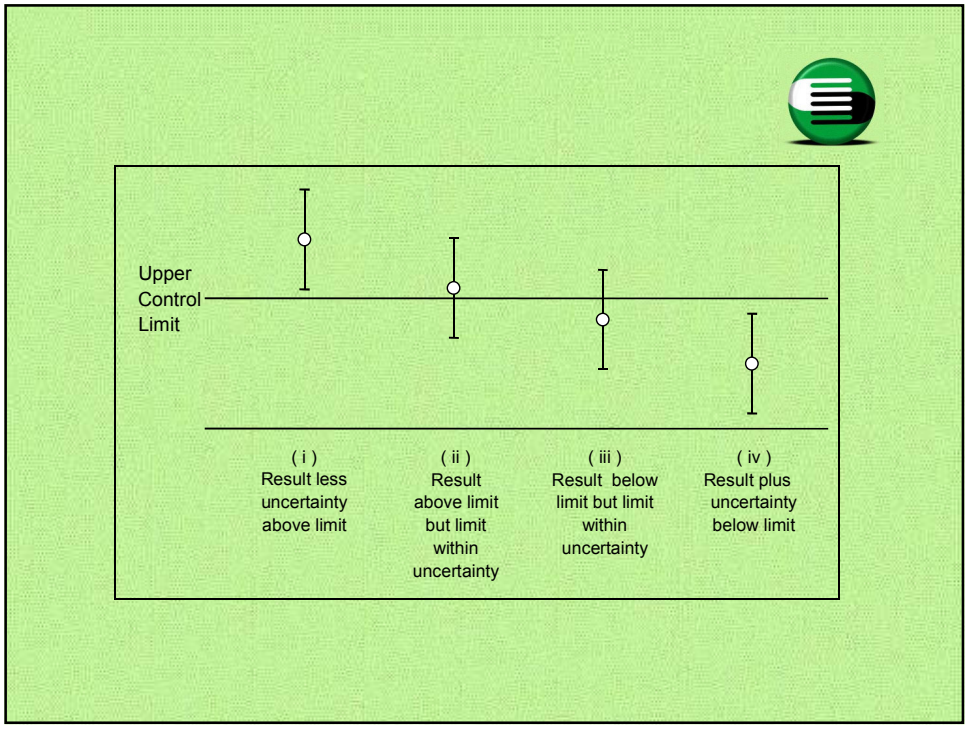
PRECAUTIONARY APPROACH

Proposed that HPLC method for the detection of paralytic shellfish poisoning in England be introduced to replace the current MBA method.

see:
http://www.food.gov.uk/consultations/consulteng/2008/hplcengland

PRECAUTIONARY APPROACH

To report the toxicity of a sample as a single value in micrograms Saxitoxin (STX) equivalent per kilogram shellfish tissue, which would equate to the upper limit of the uncertainty range of the HPLC method. This proposed reporting format reflects the measurement uncertainty associated with the extraction and HPLC methodology


Upper
Control
Limit
(i)
Result less uncertainty above limit
(ii)
Result above limit but limit within uncertainty
(iii)
Result below limit but limit within uncertainty
(iv)
Result plus uncertainty below limit