



Methodological Developments in the Codex Alimentarius Commission, EU and in CEN

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CODEX ALIMENTARIUS COMMISSION

Has recently adopted:

- Guidelines On Analytical Terminology Adopted By The Codex Alimentarius Commission, July 2009 (49 terms are defined).
- Draft Guidelines for Settling Disputes on Analytical (Test) Results (limited applicability for import/export situations).
- Guidelines for establishing methods criteria for the identification of relevant analytical methods – procedures given for developing detection/quantification limits, precision, range of the method (related to the legal limit), recovery etc.



Current Work:

Guidelines on criteria for methods for the detection and identification of foods derived from biotechnology

Guidelines for establishing methods criteria for the identification of relevant analytical methods

Revised guidelines on measurement uncertainty – simple explanation of significance

Guidance on uncertainty from sampling

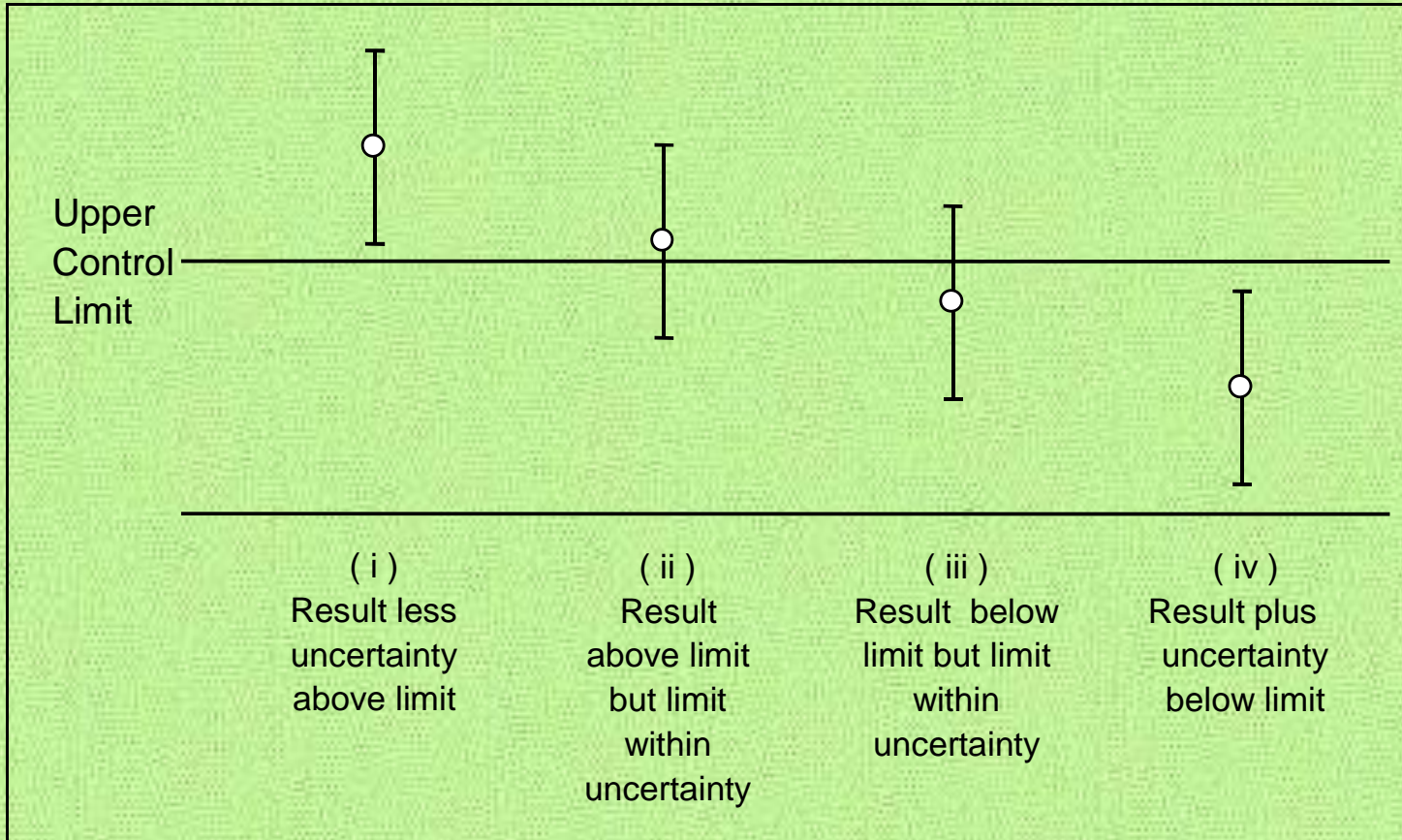


Revised guidelines on measurement uncertainty – simple explanation of significance

- What is Measurement Uncertainty?
- Does the Measurement Uncertainty have to be Estimated in Codex?
- Does Measurement Uncertainty Apply to both Sampling and Analysis?



- What is the Relationship between Measurement Uncertainty, the Analytical Result and the Method Used to Obtain the Result?
- Procedures for Estimating Measurement Uncertainty
- Values of Measurement Uncertainty Estimations
- 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, 17th Edition) (see diagram)





Draft Guidelines on Measurement Uncertainty Including Uncertainty from Sampling – simple explanation of significance

As previous and:

- procedures for estimating total measurement uncertainty. Duplicate method recommended. 8 duplicates, duplicate measurements on each duplicate.
- very significant
- difficult to demonstrate/explain to the inexperienced (even more so than the normal measurement uncertainty).



THREE EXAMPLES FROM THE FOOD SECTOR FOR TOTAL MEASUREMENT UNCERTAINTY ESTIMATES



Example 1 – Nitrate concentration in glasshouse lettuce

All values given in mg kg⁻¹

Mean: 4346

Standard deviation of analysis : 167.2

Standard deviation of sampling : 448.0



Example 2 – infant wet meals (retail survey)

All values given in $\mu\text{g kg}^{-1}$

Mean: 7.7

Standard deviation of analysis : 1.754

Standard deviation of sampling : 0.689



Example 3 – Moisture in wholesale butter (offered for EU subsidy)

All values given in g 100g⁻¹

Mean: 15.75

Standard deviation of analysis : 0.041

Standard deviation of sampling : 0.219



Upper
Control
Limit



(i)
Result less
analytical and/or
total uncertainties
above limit

(ii)
Result less analytical
uncertainty is above
limit, but limit
is within total
uncertainty

(iii)
Result above limit
but limit within both
analytical and total
uncertainties

(iv)
Result below limit
but limit
within both
analytical and total
uncertainties

(v)
Result below limit but
limit plus analytical
uncertainty
still below limit but within
total uncertainty

(vi)
Result plus
analytical or total
uncertainties below
limit



Upper
Control
Limit



(i)

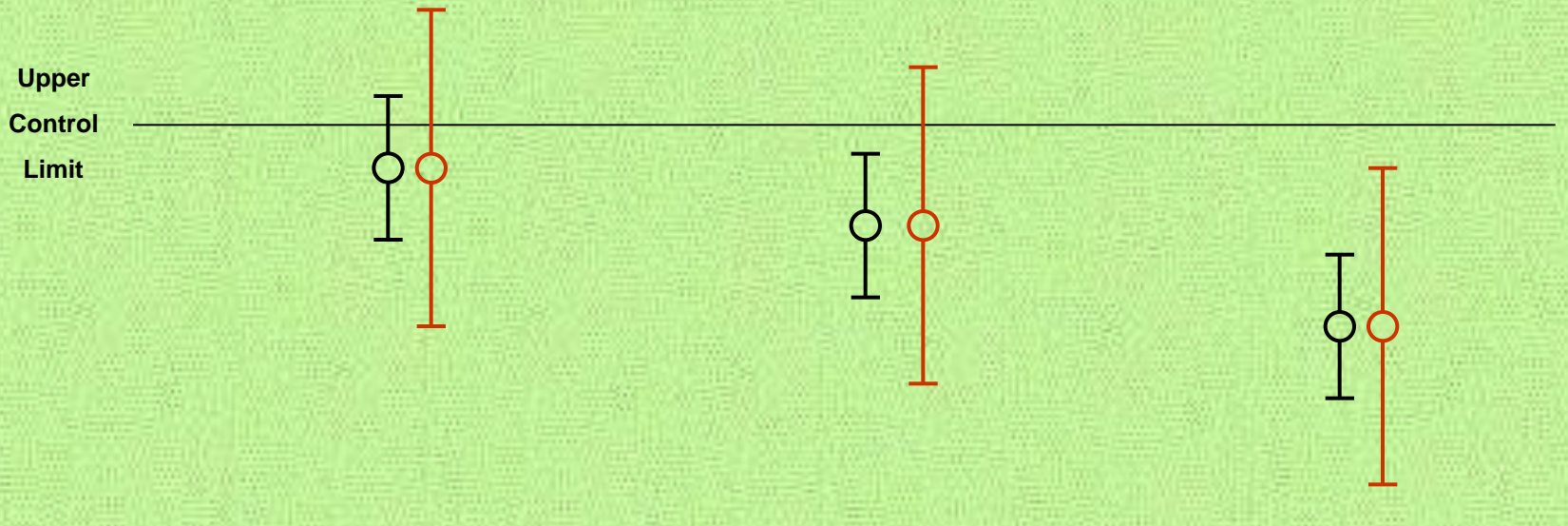
Result less analytical and/or total
uncertainties above limit

(ii)

Result less analytical
uncertainty is above limit, but limit
is within total uncertainty

(iii)

Result above limit but
limit within both
analytical and total
uncertainties



(iv)
Result below limit but limit
within both analytical and total
uncertainties

(v)
Result below limit but limit
plus analytical uncertainty
still below limit but within total
uncertainty

(vi)
Result plus analytical or total
uncertainties below limit



**WHAT IS THE CRITERIA APPROACH TO
METHODS OF ANALYSIS?**

WHY INTRODUCE IT – ANY PROBLEMS?



Need to Consider:

Codex, EU, CEN and Methods of Analysis

Criteria approach

Criteria for Acceptable Methods of Analysis



Traditional Approach (prescribing a specific method of analysis) means:

- The analyst is denied freedom of choice and thus may be required to use an inappropriate method in some situations;
- The procedure inhibits the use of automation; and
- It is administratively difficult to change a method found to be unsatisfactory or inferior to another currently available.



But is easier for the analyst!

Previously common approach in Codex and EU (still is in Codex)



Criteria Approach (prescribing performance characteristics) means:

- giving greater flexibility than the present procedure adopted by organisations such as Codex and the EU
- not being in the situation of having many methods of analysis which are available, which meet requirements as regards method performance characteristics, but which are not considered by Codex or the EU because of time constraints.



Only applicable to rationale methods, not to empirical methods (I.e. where the result is method dependent)

Need to define these better?



Regulations in EU based on Criteria Approach

Where no specific methods for the determination of contaminants in foodstuffs are prescribed at Community level, laboratories may select any validated method of analysis (where possible, the validation shall include a certified reference material) provided the selected method meets the specific performance criteria set out in the Regulation.

Note criteria approach in EU food sector first used 1998 Mycotoxin Directive; first used in UK food sector in 1994 Regulations. No problems identified.



Specified Criteria

Tend to be:

LOD

LOQ

Precision

Recovery

Selectivity



Fitness-for-purpose' Approach (Uncertainty Function Approach)

Where a limited number of fully validated methods of analysis exist, alternatively, a fitness-for-purpose' approach may be used to assess the suitability of the method of analysis. Methods suitable for official control must produce results with standard measurement uncertainties less than the maximum standard measurement uncertainty calculated using the formula below:



$$Uf = \sqrt{(CL/2)^2 + (aC)^2}$$



where: U_f is the maximum standard uncertainty

CL is the detection limit of the method

C is the concentration of interest

a is a numeric factor to be used depending on the value of C.

Results with an uncertainty less than that stipulated above will be produced by a method which is equivalent to one meeting the previous performance characteristics.



C ($\mu\text{g}/\text{kg}$)	a
$a \leq 50$	0.25
1 to 500	0.18
501 to 1,000	0.15
1,001 to 10,000	0.12
$> 10,000$	0.1



Tin at 100 mg/kg

$$Uf = ((5/2)^2 + (0.1*100)^2)^{0.5}$$
$$= (6.25 + 100)^{0.5}$$

Expanded uncertainty = 20.6



eg Pb at 100 $\mu\text{g}/\text{kg}$

$$U_f = ((10/2)^2 + (0.18*100)^2)^{0.5}$$

$$= (25 + 349)^{0.5}$$

$$= 18.7$$

Expanded uncertainty = 37.4



Measurement Uncertainty in Regulation

The analytical result shall be reported as $x \pm U$ whereby x is the analytical result and U is the expanded measurement uncertainty, using a coverage factor of 2 which gives a level of confidence of approximately 95 % ($U = 2u$).

The analyst shall note the Report on the relationship between analytical results, measurement uncertainty, recovery factors and the provisions in EU food and feed legislation' (1).



INTERPRETATION OF RESULTS

Acceptance of a lot/sublot

The lot or sublot is accepted if the analytical result of the laboratory sample does not exceed the respective maximum level as laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.



Rejection of a lot/sublot

The lot or sublot is rejected if the analytical result of the laboratory sample exceeds beyond reasonable doubt the respective maximum level as laid down in Regulation (EC) No 1881/2006 taking into account the expanded measurement uncertainty and correction of the result for recovery if an extraction step has been applied in the analytical method used.



Is this difficult to understand – sampling officers?



Need to be able to differentiate between rational and empirical (defining) methods

Proprietary methods and gluten situation.



CEN REPORT ON INFORMATION TO AND PROCEDURES FOR CEN TC 275 WORKING GROUPS TO CONSIDER WHEN SPECIFIC STANDARDS ARE BEING DEVELOPED AND ADOPTED BY THE TC AND ITS WORKING GROUPS

REPORT TO BE PREPARED BY CEN TC 275, WG 0

PROBLEMS TO BE CONSIDERED

Use of recovered data for calculation of method performance characteristics

The use of robust statistics when calculating performance characteristics



Assessment of method performance statistics - use and definition of the Horrat Value

Full collaborative trial not possible/or practical/or too expensive etc

Measurement uncertainty within CEN/TC 275 standards

Extension of method scopes - Extension of the scope of specific methods in existing CEN Standards

Extension of method scopes - Format of scopes in published CEN /TC 275 methods Modular validation of methods of analysis



Additional Performance Parameters

Problems with the Inclusion of Proprietary Aspects in CEN Standards

Expression Of Analytical Results And Rounding Rules

Validation of Qualitative Methods

Method Verification Procedures



CONCLUSIONS

- Criteria approach well established
- May lead to a fitness approach
- Need to understand the method performance characteristics
- Need to be able to differentiate between rational and empirical (defining) methods
- Sampling uncertainty likely to cause us all problems.