2013 FERA-JIFSAN ANNUAL SYMPOSIUM:
ANALYTICAL METHODS AND LABORATORY
PRACTICES FOR INTERNATIONAL FOOD SAFETY

HOW THE ANALYST IS AFFECTED BY INITIATIVES IN
THE CODEX ALIMENTARIUS COMMISSION

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A number of analytical and sampling considerations prevent the uniform implementation of Codex and national legislative standards. These include:

- How the specification is written in Codex Standard or in legislation – unambiguously described units. Number of significant figures in the specification.

- Sampling procedures need to be defined (acceptance (– variables, attributes, AQL), uncertainty of sampling, pragmatic sampling etc)

- Methods of analysis to be defined if empirical methods (Codex Type I methods)
WHAT CODEX NEEDS TO ACHIEVE - 2

• Methods of analysis or method criteria to be defined if rational methods (Codex Type II and III methods)

• Are quality standards to be applied to sampling – certification of samplers?

• Are quality standards to apply to laboratories?
  • Accreditation
  • Proficiency Testing
  • Internal Quality Control

• Are results to be expressed on a recovered or unrecovered basis?
WHAT CODEX NEEDS TO ACHIEVE - 3

• How is measurement uncertainty to be treated or estimated. Is “beyond reasonable doubt” uniformly applied?

• Quality of methods of analysis to be defined – fully validated?

• The use of proprietary methods for standardisation and official purposes?

• Method verification (use of CRMs etc)

• Tolerances – need to consider both analysis and intentional manufacturing tolerances?
TIME LINE FOR WHAT CODEX HAS ACHIEVED - 1

1976: General Principles for Sampling and Analysis

1987: Endorsement of IUPAC/AOAC/ISO Collaborative Trial Protocol

1993: Endorsement of The International Harmonised Protocol for the Proficiency Testing of (Chemical) Analytical Laboratories

1995: Endorsement of the (IUPAC) Harmonised Guidelines for Internal Quality Control in Analytical Chemistry Laboratories

1997: Publication of the Guidelines for the assessment of the competence of testing laboratories involved in the import and export control of food
TIME LINE FOR WHAT CODEX HAS ACHIEVED - 2

1999: Endorsement of the Harmonised guidelines for the use of recovery information in analytical measurement

2002: Endorsement of the (IUPAC) Harmonised guidelines for single-laboratory validation of methods of analysis

2004: Publication of the Codex General Guidelines on Sampling


2007: Adopted instructions on “The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”
TIME LINE FOR WHAT CODEX HAS ACHIEVED - 3

2008: Adopted instructions on Working Instructions for the Implementation of the Criteria Approach in Codex

2009: Publication of the Guidelines on Analytical Terminology

2009: Publication of the Guidelines for settling disputes over analytical (test) results

2010: Publication of the Guidelines on performance criteria and validation of methods for detection, identification and quantification of specific dna sequences and specific proteins in foods

2012: Publication of provisions on the use of proprietary methods in Codex Standards
WHAT THIS MEANS – 1 (Method Quality)

1976: General Principles for Sampling and Analysis

Distinction between empirical methods (Codex Type I) and rationale methods (Codex Type II/III Methods)

Performance attributes for methods have to be estimated (but not formally assessed)

2008: Adopted instructions on Working Instructions for the Implementation of the Criteria Approach in Codex

Values of LoD, LoQ, recovery, precision (HorRat) all relate to the given specification in the Codex Standard – i.e. performance attributes for methods now being formally assessed.
WHAT THIS MEANS – 2 (Method Quality)

2002: Endorsement of the (IUPAC) Harmonised guidelines for single-laboratory validation of methods of analysis

Codex Type IV – candidate methods (cf AOAC ERP First Action methods?)

Recovery?
WHAT THIS MEANS – 3 (Laboratory Quality)

1997: Publication of the Guidelines for the assessment of the competence of testing laboratories involved in the import and export control of food

- Accreditation
- Proficiency Testing
- Internal Quality Control
WHAT THIS MEANS – 4 (Result Quality)


2007: Adopted instructions on “The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”

Text is:
CODEX ALIMENTARIUS COMMISSION

instructions on:

“The Use of Analytical Results: Sampling, Relationship Between the Analytical Results, the Measurement Uncertainty, Recovery Factors and the Provisions in Codex Standards”
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.
The diagram illustrates the frequency of values within a specified range. The minimum value in the lot is 1.5 mg/kg, the mean is 1.9 mg/kg, the specification limit is 2.0 mg/kg, and the maximum value in the lot is 2.3 mg/kg.
(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
(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
Result below limit but limit within both analytical and total uncertainties

Result below limit but limit plus analytical uncertainty still below limit but within total uncertainty

Result plus analytical or total uncertainties below limit

(iv)

(v)

(vi)
OTHER ISSUES


2. Sampling to be looked at (three ways – acceptance sampling, uncertainty of sampling and auto-control)

3. Protocol on Qualitative Methods of Analysis.

4. Alternative pathway to validation of methods, e.g. use of PT data?

5. Validation of on-line procedures.
CONCLUSIONS

1. Codex has been instrumental in endorsing/adopting quality aspects for methods of analysis, methods of sampling and for laboratories.

2. Codex has been instrumental in defining how results are to be used, *cf* measurement uncertainty.

3. Codex has been instrumental in giving analyst freedom but at the cost of needing knowledge.

4. Think next issue will be sampling and all that entails – the forgotten aspect?