
Understanding Codex's problems with
method selection and performance and
how these may be overcome by the
increased use of the criteria approach

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Or: How to find analytical
methods that live up to the
relevant analytical criteria

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Background:

- Past and present - Codex endorsed methods :
 - + easily available,
 - fixed methods, inflexible,
 - maybe not fit for specific purposes, e.g. ;
 - low concentrations
 - specific matrices

Background continued:

- Just (2009) introduced: A system for the establishment of analytical performance criteria, making it possible to:
 - find methods fit for specific purposes, e.g. :
 - legal/trade requirements
 - unusual matrices
 - limitations in instrumentation

The new criteria approach: Method performance characteristics used for defining criteria

- » **precision (s_r and s_R)**
 - » **lowest validation level**
 - » **limit of detection (LOD)**
 - » **(trueness, for “total” methods)**
 - » **(recovery, for extraction methods)**
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The Criteria Approach is found in:

Codex Alimentarius Commission

Procedural Manual

Nineteenth edition
Section II

Principles for the Establishment of Codex Methods of Analysis

The criteria approach is applicable to type II and type III methods.

- Type I = Defining, or empirical, method (results are method dependent)

- Type II = Reference methods. Recommended for use in cases of dispute and for calibration purposes

- Type III = Alternative approved methods. May be used for control, inspection and regulatory purposes

- Type IV = Traditional or new method for which acceptance criteria have not been established (by CCMAS)

What are the needs

- Legal limits
- Banned substances
- To facilitate export
- Method for specific instrumentation

Where to find methods?

- AOAC
- NMKL
- CEN – ISO
- Branch organisations
 - IDF
 - OIV
 - ?
- Open literature (journals, books)
- DEFRA (MAFF), DIN
- Start with CODEX (e.g. STAN 234)

AOAC

AOAC.org

- Preview of method summary, sometimes performance criteria shown
- Method may refer to previous publications for analytical/validation details
- Full access to methods only for AOAC members

NMKL

NMKL.org

- Preview of method summary, sometimes performance criteria shown
- Each method for sale

CEN - ISO

CEN.eu

- Methods/Standards sold only by the standardisation bodies in the member states
- MS websites sometimes complicated, UK one of the better (in my view)
- CEN-methods are often originating from AOAC or NMKL

Please remember:

CODEX doesn't sit on a pile of methods! It only has a register with the references.

You have to look up each potential method at its source and check the performance data.

Example 1:

A codex committee want to control the maximum level of lead in juice, which is 0.05 mg/kg :

Criteria procedure:
Procedural manual 19th ed.
Page 53 – Table 1

A method for the determination of lead at a ML of 0.05 mg/kg must have:

- A lowest validation level of 0.03 mg/kg ($0.05 \pm 2S_R$)
- **Or** a LoD of ≤ 0.01 mg/kg ($0.05 \times 1/5$)
- **And** a RSD_R of $\leq 44\%$ (i.e. a Horrat ≤ 2)
- Trueness/Bias (*no extraction or preconcentration*)

Method performance characteristics are either statistically very precise (S_R/S_r) or rather pragmatic (lowest val. level)

Methods for lead in CXS 228-2001

FOOD	METHOD	Assessed level	LOD	RSD _R
All foods (except fats and oils)	NMKL 139 (1991) AOAC 999.11 AAS-method	≥0.045 mg/kg	0.01 mg/kg	8-47 %
All foods (except Fats and oils)	NMKL 161 (1998) AOAC 999.10 AAS-method	≥0.005 mg/kg	0.014-0.055 mg/kg	26-40 %
All foods	AOAC 986.15	≥0.03 mg/kg	0.03 mg/kg	17-106 %
All foods	AOAC 972.25	≥2.2 mg/kg	?	4.9-36 %
All foods (except Fats and oils)	AOAC 982.23	≥ 0.08 mg/kg	0.025 mg/kg	13-38 %

Method for lead not in CXS 228-2001

FOOD	METHOD	Assessed level	LOD	RSD _R	Bias
All foods	NMKL 186 (2007) ICP-MS	≥0.013 mg/kg (0.03)	0.01 mg/kg (0.01)	12-47 % (41 %)	Not significant

The method is collaboratively tested

Example 2:

Another codex committee want to control tetracycline at 200 µg/kg in cattle muscle:

Criteria procedure:
Procedural manual 19th ed.
Page 53 – Table 1

A method for the determination of tetracycline at a MRL of 200 µg/kg must have:

- A lowest validation level of 70 µg/kg ($200 \pm 3S_R$)
- **Or** a LoD of ≤ 20 µg/kg ($200 \times 1/10$)
- **And** a RSD_R of $\leq 41\%$ (i.e. a Horrat ≤ 2)
- Recovery 80 – 110 %

Methods for tetracycline in cattle muscle (CODEX Compendium of methods for veterinary drug residues)

METHOD	Assessed level	LOD	RSD _R	Recovery
AOAC 995.09	≥130 mg/kg (70)	-	11 - 27 % (41%)	57 – 79 % (80 – 110)

No other method found in AOAC, NMKL or CEN

(NFA uses its own in-house developed and validated method)

The NFA-method for tetracycline is published
(Analytica Chimica Acta, 637 (2009) 87-91)

It is validated (type III-method) and used in the official control

LOD = 8 (20)

RSD = 11% (<41%)

Recovery 86% (80-110%)

Summary

It may require a lot of work to find a suitable method.

May need some "detective" competence

Analytes can be difficult to identify (summarised/grouped headings)

"Older" methods may have validations that are difficult to read/comprehend and could be incomplete

Difficult to see the performance criteria if they are not listed in the method summary