Nordic Committee on Food Analysis
www.nmkl.org

Codex Method Performance Criteria & AOAC Standard Method Performance Requirement (SMPR)

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- Network of experts
- Analytical methods
- Guidelines
- Courses/Seminars

- FAO/WHO Food Standard
- 185 member countries
- 208 observers (GO, NGO)

Content

- Background of Codex Method Performance Criteria & AOAC Standard Method Performance Requirement (SMPR)
- The Criteria / SMPR
- Application of Criteria / SMPR
- AOAC New Pathway for Official Methods of Analysis

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Background for the Codex Alimentarius' method criteria

- Criticism on endorsing specific analytical methods in Codex standards:
  - 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
  - it is administratively difficult to change a method found to be unsatisfactory or inferior to another currently available

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Background for the Codex Alimentarius' method criteria

- Advantages with criteria" approach :
  - greater flexibility
  - In some areas of food analysis there are many methods of analysis which are available, which meet Codex requirements as regards method characteristics, but which are not considered by CCMAS and the Commission because of time constraints on the Committee, and
  - The adoption of a more generalised approach would ensure that such methods are brought into the Codex system and does not disadvantage developments being undertaken elsewhere in the analytical community.

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Background for the SMPR

- No longer method committees
- When there is a need for a method, AOAC calls the right persons together
- Calling for methods
- SMPR elaborated for choosing the right methods for validation and inclusion in OMA

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Method Criteria/Requirements: numeric values of method performance characteristics

- **applicability (analyte, matrix, conc. range)**
- selectivity
- sensitivity
- linearity
- **precision (s_r and s_R)**
- **limit of detection (LOD)**
- **limit of quantification (LOQ)**
- **recovery**
- **trueness (bias)**

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Establishing criteria

- Analyte/ Provision
- Matrix / Commodity
- Well performance around ML

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Applicability

- The method has to be applicable for the analyte, matrix, and specified level(s) (maximum and/or minimum) (ML).
- Nomenclature and CAS No – Specify the matrix, such as raw, cooked tablet, powders

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Limit of Detection (LOD)
Limit of Quantification (LOQ)

Limit of Detection (LOD):

- **For ML ≥ 0.1 mg/kg, LOD ≤ ML · 1/10**
- **For ML < 0.1 mg/kg, LOD ≤ ML · 1/5**

Limit of Quantification (LOQ):

- For ML ≥ 0.1 mg/kg, LOQ ≤ ML · 1/5
- For ML < 0.1 mg/kg, LOQ ≤ ML · 2/5

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Precision

- For ML ≥ 0.1 mg/kg, HorRat value = $RSD_R/RSD_{TR} \leq 2$
- For ML < 0.1 mg/kg, the $RSD_{TR} < 22\%$

RSD_R = relative standard deviation of reproducibility

AOAC SMPR: $RSD_r = 2/3\ RSD_R$

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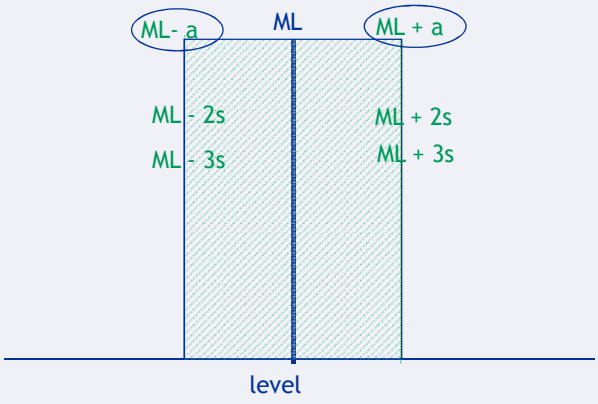
Applicability range



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Minimum applicable range

- For ML ≥ 0.1 mg/kg, [ML - 3 s_R , ML + 3 s_R]
- For ML < 0.1 mg/kg, [ML - 2 s_R , ML + 2 s_R]

s_R = standard deviation of reproducibility

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How to set s in ML ± 2s Λ ML ±3s ?

$$RSD_R(\%) = \frac{s_R \cdot 100\ \%}{c}$$

For C ≥ 10^{-7},
Horwitz eq.
$RSD_{TR} = 2C^{(-0.1505)}(\%)$

For C < 10^{-7},
Thompson
RSD_{TR} = 22%

$s_{TR} = 2C^{(-0.1505)}(\%) \cdot c/100$

$s_{TR} = 22\% \cdot c/100$
$= 0.22 \cdot c$

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Example:

ML = 0.1 mg/kg

C_{ML} = 0.1· 1mg/1000000mg =10^{-7} = 0.0000001

$ML \pm 3s_R = ML \pm 3(2 \cdot C_{ML}^{-0.1505} \cdot ML/100)$

$0.1 \pm 3 \cdot (2 \cdot 0.0000001^{-0.1505} \cdot 0.1/100)$

= 0.1 ± 0.07 i.e. **mg/kg**

[0.03 ; 0.17] mg/kg

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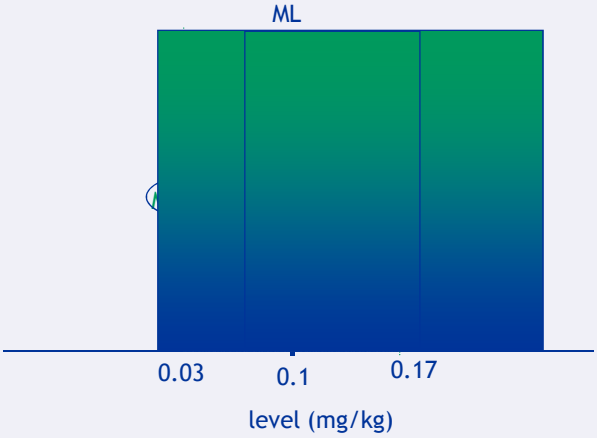
Criteria

Minimum Applicable Range



Recovery

- can be defined as the yield of extraction steps in an analytical process

$$\% \text{ recovery} = 100 \times C_f / (C_u + C_A)$$

Where
C_f = concentration of fortified samples
C_u = concentration of unfortified samples
C_A = concentration of analyte added

Concentr			
100			
≥10			
≥1			
≥0.1			
0.01			
0.001			
0.0001			80 – 110
0.00001			80 – 110
0.000001	10^{-6}	10 µg/kg	60 – 115
0.0000001	10^{-9}	1 µg/kg	40 – 120

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References

Standard Format and Guidance for AOAC Standard Method Performance Requirement (SMPR) Documents (Version 13; October 4, 2011)
www.aoac.org

Method Performance Criteria - Codex Alimentarius Procedural Manual
www. codexalimentarius.org

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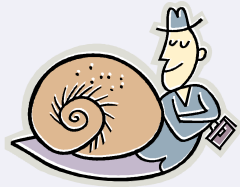
Application

Codex: TTT



AOAC International:
Many SMPRs are published

INFANT FORMULA AND ADULT NUTRITIONALS

AOAC SMPR 2011.003

Standard Method Performance Requirements for Vitamin A in Infant Formula and Adult/Pediatric Nutritional Formula

Intended Use: Global Dispute Resolution Method

1 Applicability

Determination of vitamin A in all forms of infant, adult, and/or pediatric formulas (powders, ready-to-feed liquids, and liquid concentrates). For the purpose of this SMPR, vitamin A is defined as 13-*cis* and all-*trans* retinol (CAS 68-26-8), retinyl esters [retinyl palmitate (CAS 79-81-2) and retinyl acetate (CAS 127-47-9)].

2 Analytical Technique

Any analytical technique that meets the following method performance requirements is acceptable.

3 Definitions

Adult/pediatric formula.—Nutritionally complete, specially formulated food, consumed in liquid form, which may constitute the sole source of nourishment (AOAC SPIFAN, 2010), made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

Infant formula.—Breast-milk substitute specially manufactured to satisfy, by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding (Codex Standard 72-1981), made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

Limit of detection (LOD).—The minimum concentration or

4 Method Performance Requirements[a]

Analytical range	7.0–382.6[b]	
Limit of detection (LOD)	≤2.0[b]	
Limit of quantitation (LOQ)	≤0.01[a]	
Repeatability (RSD_r)	7[b]	≤8%
	10[b]	
	100[b]	
	300[a]	
Recovery	90 to 110% of mean spiked recovery over the range of the assay	
Reproducibility (RSD_R)	10[b]	≤16%
	100[b]	
	200[b]	
	300[b]	
	383[b]	

[a] Concentrations apply to (*1*) "ready-to-feed" liquids "as is"; (*2*) reconstituted powders (25 g into 200 mL water); and (*3*) liquid concentrates diluted 1:1 by weight.

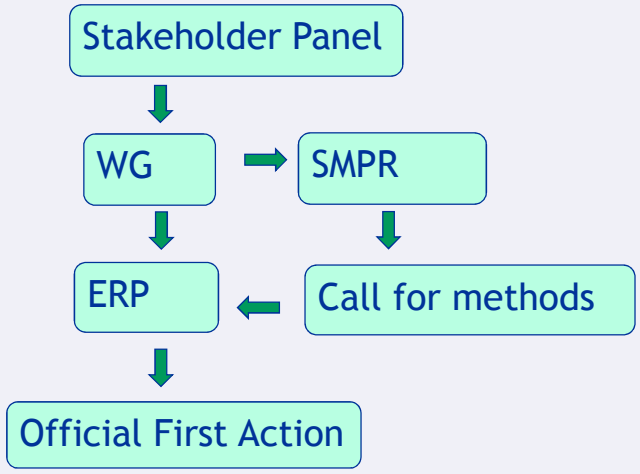
[b] µg/100 g expressed as 13-*cis* retinol and all-*trans* retinol in reconstituted final product.

5 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include blank check samples and check standards at the lowest point and midrange point of the analytical range.

6 Reference Material(s)

The pathway to AOAC official method



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The new pathway to first action

- Single Lab Validated methods can be approved for first action
  - Data demonstrating response linearity, trueness, repeatability, LOD/LOQ, and Matrix scope must be present

→ More methods made available

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The new pathway to first action cont.

- Document method performance versus SMPR
- Note which criteria are met
- For criteria not met, the ERP documents the reasoning why the method is still acceptable
- Data is present to assure the matrix and analyte scopes are covered. This is critical for methods used for dispute resolutions.

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New pathway to final action

- ERP continue to monitor for two years until method is either advanced or removed from the system
  - Using results from PT-Schemes
  - Document positive and negative feedback from users
- ERP recommends for final action to OMB
- OMB grants final action

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Criteria / SMPR

- Cost effective
- Improved quality
- Continue checking the quality

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Thank you

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www.codexalimentarius.org

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