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

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
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)

Establishing criteria

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

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

Limit of Detection (LOD):
- For $ML \geq 0.1 \text{ mg/kg}$, LOD $\leq ML \cdot 1/10$
- For $ML < 0.1 \text{ mg/kg}$, LOD $\leq ML \cdot 1/5$

Limit of Quantification (LOQ):
- For $ML \geq 0.1 \text{ mg/kg}$, LOQ $\leq ML \cdot 1/5$
- For $ML < 0.1 \text{ mg/kg}$, LOQ $\leq ML \cdot 2/5$
Precision

- For $ML \geq 0.1$ mg/kg, HorRat value = $\frac{RSD_R}{RSD_{TR}} \leq 2$

- For $ML < 0.1$ mg/kg, the $RSD_{TR} < 22\%$

$RSD_R =$ relative standard deviation of reproducibility

AOAC SMPR: $RSD_r = \frac{2}{3} RSD_R$

Applicability range

$ML - \alpha \leq ML \leq ML + \alpha$

$ML - 2\sigma \leq ML \leq ML + 2\sigma$

$ML - 3\sigma \leq ML \leq ML + 3\sigma$
Minimum applicable range

- For $ML \geq 0.1 \text{ mg/kg}$, $[ML - 3s_R, ML + 3s_R]$  
- For $ML < 0.1 \text{ mg/kg}$, $[ML - 2s_R, ML + 2s_R]$  

$s_R$ = standard deviation of reproducibility

How to set $s$ in $ML \pm 2s \land ML \pm 3s$ ?

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

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

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

$s_{TR} = 22\% \cdot c / 100 = 0.22 \cdot c$
Example:
\[ ML = 0.1 \text{ mg/kg} \]
\[ C_{ML} = 0.1 \cdot 1 \text{mg}/1000000 \text{mg} = 10^{-7} = 0.0000001 \]

\[ ML \pm 3s_R = ML \pm 3\left(2 \cdot C_{ML}^{0.1505} \cdot ML/100\right) \]
\[ 0.1 \pm 3 \cdot (2 \cdot 0.0000001^{0.1505} \cdot 0.1/100) \]
\[ = 0.1 \pm 0.07 \text{ i.e. mg/kg} \]
\[ [0.03 ; 0.17] \text{ mg/kg} \]
Recovery

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

\[ \text{% recovery} = 100 \times \frac{C_f}{C_u + C_A} \]

Where

- \( C_f \) = concentration of fortified samples
- \( C_u \) = concentration of unfortified samples
- \( C_A \) = concentration of analyte added

**Concentration Ratio Unit Recovery (%)**

- \( \geq 10 \) : \( \geq 10 \)%
- \( \geq 1 \) : \( \geq 1 \% \)
- \( \geq 0.1 \) : \( \geq 0.1 \% \)
- \( 0.01 \) : \( 97 – 103 \)
- \( 0.001 \) : \( 95 – 105 \)
- \( 0.0001 \) : \( 90 – 107 \)
- \( 0.00001 \) : \( 80 – 110 \)
- \( 0.000001 \) : \( 60 – 115 \)
- \( 0.0000001 \) : \( 40 – 120 \)

References


www.aoac.org


www.codexalimentarius.org
Application

Codex: TTT

AOAC International: Many SMPRs are published
The pathway to AOAC official method

1. Stakeholder Panel
2. WG
3. SMPR
4. ERP
5. Call for methods
6. Official First Action

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
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.

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
Criteria / SMPR

- Cost effective
- Improved quality
- Continue checking the quality

Thank you

www.aoac.org
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