Standard Method Performance Requirements (SMPRs) for Furan and Alkyl Furans in Coffee, Baby Foods, Infant Formula, Cereals, and Fruit Juices

Intended Use: Surveillance and Monitoring by Trained Technicians

1. Purpose

AOAC SMPRs describe the minimum recommended performance characteristics to be used during the evaluation of a method. The evaluation may be an on-site verification, a single-laboratory validation, or a multi-site collaborative study. SMPRs are written and adopted by AOAC stakeholder panels composed of representatives from the industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels in their evaluation of validation study data for methods being considered for Performance Tested MethodsSM or AOAC Official Methods of AnalysisSM, and can be used as acceptance criteria for verification at user laboratories.

2. Applicability

Quantitative analysis of furan, 2-methylfuran, 3-methylfuran, 2,5-dimethylfuran, 2-ethylfuran, and 2-pentylfuran in coffee, baby foods (including infant formula), cereals, and fruit juices.

3. Analytical Technique

Chromatographic separation with mass spectrometric detection.

4. Definitions

Limit of quantitation (LOQ).—LOQ is the lowest level of analyte in a test sample that can be quantified at a specified level of precision.

Repeatability.—Variation arising when all efforts are made to keep conditions constant by using the same instrument and operator (in the same laboratory) and repeating during a short time period. Expressed as the repeatability standard deviation (SDr); or % repeatability relative standard deviation (%RSDr).

Reproducibility.—Variation arising when identical test materials are analyzed in different laboratory by different operators on different instruments. The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SDR); or % reproducibility relative standard deviation (% RSDR).
Recovery.—The fraction or percentage of analyte that is measured when the test sample is analyzed using the entire method.

5. **Method Performance Requirements**

Table 1. Limit of quantitation (LOQ)

<table>
<thead>
<tr>
<th>Matrix Type</th>
<th>LOQ (µg/kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coffee (solid material)</td>
<td>≤ 20</td>
</tr>
<tr>
<td>Other matrices</td>
<td>≤ 5</td>
</tr>
</tbody>
</table>

Table 2. Recovery, repeatability and reproducibility parameters

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Requirement</th>
</tr>
</thead>
<tbody>
<tr>
<td>Recovery, %</td>
<td>80-110</td>
</tr>
<tr>
<td>RSDr, %</td>
<td>0.66 times RSDR as derived from (modified) Horwitz equation&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
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<td>As derived from (modified) Horwitz equation&lt;sup&gt;a&lt;/sup&gt;</td>
</tr>
</tbody>
</table>

<sup>a</sup>Horwitz equation for predicted relative standard deviation of reproducibility: PRSDR = 2C<sup>0.15</sup>, where C is analyte concentration expressed as mass fraction.

6. **System Suitability Tests and/or Analytical Quality Control**

Suitable methods will include blanks and appropriate check standards. Method (procedural) and solvent blanks should be below the limit of detection (LOD = 0.3 x LOQ).

7. **Validation Guidance**

Validation should be conducted at the target LOQ and 10xLOQ levels. The LOQ is determined as the lowest spiking level that meets the recovery and repeatability requirements. Suitable matrix blanks should be selected that do not contain more than 30% of the target LOQ level for each analyte.

For matrices that naturally contain higher levels of furan and alkyl furans (e.g. ground roasted coffee) and where suitable matrix blanks are not available (for all or certain analytes), spiking experiments should be conducted for the affected analytes at two concentration levels in the range of 3-10x the analyte level in the evaluated matrix. In this case, the LOQ can be estimated based on extrapolation of signal-to-noise ratio (S/N) obtained for a concentration level naturally present in the evaluated matrix to a concentration level that would correspond to S/N = 10.

Due to the high volatility of the analytes, sample homogenization step should be considered and evaluated in the method validation in addition to all other sample preparation steps.


8. Reference materials


9. Maximum Time-to-Results

None.
<table>
<thead>
<tr>
<th>Common Name</th>
<th>CAS Number</th>
<th>Molecular Structure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Furan</td>
<td>110-00-9</td>
<td><img src="image" alt="Furan Structure" /></td>
</tr>
<tr>
<td>2-Methylfuran</td>
<td>534-22-5</td>
<td><img src="image" alt="2-Methylfuran Structure" /></td>
</tr>
<tr>
<td>3-Methylfuran</td>
<td>930-27-8</td>
<td><img src="image" alt="3-Methylfuran Structure" /></td>
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<tr>
<td>2,5-Dimethylfuran</td>
<td>625-86-5</td>
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</tr>
<tr>
<td>2-Ethylfuran</td>
<td>3208-16-0</td>
<td><img src="image" alt="2-Ethylfuran Structure" /></td>
</tr>
<tr>
<td>2-Pentylfuran</td>
<td>3777-69-3</td>
<td><img src="image" alt="2-Pentylfuran Structure" /></td>
</tr>
</tbody>
</table>
Table 4. Target matrices

- **Coffee***
  - Ground roasted coffee
  - Brewed coffee
  - Ready-to-drink coffee with dairy cream (milk) and sugar

- **Baby food**
  - Fruit-based baby food
  - Vegetable-based baby food with meat
  - High carbohydrate type baby food (e.g. based on custard or yams)
  - Powdered infant formula

- **Cereals**
  - Wheat-based breakfast cereals
  - Oat-based breakfast cereals

- **Fruit juices**
  - Orange juice
  - Apple juice

*Validation data for instant coffee and decaffeinated coffee are also desirable.