AOAC SMPR® 2019.004

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 composed of representatives from 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 method being considered for *Performance Tested Methods*SM or AOAC *Official Methods of Analysis*SM, 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 (*see* Tables 1 and 2).

3 Analytical Technique

Chromatographic separation with mass spectrometric detection.

4 Definitions

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

Recovery.—Fraction or percentage of analyte that is measured when the test sample is analyzed using the entire method.

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 (SD_r); or % repeatability relative standard deviation (%RSD_r).

Reproducibility.—Variation arising when identical test materials are analyzed in different laboratories by different operators on different instruments. The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD_R); or % reproducibility relative standard deviation (%RSD_R).

5 Method Performance Requirements

See Tables 3 and 4.

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 × LOQ).

7 Validation Guidance

Validation should be conducted at the target LOQ and 10x LOQ levels. LOQ is determined as the lowest spiking level that meets 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, 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.

For MS identification criteria, refer to Part D in SANTE/11813/2017 guidelines (https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkdoc_2017-11813.pdf).

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.

Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www. eoma.aoac.org/app_f.pdf).

8 Reference Materials

Refer to Annex F: *Development and Use of In-House Reference Materials* in Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2019) 21st Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www. eoma.aoac.org/app_f.pdf).

9 Maximum Time-to-Results

None

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Posted: November 8, 2019

Table 1. Analytes

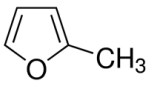
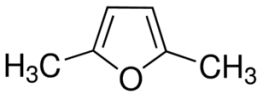
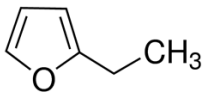
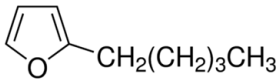
Common name	CAS No.	Molecular structure
Furan	110-00-9	
2-Methylfuran	534-22-5	
3-Methylfuran	930-27-8	
2,5-Dimethylfuran	625-86-5	
2-Ethylfuran	3208-16-0	
2-Pentylfuran	3777-69-3	

Table 2. Target matrices

Coffee[a]	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

[a] Validation data for instant coffee and decaffeinated coffee are also desirable.

Table 3. Limit of quantitation (LOQ)

Coffee (solid material)	≤20 µg/kg
Other matrices	≤5 µg/kg

Table 4. Recovery, repeatability, and reproducibility parameters

Recovery, %	80–110
RSD_r, %	0.66 times RSD_R as derived from (modified) Horwitz equation[a]
RSD_R, %	As derived from (modified) Horwitz equation[a]

[a] Horwitz equation for predicted relative standard deviation of reproducibility: $PRSD_R = 2C^{-0.15}$, where C is analyte concentration expressed as mass fraction.