

1 Version 6, 06/04/2019

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

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6 Intended Use: Surveillance and Monitoring by Trained Technicians

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8 **1. Purpose**

9 AOAC SMPRs describe the minimum recommended performance characteristics to  
10 be used during the evaluation of a method. The evaluation may be an on-site  
11 verification, a single-laboratory validation, or a multi-site collaborative study. SMPRs are  
12 written and adopted by AOAC stakeholder panels composed of representatives from the  
13 industry, regulatory organizations, contract laboratories, test kit manufacturers, and  
14 academic institutions. AOAC SMPRs are used by AOAC expert review panels in their  
15 evaluation of validation study data for method being considered for *Performance Tested*  
16 *Methods<sup>SM</sup>* or *AOAC Official Methods of Analysis<sup>SM</sup>*, and can be used as acceptance  
17 criteria for verification at user laboratories.

18 **2. Applicability**

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

22 **3. Analytical Technique**

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24 Chromatographic separation with mass spectrometric detection.

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26 **4. Definitions**

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

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31 *Repeatability.*—Variation arising when all efforts are made to keep conditions constant  
32 by using the same instrument and operator (in the same laboratory) and repeating  
33 during a short time period. Expressed as the repeatability standard deviation (SDr); or  
34 % repeatability relative standard deviation (%RSDr).

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36 *Reproducibility.*—Variation arising when identical test materials are analyzed in different  
37 laboratory by different operators on different instruments. The standard deviation or  
38 relative standard deviation calculated from among-laboratory data. Expressed as the  
39 reproducibility standard deviation (SDR); or % reproducibility relative standard deviation  
40 (% RSDr).

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*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)**

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

**Table 2. Recovery, repeatability and reproducibility parameters**

Recovery, %	80-110
RSDr, %	0.66 times RSDR as derived from (modified) Horwitz equation <sup>a</sup>
RSDR, %	As derived from (modified) Horwitz equation <sup>a</sup>

<sup>a</sup>Horwitz equation for predicted relative standard deviation of reproducibility:  $PRSD_R = 2C^{-0.15}$ , 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.

78 For MS identification criteria refer to Part D in SANTE/11813/2017 guidelines  
79 ([https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides\\_mrl\\_guidelines\\_wrkd](https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkd)  
80 [oc 2017-11813.pdf](https://ec.europa.eu/food/sites/food/files/plant/docs/pesticides_mrl_guidelines_wrkd)).

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

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86 Appendix F: *Guidelines for Standard Method Performance Requirements, Official*  
87 *Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL,  
88 Rockville, MD, USA ([http://www.eoma.aoac.org/app\\_f.pdf](http://www.eoma.aoac.org/app_f.pdf)).

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90 **8. Reference materials**

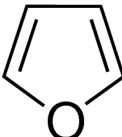
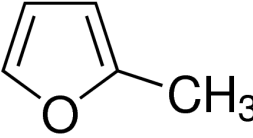
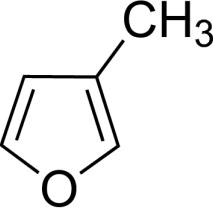
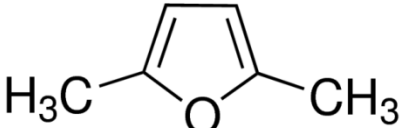
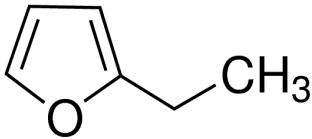
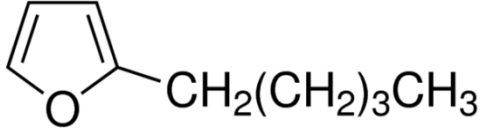
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92 Refer to Annex F: *Development and Use of In-House Reference Materials* in [Appendix F:](#)  
93 *Guidelines for Standard Method Performance Requirements*, 19<sup>th</sup> Edition of the AOAC  
94 INTERNATIONAL Official Methods of Analysis (2012). Available at:  
95 [http://www.eoma.aoac.org/app\\_f.pdf](http://www.eoma.aoac.org/app_f.pdf)

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98 **9. Maximum Time-to-Results**

99 None.  
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**Table 3: Analytes**

Common Name	CAS Number	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	

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**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.