

Standard Method Performance Requirements (SMPRs®) for Determination of Trace Elemental Contaminants in Food and Beverages

Intended Use: Surveillance and Monitoring by Trained Technicians

1 Purpose

What: AOAC Standard Method Performance Requirements (SMPRs®) are voluntary consensus standards developed in accordance with the AOAC policy, “AOAC Due Process for Development of AOAC Non-Method Consensus Standards and Documents.” SMPRs describe a scientific community’s recommended minimum method performance characteristics and analytical requirements for a specific method-related intended use.

Who: Drafted by AOAC working groups, SMPRs are adopted by AOAC by a consensus of stakeholders affiliated with its integrated science programs and projects, which are composed of volunteer subject matter experts representing academia, government, industry, and nonprofit sectors from around the world.

Use: AOAC SMPRs are used in the AOAC core science programs as a resource for AOAC method experts, including expert review panels, in the evaluation of validation study data for methods submitted to the AOAC *Official Methods of Analysis*SM and AOAC *Performance Tested Methods*SM programs. AOAC SMPRs also may be used to provide acceptance criteria for the verification of methods and serve as a resource to guide method development and optimization.

2 Applicability

Determination of total acid extractable cadmium (CAS No. 7440-43-9), total acid extractable arsenic (CAS No. 7440-38-2), total acid extractable lead (CAS No. 7439-92-1), and total acid extractable mercury (CAS No. 7439-97-6) with priority given to a variety of foods, beverages, baby food, infant formula, their

respective ingredients, and natural colorants. Additional elements may be included. For general food application, results must be submitted from all three sides of the AOAC food matrix triangle.

3 Analytical Technique

Inductively coupled plasma-based mass spectrometry (ICP-MS) instrumentation or alternative methodology that meets performance requirements.

4 Definitions

Limit of quantitation (LOQ).—Minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result.

Recovery.—Fraction or percentage of naturally present or spiked analyte that is recovered when 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, and repeating during a short time period. Expressed as the repeatability standard deviation (SD_r); or % repeatability relative standard deviation (%RSD_r).

Reproducibility.—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).

Total acid extractable.—Elemental concentration that can be extracted under an acid digestion environment using a single acid or combination of acids such as HNO₃, HCl plus H₂O₂. For food and beverage samples, the use of HF is not required in this definition of ‘total acid extractable.’

5 Method Performance Requirements

See Table 1.

6 System Suitability Tests and/or Analytical Quality Control

Suitable methods will include processed blank check samples, and second source check standards at the lowest point and/or midrange point of the analytical range.

Table 1. Method performance requirements

Parameter	Arsenic	Cadmium	Lead	Mercury
Limit of quantitation (LOQ): Food, baby food (including their respective ingredients) ^a , and infant formula ^a	5 ppb (µg/kg)	2 ppb (µg/kg)	4 ppb (µg/kg)	1 ppb (µg/kg)
LOQ: Food additives (natural colorants)	100 ppb (µg/kg)	100 ppb (µg/kg)	100 ppb (µg/kg)	100 ppb (µg/kg)
Repeatability (RSD _r)	<10 µg/kg		30%	
	10 to < 100 µg/kg		21%	
	≥100 µg/kg		15%	
Reproducibility (RSD _R)	<10 µg/kg		44%	
	10 to < 100 µg/kg		32%	
	≥100 µg/kg		22%	
Recovery	<10 µg/kg		40–120%	
	10 to < 100 µg/kg		60–115%	
	≥100 µg/kg		80–110%	

^a Concentrations apply to as consumed products. For infant formulas, these include (1) “ready-to-feed” liquids “as is” and (2) reconstituted powders (based on product instructions or using generic reconstitution of 25 g into 200 g water).

7 Reference Material(s)

A certified reference material should be used when available. Alternatively, internally produced reference materials may be used until a reference material for a specific representative commodity is made available by an internationally recognized organization, such as Institute for Reference Materials Measurements (IRMM) or United States National Institute of Standards and Technology (NIST).

8 Validation Guidance and References

Recommended level of validation:

<USP233>

*Official Methods of Analysis*SM.—All submitted methods must be accompanied by validation data upon which the ERP can undertake a comprehensive review. A minimum of a complete single-laboratory validation (SLV) is required for AOAC First Action status. However, method reproducibility will be required for any method to attain AOAC Final Action status. The method performance

parameters may be required or expected depending upon the nature of the analytes, matrices, and techniques pertinent to the method. Use the information in this SMPR to support the minimum necessary types of method validation data collection required. Additional information on other validation parameters may be found in *Official Methods of Analysis*, Appendix F: *Guidelines for Standard Method Performance Requirements*. AOAC guidelines containing information on acceptable experimental designs used to collect this data may be found in the appendices of the *Official Methods of Analysis of AOAC INTERNATIONAL*.

9 Maximum Time-to-Result

No maximum time.

Version 10. Final version: February 29, 2024. Approved by: Stakeholders interested in and affiliated with the AOAC Working Group on Heavy Metals and the affiliated AOAC Metals Community. Effective date: April 5, 2024.