**AOAC SMPR 2015.006**

## *Standard Method Performance Requirements*[SM] (SMPRs) for Quantitation of Arsenic Species in Selected Foods and Beverages

Intended Use: Reference Method for Dispute Resolution

### *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 industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. AOAC SMPRs are used by AOAC expert review panels (ERPs) in their evaluation of validation study data for methods 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. [Refer to Appendix F: *Guidelines for Standard Method Performance Requirements*, *Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed., AOAC INTERNATIONAL, Rockville, MD, USA.]

### *2 Applicability*

Quantitation of arsenic (As) species and/or total inorganic arsenic in selected food and beverage matrixes (*see* Table 1 for a list of selected foods).

### *3 Preferred Analytical Technique*

Separation of species by either ion chromatography (IC) or high-performance liquid chromatography (HPLC) and detection of separated species by inductively coupled plasma-mass spectrometry (ICP-MS).

### *4 Definitions*

*Arsenic species*.—For the purposes of this SMPR, arsenic species includes the following compounds: arsenite (As(III)), arsenate (As(V)), monomethylarsonic acid (MMA), dimethylarsinic acid (DMA), arsenobetaine (AsB), and total inorganic arsenic (I-As).

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

*Quantitative method*.—Method of analysis which response is the amount of the analyte measured either directly (enumeration in a mass or a volume), or indirectly (color, absorbance, impedance, etc.) in a certain amount of sample.

*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*.—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 (%$RSD_R$).

*Recovery*.—The fraction or percentage of spiked analyte that is recovered when the test sample is analyzed using the entire method.

*Total inorganic arsenic*.—The sum of arsenite and arsenate either reported as the sum of the individual species or converted into one form and reported as the total.

**Table 1. Selected foods**

| |
|---|
| Rice |
| Rice-based products |
| Baby food–cereals |
| Infant formula |
| Cereal |
| Fruit juice |
| Apple juice |
| Grape juice |
| Fish oil-based supplements |
| Seafood |
| Finfish |
| Shellfish |
| Seaweed |

**Table 2. Method performance requirements**

| Parameter | Range | Minimum acceptance criteria |
|---|---|---|
| Analytical range | | 10 ppb–1 ppm[a] |
| Limit of quantitation (LOQ) | | <10 ppb[a] |
| Recovery | 10–100 ppb | 60–115% |
| | ≥100 ppb–10 ppm | 80–110% |
| Repeatability ($RSD_r$) | ≤10–50 ppb | ≤20% |
| | ≥50–300 ppb | ≤13% |
| | ≥300 ppb–1 ppm | ≤12% |
| Reproducibility ($RSD_R$) | ≤10–50 ppb | ≤30% |
| | ≥50–300 ppb | ≤20% |
| | ≥300 ppb–1 ppm | ≤18% |

[a] Arsenic (As) species and/or total inorganic arsenic in selected food and beverage matrixes.

### *5 Method Performance Requirements*

*See* Table 2.

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

Suitable methods will include blank check samples, and check standards at the lowest point and midrange point of the analytical range, and a protocol to demonstrate suitability.

### *7 Reference Material(s)*

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* (2012) 19th Ed.]. Available at http://www.eoma.aoac.org/app_f.pdf.

### *8 Validation Guidance*

Validation data must include at a minimum arsenite and arsenate, or total inorganic arsenic, in the food types listed in Table 1 that

apply to the candidate method for First Action *Official Methods of Analysis* approval. It is not required to represent every arsenic species or food type.

Appendix D: Guidelines for Collaborative Study Procedures to Validate Characteristics of a Method of Analysis [*Official Methods of Analysis of AOAC INTERNATIONAL* (2012) 19th Ed.]. Available at http://www.eoma.aoac.org/app_d.pdf.

### *9 Maximum Time-to-Result*

No maximum time to result.

---

*Approved by AOAC Stakeholder Panel on Strategic Foods Analytical Methods (SPSFAM). Final Version Date: February 27, 2015. Effective Date: February 27, 2015.*