**AOAC SMPR® 2021.001**

## *Standard Method Performance Requirements* (SMPRs®) for Determination of Chlorate and Perchlorate in Baby Foods, Infant/Adult Formulas, and Their Ingredients

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 drafted by AOAC working groups composed of representatives from industry, regulatory organizations, contract laboratories, test kit manufacturers, and academic institutions. Approved by AOAC, AOAC SMPRs may be used for method development, are used by AOAC expert review panels in their evaluation of validation study data for methods being considered for AOAC *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*

Determination of chlorate and perchlorate in baby foods, infant/adult formulas. and their ingredients (*see* Table 1).

### *3 Analytical Technique*

Any analytical technique that meets the following method performance requirements is acceptable.

### *4 Definitions*

*Accuracy*.—Closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value (corresponds to the VIM definition for "trueness").

*Adult/pediatric formula*.—Nutritionally complete, specially formulated food, consumed in liquid form, which may constitute the sole source of nourishment, made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

*Baby food*.—Food intended for use by infants when they are weaned and by young children as a supplement to their diet and/or for their progressive adaptation to ordinary food. These include cereal-based products sold as dry, to be reconstituted before consumption or wet meals ready to eat, sweet or savory (*European Commission Food Labeling Guidelines*, https://ec.europa.eu/food/safety/labelling_nutrition/special_groups_food/children_en).

*Chlorate*.—Chlorate anion $ClO_{3-}$ (CAS 14866-68-3).

*Infant formula*.—Breast-milk substitute specially manufactured to satisfy, by itself, the nutritional requirements of infants during the first months of life up to the introduction of appropriate complementary feeding (Codex Standard 72–1981) made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and amino acids, with and without intact protein.

*Ingredients*.—Constituents of products used in their formulations (*see* Table 1).

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

*Perchlorate*.—Perchlorate anion $ClO_{4-}$ (CAS 14797-73-0).

*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 ($\%RSD_R$).

### *5 Method Performance Requirements*

*See* Tables 2 and 3.

*See* also *Validation Guidance.*

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

Suitable methods will include blank check samples and appropriate check standards. Method (procedural) and solvent blanks should be below 0.3 × LOQ.

### *7 Reference Material(s)*

*See* Table 4.

Additionally, remainder materials (infant formulas and infant formula ingredients) tested as part of a NIST interlaboratory comparison may be available for method developers.

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. Available at http://www.eoma.aoac.org/app_f.pdf

### *8 Validation Guidance*

Validation (including criteria from *Method Performance Requirements*) should be conducted at least 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 contain incurred levels of chlorate and where suitable matrix blanks are not available, spiking experiments should be conducted at two concentration levels in the range of 3–10x the analyte level in the evaluated matrix. The LOQ can then be estimated based on extrapolation of signal-to-noise ratio (S/N) obtained for a concentration level present in the evaluated matrix to a concentration level that would correspond to S/N = 10.

Additional guidance can be found in the following resources:

SANTE guidelines on "Analytical quality control and method validation procedures for pesticide residues analysis in food and feed" issued by the European Commission Directorate General for Health and Food Safety (SANTE/12682/2019 or recent version)

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

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

No maximum time.

*Developed by AOAC Stakeholder Program on Infant Formula and Adult Nutritionals (SPIFAN) Working Group on Chlorates and Perchlorates. Approved by SPIFAN on April 30, 2021. Final Version Date: April 30, 2021 (version 7)*

*Posted: June 2021*

**Table 1. Target matrices**[a]

| Matrix category | Matrix sub-category | Minimum representative matrices |
|---|---|---|
| Baby food | | Cereal-based<br>Fruit/vegetable-based<br>Meat-based |
| Baby food ingredients | | Cereal-based<br>Fruit/vegetable-based<br>Meat-based |
| Infant/adult formulas | | Milk-based<br>Plant-based |
| Infant/adult formula ingredients | Animal-based milk powdered protein sources | Whole milk powder<br>Whey protein concentrate |
| | Plant-based protein sources | Soy protein isolate |
| | Liquid milk | Bovine<br>One additional species |
| | Fat-based | Oil/fat |
| | Carbohydrate-based | Lactose<br>Maltodextrin<br>Oligosaccharides (galacto-oligosaccharides or fructo-oligosaccharides) |
| | Mineral- and vitamin-based (premixes) | Any |

[a] In order to include the given matrix category in the method applicability, the validation has to be conducted on all representative matrices listed as the minimum requirement for that matrix category. For example, a method has to be validated on lactose, maltodextrin, and oligosaccharides (galacto-oligosaccharides or fructo-oligosaccharides) to demonstrate applicability for the carbohydrate-based infant/adult formula ingredient category.

**Table 2. Limit of quantitation (LOQ)**

| | Chlorate, mg/kg | Perchlorate, mg/kg |
|---|---|---|
| Baby foods[a] and their major ingredients | ≤0.005 | ≤0.005 |
| Infant/adult formulas[a] | ≤0.005 | ≤0.005 |
| Infant/adult formula ingredients–major (>10% in finished powdered product)[b] | ≤0.01 | ≤0.01 |
| Infant/adult formula ingredients–minor (≤10% in finished powdered product)[b] | ≤0.1 | ≤0.1 |
| Infant/adult formula ingredients–liquid milk | ≤0.005 | ≤0.005 |

[a] Concentrations apply to as consumed products. For infant formulas, these include: (a) “ready-to-feed” liquids “as is”; (b) reconstituted powders (based on the product instructions or using a generic reconstitution of 25 g into 200 g water).

[b] Ingredient content of 10% in a finished powdered infant/adult formula product corresponds to about 1.1% content in a ready-to-feed or reconstituted (as consumed) product, using the generic reconstitution factor of 25 g into 200 g water. Examples of major and minor ingredients are whole milk powder and premixes, respectively.

**Table 3. Recovery, repeatability, and reproducibility parameters**

| | |
|---|---|
| Recovery | 80–120% |
| Repeatability ($RSD_r$) | ≤20% |
| Reproducibility ($RSD_R$) | ≤25% |

**Table 4. Reference materials**

| Source | Product | Chlorate, mg/kg | Perchlorate, mg/kg |
|---|---|---|---|
| NIST (https://www-s.nist.gov/srmors/view_detail.cfm?srm=1869) | SRM 1869 Infant/Adult Nutritional Formula II (milk/whey/soy-based) (*coming soon*) | 0.105 | — |
| NIST | RM 8260 Infant Formula (hydrolyzed protein based) (*coming soon*) | 0.281 | — |
| FAPAS (https://fapas.com/shop/product/pesticide-residues-fat-soluble-inc-eu-red-list-low-level-in-infant-formula-quality-control-material-t05140qc/1192/5511) | T05140QC Infant Formula | — | 0.052 |
| FAPAS (https://fapas.com/shop/product/biocides-quaternary-ammonium-compounds-and-perchlorates-in-salad-leaf-puree-quality-control-material-t19298qc/2059/6544) | T19298QC Salad Leaf | 0.497 | 0.338 |
| FAPAS (https://fapas.com/shop/product/pesticide-residues-fat-soluble-inc-eu-red-list-low-level-in-infant-formula-proficiency-test-05157/567/6037) | Infant Formula (2021 PT Round 05157) | — | ? |