AOAC SMPR® 2017.017

Standard Method Performance Requirements (SMPRs®) for Determination of 2- and 3-MCPD, 2- and 3-MCPD Esters, and Glycidyl Esters in Infant and Adult/Pediatric Nutritional Formula

Intended Use: Surveillance and Monitoring of Infant Formula and Adult/Pediatric Formula 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 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 in their evaluation of validation study data for method being considered for *Performance Tested Methods*℠ or AOAC *Official Methods of Analysis*℠, and can be used as acceptance criteria for verification at user laboratories.

2 Applicability

Quantitative analysis of 2-MCPD, 3-MCPD, and glycidol resulting from the separate or combined determination of free 2-MCPD and total 2-MCPD esters, free 3-MCPD and total 3-MCPD esters, and total glycidyl esters in all forms of infant, adult nutritionals, and/or pediatric formula (powders, ready-to-feed liquids, and liquid concentrates).

3 Analytical Technique

Any analytical technique (more than one technique may be required) that meets the following method performance requirements is acceptable.

4 Definitions

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

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, or amino acids, with and without intact protein.

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.

Limit of quantitation (LOQ).—The minimum concentration or mass of analyte in a given matrix that can be reported as a quantitative result. *See* reference to Appendix L in section 8 Validation Guidance.

2-MCPD.—2-Monochloropropane-1,3-diol or 2-chloropropane-1,3-diol. CAS No. 497-04-1. *See* Figure 1 for molecular structure.

2-MCPD esters.—2-MCPD esters (bound 2-MCPD) = All 2-MCPD derivatives being present as fatty acid esters (mono- and diesters).

3-MCPD.—3-Monochloropropane-1,2-diol or 3-chloropropane-1,2-diol. CAS No. 96-24-2. *See* Figure 2 for molecular structure.

3-MCPD esters.—3-MCPD esters (bound 3-MCPD) = All 3-MCPD derivatives being present as fatty acid esters (mono- and diesters).

Glycidol.—2,3-Epoxy-1-propanol. CAS No. 556-52-5. *See* Figure 3 for molecular structure.

Glycidyl esters.—Glycidyl esters (bound glycidol) = All glycidyl derivatives being present as fatty acid esters (monoesters).

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 ($\%RSD_R$).

5 Method Performance Requirements

See Tables 1–3.

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.

7 Reference Material(s)

Follow 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* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_f.pdf)

8 Validation Guidance

Appendix L: *AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation, Official Methods of Analysis of AOAC INTERNATIONAL* (2016) 20th Ed., AOAC INTERNATIONAL, Rockville, MD, USA (http://www.eoma.aoac.org/app_l.pdf)

Method developer must be able to determine recovery of free and bound forms.

Method applicability should be verified by matching assigned values of suitable reference material(s).

- Suitable reference materials should consist of infant formula that contains 2-MCPD esters, 3-MCPD esters, and glycidyl esters that originate from the commonly applied processing techniques and are not being spiked.
- Free 2-MCPD and 3-MCPD, which typically are not present in significant amounts in infant formulae, should also be present in reference materials but might be introduced by spiking.
- Analyte contents should represent ranges that are commonly present in commercially available products, and must include data for concentrations at or near the LOQ specified in this SMPR.

- As different types of infant formulae might show also very different accessibility to extraction techniques, reference materials should represent commercially available products with high demands to analyte extraction.

9 Maximum Time-to-Result

No maximum time.

Approved by the AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) on September 23, 2017. Final Version Date: October 25, 2017.

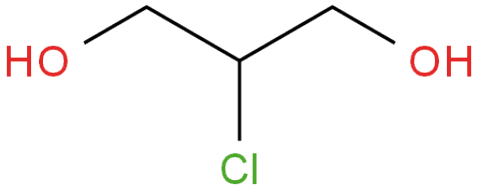

Figure 1. Molecular structure for 2-MCPD.

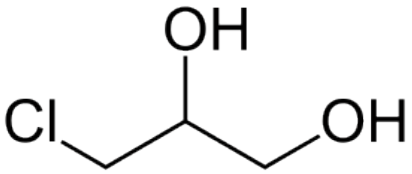

Figure 2. Molecular structure for 3-MCPD.

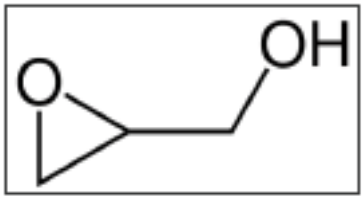

Figure 3. Molecular structure for glycidol.

Table 1. Method performance requirements: 3-MCPD

Parameter		3-MCPD[a] in dry infant formula samples (powder)	3-MCPD[a] in liquid infant formula samples
Analytical range, µg/kg		25–≥1000	3–≥120
LOQ, µg/kg		≤25	≤3
Accuracy, %		70–125	70–125
RSD_r, %		≤22	≤22
RSD_R, %	25–100 µg/kg	≤44	≤44
	>100 µg/kg	≤25	

[a] Total analyte content expressed as free 3-MCPD resulting either from separate or combined determination of free 3-MCPD and bound 3-MCPD.

Table 2. Method performance requirements: 2-MCPD

Parameter		2-MCPD[a] in dry infant formula samples (powder)	2-MCPD[a] in liquid infant formula samples
Analytical range, µg/kg		25–≥500	3–≥60
LOQ, µg/kg		≤25	≤3
Accuracy, %		70–125	70–125
RSD_r, %		≤22	≤22
RSD_R, %	25–100 µg/kg	≤44	≤44
	>100 µg/kg	≤25	

[a] Total analyte content expressed as free 2-MCPD content resulting either from separate or combined determination of free 2-MCPD and bound 2-MCPD.

Table 3. Method performance requirements: Glycidol

Parameter		Glycidol[a] in dry infant formula samples (powder)	Glycidol[a] in liquid infant formula samples
Analytical range, µg/kg		15–≥400	2–≥48
LOQ, µg/kg		≤15	≤2
Accuracy, %		70–125	70–125
RSD_r, %		≤22	≤22
RSD_R, %	15–48 µg/kg	≤25	≤44
	>48 µg/kg	≤44	

[a] Analyte content resulting from determination of bound glycidol.