STANDARD METHOD PERFORMANCE REQUIREMENTS

AOAC SMPR 2015.001

Determination of Sodium Fluoroacetate ("Compound 1080") in Infant Formula

Stakeholder Panel on Infant Formula and Adult Nutritionals

Working Group for Sodium Fluoroacetate ("Compound 1080")

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The following *Standard Method Performance Requirement*SM was championed by key subject matter experts and leading global authorities in pesticide contaminants and infant formula, including scientists from the U.S. Food and Drug Administration, Oregon State University, Florida and Maryland State Departments of Agriculture, Canadian Food Inspection Agency, NEN, contract research organizations, and the infant formula industry.

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Developed by the Working Group for Sodium Fluoroacetate ("Compound 1080") and approved by the Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN).

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Intended Use: Surveillance and monitoring of infant formula (and possibly adult/pediatric) formula by trained technicians.

1 Purpose

AOAC SMPR's 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 the 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

Determination of total fluoroacetic acid and its salts in all forms of infant formula (powders, ready-to-feed liquids, and liquid concentrates).

3 Analytical Technique

Any analytical technique that meets the following method performance requirements is acceptable. High through-put methods that can also determine total fluoroacetic acid and its salts in adult / pediatric formulas are preferable.

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, and amino acids, with and without intact protein.

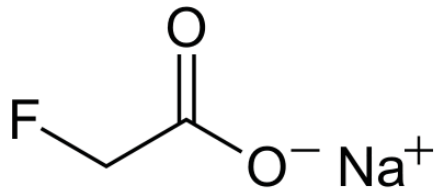

Figure 1. Chemical structure of sodium fluoroacetate (Compound 1080) molecular weight 100.24; molecular formula $C_2H_2FNaO_2$.

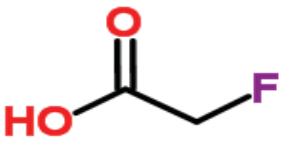

Figure 2. Chemical structure of fluoroacetic acid (cymonic acid) molecular weight 78.04; $C_2H_3FO_2$.

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 detection (LOD).—The minimum concentration or mass of analyte that can be detected in a given matrix with no greater than 5% false positive risk and 5% false negative risk.

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

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 relative standard deviation (SD_R); or % reproducibility relative standard deviation (% RSD_R).

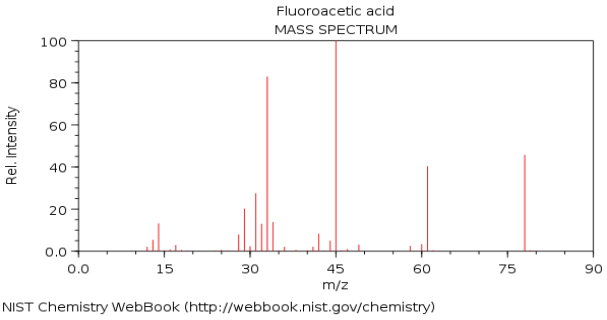

Figure 3. Mass Spectrum of fluoroacetic acid.

Table 1. Method performance requirements

Analytical range	4–100 ppm[a]
Limit of Detection (LOD)	1 ppb[a]
Limit of Quantitation (LOQ)	≤4 ppb[a]
Recovery	±20%
Repeatability (RSD_r)	≤14%
Reproducibility (RSD_R)	≤20%

[a] Fluoroacetate expressed as µg of fluoroacetic acid /1000 g of solids.

Sodium fluoroacetate.—The active ingredient in "Compound 1080", a rodenticide. IUPAC name: Sodium 2-fluoroacetate. CAS number: 62-74-8. See Figure 1.

5 Method Performance Requirements

See Table 1.

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 in the *Official Method of Analysis of the AOAC INTERNATIONAL* compendium.

8 Validation Guidance

Appendix L: AOAC Recommended Guidelines for Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN) Single-Laboratory Validation, 19th Edition of the AOAC INTERNATIONAL *Official Methods of Analysis* (2012). Available at: http://www.eoma.aoac.org/app_l.pdf

9 Maximum Time-To-Result

No maximum time, but method should be suitable for high through-put.