AOAC SMPR® 2014.004

Standard Method Performance Requirements for Minerals and Trace Elements in Infant Formula and Adult/Pediatric Nutritional Formula

Intended Use: Reference Method for Dispute Resolution

1 Applicability

Determination of minerals [total calcium (Ca), magnesium (Mg), phosphorus (P), potassium (K), and/or sodium (Na)] and trace elements [copper (Cu), iron (Fe), manganese (Mn), and/or zinc (Zn)] in all forms of infant, adult, and/or pediatric formula (powders, ready-to-feed liquids, and liquid concentrates).

2 Analytical Technique

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

3 Definitions

Adult/pediatric formula.—Nutritionally complete, specially formulated food, consumed in liquid form, which may constitute the sole source of nourishment [AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN); 2010], made from any combination of milk, soy, rice, whey, hydrolyzed protein, starch, and 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.

Milk and milk products.—Milk and milk products as defined in the General Standard for the Use of Dairy Terms (Codex Standard 206-1999).

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.

List of minerals and trace elements.—

Calcium (Ca)	CAS 7440-70-2
Copper (Cu)	CAS 7440-50-8
Iron (Fe)	CAS 7439-89-6
Magnesium (Mg)	CAS 7439-95-4
Manganese (Mn)	CAS 7439-96-5
Phosphorus (P)	CAS 7723-14-0
Potassium (K)	CAS 7440-09-7
Sodium (Na)	CAS 7440-23-5
Zinc (Zn)	CAS 7440-66-6

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 ($\%RSD_R$).

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

4 Method Performance Requirements

See Table 1.

5 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.

6 Reference Material(s)

National Institute of Standards and Technology (NIST) Standard Reference Material® (SRM) 1849a Infant/Adult Nutritional Formula or equivalent. The SRM is a milk-based, hybrid infant/adult nutritional powder prepared by a manufacturer of infant formula and adult nutritional products. A unit of SRM 1849a consists of 10 packets, each containing approximately 10 g of material. Certified values for minerals in NIST 1849a are provided in Table 2.

7 Validation Guidance

Recommended level of validation: *Official Methods of Analysis*℠.

Table 1. Method performance requirements[a]

		Mn	Cu	Fe	Zn	Mg	P	Na	Ca	K
Lower analytical range		0.001[b]	0.001[b]	0.01[b]	0.1[b]	3[b]	15[b]	10[b]	20[b]	10[b]
Upper analytical range		1.0[b]	1.2[b]	20[b]	18[b]	110[b]	800[b]	850[b]	1280[b]	2000[b]
Limit of quantitation (LOQ)		0.001[b]	0.001[b]	0.01[b]	0.1[b]	3[b]	15[b]	10[b]	20[b]	10[b]
$\%RSD_r$		5%								
Recovery		90–110% of mean spiked recovery over the range of the assay								
$\%RSD_R$	≤0.005[b]	≤16%								
	>0.005[b]	≤10%								

[a] Concentrations apply to: (a) "ready-to-feed" liquids "as is"; (b) reconstituted powders (25 g into 200 g of water); and (c) liquid concentrates diluted 1:1 by weight.

[b] mcg/100 g reconstituted final product.

8 Maximum Time-to-Result

No maximum time.

Approved by AOAC Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN). Final Version Date: March 18, 2014.

Revised: May 26, 2016 to correct unit in Table 1 footnote b.

Table 2. Certified mass fraction values for selected elements in SRM 1849a[a]

Element	Mass fraction, mg/kg	Coverage factor (*k*)
Calcium[b–d]	5253 ± 51	2.00
Copper[b–d]	19.78 ± 0.26	2.00
Chromium[b–e]	1.072 ± 0.032	2.00
Iodine[c,e,f]	1.29 ± 0.11	2.00
Iron[b–d]	175.6 ± 2.9	2.00
Magnesium[b–d]	1648 ± 36	2.00
Manganese[b–d]	49.59 ± 0.97	2.00
Molybdenum[b–e]	1.707 ± 0.040	2.00
Phosphorus[b–d]	3990 ± 140	2.00
Potassium[b–d]	9220 ± 110	2.00
Selenium[c–e]	0.812 ± 0.029	2.00
Sodium[b–d]	4265 ± 83	2.00
Zinc[b–d]	151.0 ± 5.6	2.00

[a] Each certified mass fraction value is the mean from the combination of the mean of results from analyses by NIST, the median of the results provided by collaborating laboratories, and the mean result provided by the material manufacturer, where available. The uncertainty provided with each value is an expanded uncertainty about the mean to cover the measurand with approximately 95% confidence. The expanded uncertainty is calculated as $U = ku_c$, where u_c incorporates the observed difference between the results from the methods and their respective uncertainties, consistent with the ISO Guide and with its Supplement 1, and k is a coverage factor corresponding to approximately 95% confidence.

[b] NIST ICP-OES.

[c] Collaborating laboratories.

[d] Manufacturer.

[e] NIST ICP-MS.

[f] NIST INAA.