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Standard Method Performance Requirements for Cr, Mo, and Se in Infant Formula and Adult/Pediatric Nutritional Formula

Intended Use: Global Dispute Resolution Method

1 Applicability

Determination of total chromium (Cr), molybdenum (Mo), and selenium (Se) 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 measures all three analytes simultaneously and 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, 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, 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 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.

Table 1. Method performance requirements[a,b]

	Cr	Mo	Se
Analytical range	≤20–1600	≤20–1000	≤10–500
Limit of detection (LOD)	≤7	≤7	≤4
Limit of quantitation (LOQ)	≤20	≤20	≤10
Repeatability (RSD_r)	≤5% over the analytical range		
Recovery factor	90 to 110% of mean spiked recovery over the range of the assay		
Reproducibility (RSD_R)	≤15% over the analytical range		

[a] Concentrations apply to (*1*) "ready-to-feed" liquids "as is"; (*2*) reconstituted powders (25 g into 200 mL water); and (*3*) liquid concentrates diluted 1:1 by weight.

[b] µg/kg reconstituted final product.

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.

Example protocol:

- Blank check samples (reagent blank levels <0.4 µg/L Cr, Mo; <0.2 µg/L Se).
- Calibration verification (CV) standards at the midrange point of the calibration range (valid samples must be bracketed by CVs that agree within 5% of nominal).
- Calibration error must be no more than 5% at the blank check concentration limits listed above (checked once), and all samples must have analytical solution concentrations above this lower linearity limit.
- The %RSD of duplicate results for Cr, Mo, and Se concentrations in each sample must be 10% or better (6% for the control sample). A control sample (NIST 1849 or equivalent) must be run with every set of samples. The mean of duplicate control results must be within the certified limit and within local control limits, if a control chart is in place. The relative standard deviation (%RSD) of the mean for Cr, Mo, and Se as calculated from such control chart must be <5%.

6 Reference Material(s)

National Institute of Standards and Technology (NIST) Standard Reference Material® 1849 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 1849 consists of 10 packets, each containing approximately 10 g of material.

7 Validation Guidance

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

8 Maximum Time-to-Signal

Eight hours for all three nutrients.

Approved by Stakeholder Panel on Infant Formula and Adult Nutritionals (SPIFAN). Final Version Date: September 17, 2011. Effective Date: September 20, 2011.