

## Standard Method Performance Requirements for Vitamin D in Pre-Blends, Pre-Mixes, and Pure Materials

### 1 Applicability

Determination of vitamin D, defined as vitamin D<sub>2</sub> (ergocalciferol and its previtamin isomer); and vitamin D<sub>3</sub> (cholecalciferol and its previtamin isomer), in food ingredients such as pre-blends, pre-mixes, and pure materials, including encapsulated and oil forms.

### 2 Analytical Technique

Chromatographic methods that utilize common instrumentation that are readily available worldwide.

### 3 Definitions

*Pre-blends and pre-mixes.*—Mixtures of one or more food additives, with food materials or water used as a carrier, and not intended for direct consumption by humans.

*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<sub>r</sub>); or % repeatability relative standard deviation (%RSD<sub>r</sub>).

*Reproducibility.*—The standard deviation or relative standard deviation calculated from among-laboratory data. Expressed as the reproducibility standard deviation (SD<sub>R</sub>); or % reproducibility relative standard deviation (%RSD<sub>R</sub>).

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

Analytical range	100 ppm–100%	
Limit of quantitation (LOQ)	≤100 ppm	
Repeatability (RSD <sub>r</sub> )	0.01%	≤4%
	1%	≤2%
	100%	≤1%
Recovery	90 to 110% of mean spiked recovery over the range of the assay	
Reproducibility (RSD <sub>R</sub> )	0.01%	≤8%
	1%	≤4%
	100%	≤2%

<sup>a</sup> Acceptance criteria are on a total analyte basis.

### 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)

Use suitable materials.

### 7 Validation Guidance

Recommended level of validation: *Official Methods of Analysis*<sup>SM</sup>.

### 8 Maximum Time-to-Results

No maximum time.

*Approved by the AOAC Stakeholder Panel on Strategic Food Analytical Methods (SPSFAM) on September 29, 2012. Final Version Date: September 28, 2012.*