## 1. SCOPE AND PURPOSE:

This method is for the determination of total iodine by ICP-MS in feeds, flours, mineral premixes, and pet food samples. There is no assurance that matrices other than those listed can be assayed using this method.

## 2. PRINCIPLE:

2.1. Iodine is extracted from the test portion of a sample with nitric acid using microwave digestion. The resulting solution is then neutralized using ammonium hydroxide and stabilized using a strong alkaline reagent (TMAH) and EDTA. After removing undissolved components, the solution is atomized and ionized in an ICP-MS instrument. ICP-MS analysis of iodine at mass 127 ($^{127}I$) is performed using potassium iodide as calibration standard and Germanium ($^{74}Ge$) as an internal standard.

2.2. Specific LabVantage test codes are used to indicate conditions for determining Iodine according to the following table:

| LabVantage Test Code | Used for determining |
|---|---|
| IFMS | Iodine |
| IFMSPMX | Iodine (in mineral premixes) |
| IFMS DM | Iodine Dry Matter |
| IFMSPMX DM | Iodine (in mineral premixes) Dry Matter |

2.3. The limit of quantitation for IFMS is 0.5 ppm using a 0.5 g sample with a 50 mL dilution factor.The limit of quantitation for IFMSPMX is 25 ppm using a 2 g sample with a 10,000 mL dilution factor.

2.4. Known Interferences

2.4.1. The determination of iodine via ICP-MS cannot typically be performed with a simple nitric acid digestion. Nitric acid in the presence of iodine can cause $I_2$ to be selectively evaporated in the spray chamber. The result is severe memory effects during the analytical run. Pre-treatment of the sample digest with an ammonium hydroxide solution resolves this issue. In addition to ammonium hydroxide, a matrix of 1% TMAH, 0.1% EDTA and 0.1 % Triton X is also used to keep instrument sample introduction system free of complications from precipitation of metals when switching between analysis with acidic and alkaline digestions.

2.4.2. A preferred separate and dedicated sample introduction system is recommended for Iodine ICP-MS determinations.

2.4.3. Red dye #3 will increase the total iodine content of the sample, although the iodine in the dye is not bioavailable to dogs and cats.

## 3. PRECISION:

Records of method precision based on Method Validation and/or known control summaries are located in the QA Master file for this test method. Assay precision may vary with test matrix and analyte level. Terms used to describe method precision are defined in NPSOP3000, *Validation of Quantitative Chemical Tests*.

## 4. REFERENCES:


4.1. AOAC 2012.14 Total Iodine in Infant Formula and Nutritional Products

4.2. Nestle' Method LI-00.849-2 "Total Iodine and Bromine by ICP-MS

4.3. NPAL Method "Metals and Mineral Determination using Inductively Coupled Plasma Mass Spectrometry"