

EFFECTIVE DATE	N P Analytical Laboratories	METHOD CODE
REVISED: 02/12/25	LABORATORY TEST METHOD SUMMARY	ICP, ICPPMX
REPLACES: 10/18/23	Minerals by Inductively Coupled Argon Plasma Emission Spectroscopy	PAGE 1 OF 3
KEY WORDS: ICAP, ICP, Minerals, Metals		

1. SCOPE AND PURPOSE:

This method measures minerals in foods, animal feeds, food and feed ingredients, and physiological samples. The routine minerals include calcium, copper, iron, potassium, magnesium, manganese, sodium, phosphorus, and zinc. This procedure can be expanded to include the determination of additional non-routine metals via ICP when requested.

2. PRINCIPLE:

2.1. The sample is prepared via a dry or wet ash procedure to remove organic material. The ash residue is dissolved in dilute acid. The concentrations of the various metals and minerals are determined by comparing the characteristic emission intensities of each sample to the emission intensities of a series of standard solutions, as measured by ICP spectroscopy. The characteristic wavelengths used are indicated in Table 1.

Emission Wavelengths

Table 1

Element	Wavelength, nm	Comments
Calcium	315.887, 317.933, 211.276	
Copper	324.754, 327.395	
Iron	259.940, 238.204	
Magnesium	383.829, 280.270, 279.078, 285.213	
Manganese	257.610	
Phosphorus	213.618, 178.222	
Potassium	766.491, 769.897	
Sodium	589.592, 588.995	
Zinc	213.857, 202.548	
Indium	303.936, 325.856	Used as an internal standard
Yttrium	360.074, 242.29	Used as an internal standard

- 2.2. **Note:** Alternative wavelengths may be used to adjust for sample matrix interferences and sensitivity needs.
- 2.3. Cesium Chloride is used as an ionization suppressant in combination with Indium and Yttrium (or Strontium) which are used as internal standards. The internal standard is used to reduce ionization effects, plasma loading effects and sample viscosity differences between samples. The ICP also allows for the acquisition of multiple analytical wavelengths for each element. Multiple wavelengths can be used to insure inter-element interferences are minimized in unknown or new matrices.
- 2.4. The limit of detection is calculated for each mineral based on the sample weight and lowest standard for that mineral.

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2.5. Known Interferences: The atomic lines of Na, K, and to a lesser extent Ca (422.673 nm) exhibit signal enhancement with increasing concentrations of easily ionizable elements (EIE). The effect can be minimized for the axially-viewed ICP-AES by saturating the plasma with a high concentration of another EIE such as cesium. Cesium is added as an ionization buffer to the standards and samples as a component of the internal standard (IS). Cesium was chosen as an ionization buffer as it has a low energy of ionization, is not very sensitive by ICP-AES and, therefore, spectral interference is generally not a problem. Cesium chloride is available in a very pure form and does not build up in the torch injector tube as readily as other alkali salts. Premix sample standard yields low Fe recovery when prepped using the microwave wet ash method.

3. PRECISION:

Records of method precision based on Method Validation and/or laboratory 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. Official Methods of Analysis of the AOAC International, Method 968.08, Method 984.27, Method 935.13, Method 985.35, Methods 975.03, Method 990.08, and Method 993.14
- 4.2. AOAC Official Method 2011.14: Calcium, Copper, Iron, Magnesium, Manganese, Potassium, Phosphorus, Sodium, and Zinc in Fortified Food Products. Microwave Digestion And Inductively Coupled Plasma-Optical Emission Spectrometry.
- 4.3. Agilent 5100 and 5110 ICP-OES User's Guide, G8010-90002 Issue 4, 10/16
- 4.4. Agilent 5800 and 5900 ICP-OES User's Guide, B8020-90002 Edition 1, 10/2019
- 4.5. Dolan, S. P., & Capar, S. G. (2002). Multi-element analysis of food by microwave digestion and inductively coupled plasma-atomic emission spectrometry. *Journal of Food Composition and Analysis*, 15(5), 593-615.
- 4.6. Kane, P. F., Hall Jr, W. L. (2006). Determination of arsenic, cadmium, cobalt, chromium, lead, molybdenum, nickel, and selenium in fertilizers by microwave digestion and inductively coupled plasma-optical emission spectrometry detection: Collaborative study. *Journal of AOAC International*, 89(6), 1447-1466.
- 4.7. Mars CEM Method Note Mars CEM Method Microwave digestion of fertilizer – AOAC Method 2006.03
- 4.8. Poitevin, E. (2012). Determination of calcium, copper, iron, magnesium, manganese, potassium, phosphorus, sodium, and zinc in fortified food products by microwave digestion and inductively coupled plasma-optical emission spectrometry: single-laboratory validation and ring trial. *Journal of AOAC International*, 95(1), 177-185.
- 4.9. Poitevin, E., Nicolas, M., Graveleau, L., Richoz, J., Andrey, D., Monard, F. (2009). Improvement of AOAC Official Method 984.27 for the determination of nine nutritional elements in food products by Inductively coupled plasma-atomic emission spectroscopy after microwave digestion: single-laboratory validation and ring trial. *Journal of AOAC international*, 92(5), 1484-1518.

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4.10. Webb, S., Bartos, J., Boles, R., Hasty, E., Thuotte, E., & Thiex, N. J. (2014). Simultaneous determination of arsenic, cadmium, calcium, chromium, cobalt, copper, iron, lead, magnesium, manganese, molybdenum, nickel, selenium, and zinc in fertilizers by microwave acid digestion and inductively coupled plasma-optical emission spectrometry detection: single-laboratory validation of a modification and extension of AOAC 2006.03. *Journal of AOAC International*, 97(3), 700-711.