

TRACE METALS (Flame Atomic Absorption Spectroscopy)

PRINCIPLE

A sample is dissolved or diluted in purified water and the resulting solution is atomized in the gas flame of an atomic absorption spectrophotometer equipped with a light source emitting radiation specific of the element to be quantified. The amount of radiation absorbed by the ground state atoms in the flame is proportioned to the concentration of the element in solution and is measured against a calibration curve of known reference standards.

SCOPE

This method is applicable to the determination of the following metals in water soluble carbohydrates: aluminum, antimony, arsenic, barium, beryllium, boron, cadmium, calcium, chromium, cobalt, copper, iron, lead, manganese, magnesium, molybdenum, nickel, potassium, selenium, silicon, sodium, strontium, tin, vanadium and zinc.

SPECIAL APPARATUS

1. Atomic Absorption Spectrometer (AAS): Equipped with a flame atomizer, a background corrector and an option autosampler.
2. Hollow Cathode or Electrodeless Discharge Lamps: Available from AAS manufacturers.

REAGENTS

All reagents and apparatus must be clean of the elements being tested (Note 1). In this sense, plastic ware is preferred over glassware. Excessive use of concentrated acids should be avoided.

1. Water-Highly Purified (18 Ω M): Milli-Q Purification System or equivalent.
2. Nitric Acid: AA Grade.

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3. Hydrochloric Acid: AA Grade.
4. Cesium Chloride Modifier, 2% (V/V) Solution: Weigh 10 g AA grade cesium chloride in a 500 mL flask and dilute with purified water.
5. Lanthanum Chloride Modifier, 11.3% (W/V) Lanthanum Oxide in 11% Hydrochloric Acid: Fisher reagent grade lanthanum oxide, Baker Chemical Co., Ultrex hydrochloric acid. Weigh 113 g of AA grade lanthanum oxide, add to 500 mL of purified water in a 1 L volumetric flask. Carefully add 250 mL of hydrochloric acid. Ensure lanthanum oxide is completely dissolved before diluting. Dilute to volume with water.
6. Elemental Standard Solutions, 1000 ppm: Fisher Scientific. Standards should contain the same matrix composition (dry substance, acid concentration, etc.) as the samples. Prepare a 1 ppm standard by pipetting 0.25 mL of 1000 ppm Standard Solution into a 250 mL volumetric flask and diluting with purified water. Standards should be prepared fresh daily.
7. Dextrose: ACS Grade.

PROCEDURE

Sample Preparation: Samples are diluted to 10% dry substance. For samples being analyzed for calcium and magnesium, add 2.5 mL of lanthanum chloride solution to each sample. For samples being analyzed for aluminum, potassium, sodium and strontium, add 1.25 mL of cesium chloride solution. For an alternative sample preparation see Note 2.

Standard Preparation: Standards should match the matrix of the samples in both dry substance and modifier concentration. Pipette 1.25, 2.50, 5.00 and 7.50 mL of the 1 ppm standard solution into 25 mL volumetric flasks. Include a blank as well. Add 2.5 g of dextrose to each flask. Add 2.5 mL of lanthanum chloride solution to each flask to be analyzed for calcium and magnesium. Add 1.25 mL of cesium chloride solution to each flask to be analyzed for aluminum, potassium, sodium and strontium (Do not add more than one modifier per flask).

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Analysis: Install the appropriate hollow cathode lamp (HCL) for the element of choice. The energy from the HCL should be optimized for maximum output. Elements absorb energy at several wavelengths, and although most have one wavelength that provides the best sensitivity, alternate wavelengths may not suffer from the same interferences. Choose the wavelength that is appropriate for the samples to be analyzed. Adjust the height of the flame burner to maximize the degree of absorption (which occurs in the center of the flame). The normal height of the flame from base to tip is about 3 inches. Many factors can affect the population of atoms that reach the flame, among these are droplet size, sampling rate, aspiration time, the ratio of fuel to oxygen and the type of fuel used. All of these factors must be considered during the preparation for analysis. Manufacturers can provide suggestions for optimizations of their spectrometers (Notes 3 and 4).

After the optimal parameters have been set, load the autosampler with the standards and samples. The autosampler will aspirate the samples into the flame of the AAS, and the difference between the intensity of the source (HCL) through the flame with the sample/standard present is compared to the intensity of the source through the flame without the sample present. The difference is proportional to the concentration of the chosen element.

CALCULATION

$$\text{Analyte, ppm as is} = \frac{(\text{Concentration})(\text{Volume, mL})(\text{Dilution Factor})}{\text{Sample Wt., g}}$$

NOTES AND SAFETY PRECAUTIONS

1. All glassware, plastic ware and autosampler cups should be soaked for several hrs. in dilute (5%) nitric acid solution and then rinsed three times in purified water.
2. Another method of sample preparation involves removal of all organic matter by exposing the sample to high temperatures. Weigh 10 g of sample and place in a quartz or platinum dish. Highly refined syrups give very little ash, and the initial sample can be increased to 20 g. Place the sample under a heat lamp or on a hot plate until the water has been evaporated, and the

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sample is charred. Place the sample in a 550 °C muffle furnace and hold at 550 °C until a white ash is formed. This may take up to six hrs. To speed up the process, the sample can be removed from the furnace, cooled and moistened with a little water or nitric or hydrochloric acid. Return the sample to the furnace. Once a white ash is formed, allow the sample to cool, add a few drops of concentrated nitric acid and transfer to a 25 mL volumetric flask. Dilute to volume with purified water. Ash residues needing iron analysis should be dissolved in concentrated HCl. Modifiers should not be added prior to ashing.

3. Other parameters to be considered when designing an experiment using AAS: type of background correction, type of nebulizer, type of burner/pre-mix burner system, impact devices, matrix interferences, ionization interferences and flame temperature.
4. Incorrect installation and/or operation of the spectrometer can create an explosion hazard.

REFERENCES

1. *Standard Methods for the Examination of Water and Wastewater*, 19th Edition, 3111 Metals by Flame Atomic Absorption Spectrometry: American Public Health Administration, Washington, D. C., 1993.
2. Robinson, J.W. *Atomic Spectroscopy*; Marcel Dekker, New York, 1990.
3. Beaty, Richard. *Concepts, Instrumentation and Techniques in Atomic Absorption Spectrophotometry*; Perkin Elmer, 1988.

METHOD HISTORY

Corn Syrup, Trace Metals (Flame Atomic Absorption Spectroscopy) (E-70), Date of Acceptance 2-27-1996, Revised 12-22-2006.