

3.2.05

AOAC Official Method 975.03
Metals in Plants and Pet Foods
Atomic Absorption Spectrophotometric Method
First Action 1975
Final Action 1988

(Applicable to calcium, copper, iron, magnesium, manganese, potassium, and zinc.)

A. Apparatus and Reagents

Deionized H₂O may be used. See **965.09A** and **B** (see 2.6.01), and following:

(a) *Potassium stock solution*.—1000 g K/mL. Dissolve 1.9066 g dried (2 h at 105°C) KCl in H₂O and dilute to 1 L. Use following parameters for Table **965.09** (see 2.6.01): 7665 Å (766.5 nm), air-C₂H₂ flame, and 0.04–2 g/mL range.

(b) *Calcium stock solutions*.—Prepare Ca stock solution and working standards as in **965.09B** (see 2.6.01).

(c) *Cu, Fe, Mg, Mn, and Zn stock solutions*.—Prepare as in **965.09B(b)**, (c), and (e)–(g) (see 2.6.01).

(d) *Working standard solutions*.—Dilute aliquots of solutions, (c), with 10% HCl to make 4 standard solutions of each element within range of determination.

B. Preparation of Test Solutions

(a) *Dry ashing*.—Accurately weigh 1 g test portion, dried and ground as in **922.02(a)** (see 3.1.02), into glazed, high-form porcelain crucible. Ash 2 h at 500°C, and let cool. Wet ash with 10 drops H₂O, and carefully add 3–4 mL HNO₃ (1 + 1). Evaporate excess HNO₃

on hot plate set at 100–120°C. Return crucible to furnace and ash additional 1 h at 500°C. Cool crucible, dissolve ash in 10 mL HCl (1 + 1), and transfer quantitatively to 50 mL volumetric flask.

(b) *Wet ashing*.—Accurately weigh 1 g test portion, dried and ground as in **922.02(a)** (see 3.1.02), into 150 mL Pyrex beaker. Add 10 mL HNO₃ and let soak thoroughly. Add 3 mL 60% HClO₄ and heat on hot plate, slowly at first, until frothing ceases. Heat until HNO₃ is almost evaporated. If charring occurs, cool, add 10 mL HNO₃, and continue heating. Heat to white fumes of HClO₄. Cool, add 10 mL HCl (1 + 1), and transfer quantitatively to 50 mL volumetric flask.

C. Determination

To solution in 50 mL volumetric flask, add 10 mL 5% La solution, **965.09B(d)** (see 2.6.01), and dilute to volume. Let silica settle, decant supernate, and proceed as in **965.09D** (see 2.6.01).

Make necessary dilutions with 10% HCl to obtain solutions within range of instrument.

D. Calculations

$$\text{Element, ppm (g/g)} = (\text{g/mL}) \quad F/\text{g test portion}$$

$$\text{Element, \%} = \text{ppm (g/g)} \quad 10^{-4}$$

where $F = (\text{mL original dilution} - \text{mL final dilution})/\text{mL aliquot}$ if original 50 mL is diluted.

Reference: *JAOAC* **58**, 436(1975).

Revised: March 1996