

Atomic Absorption Method Guide

Ca in Plant Materials

Key Words

- Plant Materials
- Calcium
- Flame
- Atomic Absorption

Principle

The sample is digested in mixed nitric/sulphuric/perchloric acids, and calcium is determined by flame atomic absorption spectrometry using a nitrous oxide-acetylene flame. ~ 2000 mg/L of potassium ionisation buffer is added to all solutions.

Reagents

Nitric acid (AnalaR grade, concentrated, s.g. 1.42)

Sulphuric acid (AnalaR grade, concentrated, s.g. 1.84)

Perchloric acid (AnalaR grade, concentrated, 72 %)

Potassium chloride (AnalaR grade)

Calcium master standard (1000 mg/L, Spectrosol or equivalent)

Calcium sub-stock standard solution (25.0 mg/L)

Transfer 2.5 mL of calcium master standard solution to a 100.0mL volumetric flask, dilute to volume with deionised water.

Working standards

Transfer 0, 5.0 and 10.0 mL of the calcium sub-stock standard solution into a series of 100 mL volumetric flasks containing 20 mL of deionised water. Add 1.0 mL of sulphuric acid and 0.5 g of potassium chloride to each flask and dilute to volume with deionised water. The working standards will contain 0, 1.25 and 2.50 mg/L of calcium.

Sample Preparation

Weigh 0.200 g of dry plant material into a 100 mL long necked Kjeldahl flask, add 1.0mL of sulphuric acid, 5.0 mL of nitric acid and 1.0 mL of perchloric acid. Heat gently until the initial reaction subsides, then heat more strongly until white fumes of sulphuric acid appear. Continue to heat for 15 minutes, then cool and transfer to a 100.0 mL volumetric flask and dilute to volume with deionised water. The total digestion time will be 1-1.5 hours. Transfer 10.0 mL of this solution to a 100 mL flask, add 1.0 mL of sulphuric acid and 0.5 g of potassium chloride and dilute to volume with deionised water. 2.5 mg/L in solution is equivalent to 1.25 % m/m of calcium in the original sample.

Instrument Parameters

The screenshot displays two panels of the instrument software interface for 'Ca plants (Ca)'. The top panel, titled 'Measurement Mode', shows settings for 'Absorption' with a 'Cook Book' button. Parameters include: Number of Resamples: 3; Fast Resamples: checked; Measurement Time: 4.0 s; Wavelength: 422.7 nm; Lamp Current: 100%; Bandpass: 0.5 nm. It also features checkboxes for 'Optimise Spectrometer Parameters' (unchecked), 'High Resolution' (unchecked), 'Filer Rejection' (unchecked), and 'Use Filer Rejection' (unchecked). A 'Rejection Limit (%)' is set to 95. An 'RSD Test' section includes 'Use Test' (unchecked), 'If RSD greater than' (blank), 'AND signal greater than' (0.1 Abs), and an 'Then' action of 'Flag and Continue'. The bottom panel, titled 'Flame', shows 'Flame Type' as 'Nitrous Oxide-Acetylene' and 'Fuel Flow' as 5.2 L/min. It includes checkboxes for 'Optimise Fuel Flow' (checked) and 'Backup Default' (unchecked). The 'Stabilisation' section has 'Burner Stabilisation Time (min)' at 0 and 'Nebuliser Uptake Time (s)' at 4. The 'Burner Height' section has 'Burner Height (mm)' at 11.0 and 'Optimise Burner Height' checked.

Figure 1: Instrument parameters

Results

Sample	Heather (1)	Heather (2)	Oak leaves	Peat
Calcium found (%m/m)	0.18	0.32	2.80	0.11
Reference value (%m/m)	0.17 - 0.19	0.31 - 0.33	2.74 - 2.87	0.10 - 0.11

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