

Atomic Absorption Full Method - Ca and Mg in a variety of food oils

Key Words

- Atomic absorption
- Calcium
- Magnesium
- Food oils
- FAAS

Principle:

This application note presents a straightforward system for the analysis of calcium and magnesium in a variety of food oils. Calcium and magnesium are determined directly in a variety of food oils using Flame Atomic Absorption Spectrometry. Quadline D₂ background correction is used throughout. Calibration can be performed directly using standards diluted with white spirit.

Extreme care should be taken when using solvents with AA and all applicable safety precautions should be taken as good laboratory practices employed.

Analytical range:

Methods for the direct determination of both calcium and magnesium in a variety of food oils are presented. The 3 σ method detection limit for the calibration method is 4 μ g/L for magnesium and 50 μ g/L for calcium (as calculated by the SOLAAR software Detection limit wizard).

4) Instrumentation

This analysis can be performed on any flame capable AA that is fitted with fully automatic gas control facilities.

The spectrometer was fitted with a Solvent Resistant Flame Kit.

A 100 mm titanium burner was fitted to provide greater sensitivity.

5) Methods

Reagents:

A Turpentine Substitute (White Spirit) UN number 1300, conforming to BS245, was obtained from the local hardware store.

Standards:

Both calcium and magnesium metallo-organic oil soluble standards were obtained from Conostan Speciality Product Inc. (Ponca City, Oklahoma, USA). These contained 5000 mg/Kg of each of the metals in a mineral oil base.

6) Sample collection

Calibration method:

Approximately 0.2 g of the magnesium Conostan standard and approximately 2 g of the calcium standard were accurately weighed, mixed and diluted to approximately 100 g with white spirit, to give an intermediate standard containing approximately 10 mg/Kg of magnesium and 100 mg/Kg of calcium.

Approximately 0.5 g, 2.5 g and 5 g of this intermediate standard were then weighed out exactly and each was further diluted to approximately 100 g with white spirit. This produced three working standards containing approximately 0.05, 0.25 and 0.5 mg/Kg of magnesium, and 0.5, 2.5 and 5.0 mg/Kg of calcium.

Sample preparation:

Various different types of oil were tested.

1 g portions of each of the oils were weighed exactly, and diluted to 20 g with white spirit.

7) Method settings

Ca

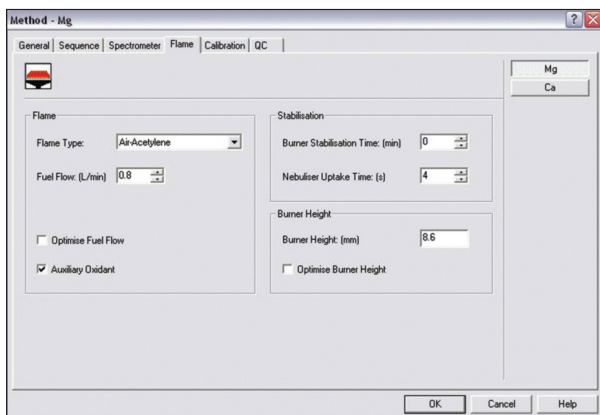
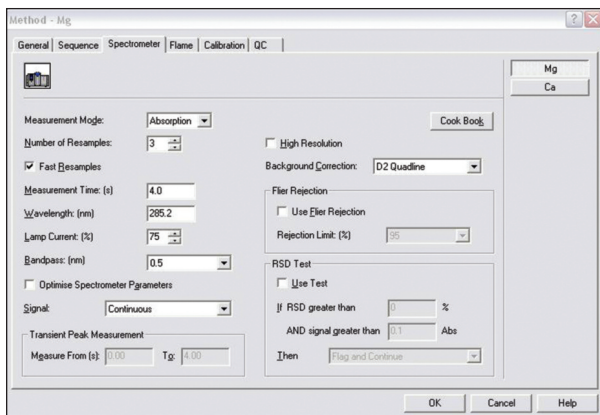
The screenshot shows the 'Method - Ca' software window with the following settings:

- Measurement Mode: Absorption
- Number of Resamples: 3
- Fast Resamples: checked
- Measurement Time (s): 4.0
- Wavelength (nm): 422.7
- Lamp Current (%): 100
- Bandpass (nm): 0.5
- Optimise Spectrometer Parameters: unchecked
- Signal: Continuous
- Transient Peak Measurement: Measure From (s): 0.00, Tg: 4.00
- High Resolution: unchecked
- Background Correction: D2 Quadline
- Filter Rejection: unchecked
- Use Filter Rejection: unchecked
- Rejection Limit (R): 95
- RSD Test: unchecked
- Use Test: unchecked
- If RSD greater than: 0 %
- AND signal greater than: 0.1 Abs
- Then: Flag and Continue

The screenshot shows the 'Method - Ca' software window with the following settings:

- Flame Type: Air-Acetylene
- Fuel Flow (L/min): 0.9
- Optimise Fuel Flow: unchecked
- Auxiliary Oxidant: checked
- Stabilisation: Burner Stabilisation Time (min): 0, Nebuliser Uptake Time (s): 4
- Burner Height: Burner Height (mm): 11.8
- Optimise Burner Height: unchecked

For simplicity an air-acetylene flame was used for the analysis of Ca.



The Auxiliary Oxidant function was selected for the flame parameters and the fuel gas flow rate was optimised by observing the appearance of the flame while aspirating clean solvent. This function provides an additional flow of air to the flame that does not pass through the nebuliser. The additional supply of air provided by the Auxiliary Oxidant compensates for the extra fuel added to the flame when an organic solvent is aspirated, allowing the normal flame chemistry to be maintained.

The burner height position was optimised using the automatic optimisation facilities provided by the SOLAAR software.

The position of the impact bead and the transverse and rotational position of the burner head were optimised with the appropriate Wizard provided in the SOLAAR software.

9) Results

Calibration curves were generated for both calcium and magnesium standards in white spirit. The data can be seen in figures 1 and 2. There is no appreciable curve in either of the fits so samples were analysed over the entire range of the standard curve.

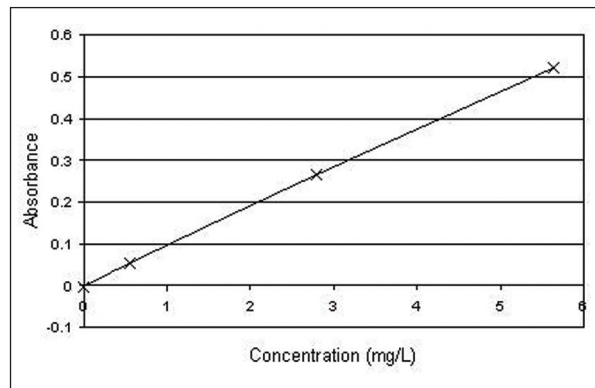


Fig 1: Calibration curve for calcium

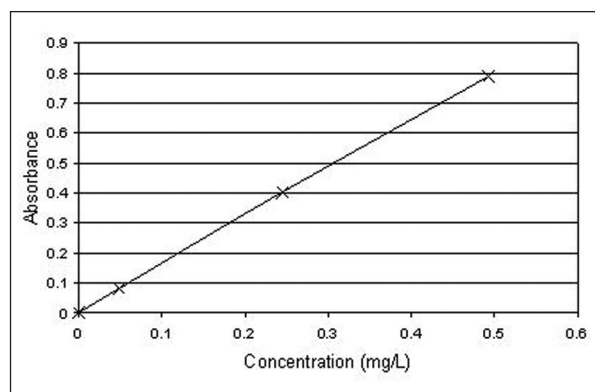


Fig 2: Calibration curve for magnesium

The results for the analysis of both calcium and calcium in the five different food oils are shown in the table below.

Sample	Groundnut oil	Vegetable oil	Rapeseed oil	Sunflower oil	Grapeseed oil
Final Mg concentration (mg/Kg)	Nd	nd	0.232	0.404	0.043
Final Ca concentration (mg/Kg)	0.640	0.619	2.28	1.64	0.612

*nd = not detected

Table 1: Concentrations of both calcium and magnesium in each of the samples tested

The analysis of spiked samples gave recoveries of 92 % or more. This could be improved further by using an oil blank to match the viscosity.

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