



The Analysis of Cement

Application Note

Atomic Absorption

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Introduction

The atomic absorption technique may be used for the determination of a wide range of elements in various samples of cement.

Several methods can be used for the preparation of sample solutions, which are suitable for analysis, by atomic absorption spectrophotometry.

When silicon has to be determined the cement samples can be fused with an alkali metal salt, but a hydrochloric acid leach is adequate when the silicon content is not required.

Both of the above procedures for obtaining cement sample solutions have been tested and are employed at Agilent Technologies, Inc.



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Experimental

Fusion Technique

Fuse 0.5 g of the sample of cement with 2 g of either NaOH or Na_2O_2 in either a platinum or a zirconium crucible over a Meker burner.

If a platinum crucible is used do not raise the temperature above a dull red heat or prolong the fusion unnecessarily.

Cool the melt and dissolve the fused cake in 100 mL of warm distilled water and acidify by the addition of 15 mL of conc. HCl, and finally add 5 mL of a 3% H_2O_2 solution. Do not boil the solution.

Transfer the solution quantitatively to a 250 mL volumetric flask and make up to volume with distilled water.

This sample solution is suitable for the determination of the following elements:

Silicon, Aluminium, Magnesium and Calcium.

Notes on the Individual Determinations

Silicon

In the determination of silicon it is necessary to match standards with samples by the addition of both sodium and aluminium, since both of these metals enhance silicon absorption additively by approximately 10%. The standards should also contain an equivalent amount of HCl to that used in the sample preparation.

It should be noted that silica does not precipitate readily from acid solutions, provided that the normality does not exceed 3N and provided that the acidic solution is not boiled.

The atomic absorption measurements are made at the 2516.1 Å resonance line, using a fuel-rich (reducing) $\text{N}_2\text{O}-\text{C}_2\text{H}_2$ flame.

Aluminium

In the determination of aluminium the standards are matched with sample solutions for sodium, calcium and silicon. Both sodium and calcium enhance aluminium absorption, while silicon depresses it.

The atomic absorption measurements are made at the 3092.7 Å, 3092.8 Å doublet, using a $\text{N}_2\text{O}-\text{C}_2\text{H}_2$ flame.

Magnesium

In the determination of magnesium the standards are matched with sample solutions with respect to their sodium content, since sodium acts as an ionization suppressant and hence its presence overcomes the slight ionization interference. If an air-acetylene flame is used the standards are also matched for aluminium and silicon content.

The atomic absorption measurements are made at the 30 times less sensitive 2025 Å resonance line rather than at the more sensitive 2852.1 Å line to avoid time-consuming dilutions, and in this case it is recommended that the lamp is operated at 8 mA.

Generally, the air- C_2H_2 flame is used in magnesium determinations, although the $\text{N}_2\text{O}-\text{C}_2\text{H}_2$ flame is especially recommended if aluminium and/or silicon is present. However, in these types of matrices, the N_2O -propane flame (~2400 °C) also appears to be quite satisfactory, from the point of view of ionization and interferences.

Calcium

As with magnesium, so with calcium, the standards are matched with the sample solutions with respect to their sodium content. If an air-acetylene flame is used they should also be matched for aluminium and silicon content.

The atomic absorption measurements are made at the 2398.6 Å resonance line, rather than at the 200 times more sensitive 4226.7 Å line, using a $\text{N}_2\text{O}-\text{C}_2\text{H}_2$ flame. It is recommended that the lamp should be operated at 10 mA since the 2398.6 Å line is weak in signal intensity.

Acid Leaching Technique

Digest in a tall beaker 2 g of a finely divided cement sample with 10 mL of 1:1 v/v HCl at just below the boiling point for 5–10 minutes. Transfer the contents of the beaker quantitatively to a 100-mL volumetric flask and make up to volume with distilled water.

Allow the sediment to settle and sample the supernatant liquid, or either filter or centrifuge the solution prior to making measurements upon the solution.

The following metals may be determined in this solution:

Iron, Sodium and Potassium (as well as Nickel or Chromium if desired).

Notes on the Individual Determinations

Iron

The atomic absorption measurements are carried out on a diluted solution (~10 times) at the 3719.9 Å line, rather than the 9 times more sensitive 2483.3 Å line, using an air-C₂H₂ flame.

The sample solution obtained from the fusion procedure could also be used. However, it is then necessary to match standards and samples for both sodium and HCl content.

Sodium

The atomic absorption measurements are made on a diluted solution (~10 times) at the 5890 Å resonance line, using the air-C₂H₂ flame and the burner rotated at 90 degrees to the optical path in order to reduce the analytical sensitivity by a factor of about fifteen.

Dilution may be avoided by the use of the much less sensitive (550 times) 3302.3 Å, 3303.0 Å doublet without burner rotation. In either case the cooler air-propane flame may also be used.

Potassium

The atomic absorption measurements are made on a diluted solution (~10 times) at the 7664.9 Å resonance line, using an air-C₂H₂ flame together with a very small rotation of the burner. Rotation may be avoided by using an air-acetylene flame burning at the 5 cm nitrous oxide-acetylene burner. This is quite safe.

Some Typical Results

Table 1. Typical Results

Component	Sample A		Sample B	
	Supplied analysis%	A. A. S.%	Supplied analysis%	A. A. S.%
SiO ₂	24.17	24.20	21.03	20.59
Al ₂ O ₃	3.3	3.10	5.38	5.02
Fe ₂ O ₃	3.07	3.05	2.07	2.11
CaO	64.43	65.5	66.6	66.8
MgO	1.39	1.37	1.12	1.12
Na ₂ O	0.2	0.17	0.08	0.07
K ₂ O	0.32	0.32	0.26	0.26

Conclusion

An estimated time of analysis for all seven elements in two cement samples, in a routine laboratory, is approximately 2½ hours from the time of receipt of the samples. The efficiency can be greatly increased by carrying out the atomic absorption measurements on a larger number of samples.

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