AAS
APPLICATION NOTES

Analysis of rubber samples by flame atomic absorption

GBC

Introduction

Determination of zinc, cobalt, lead, manganese and silicon levels in rubber is common practice for rubber manufacturers. Variations in the make-up of the matrix however, present the analyst with several paths to follow in method development.

The nature of the rubber matrix depends on the sample. Natural latex, raw synthetic rubbers and vulcanized rubbers all require different dissolution procedures.

The ASTM method D4004-82 outlines ashing and dissolution procedures for various classes of rubber. The criteria determining method choice include presence of halogens or silicon fillers (often kaolin) and whether the rubber is naturally occurring or synthetic.

The procedure chosen for this analysis was Method B. This method assumes the presence of silicon filler, but no halogen. One sample was analysed for zinc and a second sample for silicon, cobalt and zinc.

Experimental

Instrumentation

The GBC atomic absorption spectrophotometer was used for all measurements. This instrument allows direct calibration in the standard additions mode using a least squares regression, and it provides an output of the standard additions calibration graphs. It also prints the largest deviation from the fitted line to alert the analyst to calibration curvature or faulty standard preparation. The normal calibration mode allows direct concentration readout after interpolation from a calibration curve. The instrumental conditions used are shown in Table 1.

The silicon determination was carried out in a nitrous oxide-acetylene flame with a reduced sample uptake rate. Cobalt and zinc were determined using an air-acetylene flame with the maximum sample uptake rate.

Element	Background Correction	Wavelength	Slit Width	Lamp Current
Si	On	251.6 nm	0.2 nm	15 mA
Co	On	240.7 nm	0.2 nm	6 mA
Zn	On	213.9 nm	0.5 nm	5 mA

Table 1: Operating conditions

Sample preparation

Approximately 1 g of shredded rubber was placed into to a 50 cm^3 nickel crucible and left in a muffle furnace at 250°C for 30 minutes. The temperature was then raised to 550°C ± 25°C for four hours and allowed to cool once ashing was complete. 2 g of a 1:1, Na_2O_2:NaOH mixture was added to the crucible and the contents were fused using a Bunsen burner. 6 M HCl was used to wash the crucible, and the washings were transferred to a 250 mL volumetric flask. The volumetric flask was filled to the mark with distilled water. For the zinc determination, a further 1 in 50 dilution was required to bring the sample into the normal working range.

NOTE:
The ASTM method recommends a 1:3, $Na_2B_4O_7$:Na_2CO_3 fusion. This was modified because the peroxide is a more powerful oxidant with a lower fusion temperature.

Table 2 shows the metal levels expected by the manufacturer in the solid rubber samples.

Element	Sample 1	Sample 2
Si	/	<10%
Co	/	0.1%
Zn	approx. 1%	1%

Table 2: Manufacturer's specification

Results

Normal three-standard calibration curves were used to determine zinc in Sample 1, and silicon, cobalt and zinc in Sample 2. Zinc was also determined in both samples using the method of standard additions. Figure 1 shows a complete printout of the results and the standard additions graph for Sample 2. The measured concentration of 1.00 mg/L in the digested sample was equivalent to 1.20% zinc in the solid rubber. The results of all the measurements are summarized in Table 3. The excellent agreement between the results of the normal calibration and the standard additions method for zinc confirms the absence of any matrix interference.

Element	Calibration Method	Sample 1	Sample 2
Si	Normal Calibration	/	2.24%
Co	Normal Calibration	/	0.088%
Zn	Normal Calibration	1.04%	1.21%
Zn	Standard Additions	1.00%	1.20%

Table 3: Results

Standards Details	
Total Volume (µL)	25.00
Sample Volume (µL)	1.000
Number of Additions	2
Standard Volume (µL)	0.500
Standard Concentration	10.00

Calibration					
Standard/Blank	**Reading 1**	**Reading 2**	**Reading 3**	**Mean**	**RSD (%)**
Blank 1	0.021	0.015	0.016	0.018	–
Sample	0.052	0.050	0.048	0.050	4.04
Blank 2	0.005	0.005	0.002	0.004	–
Addition 1	0.201	0.201	0.197	0.199	1.16
Blank 3	–0.010	–0.009	–0.012	–0.010	–
Addition 2	0.348	0.344	0.350	0.347	0.94
Sample Concentration: 1.000 Largest Deviation: 0.4%					

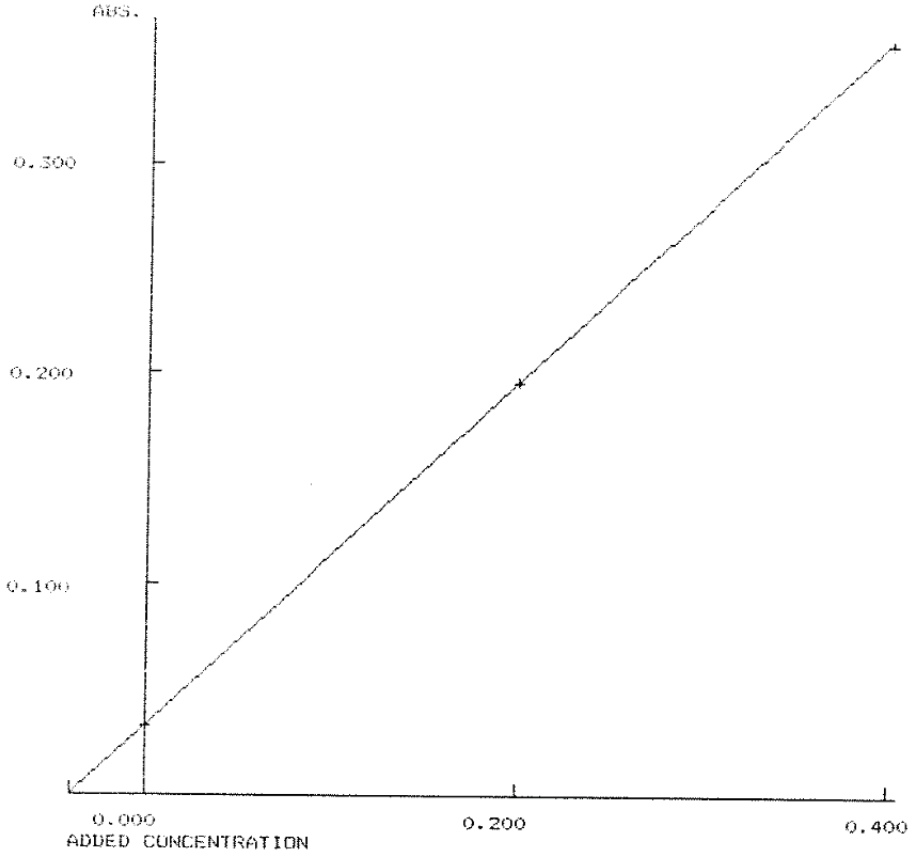

Figure 1: Output of the standard additions runs for zinc in sample 2

Conclusion

The method used for analysing silicon, cobalt and zinc in rubber proved successful, with good verification of results by the standard additions method. The ASTM-recommended sodium borohydride fusion provides adequate results, but the peroxide fusion provides greater efficiency and speed.

GBC Scientific Equipment Pty Ltd
A.B.N. 30 005 472 686
4 Lakewood Boulevard, Braeside Victoria 3195, Australia
Phone: 61 3 9588 6666 Fax: 61 3 9588 6677
e-mail: gbc@gbcsci.com Website: www.gbcsci.com

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