AAS
APPLICATION NOTES

Determination of Sb, Ba and Pb in gunpowder residue analysis

GBC

Introduction

In forensics, gunpowder analysis is used to help police determine many details about a crime, such as whether a suspect actually used a firearm, the type of firearm used, or which hand was used to pull the trigger.

Fired smokeless powder can be collected from the hands of shooters, clothing, spent shell casings or explosive remains.

The elements Antimony, Lead and Barium are found in the primers of nearly all centrefire cartridges but depending on the type of gun, the ratio that these will be found in will vary.

This application shows how Antimony, Barium and Lead can be determined using the GBC Zeeman atomic absorption spectrometer with the PAL autosampler.

Sample collection and preparation

Four drops of concentrated HNO_3 were placed on a cotton bud. The swabbed cotton bud was then placed in a 100 mL beaker containing 25 mL of 5% HNO_3. The beaker containing the swab was then covered with Parafilm™ and heated to 65°C for five minutes. Care was taken not to exceed 70°C. The beaker was then left to stand overnight.

The swab was removed and the 25 mL of solution was quantitatively transferred to a 100 mL volumetric flask and made to volume using Distilled/De-ionized water.

In forensics more than one swabbing is usually taken. A Control Swab must always be taken. This swab is the cotton bud, without swabbing, taken through the entire procedure described above to determine whether any antimony, barium or lead comes from the cotton bud, acid or water.

Standard preparation

A bulk standard was prepared as per table 1. For each element the PAL was programmed to prepare a calibration curve from the one bulk standard.

Instrumentation

The GBC Zeeman atomic absorption spectrometer and PAL were used. The instrument conditions, modifiers and sample volumes used are shown in Table 1. Furnace programs are shown in Figures 1 to 3.

Element	**Sb**	**Ba**	**Pb**
Wavelength	217.5 nm	553.6 nm	217.0 nm
Lamp Current	15 mA (superlamp)	20 mA	5.0 mA
Slit Width	0.2 nm	0.2 nm	1.0 nm
Measurement Mode	Peak Height	Peak Height	Peak Height
Matrix	1% HNO_3	1% HNO_3	1% HNO_3
Bulk Standard	50 ppb	50 ppb	25 ppb
Modifier	1% $NH_4H_2PO_4$	No modifier	1% $NH_4H_2PO_4$
Sample Volumes	10 µL sample 5 µL modifier	15 µL sample	10 µL sample 5 µL modifier

Table 1: Instrumental parameters used

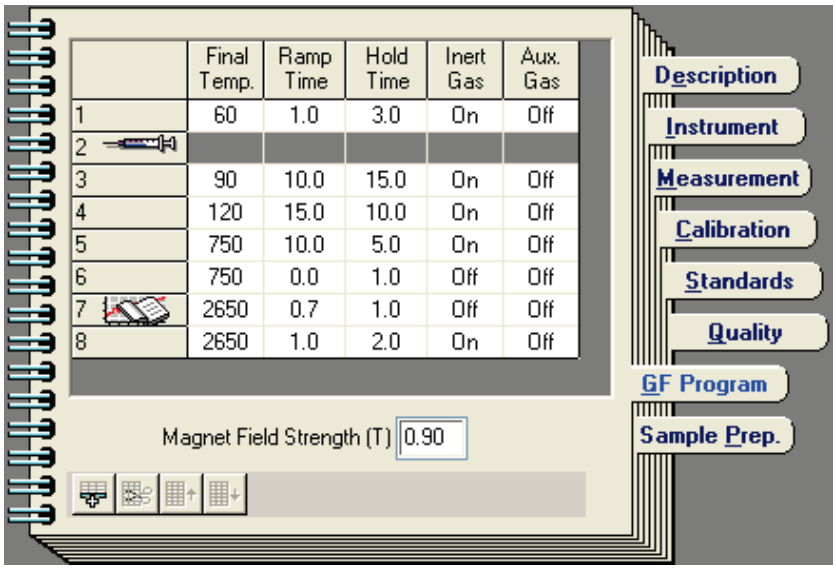

Figure 1: Antimony furnace program

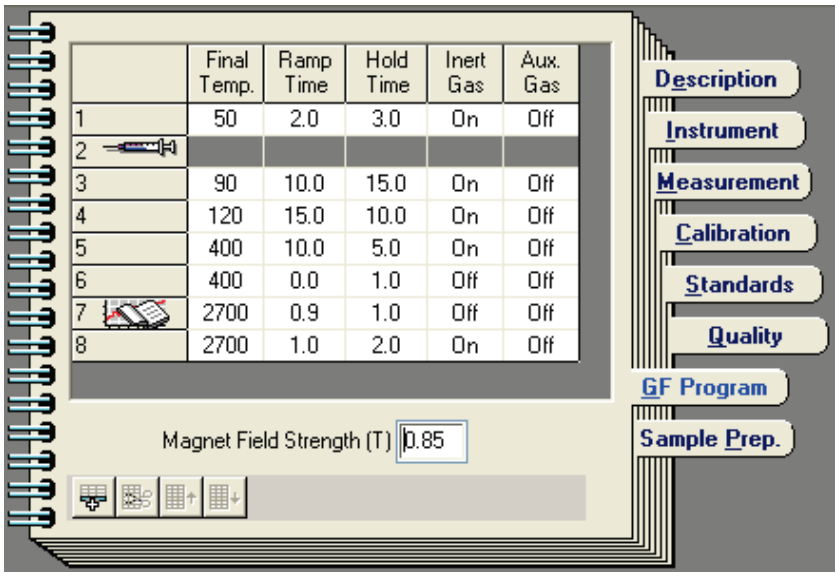

Figure 2: Barium furnace program

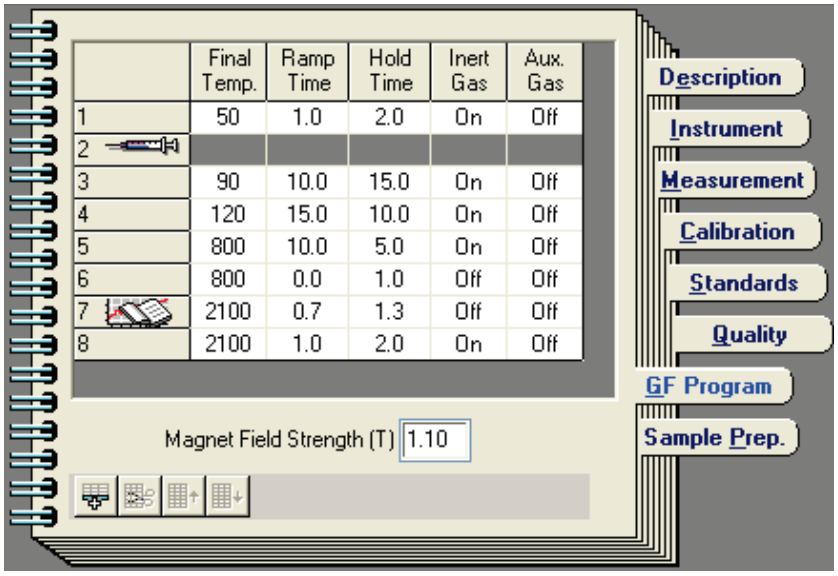

Figure 3: Lead furnace program

Results

Figures 4 to 6 shows the various dilutions performed and the excellent peak shapes and reproducibility obtained for the various elements.

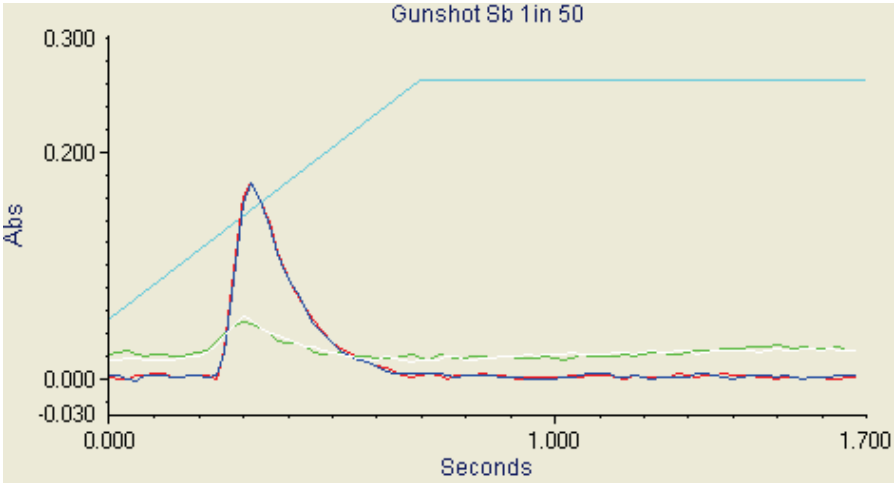

Figure 4: Antimony peak for a 1 in 50 sample dilution

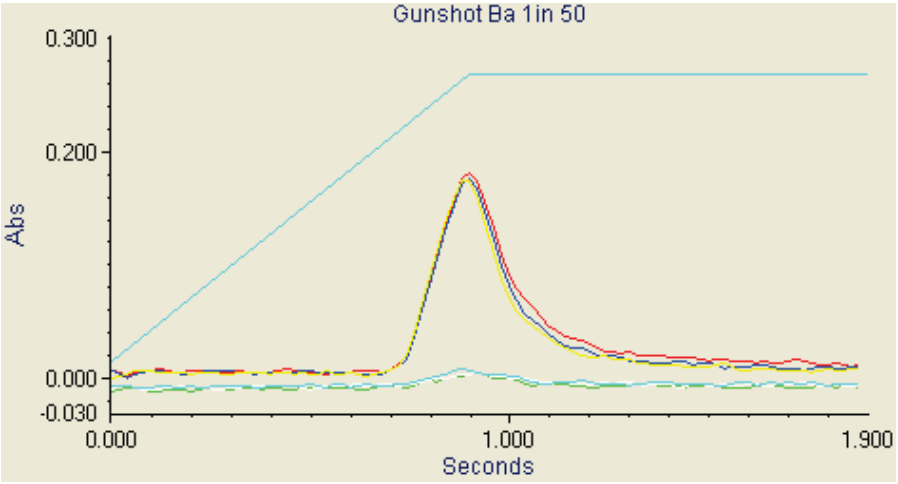

Figure 5: Barium peak for a 1 in 50 sample dilution

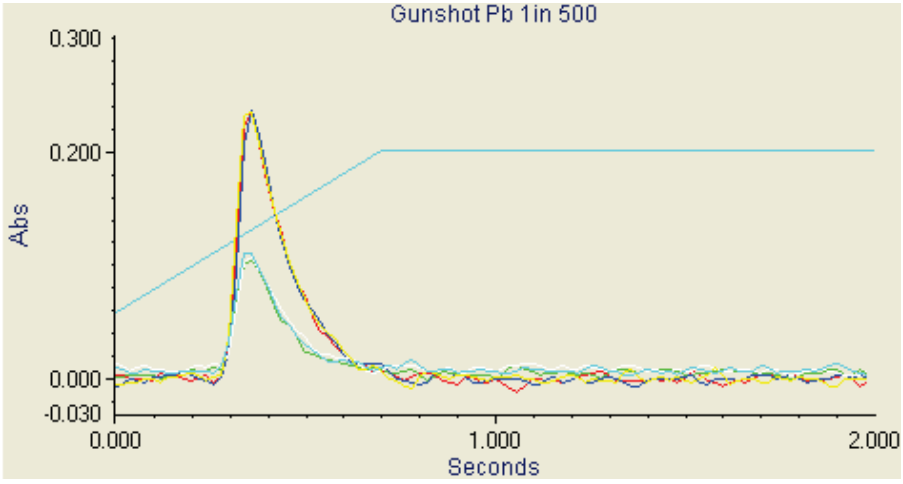

Figure 6: Lead peak for a 1 in 500 sample dilution

Discussion

While there are many different techniques for measuring the ratios of Sb, Pb and Ba in gunshot residues, using the GBC Zeeman atomic absorption spectrometer is by far one of the easiest and most sensitive. It is also one of the most inexpensive techniques compared to others such as neutron activated analysis, ICP or scanning electron microscopes.

The sample preparation technique gives a final sample matrix of approximately 1–2% nitric acid. These samples do not require complicated furnace programs required for complicated matrices. The final concentration of the different elements will depend upon the amount of residue swabbed.

Multi-standard calibrations were performed using the one bulk multi-element standard by injecting various volumes. The samples were diluted so that their absorbance was within the linear concentration range.

Conclusion

The sensitivity of the GBC Zeeman atomic absorption spectrometer is very useful to the analyst particularly for the analysis of gunpowder residues.

With characteristic masses of 6.0 pg for antimony, 3.0 pg for lead and 8.0 pg for barium, very low concentrations of gunpowder residue can be determined with confidence.

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Part No. 01-1091-00
October 2021