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APPLICATION NOTES

The determination of lead in blood by graphite furnace AAS

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Introduction

The determination of lead in blood is of considerable interest due to the toxicity of lead and its widespread presence in the environment and the workplace. Atomic absorption, because of its simplicity, sensitivity and specificity, is usually the method of choice for lead determinations.

In blood, the normal level for an unexposed person is less than 0.25 mg/L (ppm). The level for occupationally exposed personnel is 0.25 – 0.80 mg/L and greater than 0.80 mg/L is regarded as excessive. These levels are too low to be accurately measured by flame AA without pre-concentration and so the graphite furnace is usually used. Additional advantages of the graphite furnace for this analysis are its ability to handle viscous samples and the small sample volume needed.

Direct injection of blood into the graphite furnace can cause problems due to frothing of the sample while it is drying. For this reason, the blood is usually mixed with a detergent such as Triton X-100[1,2] or with nitric acid[3] prior to analysis. For this study, the Triton X-100 method was used.

Experimental

Sample preparation

The sample was venous blood from a local hospital and was diluted 1:1 with 1% Triton X-100. Blood is known to produce chemical interferences in lead determinations, so it was necessary to use the method of standard additions for calibration. The additions were prepared automatically using the PAL programmable automatic sample loader.

Instrumentation

The GBC atomic absorption spectrometer and the GBC automated graphite furnace system was used. The GBC graphite furnace system comprises the graphite furnace (GF) and programmable automatic sample loader (PAL). This combination of equipment is particularly suited to the determination of lead in blood since the graphite furnace system can automatically prepare a range of additions and the atomic absorption spectrometer allows direct calibration in the standard additions mode.

An output of the operating parameters for the spectrophotometer, furnace and sampler is shown in Figure 1. The 283.3 nm lead wavelength was used in preference to the 217.0 nm line. Although the sensitivity at 283.3 nm was only half that at 217.0 nm, the noise was lower, the calibration curve was more linear and the background absorbance was lower.

The automatic sampler was programmed to inject a sample volume of 2 μL, a first addition comprising 1 μL of 0.25 mg/L lead and a second addition comprising 2 μL of 0.25 mg/L lead. The appropriate volume of blank solution was automatically added to bring the total aliquot volume to 5 μL in each case. An ash temperature of 450°C was found to be the highest that could be used without a loss of lead in the ash step.

Operating Parameters	
Element	Pb
Beam Mode	Double Beam
Wavelength	283.3 nm
Slit Width	0.5 nm
Atomization	Furnace
Lamp Current	6.0 mA
EHT (gain)	– 328 V
Scale Expansion	1.000
Integration Time	0.02 s

Graphite Furnace Parameters					
Step No.	**Final Temp °C**	**Ramp Time (sec)**	**Hold Time (sec)**	**Gas Type**	**Read On**
1	80	10.0	1.0	None	No
2	120	30.0	5.0	None	No
3	200	10.0	2.0	None	No
4	450	30.0	10.0	Inert	No
5	2000	1.0	3.0	Inert	Yes

Autosampler Details	
Sample Volume (µL)	5.0
No. of Multiple Injections	1
No. of Sample Repeats	3
Blank Repeat Rate (zero on blank)	1
Dry Steps for Multiple Injections	1
Inject on Step Number	1

Aliquot Volumes	
Aliquot Volume (µL)	5.0
Sample Volume (µL)	2.0
Number of Additions	2
Standard Volume (µL)	1.0
Standard Concentration (µg/L)	250

Figure 1: Operating parameters for determining lead in blood

Results

Figure 2 shows the output of the results for this analysis. The automatic sample was programmed for three replicate measurements of each solution with a blank between each solution. The relative standard deviations ranged from 1.3 to 4%.

Results					
Sample/Blank	**Reading 1**	**Reading 2**	**Reading 3**	**Mean**	**RSD (%)**
Blank 1	0.025	0.020	0.019	0.021	–
Sample 1	0.126	0.136	0.132	0.131	3.99
Blank 2	0.037	0.029	0.024	0.030	–
Addition 1	0.245	0.240	0.239	0.242	1.31
Blank 3	0.036	0.027	0.031	0.031	–
Addition 2	0.329	0.320	0.316	0.322	2.08

Figure 2: Results for determining lead in blood

Figure 3 is a graph of the measurement of addition 2. This graph shows a clean, undistorted lead peak.

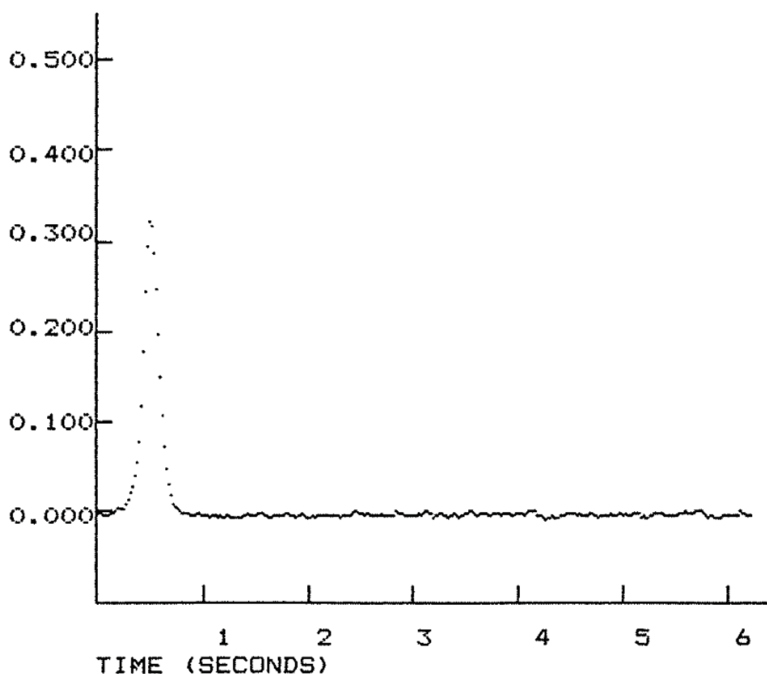

Figure 3: Peak graph, addition 2

An output of the standard additions graph is shown in Figure 4. A feature of the GBC atomic absorption spectrometer is the display of the largest deviation from the line of best fit. This instantly alerts the operator if there is a faulty standard or a curved calibration. The largest deviation with this analysis was an acceptable 3.4%. The calculated sample concentration was 157.5 µg/L. This corresponds to 0.315 mg/L in the undiluted blood. This level is at the top of the normal unexposed range or the bottom of the range for a person with some exposure.

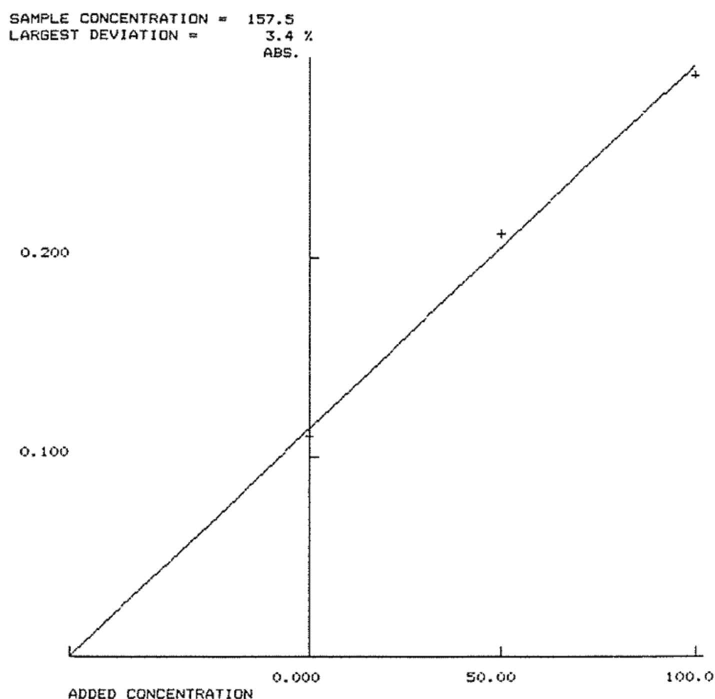

Figure 4: Standard additions graph

Conclusion

A method for the determination of lead in blood has been developed. The method was conveniently automated using the GBC atomic absorption spectrometer and GBC automated graphite furnace system. This system was programmed to automatically load the sample, add the additions and compute the sample concentration. The measure precision of 1–4% was considered very good for the determination of a low concentration of lead (0.3 ppm) in such a complex matrix as whole blood.

References

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