SHIMADZU APPLICATION NEWS

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● SPECTROPHOTOMETRIC ANALYSIS

No.A352

Pb Analysis by Atomic Absorption

Lead has been used in diverse applications since ancient times because it displays excellent workability due to its low melting point and its softness. In addition, due to its high density, it is used as a shielding material for blocking X-rays or γ-rays, as well as in lead glass.

On the other hand, lead is hazardous to human health, its acute poisoning appears as anemia, neuralgia or neurological brain disorders. When lead is ingested orally, it is said that the body absorbs about 10% of the total amount. While most of that is expelled in the feces in the form of lead phosphate or lead carbonate, a portion enters the blood and adheres to the bones and soft tissues.

In the past, lead was used in tap water pipes, however, since it was discovered that it dissolves in the slightly acidic solution produced in the presence of oxygen, lead is now being replaced with stainless steel and synthetic plastic, etc. in tap water plumbing. In addition, the tetraethyl lead that had been added to gasoline as an octane rating enhancing antiknock substance to suppress excessive combustion in automobile engines are now prohibited due to environmental pollution concerns.

In recognition of the hazardous nature of lead, lead-free movements are accelerating in various industries. For example, the RoHS* directive bans the use of specified harmful substances in electrical and electronic products marketed in the EU from the 1st July 2006. The specified hazardous substances include organic halides (brominated flame retardants) and hazardous metals, including mercury, cadmium, hexavalent chromium, along with lead.

Lead analysis is conducted in a variety of fields, including water quality-related analysis on tap water and wastewater, environmental-related analysis on underground water and soil, and quality management analysis on food and pharmaceutical and industrial products, etc.

Introduced here are examples of lead analysis in soil using the flame method, and in plastic using the furnace method.

*RoHS Directive: Restrictions on Hazardous Substances

■ Basic Pb-related Data

Atomic Weight	: 207.2
Melting Point	: 328˚C($PbCl_2$: 501˚C, $PbSO_4$: 1070˚C)
Boiling Point	: 1740˚C ($PbCl_2$: 95˚C)
Oxidation Number	: +2 (e.g. PbO, PbS, $PbCl_2$, etc.)
	+4 (e.g. PbO_2, Pb_3O_4, PbS_2, $PbCl_4$, etc.)
Solubility	: $PbCl_2$ 10.8g/L (25˚C)
	$PbSO_4$ 40mg/L (15˚C)

■ Wavelength of Pb

Wavelength	Sensitivity Ratio
283.3nm	0.4
217.0nm	1.0

Note: Compared to measurements at 217.0nm, measurements at 283.3nm are less influenced by the background due to the longer wavelength. In addition, since the light intensity is greater and baseline noise is smaller, the 283.3nm wavelength is more often used in official methods. The analyses introduced here use the 283.3nm wavelength.

■ Flame Analysis of Pb

A 0.5g sample of volcanic ash/soil standard JSAC-0411 (Elements, Fig. 1) was weighed out, dissolved in aqua regia on a hot plate, and after filtering, was brought to 50mL in a volumetric flask using distilled water for flame analysis (calibration curve method). High sensitivity analysis was performed using the booster technique. The sample peak and the calibration curve are shown in Fig. 2 and Fig. 3, respectively. The analysis result obtained was 19mg/kg, consistent with the guaranteed value.

	As	Be	Cr	Cu	Mn	Ni	Pb	Se
mg/kg	11.3	1.04	23.5	26.7	943	11	18.9	1.32

Fig.1 Constituent Elements of JSAC-0411

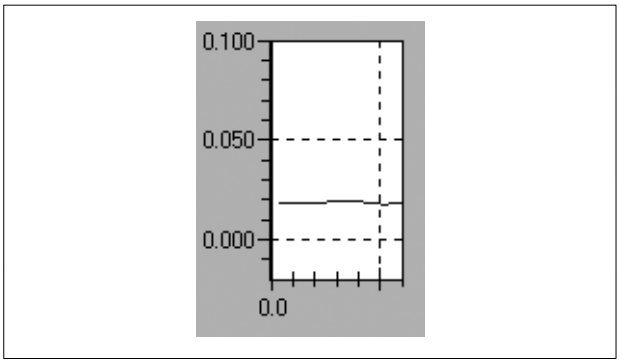

Fig.2 Profile of Flame Method

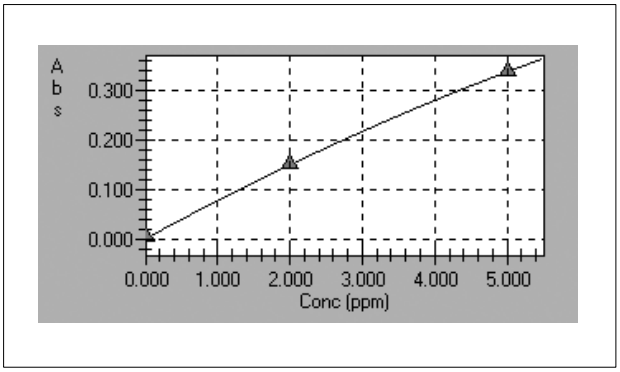

Fig.3 Calibration Curve of Pb (Flame Method)

■ Furnace Analysis of Pb

A 0.1g sample of polyethylene standard BCR-681 (Elements, Fig. 4) was weighed out, dissolved in nitric acid on a hot plate, and after allowing to cool down, was brought to 100mL in a volumetric flask using distilled water for furnace analysis (standard addition method). The sample injection volume was 6 mL. The sample (without added standard) peak and the calibration curve are shown in Fig. 5 and Fig. 6, respectively. The analysis result obtained was 13.7mg/kg, consistent with the guaranteed value. For this analysis, a high density tube was used for the graphite tube, and analysis was conducted using the heating conditions shown in Fig. 7.

	As	Br	Cd	Ci	Cr	Hg	Pb	S
mg/kg	3.93	98	21.7	92.9	17.7	4.50	13.8	78

Fig.4 Constituent Elements of BCR-681

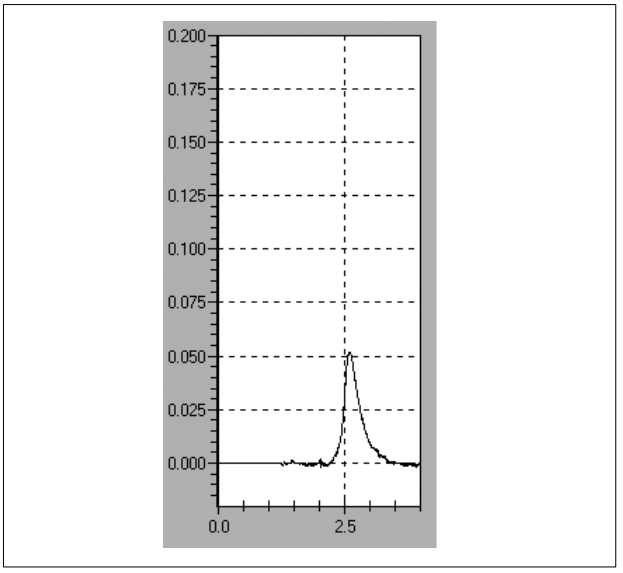

Fig.5 Profile of Furnace Method

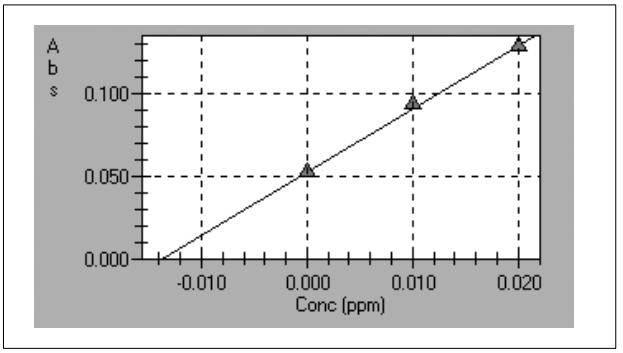

Fig.6 Calibration Curve of Pb (Furnace Method)

	Temperature	Heating Time	Heating Method	Ar Flow Rate
1	150	20	RAMP	0.1
2	250	10	RAMP	0.1
3	800	10	RAMP	1.0
4	800	10	STEP	1.0
5	2400	2	STEP	0.1
6	2500	2	STEP	1.0

Fig.7 Heating Conditions

■ Conclusion

In atomic absorption analysis, it is always necessary to be careful of interference due to substances existing in the sample, regardless of the target element. One effective method used to suppress this interference is to add a matrix modifier.

In case of the flame analysis, if carbonates, phosphates, iodide or fluoride compounds are present in the sample solution at more than 10 times the amount of the lead, negative interference can occur. In this situation, the interference can be suppressed by adding about 0.1M of EDTA.

In case of the furnace analysis, the presence of chloride ions causes the formation of lead chloride. Since the boiling points of chlorides are generally low, so they are easily sublimated during the charring stage, and that causes deterioration in sensitivity and reproducibility. To suppress this, the addition of Pd nitrate to the sample solutions is widely adopted. Pd belongs to the platinum group, and is also used as a catalyst. Since it forms compounds with lead that are thermally stable, sublimation of the lead is effectively suppressed.

The addition of Pd is adopted in official test methods, including JIS K 0101 (Testing methods for industrial water) and JIS K 0102 (Testing methods for industrial wastewater).

It is expected that high-sensitivity analysis of lead will be increasingly important with the tightening of regulations. Lead is said to be comparatively free from contamination during analysis. However, when analyzing trace amounts of lead, care is required for contamination from the environment, equipment, and reagents.

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