

Detection of Pb in Polydimethylsiloxane

1 Sample solution preparation :

About 2.0g liquid sample was weighed in a porcelain crucible, heated on an electric hot plate to carbonize on small heat until smoke stops, then moved into a muffle furnace and ashed at $600\text{ }^{\circ}\text{C} \pm 5\text{ }^{\circ}\text{C}$ for about 5 hours. After cooling down, 1 mL of mixed acid (nitric acid + perchloric acid = 9 +1) was added to dissolve the residue, washed with deionized water and transferred into a 100 mL PTFE beaker. 2 mL hydrofluoric acid was added, then heated on hot plate for digestion and to reduce acid to near dry. The crucible wall was rinsed with deionized water, then evaporated again until dry, then rinsed with deionized water and transferred into a 10mL volumetric flask. Shaken well and spared for later use.

2 Experimental equipment and reagents

AA7000 series atomic absorption spectrophotometer (with Pb, Cd hollow cathode lamp, EWAI Inc.)

Temperature-controlled hot plate

Muffle furnace

PTFE beaker

Nitric acid (HNO₃): excellent grade purity

Perchloric acid (HClO₄): excellent grade purity

Hydrofluoric acid (HF): excellent grade purity

2% ammonium dihydrogen phosphate: 2.0g analytical-grade ammonium dihydrogen phosphate was weighed, dissolved and transferred to a 100mL volumetric flask with deionized water, and then diluted up to the volume with deionized water.

Lead standard solution (National Reference Materials Research Center)

3 Instrument conditions

Element	Wavelength (λ /nm)	Lamp current (I/mA)	D2 current (I/mA)	bandpass width ($\Delta\lambda$ /nm)	Background correction
Pb	283.31	1.3	100	0.2	D2 lamp

Pb graphite furnace heating program

Id	Step	Start temp	End temp	Heating time	Inner gas	Auxiliary gas	Mode
1	Drying	50	100	30	Open	Off	Power
2	Drying	100	120	10	Open	Off	Power
3	Ashing	120	600	10	Open	Off	Power
4	Ashing	600	600	10	Open	Off	Power
5	Ashing	600	600	3	Off	Off	Power
6	Atomization	2000	2000	3	Off	Off	Power
7	Cleaning	2200	2200	3	Open	Off	Power
8	Cooling	0	0	18	Open	Off	Power
9	Cooling	0	0	1	Off	Off	Power

4 Standard solution preparation

Element	Concentration (ng/mL)					
Pb	0	2.5	5.0	10.0	20.0	30.0

5 Standard curve

