



Determination of lead in soil using the Thermo Scientific iCE FIOS AAS

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Keywords

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Goal

Demonstrate the suitability of AAS for the determination of lead in soils.

Introduction

Lead is naturally present in soil. It generally occurs in the range of 15 to 40 mg·kg⁻¹. Pollution can increase soil lead levels to several thousand mg·kg⁻¹, the toxic properties of lead have been widely recorded and its use in recent years has declined. The major cause of soil lead contamination in populated areas is the weathering, chipping, scraping, sanding, and sand-blasting of structures bearing lead-based paint.

Due to the nature of the contamination process, lead in soil may be very unevenly distributed. The lead in paint removed from a structure will generally be concentrated near the source, but levels may vary greatly over small distances. Another source of lead may be from lead arsenate which has been used to treat trees in orchards.

Therefore, accurate measurement of lead content in soil is very important. The maintenance of low lead content in soil is extremely important to grow contamination free and healthy fruits, vegetables and other crops.

Here, a fast, accurate and precise method for determination of total lead in soil sample is presented.

Standard and sample preparation

Samples

A soil CRM sample (PID: SQC001-30G, Lot: LRAB7490) was weighed (0.25 g) into PTFE microwave digestion system (Multiwave PRO, Anton Paar) vessels and digested with 3 ml nitric acid (concentrated, trace metal grade), 1 ml hydrochloric acid (concentrated, trace metal grade) and 0.5 ml hydrofluoric acid (concentrated, trace metal grade) using the program in Table 1. The resulting solution was then diluted to 50 ml using with 18 MΩ ultra-pure water (followed by filtration using nylon syringe filter to obtain a clear solution). Three independent samples were prepared to check the method repeatability and reproducibility.

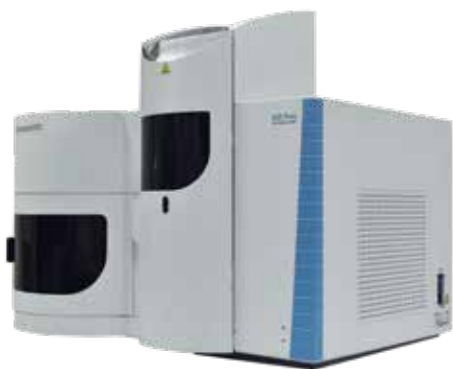
Table 1. Microwave digestion program.

Steps	Temperature (°C)	Time (mm:ss)	Fan level
Temperature ramp	100	10:00	1
Temperature hold	-	5:00	1
Temperature ramp	185	10:00	1
Temperature hold	-	30:00	1
Cooling	65	20:00	3

Standards

Working Standards of lead (1, 1.5, 2 & 2.5 mg·kg⁻¹)

0.05, 0.075, 0.1 and 0.125 mL of the lead standard stock solution (1000 mg·kg⁻¹) was transferred into a series of 50 mL volumetric flasks and diluted to volume with 1% (v/v) nitric acid to prepare working standards with 1, 1.5, 2 and 2.5 mg·kg⁻¹ of lead respectively.



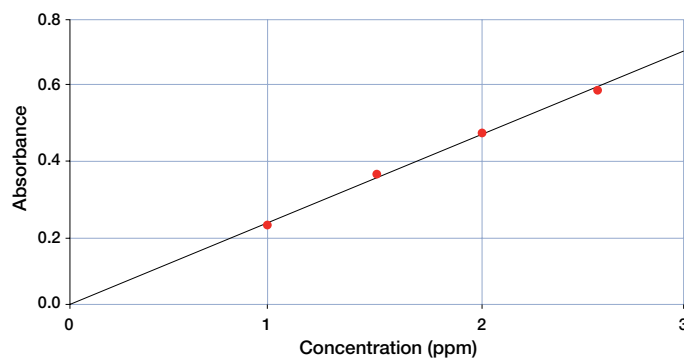
Method

The Thermo Scientific™ iCE™ FIOS™ AAS was set up with method parameters applicable for lead analysis using a lead coded hollow cathode lamp. Method parameters are presented in Table 2.

Table 2. Instrument parameters.

Instrument conditions for Pb	
Turret No.	8
Current (mA)	9.8
D2 Cur (mA)	107
Pmt (v)	423.9
Burner horizontal	3.00
Wavelength (nm)	217.00
Slit (nm)	0.5
Fuel (litre/min)	0.80
Burner height (mm)	6.0
Burner angular	5.00
Calculation mode	LINEAR
R-squared	0.999

Initially, the instrument was calibrated using working standards of lead in the range of 1 mg·kg⁻¹ to 2.5 mg·kg⁻¹, then the sample solutions were aspirated into the iCE FIOS AAS. Absorbance values were recorded for the blank, standard and sample solutions. Concentration values of the unknown samples were calculated directly through software using slope and intercept of linearity plot (Figure 1).



Calibration Graph : (LINEAR) 1.75623 R-squared = 0.999
Eqn (-0.0001+ 0.0238*X)

Figure 1. Standard calibration of lead using the iCE FIOS AAS.

Results

Table 3. Absorbance and concentration values.

Sample name	Absorbance (A)	Concentration (mg·kg ⁻¹)	Weight of sample (g)	Volume made (ml)	Pb content (mg·kg ⁻¹)
Blank	0	0	0	50	0
Standard 1	0.023	1	0	50	0
Standard 2	0.037	1.5	0	50	0
Standard 3	0.048	2	0	50	0
Standard 4	0.059	2.5	0	50	0
Soil sample prep -1	0.039	1.65	0.2585	50	319.1
Soil sample prep -2	0.041	1.68	0.2579	50	325.7
Soil sample prep -3	0.042	1.71	0.2602	50	328.6

Table 4. Comparison of sample results with certified value.

Sample name	Obtained concentration (mg·kg ⁻¹)	Reference value (mg·kg ⁻¹)	% Accuracy
Soil sample prep -1	319.1	280	114.0
Soil sample prep -2	325.7		116.3
Soil sample prep -3	328.6		117.4

Conclusion

The data obtained clearly indicates that, values of lead content in all three-replicate preparation of samples obtained by this method are in close agreement with each other as well as with the certified value given for the CRM.

It concludes that, method presented here using the iCE FIOS AAS is accurate, precise and reproducible for determination of lead content in soil sample.

References

1. <https://ag.umass.edu/soil-plant-nutrient-testing-laboratory/fact-sheets/soil-lead-testing-interpretation-recommendations>

Find out more at thermofisher.com/TEA

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