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

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Goal

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

Introduction

The amount of trace elements found in soil are sometimes so small that they are barely detectable, but without them, plants fail to thrive. Zinc is one of those essential trace elements. Zinc plays a substantial role in many biological processes and is an essential trace element for proper growth and reproduction of plants, and health of animals and humans. It has also been reported to cause contamination of soil, water, and food chains when levels exceed specific concentrations. In human beings zinc deficiency is associated to diet quality, exacerbated by zinc-deficient soils. Soils vulnerable to zinc deficiency, are sandy, calcareous, saline and wetland soils, compacted and rich in organic matter with high nitrogen and phosphate levels. In unfertilized and uncontaminated soil the content of zinc ranges from 10 to 300 $mg \cdot kg^{-1}$ (overall mean of around 50–55 $mg \cdot kg^{-1}$).

The function of zinc is to help the plant produce chlorophyll. Leaves discolor when the soil is deficient in zinc and plant growth is stunted. Zinc deficiency causes a type of leaf discoloration called chlorosis, which causes the tissue between the veins to turn yellow while the veins remain green. Chlorosis in zinc deficiency usually affects the base of the leaf near the stem.

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Chlorosis appears on the lower leaves first, and then gradually moves up the plant. In severe cases, the upper leaves become chlorotic and the lower leaves turn brown or purple and die. When plants show symptoms this severe, it is best to pull them up and treat the soil before replanting.

Therefore, accurate measurement of zinc content in soil is very important to decide proper course of action either to increase or decrease zinc concentration in soil. The maintenance of appropriate zinc content in soil is extremely important to grow crops in a profitable way.

Here, a fast, accurate and precise method for determination of total zinc 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). The solutions were further diluted 10 times (5 ml diluted to 50 ml) to achieve zinc concentration within the linearity range. 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

Intermediate stock solution of zinc (10 mg·kg^{-1})

1.0 mL of zinc standard (1000 mg·kg^{-1}) solution was transferred to a 100.0 mL volumetric flask and diluted to volume with 18 MΩ ultra-pure water.

Working Standards of zinc (0.1, 0.2 and 0.5 mg·kg^{-1})

0.5, 1.0 and 2.5 mL of the zinc intermediate stock solution (10 mg·kg^{-1}) was transferred into a series of 50 mL volumetric flasks and diluted to the volume with 1% (v/v) nitric acid to prepare working standards with 0.1, 0.2 and 0.5 mg·kg^{-1} of zinc respectively.

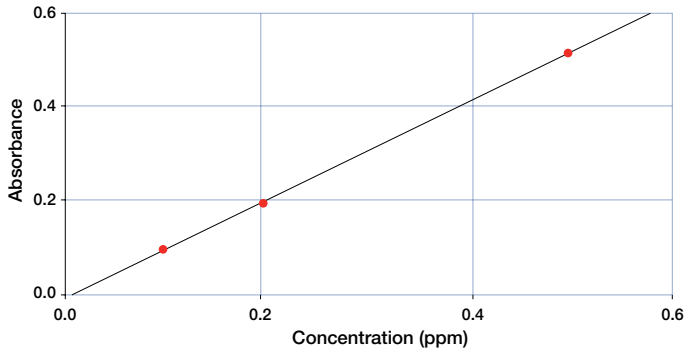
Method

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

Table 2. Instrument parameters.

Instrument conditions for Zn	
Turret No.	2
Current (mA)	5.0
D2 Cur (mA)	128
Pmt (v)	337.7
Burner horizontal	3.00
Wavelength (nm)	213.90
Slit (nm)	1.0
Fuel (litre/min)	0.58
Burner height (mm)	2.0
Burner angular	5.00

Initially, the instrument was calibrated using working standards of zinc in the range of 0.1 mg·kg^{-1} to 0.5 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) 1.38033 R-squared = 1.000
Eqn (-0.0029+ 1.0235*X)

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

Results

Table 3. Absorbance and concentration values.

Sample name	Absorbance (A)	Concentration ($mg \cdot kg^{-1}$)	Weight of sample (g)	Volume made (ml)	Dilution factor	Zn content ($mg \cdot kg^{-1}$)
Blank	0	0	0	50		0
Standard 1	0.101	0.1	0	50		0
Standard 2	0.195	0.2	0	50		0
Standard 3	0.511	0.5	0	50		0
Soil sample prep -1	0.286	0.28	0.248	50	10	564.5
Soil sample prep -2	0.288	0.28	0.2498	50	10	560.4
Soil sample prep -3	0.284	0.28	0.2501	50	10	559.8

Table 4. Comparison of sample results with certified value.

Sample name	Obtained concentration ($mg \cdot kg^{-1}$)	Reference value ($mg \cdot kg^{-1}$)	% Accuracy
Soil sample prep -1	564.5	590	95.7
Soil sample prep -2	560.4		95.0
Soil sample prep -3	559.8		94.9

Conclusion

The data obtained clearly indicates that, values of zinc 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 zinc content in soil sample.

References

1. https://www.gardeningknowhow.com/garden-how-to/soil-fertilizers/zinc-and-plant-growth.htm

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