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

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Goal

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

Introduction

Copper is an essential and important element for plant growth. Soils naturally contain copper in various available forms at concentrations ranging from 2 to 100 parts per million ($mg \cdot kg^{-1}$) and averaging at about 30 $mg \cdot kg^{-1}$. If there is a lack of copper in soil it will affect plant growth. Therefore, maintaining the necessary amounts of copper for healthy plant growth is important.

Soil rarely contains excessive amounts of copper through natural processes, copper toxicity can occur from the repeated use of fungicides that contains copper. Plants subjected to excessive copper appear stunted, are usually bluish in color, and eventually turn yellow or brown. Toxic copper levels reduce seed germination, plant vigor, and iron intake. Removal of excessive copper from soil is extremely difficult as copper has low solubility, which enables it to persist in the soil for many years.

Accurate measurement of copper content in soil is therefore very important in the decisions involving further copper applications to soil. The maintenance of appropriate copper content in soil is extremely important to ensure plant health especially in commercial crops to ensure profitability.

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

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Standard and sample predation

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 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

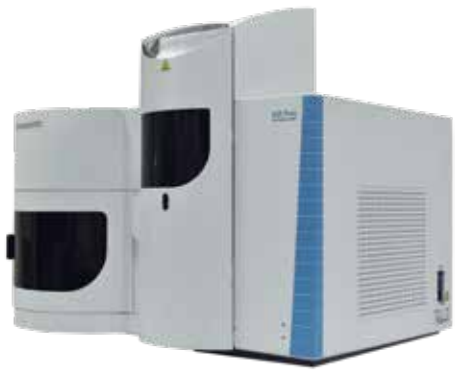
Standards

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

1.0 mL of copper 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 copper (0.1, 0.2, 0.5 and 1 mg·kg^{-1})

0.5, 1.0, 2.5 and 5.0 mL of the copper intermediate stock solution (10 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 0.1, 0.2, 0.5 and 1 mg·kg^{-1} of copper respectively.



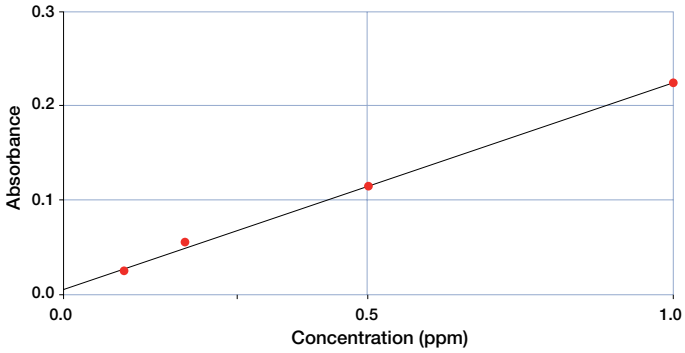
Method

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

Table 2. Instrument parameters.

Instrument conditions for Cu	
Turret No.	1
Current (mA)	2.3
D2 Cur (mA)	219
Pmt (v)	390.7
Burner horizontal	3.00
Wavelength (nm)	324.70
Slit (nm)	0.5
Fuel (litre/min)	0.58
Burner height (mm)	2.0
Burner angular	5.00

Initially, the instrument was calibrated using working standards of copper in the range of 0.1 mg·kg^{-1} to 1 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) 4.33781 R-squared = 0.998
Eqn (0.0042+ 0.2212*X)

Figure 1. Standard calibration for copper 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)	Cu content ($mg \cdot kg^{-1}$)
Blank	0	0	0	50	0
Standard 1	0.022	0.1	0	50	0
Standard 2	0.048	0.2	0	50	0
Standard 3	0.106	0.3	0	50	0
Standard 4	0.196	0.4	0	50	0
Soil sample prep -1	0.090	0.44	0.248	50	88.7
Soil sample prep -2	0.089	0.43	0.2498	50	86.1
Soil sample prep -3	0.093	0.46	0.2501	50	92.0

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	88.7	89.6	99.0
Soil sample prep -2	86.1		96.1
Soil sample prep -3	92.0		102.6

Conclusion

The data obtained clearly indicates that the values of copper 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 the method presented here using the iCE FIOS AAS is accurate, precise and reproducible for determination of copper content in soil sample.

References

1. https://www.gardeningknowhow.com/garden-how-to/soil-fertilizers/copper-for-the-garden.htm

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