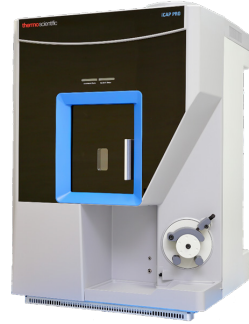


Elemental analysis in Soil and Fertilizer



Mahitti Puanngam



Introduction

Agronomy

(field crop production and soil management)

- Nitrogen and Carbon in soil → evaluate organic matter and calculate the amount of fertilizer to be added.
- Nutrient elements in soil → growth rate.
- Sulfur in soil → its deficiency can have negative influence for the growth of vegetables, particularly in the quality of proteins.
- Toxic elements in soil → Food safety and Human protection.

Soil

- The differentiation of Total Carbon (TC) and Total Organic Carbon (TOC) → evaluate the quality of soils
- Environmental protection → agricultural land, construction sites, playgrounds, forests, and gardens, as well as wastelands.

Fertilizer

- In the production process, the elemental composition of fertilizers is periodically monitored for their characterization.
 - Raw materials.
 - Finish products.

Techniques routinely used for soil analysis

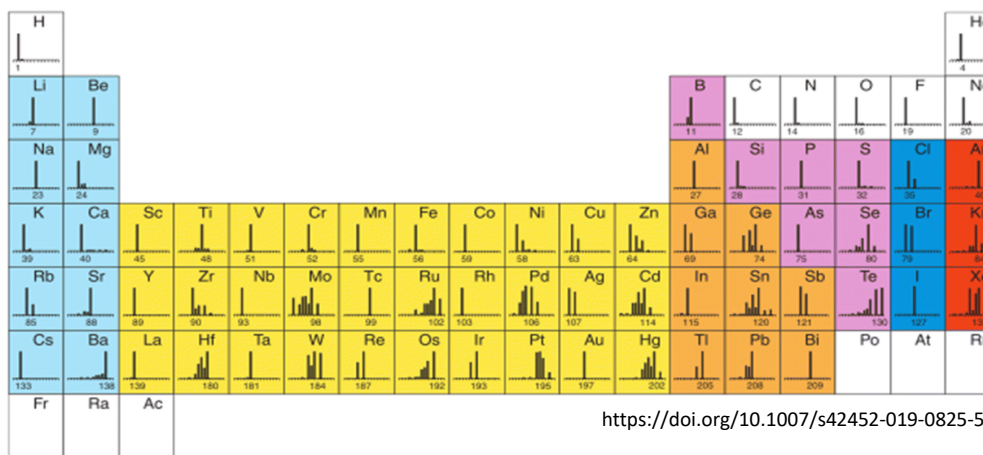
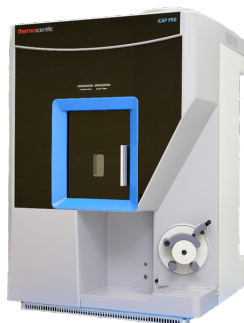
	Organic element analyzer	Discrete analyzer	IC	Accelerated Solvent Extraction	GC & GC-MS	X-ray Fluorescence	ICP-OES & ICP-MS
Nutrient Analysis	●	●	—	—	—	●	●
Metal Contaminants	—	—	—	—	—	●	●
Inorganic Anions	—	—	●	—	—	—	—
Organic Contaminants	—	—	—	●	●	—	—



What technique should be used ?

The Organic Elemental Analyzer is used for Carbon, Hydrogen, Nitrogen, Sulphur and Oxygen analysis.

The ICP-OES and ICP-MS are used to provide information of major plant nutrients (N, P, K), secondary plant nutrients (Ca, S, Mg), micronutrients such as B, Mn, Fe, Cu, Zn, Mo and Se, also the toxic elements (As, Cd, Pb and Hg).



CHNS/O

The Organic Elemental Analyzer



Elemental characterization for agricultural purposes give information useful for determining agronomy management plans. The determination of **nitrogen** and **carbon**, **Total Carbon** (TC), **Total Organic Carbon** (TOC) and **sulfur** enables to characterize soils, leaves, plants, crops and other materials and to setup fertilization plans.

Nitrogen, carbon and TOC determinations give also information on the deficiency or excess of nutritional elements in soils and plants.

Sulfur is an essential component of living matter and the lack of sulfur in vegetables, for example, affects their growth and the quality of proteins through the synthesis of amino acids such as methionine, cysteine and cystine, and in the synthesis of vitamins.

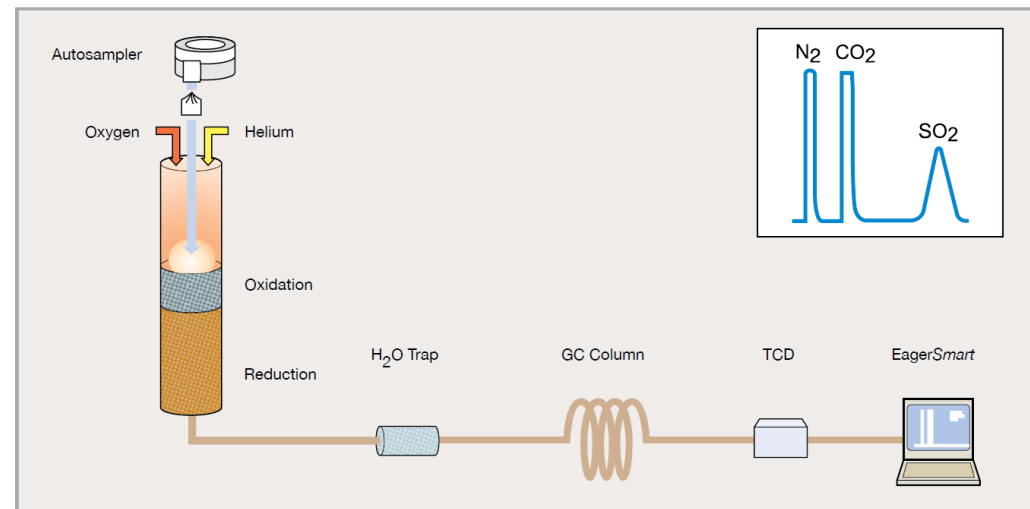
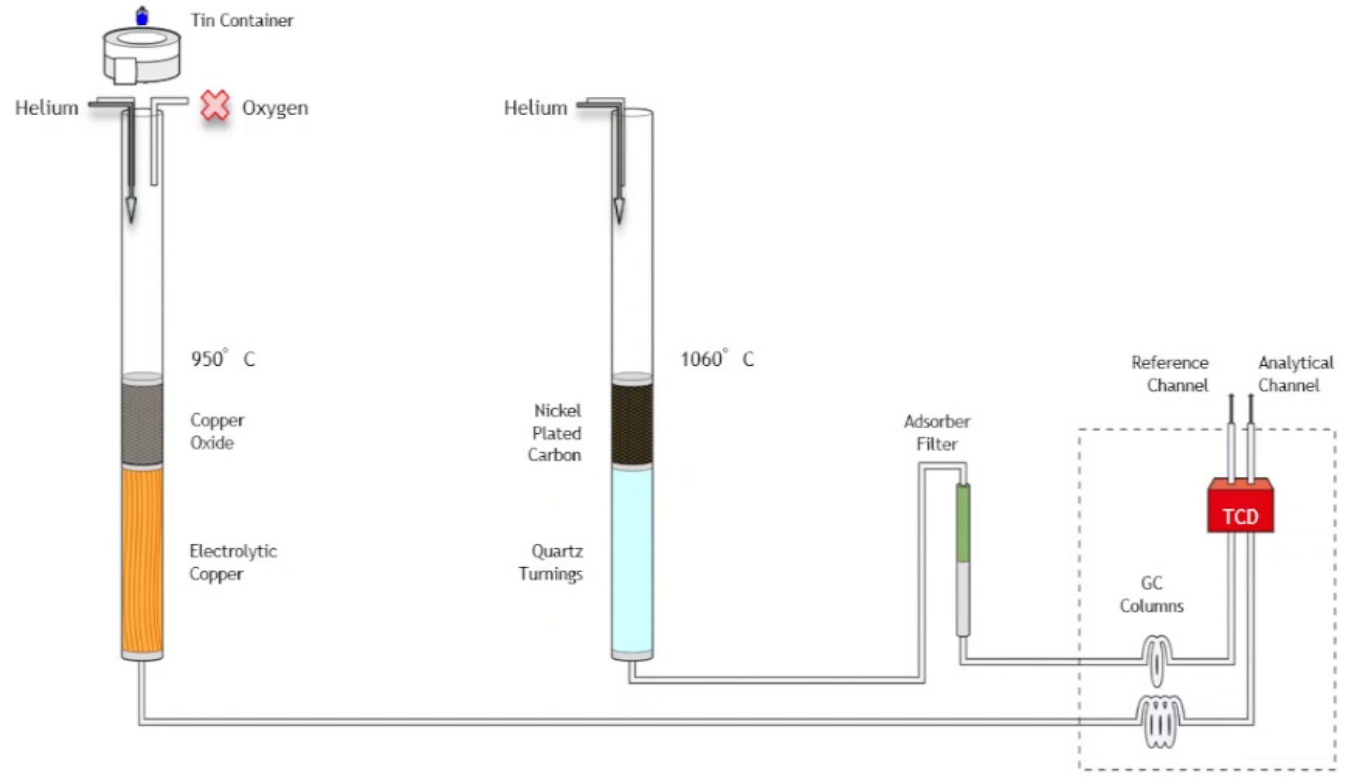
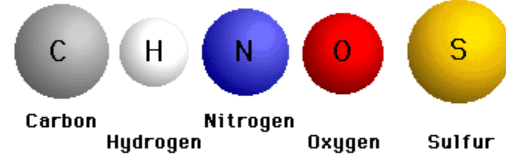


Figure 3. FlashSmart NCS configuration.

FlashSmart analyzer



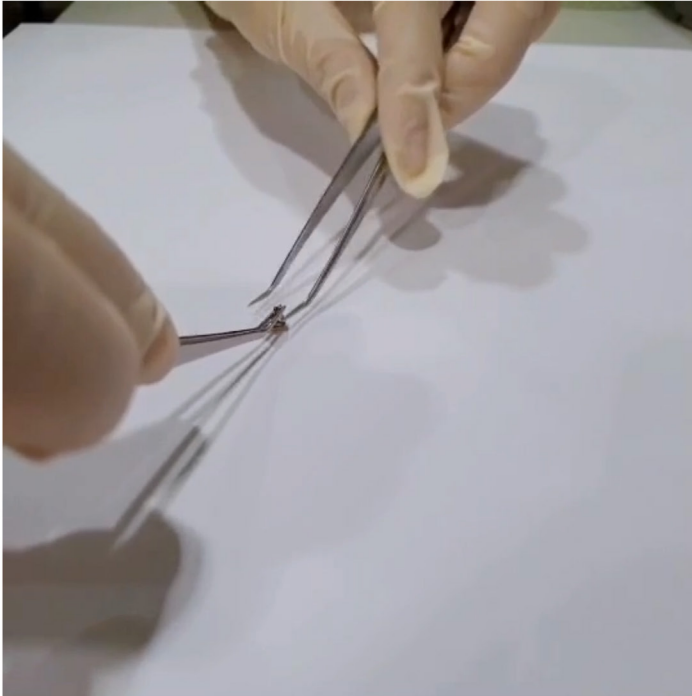
Organic Elemental Analyzer "OEA" is a simultaneous technique to determination of contained in organic and inorganic materials in solid liquid and gas form.



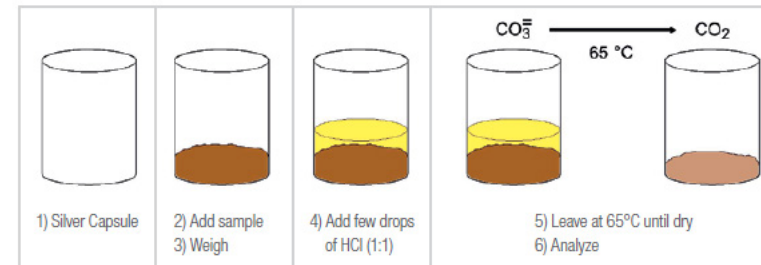
FlashSmart analyzer



Sample Preparation



Tin capsule



TOC was determined after removing carbonate minerals by acidification of the sample with HCl

Application Information

Please refer to the QR code or click on the link for more details in the application notes.



[AN42198 NCS characterization of fertilizers](#)

[AN42264 High accuracy of nitrogen, carbon and sulfur analysis for agronomy applications](#)



[AN42219 NC determination in soils and plants using argon as carrier gas](#)

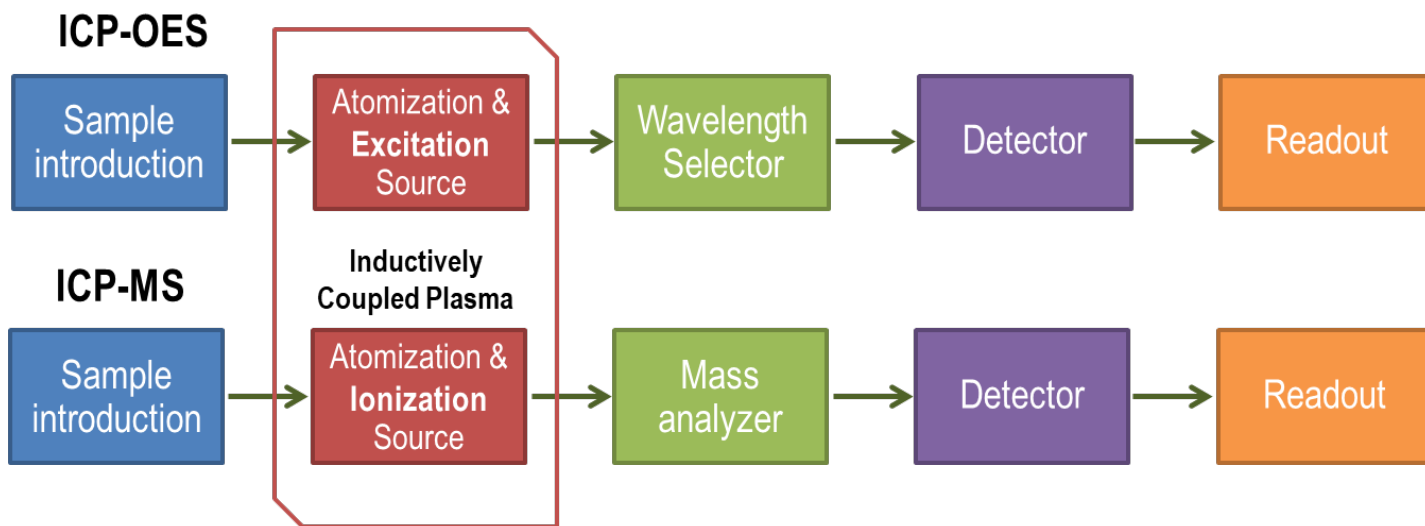
[AN42252 CHN characterization of soils and plants using argon as carrier gas](#)



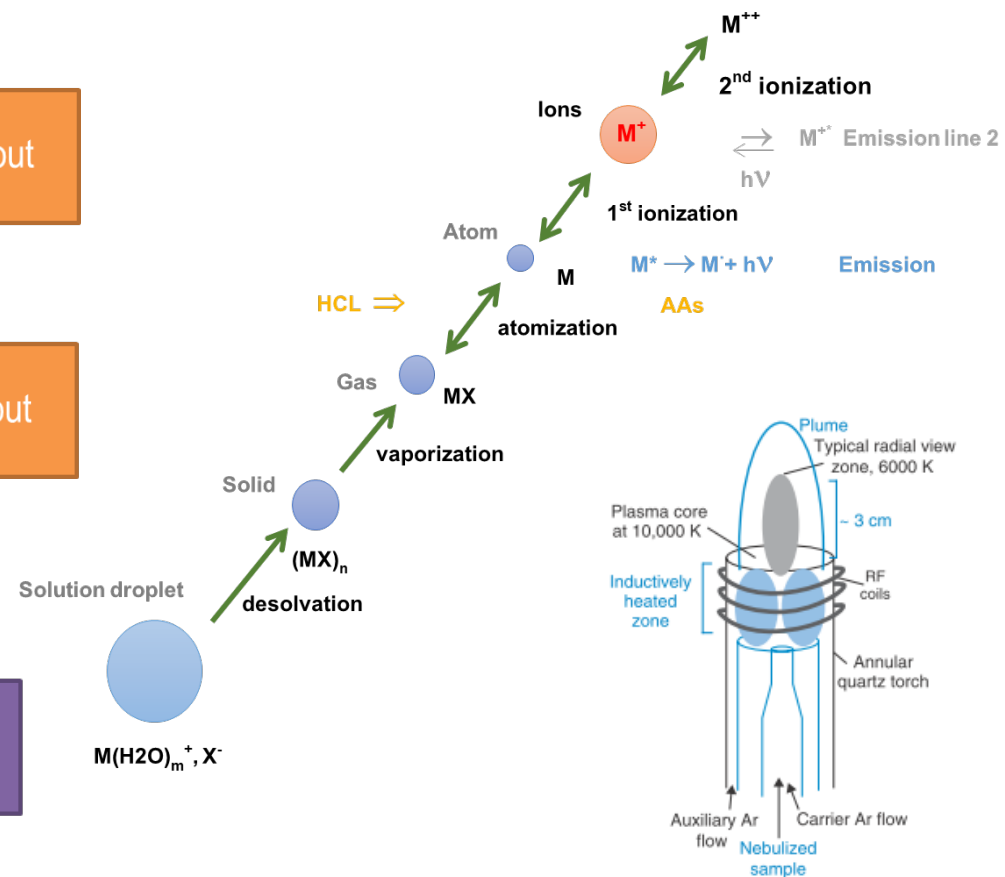
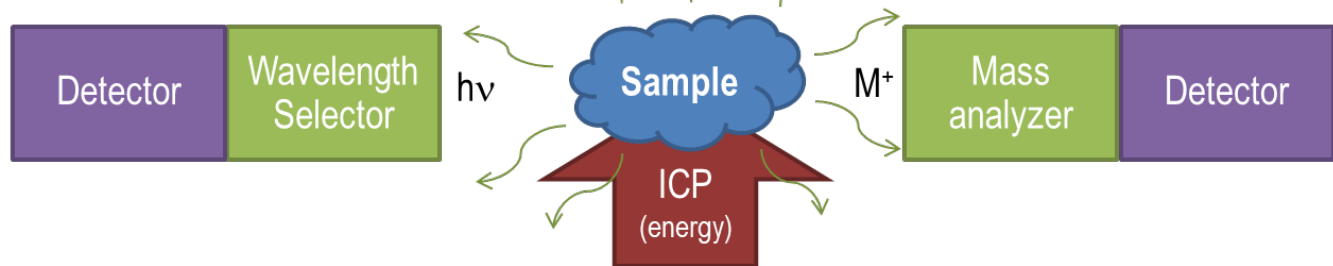
[AN42499 CHNS/O characterization of solid and liquid fertilizers](#)

The ICP-OES and ICP-MS

Inductively coupled plasma mass spectrometry (ICP-MS) and Inductively coupled plasma optically emission spectrometry (ICP-OES) are the common technique used for metals and nutrients analysis.



(Atomization, Excitation & Ionization)



Method 200.7: Determination of Metals and Trace Elements in Water and Wastes by Inductively Coupled Plasma-Atomic Emission Spectrometry

EPA Method 6010D (SW-846) Inductively Coupled Plasma - Atomic Emission Spectrometry

EPA Method 200.8 Determination of Trace Elements in Waters and Wastes by Inductively Coupled Plasma-Mass Spectrometry

EPA Method 6020B (SW-846) Inductively Coupled Plasma - Mass Spectrometry, part of Test Methods for Evaluating Solid Waste, Physical/Chemical Methods

Hazardous Waste Test Methods / SW-846

Elemental analysis workflow



Be aware of contamination sources.



Minimize handling and transfer steps.



Use high-purity reagents.



Use ultrapure water.



Measure weights and volumes with accuracy.



Apply proper skill, technique, and attention to detail.



Sample Preparation

Hot plate acid digestion



- ✓ Simple and inexpensive set-up involving the use of commonplace laboratory apparatus and a hot plate.
- ✓ Procedures are standardized and uncomplicated.
- ✓ Higher sample sizes (e.g., > 1 gram) are possible, which may be required for multiphasic, heterogeneous samples.



Long digestion time.



Incomplete digestion.



Exposure to contamination.



Loss of analytes.



High reagent consumption.



Constant monitoring and addition of acids.



Numerous sample handling and transfer steps.



Overall inefficiency.

Hot block acid digestion



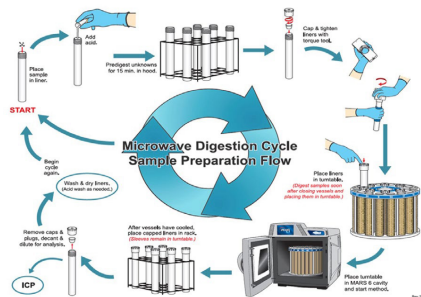
- ✓ Reduced sample handling and transfers
- ✓ Exposure to contamination is reduced.
- ✓ Elimination of issues associated with glassware.
(adsorption of analytes to container walls or leaching of elemental impurities to the sample solution, are eliminated)
- X However, the duration of the digestion process is still extensive, reagent consumption is high, and exposure to contamination from the atmosphere may be possible since it is an open system.

Sample Preparation

Microwave assisted acid digestion



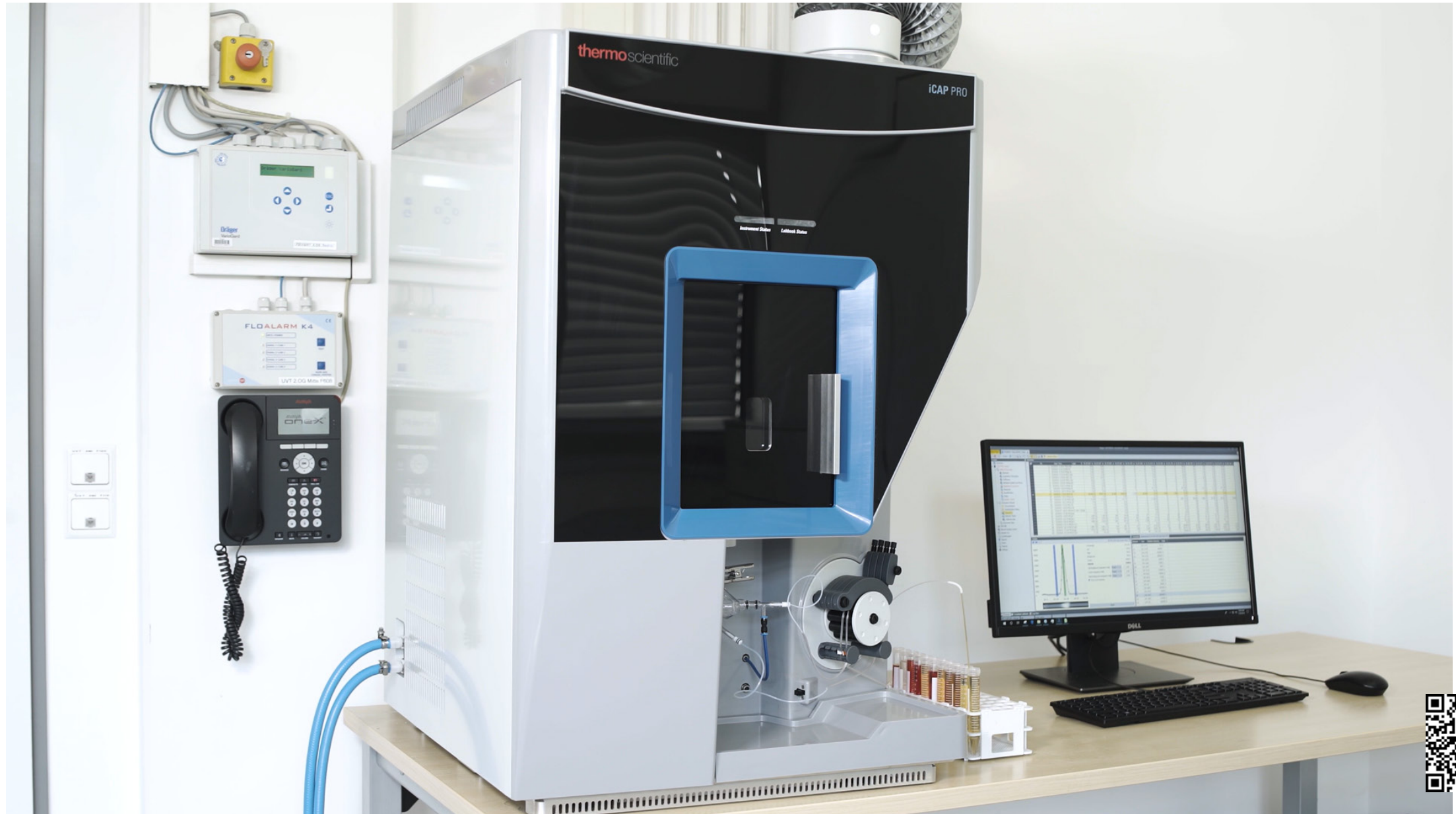
- ✓ Speed of digestion
- ✓ Quality digestion
- ✓ Reduced exposure to contamination
- ✓ Reduced reagent consumption
- ✓ Retention of analyte



Microwave assisted acid digestion is a closed vessel system and has become widely used and known as the best solution for clean chemistry when preparing samples for trace and ultra-trace elemental analyses.

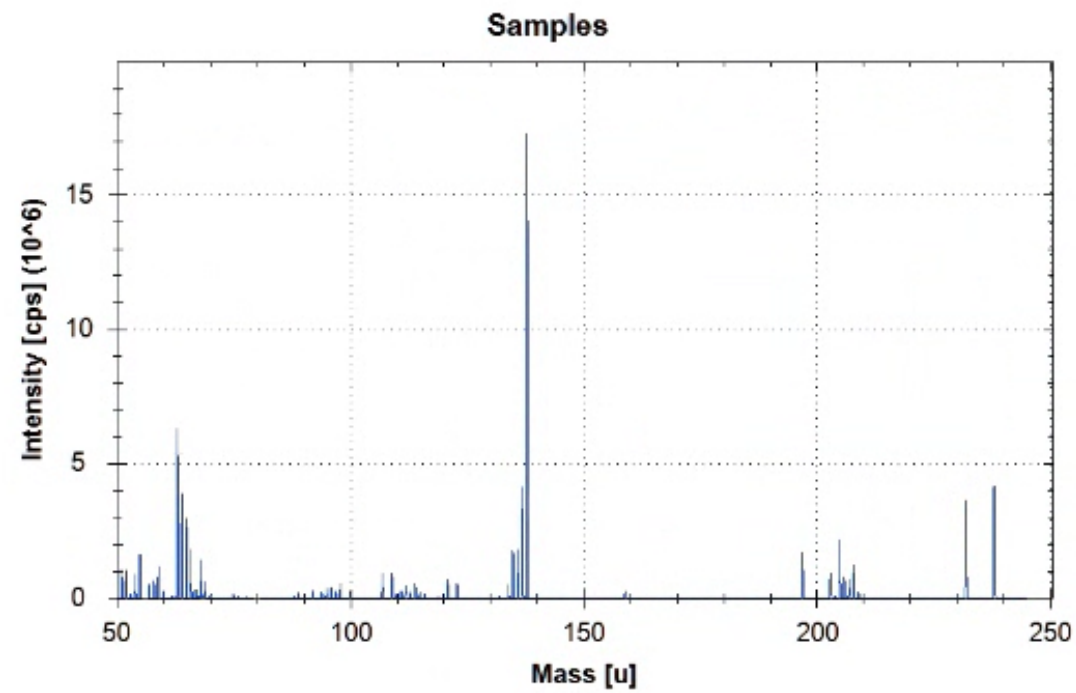
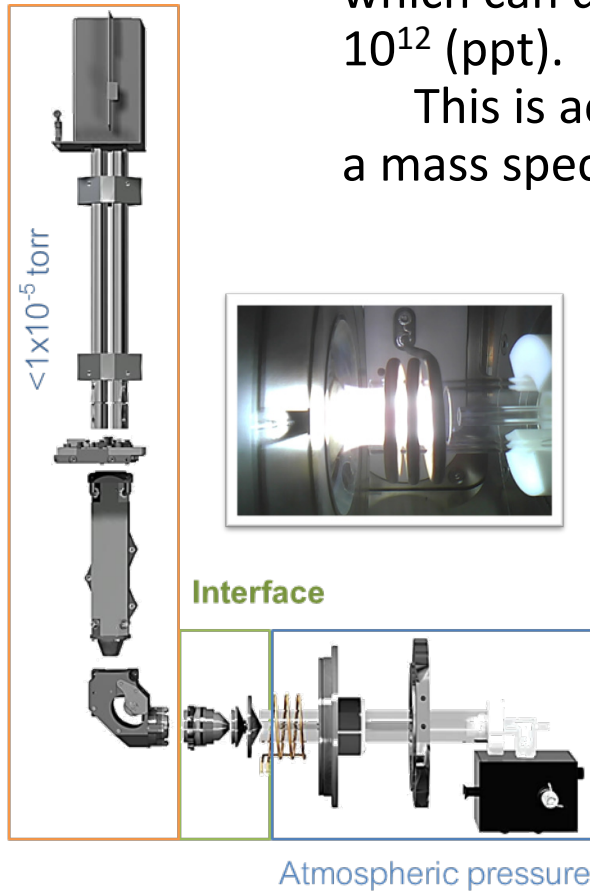
Key considerations	Open vessel acid digestion		Closed vessel acid digestion
	Hot plate	Hot block	Microwave
Initial investment	\$	\$\$	\$\$\$
Ease of set-up	Easiest	Easier	Easy
Consumables	N/A	Required	Optional
Maintenance and cleaning	High	Low	Medium
Sample handling	Highest handling	Lowest handling	Medium handling
Contamination exposure	Highest risk	Medium risk	Lowest risk
Reagent consumption	High	High	Low
Retention of analyte	Lowest	Medium	Highest
Digestion quality	Low	Medium	High
Batch size	Lowest	Highest	Medium
Digestion time	Hours	Hours	Minutes
Sample throughput	Lowest	Medium	High
Recommended for ultra-trace elemental analysis	Not recommended	Recommended	Highly recommended
Overall efficiency	Low	Medium	High

iCAP Pro ICP-OES



Inductively coupled plasma mass spectrometry (ICP-MS) is a type of mass spectrometry which can detect metals and several non-metals at concentrations as low as one part in 10^{12} (ppt).

This is achieved by **ionizing** the sample **with** inductively coupled **plasma** and then using a mass spectrometer to separate and quantify those ions.





Guide for environmental sample analysis by ICP-MS:

Recommendations for getting started and best practices to streamline workflow

by Sabrina Antonio

thermo scientific

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Please refer to the QR code or click on the link for more details in the application notes.



APPLICATION NOTE 44358

[US EPA SW-846 Method 6020B using the iCAP RQ ICP-MS](#)



APPLICATION NOTE 74146

[Fast, accurate, and robust analysis of environmental samples according to US EPA Method 6010D](#)



APPLICATION NOTE 44492

[Fast, simple analysis of soil and sediment according to German soil protection regulations](#)

Thank you for your attention