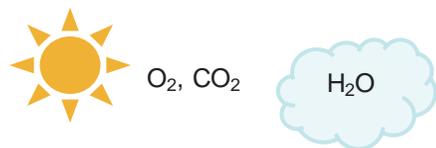




# Elemental Analysis in Soil and Fertilizer

Kantima Sitlaothaworn  
TEA Product Specialist

# Introduction



O<sub>2</sub>, CO<sub>2</sub>

H<sub>2</sub>O



Carbon (C)  
Oxygen (O)  
Hydrogen (H)

## Major nutrients

Nitrogen (N)  
Phosphorus (P)  
Potassium (K)



## Toxic

Arsenic (As)  
Cadmium (Cd)  
Mercury (Hg)  
Lead (Pb)  
Copper (Cu)  
Chromium (Cr)

## Secondary nutrients

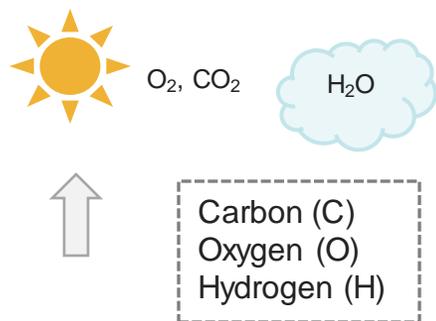
Calcium (Ca)  
Magnesium (Mg)  
Sulfur (S)

## Micronutrients

Boron (B)  
Copper (Cu)      Cobalt (Co)  
Iron (Fe)        Chromium (Cr)  
Manganese (Mn)    Selenium (Se)  
Molybdenum (Mo)    Vanadium (V)  
Zinc (Zn)        Sodium (Na)  
Nickel (Ni)        Silica (Si)



# Introduction



**Major nutrients**

- Nitrogen (N)
- Phosphorus (P)
- Potassium (K)



**Secondary nutrients**

- Calcium (Ca)
- Magnesium (Mg)
- Sulfur (S)

**Micronutrients**

Boron (B)	Cobalt (Co)
Copper (Cu)	Chromium (Cr)
Iron (Fe)	Selenium (Se)
Manganese (Mn)	Vanadium (V)
Molybdenum (Mo)	Sodium (Na)
Zinc (Zn)	Silica (Si)
Nickel (Ni)	



Soil, plant, and fertilizers



Animal feed



Human food

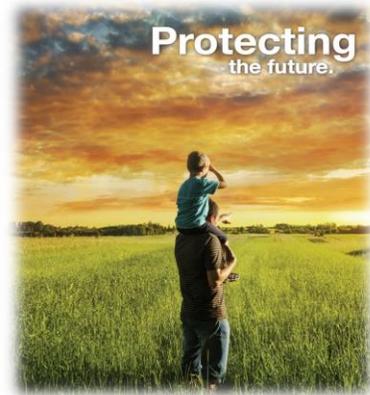
## Soil

- The differentiation of Total Carbon (TC) and Total Organic Carbon (TOC) → evaluates 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



# Introduction



Toxic elements	Major uses and sources of soil contamination
Arsenic (As)	Pesticides, plant desiccants, animal feed additives, coal and petroleum, mine tailings and detergents
Cadmium (Cd)	Electroplating, pigments for plastics and paints, plastic stabilizers and batteries, fertilizers
Chromium (Cr)	Stainless steel, Chrome-plated metal, pigments and refractory brick manufacture
Lead (Pb)	Combustion of oil, gasoline and coal: Iron and steel production
Mercury (Hg)	Pesticides, catalysts for synthetic polymers, metallurgy, thermometers
Nickel (Ni)	Combustion of coal, gasoline, and oil; alloy manufacture, electroplating, batteries

## Soil: Regulation in Thailand

Element	Concentration (mg/kg)	Element	Concentration (mg/kg)
As	6	Hg	22
Cd	67	Ni	436.5
Cr	17.5	Mn	1710
Cu	2920	Se	365
Pb	400		

ประกาศคณะกรรมการสิ่งแวดล้อมแห่งชาติ เรื่องกำหนดมาตรฐานคุณภาพดิน

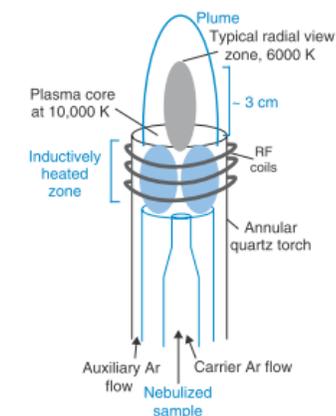
## Fertilizers: Regulation in Thailand

Element	Concentration (mg/kg)
As	50
Cd	5
Cr	300
Cu	500
Pb	500
Hg	2

ประกาศกรมวิชาการเกษตร เรื่อง กำหนดเกณฑ์ปุ๋ยอินทรีย์

# 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	—	—	—	●	●	—	—



ppb to %

100 ppm to 100 %



CHNS

+



Oxygen



1800°C

Flash Combustion

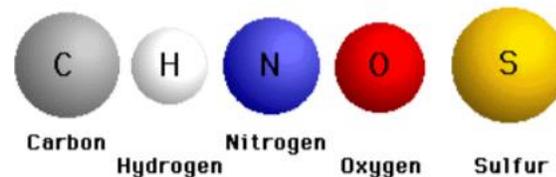
Organic Elemental Analysis (Combustion)

- 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)

# What is Organic Elemental Analyzer?

Elemental characterization for agricultural purposes gives information useful for determining agronomy management plans. The determination of **Nitrogen** and **carbon**, **Total Organic Carbon (TOC)**, and **sulfur** enables to characterization of

- Soils
- Leaves, Plants, Crops, and other materials
- Fertilizer



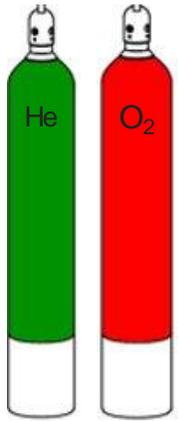
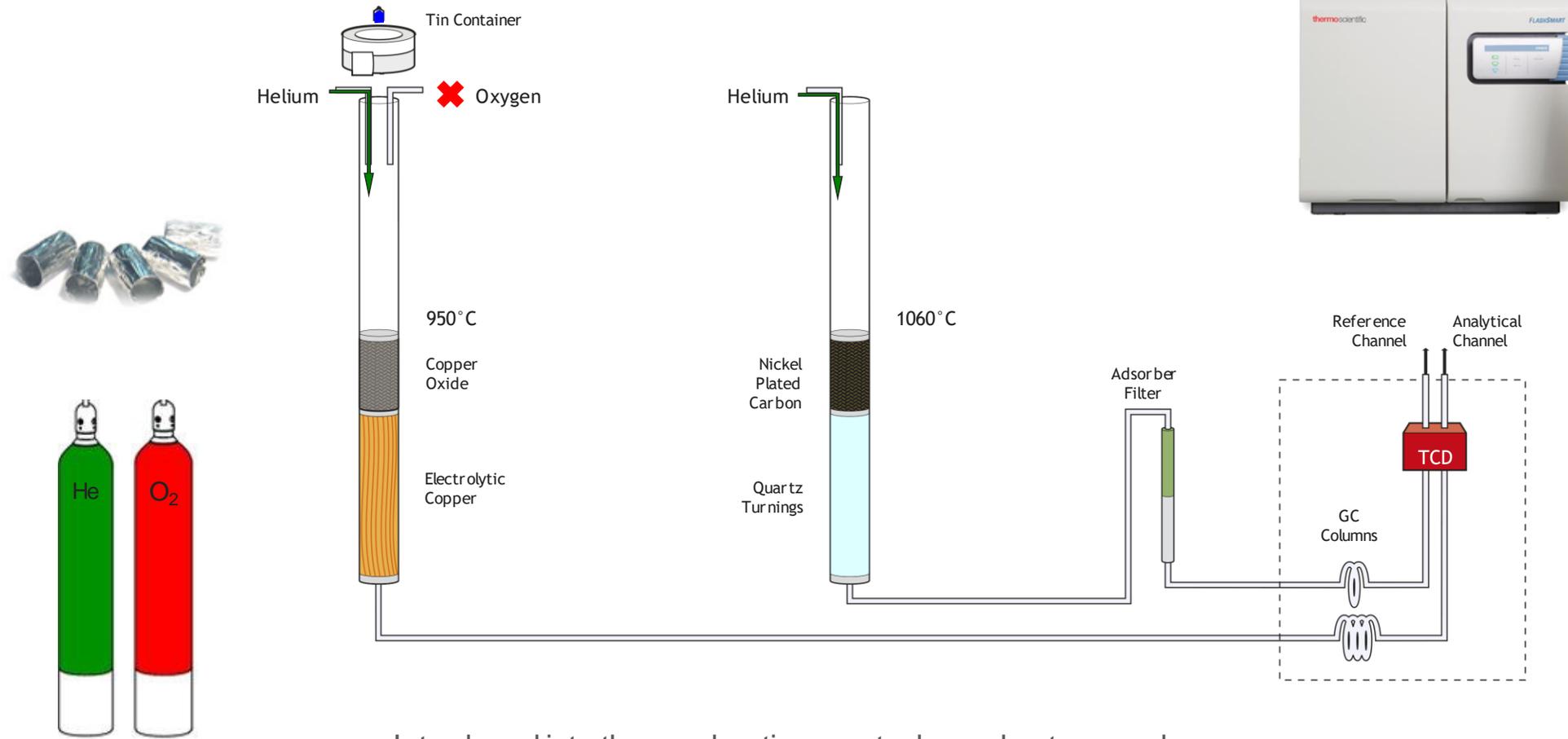
Based on the combustion of the sample. Upon combustion, the sample generates uniform compound gases of the elements C, H, N, and S. These combustion products are measured using gas chromatography and thus the ratio of the elements in the original sample is determined. C, H, N, and S can all be determined simultaneously whereas O by pyrolysis.



Quantification of the sample	Weighting
Quantitative oxidation of the sample	Combustion
Reduction of combustion gases	Reduction
Separation of the oxidation gases	Chromatography
Generation of signal	Detection



# CHNS Analytical configuration

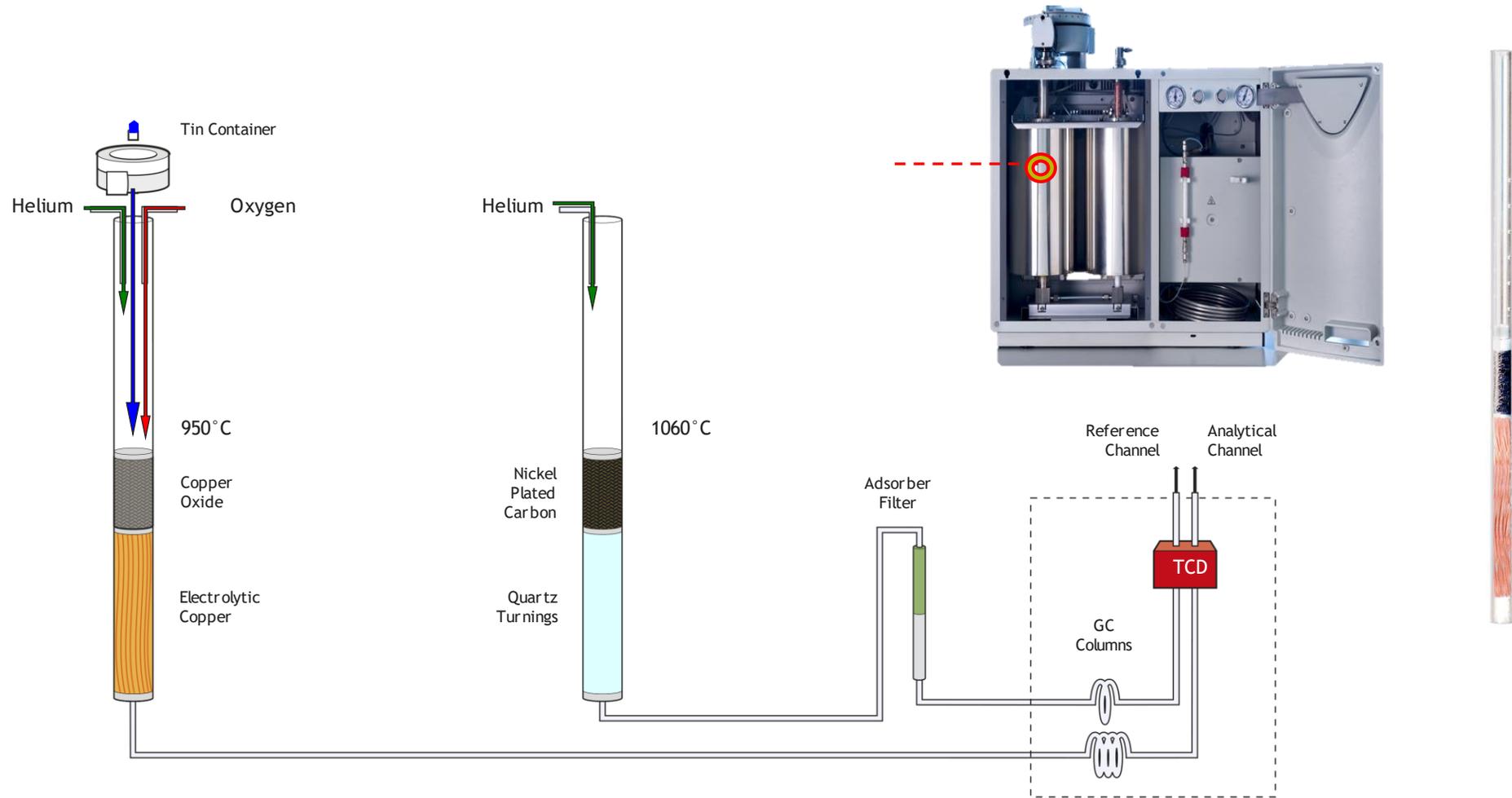


High purity

- Introduced into the combustion reactor by and auto sampler



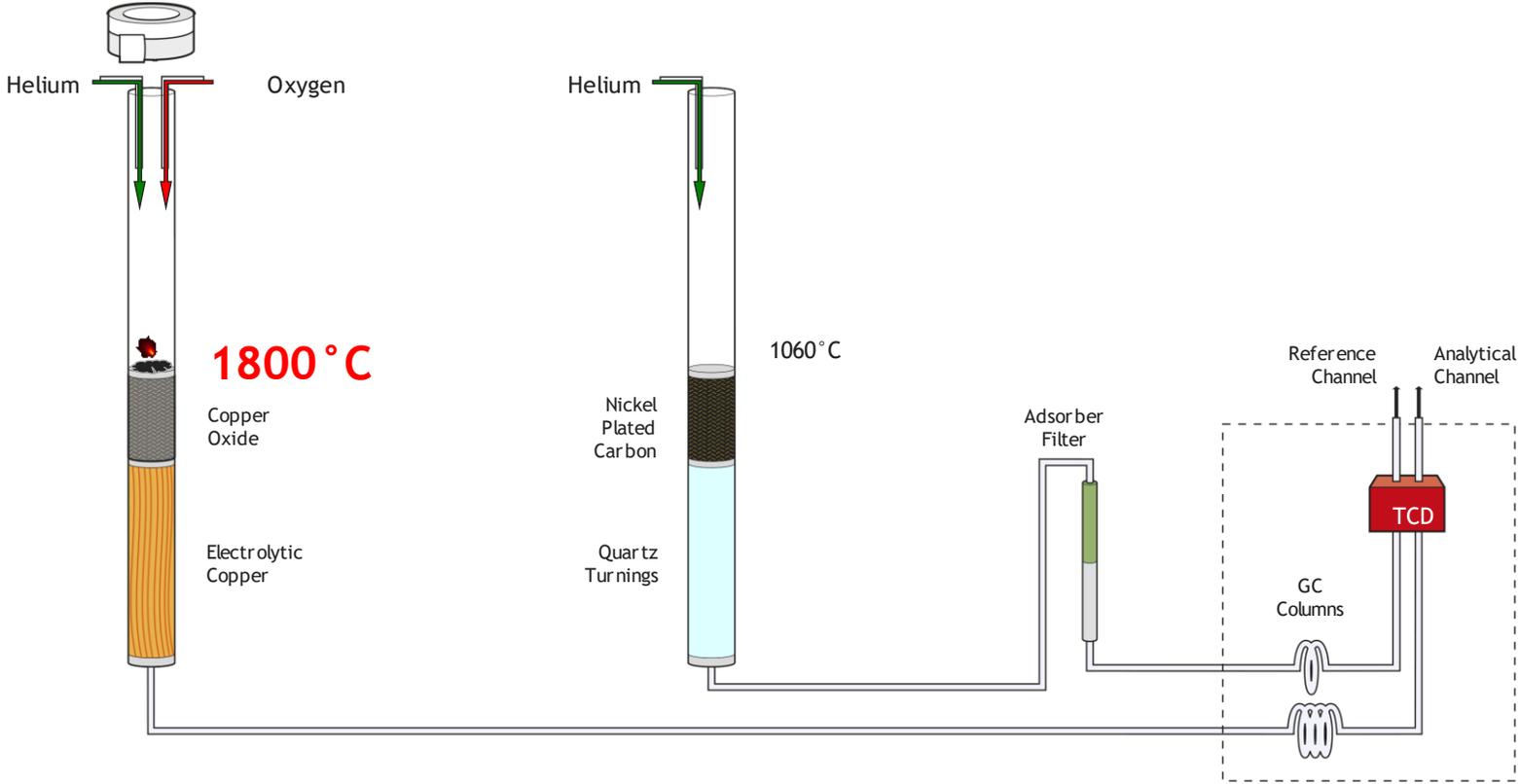
# CHNS Analytical configuration



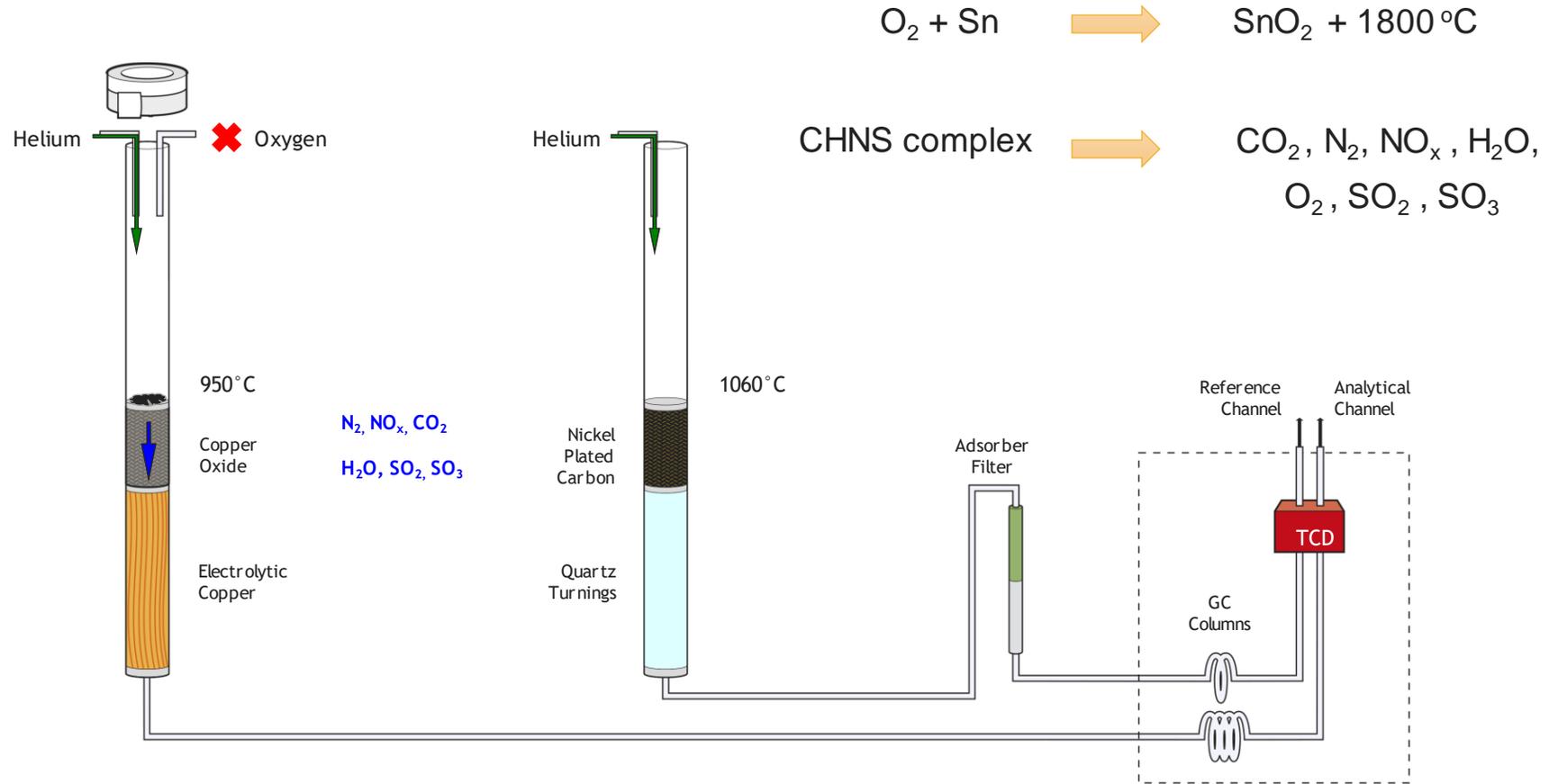
- Inserted in the special furnace heated at 950 °C
- A small volume of pure oxygen is added to the system and helps to burn the sample



# CHNS Analytical configuration



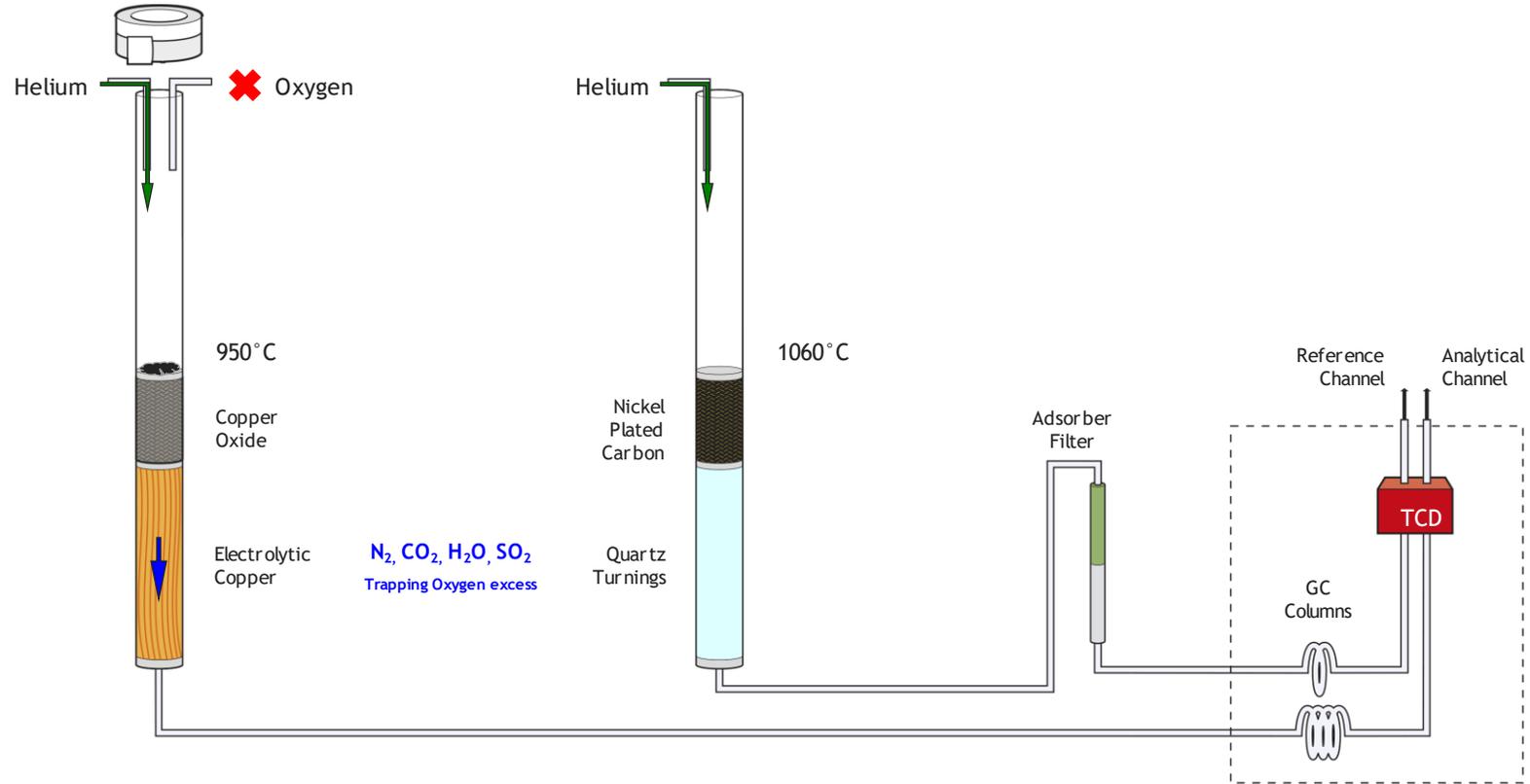
# CHNS Analytical configuration



CuO Convert any CO to CO<sub>2</sub>, N to, NO<sub>x</sub>, S to SO<sub>x</sub>, and H<sub>2</sub> to H<sub>2</sub>O



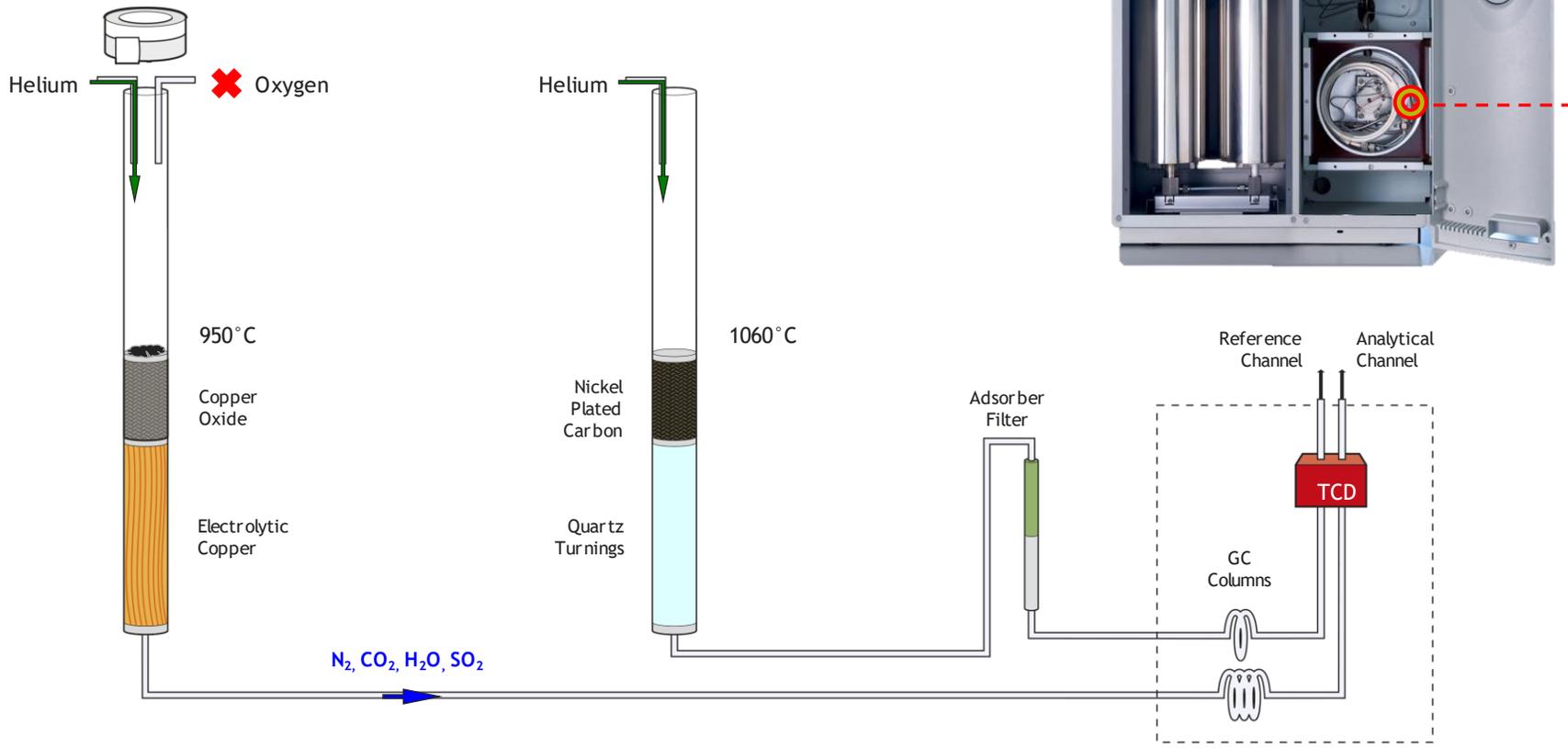
# CHNS Analytical configuration



- Reduction “using Copper” converting the sample into element gases
- Reduces  $NO_x$  to  $N_2$  and  $SO_x$  to  $SO_2$  and removes the excess  $O_2$



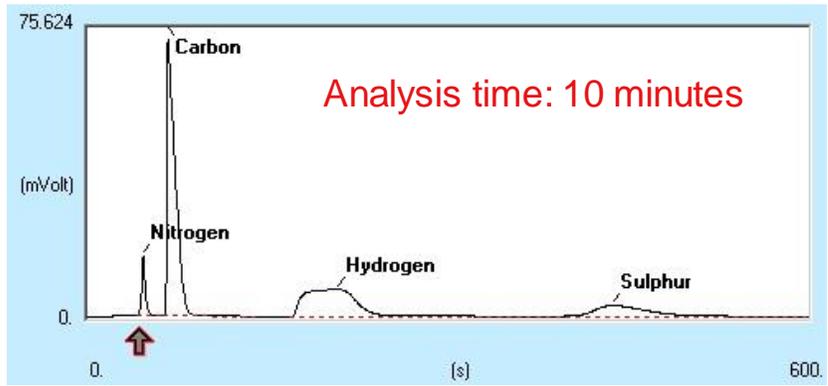
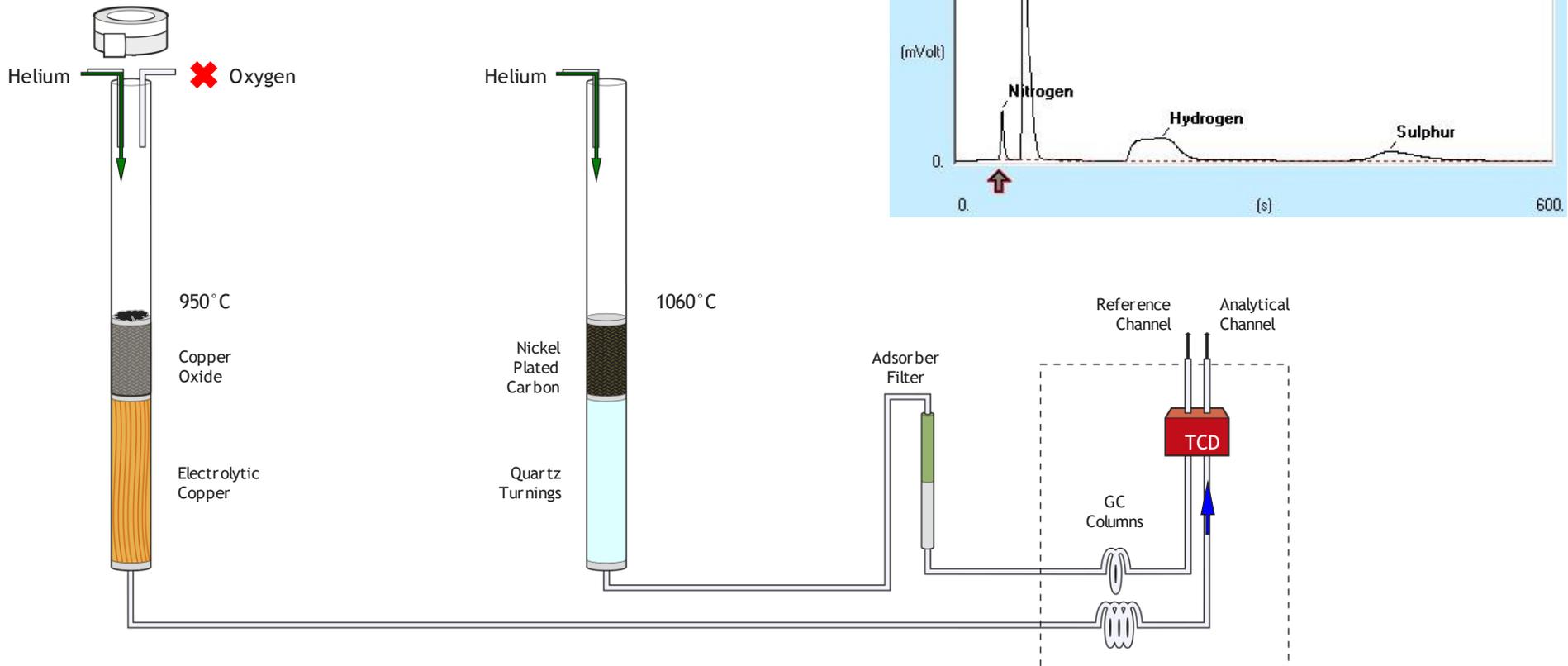
# CHNS Analytical configuration



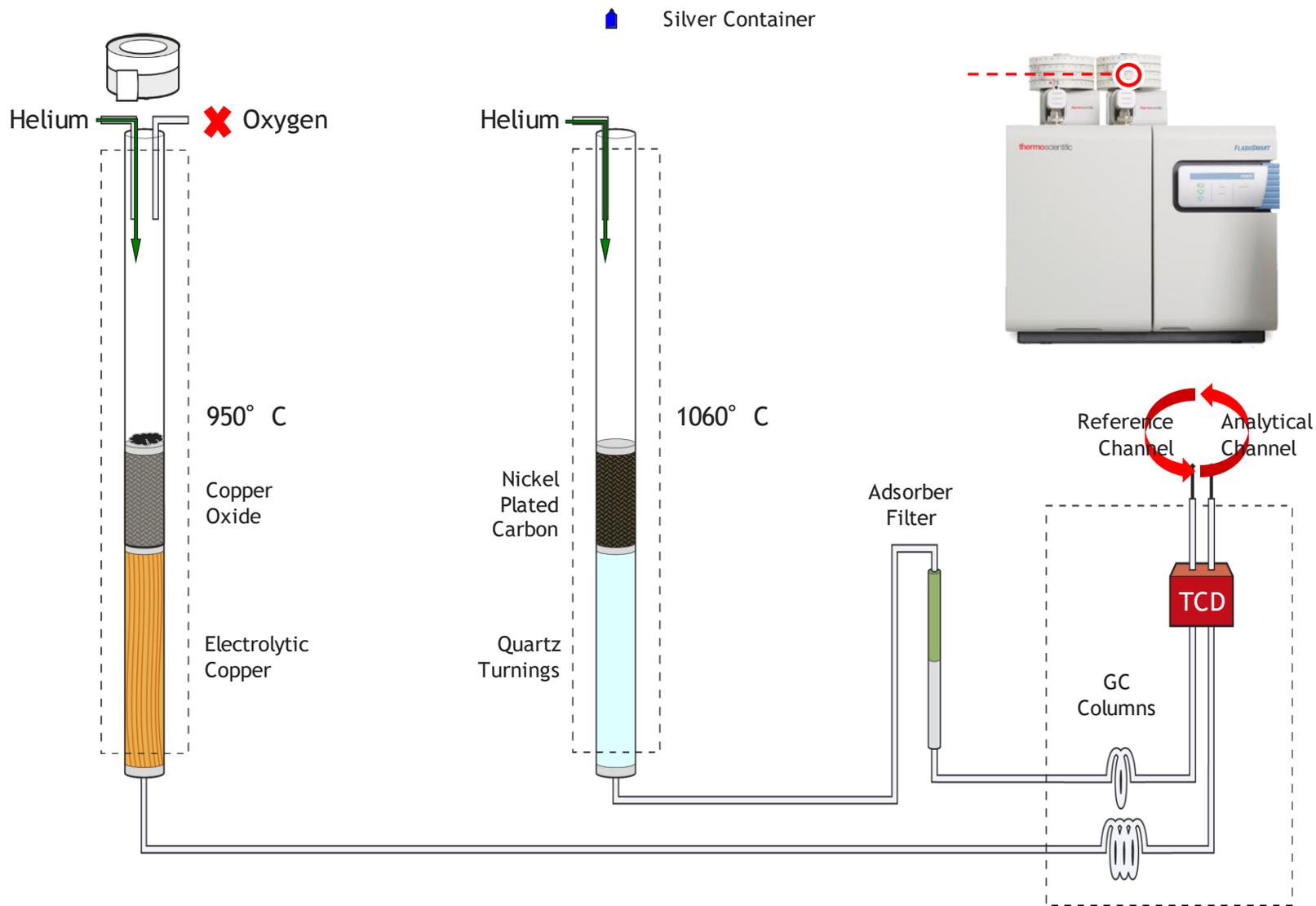
- A separation column and TCD detector allows the user to determine elements



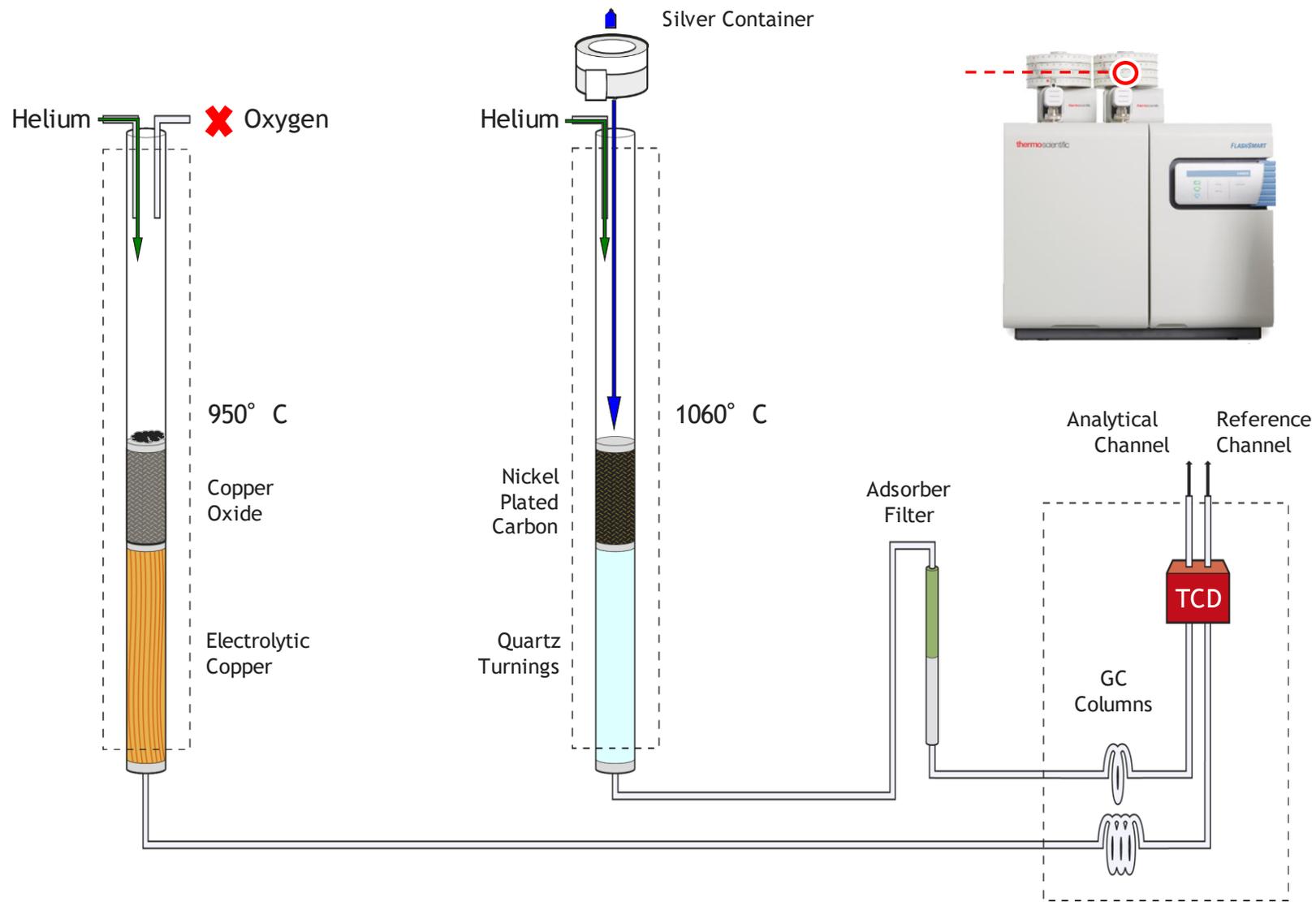
# CHNS Analytical configuration



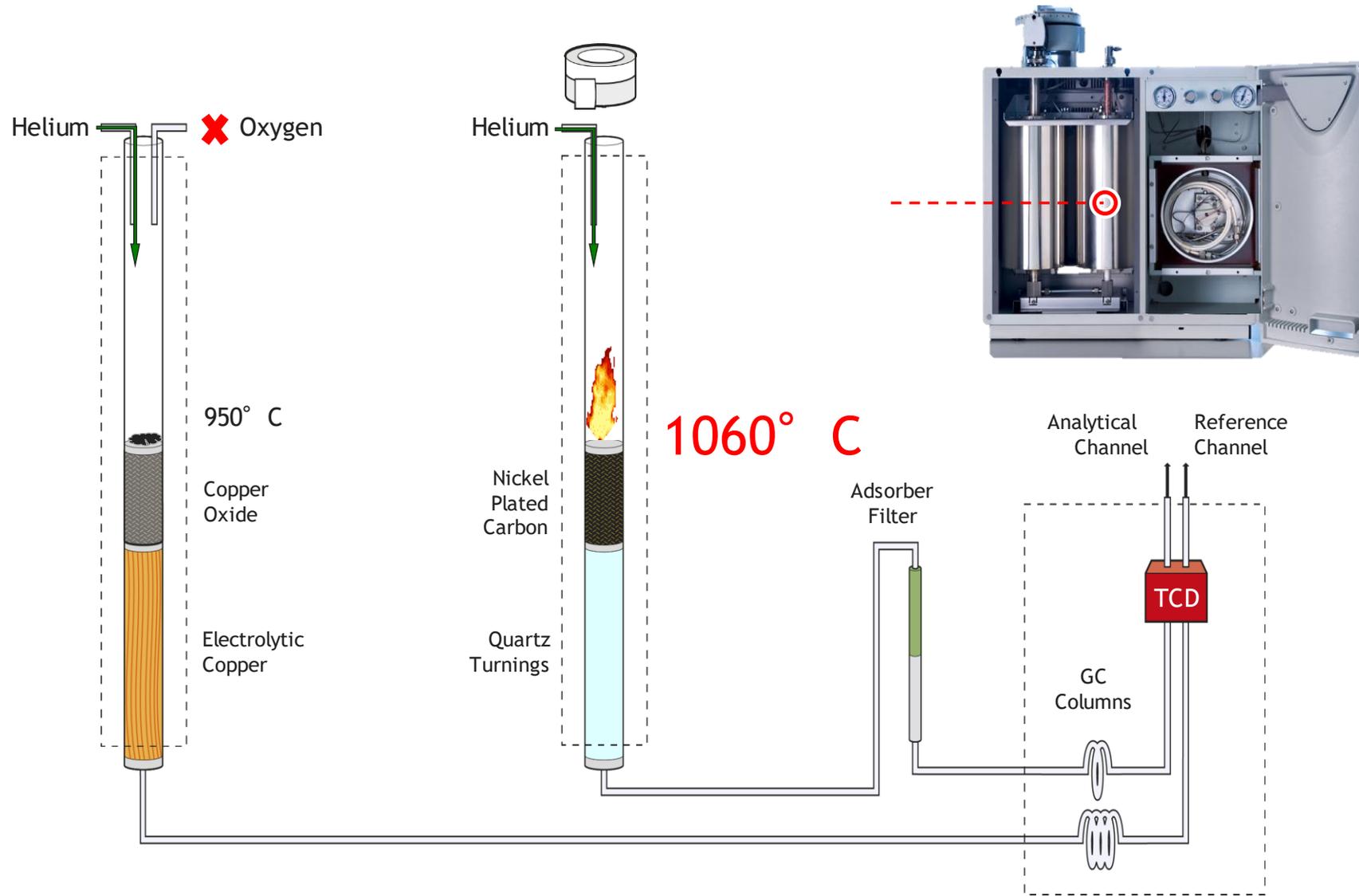
# Oxygen Analytical configuration



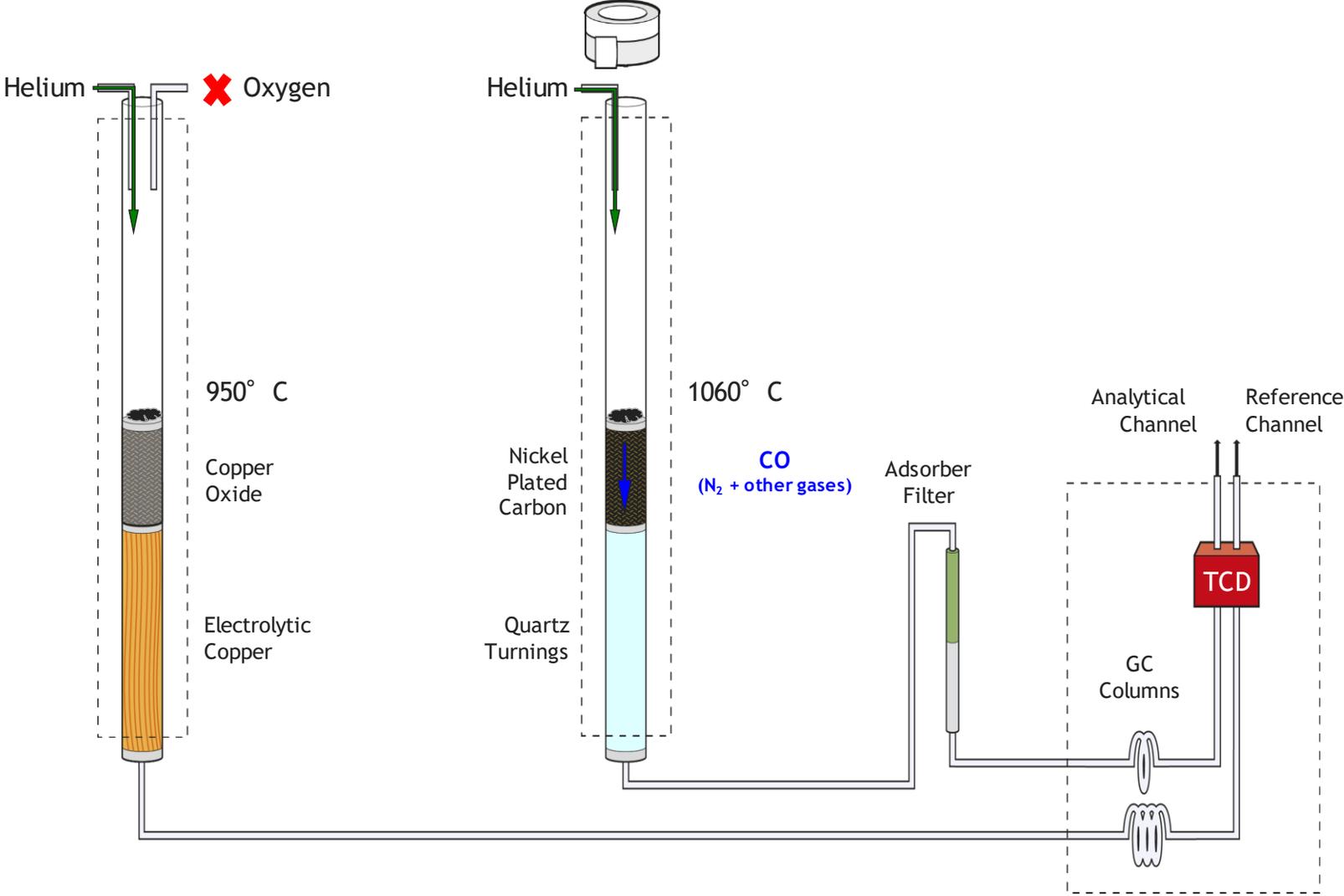
# Oxygen Analytical configuration



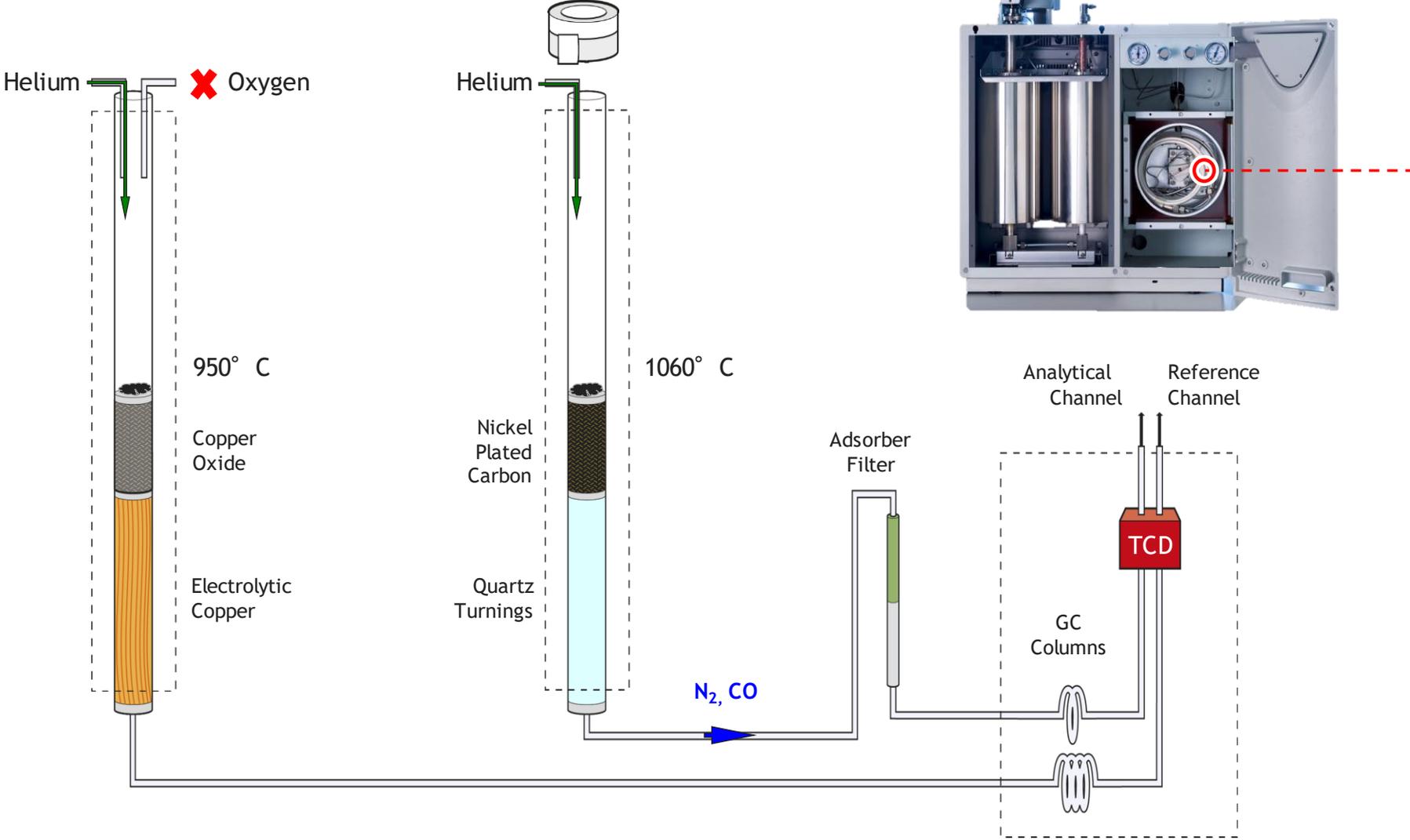
# Oxygen Analytical configuration



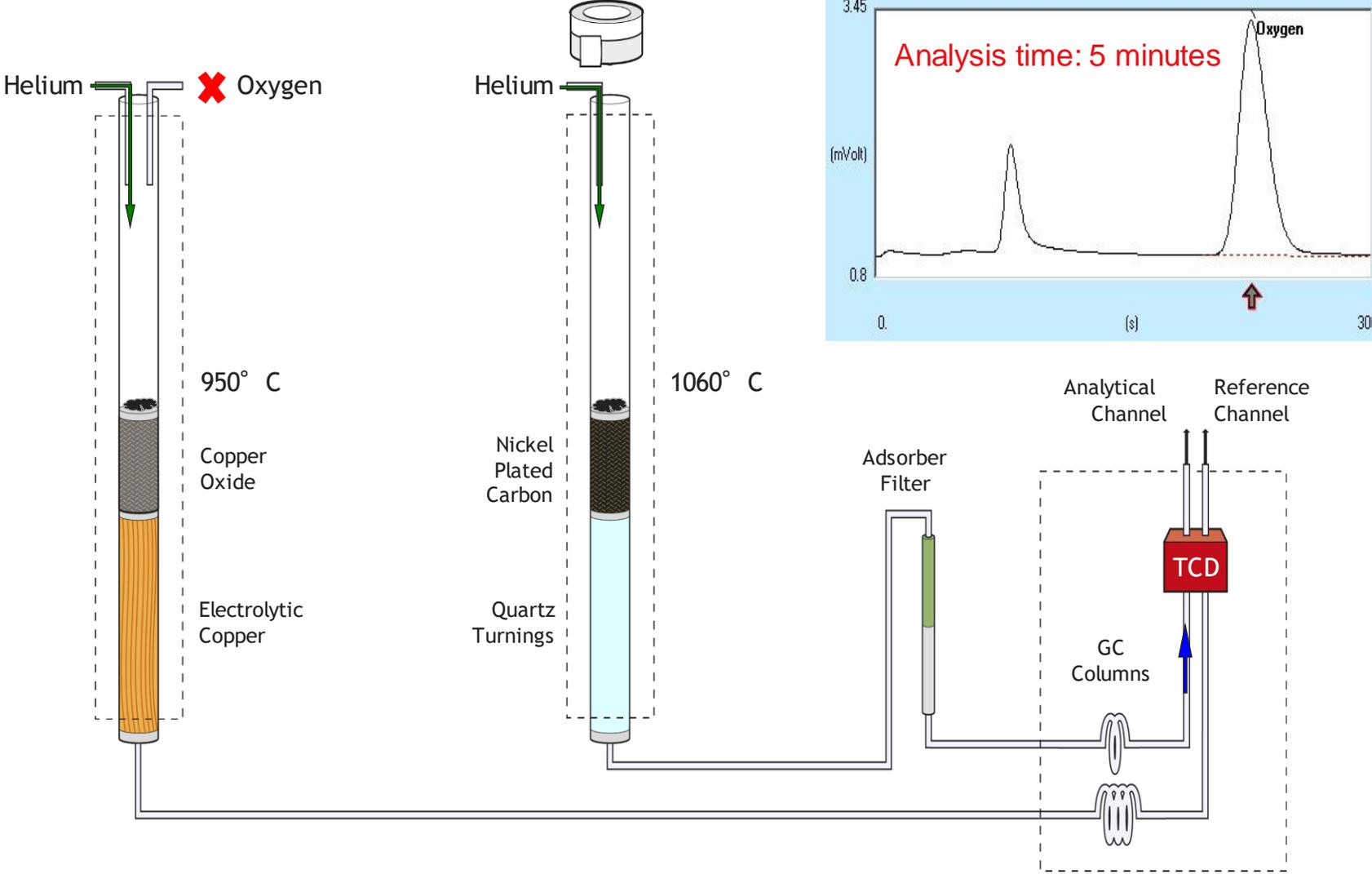
# Oxygen Analytical configuration



# Oxygen Analytical configuration



# Oxygen Analytical configuration



# Sample Preparation

Most materials are often non-homogeneous, and an appropriate homogenization procedure is required to obtain high-quality data. The reproducibility of results is independent of the sample size but strongly dependent on the sample homogeneity.



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



# Agronomy: Soil NCS and TOC



Drill in several points



Representative sample



Sieve 1.5 cm



Take 1 kg

Dry at room temperature or at 35-40 °C in a oven up to constant weight



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Thermo Fisher Scientific, Milan, Italy

**Keywords**  
Argon, CHN Determination, Elemental Analysis, Heaters, Plants, Soil

**Introduction**  
Carbon, hydrogen and nitrogen are regularly characterized in soils and plants to determine agricultural and environmental practices. As the demand for soils and plants testing has grown in the last years, the classical analytical methods showed to be no longer suitable, for their time-consuming sample preparation and for their cost of reagents. For this reason a simple and automated technique is the requirement for modern laboratories dealing with routine analysis.

**Conclusion**  
The Thermo Scientific® FlashDart™ Elemental Analyzer (Figure 1), using typically helium gas carrier and based on the dynamic flash-combustion of the sample, meets laboratory requirements such as accuracy, day to day reproducibility and high sample throughput. Considering the need for cost efficiency and the likely increase in helium gas cost, an alternative gas to be used as carrier gas is needed. Argon can be used as alternative to helium in the FlashDart CA.

**Goal**  
This application note shows nitrogen, carbon and hydrogen determination for soils and plants with the FlashDart CA using argon as carrier gas.

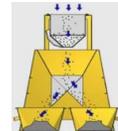
This note presents data on CHN determination in soils and plants reference materials with different concentration to show the performance of the system using argon as carrier gas and to show the reproducibility of the results obtained.



Homogenization by a mortar or ball mill

Take 20 grams

Take 250 grams by a sample splitter



Sieve to 2 mm

Crush with wooden rolling pin on brown paper.



**Analysis**



Sieve to 500 um



Tin caps.

For NC, S an.



Weight the sample



Introduce the sample into the MAS Plus Autosampler

Analysis of NC, S and TOC det.

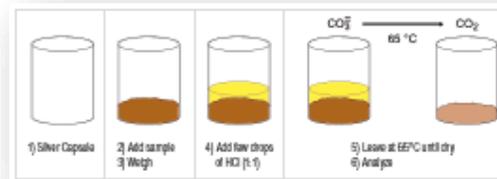


Silver caps. for TOC an.

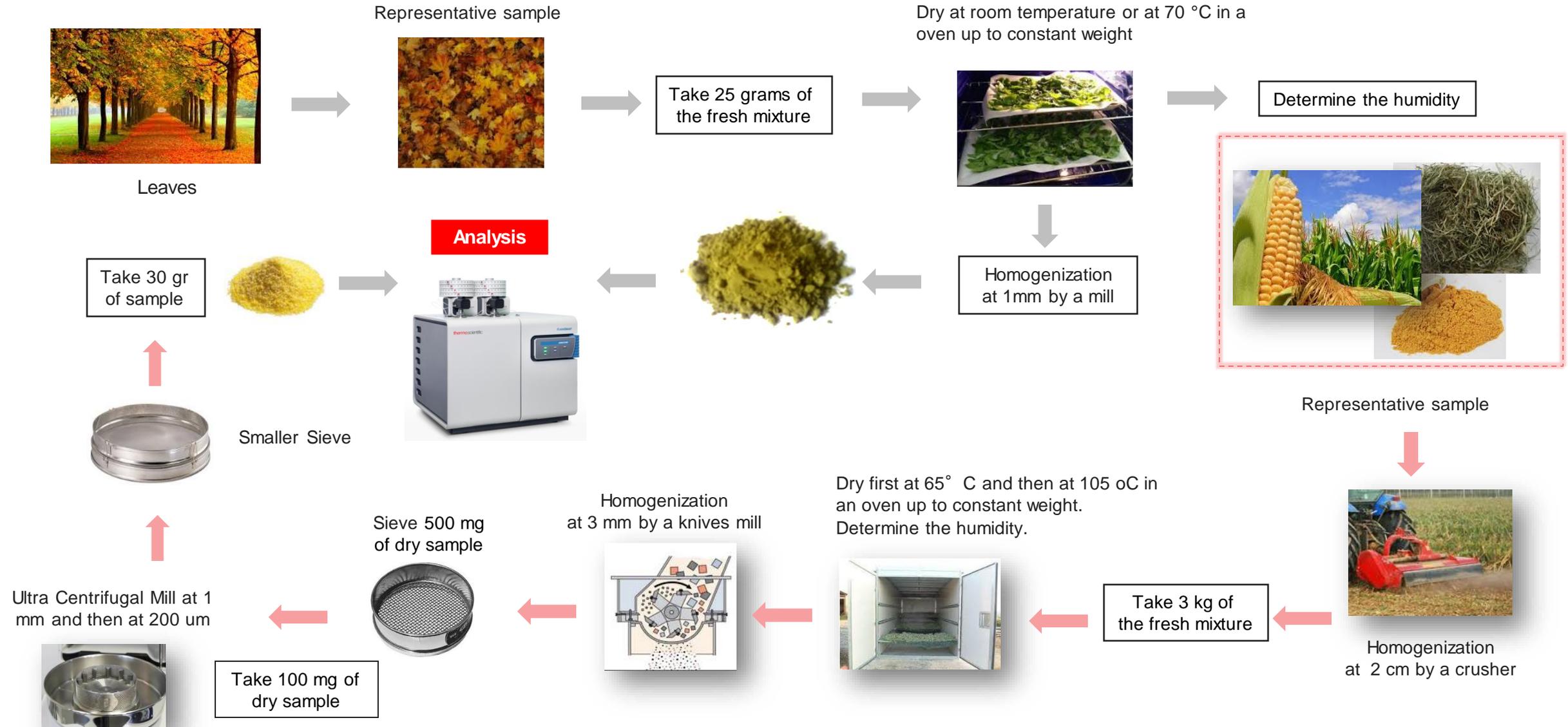
Dry at 105°C up to constant weight. Determine the humidity.



Acidification with HCl 1:1 for TOC

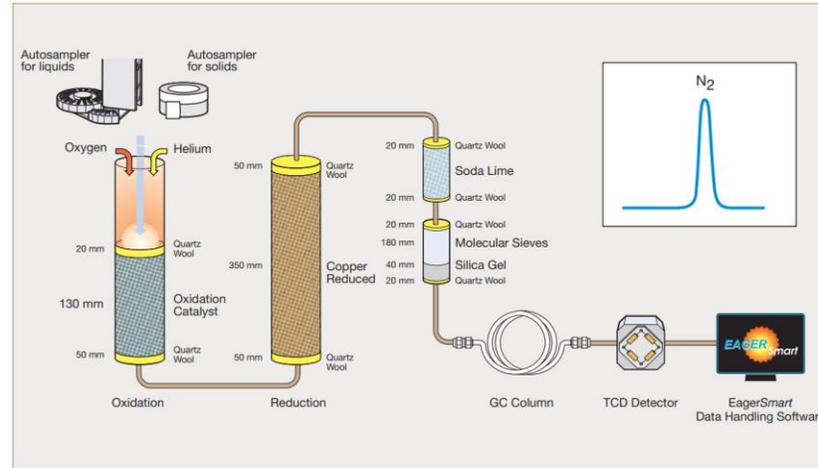


# Agronomy & Animal Feed: NCS

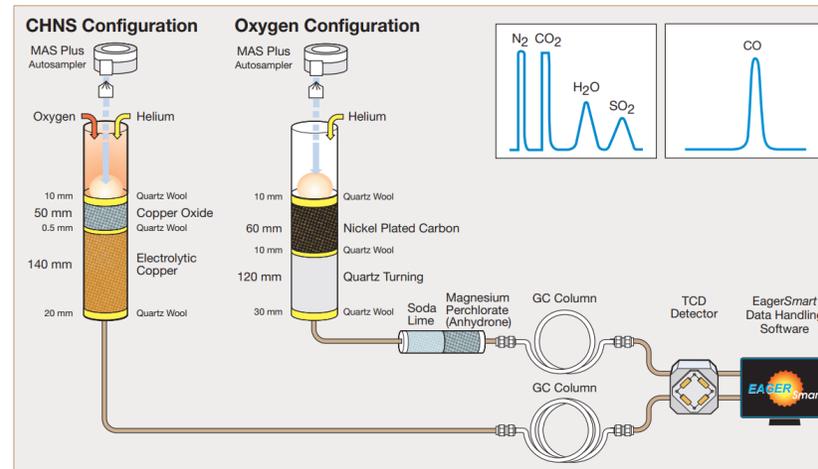


# Fertilizer

**Nitrogen determination of fertilizers according to AOAC 993.13 Official Method** : applicable to the determination of 1 – 67 % total nitrogen content in liquid and solid fertilizer materials



\* ประกาศกระทรวงเกษตรและสหกรณ์ เรื่อง กำหนดกรรมวิธีการตรวจวิเคราะห์ปุ๋ยเคมี



Application	Official Association	Official Method
	Official Italian Method on Soils Analytical Chemistry (Gazzetta Ufficiale)	Method 248, 1999. Nitrogen, Carbon and Organic Carbon in Soils
	AOAC (Association of Official Analytical Chemists)	Official Method 993.13. Nitrogen (Total) in Fertilizer 2.4.02
	ISO 10694, 1995 UNE 77321:2003	Soil Quality - Determination of Organic and Total Carbon After Dry Combustion (elementary analysis)
	ISO 15878, 1998 UNE 77328:2003	Soil Quality - Determination of Total Nitrogen Content by Dry Combustion (elementary analysis)
	UNE 77325:2003	Soil Quality - Determination of Total Sulfur by Dry Combustion
	UNI EN 13654-2	Soil Improvers and Growing Media. Determination of Nitrogen by Combustion Method
	Official Italian Method on Soils Analytical Chemistry (Gazzetta Ufficiale)	Method 146, 1998 Nuovo Norme per la Classifica Dei Fertilizzanti (New regulations for fertilizer's control)
	EPA (Environmental Protection Agency)	Method 440.0, 1997 Determination of Carbon and Nitrogen in Sediments and Particulates of Estuarine/Coastal Waters using Elemental Analysis

# Applications: Access more applications: become the reference laboratory in your field



## Petrochemistry: gasoline, diesel, lubricants, oils



## Environmental and Marine Science: Wastewater, Particulate matter, Biomass, Plankton



**Elemental Analysis: C, H, N, S determination in carbon**

**Elemental Analysis: CHNS determination of organic liquids and fuels by the FlashSmart Elemental Analyzer**

**Characterization of lubricants and oils by the Thermo Scientific FlashSmart Elemental Analyzer**

**Diesel and bio-diesel characterization by the Thermo Scientific FlashSmart Elemental Analyzer**

**Elemental Analysis: CHNSO determination in coals**

**Thermo Scientific FlashSmart Fully Automated Double Channel Analysis for Petrochemical Applications**

## Food: cheese, chocolate, cocoa, flours, pasta, starch, juice, wine, milk, yoghurt



**Elemental analysis: NC determination by single and double reactor of high weight sample with the FlashSmart EA**

**High Accuracy of Nitrogen, Carbon and Sulfur Analysis for Agronomy Applications using the Thermo Scientific FlashSmart Elemental Analyzer**

**Elemental Analysis: CHNSO characterization of biomass**

**Elemental Analysis: Nitrogen determination of inorganic fertilizers using argon as carrier gas**

**Elemental Analysis: Nitrogen determination in soils and plants using argon as carrier gas**

**Elemental Analysis: NCS characterization of fertilizers**

## Organic Chemistry & Pharm



**Nitrogen and carbon determination in focus by the Thermo Scientific FlashSmart Elemental Analyzer using argon as carrier gas**

**Nitrogen/Protein determination in dairy products using the Thermo Scientific FlashSmart Elemental Analyzer**

**Nitrogen/Protein determination of infant food by the Thermo Scientific FlashSmart Elemental Analyzer using helium or argon as carrier gases**

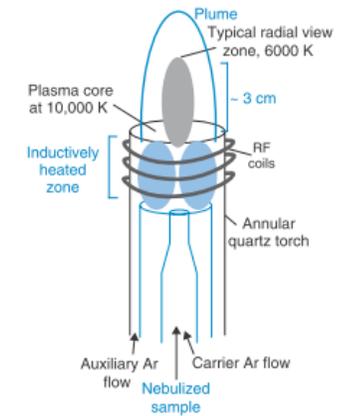
**High protein content determination in food and animal feed by combustion using the Thermo Scientific FlashSmart Elemental Analyzer**

## Materials: paper, cotton, textile fibers, polyethylene film



# 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	—	—	—	●	●	—	—



ppb to %

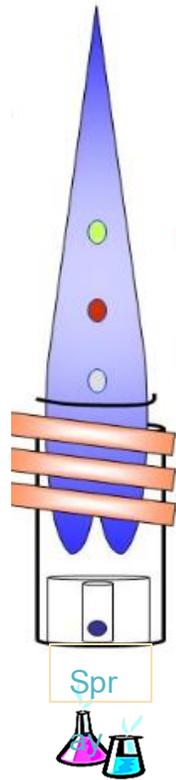
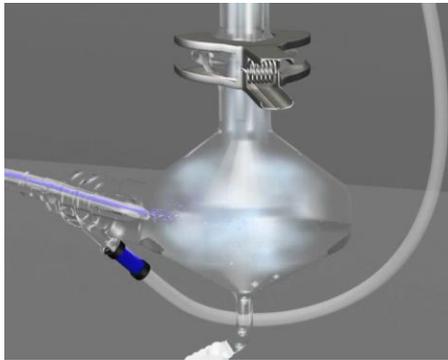
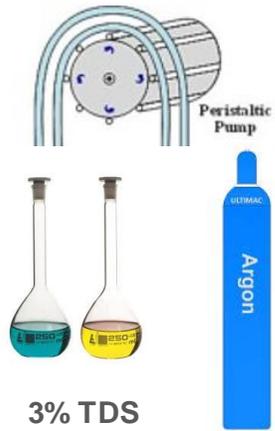
- The ICP-OES and ICP-MS are used to provide information of major plant nutrients (P, K), secondary plant nutrients (Ca, S, Mg), micronutrients such as B, Mn, Fe, Cu, Zn, Mo and Se, also the toxic elements (As, Cd, Pb and Hg)

# What is ICP-OES?

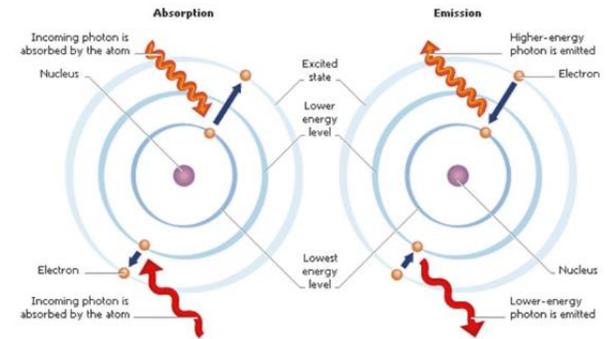
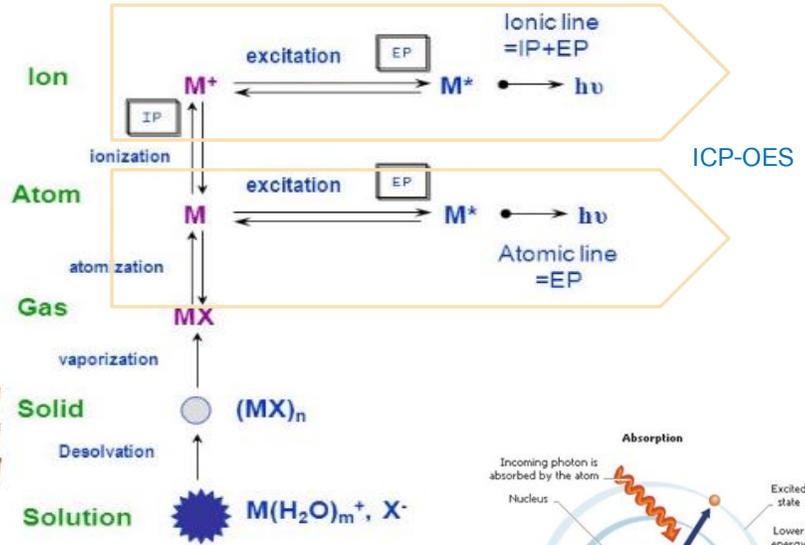


Delivers all the sample to the plasma which provide a *non-effect to plasma stability*

Convert all forms of sample into *free Atom/Ions* and Excite free Atom/Ions turn to *Excited state*



SOLUTION SAMPLE



# What is ICP-OES?

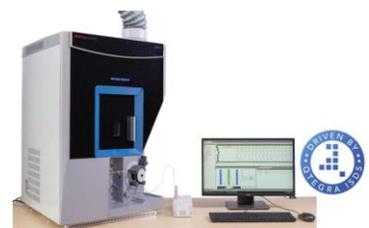
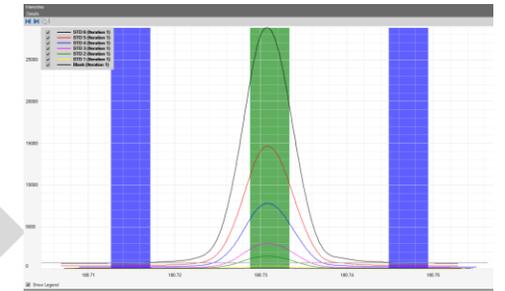
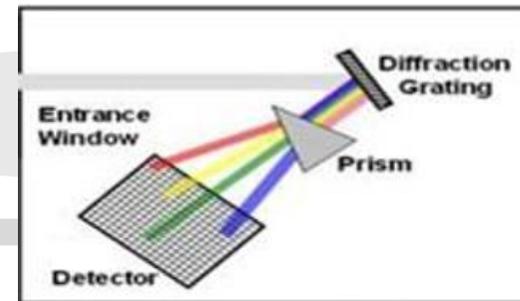
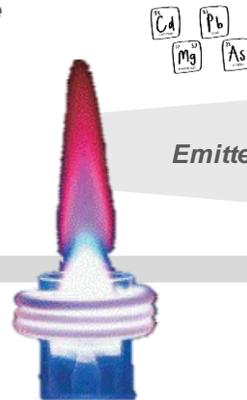
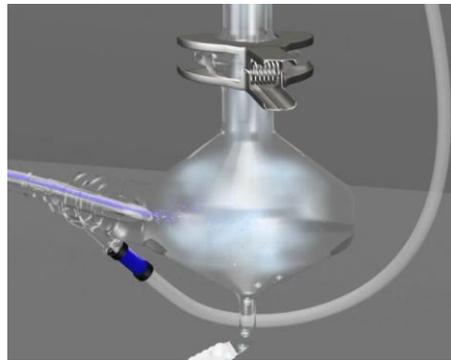
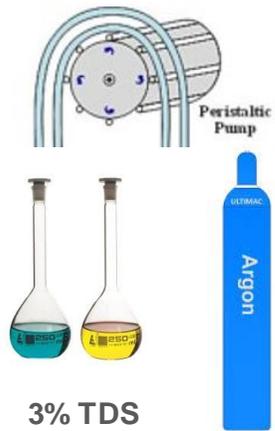


Delivers all the sample to the plasma which provide a *non-effect to plasma stability*

Convert all forms of sample into *free Atom/Ions* and Excite free Atom/Ions turn to *Excited state*

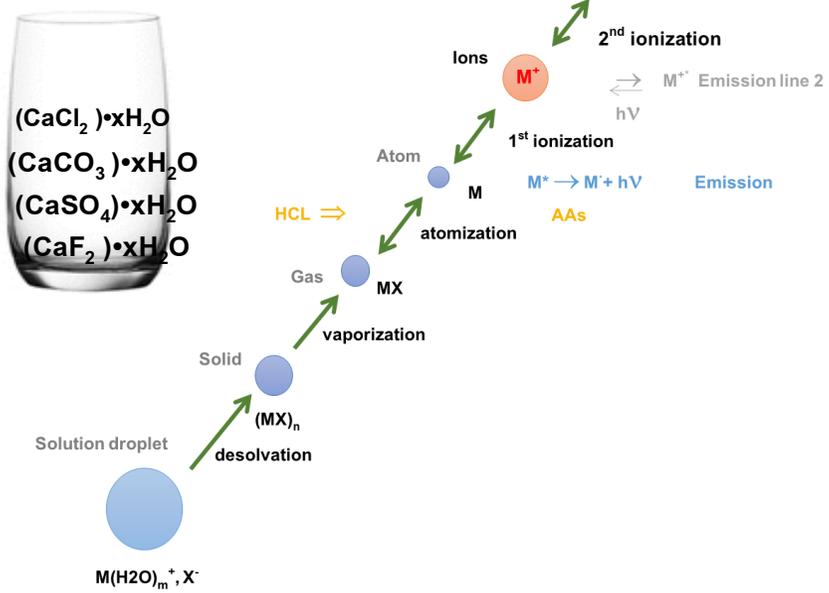
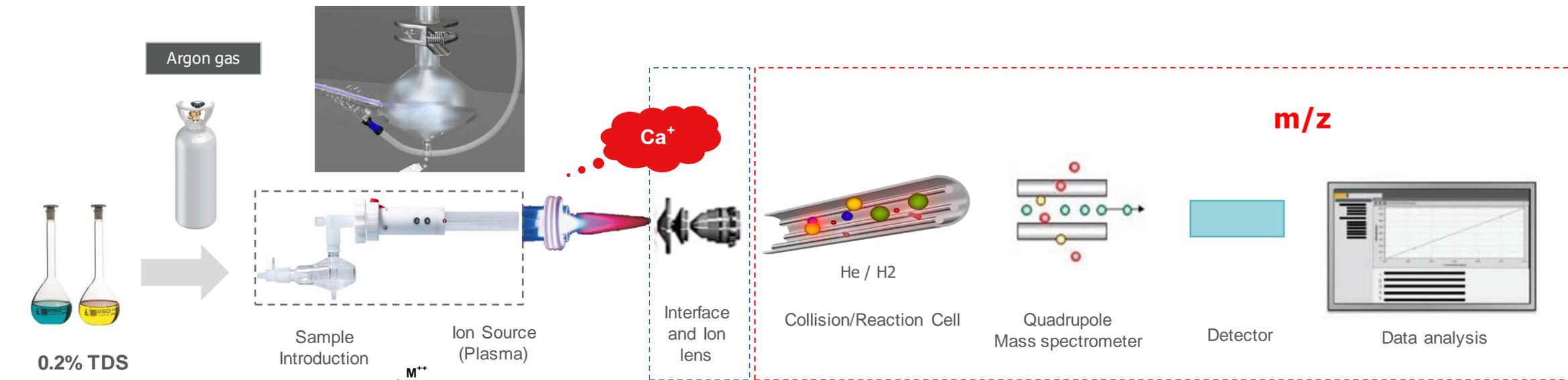
Distinguish emission light from each elemental Atom/Ions and Detect the intensity of each wavelength

Provide all important information when required but in a simple way.



# What is ICP-MS?

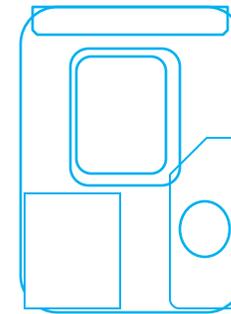
ICP-MS is an elemental analysis technique and uses an argon (Ar) plasma – the ICP – to convert the sample into ions that are then measured using a mass spectrometer



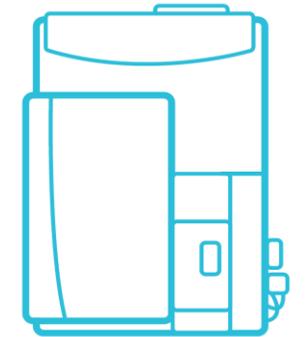
1. Sample introduction system to form a fine aerosol mist from the liquid sample
2. Plasma (ICP) to convert the elements in the sample aerosol to ions
3. Interface to extract the ions into the vacuum system
4. Ion lens to focus the ions and separate them from background signals
5. Collision/reaction cell (CRC) to resolve the analyte ions from interfering ions
6. Mass spectrometer (MS) to filter the analyte ions by mass
7. The electron multiplier detector
8. Data processing

# Select the right techniques for metal analysis in your lab

Field	Typical Applications	Commonly used Techniques		
		AA	ICP-OES	ICP-MS
Environmental	Water	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Soil	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Air	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Food	Food safety	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Nutritional labeling	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Pharmaceutical	Drug / Clinical	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Petrochemical	Petroleum refining	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Lubricants and oil	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input type="checkbox"/>
Chemical / Industrial	QC/Product testing	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Agriculture	Soil / Fertilizer / Plant	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Geochemical/Mining	Exploration	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Research	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Bio-monitoring	Biological Fluids	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Semiconductor	Wafers	<input type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
	High-Purity Chemicals	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>
Nuclear Energy	Low-level waste	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Process water	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Renewable Energy	Biofuels	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
	Solar panels	<input type="checkbox"/>	<input checked="" type="checkbox"/>	<input checked="" type="checkbox"/>
Nano materials	Research	<input checked="" type="checkbox"/>	<input type="checkbox"/>	<input checked="" type="checkbox"/>



**ICP-OES**



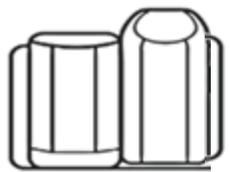
**ICP-MS**



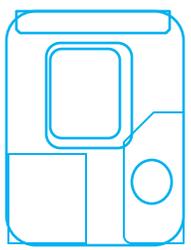
# Select the right techniques for metal analysis in your lab

Multi-element technique

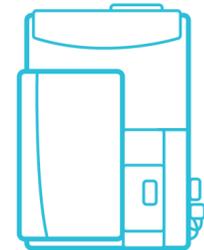
Single element technique



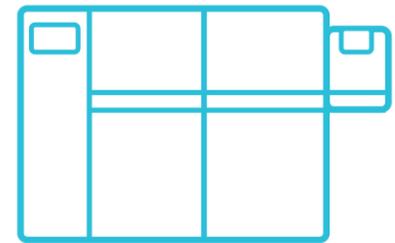
AAs



ICP-OES



ICP-MS



High-Resolution ICP-MS



PERFORMANCE



SENSITIVITY AND  
DETECTION  
LIMIT



ANALYSIS  
SPEED



EASE OF USE  
AND  
MAINTENANCE



COST PER  
SAMPLE

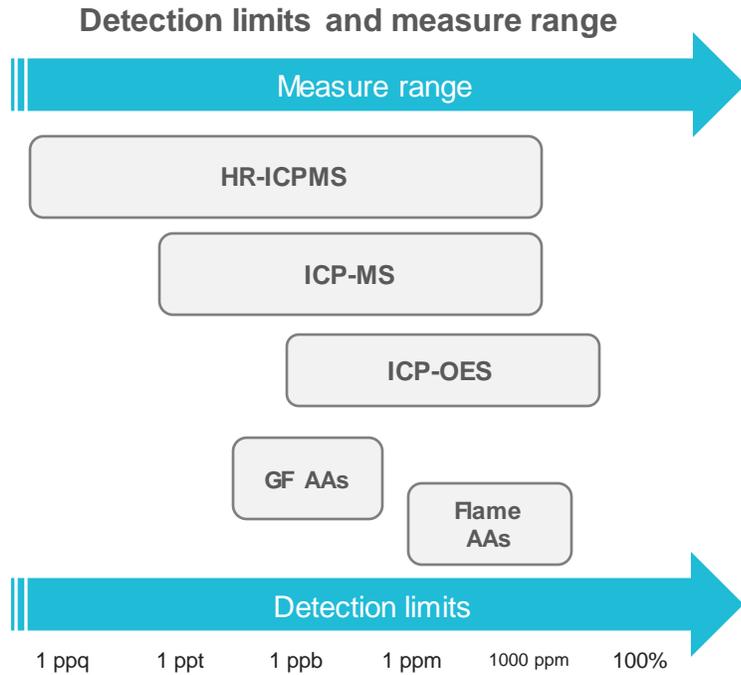
## ICP-OES

- Simultaneous
- Multi-elements
- ppb to %
- Highest matrix tolerance
- High sample throughput
- Ease to use

## ICP-MS

- Multi-elements
- High dynamic range and lowest limits of detection
- Detection limits in ppt range
- High sensitivity
- High sample throughput
- Ease to use

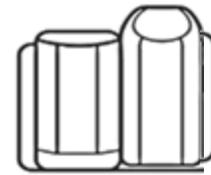
# Considers for instrument selection



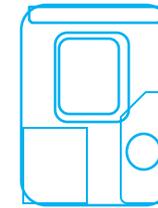
- ✓ ICP-MS offers high dynamic range and lowest limits of detection
- ✓ ICP-OES has highest matrix tolerance
- ✓ GFAAs offers sensitivity, best for few elements
- ✓ Flame AA offers fast analysis, and economical sol.

## Analysis Speed

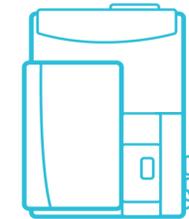
ICP-OES 2-5 min per sample	ICP-MS 1-3 mins per sample
Flame AA 15 sec per element per sample	GF AAs 2-3 min per element per sample



AAs



ICP-OES



ICP-MS

Low (<5) ← Number of Elements → High (>15)

One matrix ← Sample Matrix → Difference matrix

ppm ← Detection limits → ppb or lower

Low (<20) ← Sample throughput → High (>100)

# 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.



# Elemental analysis workflow



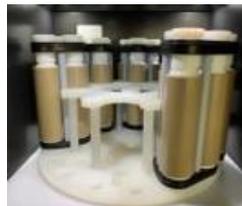
Acid Leaching



Wet digestion :  
Hot plate



Block Acid  
digestion



Microwave  
digestion



Dry ashing



# Standard Methods

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

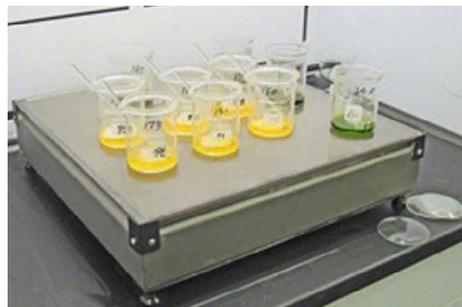
Hazardous Waste Test Methods / SW-846

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

# Sample preparation techniques

## 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.

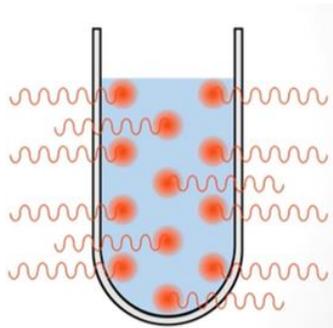
(adsorption of analytes to container walls or leaching of elemental impurities to the sample solution, are eliminated)

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 techniques - Microwave digestion

- Microwaves are a form of electromagnetic radiation with relatively low energy. It promotes the rotation of specific molecules in a reaction mixture, this rotation results in increased molecular collision and the generation of heat
- Combining the sample matrix and acids (HCl, HNO<sub>3</sub>, H<sub>2</sub>SO<sub>4</sub>, and H<sub>2</sub>O<sub>2</sub>) in a pressurized close container and elevating the solution past the boiling point of the acid
- Temperatures in the range of 200-260 °C
- Typical microwave digestion takes just 20-40 minutes

## Microwave heating



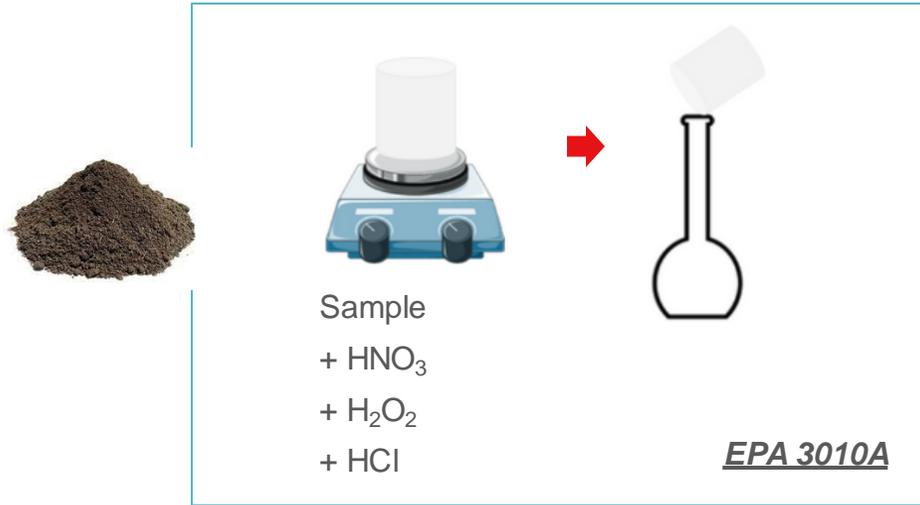
- ✓ Speed of digestion
- ✓ Quality digestion
- ✓ Reduced exposure to contamination
- ✓ Reduced reagent consumption
- ✓ Retention of analyte

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

# Soil, Sediment and Solid waste

- Nutritional elements: Al, Ba, Ca, Fe, K, Mg, Mn, P and Zn
- Trace elements: Cd, Co, Cr, Pb, Mo, Ni, V, etc.
- EPA Method 6010D (SW-846) – ICP-OES

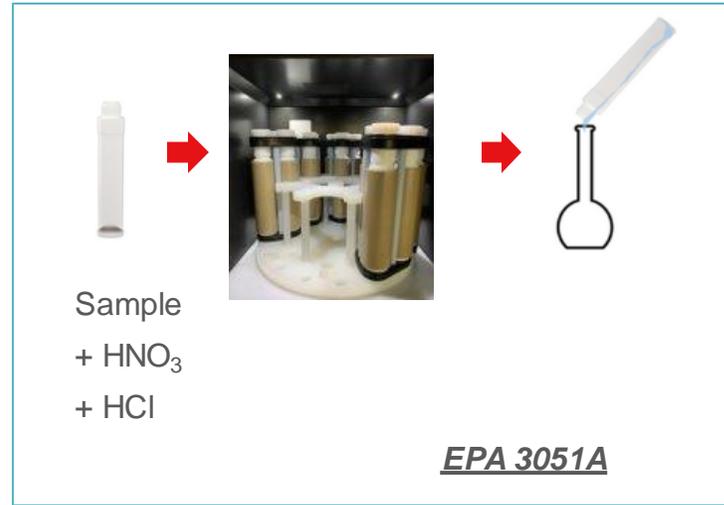
## Wet digestion



Sample  
+ HNO<sub>3</sub>  
+ H<sub>2</sub>O<sub>2</sub>  
+ HCl

**EPA 3010A**

## Microwave digestion

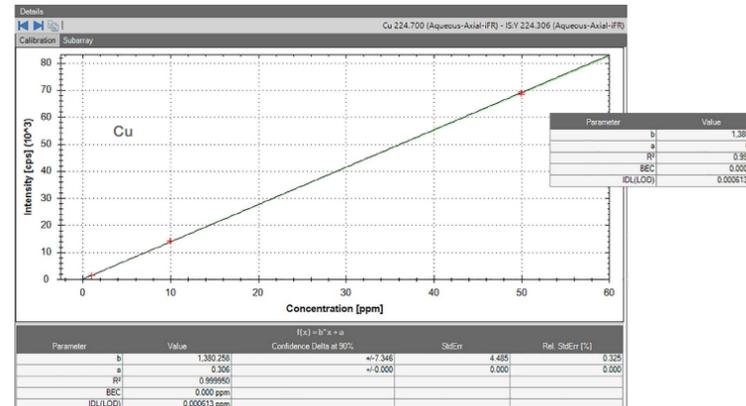


Sample  
+ HNO<sub>3</sub>  
+ HCl

**EPA 3051A**

Analysis by ICP-OES

**All elements in single run**



## thermo scientific APPLICATION NOTE

Fast, accurate, and robust analysis of environmental samples according to US EPA Method 6010D

Authors: Jierling Cui and Timothy Traylor  
Thermo Fisher Scientific, Germany  
Trace Analytical Services, EUSA Laboratory, United States  
Keywords: EPA Method 6010D (SW-846), environmental analysis, trace element correction (EC), EPA 3015A, EPA 3051A, microwave assisted acid digestion

**Goal**  
This application note describes the performance of the Thermo Scientific™ CAP™ PRO ICP-OES Duo system for the analysis of environmental samples by following the US Environmental Protection Agency (EPA) Method 6010D.



Since Congress enacted the Resource Conservation and Recovery Act (RCRA), which governs the disposal of solid and hazardous waste, suitable methods for the analysis of these types of samples are called into "Test Methods for Evaluating Solid Waste, Physical/Chemical Methods," more commonly known as SW-846. One of the specified methods is EPA Method 6010D (SW-846) "Trace Metals by Direct Plasma Atomic Emission Spectrometry." This method prescribes the use of inductively coupled plasma optical emission spectrometry (ICP-OES) instrumentation for the determination of trace elements in groundwater, industrial and organic wastes, soils, sludge, and sediments. Although EPA Method 6010D (SW-846) is used

## thermo scientific APPLICATION NOTE

Fast, simple analysis of soil and sediment according to German soil protection regulations

Authors: Sukanya Sengupta, Ralf Rohrling, and Cristian Copcariu  
Thermo Fisher Scientific, Bremen, Germany  
Laboratorien Dr. Döring, Bremen, Germany  
Thermo Fisher Scientific, UK

Keywords: ICAP-PRO X ICP-OES Duo, ICP-OES, ICP-MS, analytical testing, fast analysis, limits of quantification, method validation, robustness, sensitivity

**Goal**  
To demonstrate the analytical performance of the Thermo Scientific™ CAP™ PRO X ICP-OES Duo system for heavy metal analysis in soil samples based on the regulatory norms and methods in the EU and Germany.

**Introduction**  
Soil protection laws in Germany were introduced in 1998-2000 in the form of the Federal Soil Protection Act and Federal Soil Protection and Contaminated Sites Ordinance.<sup>1</sup> These laws were created with the goal of enhancing environmental protection as well as effective waste management and recycling. The guidelines prescribe the monitoring of trace elements to verify that risk to human and animal health hazards as well as leading to deteriorating environmental conditions in the long run. Therefore, it is of vital importance to conduct investigations of trace element concentrations in soils from agriculture

land, construction sites, agricultural, forests, and gardens, as well as wastelands. This may be ongoing for a specific purpose, such as land development.<sup>2</sup>  
Soil measurements demand analysis that can provide the sensitivity required, but speed and robustness are of equal importance. ICP-OES is ideal for such analyses. In this application note, the CAP PRO X ICP-OES Duo system was used to measure three samples with known trace elemental compositions and four standard reference materials (SRM) with certified reference trace elemental contents. These two independent soil sample analyses were substituted in a more extensive interlaboratory comparison with data of the same exact samples analyzed in different analytical testing laboratories using both ICP-OES as well as ICP-MS techniques. The SRM were additionally digested and analyzed using a



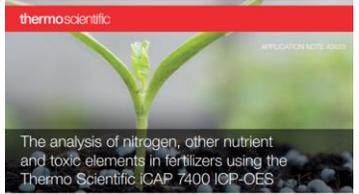
# Fertilizer (AOAC 2017.02)

## Elemental Categories in Fertilizer

Class	Elements
Primary Nutrients	N, P, K
Secondary Nutrients	Ca, Mg, Fe, Mn, Na, Cu, Zn, Mo, B, S,
Micronutrients	Al, Co, V, Se, Ni
Harmful trace elements	As, Cd, Cr, Pb

## Regulatory Limits of Elements in Fertilizers in Thailand

Element	Regulatory Concentration (mg/kg)
As	50
Cd	5
Cr	300
Pb	500

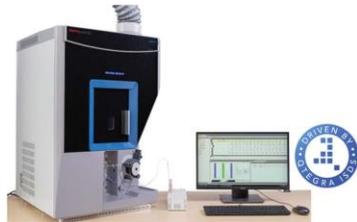
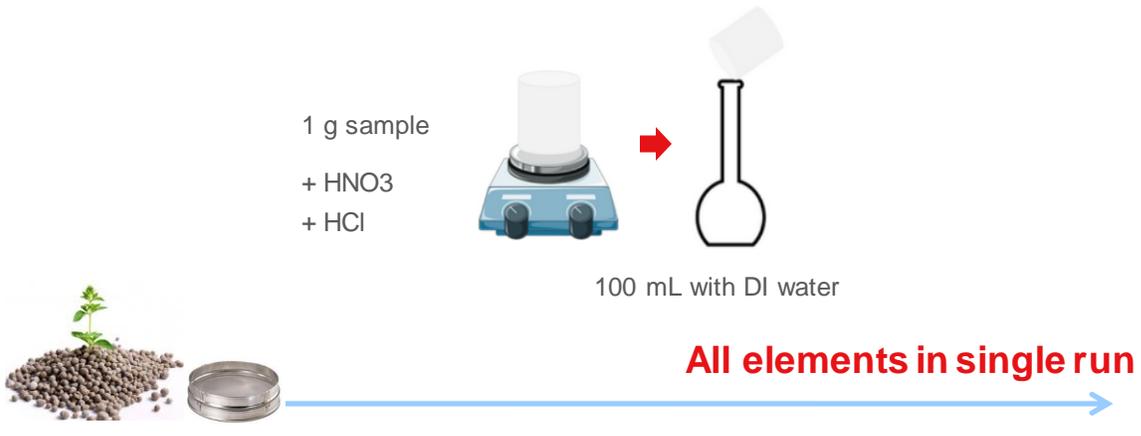


**Authors**  
Mathias Casale,  
Product Manager ICP-OES/AA,  
Thermo Fisher Scientific,  
Thermo, Germany

**Introduction**  
Fertilizers are used to provide major plant nutrients (N, P, K), secondary plant nutrients (Ca, S, Mg) and micronutrients such as B, Mn, Fe, Cu, Zn, Mo and Se. Accurate determination of the composition of fertilizers is essential so that the correct dose can be applied to the soil. An insufficient application of a fertilizer may result in poor crop yield. In contrast, an excessive application may result in environmental damage such as eutrophication - caused by dissolved phosphates and nitrate entering water courses or soil - contamination from non-nutrient elements within the fertilizers. ICP-OES facilitates the cost-effective analysis of fertilizers due to the powerful multi-element capability of this technique. The Thermo Scientific iCAP 7400 Plus Series ICP-OES enables the simultaneous analysis of all the plant nutrient elements (including nitrogen) as well as potential harmful elements like Cd, Cr or Pb in fertilizers.

**Keywords**  
Fertilizer, Nitrogen, Nutrients

**Goal**  
To demonstrate accurate analysis of nitrogen and other elements in fertilizers using the Thermo Scientific iCAP 7400 ICP-OES Duo.



Analysis by ICP-OES

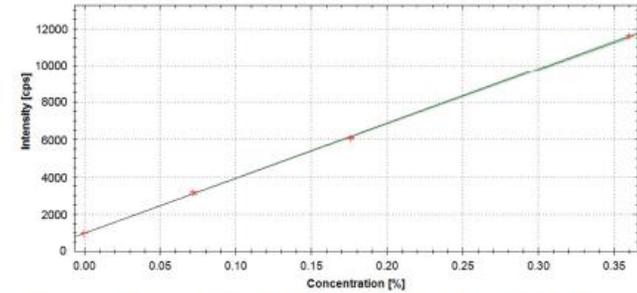


Figure 2. Calibration curve for N 174.272 nm with an R<sup>2</sup> of 0.9998.

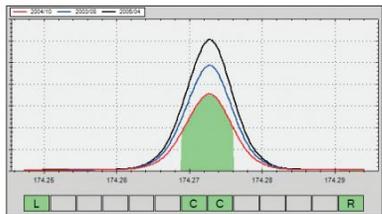
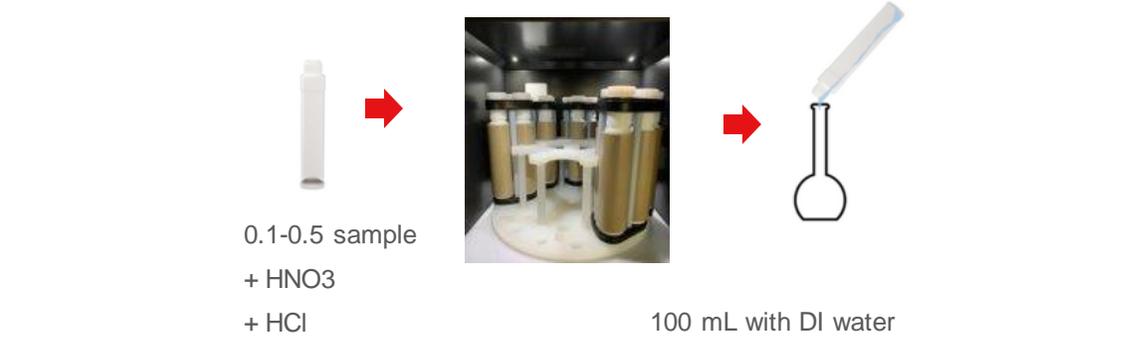


Figure 1. Subarray plot of N 174.272 nm.



# Cannabis / Kratom / Herbal

## Raw Materials and Products



- Liquid soap
- Soap bar
- Cannabidiol oil
- Caring oil

- Face cream
- Skin cream organic
- Gel cream organic

หน้า ๑๑  
เล่ม ๑๔๐ ตอนพิเศษ ๑๘๑ ง ราชกิจจานุเบกษา ๒๖ กรกฎาคม ๒๕๖๖

**ประกาศคณะกรรมการผลิตภัณฑ์สมุนไพร**  
เรื่อง ควบคุมคุณภาพและข้อกำหนดเฉพาะของผลิตภัณฑ์สมุนไพร  
และหลักเกณฑ์ วิธีการ และเงื่อนไขเกี่ยวกับหนังสือรับรองผลการวิเคราะห์ผลิตภัณฑ์สมุนไพร  
(ฉบับที่ ๒) พ.ศ. ๒๕๖๖

โดยที่เป็นการสมควรแก้ไขเพิ่มเติมข้อความแสดงรายละเอียดวิธีการทดสอบและเกณฑ์มาตรฐาน  
ของแต่ละชนิดทดสอบ ให้สอดคล้องกับตำราเกี่ยวกับผลิตภัณฑ์สมุนไพรฉบับปัจจุบัน  
อาศัยอำนาจตามความในมาตรา ๑๕ (๔) และ (๑๒) แห่งพระราชบัญญัติผลิตภัณฑ์สมุนไพร  
พ.ศ. ๒๕๖๒ คณะกรรมการผลิตภัณฑ์สมุนไพร ในการประชุมครั้งที่ ๗/๒๕๖๕ เมื่อวันที่ ๒๖ ธันวาคม ๒๕๖๕  
ออกประกาศไว้ ดังต่อไปนี้

ข้อ ๑ ประกาศนี้เรียกว่า “ประกาศคณะกรรมการผลิตภัณฑ์สมุนไพร เรื่อง ควบคุมคุณภาพ  
และข้อกำหนดเฉพาะของผลิตภัณฑ์สมุนไพร และหลักเกณฑ์ วิธีการ และเงื่อนไขเกี่ยวกับหนังสือรับรอง  
ผลการวิเคราะห์ผลิตภัณฑ์สมุนไพร (ฉบับที่ ๒) พ.ศ. ๒๕๖๖”

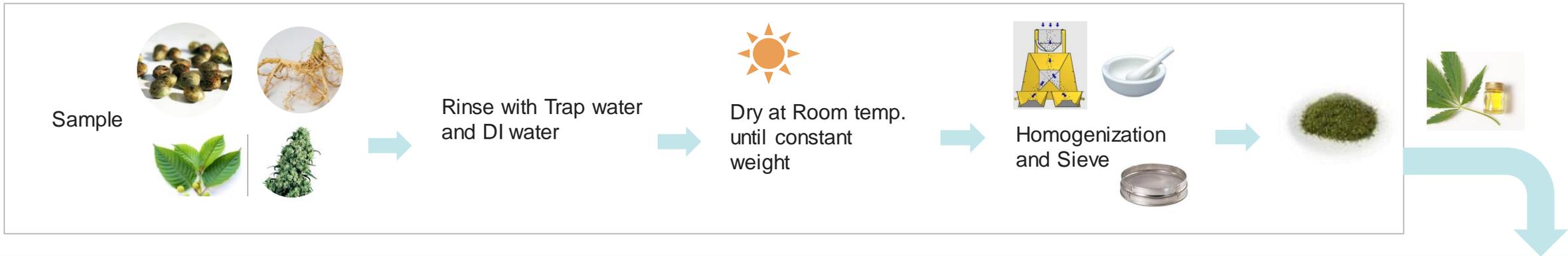
ข้อ ๒ ประกาศนี้ให้ใช้บังคับตั้งแต่วันถัดจากวันประกาศในราชกิจจานุเบกษาเป็นต้นไป

ข้อ ๓ ให้ยกเลิกความในบัญชี ๒ การแสดงรายละเอียดวิธีการทดสอบและเกณฑ์มาตรฐาน  
ของแต่ละชนิดทดสอบของเอกสารแนบท้ายประกาศคณะกรรมการผลิตภัณฑ์สมุนไพร เรื่อง ควบคุม  
คุณภาพและข้อกำหนดเฉพาะของผลิตภัณฑ์สมุนไพรและหลักเกณฑ์ วิธีการ และเงื่อนไขเกี่ยวกับหนังสือ  
รับรองผลการวิเคราะห์ผลิตภัณฑ์สมุนไพร พ.ศ. ๒๕๖๔ และให้ใช้ความในบัญชี ๒ แนบท้ายประกาศนี้แทน

ประกาศ ณ วันที่ ๔ กรกฎาคม พ.ศ. ๒๕๖๖  
พงศ์เกษม ไข่มุกด์  
รองปลัดกระทรวงสาธารณสุข  
หัวหน้ากลุ่มภารกิจด้านสนับสนุนงานบริการสุขภาพ  
ประธานกรรมการผลิตภัณฑ์สมุนไพร

*	Type	Pb	As	Cd	Hg
	Herbal medicines	10 mg/kg	-	0.3 mg/kg	-
	Raw herbal materials	10 mg/kg	5 mg/kg	0.3 mg/kg	0.2 mg/kg
	Raw herbal materials	10 mg/kg	5 mg/kg	1.0 mg/kg	0.5 mg/kg
	Finished herbal materials	10 mg/kg	5 mg/kg	-	0.5 mg/kg
	Finished herbal materials	20 mg/kg	5 mg/kg	-	0.5 mg/kg
	Raw herbal materials	10 mg/kg	5 mg/kg	0.3 mg/kg	0.5 mg/kg

# Cannabis / Kratom / Herbal



## Acid digestion & Analysis by ICP-MS

Weight 0.25 - 0.5 g



Add conc. HNO<sub>3</sub>, HCl, H<sub>2</sub>O<sub>2</sub>



Microwave digester



Adjust the final volume to 25 ml with ultrapure water



Qtegra Intelligent Scientific Data Solution (ISDS) Software

Raw mat.	HNO3	HCl	H2O2
Flower	✓	✓	✓
Leave	✓	✓	
Seed	✓		✓
Root	✓		✓
Products	✓	✓	✓

Step	Temperature (°C)	Heat time (min)	Hold time (min)
1	160	5	5
2	200	5	25

Technical Note: 43106  
Thermo Scientific Qtegra ICP-MS software for 21 CFR Part 11 compliant labs



# *Thank You* **Q & A**



 The world leader in serving science

