

Thermo Scientific TC/EA



*High Temperature Conversion
Elemental Analyzer*

Thermo Scientific TC/EA High Temperature Conversion Elemental Analyzer

For many years the on-line isotope ratio analysis of organic bulk samples for $^{13}\text{C}/^{12}\text{C}$, $^{15}\text{N}/^{14}\text{N}$ and $^{34}\text{S}/^{32}\text{S}$ have been performed rapidly, easily and precisely using a conventional Elemental Analyzer – continuous flow isotope ratio MS (IRMS). Measurement of $^{18}\text{O}/^{16}\text{O}$ and D/H ratios in organic and inorganic matter had been restricted to off-line sample preparation in the past. The Thermo Scientific TC/EA is the technological breakthrough offering the benefits of continuous flow IRMS to on-line oxygen and hydrogen isotope ratio analysis of solid and liquid bulk samples.

Direct Analysis of $^{18}\text{O}/^{16}\text{O}$ and D/H in Organic and Inorganic Samples

- Automated analysis with high sample throughput
- Only small sample amounts required
- Low blanks and negligible memory
- Oxygen isotope ratios from organic compounds, water and selected inorganic compounds
- Hydrogen isotope ratios from organic compounds and water
- Nitrogen and oxygen isotope ratios from nitrates



Principle of Operation

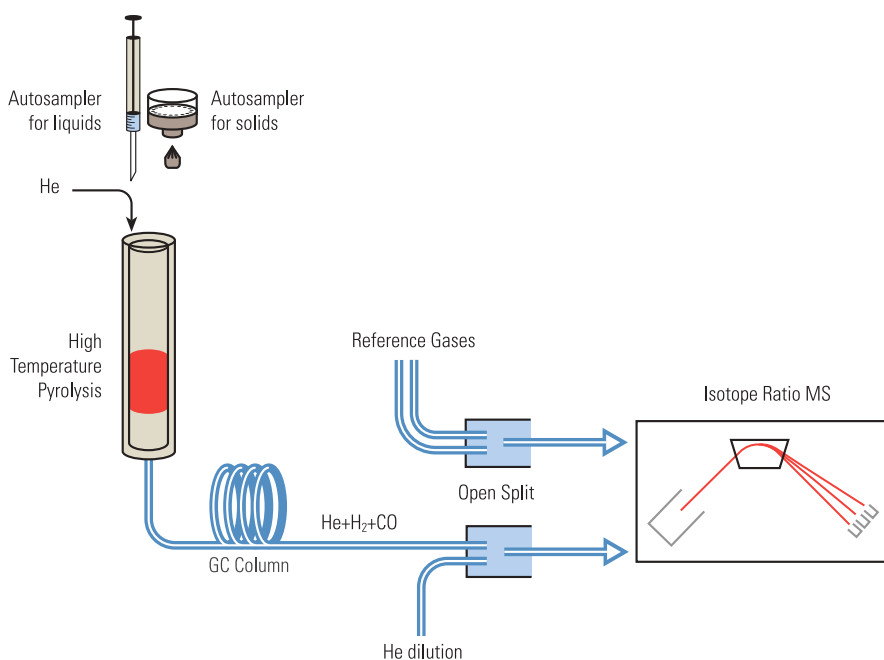
Quantitative high temperature conversion, also referred to as pyrolysis, is a new technique in which oxygen present in a compound is converted to CO, and hydrogen contained in a compound is converted to H₂. The process is rapid and quantitative in a reducing environment at high temperatures, typically exceeding 1400 °C.

The Thermo Scientific TC/EA preparation system and Thermo Scientific ConFlo III interface are outlined in the schematic diagram. This combination facilitates the conversion reaction, separation of reaction gases, transfer into the IRMS and referencing against standard gases.

The patented reactor⁽¹⁾ consists of a glassy carbon tube with glassy carbon filling, ensuring that neither sample nor reaction gases can get into contact with oxygen containing surfaces (e.g. Al₂O₃) while at high temperatures. Only this technology enables memory-free conversion reactions with no restrictions on compound type.

The reaction gases are separated in an isothermal gas chromatograph, which is also part of the Thermo Scientific TC/EA. The gases are admitted to the IRMS via the Thermo Scientific ConFlo III interface. The Thermo Scientific ConFlo III interface allows automatic sample gas dilution and generation of reference gas pulses, enabling individual referencing of each sample gas peak.

- No fractionation
- Lowest memory
- No restrictions on organic samples
- Usable for selected inorganic samples
- Referencing of each peak using reference gas



⁽¹⁾J. Koziet et E. Falou, French patent No. 2 734 363 "Méthode de mesure de la teneur en ¹⁸O et/ou ¹⁵N d'une substance chimique, appareillage et dispositif de pyrolyse"

Principle of High Temperature Conversion



Specifications and Installation Requirements

The Thermo Scientific TC/EA can be connected to any current Thermo Scientific IRMS equipped for continuous flow applications. If hydrogen isotope ratios are to be analyzed, the IRMS must be equipped with an energy filter to suppress $^4\text{He}^+$ ions on the DH collector (Thermo Scientific MAT 253, Thermo Scientific DELTA V Plus, Thermo Scientific DELTA V Advantage). A differential pumping system is required.

Gases

High purity helium (99.999% or better).

Reference gases (CO , N_2 , H_2) with pressure regulators.

To use the Thermo Scientific TC/EA with CO and H_2 reference gas, the laboratory must be equipped with CO and H_2 detectors.

Power

230 V, single phase, 8 A.

Dimensions and Weight

45 x 70 x 50 cm (W x D x H), 59 kg.

External Precision for Isotope Ratios, H, O, ($n = 10$), δ -Isotope

Benzoic acid at natural abundance for H and O - IRMS

	$^{18}\text{O}/^{16}\text{O}$	D/H
50 μg O, 25 μg H	0.4 ‰	3 ‰

Water for H and O - IRMS²

	$^{18}\text{O}/^{16}\text{O}$	D/H
0.5 μL	0.2 ‰	2 ‰



Applications

The Thermo Scientific TC/EA can be used for simultaneous hydrogen and oxygen isotope ratio determination of all organic compounds. Due to its very high maximum operation, temperature restrictions to individual sample classes do not exist.

For the analysis of hydrogen isotope ratios the IRMS must be capable of 4He^+ suppression at the DH collector (Thermo Scientific MAT 253, Thermo Scientific DELTA V Plus, Thermo Scientific DELTA V Advantage). Selected inorganic compounds can also be analyzed, such as nitrates (N and O), phosphates (O) and sulfates (O).

One important application is the isotope ratio analysis of hydrogen and oxygen in water samples. For this application an autosampler for liquid samples can be added to the system, replacing the standard autosampler for solid samples. Typical sample amounts for water are $< 0.5 \mu\text{L}$. The analysis of smaller water samples depends on the quality of syringe transfer and injection. Both isotope ratios can be determined within one run, making this setup perfectly suitable for e.g. high throughput doubly-labeled water analysis or screening of water resources.

Typical measurement time is about 5 minutes per sample for oxygen and less than 3 minutes for hydrogen isotope ratio determination. A determination of both isotope ratios in one run takes about 6 minutes.

Isotope Ratios of Single Elements

1. O	→	CO	→	$\delta^{18}\text{O}$, wt% O
2. H	→	H_2	→	δD , wt% H

IRMS, Isotope Ratios from Two Elements

3. H + O	→	H_2	→	δD , wt% H
		+ CO	→	$\delta^{18}\text{O}$, wt% O, H/O

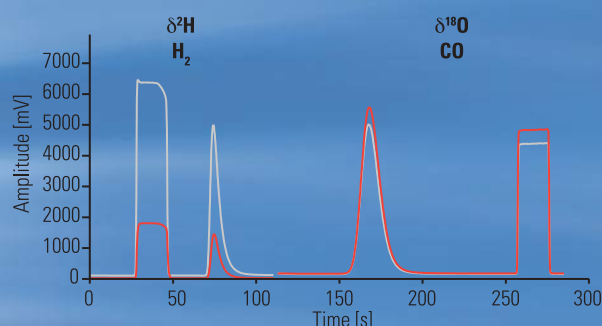
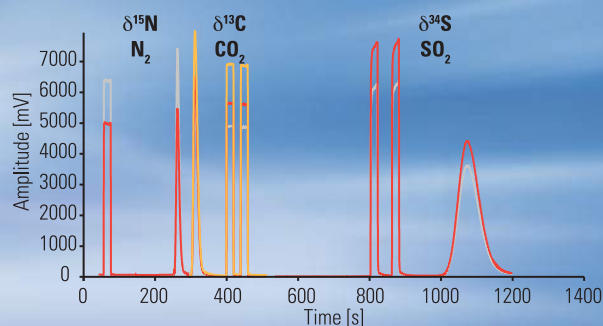
a. solids: organic and inorganic samples

b. water: $\text{H}_2\text{O} + \text{C} \rightarrow \text{CO} + \text{H}_2$ (requires liquid injector)

4. N + O	→	N_2	→	$\delta^{15}\text{N}$, wt% N
		+ CO	→	$\delta^{18}\text{O}$, wt% O, N/O

e.g. $2 \text{AgNO}_3 + 6 \text{C} \rightarrow 2 \text{Ag} + 6 \text{CO} + \text{N}_2$

Multi Element System: One Interface - Five Elements CNOHS



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