MASS SPECTROMETRY

Thermo Scientific TC/EA

High Temperature Conversion Elemental Analyzer

Part of Thermo Fisher Scientific

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For many years the on-line isotope ratio analysis of organic bulk samples for $^{13}C/^{12}C$, $^{15}N/^{14}N$ and $^{34}S/^{32}S$ has been performed rapidly, easily and precisely using a conventional Elemental Analyzer – continuous flow isotope ratio mass spectrometers (IRMS). Measurement of $^{18}O/^{16}O$ and D/H ratios in organic and inorganic matter had been restricted to off-line sample preparation in the past. The Thermo Scientific high temperature conversion elemental analyzer (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 $^{18}O/^{16}O$ 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 H_2. The process is rapid and quantitative in a reducing environment at high temperatures, typically exceeding 1400 °C.

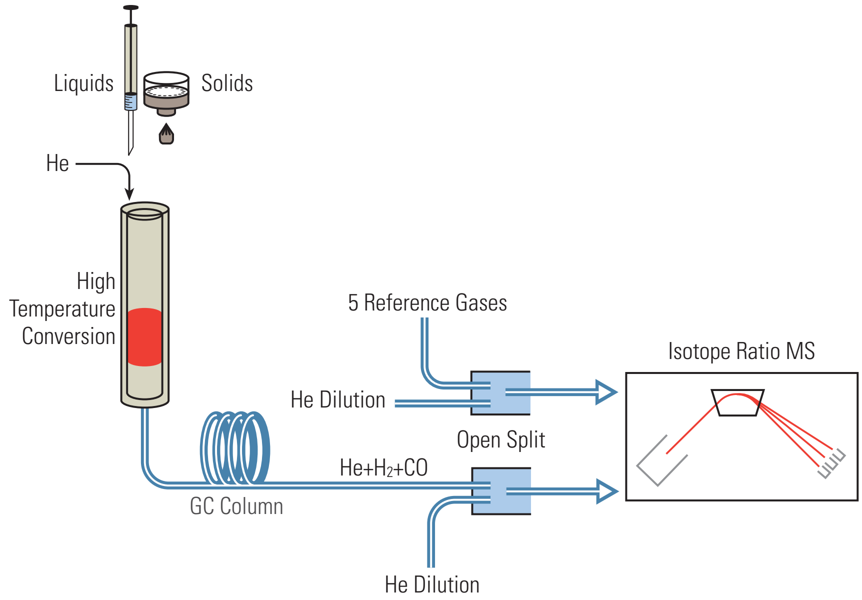
The Thermo Scientific TC/EA preparation system and Thermo Scientific ConFlo IV universal interface are outlined in the schematic diagram. This combination facilitates the conversion reaction, separation of reaction gases, transfer into the IRMS and referencing against standard gases.

The reactor 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. Al_2O_3) while at high temperatures. Only this technology enables memory-free conversion reactions with no restrictions on compound type.

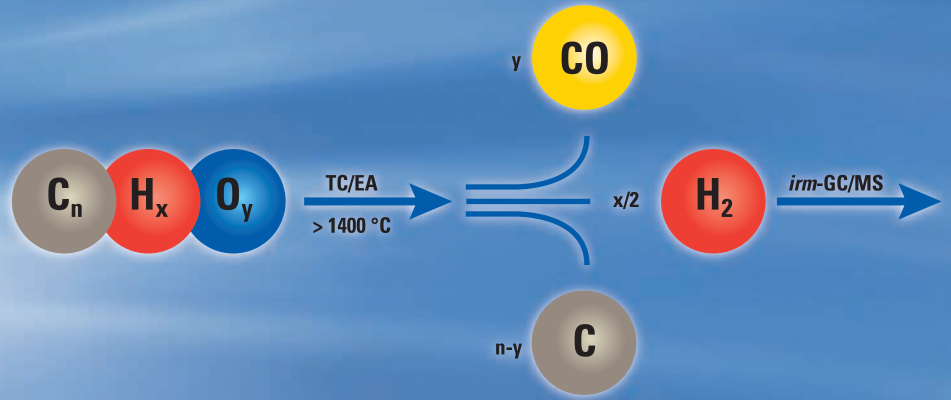
The reaction gases are separated in an isothermal gas chromatograph, which is also part of the TC/EA. The gases are admitted to the IRMS via the ConFlo IV universal interface. The ConFlo IV allows automatic sample gas dilution and generation of reference gas pulses, enabling individual referencing of each sample gas peak. The reference gas pulses can be automatically adjusted in signal intensity which also allows fully automated H^{3+} factor determination at any time within a sample sequence.

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

Schematic of the TC/EA, ConFlo IV and IRMS



Principle of High Temperature Conversion



Specifications and Installation Requirements

The Thermo Scientific TC/EA can be connected to any current Thermo Scientific Isotope Ratio MS equipped for continuous flow applications. If hydrogen isotope ratios are to be analyzed, the IRMS must be equipped with an energy filter to suppress $^4He^+$ ions on the DH collector (Thermo Scientific MAT 253, DELTA V Plus, and DELTA V Advantage IRMS).

Gases

High purity helium (99.999% or better).
Reference gases (CO and H_2) with pressure regulators.
To use the 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		
	$^{18}O/^{16}O$	D/H
50 µg O, 25 µg H	0.4 ‰	3 ‰
Water for H and O*		
	$^{18}O/^{16}O$	D/H
0.5 µL	0.2 ‰	2 ‰

** requires liquid injector*

Applications

The Thermo Scientific TC/EA high temperature elemental analyzer 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 $^{4}He^{+}$- suppression at the DH collector (Thermo Scientific MAT 253, DELTA V Plus, 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 µL. 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.

Options

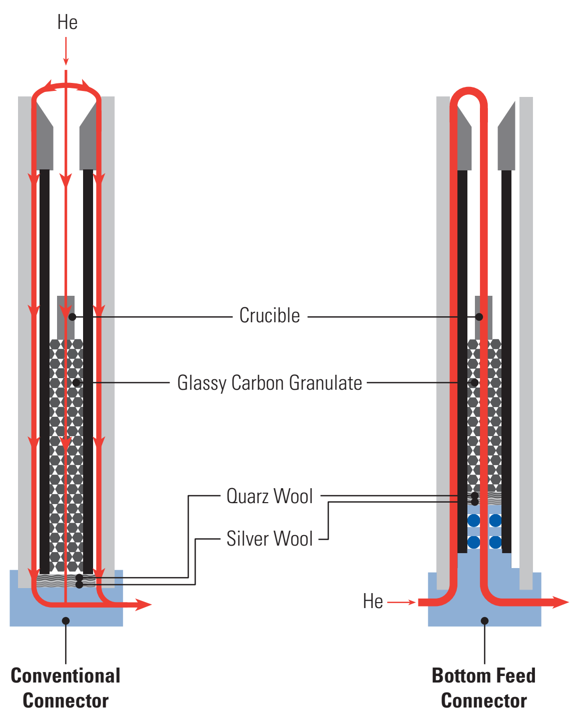
Liquid Injection Kit

Isotope analysis of water and liquids by high temperature conversion requires rapid transfer of the injected liquid into the reaction zone. The H_2O kit includes a special insert for the reactor, a septum-equipped injection head, a microliter syringe, screw top vials and spare parts. A special autosampler for liquids, e.g. Thermo Scientific AS3000, is recommended achieving high throughput and repeatedly reproducible injection for highest precision.

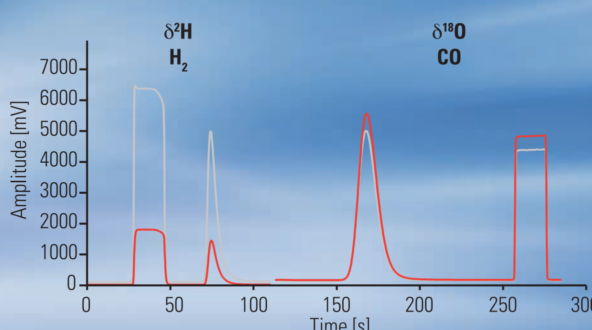
Bottom Feed Connector

The bottom feed connector (BFC) is the most recent improvement for high temperature conversion elemental analysis. By feeding the carrier gas flow from the bottom instead of the top, as in the conventional design, maintenance intervals become significantly less. Channeling of the complete flow through the glassy carbon tube reduces the effects of ash deposition which makes cleaning procedures occasional (less frequent). The BFC increases the analytical capacity of the high temperature conversion by at least a factor of five for solid samples and improves peak shapes. The design of the Thermo Scientific BFC allows the use of the original reactor material. No change of dimensions of the tubes is required.

Flow Path with Conventional and Bottom Feed Connector



One Injection - Two Isotope Ratios



Isotope Ratios of Single Elements

O	➡	CO	➡	$\delta^{18}O$, wt% O
H	➡	H_2	➡	δD, wt% H

Isotope Ratios from Two Elements

H + O	➡	$H_2 + CO_2$	➡	δD + $\delta^{18}O$, wt% H + wt% O
Solids: organic and inorganic samples				
Water*: $H_2O + C$	➡	$CO + H_2$		

** requires liquid injector*

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