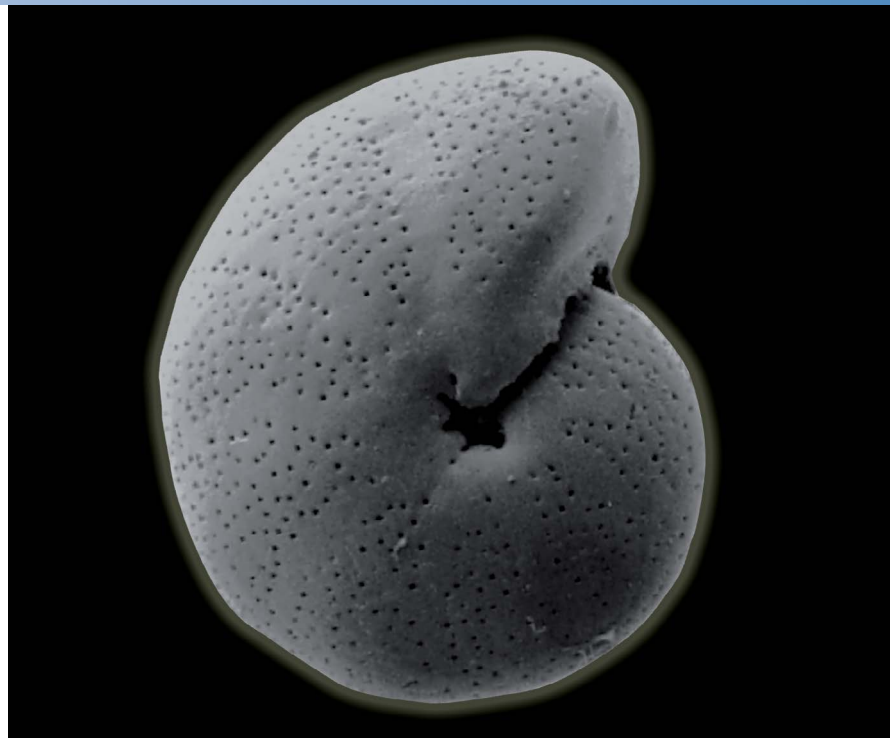


Thermo Scientific KIEL IV

Carbonate Device

The Thermo Scientific KIEL IV Carbonate Device provides fully automated long term performance at ultimate isotope precision for more than 10,000 samples per year.



Introduction

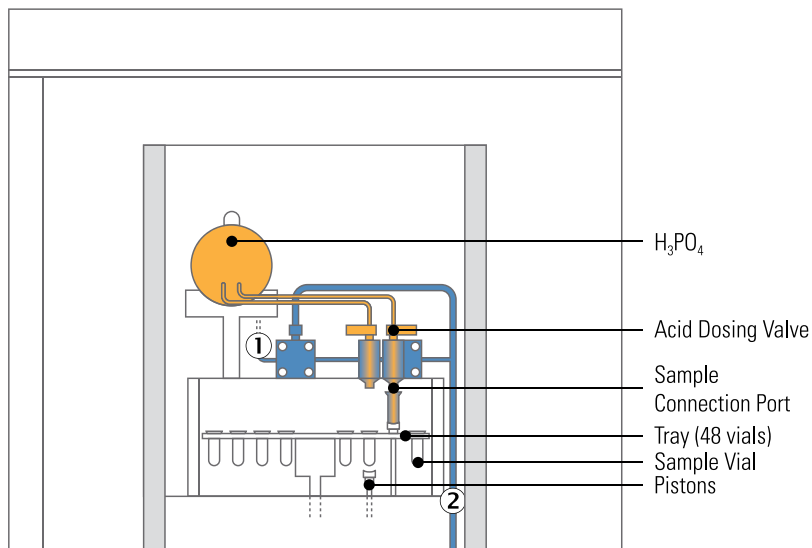
The KIEL IV Carbonate Device coupled to either the 10-kV Thermo Scientific MAT 253 or the 3-kV Thermo Scientific DELTA V isotope ratio mass spectrometer meets the requirements of such work by providing the highest linearity at highest sensitivity, while enabling high throughput through full automation of the analytical process, from the reaction to the reporting of measurement results.

- Unique temperature controlled reaction cabinet for ultimate isotope precision of carbonates

- Two independent reaction lines with acid dosing valves for high sample throughput
- New microvolume ensuring viscous flow of CO₂ from small samples of carbonate
- “Fast bellows” strategy reducing CO₂ sample consumption during standard-to-sample pressure adjustment
- Total process control and data log file for complete insight to all preparation and measurement processes at any time
- Time slicing for realtime information of data acquisition with a resolution up to 80 data points per integration cycle

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Temperature controlled reaction cabinet (front view)



Gas trapping and cleaning cabinet (side view)

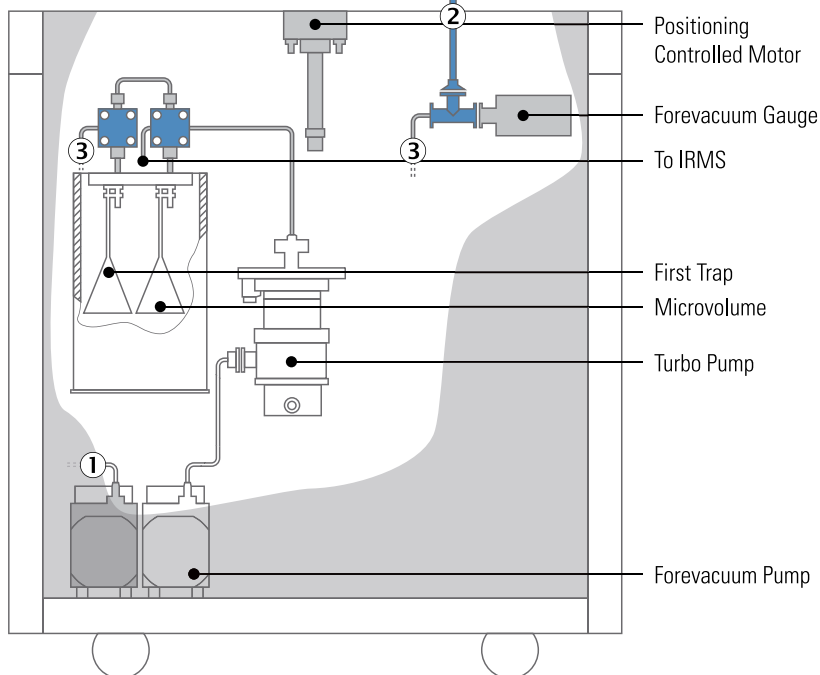


Figure 1. Inside views.

Inside Views of the KIEL IV Carbonate Device

The inside views of the temperature controlled reaction cabinet (top) and the gas trapping and cleaning cabinet (bottom) show:

1. The connection to four vacuum pumps for vial pre-evacuation
2. The connection of reaction region and first trap
3. The connection to high vacuum region for first trap and microvolume evacuation

CO₂ Sample Gas Preparation and Transfer

The reaction of carbonates with phosphoric acid produces CO₂ and H₂O plus non-condensable gases from impurities in the sample. The cryogenic trapping system consists of a temperature controlled first trap with associated valves, ultra-high vacuum system, pressure gauge and a microvolume.

- Carbonate samples, phosphoric acid storage, acid transfer and chemical reaction under identical conditions in a precision temperature controlled oven (+/- 0.1 °C)
- Storage of phosphoric acid in a 500 mL reservoir in the thermostated environment of the KIEL IV to keep the acid under absolutely dry conditions and for a long measurement period
- Additional drying storage of prepared sample vials in the same temperature controlled environment as sample analysis (+/- 0.1 °C)
- Full temperature controlled and CO₂ memory-free individual acid bath reaction
- System uses 105% dry phosphoric acid
- Septum-free and reusable borosilicate vials
- Fully observable vial content at any time
- High sample throughput for high precision carbonate analysis (46 samples a day)
- Drop test under vacuum and reaction conditions before chemical reaction
- Control and monitoring of ultra-high vacuum in the complete reaction and preparation system
- Automated high vacuum system check before each sample reaction from the sample vial to the CO₂ freezing trap

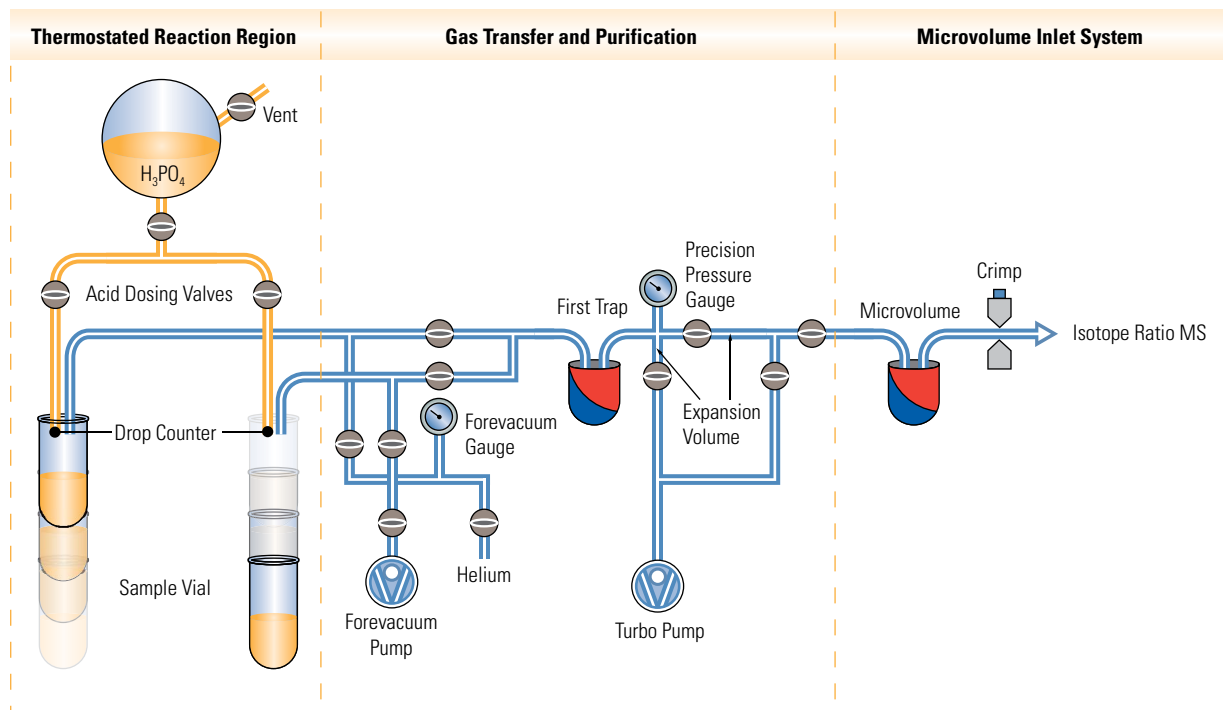


Figure 2. Schematic of the KIEL IV Carbonate Device.

In the first step, CO_2 and H_2O are trapped into the first automated liquid nitrogen trap at $-190\text{ }^\circ\text{C}$ while any non-condensable gases are removed. The CO_2 is then released at $-90\text{ }^\circ\text{C}$ for transfer into the microvolume, while the water is completely retained in the first trap. Based on the pressure of the released CO_2 , the Isodat software suite defines the portion of CO_2 being transferred into the microvolume. This software process assures the optimal sample gas pressure in the Isotope Ratio MS. In parallel the reference gas bellow in the dual inlet is pre-adjusted to the expected inlet pressure.

- Low liquid nitrogen consumption (0.5 L/sample) by automated liquid nitrogen refill into a low volume Dewar with a new level detector mechanism
- High throughput of carbonate in routine operation (> 10,000 analyses per year) with the only consumable being LN_2
- Redesigned trapping and gas cleaning system with new transfer line, LN_2 level indicator and trap temperature control
- Water and temperature resistant compact cartridge design which includes both the temperature sensor and heating cartridge
- Perfect temperature regulation through direct positioning of the cartridge to the spot of sample freezing
- CO_2 sample yield determination with a precision pressure gauge at the first trap
- Complete bake-out of the system

Sample Analysis and Referencing

The microvolume is heated to $+30\text{ }^\circ\text{C}$ releasing the CO_2 via a dedicated stainless steel capillary to the changeover valve and into the IRMS for $\delta^{13}\text{C}$ and $\delta^{18}\text{O}$ analysis. In parallel, water is removed from the gas cleaning and trapping system by baking the first trap and evacuating all valves and gas lines.

- New small microvolume inlet systems for KIEL IV and Dual Inlet
- New microvolume design for smallest amount of samples with highest precision (e.g. in the range of $6\text{ }\mu\text{g}$ to $130\text{ }\mu\text{g}$). See Thermo Scientific Application Note AN30176.
- Regulated CO_2 transfer to the KIEL IV microvolume to achieve the desired inlet pressure for measurement
- Same CO_2 sample gas flow from the chemical reaction to the IRMS guarantees the principle of identical treatment of samples
- Fast bellow reference gas pressure pre-adjustment using the CO_2 sample yield determination
- Highest precision isotope measurements with a dedicated stainless steel inlet capillary

An overall external precision of 0.04‰ for $\delta^{13}\text{C}$ and 0.08‰ for $\delta^{18}\text{O}$ using the Thermo Scientific MAT 253 is reached for samples greater than $20\text{ }\mu\text{g}$. In routine operation, laboratories have shown throughputs up to 15,000 analyses per year with the only consumable being LN_2 .

Analytical Performance

With a sample size of > 20 µg for MAT 253 or > 40 µg for DELTA V and MAT 253, the following performance for one full autosampler run (46 samples) will be achieved.

External Precision 1 σ

Carbon ($^{13}\text{C}/^{12}\text{C}$)	0.04‰
Oxygen ($^{18}\text{O}/^{16}\text{O}$)	0.08‰

For more details on analytical performance, please contact your local sales representative.

Installation Requirements

Instrumentation

The KIEL IV Carbonate Device connects to MAT 253 and DELTA V stable isotope ratio MS equipped with a dual inlet system.

Liquid Nitrogen

0.5 L/sample

Compressed Air

350 to 500 kPa (50 to 75 psi)

Gases

Helium or argon, about 15 mL per sample (for pressurized release of sample containers from reaction position)

Power

50/60 Hz

Voltage

230 V, single phase, 10 A = 2,200 Watt

Environment

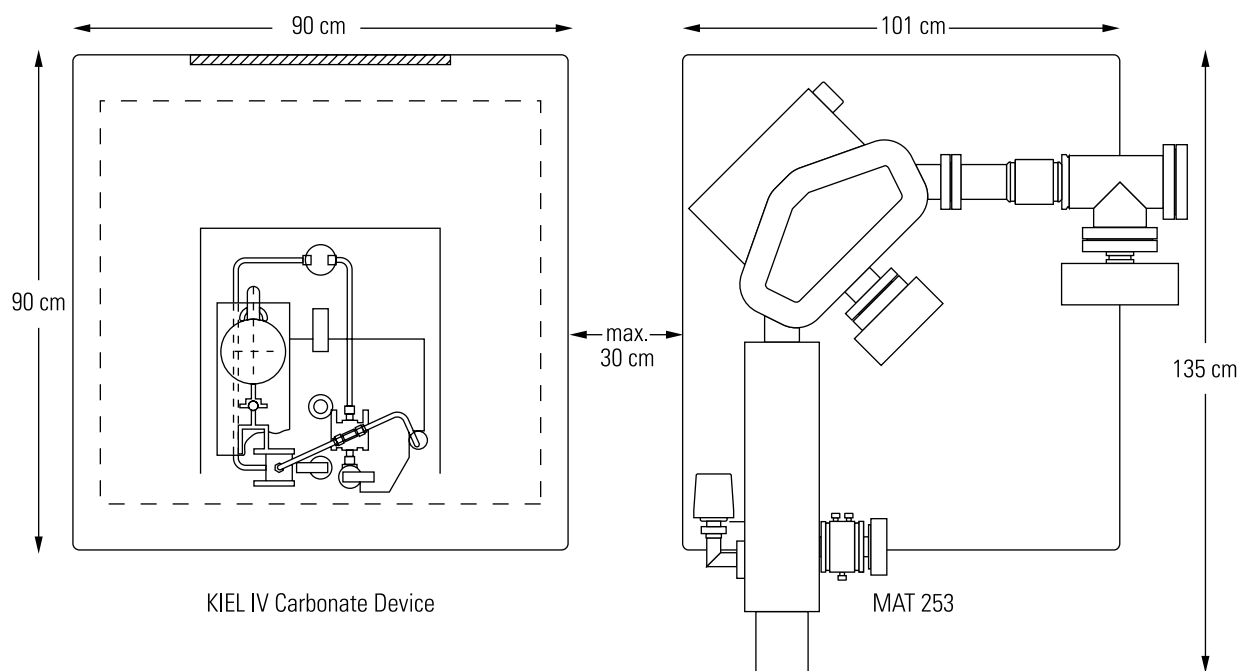
Ambient temperature between 18 °C and 28 °C, with a relative humidity between 20 and 70%

Dimensions

90 cm (width) × 90 cm (depth) × 190 cm (height)

Weight

Approximately 100 kg (220 lbs)



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