

LIENSs Stable Isotope Facility

Appendix 1 : Instrumentation and facilities

1. Analytical equipments (room B106/107)

The Isotope Ratio Mass Spectrometer (IRMS) Delta V Advantage

The **Delta V Advantage** IRMS (Thermo Scientific, Bremen, Germany) has been set up on April 2008. It is fitted with a 5 collectors system for the measurement of the masses from 1 to 80, allowing the analysis of the isotope ratios of C, N, O, S and H elements.



Delta V features in Continuous Flow Mode

Sensitivity : 1500 M/l Molecules CO₂ / mass 44
(linearity 0,02 ‰ / nA)
Mass range 1 – 80 Dalton at 3 kV
Mass Resolution 110m/ Δ m (10 % valley)
Stability < 10 ppm
H₃⁺ Factor < 10 ppm / nA
H₃⁺ Factor Stability < 0,03 ppm / nA / h
Noise level < 50 dB(A)
External precision (specifications : <0,15 ‰ (CO₂) ;
<0,20 ‰ (N₂) (for running standards)

For isotope ratio mass spectrometry, samples either organic or inorganic have to be converted into simple gases. Diverse preparation devices and interfaces are available for many different applications.

The Delta V can be hooked permanently to up to 3 peripherals and 5 reference gas through the universal interface **ConFlo IV**. This interface allow controlling and adjusting the reference gas level, and using the Smart-EA option to dilute each sample gas to the reference gas level based on the signal of the EA-TCD.



The Isotope Ratio Mass Spectrometer (IRMS) Delta V Plus

The **Delta V Advantage** IRMS (Thermo Scientific, Bremen, Germany) has been set up on April 2008. It is fitted with a 5 collectors system for the measurement of the masses from 1 to 80, allowing the analysis of the isotope ratios of C, N, O, S and H elements.



Delta V features in Continuous Flow Mode

Sensitivity : 1100 M/l Molecules CO₂ / mass 44
(linearity 0,02 ‰ / nA)
Mass range 1 – 96 Dalton at 3 kV
Mass Resolution 110m/ Δ m (10 % valley)
Stability < 10 ppm
Noise level < 50 dB(A)
External precision (specifications : <0,15 ‰ (CO₂) ;
<0,20 ‰ (N₂) (for running standards)

The Delta V Plus IRMS is coupled to peripherals via an universal interface ConFlo IV.

Peripherals :

- The **Flash EA 1112** Elemental Analyzer (Thermo Scientific, Milan, Italy) allow the conversion of solid (or liquid) samples placed in tin or silver capsules, to gases, the purification and the separation of these gases, and their subsequent quantification before their introduction into the mass analyzer for the C, N and S isotope ratio measurement.

C and N isotope ratios can be measured on the same sample, the S isotope ratio is analyzed apart, using different reactors and CPG column.

This analyzer is connected to the Delta V Plus IRMS.

The present autosampler is a Zero Blank (Costech, Milan, Italy) that replaced the original autosampler and NoBlank option during October 2009. Two sample trays can be used with this sampler : a 50-positions tray (mainly for sediment or filter samples) and a 100-positions tray (for plant and animal samples).

Standards are analyzed along with samples (2 every 20 samples) and 3 blanks are measured at the beginning of every run.



The elemental analyzer is fitted with the SmartEA option which, in conjunction with the ConFlo IV, allows the automatic dilution of the sample gas as to adjust them to the reference gas level, for each element. The range of sample weight is rather wide thanks to the automatic dilution, but a minimum of 10 µg of C and N is needed to achieve the required precision.

Taking account of all working procedures (insert replacement, sample loading, helium purge, stability test ..), from 65 to 120 samples can be analyzed per day (C & N), depending of their type and the autosampler tray used.

The overall precision achieved for the acetanilide standard on the long term is better than 0,04 ‰ for C and 0,07 ‰ for N.

- **The Flash EA 2000** elemental analyzer (Thermo Scientific, Milan, Italy) has the same features as the Flash EA 1112. It is also fitted with a Costech ZeroBlank autosampler and with the SmartEA option. It is coupled to the Delta V Plus IRMS.



- The **GC-Isolink** system links a **GC TRACE Ultra™** to the DELTA V for measuring isotope ratios (C, N) of specific biochemical compounds within complex mixtures (presently used for fatty acids).



- a **carbonate preparation line is linked to the Delta V** for the determination of isotope ratios (C, O) on very small (10-300 µg) samples of carbonates. It is constituted by a reactor, a line including gas traps, a 6-way valve, a Poraplot Q Varian column, and a high vacuum system TPS-Compact Varian.

The overall precision achieved is < 0,1 ‰ for C and O isotope ratios.

- The High Temperature Conversion Elemental Analyzer **TC/EA Thermo Scientific** allows the high precision on-line stable isotope determinations of $^{18}\text{O}/^{16}\text{O}$ and D/H in small organic and some inorganic solid and liquid bulk samples using pyrolysis.



2. Sample preparation

- **Christ Alpha 1-4 freeze-dryer** (room B109): for sample drying before grinding. For a better efficiency of the ball mill, samples are kept overnight in a desiccator under vacuum and over P_2O_5 after freeze-drying.

- **Retsch MM400 ball mill** (room B111) : for isotopic analysis, samples must be ground as to get a very fine powder (like flour) for a better combustion and a better representativity of the sample aliquote. Grinding is generally very fast (1 to 3 minutes).

Various jars are available : inox 10 and 25 ml, zirconium 35 ml, and racks for 20 x 2 ml microtubes.



- **Rotary agitators** (x2) (room B 108) for removing lipids from 2 x 20 samples



- **Jouan / Thermo CR4i centrifuge** (for large sample, room B109) or **MicroStar centrifuge** (for very small samples, room B108), required by the protocol of lipid removal

- The 2 **dry baths** are used (room B108): one for evaporating the rest of solvents after lipid removal (a); the other for evaporating HCl after carbonate removal (b). This latter is fitted with a system for flushing each vial with filtered air to speed up evaporation.



- **Microbalance Sartorius ME5** (room B107) : its precision is $\pm 1 \mu\text{g}$, not really required for the isotopic analyses, but needed for a good precision of %C and %N data.



- **Static eliminator AD-1683** (A & D Company) (room B107) : this equipment allows to eliminate static electricity and thus facilitates weighing of certain samples. Placing the tubes during about 15 s between 10 and 30 cm in front of the static eliminator is sufficient to remove static electricity.



- **Drying oven with forced convection Froilabo** (x2) (room B111) : the small one (50 L) is specifically dedicated to the drying of the grinder accessories, the large one (200 L) to the drying of the usual glassware.

These ovens are dedicated to clean materials, and must not be used for sample drying.



- **Static oven Memmert** (room B111) :
This drying oven is specifically dedicated to the drying of samples (in peculiar to samples preserved with ethanol).

