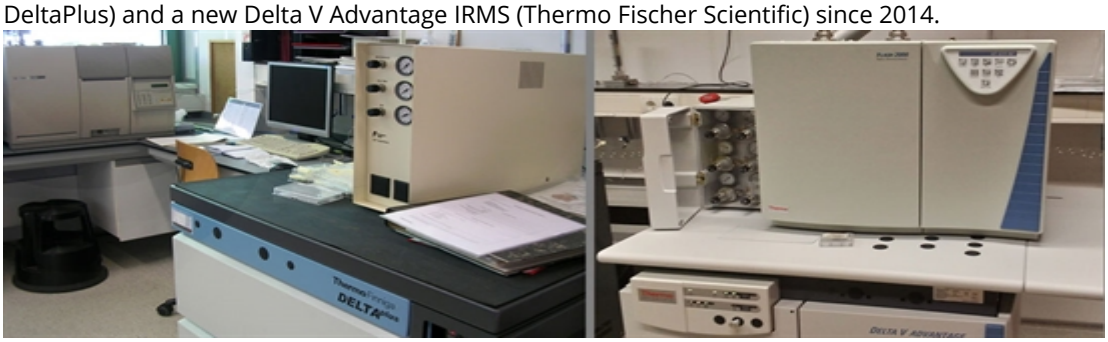
IRMS

Isotopes Ratio Mass Spectrometry

Stable isotopes can be measured by a coupling of the **CN elemental analyzer** (EA NA1500 - EA 1110 device, Carlo Erba and Thermo Fisher Scientific) through an **interface** (the Conflo III) to a **mass-spectrometer** (a Finnigan DeltaPlus) and a new Delta V Advantage IRMS (Thermo Fischer Scientific) since 2014.

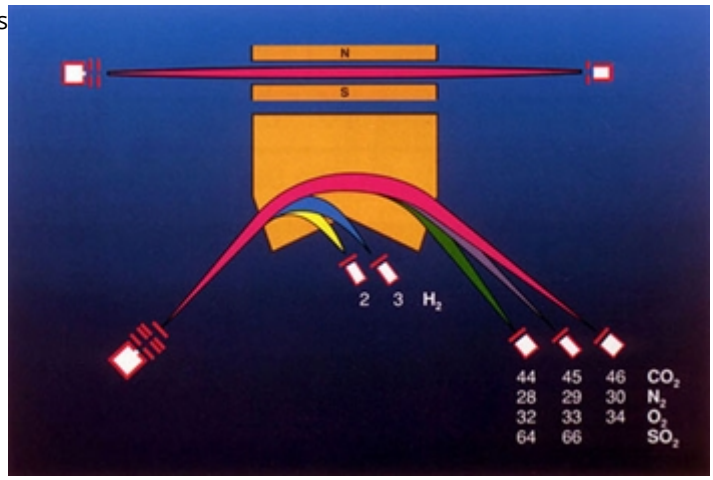


Release of N_2 and CO_2 in the CN elemental analyzer

In the first step samples consisting of solid material (e.g. plant tissue, animal material, soil, sediments) are very rapidly combusted and transformed into gaseous products, in particular pure N_2 and CO_2, of the elements from which they are constituted. This conversion occurs in the CN elemental analyzer (https://www.ru.nl/science/gi/facilities-activities/elemental-analysis/cn-elemental/) (see page with an introduction on this technique). The sample preparation for radio isotopes resembles that for C and N analysis, but there are specific conditions. Contact Paul van der Ven.

Separation of isotopes in the IRMS

In second instance the **δ** N and **δ** C are determined. This occurs in the **IRMS** (isotopes ratio mass-spectrometer). Products of the CN elemental analyzer are introduced into the mass-spectrometer, where they are ionized, accelerated and separated on account of their mass. At the end of the pathway these ions are detected by Faraday cups, positioned in such a way that three different masses can be simultaneously caught. For nitrogen (N_2) the masses are 28, 29 and 30 and for carbon (CO_2) the masses are 44, 45 and 46.



Dual isotope analysis

For this analysis the C:N ratio should be in balance, but often the C amount is too high. When necessary, the CO_2-signal can be diluted with an additional helium flow in the Conflo unit, so that the contribution of CO_2 is comprised in the right measurement range. At high C:N ratio one should monitor that the signal of mass 28 and 29 of nitrogen does drop too much, as then the delta N cannot be ascertained with precision. The decision has to be made to perform the stable isotope measurements for C and N separately.

CNS – OH IRMS

A new IRMS instrument, a DELTA V Advantage Isotope Ratio Mass Spectrometer (Thermo Fisher Scientific) has been installed in November 2014.

This device is coupled to a Flash HT 2000 elemental analyser (Thermo) through the Thermo Conflo IV interphase . The elemental analyser contains two ovens: one for isotope ratio analysis of CNS by means of the “Dynamic flash combustion” method (see elemental analyzer (https://www.ru.nl/science/gi/facilities-activities/elemental-analysis/cn-elemental/) page for further explanations), and one oven for isotopes ratio analysis of H and O by means of “High Temperature Conversion”, also called pyrolysis. The breakdown of the samples into carbon dioxide and hydrogen at high temperature (1450 °C) happens in a special chamber (pyrolysis reactor) under interaction of quartz with oxygen and hydrogen. The

emitted gases are separated on the gas chromatograph of the analyzer before being conducted to the IRMS. The IRMS is equipped with a triple collector (Faraday cups) for N_2, CO, NO, O_2, CO_2 , N_2O and SO_2, as well as a H_2 collector; there are two additional Faraday cups for m/z 2 en 3.

The DELTA V Advantage is at present operational; for isotope analyses of **δ** N **δ** C and **δ** S. The instrument should also function for **δ** O and **δ** H from mid 2016 on.

What are stable isotopes and why employing them?

Isotopes are atoms with the same number of protons, but with a different number of neutrons, so these atoms differ in mass, but hardly in chemical behavior. Isotopes can exist in both stable and unstable forms. The unstable atoms are radioactive. The stable forms that are of biological interest regard light elements, i.e. H, C, N, O and S. These elements have different proportion of at least two isotopes, where the lightest of these isotopes is predominantly present. The prevalence of various isotopes of an element is called abundancy. Small variations in abundance of certain isotopes is found through time, at different locations in the world and in different organisms. One factor playing an important role in this phenomenon is that δ12C is enriched relative to δ13C during the process of photosynthesis, while also a different yield is observed between C3 and C4 plants. Isotopes of carbon and nitrogen, often added to a system in enriched form, are powerful tools to trace relationships in a food chain and to assess physiological metabolic processes.

Sample preparation and delivery

Discuss the sample preparation and the planning in advance with Paul van der Ven.

For the analysis of isotopes it is of great importance that **enriched samples** (C^{13} or N^{15}) are kept separate from non-enriched (natural abundancy) samples. Prevent contamination. Three important steps are distinguished: drying, grinding and weighting.

1. Drying. Samples should be dried before grinding them, but also for another hour or so, just before weighting them.

2. Grinding. For analysis, it is of course, very important to use samples with a uniform composition, which moreover are representative for the object to study. In order to obtain a homogenous composition and structure of the sample-matrix, one should take care to have fine particles of uniform size, chemical composition and purity. In practice, this means grinding the material to talk dust-sized particles. For this purpose, one can use the present at the GI.

3. Weighting. The samples are weighed into tin containers of 8x5mm using the analytical micro-balance and forceps. For carbon measurements on the IRMS the containers should be pre-glowed due to carbon contamination. Ask Paul which cups to use (these cups are brownish-purple).The containers with weighted samples should be shaped to little round balls. The shape is relevant for a good 'Flash Combustion'. Moreover, flat or misshapen balls can get entangled in the auto-sampler! Balls can be shaped with help of a forceps, but it is also allowed to do this by hand, using gloves. Use also clean tools, weigh in a dust free environment, and prevent contamination from one to another sample.
For the first IRMS analyses of new material, one should perform a pre-analysis on test samples to determine the best amount of sample to weight per container. The amount of sample needed per cup depends on the type of samples:
- 1 mg for animal material,
- 3 mg for aerial parts of plants (e.g. leaves),
- 10 mg for root material and
- 5-40 mg for soil and sediment samples (depending whether from mineral or organic origin; some soils consist of 90% of plant materials).
- Samples heavier than 50 mg can cause problems during combustion.

4. Delivery and submission of samples. A good way to store and deliver samples is to use 96-wells titer plates. The corresponding weights should be entered in an excel sheet (WEIGHTS CN IRMS (xls, 43 kB) (/publish/pages/635552/weights_cn_versie13-03-2016.xls)). Internal users are requested to enter their demand for analysis of samples in the bookings (https://bookings.science.ru.nl/public/auth/login/) system, together with the filled weight sheet. External users can send a submission form.

DELTA V Advantage samples

The sample preparation for the DELTA V Advantage IRMS requires special attention, in particular for the **δ** S analysis; the concentration of S has to be high enough, as measurement of S is impeded in large weighted samples. Discuss the possibility to add Vanadium pentoxide to the weighted samples in order to achieve a better combustion.

IRMS (https://www.ru.nl/fnwi/gi/faciliteiten-activiteiten/element-analyse/irms/)

Location of equipment

Room HG 01.210

Assistance

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Internal users

- Download weight sheet (xls, 43 kB) (/publish/pages/635552/weights_cn_versie13-03-2016.xls) for IRMS samples
- Bookings (https://bookings.science.ru.nl/) of microbalance
- Submit samples and upload filled weight sheet in Bookings (https://bookings.science.ru.nl/)

External users

- Submission sheet for IRMS samples (docx, 19 kB) (/publish/pages/635552/irmssample_submission_sheet.docx)
- Weight sheet (xls, 43 kB) (/publish/pages/635552/weights_cn_versie13-03-2016.xls) for IRMS samples

Contact Paul van der Ven in advance to discuss sample preparation and planning

Links

- Stable isotope laboratoria (http://isogeochem.wikispaces.com/SIRMS+Labs)
- RFF (http://www.ru.nl/radboudresearchfacilities/english/facilities/nano-and/ir-mass-spectrometer/) for shared facilities

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