



# irm-EAMS routine - measurements: high precision; high accuracy; high throughput

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## Introduction

Since about six years we analyse  $\delta^{13}\text{C}$  and  $\delta^{15}\text{N}$  of bulk organic and inorganic sample material with high precision. Hence, these measurements, which are made using an EA-IRMS system, are daily routine in BGC-IsoLab. More than 25000 measurements in 2004 demonstrate, that the interest in stable isotope analyses inside the institute as well as from outside partners has remained strong and has even increased over time.

In order to cope with the increased request for measurements we have acquired a second EA combustion unit in the second half of 2004. With this additional capacity, which we activate during times of high sample backlog, we have been able to shorten the average dwell time of submitted samples considerably.

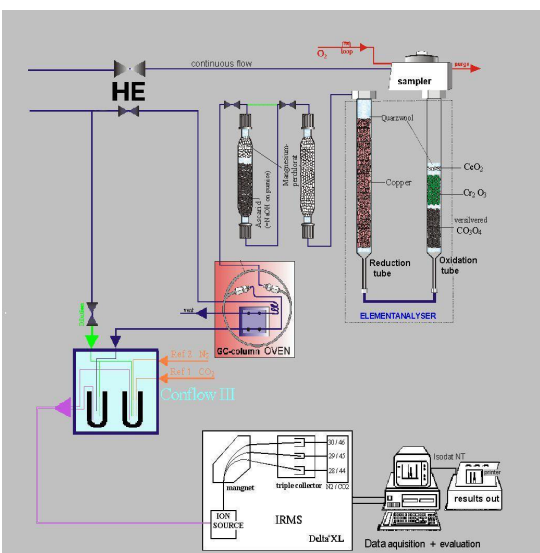
Typical samples we analyse derive from plants or from soil, but these are by no means the only sources. Special attention of the operator is required for some soil samples, when the C and / or N content is very low, or for samples with a high volatility like methanol or alkanes, which we measure as a service for other techniques like GC-combustion.

A considerable effort went into new reference materials or into round robin checks of existing reference materials. The latter activities have been in collaboration with the IAEA in Vienna, with the USGS and other international partners. It is within this context that we have made a large number of  $\delta^{13}\text{C}$  analyses on carbonates, oils, sugars, cellulose samples etc.

All measurements are routinely controlled using QA-standards as well as internal working reference materials. High precision data require that the signals of working reference materials and measured samples are of comparable size. Hence, the carbon or nitrogen content of the materials must be known a priori and weighing of samples and standards must be made with utmost care.



**Simon (Delta C; 'Stable Isotope Monitoring System')**  
Delta C prototype IRMS (Finnigan MAT); the instrument is coupled on line with an EA 1100 (CE, Maastricht) via a ConFlo III interface



## How do we ensure accuracy and avoid problems during measurement?

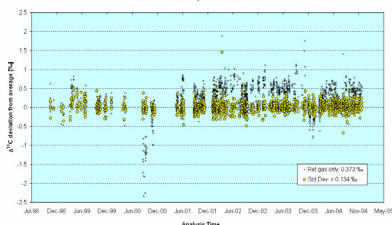
- > we measure  $\delta^{13}\text{C}$  or  $\delta^{15}\text{N}$  separately. The concentration of C and N usually are so different, that accurate measurement of  $\delta^{15}\text{N}$  requires removal of  $\text{CO}_2$  with Ascarite (NaOH on pumice)
- > every day samples, standards, and blanks are measured together in one sequence
- > blanks in general must be very low. Their measured values are used to correct samples for blank contribution.
- > every day we control the system background for water and argon and check the status of the scavenging chemicals ( $\text{Mg}(\text{ClO}_4)_2$  and Ascarite)
- > for every sequence we can select an appropriate He dilution dependent on sample amount
- > the working range by weight varies from a maximum of 100 mg to less than 100  $\mu\text{g}$ . However, no more than 5 mg C are allowed due to the amount of oxygen available during combustion. The minimum amount of C should not be lower than 50  $\mu\text{g}$  C (without dilution).
- > All consumables (i.e. tubes and reagents) inside the EA are exchanged regularly. On the CE-EA, combustion and reduction reactors are always replaced with new ones as a pair after 700 analyses. In 2004, the reactors were renewed 35 times. The new Eurovector EA allows to exchange the reactors separately (and fast). Exchange of the  $\text{Mg}(\text{ClO}_4)_2$  water traps occurs rather infrequent due to the integration of a Nafion dryer unit.
- > ISODAT NT simplified the work with the EA TC-MS considerably; it is much more economical in terms of the time spent to prepare sample sequences. Moreover, copy & paste from the weighing list to the sequence table results in a reduction of errors and an improved consistency of sample names.
- > It is always very important for us and for the whole analytical process to interact efficiently with the person submitting samples to IsoLab on a personal basis...

## What kind of samples can we analyse?

- organic bulk samples (well prepared, i.e. dry and homogeneous)
- soil samples with known concentrations of C and N within the specified ranges for sample amounts (C > 0.5%, N > 0.2%)
- samples from tracer experiments, however only after intense discussion. Tracer amount should be as low as feasible. Samples must be prepared and weighed in a special lab with dedicated tools (danger of contamination of natural isotope ratio samples)
- volatile samples must be analysed by hand. Solvents like methanol, acetone or alkanes with low boiling points are most critical due to possible alteration of the isotopic composition by evaporative loss.
- reference materials, e.g. carbonates or oil, demand special care due to the requirement for high precision.
- samples with acid treatment are difficult to analyse and require special awareness.

### $\delta^{13}\text{C}$ Performance of caf-j1 and caf-j3

Scale based on all-j1 lab reference material

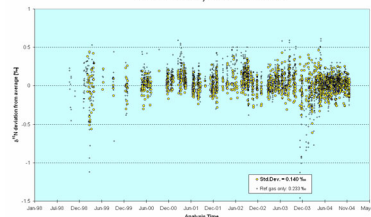


### Performance Charts:

$\delta^{13}\text{C}$ -values and  $\delta^{15}\text{N}$ -values of a quality control standard as a function of measurement time. The scale is based on a carefully calibrated working reference material that passed through the same preparation channel as the QA standard. Each sequence of 32 samples measured holds one QA standard that is represented by a single point.

### $\delta^{15}\text{N}$ Performance of caf-j1 and caf-j3

Scale based on all-j3 lab reference material



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