Automatic, sensitive determination of the ^{15}N abundance of inorganic N compounds in aqueous samples using the SPINMAS measuring system

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The ^{15}N determination of tiny amounts of N in the form of nitrite and nitrate can be carried out quickly and precisely in a single step by chemically converting the nitrogen in these compounds selectively into nitrogen monoxide (NO), which is then introduced into a suitable mass spectrometer using helium as carrier gas (Russow, 1999). In order to use this method for automated routine analysis, a set-up was developed in which the chemical conversion of the inorganic nitrogen compounds nitrite and nitrate as well as ammonium and hydroxylamine to form the gases NO as well as N_2 and N_2O takes place automatically under PC control (SPIN – **S**ample **P**reparation of **I**norganic **N** compounds, Russow et al., 1999). The SPIN unit is connected to a GAM 400 quadrupole mass spectrometer (InProcess Instruments GmbH, Bremen) and an automatic 222 XL Liquid Handler (GILSON). The entire measuring process is PC-controlled thanks to measuring sequences being programmed. The material problems caused by the very aggressive reaction solutions call for the use of especially corrosion-resistant materials.
Each measurement takes no longer than 8 min. The concentration of the respective N compound can also be determined by the measuring system described here, if medium accuracy is sufficient.

References

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Factors controlling the performance of IRMS Systems

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Since 1950 the focus in isotope ratio mass spectrometry (IRMS) is to enhance the precision of isotope ratio determination while reducing the sample size. However, the "classical" dual inlet technique limits the minimum sample size in the range of 5 bar- microliter.

With the introduction of continuous flow applications in 1988 for compound specific isotope analysis (CSIA) using GC combustion a major breakthrough for smallest sample sizes was achieved. This was followed by bulk stable isotope analysis (BSIA) coupling an elemental analyzer via an open split interface to the IRMS. This boost in overall sensitivity had to be balanced by accepting a lower precision due to the transient GC peaks used in continuous flow analysis when compared to dual inlet applications.

Today all major isotopes in organic samples (CHNOS) are available in continuous flow IRMS heavily reducing the workload of sample preparation. Subsequently the focus of CF-IRMS research moves towards reading very small isotope signatures in nature within total ranges of a few ‰, like for CO_2 and other trace gases in air and in water currents, seasonality in tree rings and calcareous materials.

The research and monitoring of such minor isotope signals requires improved sensitivity, stability and linearity of the IRMS as well as of the continuous flow interfaces providing sample preparation, transfer and automation.
For all these applications the long term accuracy and precision is one of the most crucial parameters combining high performance with robustness.

The factors controlling the performance of continuous flow IRMS systems will be demonstrated on basic tests. Improved routine performance will be shown on specific applications.

Temperature effects on delta 13C of soil-respired CO2: an incubation study with arctic soils

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The delta 13C value of soils is mainly determined by the delta 13C value of the incoming litter and fractionation processes during organic matter decomposition. Thus, the C isotopic composition of soils and respired CO2 varies with composition of SOM, activity of heterotrophs and soil depth.
Arctic ecosystems store vast amounts of the earth`s soil carbon. In a global warming scenario this represents a ticking time bomb of potential increase of atmospheric CO2, one of the most important greenhouse gases. Although a loss of CO2 from arctic ecosystems to the atmosphere has been reported recently, it is as yet unclear whether microorganisms are able to utilze the large pool of more stable, recalcitrant C compounds.
In order to analyze the effect of temperature on the biological sources of heterotrophs in arctic soils we measured respiration rates and delta13C values of respired CO2 and soils incubated at 2, 12 and 24°C. We found a consistent increase in respiration rates across the entire temperature range in organic horizons. In mineral horizons, respiration rates increased between 2°C and 12°C, but were similar between 12°C and 24°C. The relationship between soil temperature and the stable C isotope ratio of CO2 produced by heterotrophs was linear, with depleted delta13C values of CO2 at higher temperatures relative to lower temperatures. Additionally, the delta13C values of CO2 were highly correlated to the delta13C value of bulk soil at 24°C but not at 2 and