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Titel

SPIN-MIMS simplifying the SPIN-MAS instrumentation for online measurement of ¹⁵N-abundances of ammonium, nitrite and nitrate in aqueous solutions

Abstract

Common methods for measuring selectively the ¹⁵N abundances in individual N-species such as NH₄⁺, NO₂⁻ and NO₃⁻ in samples with multiple N-species are laborious and time consuming.

The SPIN-MAS technique (Stange et al. 2007) offers an automated, rapid and selective determination of ¹⁵N abundances in NH₄⁺, NO₂⁻ and NO₃⁻ in aqueous samples. During a SPIN-MAS measurement one of three different reaction solutions is mixed with the aqueous sample in a Sample Preparation unit for Inorganic N-species (SPIN). The reaction solution is chosen in dependence on the N-species of interest. The gaseous reaction products (N₂ or NO) are then conducted to a quadrupole mass spectrometer (MAS) in a helium stream. This measurement technique is not commonly used due to its complex instrumentation.

The instrumentation can be significantly simplified by the use of a membrane inlet mass spectrometer (MIMS). The presented SPIN-MIMS approach relies on the use of a reaction capillary in which the sample containing the N-species of interest is mixed with the corresponding reaction solution. The mixture of reaction solution and sample is pumped from the reaction capillary directly to the membrane inlet of the mass spectrometer. The reaction products (N₂ or NO) formed during the reaction of NH₄⁺, NO₂⁻ and NO₃⁻ with the reaction solutions are passed through the gas-permeable membrane of the inlet directly into the ion source of the mass spectrometer.

¹⁵N standards with different at% ¹⁵N (NH₄⁺, NO₂⁻ and NO₃⁻ respectively in dist. Water) were used to assess the performance of the system. Overall, SPIN-MIMS measurements showed a good agreement between measured and expected ¹⁵N abundances (range 0.36 – 10 at% ¹⁵N deviations: <0.5 at% ¹⁵N for NH₄⁺, <0.23 for NO₂⁻ and <0.15 at% ¹⁵N for NO₃⁻ standards).

Literatur

Stange, C.F., Spott, O., Apelt, B. und Russow, R.W.B. (2007) Automated and rapid online determination of ¹⁵N abundance and concentration of ammonium, nitrite, or nitrate in aqueous samples by the SPINMAS technique. *Isotopes in Environmental and Health Studies*. 43, 227-236.