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STABLE ISOTOPES OF OXYGEN AND NITROGEN IN NITRATE IDENTIFICATION. MEASURING TECHNIQUES

A widespread contamination of water with nitrate is of global concern to environmentalists. The ratio of nitrogen to oxygen isotopes can be used to distinguish between different sources of nitrate. This paper describes analytical methods and field applications of isotope measurements in environmental studies. New reference materials combined with previously determined standards are set up. A proper calibration of different techniques is essential to improve interlaboratory comparability. The most proper method was chosen for our own studies of nitrates in rivers.

1. INTRODUCTION

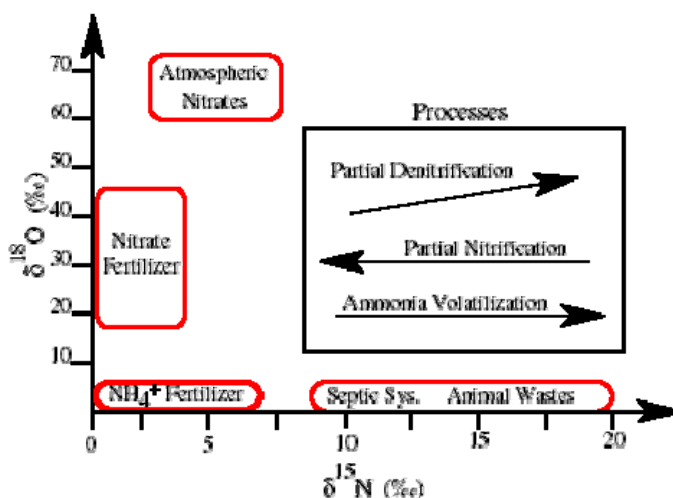
The analyses of oxygen and nitrogen isotopes of nitrate in the atmosphere and hydrosphere can provide important information about sources, transport and reactions of these elements in the environment. Identification of nitrate sources is important to elucidate, among others, the causes of increased nitrate fluxes in aquatic ecosystems in recent years. High concentration of nitrate in rivers and lakes can be responsible for eutrophication and acidification of sensitive local environment and degradation of drinking water.

Recently, several analytical methods have been described that permit the use of isotope ratio R measurements. Absolute differences in R can be very small and difficult to detect. A larger value being easier to measure is the difference between the ratio of the substance of interest and a common standard. For practical reasons, instead of using the isotope ratio R , isotopic composition is generally given as δ value, a relative deviation with respect to a standard value, which is defined by:

$$\delta [\text{‰}] = (R_{\text{sample}}/R_{\text{standard}} - 1) \cdot 1000. \quad (1)$$

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Recently, techniques have been developed to determine $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ values to find the sources of nitrate in water [1]–[4], [6], [7], [9], [10], [13], [17], [25], [29], [35], [36]. Nitrate $\delta^{18}\text{O}$ values show a wide range, i.e., from about -10 to $+80\text{‰}$. Previous studies have shown that $\delta^{18}\text{O}$ value of nitrate from inorganic fertilizer is usually $+23 \pm 3\text{‰}$, for atmospheric oxygen it varies between 25 and 75‰, and for nitrates produced by nitrification in soil it ranges from -2 to $+6\text{‰}$ [22], [24], [29], [35], [38]. Experiments have shown that in biologically produced nitrates, one oxygen atom comes from atmospheric O_2 , and other two ones come from the water [11], [22], [28]. This contrasts sharply with nitrates in synthetic fertilizers, which receive their oxygen primarily from atmospheric O_2 . However, interpretation of nitrogen isotope ratios has not always led to unequivocal results, since many sources of nitrate have wide and overlapping ranges of $\delta^{15}\text{N}$ values [22]. Isotopic fractionation effect which occurs during different nitrogen transformations, such as nitrification, ammonia fixation, denitrification, further complicates the $\delta^{15}\text{N}$ value interpretation for soil, groundwater and surface water. Despite the above, $\delta^{15}\text{N}$ values for inorganic fertilizer nitrate ranged between -1 and $+2\text{‰}$ and for animal waste sources or sewage, from $+8$ to $+16\text{‰}$ [38]. These applications are most successful in well-drained soils and oxygenated groundwaters where nitrification is rapid and denitrification is minimal – under these conditions $\delta^{15}\text{N}$ values are relatively conservative [17]. Such processes as ammonia volatilization, ion exchange, denitrification often modify δ values of nitrogen, hence indentifying the source of compounds or tracing their origin is very difficult. But simultaneous analysis of both $\delta^{15}\text{N}$ and $\delta^{18}\text{O}$ can distinguish denitrification process from other transformations. The figure shows typical ranges of δ values from various sources.



Schematic isotopic composition of nitrate sources [16]

2. METHODS FOR ANALYSING STABLE ISOTOPES OF NITROGEN AND OXYGEN

Isotopes of different elements are usually analysed by mass spectrometry techniques which separate ions on the basis of their differing mass/charge ratio (IRMS). A promising new method for measuring isotope ratios in water is laser absorption spectroscopy (LS). KERSTEL et al. [19] conclude that LS yields the results comparable to or better than IRMS. This is a highly precise method with accuracy of $\pm 0.7\%$ for $\delta^2\text{H}$ and $\pm 0.5\%$ for $\delta^{18}\text{O}$ and $\delta^{17}\text{O}$, while calibration accuracy is ± 3.0 and $\pm 1.0\%$, respectively.

Despite a high precision of LS method, it is not the most common method for measuring the isotope ratios in water. These ratios are still determined by mass spectrometry analyses. Different techniques for measuring the isotope ratios are among those methods which involve the sample preparing and converting into an applicable form of the element analysed. For years the methods for preparing nitrate from natural water for $\delta^{15}\text{N}$ analysis have involved evaporation or freeze-drying of filtered samples. The resulting solid samples are combusted to produce pure nitrogen. In 2003, the National Water Quality Laboratory (NWQL) in USA has arranged for the Reston Stable Isotope Laboratory (RSIL) to carry out the analyses which were performed in the past. The RSIL provided new analytical capability for oxygen isotope ratios of NO_3^- in water and soil and N and C isotope ratios of solid samples. In the laboratories, special codes were used for analyses and when the new procedures were introduced, new lab codes and schedule were presented (see the table [37]).

Table

The lab codes and schedule for nitrogen isotope analyses

Description	Old lab code/ schedule	New lab code/ schedule
$^{15}\text{N}/^{14}\text{N}$ of nitrate dissolved in water	LC1718	LC 2899
$^{15}\text{N}/^{14}\text{N}$ and $^{18}\text{O}/^{16}\text{O}$ of nitrate dissolved in water	–	LC 2900
$^{15}\text{N}/^{14}\text{N}$ of ammonium dissolved in water	LC1717	LC 2898
$^{15}\text{N}/^{14}\text{N}$ of nitrate in soil	LC1719	LC 2894
$^{15}\text{N}/^{14}\text{N}$ and $^{18}\text{O}/^{16}\text{O}$ of nitrate in soil	–	LC 2897
$^{15}\text{N}/^{14}\text{N}$ of ammonium in soil	LC1720	LC 2895
$^{15}\text{N}/^{14}\text{N}$ and N concentration of solids	–	LC 2893
$^{15}\text{N}/^{14}\text{N}$, $^{13}\text{C}/^{12}\text{C}$, N and C concentration of solids	–	S 1832

According to new procedures analytical methods described by lab codes LC 2899, 2900, 2894 and 2897 allow measurement of $^{15}\text{N}/^{14}\text{N}$ and some $^{18}\text{O}/^{16}\text{O}$ isotope ratios. They involve a bacterial conversion of nitrate into nitrous oxide and nitrogen isotope analysis by continuous flow isotope ratio mass spectrometry (IRMS) [8], [33]. LC

2898 method involves the collection of ammonium in ion exchangers, combustion by elemental analyser, purification of N₂ by gas chromatography (GC), and isotopic determination by continuous flow isotope ratio mass spectrometry [5]. Determination of ¹⁵N/¹⁴N of ammonium in soil (LC 2895) involves aqueous dissolution of ammonium in KCl solution, distillation and collection of ammonium in ion exchanger, combustion by elemental analyser, purification of N₂ by GC, and isotopic analysis by IRMS [5]. New LC 2893 procedure involves combustion by elemental analyser, purification of N₂ by GC, and isotopic determination as above. The last procedure in the table was also described by BRAND [5]. It is the same procedure as LC 2893, but additionally CO₂ is taken into account. But in many laboratories, numerous modifications to the standard procedures were adopted because of sample character or usability. According to KENDALL [17] ammonium can be removed from the sample and converted into N₂ for MS analysis by steam distillation of ammonium followed by oxidation and purification of N₂ in a Cu/CuO furnace, distillation followed by collection of ammonium on a specific zeolite and sealed-tube combustion using Cu/CuO and CaO to produce pure N₂, and the so-called “micro-diffusion method” where ammonium is slowly diffused into an acid solution to produce (NH₄)₂SO₄ and combusted or reacted as above to form N₂. Automated combustion of solid samples for C, N and S isotopes with an elemental analyser connected to a continuous-flow MS has recently become a very popular method. Nitrate has also been prepared by distilling off any existing ammonium, reducing nitrate to ammonium by a Kjeldahl reaction, and then converting NH₄⁺ into N₂ by using any of the above methods. To collect nitrate the water sample is passing through anion exchanging column, then eluted from the columns with HCl, converted into AgNO₃, freeze-dried to obtain solid AgNO₃, combusted to N₂ in sealed quartz tubes for δ¹⁵N analysis. This method is suitable for fresh water [34].

The first reliable method for δ¹⁸O analysis involves combustion with Hg(CN)₂ at 550 °C. But quantitative recovery of oxygen from nitrate as free CO₂ has not been achieved. Additionally very toxic Hg(CN)₂ poses serious environmental hazards and mercury gets through vacuum system. This problem has been solved by replacing mercury cyanide with argentum cyanide [29]. WASSENAAR [38] and also SILVA et al. [34] analysed δ¹⁸O in silver nitrate which was prepared by being combusted with graphite at 900 °C. KORNEXL et al. [20] also prepared potassium nitrate by its combustion with graphite at 1400 °C. These methods reported oxygen yield less than 100%, and N isotope analyses of nitrate samples combusted for O isotope analyses have not been precise. So the next procedure was improved by combustion with the catalysed graphite (C+Pd+Au). Quantitative recovery of N and O from NO₃⁻ as free CO₂, K₂CO₃ and N₂ can approximately be represented by the following reaction:



Stoichiometric conversion of KNO₃ requires a C/KNO₃ molar ratio of 1.25, but

experimental data indicate that the conversion is not stoichiometric and excess graphite is advantageous for improving both O and N yields [31].

A new method for collection of nitrate from fresh water and the analysis of isotope ratios of nitrogen and oxygen was developed by SILVA et al. [35]. The benefits of the technique are as follows: no need to transport large volumes of water to laboratory, no need for hazardous chemicals and the ability to concentrate nitrate from fresh waters. Experimental results indicate that the method is most applicable to fresh waters with moderate to high nitrate concentration, although it may be modified for lower concentrations [9]. The greatest obstacle to $\delta^{18}\text{O}-\text{NO}_3^-$ analysis is the presence of other oxygen-bearing substances, particularly DOC (dissolve organic carbon) in the water sample.

Equilibrating CO_2 with the water sample is the most common method for measuring $^{18}\text{O}/^{16}\text{O}$ in water. There are different combustion techniques: low-temperature graphite off-line combustion method, high-temperature method, on-line carbon method, silica additive experiments with low and high temperature, graphite method and pure CO_2 gas/quartz glass isotope-exchange experiment [30]. There are other methods which are in an experimental stage or specifically applicable to certain problems:

- water is converted quantitatively into O_2 by fluorination with bromine pentafluoride [27],
- water reacts in nickel tubes with graphite at high temperature to produce CO_2 [12],
- O_2 can be produced by electrolysis of water [23],
- measurement of isotope ratios directly by laser absorption spectroscopy [19].

3. REFERENCE MATERIALS

Despite a rapid growth in the applications of analytical methods there is evidence that the calibration of isotope measurements in nitrate is not reliable. For this reason, large aliquots of nitrate salts similar to the ones used in studies have been prepared for distribution as isotope reference materials.

These reference materials are available from the National Institute of Standards and Technology (NIST) in the USA and from the International Atomic Energy Agency (IAEA) in Vienna [15]. International nitrate reference material with a wide range of $\delta^{18}\text{O}$ values gives very precise comparison of the data obtained in different laboratories. At present these are nitrate salts known as USGS-34 and USGS-35. Previous reference materials with a narrow range of $\delta^{18}\text{O}$ values have the USGS-32 or IAEA-N3 (IAEA-NO-3) symbol. USGS-34 means KNO_3 being prepared by equilibrating HNO_3 with Antarctic Snow-melt Water, and USGS-35 is commercially purified NaNO_3 from the natural nitrate ore deposits of the Atacama Desert, Chile. USGS-

34 (KNO_3) has low $\delta^{18}\text{O}$ values (from ca -50 to -28%), and USGS-35 (NaNO_3) has higher $\delta^{18}\text{O}$ values (from ca $+36$ to $+52\%$). USGS-32 is also known as RM 8558 (NIST – the National Institute of Standards and Technology, USA). IAEA-NO-3 has a $\delta^{18}\text{O}$ value ranging from $+22$ to $+26\%$. These values are similar to that of atmospheric O_2 ($+23.8\%$) [4], [30], [38].

The $\delta^{15}\text{N}$ values for USGS-34, USGS-35 and IAEA-N3 are similar to that of atmospheric nitrogen. $\delta^{15}\text{N}$ values of both new materials are similar to the values commonly reported for normal artificial nitrate reagents and also to those of atmospheric NO_3^- and N_2 .

Interlaboratory comparison based on these standards will be useful for determining discrepancies among the techniques. Thus local calibration and constant experimental conditions are necessary for both accuracy and precision.

4. SUMMARY

Of the methods for determining isotopic ratios in nitrate presented above, combustion techniques to final N_2 and CO_2 products are the most common methods. Depending on analytical possibilities and sample character, these techniques are subjected to different modifications. So it is very difficult to choose unambiguously the best method. These modifications carried in order to satisfy various needs allow dangerous reagents or onerous elements of work to be eliminated.

Nitrate sources in the rivers in south-east Poland will be studied in the Department of Engineering and Environmental Chemistry of Rzeszów University of Technology. Nitrates from different sources have different isotopic composition of oxygen and nitrogen, so they have different $\delta^{18}\text{O}$ and $\delta^{15}\text{N}$ values. Both isotopic ratios will be determined using IRMS. The most suitable technique seems to be combustion with CaO to complete water and CO_2 removing [18]. After nitrogen selection and precipitation as AgNO_3 it will be converted into N_2 in elemental analyser coupled with IRMS. Oxygen after further thermal transformation into CO_2 will be analysed with a gas chromatograph also coupled with IRMS.

The nitrogen isotopic ratio ($\delta^{15}\text{N}$) has been used extensively as an indicator of the source of nitrates in the hydrosphere and as a measure of the degree of isotopic fractionation caused by chemical transformations such as denitrification process. The $\delta^{18}\text{O}$ value of NO_3^- has the potential to resolve some of the ambiguities presented by $\delta^{15}\text{N}$ data, because some sources of oxygen in nitrate are isotopically distinctive. The analysis of $\delta^{18}\text{O}$ in combination with $\delta^{15}\text{N}$ provides additional information about nitrates in water and soil, specifically about the relative contributions of fertilizers and manure/septic waste. Because nitrate origin determined by isotopic composition of oxygen atoms and $\delta^{18}\text{O}$ value is strongly correlated with denitrification process – the in-

terpretation of isotopic ratio both N and O gives more complete, but not fully unambiguous view of the changes in the environment.

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STABILNE IZOTOPY TLENU I AZOTU W IDENTYFIKACJI AZOTANÓW. TECHNIKI POMIAROWE

Problem zwiększającego się wciąż zanieczyszczenia wód azotanami staje coraz poważniejszy. Za pomocą analizy stosunków izotopowych tlenu i azotu można rozróżnić źródła pochodzenia azotanów. Praca obejmuje przegląd metod analizy oraz ich zastosowanie w badaniach środowiskowych. Uwzględnia zarówno nowe materiały odniesienia, jak i stosowane wcześniej. Wykazano, że ze względu na różnorodność stosowanych technik właściwa kalibracja umożliwia porównywanie wyników uzyskiwanych w różnych laboratoriach. Wybrano metodę IRMS, która wydaje się właściwa do naszych badań izotopowych azotanów prowadzonych w rzekach Polski południowo-wschodniej.