Stable isotope amount ratio analysis by using high-resolution continuum source graphite furnace molecular absorption spectrometry

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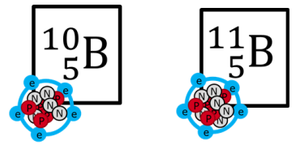
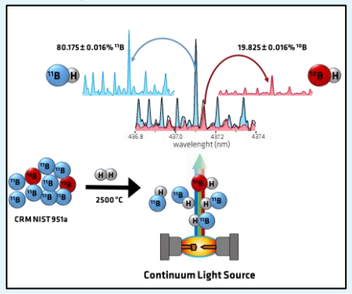


Motivation: variations in the boron and magnesium isotope amount ratio are used as proof of provenance, study of geological processes and for boron, control/monitoring of neutron radiation in nuclear plants.

Up to now, isotope analysis is a field dominated by mass spectrometry (MS) which is expensive, time consuming and requires a highly qualified operator.

However, the recently introduction of high resolution continuum source molecular absorption spectrometry (HR-CS MAS) provides a new way for the determination of non-metals and now isotope amount ratios. [1]



Approach: transient diatomic molecules like boron monohydride (BH) and magnesium monohydride (MgH) present a large isotopic shift (Δν) in their molecular absorption spectra and it is proportional to their reduced mass (μ) (Fig.1). The resulting molecular spectrum is a linear combination of the isotopologues spectra ^{10}BH and ^{11}BH for boron and ^{24}MgH, ^{25}MgH and ^{26}MgH for magnesium . [2,3]

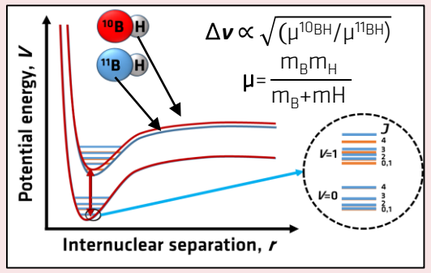


Fig. 1. Energy diagram for the first BH electronic transition $X^1\Sigma^+ \rightarrow A^1\Pi$

By using HR-CS-MAS, isotopic shifts in the BH and MgH molecules can be measured to deconvolute its components (isotopes) at the two most sensitive wavelengths: 433 nm and 437 nm for BH and 513.45 nm for MgH (Fig. 2).

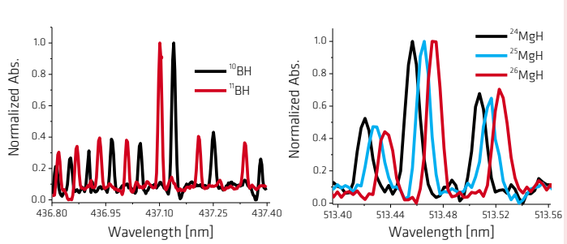


Fig.2. (Right) Comparison of the average spectra of ^{11}BH (^{11}B relative isotope abundance 99 %) and ^{10}BH (^{10}B 98 %) and (left) Comparison of the average spectra of ^{24}MgH (^{24}Mg 99 %), ^{25}MgH (^{25}Mg 99 %) and ^{26}MgH (^{26}Mg 99 %)

Methodology: molecular spectra of BH and MgH are recorded by vaporization of a sample in a graphite furnace (Fig. 3).

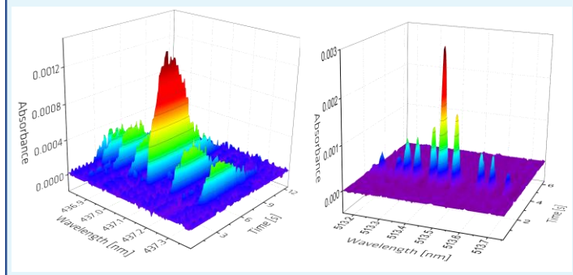


Fig. 3. (Right) time resolved spectra of the ^{11}BH molecule (^{11}B relative isotope abundance 99%) and (left) time resolved spectra of the ^{24}MgH molecule (^{24}Mg 99%)

As example, analysis of the recorded spectra for boron is schematized in Fig. 4 as follow: (1) a spectral data bank is established using enriched boric acid solutions in ^{10}B and ^{11}B for fractions of ^{11}B from 2 to 99 %. (2) Spectrum of a given sample is recorded. (3) Spectrum of sample is analyzed by partial lest square regression (PLS) using the data bank. The same procedure applies to isotope analysis of magnesium.

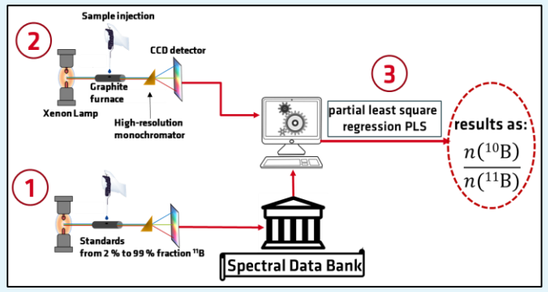


Fig.**4.** Schematic procedure for isotope analysis of boron.

Results: for exemplification; results for standard reference materials NIST SRM 951a (boric acid) and SRM 980 (magnesium) are shown in Table 1 and 2.

Table 1. Results for NIST SRM 951a

	Calculated values @ 433 nm	Calculated values @ 437 nm	Reported values (MS)
^{10}B %	19.3 ± 3.1	19.825 ± 0.016	19.827 ± 0.013
^{11}B %	80.7 ± 3.1	80.175 ± 0.016	80.173 ± 0.013
Ratio ^{10}B/^{11}B	0.240 ± 0.040	0.24728 ± 0.00010	0.2473 ± 0.0002

Table 2. Results for NIST SRM 980

	Calculated values @ 513 nm	Reported values (MS)
^{24}Mg %	78.64 ± 0.08	--
^{25}Mg %	10.20 ± 0.08	--
^{26}Mg %	11.16 ± 0.08	--
Ratio ^{26}Mg/^{24}Mg	0.14190 ± 0.00028	0.13932 ± 0.00026
Ratio ^{25}Mg/^{24}Mg	0.12976 ± 0.00026	0.12663 ± 0.00013

Conclusions: this work demonstrates the capabilities of HR-CS-MAS for the determination of isotope amount ratios of boron and magnesium by monitoring the absorption spectra of the BH molecule with high reproducibility and accuracy and compared with those obtained by mass spectrometry. Next step: to study the matrix effect in real samples.

References
[1] F.V. Nakadi *et al.*, Chlorine isotope determination via the monitoring of the AlCl molecule by high-resolution continuum source graphite furnace molecular absorption spectrometry – a case study, J. Anal. At. Spectrom., 30 (2015) 1531-1540.
[2] C. Abad *et al.*, Determination of boron isotope ratios by high-resolution continuum source molecular absorption spectrometry using graphite furnace vaporizers, Spectrochim. Acta, Part B, 136 (2017) 116-122.
[3] C. Abad *et al.*, unpublished results, 2017.

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