



Improved determination of the oxygen isotopic composition of nitrates and other nitrogen containing compounds by TC-EA-IRMS.

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On-line determination of the oxygen isotopic composition ($\delta^{18}\text{O}$ value) in organic and inorganic samples is commonly performed using a thermal conversion elemental analyzer linked to a continuous flow isotope ratio mass spectrometer (TC-EA-IRMS). Accurate $\delta^{18}\text{O}$ analysis of N-containing compounds (like nitrates) by TC-EA-IRMS may be complicated because of interference of the N_2 -peak on the m/z 30 signal of the CO peak, which is used for calculation of the $\delta^{18}\text{O}$ value. The objective of this study was to evaluate the effectiveness of different strategies to eliminate this interference.

We evaluated two strategies to eliminate the interference of the N_2 peak on the oxygen isotope ratio determination by TC-EA-IRMS. A first strategy consisted of an optimization of the operational settings of the gas chromatographic (GC) separation in the TC-EA system (GC column length, GC column temperature and He flow rate) in order to achieve better chromatographic separation of the N_2 and CO peaks. In addition to this, we evaluated the effect of an improved background correction on the CO m/z 30 sample peak integration. A second strategy consisted of (1) reducing or (2) eliminating the introduction of N_2 into the ion source. This was achieved by (1) diluting the N_2 peak with He or (2) diverting the N_2 peak to waste while providing make-up He during elution of the N_2 peak. $\delta^{18}\text{O}$ analysis results of nitrate isotope reference materials (IAEA-N3, USGS34 and USGS35) relative to VSMOW and SLAP were compared to $\delta^{18}\text{O}$ values obtained using diversion of the N_2 -peak (without interference of the N_2 -peak), in order to evaluate the effectiveness of these strategies.

Our results indicate that the observed interference could not be completely eliminated by optimizing the chromatographic separation. With an additional improved background correction on the CO m/z 30 sample peak integration, however, the $\delta^{18}\text{O}$ results obtained with optimized TC-EA operational settings were more in agreement with values obtained using N_2 -peak diversion. Based on our results we conclude that He-dilution during N_2 peak elution, in order to reduce the introduction of N_2 into the ion source, is a viable method for $\delta^{18}\text{O}$ analysis of nitrate and other N-containing samples using TC-EA-IRMS. Since activation of the He-dilution during a measurement sequence can easily be programmed in the standard software of IRMS systems, this method is an easy solution to eliminate the interference of the N_2 -peak on the m/z 30 signal of the CO peak (for samples not routinely measured using He-dilution).