Contribution ID: **279**   Type: **Poster**

QA/QC PROCEDURES FOR STABLE ISOTOPES ANALYSIS OF NITROGEN (δ15N-NO3) AND OXYGEN (δ18O-NO3) IN ENVIRONMENTAL SAMPLES AT CIRCE lab, Italy

The dual measurement of stable isotopes of nitrogen (δ15N-NO-3) and oxygen (δ18O-NO-3) in nitrates is currently used to identify sources of nitrates in environmental samples (e.g.: soil, fertilizers, groundwater, surface water, sewage, etc.). At CIRCE (Centre for Isotopic Research on Cultural and Environmental heritage, Caserta, Italy) lab, δ15N-NO-3 and δ18O-NO-3 measurements are performed by means of Temperature Conversion/Elemental Analyzer Isotope Ratio Mass Spectrometry (TC/EA-IRMS), quality controlled by means of Quality Assurance/Quality Control (QA/QC). This procedure involves data normalization through raw data calibration with δ values of Reference Materials (RM) and drift/QC sample analyses. Before analysis, nitrates are extracted from the bulk material and converted to AgNO3. The sample preparation can affect the original isotopic ratios of nitrates. In order to i) test the accuracy and the reproducibility of nitrates extraction procedure; ii) check the quality (i.e. accuracy and precision) of measurements, we currently apply a QA/QC method based on the analysis of different Reference Materials, undergoing the extraction protocol, for data normalization. The RMs must be chemically similar to the sample to simulate possible isotopic fractionations eventually occurring during preparation. International Reference Materials covering a range of δ values (USGS34: δ15N -1.8 ± 0.2‰ and δ18O -27.9 ± 0.6‰, USGS32: δ15N 180 ± 1‰ and δ18O 25.7 ± 0.4‰, USGS35: δ15N 2.7 ± 0.2‰ and δ18O 57.5 ± 0.6‰, IAEA NO3: δ15N 4.7 ± 0.2‰ and δ18O 25.6 ± 0.4‰) were used to characterize a number of Internal Standards (SIAL KNO3: δ15N 2.5 ± 0.5‰ and δ18O 24.8 ± 0.5‰, CIRCE KNO3 1: δ15N 5.3 ± 0.4‰ and δ18O 23.5 ± 0.1‰, CIRCE KNO3 3: δ15N 26.9 ± 0.8‰ and δ18O 23.8 ± 0.1‰).

In this paper machine, protocol and overall performances (e.g. accuracy and precision) based on experimental distributions of measured RM and QC datasets will be discussed.

Preliminary results show a precision of extraction protocol, determined as the standard deviation (1σ) of measures of AgNO3, equal to 0.8‰ and 0.2‰ for δ15N and δ18O, respectively. The accuracy, obtained by the comparison between the direct combustion and the extraction protocol of RMs at different δ15N and δ18O, results to be 14.3 ± 1.7% for δ15N and 4.9 ± 0.3% for δ18O.

The machine precision obtained by means of QA samples (n= 120) is 0.13‰ (mean error) for δ18O and of QA samples (n=103) 0.07‰ for δ15N.

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