STABLE ISOTOPES δ^{13}C & δ^{15}N IN SOILS & SOLIDS BY CONTINUOUS FLOW ISOTOPE RATIO MASS SPECTROMETRY

1. Principle – This method describes the determination of Delta ^{15}N vs Air and/or Delta ^{13}C vs VPDB in soil, sediments, plant, or biologic material by flash combustion. There are two naturally occurring stable isotopes of both nitrogen - ^{14}N (99.634%) and ^{15}N (0.366%), and carbon ^{12}C (98.89%) and ^{13}C (1.11%). An aliquot of sample is combusted under oxygen, and the carbon and nitrogen present in the sample is converted to CO\textsubscript{2} and N\textsubscript{2}. CO\textsubscript{2} and N\textsubscript{2} are separated chromatographically, then analyzed by continuous flow IRMS. Intensities of mass 46/45/44 for CO\textsubscript{2} and mass 28/29/30 for Nitrogen are measured. Working standards are calibrated against the International Reference scale (i.e. ^{13}C vs. VPDB and ^{15}N vs. Air), either determined in-lab or obtained (certified) commercially. Raw data from the mass spectrometer is then referenced to VPDB or Air using a linear regression calculated from the working standard results.


3. References:


   3.5 Thermo Scientific Delta V Advantage IRMS User Manual

4. Standards Used:
   4.1 Primary Calibration Standards: For ^{13}C: NBS22 (-29.7 δ^{13}C\textsubscript{VPDB}), LSVEC (-47 δ^{13}C\textsubscript{VPDB}), and NBS19 (+1.95 δ^{13}C\textsubscript{VPDB}), purchased from NIST. For ^{15}N: IAEA-N1 (+0.4 δ^{15}N\textsubscript{AIR}), IAEA-N2 (+20.3 δ^{15}N\textsubscript{AIR}), IAEA-N3 (+2 δ^{15}N\textsubscript{AIR}), purchased from NIST. These standards are periodically used to check laboratory working standards.

   4.2 Laboratory Working Standards: Protein (-26.98 δ^{13}C\textsubscript{VPDB}/+5.94 δ^{15}N\textsubscript{AIR}), Wheat Flour (-27.21 δ^{13}C\textsubscript{VPDB}/+2.85 δ^{15}N\textsubscript{AIR}), Sorghum Flour (-13.68 δ^{13}C\textsubscript{VPDB}/+1.58 δ^{15}N\textsubscript{AIR}),& High Organic Soil (-26.27 δ^{13}C\textsubscript{VPDB}/+4.42 δ^{15}N\textsubscript{AIR}), Urea (-48.63 δ^{13}C\textsubscript{VPDB}/+0.3 δ^{15}N\textsubscript{AIR}), EMPA-P2 (-28.19 δ^{13}C\textsubscript{VPDB}/+1.57 δ^{15}N\textsubscript{AIR}). This list of internal standards is representative of those used day-to-day; please contact the lab for current certified reference values.