

## STABLE ISOTOPES $\delta^{13}\text{C}$ & $\delta^{15}\text{N}$ IN SOILS & SOLIDS BY CONTINUOUS FLOW ISOTOPE RATIO MASS SPECTROMETRY

- 1. Principle** – This method describes the determination of Delta  $^{15}\text{N}$  v Air and/or Delta  $^{13}\text{C}$  v VPDB in soil, sediments, plant, or biologic material by flash combustion. There are two naturally occurring stable isotopes of both nitrogen -  $^{14}\text{N}$  (99.634%) and  $^{15}\text{N}$  (0.366%), and carbon  $^{12}\text{C}$  (98.89%) and  $^{13}\text{C}$  (1.11%). An aliquot of sample is combusted under oxygen, and the carbon and nitrogen present in the sample is converted to  $\text{CO}_2$  and  $\text{N}_2$ .  $\text{CO}_2$  and  $\text{N}_2$  are separated chromatographically, then analyzed by continuous flow IRMS. Intensities of mass 46/45/44 for  $\text{CO}_2$  and mass 28/29/30 for Nitrogen are measured. Working standards are calibrated against the International Reference scale (i.e.  $^{13}\text{C}$  vs. VPDB and  $^{15}\text{N}$  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.
- 2. Instrument Used:** Thermo Delta V Advantage Isotope Ratio Mass Spectrometer (IRMS). Thermo Scientific Inc., Bremen, Germany 2016.  
**Instrument Used:** Thermo FLASH HT Plus 2000 Organic Elemental Analyzer, Thermo Fisher Scientific Inc., Milan, Italy, 2016.  
**Instrument Used:** ConFlo IV (for CF-IRMS), Thermo Scientific Inc., Bremen, Germany 2016.
- 3. References:**
  - 3.1** Official Methods of Analysis of AOAC International, 17<sup>th</sup> Edition (2000), AOAC International, Arlington, VA. Method 972.43, Micro-chemical Determination of Carbon, Hydrogen, and Nitrogen, Automated Method.
  - 3.2** Methods of Soil Analysis, Part 3 – Chemical Methods, Soil Science Society of America Book Series #5, Soil Science Society of America, Inc., Madison, Wisconsin. Dumas Methods, Determination of Total Nitrogen and Total Carbon by Combustion.
  - 3.3** ECS 4010 Elemental Combustion System CHNS-O Operating Manual, Costech Analytical Technologies Inc., Valencia, CA.
  - 3.4** P.A. de Groot, Handbook of Stable Isotopic Analytical Techniques, Volume 1, Elsevier, 2004, ISBN:0 444 511148
  - 3.5** Thermo Scientific Delta V Advantage IRMS User Manual
- 4. Standards Used:**
  - 4.1 Primary Calibration Standards:** For  $^{13}\text{C}$ : NBS22 ( $-29.7 \delta^{13}\text{C}_{\text{VPDB}}$ ), LSVEC ( $-47 \delta^{13}\text{C}_{\text{VPDB}}$ ), and NBS19 ( $+1.95 \delta^{13}\text{C}_{\text{VPDB}}$ ), purchased from NIST. For  $^{15}\text{N}$ : IAEA-N1 ( $+0.4 \delta^{15}\text{N}_{\text{AIR}}$ ), IAEA-N2 ( $+20.3 \delta^{15}\text{N}_{\text{AIR}}$ ), IAEA-N3 ( $+2 \delta^{15}\text{N}_{\text{AIR}}$ ), purchased from NIST. These standards are periodically used to check laboratory working standards.
  - 4.2 Laboratory Working Standards:** Protein ( $-26.98 \delta^{13}\text{C}_{\text{VPDB}}/+5.94 \delta^{15}\text{N}_{\text{AIR}}$ ) Wheat Flour ( $-27.21 \delta^{13}\text{C}_{\text{VPDB}}/+2.85 \delta^{15}\text{N}_{\text{AIR}}$ ), Sorghum Flour ( $-13.68 \delta^{13}\text{C}_{\text{VPDB}}/+1.58 \delta^{15}\text{N}_{\text{AIR}}$ ), & High Organic Soil ( $-26.27 \delta^{13}\text{C}_{\text{VPDB}}/+4.42 \delta^{15}\text{N}_{\text{AIR}}$ ), Urea ( $-48.63 \delta^{13}\text{C}_{\text{VPDB}}/+0.3 \delta^{15}\text{N}_{\text{AIR}}$ ), EMPA-P2 ( $-28.19 \delta^{13}\text{C}_{\text{VPDB}}/+1.57 \delta^{15}\text{N}_{\text{AIR}}$ ). This list of internal standards is representative of those used day-to-day; please contact the lab for current certified reference values.