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University of California, Santa Cruz **March 9, 2018**
Stable Isotope Laboratory

Standard Operating Procedures: For analyzing marine sediments and tissues for carbon and nitrogen isotopic analysis and weight percent determination.

Sample preparation:
Untreated samples, both tissue and sediments, are frozen overnight prior to freeze dried in a Labconco lyophylizer. Dried samples are then ground and homogenized using a agate mortar and pestle. If the sediments contain inorganic carbon then the sediments are acidified using the sulfurous acid procedure of Verado et al. 1990. Samples requiring acidification are pre-weighed into silver capsules prior to de-carbonation.

Samples are then weighed into tin capsules (Costech Analytical 5x9) so that the target 50 µg nitrogen is reached based on the nitrogen content of the sample. For acidified samples, a goal of 50 µg carbon yield dictates the target sediment sample mass.

Analysis:
Solid materials are analyzed for $\delta^{15}N$ and $\delta^{13}C$ isotopes using a Carlo Erba 1108 elemental analyzer interfaced to a ThermoFinningan Delta Plus XP isotope ratio mass spectrometer (IRMS). Tin (or silver) encapsulated samples are flash (Dumas) combusted at 960°C in a quartz column containing chromium oxide (acting as an oxygen source to aid combustion) and silvered colbatous/cobaltic oxide (acting as a scrubber to "clean" the combustion products of sulfur bearing compounds and halides). Following combustion, excess oxygen and oxides of nitrogen are reduced in a reduction column (reduced copper at 650°C). Helium carrier flows through a water trap containing magnesium perchlorate. N_2 and CO_2 are separated on a Carbosieve GC column (45°C, 100 mL/min) before introduction to the IRMS.

Quality Control/Assurance:
During analysis, internationally calibrated in-house standards precede and are interspersed between samples to correct for linearity (size) effects and drift. A second calibrated laboratory standard is run "as-a-sample" to monitor quality control and long-term performance. These laboratory standards, which are selected to be compositionally similar to the samples being analyzed, have been previously calibrated against National Institute of Standard and Technology (NIST) and International Atomic Energy Agency (IAES) Standard and Reference Materials (IAEA-N2, IAEA-N3, IAEA-USGS25, and IAEA-USGS26 of $\delta^{15}N$ and IAEA-CH7, and NBS-22 and IAEA-USGS25 for $\delta^{13}C$). The isotopic values of the international $\delta^{15}N$ and $\delta^{13}C$ standards are taken from the Commission on Isotopic Abundances and Atomic Weights (ciaaw.org) published values. Corrected delta values, $\delta^{13}C$ and $\delta^{15}N$, are expressed relative to international standards VPDB (Vienna PeeDee Belemnite) for $\delta^{13}C$ and Air for $\delta^{15}N$. Long term $\delta^{13}C$ and $\delta^{15}N$ precision and accuracy of our quality control standards are better than 0.2 ‰. The lab adheres to the guidelines of Carter and Barwick (2011).

Verado et al. Determination of Organic-Carbon and Nitrogen in Marine-Sediments using the Carlo-Erba-NA-1500 Analyzer, Deep-Sea Research Part a-Oceanographic Research Papers (1990) vol. 37 pp. 157-165

J. F. Carter and V. J. Barwick (Eds), Good practice guide for isotope ratio mass spectrometry, FIRMS (2011). ISBN 978-0-948926-31-0