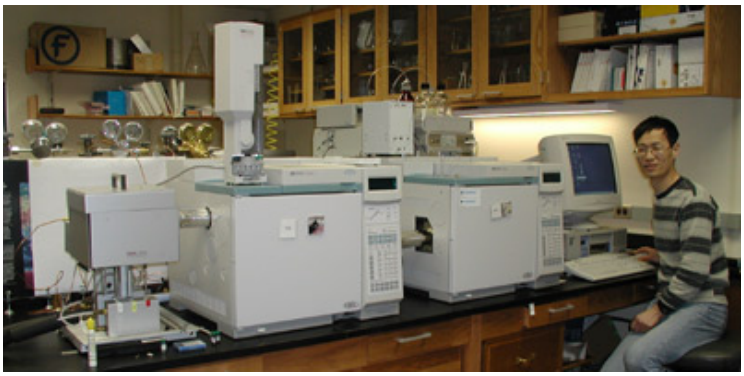
Print

NOSAMS: Associated Instruments

Overview

The NOSAMS facility provides proven designs for sample-preparation systems and our staff have trained many students, technicians and investigators to use them. In some cases we work with investigators to evolve procedures tailored to their needs. However, because of limitations imposed by working space, we sometimes have limited ability to welcome outside investigators into our laboratories.

Visitors have used the facility's chromatographic systems to prepare samples for compound-specific analyses. Those with large numbers of samples can be offered favorable rates and arrangements for preparing their own samples at NOSAMS.



Dr. Li Xu, a specialist skilled in the preparative separation of organic compounds, shown here operating the two-dimensional PCGC in 2004.

Preparative capillary gas-liquid chromatography (PCGC)

With the advent of AMS, and the additional efforts at NOSAMS to greatly reduce sample size requirements, PCGC technology has allowed the development of compound-specific radiocarbon analysis (CSRA). This technique in which natural-abundance levels of ^{14}C are determined in specific organic compounds was developed by Dr. Timothy Eglinton and his coworkers at WHOI. The PCGC allows recognition and resolution of sources of compounds, providing information about processes within the biogeochemical cycle of carbon and about paleoenvironmental conditions that was unavailable using previous techniques.

Two-dimensional PCGC

A multi-dimensional PCGC system was installed at NOSAMS in December 1998. This more precise comprehensive two-dimensional gas chromatography is capable of separating one order of magnitude more compounds than the traditional gas chromatography method. The system consists of two HP6890 gas chromatographs (GC) in series with a Gerstel preparative fraction collector attached at the end. Sample extracts containing suites of very similar molecules, e.g., a homologous series of n-alkanes, are injected repeatedly onto a high-capacity megabore capillary column in the first GC to separate and resolve individual molecules. In instances where separations are difficult to effect with one GC, targeted compounds or regions of the chromatogram are isolated and re-injected into the second GC. Finally, up to six specified peaks are isolated in the preparative fraction collector. Even with the power of capillary GC resolution and the high capacity of megabore columns, it is still difficult to collect enough sample for a radiocarbon measurement. Typical runs for up to a week often yield only 20-100 µg C samples. This system provides NOSAMS with a unique and powerful means of isolating and measuring the radiocarbon content of individual organic compounds isolated from complex natural matrices, e.g., sediments and soils. The ability to repeatedly inject sample extracts, collect target compounds, and analyze microgram-sized samples has made compound-specific radiocarbon analyses a reality. The addition of a second GC further improves the resolution, and therefore, the power of the technique.

HPLC - High-performance liquid chromatography

Sean Sylva prepares to use the High Peformance Liquid Chromatograph (HPLC) to determine which organic compounds from an *Archaea* bacteria sample (collected at a Lost City hydrothermal vent) can be isolated and analyzed for ^{14}C.

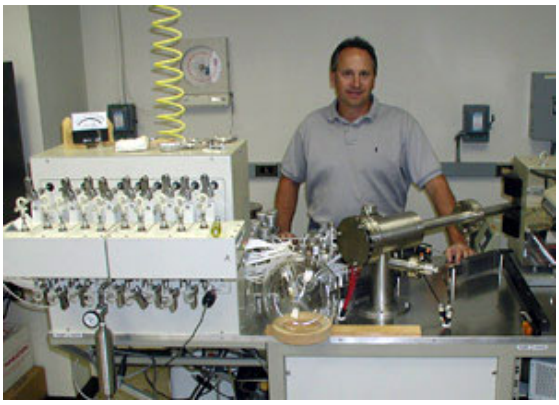
A liquid-chromatographic system was installed at NOSAMS in 2003 to supplement the capabilities of the preparative capillary gas chromatographic system. This instrument is capable of separation, identification, purification, and quantification of various compounds and is not limited by the volatility or stability of the sample compound.

Elemental analyzer

(> 1% C, > 25 µmol)

To improve processing of samples for total organic carbon (TOC) ^{14}C analysis, we built an automated system for the collection of CO_2 gas generated by the combustion of organic carbon samples. It is based on a commercial Carlo Erba NA1500 elemental analyzer and in many cases takes the place of our standard closed-tube combustion technique. CO_2 produced during sample combustion in the elemental analyzer is separated from the carrier gas and cryogenically trapped. The gas (sample size approximately 500 µg C yield) is transferred

cryogenically to a calibrated volume in vacuo, quantified, and stored in a modular gas collection manifold. These manifolds are removed and re-connected to the automated graphite reactor vacuum systems.



Al Gagnon operates and maintains The PRISM and Optima mass spectrometers.

Optima Stable Mass Spectrometer

The Optima is the primary stable mass spectrometer used for analyzing a split of CO_2 in-line as a a sample is sent through the vacuum system to a reactor for reduction to graphite.

VG Prism Stable Mass Spectrometer

A PRISM Series II system provides auxilary capacity for automated high-precision analysis of the stable isotopes of ^{13}C and ^{18}O in carbon dioxide in association with radiocarbon analysis.

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