Facility Overview

The Northeast National Ion Microprobe Facility (NENIMF) was established at Woods Hole Oceanographic Institution by Nobumichi Shimizu in 1996. It is a multi-user facility specializing in Secondary Ionization Mass Spectrometry (SIMS), a powerful micro-analytical technique with broad applications to both earth and ocean sciences. The NENIMF specializes in in situ determinations of trace element abundances in minerals, glasses, biogenic carbonates and oxides, as well as industrial materials. We also carry out in situ determination of isotopic compositions of H, B, C, O, S, Cl, Pb in geological materials. The centerpiece of the facility is the Cameca IMS 1280, a new generation, high transmission-high mass resolution SIMS instrument, the latest version of a double focusing mass spectrometer with a large radius magnetic sector (585 mm), produced by Cameca Instruments. It is one of only five such instruments in the United States. The ion optics of the IMS 1280 are optimized to attain a mass resolving power (MRP = M/∆M, where M is the observed mass and ∆M is the difference between 2 masses that can be separated) of 6,000 without significant loss of secondary ion intensity. It is useful for chemical and isotopic analysis of wide varieties of materials. The instrument is flexible in that both the energy filtering and high-resolution approaches can be used, dependent on where analytical advantages lie. The NENIMF also operates a Cameca IMS 3f that has been used since 1978 for a wide spectrum of geochemical studies, and remains highly effective for in situ trace element analysis of rock-forming minerals and glasses (spatial resolution of 10 μm or less). Some technical aspects of the NENIMF operation are illustrated in this handout.

Management of the NENIMF is overseen by a steering committee, chaired by G. Gaetani, that is responsible for all financial and scheduling decisions, and is dedicated to outreach and to increasing the NENIMF user base. Nobu Shimizu, a pioneer in SIMS techniques, is the Chief Scientific Advisor of the NENIMF, consulting on all technical aspects of the facility. The steering committee works closely with an external advisory committee, chaired by D. Ebel of the American Museum of Natural History.

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NENIMF offers Secondary Ionization Mass Spectrometry (SIMS) for both earth and ocean sciences, specializing in in situ determinations of trace element abundances in minerals, glasses, biogenic carbonates and oxides, as well as industrial materials.

NENIMF Fee Structure
(for government funded projects)

<table>
<thead>
<tr>
<th>Instrument</th>
<th>Hourly Rate</th>
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<tbody>
<tr>
<td>IMS 1280</td>
<td>$150/hr</td>
</tr>
<tr>
<td>IMS 3f</td>
<td>$75/hr</td>
</tr>
</tbody>
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Weekends & holidays discounted 15%.
Non-federally funded projects, including foreign funding, are subject to a 15% administrative fee.
See website for non-academic user rates. The facility also offers analytical services (i.e., fixed per-sample measurements) with a limited scope and a different fee structure.

https://www2.whoi.edu/site/nenimf/
The analytical capabilities and expertise of the NENIMF are focused in 2 areas: determination of magmatic volatiles in silicate glasses and analysis of biogenic carbonates as recorders of climate change and its impacts on marine organisms. Analytical protocols and the requisite standard materials are available for determination of H₂O, CO₂, F, S, and Cl in silicate glasses and of B, Mg, Sr, Ba, and U in carbonates. There are many additional analytical protocols and applications available. The emphasis on these areas makes NENIMF a unique interdisciplinary resource to the geosciences community, with minimal overlap to other ion microprobe facilities.

**Determination of Magmatic Volatiles in Silicate Glasses**

Magmatic volatiles play a key role in driving volcanism at plate margins and in facilitating mass transfer between the mantle and its exospheric reservoirs, and thus are of fundamental importance to earth and ocean sciences. SIMS techniques have a number of advantages over Fourier Transform Infrared spectroscopy (FTIR) and electron microprobe techniques for measuring volatiles. First, the spot size required for ion microprobe analyses is significantly smaller than for FTIR measurements. Second, ion microprobes have a higher sensitivity, as compared to electron microprobes, for measuring F and Cl at low concentrations. Finally, unlike other techniques, SIMS allows acquisition of all analyses in a single analysis. The NENIMF is the only open, multi-user facility in the eastern United States that presently offers this analytical capability.

The analytical protocol developed for the IMS 1280 use the Cs⁺ primary ion beam. The concentration of H₂O is determined by measuring O/H⁺, rather than H++. The H⁺ ion trajectory in the secondary ion optics of the large-radius IMS 1280 is very different from higher-mass ions, so that measuring the O/H⁺ ion is more convenient. An MRPC of 5,500 is routinely used for separation of O/H⁺ from 16O1H (MRP required = 4711). At such an MRPC, all molecular ion interferences are completely separated: 29Si18O* denotes two SiO molecular ions, 30Si19O from 16O1H (MRP required = 4711). At such an MRP, all molecular ion interferences are sufficiently suppressed so that measured mass ratios are analyzed with a negatively charged oxygen ion (O⁻) with a current of ~15 nA focused to a spot ~20μm in diameter. With an energy offset of ~60V, molecular ion interferences are sufficiently suppressed so that the measured 87Sr/86Sr ratio is identical to the natural abundance ratio (0.1194) within counting statistic uncertainties. The energy filtering-based approach has since been transferred to the IMS 1280, which has a greater sensitivity relative to the 3f. Working curves for these ratios are determined based on three carbonate standard samples (OKA carbonatite, 0785 “Blue” calcite, and AG-1 aragonite).

**Biogenic Carbonate Records of Climate Change and Related Impacts on Marine Organisms**

The second key focus area for NENIMF analytical capabilities and expertise is the analysis of biogenic carbonates as recorders of climate change and related impacts on marine organisms. Much of what we know about past climate variability and the role of the oceans in climate change on decadal through orbital timescales, comes from proxy data contained within the carbonate skeletons of marine organisms. Nevertheless, there are fundamental questions concerning the detailed interpretation of geochemical proxies, the role of diagenesis in distorting the climate signals and the impacts of climate change on marine organisms. Many of these questions can only be addressed with the unparalleled spatial resolution and precision afforded by SIMS. Since its first application to the fine-scale analysis of biogenic carbonates, SIMS techniques have played a key role in shaping our understanding of the relationships between skeletal composition and environmental variables and the factors that distort them (so-called “vital effects”).

Trace element analysis in marine carbonates began at NENIMF more than 15 years ago, using the IMS 3f with the energy filtering approach. Mg/Ca, Sr/Ca, and Ba/Ca ratios are analyzed with a negatively charged oxygen ion (O⁻) with a current of ~15 nA focused to a spot ~20μm in diameter. With an energy offset of ~60V, molecular ion interferences are sufficiently suppressed so that the measured 87Sr/86Sr ratio is identical to the natural abundance ratio (0.1194) within counting statistic uncertainties. The energy filtering-based approach has since been transferred to the IMS 1280, which has a greater sensitivity relative to the 3f. Working curves for these ratios are determined based on three carbonate standard samples (OKA carbonatite, 0785 “Blue” calcite, and AG-1 aragonite).