

This plan is presented to comply with NSF policy and directions on the dissemination and sharing of research results as described in Grant Proposal Guide (GPG) Chap. II.C.2.j.

Data Policy Compliance

Our research into the incorporation of Fe into biogenic silica will generate multiple complementary data sets, including (a) dissolved trace metal concentrations, (b) labile and total particulate trace element concentrations, (c) single-cell trace element concentrations, and (d) single-cell trace element XANES spectra.

Sample types

Sample archive management: The samples to be analyzed are archived from the 2011 CLIVAR S04P Southern Ocean cruise. Log sheets were completed shipboard for each station and included relevant sampling details such as location, depth, and sample volumes collected for each analyte and type (e.g., dissolved or particulate).

Dissolved Trace Metal samples: water column samples from 12 depths between 0 m and 1400 m were collected approximately every 2° along the cruise transect. Dissolved samples are operationally defined as water that passes through a 0.4 µm Nuclepore PCTE filter. Elemental concentrations will be determined by HR-ICP-MS analyses (Thermo ELEMENT 2) and quantified by isotope dilution (for Fe, Cd, Cu, Ni, Zn, and Pb) or standard additions (for Mn and Co).

Dissolved concentrations will be reported in nmol/L or pmol/L. **Blank concentrations** (as determined by similarly extracting and analyzing acidified UHP water) will be determined for each batch of extractions and reported for each element. Procedural validation will be assessed using GEOTRACES consensus **reference materials** (e.g., SAFe S and D2, GEOTRACES GS and GD). These concentrations will be included in the data report to BCO-DMO and also submitted to Dr. Jim Moffett (U. Southern California) and the GEOTRACES Standards and Intercalibration Committee for ongoing updates of the reference material values.

Particulate Trace Metal samples: Particles greater than 0.4-µm that were collected on the PCTE filters were stored cleanly in petri dishes dry and at room temperature until processing and analyses. Filter volumes were recorded in the shipboard log sheets and Excel database.

Particulate samples will be processed by four different methods, and will be reported with figures of merit. **Total elemental concentrations** will be determined using a total digest (HNO₃, HCl, and HF; Ohnemus et al., 2014), and **labile elemental concentrations** will be determined using an ammonium acetate-hydroxylamine hydrochloride leach (Berger et al., 2008). Biogenic silica (bSi) will be determined using a base dissolution and fluorometric detection (Krause et al., 2009). **Single-cell elemental concentrations** of Fe, Si, and other trace elements will be determined by Synchrotron X-Ray Fluorescence (SXRF).

Concentration information will be reported in nmol/L or pmol/L. **Reagent blank** concentrations will be determined for each round of digestions and reported for each element. In addition, **filter blanks** (acid washed but unused) will be processed like marine particulate samples. Procedural validation will be assessed using multiple **reference materials**: PACS-2 sediments (NRCC), BCR-414 plankton (JRC, EU), and a consensus “ATD aerosol dust” reference material (Powder Technologies, Inc.). These concentrations will be included in the data report to BCO-DMO. The single-cell SXRF concentrations will be quantified using geologic minerals and powdered standards (Micromatter Technologies, Inc.).

X-ray fluorescence data files are processed and interpreted using free, publically available software (e.g. MAPS and ATHENA). Key analytical parameters utilized to acquire X-ray fluorescence data (e.g., dwell times, X-ray optics configuration, detectors, etc.) will be recorded in laboratory notebooks and then organized into searchable electronic spreadsheets. We will upload all data (X-ray fluorescence data and

analytical spreadsheets, as well as spreadsheets) to the above database with notes on how and where to download the MAPS and ATHENA software programs.

Data Management

We will conform to the metadata standards established by the CLIVAR and Carbon Hydrographic Data Office (<https://cchdo.ucsd.edu/>) and the Biological and Chemical Oceanography Data Management Office (BCO-DMO). Shipboard data was organized by the Scripps Institution of Oceanography Ocean Data Facility in accordance with these formats. As much as possible, trace element concentration data will be archived in the same ASCII format (comma-delimited TXT files) to conform to existing CCHDO data sets.

Digital storage of relevant sampling parameters: Relevant sampling information was transferred from the handwritten log sheets to an Excel database and included: station latitude and longitude, date and time of sampling events, Go-Flo bottle number, targeted and actual sampling depth, subsample type (filtered or unfiltered) and volumes drawn from each Go-Flo bottle.

All digital data *in progress* will be backed up daily by the National High Magnetic Field Laboratory's (NHMFL) InSync system. This system is password protected and is a mandatory practice for all NHMFL networked computers. In addition, there is an internal network drive ("Z Drive") where data will be uploaded for long-term storage.

Data Archiving

Public Data Repositories: All dissolved and particulate data will be posted to BCO-DMO and CCHDO by the end of the project. Public access will be granted to all data sets following publication or at most two years after collection.

Sample Repositories: Dissolved and particulate samples from this study have been archived at Florida State University since their collection. Particulate samples processed and analyzed for trace element concentrations will be completely consumed, so no sample will remain for archiving. However, particulate samples that are not analyzed will remain in archive at FSU. Dissolved samples are archived in 125-mL LDPE bottles, and in general only 15-50 mL of sample are required for analysis. The remaining dissolved sample will be returned to the archive and available for sharing with other investigators upon request.

Berger, C. J. M., Lippiatt, S. M., Lawrence, M. G., and Bruland, K. W. (2008). Application of a chemical leach technique for estimating labile particulate aluminum, iron, and manganese in the Columbia River plume and coastal waters off Oregon and Washington. *J. Geophys. Res.* 113, C00B01. doi:10.1029/2007JC004703.

Krause, J. W., Nelson, D. M., and Lomas, M. W. (2009). Biogeochemical responses to late-winter storms in the Sargasso Sea, II: Increased rates of biogenic silica production and export. *Deep Sea Res. Part I Oceanogr. Res. Pap.* 56, 861–874. doi:10.1016/j.dsr.2009.01.002.

Ohnemus, D. C., Auro, M. E., Sherrell, R. M., Lagerström, M., Morton, P. L., Twining, B. S., et al. (2014). Laboratory intercomparison of marine particulate digestions including Piranha: a novel chemical method for dissolution of polyethersulfone filters. *Limnol. Oceanogr. Methods* 12, 530–547. doi:10.4319/lom.2014.12.530.