

## Environmental Chemistry Facility Instrumentation

<http://www.brown.edu/Research/Evchem>

David Murray, Director

MacMillan 114, [dmurray@brown.edu](mailto:dmurray@brown.edu), 401-863-3531

### CE Instruments NC2100 Elemental Analyzer (MacMillan 221):

analysis of carbon, nitrogen, and sulfur concentration of solid samples

Sample type: dried, homogenized, ground

Sample amount: 5-15 mg- depending on concentration

Detection: 5-10  $\mu\text{g}$  of C or N, 50-100  $\mu\text{g}$  S

Time: dry, grind, and homogenize samples, weigh out standards and samples, calibrate analyzer (9 analyses), analysis: 3.5 minutes per sample, 63 position autosampler.

Cost: \$2.50 / sample

### INNOV-X Systems Alpha 4000 Handheld XRF (in GeoChem 134, with use coordinated by David Murray):

Non-destructive elemental analysis on a variety sample types for concentrations of some metals ranging from about 20 ppm to 10%.

Sample type: most items with a surface  $\sim$ 1 cm diameter, highest precision and accuracy on dried, ground, and homogenized samples

Sample amount:  $\sim$ 1 cm diameter and 1 mm thick

Detection: 10 ppm-10% for a suite of about 20 element- some more accurate than other

Time: 2 minutes per sample,  $\sim$ 30 minutes to set up, calibrate, and to put away instrument

Cost: N/A

### Milestone DMA80 Mercury Analyzer (in Barus&Holly 031, with use coordinated by David Murray):

analysis of mercury in solid or liquid samples

Sample type: solids should be ground and homogenized

Sample amount: 50-400 mg- depending on concentration

Detection: 0.1 ng Hg

Time: prepare and weigh samples, calibrate analyzer (2-3 standards), analysis: 5 minutes per sample, 40 position autosampler.

Cost: \$2/sample

### Lachat QuikChem 8500 Flow Injection Analyzer (FIA (in Osvaldo Sala's lab with use coordinated by David Murray)- This will be replaced by a Westco Smartchem 200 Discrete Analyzer in MacMillan 103, with use coordinated by David Murray):

Analysis of Nitrate/Nitrite, Phosphate, and Ammonia in waters and chemical extracts, and silicate in water samples

Sample type: liquid

Sample amount: 5-10 ml

Detection: 0.1 ppm

Time: prepare sample and standards, calibrate analyzer, 1-3 minutes per analysis

Cost: expendable supplies, mostly chemical reagents associated with analyses

**Jobin Yvon JY2000 Inductively Coupled Plasma Atomic Emission Spectrometer (ICP-AES) MacMillan 221:**

analysis of most elements with atomic weights above 10.

Sample type: filtered liquid, either natural waters or digested soil and plant material in an acid matrix (2-10%).

Sample amount: 10 ml, 40-500 mg depending on sample preparation and concentrations of elements of interest

Detection: 10-20 ppb, note that digested soils and plants are diluted by at least 140x before analysis.

Time: dry, grind, and homogenize samples, digest samples (6 hrs/10 samples), calibrate analyzer (30 minutes), analysis: 2-5 minutes per sample depending on number of elements up to 12 elements, 176 position autosampler

Cost: \$3-\$5/sample with minimum charge of \$25.

**Perkin Elmer 4100ZL Graphite Furnace-Atomic Absorption Spectrometer (AAS-GF) (MacMillan 221):**

analysis of metals in liquids

Sample type: filtered liquid, either natural waters or digested soil and plant material in an acid matrix (2-10%)

Sample amount: 2 ml, 40-500 mg depending on sample preparation and concentrations of elements of interest

Detection: 1-4 ppb, concentrations need to be below 100 ppb

Time: dry, grind, and homogenize samples, digest samples (6 hrs/10 samples), calibrate analyzer (1 hr), analysis: 5 minutes/sample, 80 position autosampler

Cost: \$3.00/sample

**Dionex Modular DX500 Ion Chromatography/HPLC system (MacMillan 221):**

Column separation of chemical species in liquids

Detectors are conductivity, UV/Vis, and fluorescence.

Sample type: liquid, free of high concentrations of interfering components (e.g. salts, acids)

Sample amount: 2-3 ml

Detection: 0.2-0.5 ppm

Time: filter samples, calibrate analyzer (5-10 standards), analysis: 15 minutes per sample, 72 position autosampler for IC, manual injection for HPLC.

Cost: \$4-\$5/sample with additional with additional costs for interfering component removal.

**Phillips PW1480 X-ray Fluorescence Spectrometer (XRF) (MacMillan 312):**

Analysis of most elements with atomic weights above 10 (accurate for Si, Al, Fe, Mn, Mg, Ca, Na, K, Ti, Co, Cr, Cu, Ni, Pb, Sr, V, Zn).

Sample type: dried, homogenized, fine powder

Sample amount: 4-5 g

Detection: 5 ppm- requires some processing of data to get low detection

Time: dry, grind, homogenize samples, mix with binder (2 samples/10 minutes), make pellet (5 minutes/pellet), analysis: 25 minutes per sample, 30 position autosampler

Cost: \$7-\$10/sample

**Agilent Technologies 6890+ Series Gas Chromatograph (GC) with flame ionization, electron capture, or mass spectrometer detectors and Purge and Trap sample concentrator (Available in Yongsong Huang's lab with use coordinated by David Murray):**

Analysis of volatile organic compounds extracted from solid or liquid samples including SVOC's (PAH's), VOC's (MTBE), and chlorinated organic compounds (PCB's, DDT, and Dioxin)

Sample type: dried, homogenized, fine powder or organic solvent extraction from soils or water.

Sample amount: 1 g dry weight

Detection: varies depending on detector (ng to  $\mu\text{g/g}$ )

Time: Can be long lead time to schedule machine. 1-2 days of sample preparation, analysis on GC (1 day setup for nonstandard detection 1 hour per sample analysis, 6 position autosampler on GC/MS and 100 position autosampler on GC/FID, 50 position autosampler for Purge and Trap.

Cost: reimburse lab for expendable supplies, primarily gas and organic solvents

**Note-** the Purge and Trap sample concentrator used for VOC analysis was moved to Eric Suuburg's Lab (PL127) during the Summer 07 and attached to a Shimadzu 2010 GC-ECD, FID Yongsong Huang also has an Agilent Tech., 1200 LC/MS system that is used to measured organic biomarkers and contaminants. Use is arranged by Yongsong Huang and his technician,

**Sample preparation**

**Drying Ovens (MacMillan 010):** mechanical and gravity convection, dry overnight

**Drying racks (MacMillan 010):** for air-drying samples

**Large chest freezers (MacMillan 010):** two for storing samples

**Cold room (MacMillan 010):** 4°C room for storing samples

**Freeze Dryer (MacMillan 010):** used to dry samples, freeze overnight, then minimum 48 hrs to dry

**Wiley Mill (MacMillan 011B):** used to grind plant material into a fine powder

**Mixer Mill (MacMillan 011A):** used to grind soils and rock material into a fine powder

**Mortar and Pestle:** used to grind plants and soils into a coarse powder

**Milestone Ethos 1600 Microwave digestion (MacMillan 316):** used to digest solid material in concentrated acid, 6-7 hr continuous process w/2 hr break in middle, 12-sample rotor, 10 samples per 6 hrs, Cost: \$5/sample

**Dionex Advance Solvent Extraction (ASE) (Available in Yongsong Huang's lab – GeoChem 123, with use coordinated by David Murray):** used to extract organic compounds from solid samples, 3 hours load time for up to 24 samples, 40 minutes/sample for extraction, 4 hours to unload samples and clean up cells, Cost: expendable supplies and some hardware.