

Environmental Chemistry Facility Instrumentation

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

David Murray, Director
MacMillan 114, 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 μg of C or N, 50-100 μg 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: \$3.25 / sample

INNOV-X Systems Alpha 4000 and Olympus Delta Handheld XRF (MacMillan 109B, 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% (Alpha) and >10 ppm (Delta).

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

Sample amount: ~1 cm diameter and 1 mm thick

Detection: >10 ppm for a suite of elements including Pb, Zn, Cu, Cr, Mn, Fe, Zr, Sr, Ti, K, and Ca; some more accurate than others

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

Cost: N/A

Milestone DMA80 Mercury Analyzer (MacMillan 109B, 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: 7 minutes per sample, 40 position autosampler.

Cost: \$4.50/sample

Westco Smartchem 200 Discrete Analyzer (MacMillan 103, with use coordinated by David Murray and Ruby Ho):

Analysis of Nitrate/Nitrite, Phosphate, Ammonia, Silicate, and other nutrients in waters and chemical extracts

Sample type: liquid

Sample amount: 1-4 ml

Detection: 50-100 ppb

Time: prepare sample and standards, calibrate analyzer, minimum of ½ hr to calibrate and 1-3 minutes per analysis

Cost: expendable supplies, mostly chemical reagents associated with analyses

Thermo Scientific iCAP 7400 Duo 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 radial depending on element, 0.5-1 ppb axial, 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, 240 position autosampler

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

Perkin Elmer AAnalyst 600 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 (4 hrs/10-12 samples), calibrate analyzer (1 hr), analysis: 5 minutes/sample, 80 position autosampler

Cost: \$2.50/sample

Thermo Scientific X-series-2 Inductively Coupled Plasma Mass Spectrometer (GeoChem 040, use coordinated by David Murray and Soumen Mallick)

Analysis of trace elements in liquid samples

Sample type: filtered liquid, either natural waters or digested soil and plant material in an acid matrix (2-10%) - use distilled metal free acids and samples need to be low in total dissolved solids

Sample amount: 2-5 ml, 40-500 mg depending on sample preparation and concentrations of elements of interest

Detection: 0.25 ppb, concentrations need to be below 10 ppb

Time: dry, grind, and homogenize samples, digest samples (4 hrs/10-12 samples, include blanks and reference standards), calibrate analyzer (1 hr), analysis: 5 minutes/sample

Cost: \$40/hr

Dionex Modular DX500 Ion Chromatography system (MacMillan 312):

Column separation of chemical species in liquids- primarily used of anions in water samples

Detectors are conductivity and UV/Vis.

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.

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

UIC, INC Inorganic Carbon Analyzer (MacMillan 221, Use coordinated by Joe Orchardo):

Analysis of inorganic carbon in solid samples

Sample type: ground sediments, soils, or other solids

Sample amount: 10-50 mg

Detection: 5 µg

Time: dry, grind, and homogenize samples, weigh out standards and samples, 5 standards, analysis: 15-35 minutes per sample, 45 position autosampler.

Cost: \$3.50

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

Analysis of volatile organic compounds extracted from solid or liquid samples including SVOC's (PAH's) 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: 1-5 g dry weight

Detection: varies depending on detector (ng to µg/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, 100 position autosampler on GC/MS and 100 position autosampler on GC/FID)

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

SEE WEBSITE FOR OTHER ANALYTICAL EQUIPMENT

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 Ultrawave Microwave digestion (MacMillan 316): used to digest solid material in concentrated acid, 3-4 hr continuous process, 15-sample holder, 10-12 samples per run, then at least 24 hr drydown, Cost: \$4/sample

Dionex Accelerated Solvent Extractor (ASE) (Available in Yongsong Huang's lab –

GeoChem 148, with use coordinated by David Murray and Rafael Tarozo): 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.