1.c. Analytical Methods

Sample Preparation Overview

Analyses of total phosphorus and Total Kjeldahl nitrogen are done on whole water samples that include both dissolved and particulate materials (i.e. suspended solids). Analyses of the remaining nutrients are done on filtrates that have passed through a 0.45 micron membrane filter.

Pesticide analyses are completed following solid phase extraction of whole water samples. For the immunoassay procedures, whole water samples are used.

For the metals analysis, whole water samples are digested with nitric/hydrochloric acid and decanted prior to analysis.

Analytical Procedures

The specific methods currently used in the tributary loading program are shown in the adjacent table. In the early days of the program, all nutrients were done using Autoanalyzer II systems. Subsequently, we shifted to Technicon TRACCS systems for all but TP and TKN. Later, the anions were switched to Dionex Ion Chromatography.

Analytical Group	Equipment	Method Reference*
Suspended Sediment	Mettler Balance	EPA Method 160.2
Nutrients and major ions		
Total phosphorus	Technicon AAII	EPA Method 365.3
Total Kjeldahl nitrogen	Technicon AAII	EPA Method 351.2
Ammonia nitrogen	Technicon TRAACS	EPA Method 350.1
Soluble reactive phosphorus	Technicon TRAACS	EPA Method 365.3
Silica	Technicon TRAACS	EPA Method 370.1
Specific Conductance	Technicon TRAACS	EPA Method 120.1
Nitrate nitrogen	Dionex Ion Chromatograph	EPA Method 300.1
Nitrite nitrogen	Dionex Ion Chromatograph	EPA Method 300.1
Chloride	Dionex Ion Chromatograph	EPA Method 300.1
Fluoride	Dionex Ion Chromatograph	EPA Method 300.1
Sulfate	Dionex Ion Chromatograph	EPA Method 300.1
Current generation pesticides EPTC, Butylate Phorate, Simazine Atrazine, Terbufos Fonofos, Metribuzin Alachlor, Linuron Metolachlor, Chlorpyrifos Cyanazine, Pendamethalin Acetochlor	Gas Chromatography/ Mass Spectroscopy (GC/MS) using a Varian Saturn II	EPA Draft Method 507, solid phase extraction
Current generation herbicides Atrazine, Alachlor Metolachlor, Cyanazine	Immunoassay, Ohmicron RPA1 reader and tubes	Ohmicron Methods
Metals (major) Calcium, Magnesium Sodium, Potassium Strontium, Barium Aluminum, Iron Trace metals Copper, Cadmium Lead, Manganese Zinc	Varian Liberty 100 ICP, with ultrasonic nebulizer	Standard Methods for the Examination of Water and Wastewater, 17th edition, Method 3120

*Methods for Analysis of Water and Wastes, EPA 600/4-79-020, Cincinnati, OH, 1979

Some Views of the WQL's Analytical Equipment and Laboratories

Since the onset of its tributary loading programs, the WQL has used automated analytical systems for analysis of nutrients and subsequently for pesticides and metals. In the early years of the program, bookkeeping was done manually. However, relatively early in its history, the WQL switched to electronic transfer of all analytical data to its computer data base. Several components of our quality control program are also automated, either by the commercial software that accompanies the analytical equipment or by software developed within the WQL.

In December 2004, the WQL moved into laboratories on the third floor of the newly constructed Gillmor Science Hall. Previously, the laboratory had occupied space in the basement of Bareis Hall.

Varian ICP-MS used for trace metals analyses.

Technicon TRAACS Autoanalyzer for anlyses of ammonia, soluble reactive phosphorus, silica, and specific conductance.

Dionex Ion Chromatograph for nitrate, nitrite, chloride, fluoride and sulfate.

GC-MS system and autosampler for volatile organic compounds.

A general view of the suspended sediment laboratory, showing filtering racks, drying oven and balances.

Solid phase extraction filters for pesticides.

Technicon Autoanalyzer II systems are used for the analysis of total phosphorus and total Kjeldahl nitrogen (TKN).