CHAPTER 1. MATERIALS AND METHODS

COLLECTION OF WATER SAMPLES

Water samples were collected at passive treatment systems from their influent and effluent points, and, if applicable, between treatment cells within the system. Raw and acidified (2 mL of concentrated HCl) water samples were collected in 250 mL plastic bottles at each sampling point. Measurements of pH and temperature were made in the field with a calibrated Orion SA 270, SA 250 or SA 290 portable pH/ISE meter.[5] Alkalinity was measured in the field using a pH meter and an Orion Total Alkalinity Test Kit. At sites where particulates were visible in water samples, an extra sample was collected that was filtered through a 0.22-μm membrane filter before acidification. All samples were immediately placed on ice in an insulated cooler and returned to the laboratory within 36 h of collection. Samples were refrigerated at 4° C until analysis.

Substrate pore water samples were collected using a dialysis method similar to that described by Wheeler and Giller (*19*). Lengths of 6,000-8,000 molecular weight dialysis tubing were filled with 250 mL of deionized, deoxygenated water and buried 30-45 cm deep in the organic substrate of the wetland. Three weeks later, the dialysis tubes were retrieved and the contents immediately filtered through a 0.45-μm membrane filter. Laboratory experiments established that the chemistry of water within the sampling tubes equilibrated with surrounding pore water within 24 h. The 3-week equilibration period was allowed so that chemical anomalies caused by the burial process would dissipate. Portions of the filtered water samples were preserved with NaOH (for dissolved sulfide determinations), HCl (for cation analysis), or were left unpreserved (for alkalinity, acidity, and sulfate analyses).

ANALYSIS OF WATER SAMPLES

Concentrations of Fe, Mn, Al, Ca, Mg, and Na were determined in the acidified samples using Inductively Coupled Argon Plasma Spectroscopy, ICP (Instrumentation Laboratory Plasma 100 model). The acidified samples were first filtered through a 0.45-μm membrane filter to prevent clogging of the small diameter tubing in the ICP.

Ferrous iron concentrations were determined on acidified samples by the potassium dichromate method (*20*). Sulfate concentrations were determined by reaction with barium chloride (BaCl) after first passing the raw sample through a cation exchange resin. Thorin was used as the end-point indicator. Dissolved sulfide species were determined using a sulfide-specific electrode.

Acidity was determined by boiling a 50-mL raw sample with 1 mL of 30% H_2O_2 (hydrogen peroxide), and then titrating the solution with 0.1 *N* NaOH (sodium hydroxide) to pH 8.3 (*21*). Acidity and alkalinity are reported as $mg \cdot L^{-1}$ $CaCO_3$ equivalents.

ANALYTICAL QUALITY CONTROL

For each set of samples for a particular site, a duplicate, standard, and spike were analyzed for quality control purposes. The relative standard deviation for the duplicate was always at least 95%. Percent recovery for the standards were within 3% of the original standard. Spike recoveries were within 5% of the expected values.

FLOW RATE MEASUREMENTS

Mine water flow rates were determined by several methods. Whenever possible, flow was determined with a bucket and stopwatch. In all cases, three to five measurements of the time needed to collect a known volume of water were made at each sampling location, and the average flow rate of these measurements was reported. At two sites where flows were occasionally too high to measure with a bucket (the Latrobe and Piney Wetlands), 0.50 or 0.75 ft H-type flumes were installed and flows were determined from the depth of water in the flume. At the Keystone site, flows were determined by measuring the depth of water in a drainage pipe and then using the Manning formula for measurement of gravity flow in open channels (*22*).

ANALYSIS OF SURFACE DEPOSITS

The chemical composition of surface deposits collected from several constructed wetlands were determined by the following procedure. The samples were rinsed with deionized water, dried at 100° C, and weighed. The acid-soluble component was extracted by boiling 5 g of dry sample in 20 mL of concentrated HCl for 2 min. The acid extractants were filtered and analyzed for metal content by ICP Spectroscopy and for sulfate content by liquid chromatography. The acid-insoluble material was dried at 100° C and weighed. The acid-soluble component was determined by subtracting the dry weight of the insoluble material from the original dry weight.

[5]Reference to specific products does not imply endorsement by the U.S. Bureau of Mines.