Appendix 1. Sampling and Sample Processing Techniques

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Sampling techniques included automatic, flow-proportional, fixed point sampling of water and the collection of bottom-sediment grab, fish-tissue, stormwater, and passive in situ chemical-extraction samples.

Bottom-Sediment Grab Sampling and Sample Processing

Bottom-sediment grab samples were collected at 23 locations in the Neponset River, Neponset River Estuary, and Mother Brook. In water deeper than about 3 ft, the top 4 in. of sediment was collected by means of a stainless-steel dredge. In water shallower than about 3 ft, bottom sediment was scooped by a Teflon scoop directly into a precleaned Teflon bag. A minimum of three samples was collected at each sampling location. In the lab, grab samples were manually homogenized in the Teflon bag and squeezed out of the bag into 500-mL amber-glass jars. Sediment grab samples were sent on ice to a commercial laboratory for PCB-congener analysis. Subsamples were also collected and sent to a commercial laboratory for elemental analysis.

Automatic, Flow-Proportional, Fixed-Point Sampling of Water and Sample Processing

The mass of PCBs transported in river water to the estuary was measured for one year (May 2005 to April 2006) by outfitting a USGS streamgage (Neponset River at Milton Village, 011055566) with an ISCO automated sampler and Campbell Scientific, Inc.[14] data logger. Each time a specified volume of water passed the streamgage, the data logger initiated a sequence that included purging the intake line; rinsing the intake line with river water; and opening a two-way valve, which directed a 50-mL sample of river water into a 20–L precleaned Teflon bag. The specified volume was 1.6, 3.2, or 6.4 Mft^3, predicted on the basis of long-term discharge records and local weather forecasts. The 50-mL samples were composited in this way for 1 month. At the end of each month, the samples were retrieved and brought to the USGS laboratory in Northborough. One exception to this procedure was implemented during March and April 2006, when low streamflow necessitated combining the water samples collected during these two months into one sample. For this reason, 11 (instead of 12) flow-proportional water samples were collected between May 2005 and April 2006, with each sample consisting of 120 to 617 discrete samples. These samples comprise a total of 2,656 discrete water samples or about 133 L of river water collected over a period of one year. Ideally, similar numbers of samples would have been collected each month; however, unpredictable changes in the flow regime as a result of changes in weather (for example, storms or dry spells) and streamflow regulation (for example, diversion of water from the Charles River into Mother Brook) resulted in variation in the number of discrete monthly samples collected.

Dissolved and particulate PCBs in water samples were extracted from the water phase onto a glass-fiber filter (GFF; particulate) and an XAD–2 resin column (dissolved). Samples were pumped slowly at less than 3 mL/min by a peristaltic pump with C–Flex tubing in the pump head and Teflon tubing through a GFF with 0.1-µm pore size that had been baked at 400°C for 4 hours and a XAD–2 resin extraction column (fig. 1–1). Teflon tubing and the filter housing were cleaned by soaking in acetone, hexane, and methanol, in that order (Litten, 1993); C–Flex tubing was soaked in methanol only. After being soaked, the tubing and filter housings were rinsed with tap water. After the sample was filtered, the Teflon sample bag was rinsed with 1 L of tap water, which was also filtered through the GFF cartridge and XAD–2 extraction column, to remove any remaining solids left behind in the bag. GFF cartridges, XAD columns, and Teflon sample bags were wrapped in hexane-rinsed aluminum foil, put on ice, and shipped to a commercial laboratory for PCB analysis.

Fish Sampling and Processing for Tissue Samples

White sucker *(Catostomus commersoni)* were collected twice (in August 2003 and September 2005) from the Tileston and Hollingsworth and Walter Baker Impoundments in gill nets along the bank and across the river. Eight fish were collected from each impoundment on each sampling date (a total of 32 fish), stored on ice, and brought back to the USGS laboratory in Northborough. Common mummichog were collected from the Neponset River Estuary on July 6, 2006, in minnow traps baited with cat food. Cat-food containers were perforated with a sharp knife so that the fish could smell, but not eat, the bait and were placed in the minnow traps.

In the lab, fish were measured and weighed. Fish were then wrapped in aluminum foil, packed on ice, and shipped overnight to a commercial laboratory for PCB-congener analysis. Fish collected in 2003 were skinned and filleted prior to analysis, whereas fish collected in 2005 were analyzed whole. White sucker collected in 2005, however, had their stomach contents emptied prior to analysis by removal of the intestinal tract, extrusion of the contents, and replacement of the intestines. Intestinal contents were removed so that PCB-congener concentrations and patterns measured in fish tissue were not biased by PCB-contaminated bottom sediment that may have been ingested by the fish just prior to capture. This procedure was done to determine the primary pathway(s)—PCB-contaminated water and (or) PCB-contaminated bottom sediment—through which fish, white sucker in particular, became contaminated with PCBs in the Neponset River. Five samples of filleted and whole fish were homogenized by sampling location and date.

[14] Any use of trade, product, or firm names is for descriptive purposes only and does not imply endorsement by the U.S. Government.

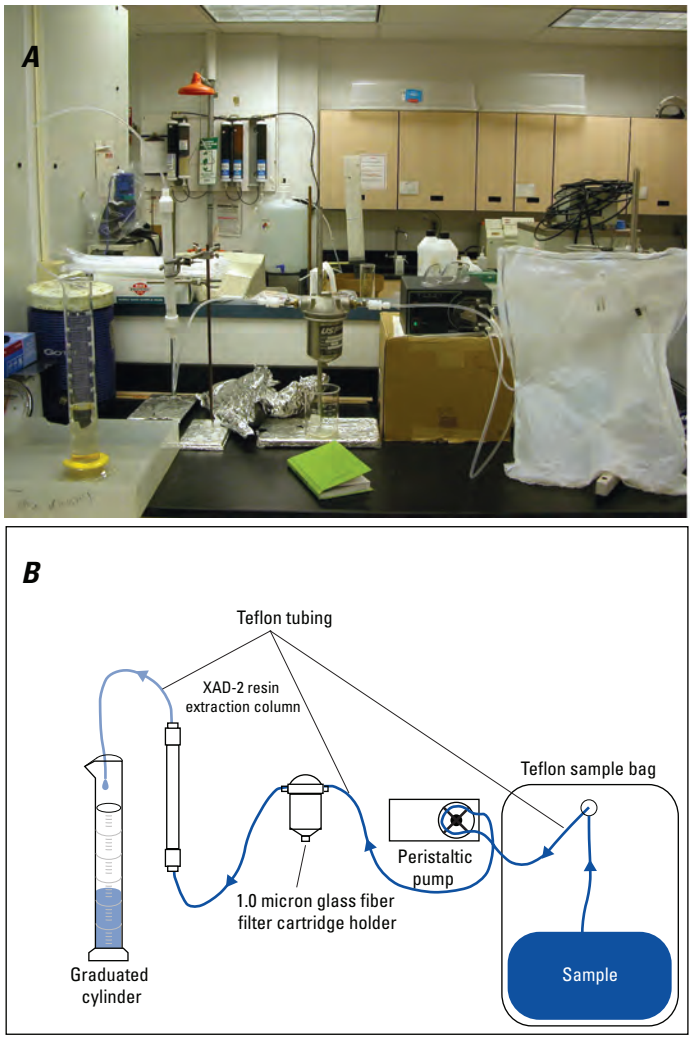

Figure 1–1. *(A)* Apparatus and *(B)* schematic diagram of apparatus for the separation of particulate (with diameters greater than one micron) and dissolved (with diameters less than one micron) polychlorinated biphenyls.

Stormwater Sampling and Sample Processing

Isokinetic, equal-width integrated (EWI) samples were collected by means of a USGS DH–81 sampler with a 1–L precleaned (acetone, hexane, and DIW rinse) Teflon bottle at about 20 locations along the cross section of the river. Once filled, the 1–L Teflon bottle was poured into a 20–L Teflon bag. While water quality was being sampled, concurrent measurements of streamflow were made with an acoustic doppler current profiler (ADCP). Water samples were brought back to the USGS laboratory in Northborough and processed as described previously.

Passive Chemical-Extraction Samplers

PISCES were deployed at 15 sampling locations in the Neponset River, Neponset River Estuary, and Mother Brook. Prior to deployment, samplers were cleaned in the laboratory with soap and water and a deionized water (DIW) rinse and then were air dried. Low-density polyethylene membranes and Viton O–Rings were cleaned by a 7-hour Soxhlet extraction with hexane. After being cleaned, the samplers were assembled and wrapped in hexane-rinsed aluminum foil. In the field, the assembled samplers were rinsed with hexane again and were filled with 0.2 L of hexane just before deployment. PISCES were attached to cinder blocks, buoys, bridges, or pilings about 6 in. below the surface of the water. Dissolved PCBs diffuse from the water column through the membrane during the time the samplers are deployed, thus providing time-integrated samples of dissolved PCBs. PISCES were retrieved after nearly two weeks. At the time of sample collection, hexane from each PISCES was carefully poured into a 125-mL amber-glass vial and sent on ice to AXYS Analytical Laboratory for PCB-congener analysis. At the time of retrieval, water temperature and specific conductance were measured, and the condition of each sampler was noted.

Reference

Litten, S., Mead, B., and Hassett, J., 1993, Application of passive samplers (PISCES) to locating a source of PCBs on the Black River, New York: Environmental Toxicology and Chemistry, v. 12, p. 639–647.

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