

Summary of 2,4-D Survey

Sediment samples from various locations in Paradise Lake were collected and delivered to the lab for analysis of 2,4-D. 2,4-D (2,4-dichlorophenoxyacetic acid) is a commonly used herbicide used for the control of many broadleaf plants. It is approved for use in aquatic environments and has proved effective in the control of many nuisance macrophytes. It can only legally be applied by licensed applicators. The samples were analyzed using a Perkin-Elmer high pressure liquid chromatograph (HPLC) fitted with a 5 μm C_{18} column (150 x 4.6 mm) using a mobile phase of aqueous methanol and acetic acid (300:190:10) methanol: water: acetic acid (v/v). The flow rate was 1.0 ml/minute. The compounds of interest were detected by monitoring the column effluent at 280 nm. Instrument control and data collection was with TotalChrom, v. 6.3. Under these conditions the retention time of 2,4-D was approximately 9.6 minutes and was baseline resolved.

Samples were dried for approximately three days in a lyophilizer followed by sieving the dried material through a 1.0 mm screen to remove large extraneous material (pebbles, shell fragments and woody debris). The sieved samples were ground to a fine powder using a SPEX ball mill. 1-5 grams of pulverized material was weighed into 50-mL centrifuge tube and 10 mL of aqueous acetonitrile (50/50 v/v) added to extract the 2,4-D along with any degradation products. Samples were placed on a reciprocating shaker and extracted at room temperature for 30 minutes. Extracted samples were centrifuged at 2000 rpm for 10 minutes and an aliquot of the clear supernatant was filtered through a 0.5 μm glassfiber filter fitted onto a syringe. A 20 μL sample of the filtered material was used in the analysis.

Standard materials included 2,4-D as well as the major degradation products: 4-chlorophenoxy acetic acid, 2,4-dichlorophenol and 4-chlorophenol. Each standard was baseline resolved under the chromatographic conditions and the molecules had retention times of 9.6, 5.7, 8.5 and 4.7 minutes respectively. The detector response for 2,4-D was linear from 0 to 5 ppm (mg/L) and the detection limit was determined to be approximately 0.05 ppm. The standard curve is shown in Figure 1.

Sample results are shown in Table 1. Samples 2, 7 and 8 had trace amounts of 2,4-D although no degradation products were found. Retention times were matched with standards for peak identification and all samples that showed the presence of 2,4-D were spiked with authentic material to verify identification. 2,4-D is a polar molecule and quite soluble in water (solubility is about 900 mg/L at 25°C). Because of its polar nature, it would be expected to readily adsorb onto sediment samples. Given that the samples analyzed were collected fairly quickly after application but showed only trace amounts of the material, most of the herbicide may still have been in the water column. Future sampling should include both water and sediment samples. Additionally, much larger sediment samples should be collected. The small sample amounts analyzed made identifying and quantifying the 2,4-D difficult.