A Tool to Monitor Sediment Loading by EPA Method 8082

Due to growing water quality and sediment loading concerns, many regulators are requiring industries to achieve risk-based detection limits on their discharge water analyses. One of these analyses, that for low-level PCB-Aroclors in outfalls, is of special concern for ecological and human health protection. There is also interest in identifying Aroclor point source loading of the sediment adjacent to and downriver of the outfall plume. Columbia Analytical has developed analytical techniques to ensure our ability to identify and quantify Aroclors at risk-based detection limits.

While other analytical options exist, PCB Aroclors and Congeners are generally determined using Gas Chromatography/Electron Capture Detection (GC/ECD) following EPA Method 8082. Typically, increased method sensitivity is achieved via three mechanisms:

1. Hardware modifications and/or optimization of the operating conditions of the instrument;
2. Use of sample clean-up procedures to eliminate or minimize interferences; and
3. Introduction of more sample into the instrument by:
   a. extracting a larger volume of water or sediment;
   b. concentrating the sample extract; and/or
   c. use of a Large Volume Injector (LVI).

The primary objective is to perform sample preparation techniques that result in reliable, ultra low-level Aroclor quantitation. However, interferences inherent to the sample matrix or introduced during the sample preparation may be enhanced by use of these techniques. If non-target background signal and laboratory contamination can be controlled, significant gains in sensitivity can be achieved.

A relatively large volume or mass of sample is solvent-extracted by the laboratory. After concentration of the sample extract to a small volume, the extract is subjected to Gel Permeation Chromatography (GPC) followed by a large-volume silica gel column cleanup to remove PCB-interfering pesticides and other non-target interferences. These cleanups are followed by concentrated sulfuric acid treatment to remove biogenic interferences.

Further cleanup is performed using elemental mercury to precipitate sulfur, which interferes with the accurate quantitation of Aroclors. Samples are injected into the gas chromatograph using a pressure-programmed LVI that allows the injection of up to 100 L of sample extract, which essentially results in further concentration of the sample in the instrument. This technique has been used successfully on discharge water, receiving water, sewage influents/effluents, sediment, and tissue. Aroclor reporting limits are 5 ng/L (ppt) for water, 2.0 g/kg (ppb) for tissue and sediment. Experimentally derived detection limits are 2-5 times lower than the reporting limit, depending on the Aroclor.

End users of the low-level Aroclor outfall data have been able to correlate discharge total mass to sediment loading, particularly when PCB control measures have been implemented. These data have also been helpful to potentially responsible parties when allocation disputes occur.

Other items of importance:

- Laboratory cleanliness is critical to prevent background/blank contamination. The Kelso, Washington Laboratory has segregated all ultra-low level analyses in an ultra-trace organics extraction facility. Much of the extraction and concentration glassware is disposable to prevent potential cross-contamination. Non-disposable glassware is washed, solvent rinsed, subjected to high heat in a muffle furnace, and solvent rinsed again prior to use. Glassware designated for this analysis is segregated from other equipment used for routine applications. In some cases on critical projects, it may be advisable to purchase glassware specifically for the project.
- Although extensive extract cleanups are performed, applications of this technique can still be limited by matrix.
- These analyses should be performed by experienced analytical chemists. Columbia Analytical/Kelso uses a panel of experienced analysts to review and report the data from difficult samples.

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