

IV. Project Summary

Title: On-line SFE/GC for Improved Detection of Trace Organic Pollutants in Ground Water Monitoring

Project ID: DATCP 98-02, UWS (no number specified)

Investigators:

Principal Investigator: Dr. David E. Armstrong, Professor, Water Chemistry Program, University of Wisconsin-Madison

Co-Principal Investigator: Dr. Robert J. Noll, Post-Doctoral Research Associate, Water Chemistry Program, University of Wisconsin-Madison; Current Position: Assistant Professor, Chemistry Department, Lawrence University, Appleton, Wisconsin.

Period of Contract: July 1998-Sept 1999

Background/Need: Scientists need to measure environmental pollutants with greater accuracy and with lower limits of detection.

Objectives: To improve the detection of trace organic contaminants in ground water by developing a new approach to concentrating sample analytes (on-line SFE/GC) while minimizing interferences.

Methods: Optimize the quantitative transfer of analytes from a supercritical fluid extractor (SFE) to a gas chromatograph (GC). SFE will be conducted on sorbents, such as XAD-2 or Tenax which have pre-concentrated contaminants from water matrices. An SFE/GC interface was constructed. This consisted of an independently controlled trap. A sorbent (such as Tenax) trapped analytes from the supercritical fluid extractor. Subsequent heating of the trap would desorb the analytes into a stream of chromatographic carrier gas and into the gas chromatograph (GC). A chromatographic separation could then be carried out.

Results and Discussions: The trap and SFE/GC interface was constructed. Testing of the apparatus showed that massive interferences resulted from a key valve controlling both supercritical fluid flow and chromatographic carrier gas flow. The valve also served as a sink for analytes during transfer from the trap to the GC.

Conclusions/Implications/Recommendations: This method may still be feasible with modifications. These include replacing the old valve with a Valco-type two-position, multiport valve; better and automatic temperature control of heated and cooled regions of the interface; using a commercial cryotrap at the head of the GC column; shortening transfer lines; using more inert materials for transfer lines; and improving flow control of the desorption gas and chromatographic gas.

In addition, other workers (Stone and Taylor, 2000, *Anal. Chem.*, **72**: 3085-3092) have successfully demonstrated a slightly different approach, involving the direct trapping of analytes from a supercritical fluid stream into the stationary phase of a gas chromatographic column. We recommend pursuing this method, except for where the highest level of chromatographic resolution would be required.

Finally, SFE/GC of water sampling sorbents may not be the best method for sampling water matrices for pollutants. Instead, Solid phase micro-extraction (SPME) and head space-SPME (HS-SPME) may be better methods. SPME realizes the main benefits of SFE but is less expensive. HS-SPME may more easily and effectively discriminate against ubiquitous interferents such as lipids. We recommend that future work concentrate on optimizing SPME-based methods

Related Publications: None

Key Words: PCB's, polychlorinated biphenyls, surface water, XAD, Tenax, SFE, supercritical fluid extraction, GC, gas chromatography, ECD, electron capture detection,

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