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[DNR-128]

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MEMORANDUM

DATE: August 15, 1997

TO: Randell Clark

FROM: William C. Sonzogni

RE: Final Report- Ground Water Research Program on Improved Detection Limits for Ground Water Monitoring

CC: Ron Arneson, Mike Zorn

Please find enclosed a final report on our research project. We believe the project has been very successful. We still have some more research we expect to finish up in the next few months. It will be included in Michael Zorn's Ph.D. thesis, and I will be sure to make that information available to the Groundwater Coordinating Council and the Department.

On behalf of Mike and I, I want to thank the Groundwater Coordinating Council and the Department for sponsoring our research. We hope we have made some contributions that will result in an appreciable return on the investment.

051204

Improved Detection Limits for Ground Water Monitoring

Final Report

This study was designed to improve the detection of trace contaminants in ground water. The objectives were to (1) develop a new approach to concentrating sample analytes (while minimizing interferences) for the purpose of reducing detection limits, and (2) apply a more rigorous, but practicable, computational method for the analytical limit of detection.

We have accomplished our first objective by developing an on-line supercritical fluid extraction/gas chromatographic (SFE/GC) technique for determining trace quantities of pesticides and PCB's in water. This procedure is faster, cleaner, and cheaper than the conventional Soxhlet extraction/gas chromatographic technique. Also, the on-line SFE/GC method should realize gains in sensitivity of three orders of magnitude, resulting in greatly improved detection limits, much smaller sample size requirements, or both.

One of the developments in this work is a procedure for physically handling and transferring the XAD-2 resin at a very small scale. Rather than passing 80 L-160 L of water through 125 g of resin, the developed procedure uses 50 to 200 mL of water passed through approximately 350 mg of resin. The water sample is passed through a stainless steel extraction column where the analytes are retained by the XAD-2. The water remaining in the extraction column is removed prior to the SFE/GC analysis by passing high purity nitrogen through the sample at 50°C. The time required for the nitrogen drying process to quantitatively extract the PCB's from the XAD-2 resin still needs to be optimized.

Experiments designed to recover a PCB standard spiked onto an inert matrix yielded near quantitative recovery (between 85 and 115 percent). This suggests that the analytes are being quantitatively trapped for subsequent gas chromatographic analysis. Experiments involving a PCB standard spiked onto a clean sample of XAD-2 adsorbent resin have also yielded near quantitative recovery. Extraction of PCB's spiked onto a sample of XAD-2 may not completely mimic the process of extracting PCB's from an actual water sample, so we have some more work to do before completely verifying

the usefulness of this technique. However, we expect to test a water sample containing measurable levels of pesticides and PCB's and finalize the technique in the next few months. We are very excited about this technique as it could greatly improve our ability to do trace analyses of groundwater by chromatographic techniques. It also has tremendous potential for improving our ability to measure trace organics in surface waters, such as Lake Michigan.

We have also accomplished our second objective, completing work on a statistically rigorous method for calculating the limit of detection. We believe this procedure provides a significant improvement to the current method of calculating the detection limit based on the standard deviation of replicate measurements at a single concentration. This work was recently published in *Analytical Chemistry*. A copy of the paper is included with this final report; we will be happy to provide additional reprints if needed. This material was also presented at the American Water Resources Association (AWRA) meeting March 6 and 7, 1997 and at the Society of Environmental Toxicology and Chemistry (SETAC) meeting April 2-4, 1997. Also, we made a presentation on the topic to the Integrated Science

Services staff of the Department of Natural Resources. We are currently developing simplifications to the technique so that it can be applied routinely in analytical chemistry laboratories.

Finally, I want to mention that this grant helped support Michael Zorn in his pursuit of a Ph.D. Mike has performed extremely well, receiving straight A grades in his graduate courses. He has also made great progress on his dissertation, and he expects to be finished by the end of the year. The Wisconsin Coordinating Council deserves credit for helping to provide this opportunity to Mike (a Wisconsin native, by the way).

Final report included a full text journal article Zorn, Michael E; Gibbons, Robert D; Sonzogni, William C (1997). Weighted Least-Squares Approach To Calculating Limits of Detection and Quantification by Modeling Variability as a Function of Concentration. Analytical Chemistry, 69 (15), p. 3069-3075.

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