

Report for 2001ND3021B: New Methods to Detect Chlorinated Organic Pollutants in Water

- Conference Proceedings:
 - A. F. Arruda and A. D. Campiglia. 2001. Time Resolved High-Resolution Phosphorescence Spectroscopy of Organochlorinated Pollutants in Shpol'skii Matrices with Fiber Optic Probes, 3rd biennial Joint EPSCoR Conference, Brookings SD, Abstract Book 27.
- Other Publications:
 - A. F. Arruda, T. L. Martin, A. D. Campiglia. 2001. Phosphorescence Analysis of Organochlorinated Pollutants by Shpol'skii spectroscopy", 28th Annual Conference of Analytical Chemistry and Spectroscopy Societies (FACSS), Detroit, MI, Abstract Book 288, pg. 166.

Report Follows:

New Methods to Detect Chlorinated Organic Pollutants in Water

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Polychlorinated biphenyls (PCB) and polychlorinated dibenzofurans (PCDF) are organic pollutants that can occur in surface, ground and drinking water. The initial objective of this project was to provide a rapid, simple and cost effective screening method for determining total polychlorinated biphenyls (PCB) and polychlorinated dibenzofurans (PCDF) in water samples. The objective was accomplished coupling solid-liquid extraction (SLE) and room temperature phosphorimetry (RTP). The method includes a three-step sample procedure, namely water extraction, application of phosphorescence enhancers to the extraction membrane, and direct RTP detection on the extracting substrate. The water sample (10-100 mL) is processed through an octadecyl extraction membrane with a syringe. The excess of water is removed from the membrane applying positive pressure to the syringe. 5 mL of phosphorescence enhancer is applied to the extracting substrate previous to phosphorescence detection. RTP is performed with a commercial spectrophosphorimeter under a nitrogen flow. Total analysis time varies from 8 to 10 minutes per sample. Limits of detection with 100mL of water sample were estimated at the parts-per-trillion level ($10^{-12}\text{g}\cdot\text{mL}^{-1}$). Lower limits of detection are obtainable with larger water volumes. Environmental pollutants commonly encountered in water samples do not interfere with the screening method. As an alternative to achieve specific compound identification, Laser-Excited Time-Resolved Spectrometry (LETRS) was investigated. The process involves SLE followed by laser spectroscopy measurements in the membrane at low temperature. SLE are performed, as previously described, in water sample spiked with PCB and PCDF. The extraction membrane (5.5mm diameter) is placed in a vial and a fiber optic probe is positioned above the membrane. The sample is then frozen by vial immersion into liquid nitrogen (77K) or liquid helium (4.2K). The spectroscopy is directly performed from the frozen matrix. Analysis at low temperature offered narrow spectral bands, more intense signals and larger difference between the lifetimes compared to room temperature measurements. The total procedure takes 10 minutes per sample and provides excellent reproducibility of measurements (2-5%). For 100mL water sample, limits of detection were at parts-per-trillion level ($10^{-12}\text{g}\cdot\text{mL}^{-1}$). Lifetimes obtained of PCB and PCDF in the membrane showed considerably difference, not only between PCB and PCDF, but also between their congeners. These differences facilitate time resolution of PCB and PCDF congeners.

The support from ND-WRRI permitted the development of a screening method, that can determine total PCB and PCDF in water sample and tell before hand whether the sample merits detailed chromatographic analysis. And also, permitted the investigation of LETRS with fiber optic probes to analyze PCB and PCDF in aqueous samples and overcome the lack of selectivity from the RTP method.