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Project Summary

Validation of Soxhlet Extraction Procedure for SW-846

Larry C. Michael, M. Arthur Mosely, John W. Hines, and Edo D. Pellizzari

The Soxhlet Extraction Procedure (Method 3540) was evaluated for the analysis of hazardous waste components in diatomaceous earth filter materials. Several fortification techniques were examined to produce a uniform matrix. A tumbling technique with methylene chloride spiking was judged optimal. The accuracy and precision of the analysis of this fortified matrix were evaluated. Lower recoveries were determined for toluene and the xylenes (< 50%); however, for the less volatile materials, recoveries of 90% or greater were recorded with an average relative standard deviation (% RSD) of 7.6%. Replicate analyses of four industrial waste mixtures were somewhat less precise with an RSD of 27%. These reduced precisions were attributed to a lack of homogeneity in the sample.

This Project Summary was developed by EPA's Environmental Monitoring and Support Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

Adsorption of waste materials on diatomaceous earth is a common practice in the chemical industry. In the process, a waste solution is filtered through a bed of diatomaceous earth to remove the impurities prior to discharge or reuse. The resultant filter bed contains relatively large amounts of organic compounds, typically up to 30 percent of the original filter weight with individual compounds ranging up to 10 percent.

The Soxhlet Extraction Procedure has been used extensively to monitor hazardous waste constituents in diatomaceous earth filter materials, but little information is available on the accuracy and precision of these analyses. The primary objective of this task was to provide data to demonstrate the validity of the Soxhlet Extraction Procedure for the analysis of such filter media.

The validation was conducted in two phases. Phase 1 involved evaluation of techniques for fortification of reagent diatomaceous earth filter material. Twenty organic analytes representative of the pesticide manufacturing industry were spiked onto filter material and the production of a uniform matrix was assessed. Utilizing the optimal fortification technique, the accuracy and precision of the Soxhlet Extraction Procedure was assessed. In Phase 2, method precision was further verified by analysis of actual waste samples from pesticide manufacturers.

Development of Fortification Techniques

Fortification experiments were conducted to develop a procedure for spiking diatomaceous earth with analytes that would produce a homogeneous matrix. The parameters to be assessed were: (1) spiking solvent, (2) mixing procedure, and (3) mixing time. Reproducibility of recovery was the primary measure of homogeneity, followed by quantitativeness of recovery.

Four aliquots of diatomaceous earth were spiked at the 1% level (total analyte mass). In two aliquots, methylene chloride was the spiking solvent. The samples were tumbled at 45 rotations per minute for 0.5 and 24 hours. The third aliquot was also tumbled for 0.5 hours, but differed in that methanol was the spiking solvent. The fourth experiment employed

high-speed blending as the homogenization method. Three aliquots of each homogenized sample were carried through the Soxhlet extraction. Analyses were performed by capillary column gas chromatography with flame ionization detection (GC/FID). Recoveries of analytes were determined by comparison of concentration found in the extracts with those in the original spiking solution.

No appreciable difference was shown between fortification solvents, duration of homogenization or method of mixing. Distribution of analytes in diatomaceous earth was essentially independent of these parameters. Hence, the most facile approach, methylene chloride solvent tumbled for 30 minutes, was used in subsequent work.

Soxhlet Extraction with Fortified Filter Materials

Diatomaceous earth samples from two different suppliers were spiked with each analyte at three different levels. For fifteen of the analytes, the levels were 0.1, 0.5, and 1% by weight (w/w); for the remaining five analytes, concentration levels were 1, 5, and 10%. Five replicates for each level and supplier were extracted and analyzed along with a blank. Method 3540 was followed precisely throughout extraction and solvent reduction. Internal standards were added to the concentrated extract and analyzed by GC/FID.

Soxhlet Extraction with Pesticides Manufacturing Waste

The original experimental design included analyses of actual pesticide manufacturing filter cakes. When this type of sample was not available, solid and liquid wastes from the industry were substituted. These samples were then incorporated into precleaned diatomaceous earth at a 10% (w/w) level using the homogenization procedure previously developed. Aliquots of the mixtures were dispensed to cellulose extraction thimbles and extracted by Method 3540. Six replicates of each sample were analyzed by GC/FID.

Results and Discussion

In general, the recoveries of all 20 target analytes for the spiked samples were quite high and precise. The mean recovery for all analytes was 83.6 percent with a mean percent relative standard deviation of 7.5%. There were distinctly lower recoveries for toluene, and o- and p-xylene. These analytes are suspected of being lost during solvent evaporation. If one excludes these compounds, the mean recovery of all other analytes is 90.7 percent. Neither recovery nor % RSD shows any dependence on fortification level or diatomaceous earth source.

The analyses of the pesticides waste—diatomaceous earth mixtures were significantly less precise than the spiked samples. The % RSD of the analyses of six replicate samples ranged from 10 to 46 with a mean of 27%. This is most likely a result of non-homogeneity of the sample. In three of the samples, the pesticide waste was a solid and was, therefore, not thoroughly distributed throughout the matrix. However, when a liquid waste was mixed with diatomaceous earth, replicate analyses demonstrated high precision with a mean % RSD of 10 percent.

L. C. Michael, M. A. Mosely, J. W. Hines, and E. D. Pellizzari are with Research Triangle Institute, Research Triangle Park, NC 27709.
***Robert W. Slater, Jr.** is the EPA Project Officer (see below).*
The complete report, entitled "Validation of Soxhlet Extraction Procedure for SW-846," (Order No. PB 86-118 585/AS; Cost: $9.95, subject to change) will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
Telephone: 703-487-4650

The EPA Project Officer can be contacted at:

Environmental Monitoring and Support Laboratory
U.S. Environmental Protection Agency
Cincinnati, OH 45268

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