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Project Summary

Feasibility of Using Solid Adsorbents for Dioxin Sampling

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Recovery efficiencies from XAD-2 resin (Amberlite) and Florisil of spiked 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) were determined to assess the suitability of these adsorbents for sampling. Two spiking methods were used: Method A consisted of uniformly depositing 1 mL of the spiking solution onto the adsorbent, and Method B consisted of covering the whole adsorbent sample with the spiking solution. There was no significant difference in recovery efficiencies between the two methods or between the two adsorbents. The overall recovery from XAD-2 resin was 92 ± 8%, and 95 ± 6% from Florisil.

This Project Summary was developed by EPA's Industrial Environmental Research Laboratory, Research Triangle Park, NC, 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

Due to the well known toxicity and ubiquitous nature of polychlorinated dibenzo-p-dioxins (PCDDs) in industrial manufacturing streams and (in particular) combustion sources, EPA initiated this program as part of a general effort to demonstrate the reliability of the sampling and analysis procedures. The specific goal of this program is to assess the extraction efficiency of 2,3,7,8-tetrachlorodibenzo-p-dioxin (2,3,7,8-TCDD) (chosen to represent this group of compounds) from XAD-2 resin or Florisil. These adsorbents are normally used in collecting organics emitted from combustion sources in either the source assessment sampling system (SASS) train or the Modified Method 5 (MM5).

Experimental Procedures

The spiking of 2,3,7,8-TCDD into XAD-2 and Florisil was carried out using two methods:

Method A consisted of uniformly depositing 1 mL of methylene chloride containing either 3 ng or 30 ng of 2,3,7,8-TCDD onto the surface of a 15 g portion of the adsorbent (XAD-2 or Florisil).

Method B consisted of depositing approximately 25 mL of the methylene chloride spiking solution containing either 3 ng or 30 ng of 2,3,7,8-TCDD to cover the whole body of the adsorbent.

In both cases, methylene chloride was removed by evaporation at room temperature.

Sample Extraction and Cleanup

The spiked samples were Soxlet-extracted for 16 hours with 250 mL of methylene chloride. The methylene chloride extract was spiked with 2 ng of 2,3,7,8-TCDD-$^{13}C_{12}$ to serve as an internal standard to quantify the native 2,3,7,8-TCDD and to correct for any losses during experimental workup. The extracts were concentrated to approximately 1 mL using the Kuderna-Danish (K-D) apparatus.

The extracts were cleaned up using two chromatography columns. The first column was a multilayered silica column containing alternate layers of activated silica, 44% concentrated sulfuric acid on silica, and 33% 1M potassium hydroxide on silica in order to temper acidic and basic compounds as well as easily oxidized materials that might have been co-extracted along with the 2,3,7,8-TCDD. A hexane/benzene (1:1) mixture was used as the eluent. The eluate was concentrated using K-D and solvent-exchanged into 1-2 mL of hexane prior to

adding it to the second chromatographic column containing approximately 5 g of activated basic alumina. The column was eluted sequentially with hexane, hexane/carbon tetrachloride and hexane/methylene chloride. 2,3,7,8-TCDD collected in the hexane/methylene chloride eluate was stored at 0°C after solvent-exchanging into n-decane.

Instrumental Analysis

High resolution gas chromatography/high resolution mass spectrometry (HRGC/HRMS) was used to quantify the 2,3,7,8-TCDD. The HRMS system was a VG Model MM-7070H in conjunction with a VG Model 2035 data system.

Results

Results of the 28 spiking experiments are summarized in Table 1 for XAD-2 resin, and Table 2 for Florisil. The 2.1 ng and 16.5 ng levels were the actual determined levels in the spiking solution targeted at 3 ng and 30 ng, respectively. A possible explanation for the determined levels is loss of the 2,3,7,8-TCDD due to degradation during long storage periods. The data show no significant difference in spike recoveries based on the spiking method: solution deposited on the adsorbent (Method A), or solution covering the whole adsorbent sample (Method B). The average recovery of spiked 2,3,7,8-TCDD from XAD-2 was 94 ± 6% when Method A was used, and 91 ± 10% when Method B was used. The average recovery from Florisil was 102 ± 6% when Method A was used, and 89 ± 6% when Method B was used.

Overall recovery from XAD-2 is 92 ± 8%, and 95 ± 6% from Florisil.

Table 1. Recovery Data for Spiked Native 2,3,7,8-TCDD on XAD-2 Resin

Native Spike Level (ng)	Native Determined Level (ng)			Recovery Percent				RSD[a] (%)	Spiking Method
	Rep. 1	Rep. 2	Rep. 3	Rep. 1	Rep. 2	Rep. 3	Avg.		
0	0.08	ND[b]	ND	ND	ND	ND	ND	ND	A
2.1	2.0	1.8	1.7	95	86	81	87	8	A
16.5	17.4	16.3	15.6	105	99	95	100	5	A
0	0.010	ND	ND	ND	ND	ND	ND	ND	B
2.1	1.8	1.8	2.2	86	86	105	92	12	B
16.5	14.2	14.3	16.0	86	87	97	90	7	B
							92[c]	8[c]	

[a]RSD = Relative standard deviation.
[b]ND = Not determined.
[c]Overall recovery.

Table 2. Recovery Data for Spiked Native 2,3,7,8-TCDD on Florisil

Native Spike Level (ng)	Native Determined Level (ng)			Recovery Percent				RSD[a] (%)	Spiking Method
	Rep. 1	Rep. 2	Rep. 3	Rep. 1	Rep. 2	Rep. 3	Avg.		
0	0.017	ND[b]	ND	ND	ND	ND	ND	ND	A
2.1	2.2	2.0	1.8	105	95	86	95	11	A
16.5	17.9	17.8	18.0	108	108	109	108	1	A
0	0.034	ND	ND	ND	ND	ND	ND	ND	B
2.1	1.7	1.7	1.9	81	81	90	84	6	B
16.5	16.8	14.9	15.0	102	90	91	94	7	B
							95[c]	6[c]	

[a]RSD = Relative standard deviation
[b]ND = Not determined
[c]Overall recovery

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***Merrill D. Jackson** is the EPA Project Officer (see below).*
The complete report, entitled "Feasibility of Using Solid Adsorbents for Dioxin Sampling," (Order No. PB 84-215 482; Cost: $7.00, subject to change) will be available only from:

National Technical Information Service
5285 Port Royal Road
Springfield, VA 22161
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The EPA Project Officer can be contacted at:

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