

Rapid Extraction of Dioxins from Soil, Fly Ash and XAD-2 Resin Using Accelerated Solvent Extraction (ASE) and Hot Extraction

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Introduction

Soxhlet extraction is generally used to extract polychlorinated dibenzo-p-dioxins (PCDDs) and polychlorinated dibenzofurans (PCDFs) from solid samples, but it is time consuming and requires a large amount of organic solvents. Therefore, several methods for rapid extraction have been developed in order to reduce the extraction time and the volume of required solvents (1-6). In this paper, we present improved analytical methods using Accelerated Solvent Extraction (ASE) and Hot Extraction of PCDDs and PCDFs from soil, fly ash and XAD-2 resin.

Materials and Methods

Soil samples were air-dried, sieved through a 2-mm mesh and homogenized.

Fly ash samples, which originated from municipal waste incinerators, were pretreated with 2M HCl, rinsed with water and air-dried.

To Amberlite XAD-2 resin, the recovery standard of native PCDDs and PCDFs was added previously.

The ¹³C-labeled PCDDs and PCDFs were spiked into each sample before extraction.

The ASE extraction was performed on a Dionex ASE 200.

The Hot Extraction was performed on a BÜCHI B-811 of Hot Extraction mode.

Conventional Soxhlet extraction was also carried out for comparison.

The extraction conditions are shown in Tables 1 and 2.

Each extract was cleaned up with multilayer silica gel column and alumina column.

Analyses of PCDDs and PCDFs were performed by HRGC/HRMS using a Hewlett Packard 6890 gas chromatograph and a Micromass Autospec mass spectrometer.

Table 1. Extraction Conditions for Soil and Fly ash

	ASE		Hot Extraction	Soxhlet Extraction	
	Soil	Fly ash	Fly ash	Soil	Fly ash
Sample weight	10 g	10 g	10 g	10 g	10g
Solvent	Acetone	Toluene	Toluene	2-Propanol, Toluene	Toluene
Solvent volume	50ml ×3	50ml ×3	120 ml	250 ml	250 ml
Heating temperature	150 °C	150 °C	ca. 280°C at upper heater ca. 200°C at lower heater	< 110 °C	< 110 °C
Pressure	2000 psi	2000 psi	Atmospheric	Atmospheric	Atmospheric
Extraction time	15 min × 3	15 min × 3	2 hr	24 hr	16 hr

Table 2. Extraction Conditions for XAD-2 Resin

	Hot Extraction	Soxhlet Extraction
Sample	XAD-2 resin	XAD-2 resin
Sample weight	7 g	7 g
Solvent	Toluene	Toluene
Solvent volume	120 ml	250 ml
Heating temperature	ca. 280°C at upper heater ca. 200°C at lower heater	< 110 °C
Pressure	Atmospheric	Atmospheric
Extraction time	5 hr	16 hr

Results and Discussion

ASE Extraction from Soil:

Figure 1 shows the results for ASE and Soxhlet extraction from soil. The results of ASE were equivalent to those of Soxhlet extraction. Use of ASE reduces the extraction time from 24 hours to about 1 hour and decreases the solvent volume from 250ml to 150ml.

ASE and Hot Extraction from Fly Ash:

Figure 2 shows the results for ASE and Soxhlet extraction from fly ash. The extraction yields obtained with the ASE were higher than those from Soxhlet extraction. It is considered that the ASE for fly ash is more efficient than the Soxhlet extraction. However, the extraction efficiency of ASE depended on the properties of the fly ash.

Figure 3 shows the results for Hot Extraction and Soxhlet extraction from fly ash. The results of Hot Extraction were equivalent to those of Soxhlet extraction. Use of Hot Extraction reduces the extraction time from 16 hours to 2 hours and decreases the solvent volume from 250ml to 120ml.

Hot Extraction from XAD-2 Resin:

To develop Hot Extraction to XAD-2 resin that adsorbed gaseous dioxins, we used the improved extractor with large sample capacity. Spiked PCDDs and PCDFs in XAD-2 resin were completely extracted by using Hot Extraction for 5 hours.

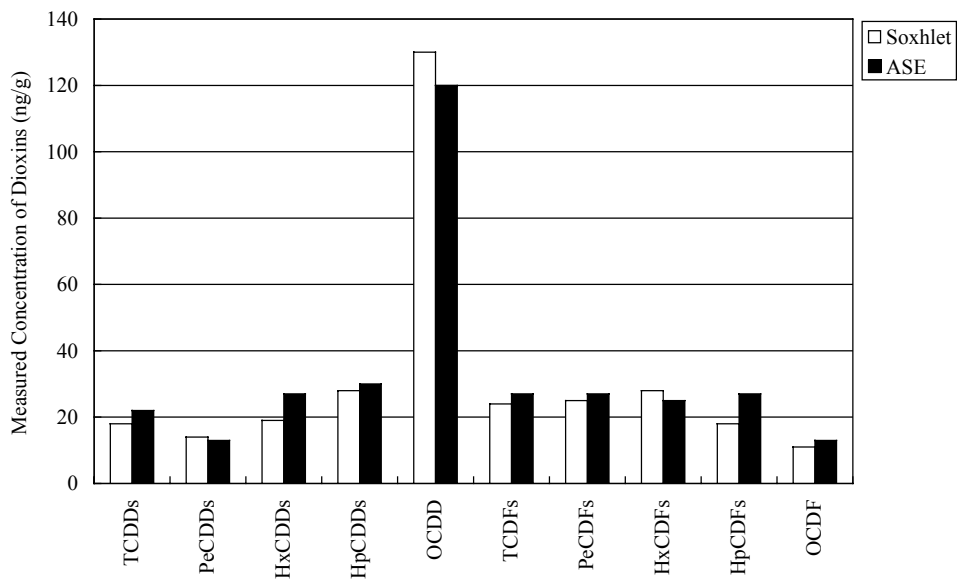
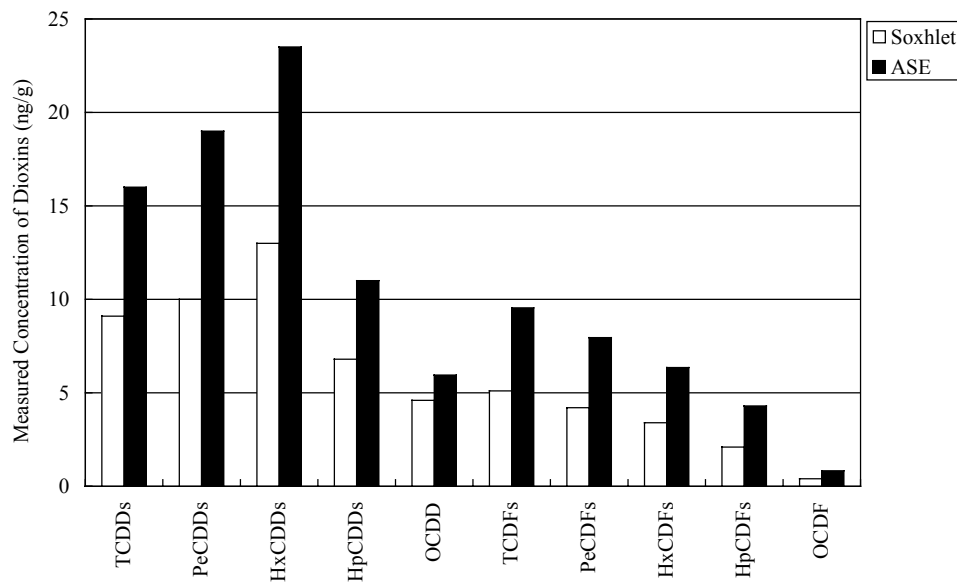


Figure 1. Comparison of ASE and Soxhlet Extraction of Dioxins from Soil

Figure 2. Comparison of ASE and Soxhlet Extraction of Dioxins from Fly Ash



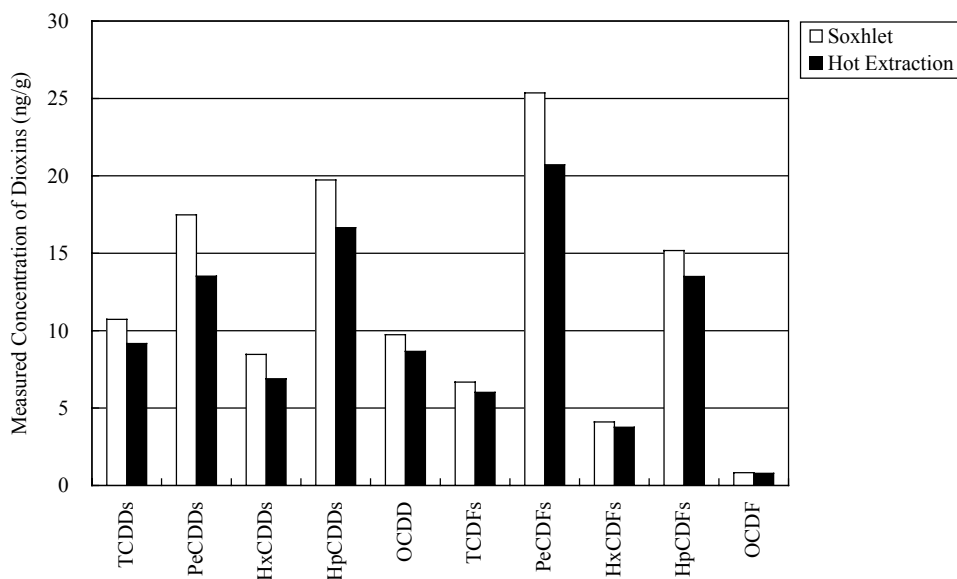


Figure 3. Comparison of Hot Extraction and Soxhlet Extraction of Dioxins from Fly Ash

References

1. Djien Liem, A.; van der Velde, E.; Linders, S.; Hijiman, W.; Marsman, J.; der Hartog, R. *Organohalogen Compounds* **1998**, 35, 1
2. Eppe, G.; Windal, I.; Gridelet, A-C.; Garcia-Ayuso, L.; Luque de Castro, M.; De Pauw, E. *Organohalogen Compounds* **1998**, 35, 199
3. Davis, M.; Paper, J.; Shafiei, H.; Stanley, J. *Organohalogen Compounds* **1998**, 35, 105
4. Eljarrat, E.; Caixach, J.; Rivera, J. *Chemosphere* **1998**, 36, 2359
5. Richter, B.E.; Ezzell, J.L.; Knowles, D.E.; Hoefler, F.; Mattulat, A.K.R.; Scheutwinkel, M.; Waddell, D.S.; Gobran, T.; Khurana, V. *Chemosphere* **1997**, 34, 975
6. Popp, P.; Keil, P.; Möder, M.; Paschke, A.; Thuss, U. *J. Chromatogr. A* **1997**, 774, 203