

An Improved Sampling System for PCDDs/Fs in Flue Gas  
of Municipal Solid Waste Incinerators

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INTRODUCTION

In Japan, PCDDs/Fs have been found even in human adipose tissue and mother's milk at low levels, and a main source of environmental contamination by PCDDs/Fs is thought to be municipal solid waste incinerators. In order to prevent environment from PCDDs/Fs contaminating, guidelines to control PCDDs/Fs formation in various kinds of incinerators were published by the Welfare Ministry in 1991. In the guidelines, combustion parameters related to PCDDs/Fs formation in incinerators have been controlled, and in incineration plants many efforts have been made to minimize PCDDs/Fs emission from the stack. Therefore, it has been required to develop a more efficient adsorbent to trap gaseous PCDDs/Fs in flue gas than Amberlite XAD2 and so simple sampling system that PCDDs/Fs can be analyzed as rapidly and easily as possible.

We developed an improved adsorbent dodecyl sulfuric acid-binding DEAE-Sephadex for uptake of gaseous PCDDs/Fs in flue gas and could devise a simple sampling system for the analysis thereby.

EXPERIMENTAL

Reagents and Apparatus

*Adsorbent* Dodecyl sulfuric acid binding DEAE-Sephadex (DS-Sephadex) was prepared by exchanging DEAE-Sephadex (Cl form) with dodecyl sulfuric acid in a giant chromatographic tube. After washing with several solvents, it is stored in ethanol.

*Phenol Red-DEAE-Sephadex (PR-DEAE-Sephadex)* PR-DEAE-Sephadex was prepared by exchanging DEAE-Sephadex (OH form) with phenol red partially so that the color would change to yellow when immersed in an acidic solution.

*DS-Sephadex Column* Before use, DS-Sephadex was equilibrated with water, and the DS-Sephadex suspension was poured into a chromatographic tube under reduced pressure to remove excess water to make the column (3.5 i.d. x 3.0 cm).

Procedure

*Sampling* A sampling system was composed of a cylindrical silica fiber filter, three impingers (the first was empty, the second containing water and the third containing a PR-DEAE-Sephadex and dichloromethane extracted cellulose powder suspension), the DS-Sephadex column, a vacuum pump and a gas flow volume meter.

The silica fiber filter was heated with a ribbon heater to approximately 120°C, the impingers were cooled with water at below 30°C and the DS-Sephadex column was cooled with water containing ice. Gas sampling speed was kept to flue gas speed, and gas sample volumes were 1-3 Nm<sup>3</sup>.

*Analysis* The PR-DEAE-Sephadex and cellulose powder suspension was separated by filtrating with a chromatographic tube. From the residue and the DS-Sephadex column, PCDDs/Fs were eluted with ethanol followed by n-hexane. After dilution of the eluate and washing solutions of inside walls of the impingers and connecting tubes, PCDDs/Fs were extracted with benzene. PCDDs/Fs in dust trapped on the filter were separately extracted. After cleanup with a KOH solution followed by concentrated sulfuric acid, the extracts were submitted to silica gel and alumina chromatography. PCDDs/Fs were analyzed isomer specifically using HRGC/LRMS/SIM.

## RESULTS AND DISCUSSION

### *Elution of PCDDs/Fs from DS-Sephadex Column*

The elution of T<sub>4</sub>-OCDDs/Fs from the spiked columns (3.5 i.d. x 3.0 cm) was carried out with ethanol.

It was indicated from elution patterns of PCDDs/Fs that they were easily eluted with ethanol from the column even when the elution had been started in the presence of water in the Sephadex. In recovery tests of PCDDs/Fs from the spiked DS-Sephadex column, quantitative results were obtained.

### *Removal of Acidic Substances in Flue Gas before introducing to DS-Sephadex column*

In order to prevent DS-Sephadex from releasing of DS by acidic substances in flue gas, the flue gas was introduced to the impinger containing the PR-DEAE-Sephadex suspension before introducing to the DS-Sephadex column. PR is an indicator for activity of the DEAE-Sephadex (OH form) to remove acidic substances.

### *Uptake of PCDDs/Fs in Flue Gas by DS-Sephadex*

In order to investigate uptake efficiency of the DS-Sephadex column for PCDDs/Fs, approximately 1 Nm<sup>3</sup> volume of flue gas was collected by use of a DS-Sephadex column (1.0 i.d. x 3.0 cm) at the inlet of the electrostatic precipitator (EP) in an incinerator. After sampling, distribution of PCDDs/Fs trapped on the column was investigated by eluting from fractions of the used column.

99.9% of total amount of PCDDs/Fs trapped were distributed to a top layer of 5 mm, and the other were trapped in the next fraction. From any lower fractions, PCDDs/Fs were not detected at all.

### *Retention of DS-Sephadex Column for PCDDs/Fs*

To validate that PCDDs/Fs trapped on the DS-Sephadex column would be retained without loss during sampling, the retention was investigated by use of DS-Sephadex spiked by <sup>13</sup>C-labeled surrogates. After sampling of approximately 3 Nm<sup>3</sup> volume of flue gas at the EP inlet, the surrogates on the column were determined.

Recovery of the surrogates were 95-104% in the native PCDDs/Fs at 14 and 28 µg levels, respectively.