Analysis of Dioxins, Furans and dl-PCBs in stack emissions using Automated Pressurized Liquid Extraction PLE<sup>®</sup> and the EconoPrep<sup>®</sup> Cleanup System.

### Introduction

Waste management comprises a set of operations, such as the collection, transport, recovery and disposal of waste, including the supervision of such operations and the aftercare of disposal sites. These operations are carried out in order to give the most appropriate destination to the waste produced in a particular area according to their characteristics and marketing opportunities, cost treatment and legal regulations. PCDD/Fs have been the target of many studies in the last few decades and are well known as unintentionally generated POPs, which pose serious health and environmental risks on a global scale

### Instrumentation

- FMS, Inc. PLE®
- FMS, Inc. EconoPrep NG.
- FMS, Inc. TurboTrace®
- FMS, Inc. SuperVap<sup>®</sup> 6 Concentrator

■ FMS, Inc. 250 mL concentrator tubes (1 mL termination)

■ Thermo Scientific Trace GC 1310 w/DFS MS and AS Triplus RSH Autosampler

### Consumables

FMS, Inc. PLE Teflon end caps

■ FMS, Inc. SNCL-ACD-006 - Classical Acid Silica Column, SNLC-BAN-004 - Basic and Neutral Silica Column

■ FMS,Inc. SNCL-BAS-004 - 4G Basic Alumina Column.

■ FMS,Inc. SNCA-C34-034 - Carbon/Celite Column

- Fisher Dichloromethane pesticide grade
- Fisher Hexane pesticide grade.
- Fisher Acetone pesticide grade
- Fisher Toluene optima grade

Relevant native and labeled spiking standards

# PLE:

- PLE CAR SS100, 100mL stainless steel PLE Cartridge.
- 30g of resin and glass filter of containers # 1 and # 2 (containing stack emissions) are placed in 100 mL extraction cell and void space filled with pre-cleaned Hydromatrix.
- Spiked with relevant recovery standards.
- Capped with disposable Teflon end caps
- Extracted with a mixture of Dichloromethane: Hexane 1:1. Run two cycles of 15 min at 120 °C and 1700 psi.
- 15 min cool down
- Nitrogen flush to transfer analytes and extract to 250 mL collection tubes

# SuperVap Concentration

- Pre-heat temperature: 55 °C
- Pre-heat time: 15 min
- Heat in Sensor mode: 55 °C
- Nitrogen Pressure: 8-9 psi
- Solvent exchange to hexane

# Dilute sample extracts to 5 mL in hexane

## Econo Prep NG Cleanup

Put SNCL-ACD-006 - Classical Acid
Silica Column, SNLC-BAN-004 - Basic and
Neutral Silica Column, SNCL-BAS-004 4G Basic Alumina Column, SNCA-C34-034
- Carbon/Celite columns in place.

 Condition columns with 20 mL Hexane.

 Load sample extracts onto silica columns
Elute the silica and alumina columns with 100 mL hexane.

■ Elute the Alumina column with the 120 mL of the 10% Dichloromethane in Hexane and collect in the concentrator tube. Concentrate until dryness and spike with the dI-PCBs internal standard.

Elute the carbon column with 110 mL of Toluene.

■ Collect in the concentrator tube. Concentrate until 1 ml and spike with the PCDD/F standard. Then move all vials to micro (vial) evaporator to concentrate to 10uL.



# Application Note



Recoveries in percent	
2.3.7.8-TCDD	85
1.2.3.7.8-PeCDD	82
1.2.3.4.7.8-HxCDD	70
1.2.3.6.7.8-HxCDD	72
1.2.3.7.8.9-HxCDD	**
1.2.3.4.6.7.8-HpCDD	89
OCDD	91
2.3.7.8-TCDF	73
1.2.3.7.8-PeCDF	82
2.3.4.7.8-PeCDF	70
1.2.3.4.7.8-HxCDF	70
1.2.3.6.7.8-HxCDF	72
1.2.3.7.8.9-HxCDF	74
2.3.4.6.7.8-HxCDF	74
1.2.3.4.6.7.8-HpCDF	78
1.2.3.4.7.8.9-HpCDF	82
OCDF	**
PCB # 81	72
PCB # 77	76
PCB # 123	82
PCB # 118	84
PCB # 114	86
PCB # 105	88
PCB # 126	87
PCB # 167	88
PCB # 156	92
PCB # 157	96
PCB # 169	94
PCB # 189	98

**Table 1.** Dioxins, Furans and dl-PCBsrecoveries for stack emission samples



**Figure 1.** Pressurized Liquid Extraction Apparatus.



Figure 2. EconoPrep Automated Cleanup System



### Application Note



When a large volume of water is present in the sampling system, PCDDs/PCDFs and dl-PCBs can be extracted using the FMS TurboTrace Solid Phase Extraction (SPE) system (Figure 3).

When the presence of water is just related to moisture, samples can be dried over sodium sulfate which will increase recoveries.

In case the moisture is not more than 2% of the ambertile resin weight, Hydromatrix is used during the PLE extraction to remove the moisture.



**Figure 3.** TurboTrace for SPE of diethylene glycol/water samples.

#### Discussion

Results for extraction and cleanup of the stack emission samples are given in Table 1. Excellent recoveries were obtained. The extraction using PLE and cleanup using Econo Prep NG are reproduceable and very suitable when analyzing Dioxins, Furans and dl-PCBs on glass filter and amberlite resin. (XAD-2).

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