

### Pittcon 2016 1090-1

William Hedgepeth, Ken Tanaka Shimadzu Scientific Instruments, Inc., Columbia, Maryland

PO-CON1624E

### Introduction

There are a large number of explosives-contaminated sites in the US, Europe, and Asia. High levels of explosives in soil can threaten the health of humans, livestock, and wildlife. A number of remediation efforts are underway, which require the analysis of explosives in soil samples. Recently, a new technique was introduced the allows the automated supercritical extraction and SFC analysis of samples with minimal sample preparation and handling requirements to save analyst time and sample preparation expenses. This technique was applied to the analysis of explosives in soil samples and showed good recoveries of the explosives tested in a number of different soil samples. Automated analysis of up to 48 samples is possible without the need for manual sample preparation to allow quick screening of explosives in numerous soil samples.

### Experimental

Fig.1 shows a diagram of the SFE-SFC system that was used in this experiment. This system consists of a combination of supercritical fluid chromatography and extraction systems. Method development was initially performed with the SFC method scouting system that automatically allows screening of up to 12 analytical columns with a number of different modifiers.

After determination of the optimal column and modifier combination for an explosives mix (AccuStandard

M-8330) was completed, the analysis was moved to the SFE portion of the system for study of the explosives mix from a variety of soil types.

The SFE portion of the system allows the automated analysis of up to 48 soil samples by combining the sample preparation portion with the chromatographic analysis. Samples are extracted from extraction vessels and automatically transferred to an analytical column for analysis.

### Instrument Design



1A: Supercritical fluid chromatography (SFC) system for analytical method development

#### 1B: On-line Supercritical fluid extraction/chromatography (SFE/SFC) system



### Method Development

Conditions	
Flow rate	: 3 mL/min
Detector	: Photodiode array
Column Temp	: 35°C
Backpressure	: 15 MPa
Mobile Phase	
A	: CO2
В	: MeOH
Gradient	: 1 to 10 min, 0 to 40% MeOH

#### Columns: 4.6 x 250mm, 5 um

Nexera UC Basic Nexera UC PFP Nexera UC Diol Nexera UC Ethyl Pyridine Nexera UC Naphthyl Nexera UC Nitro

### SFC Method Scouting



SFC Column Scouting of Explosives Mix

# Optimized SFC Chromatogram

### Nexera UC Nitro column



### Samples

AccuStandard explosives standards M-8330-05 were used to prepare an explosives mixture. One gram of each soil sample was spiked with 100 uL of a 50 ppm explosives mixture.

#### Standards

RDX, TNT, HMX, Tetryl, Nitrobenzene, 1,3-Dinitrobenzene, 2-Nitrotoluene, 3-Nitrotoluene, 2,4-Dinitrotoluene, 4-Amino-2,6-dinitrotoluene, 2,6-Dinitrotoluene, 1,3,5-Trinitrobenzene, 2-Amino-4,6-dinitrotoluene

#### Soil Samples

- 1. Clean Sandy Loam
- 2. Clean Clay Loam
- 3. Clean Sandy Soil
- 4. Clean Loam Soil





### SFE-SFC Conditions

Extraction Conditions		Chromatograp	Chromatography Conditions	
Flow rate	: 5 mL/min	Column	: NexeraUC Nitro	
95/5 CO2/MeO	Н	Flow rate	: 3 mL/min	
0-3 min	: Static extraction	6-15 min	: 0 to 40% MeOH	
3-6 min	: Dynamic extraction	17-25 min	: Wash and equilibration	

### Blank Soil Sample Extracts



SFE extracts of blank soil types



### Clean Clay Loam Extract



### Clean Sandy Loam Extract





### Clean Loam Soil Extract



### Clean Sandy Soil Extract





## Spiked Soil Extracts Overlay



Spiked explosives standard into four different soil types

## Linearity Study



One gram of soil was spiked with 50, 100, and 200 uL of explosives mix



### Linearity Results



### Discussion

- A variety of soil samples showed little interference with spiked explosive standards.
- Clean Loam Soil provided poor recovery of the late eluting RDX peak.
- Good reproducibility was observed with the explosive standard extracts from a variety of soil samples.
- Good linearity was observed for the explosive compounds.
- Automated SFE-SFC can be a quick way to screen up to 48 soil samples for explosives in a variety of soil types with minimal sample prep.

First Edition: March, 2016



For Research Use Only. Not for use in diagnostic procedure.

This publication may contain references to products that are not available in your country. Please contact us to check the availability of these products in your country.

The content of this publication shall not be reproduced, altered or sold for any commercial purpose without the written approval of Shimadzu. Company names, product/service names and logos used in this publication are trademarks and trade names of Shimadzu Corporation or its affiliates, whether or not they are used with trademark symbol "TM" or "®". Third-party trademarks and trade names may be used in this publication to refer to either the entities or their products/services. Shimadzu disclaims any proprietary interest in trademarks and trade names of the names of the

The information contained herein is provided to you "as is" without warranty of any kind including without limitation warranties as to its accuracy or completeness. Shimadzu does not assume any responsibility or liability for any damage, whether direct or indirect, relating to the use of this publication. This publication is based upon the information available to Shimadzu on or before the date of publication, and subject to change without notice.

Shimadzu Corporation www.shimadzu.com/an/