

Automated Analysis of Explosives in Soil Samples

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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 that 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.

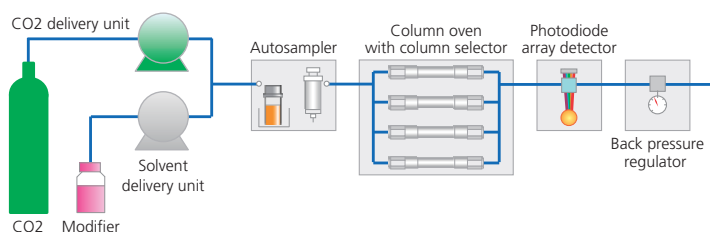
After determination of the optimal column and modifier combination for an explosives mix (AccuStandard

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.

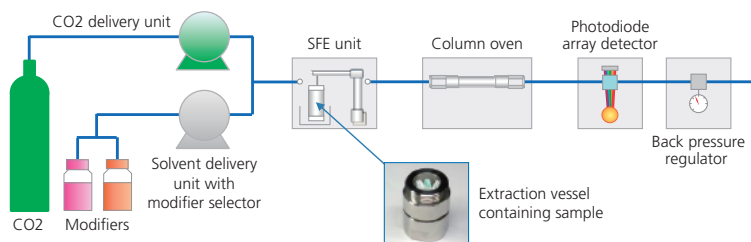
The SFE portion of the system allows the automated analysis of up to 48 soil samples by combining the sample preparation portion with the chromatographic analysis. Samples are extracted from extraction vessels and automatically transferred to an analytical column for analysis.

Instrument Design

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



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

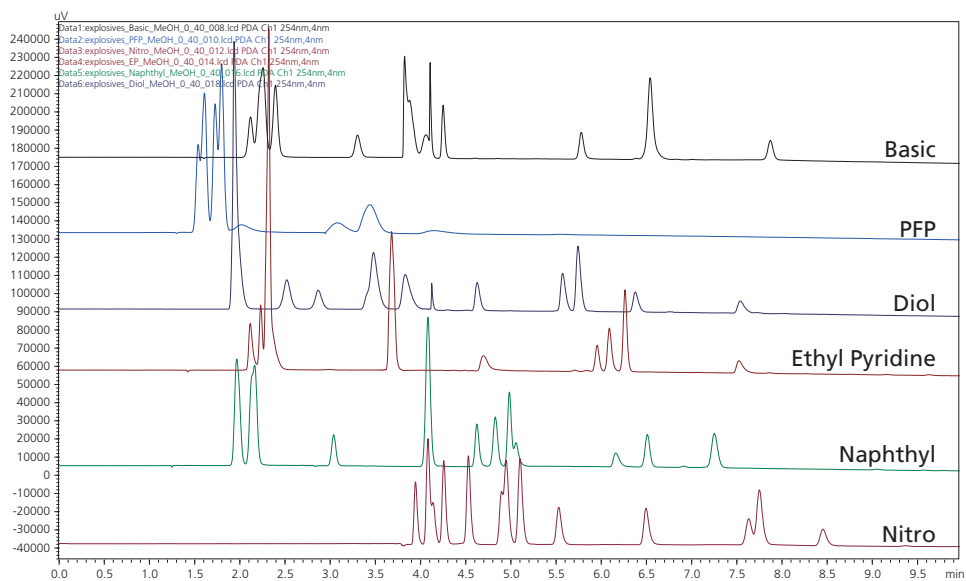


Automated Analysis of Explosives in Soil Samples

Method Development

Conditions		Columns: 4.6 x 250mm, 5 um	
Flow rate	: 3 mL/min	Nexera UC Basic	
Detector	: Photodiode array	Nexera UC PFP	
Column Temp	: 35°C	Nexera UC Diol	
Backpressure	: 15 MPa	Nexera UC Ethyl Pyridine	
Mobile Phase		Nexera UC Naphthyl	
A	: CO ₂	Nexera UC Nitro	
B	: MeOH		
Gradient	: 1 to 10 min, 0 to 40% MeOH		

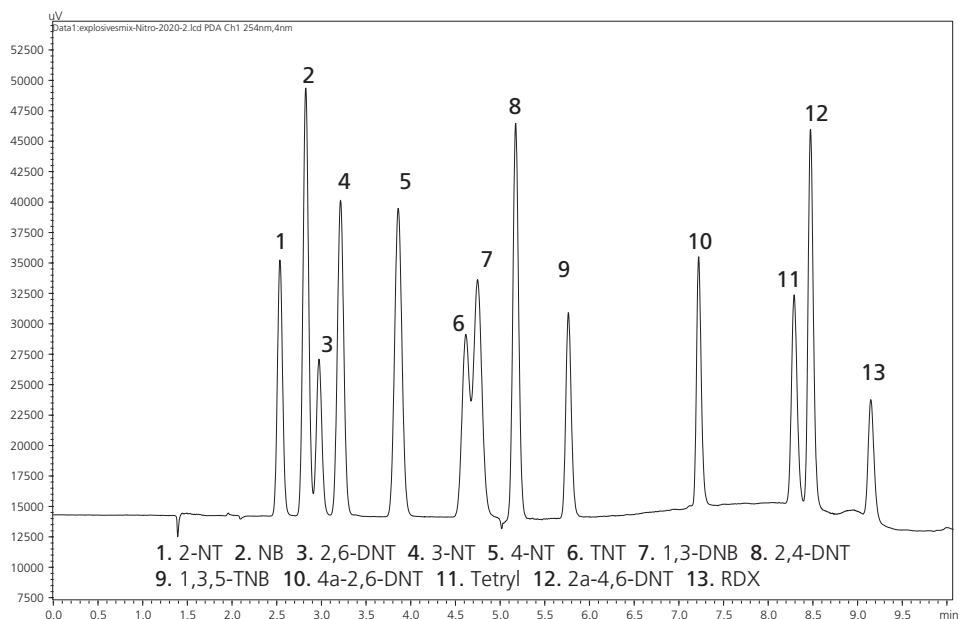
SFC Method Scouting



SFC Column Scouting of Explosives Mix

Optimized SFC Chromatogram

Nexera UC Nitro column



Samples

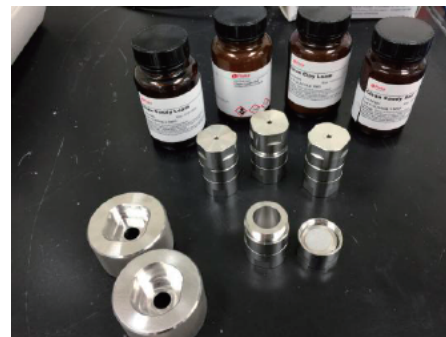
AccuStandard explosives standards M-8330-05 were used to prepare an explosives mixture. One gram of each soil sample was spiked with 100 uL of a 50 ppm explosives mixture.

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

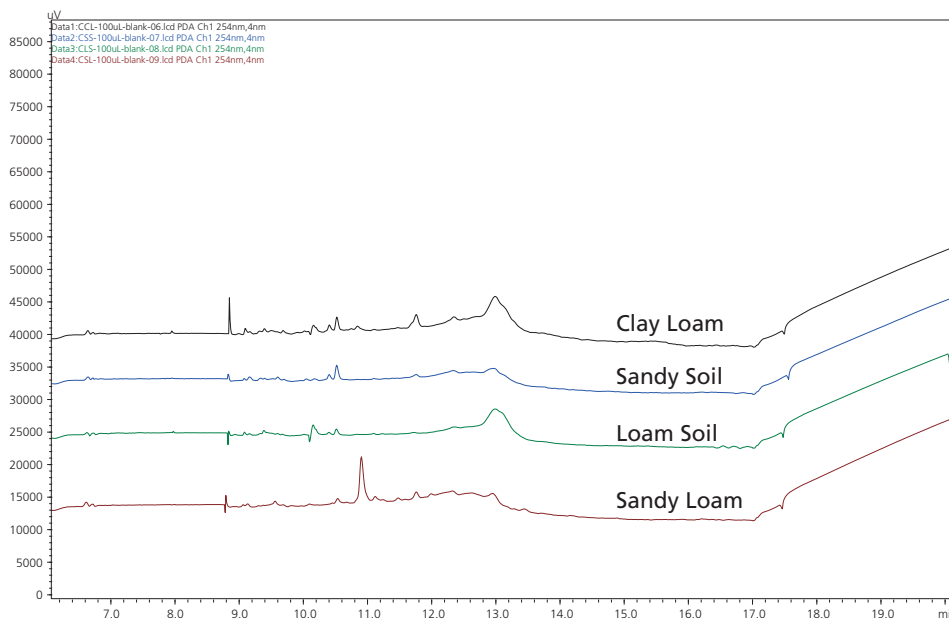


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SFE-SFC Conditions

Extraction Conditions		Chromatography Conditions	
Flow rate	: 5 mL/min	Column	: NexeraUC Nitro
95/5 CO ₂ /MeOH		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

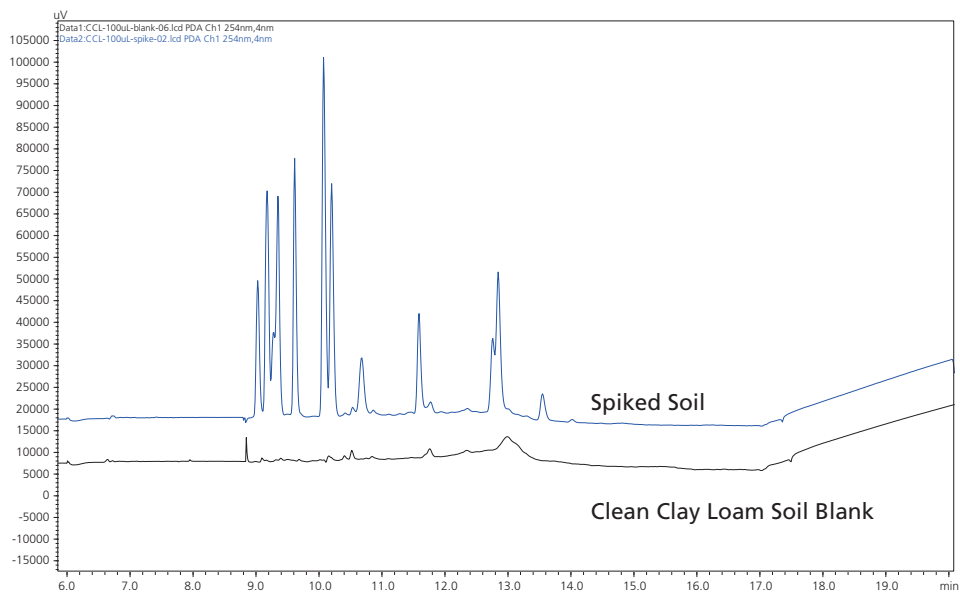
Blank Soil Sample Extracts



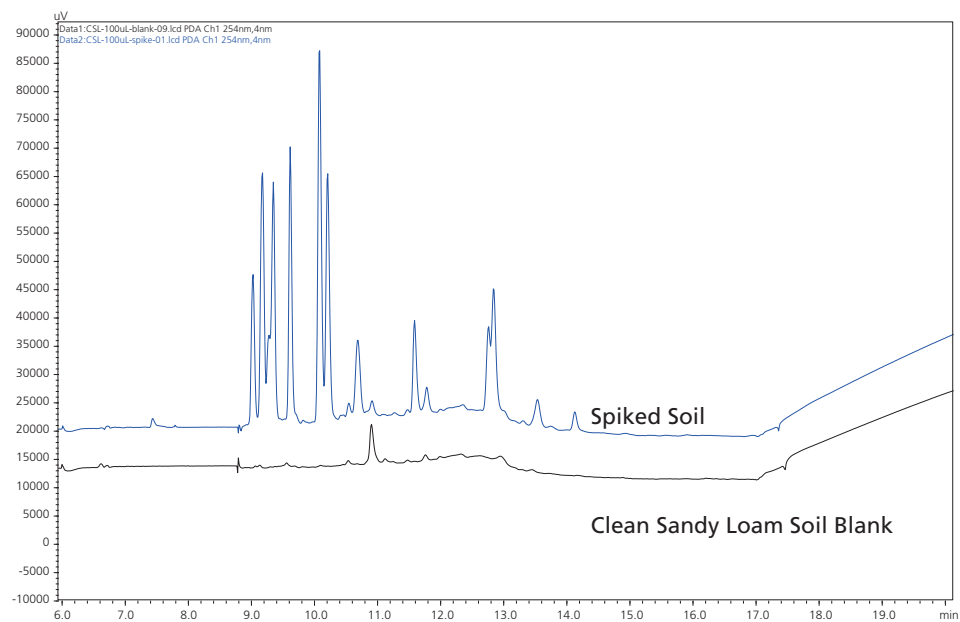
SFE extracts of blank soil types

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Clean Clay Loam Extract

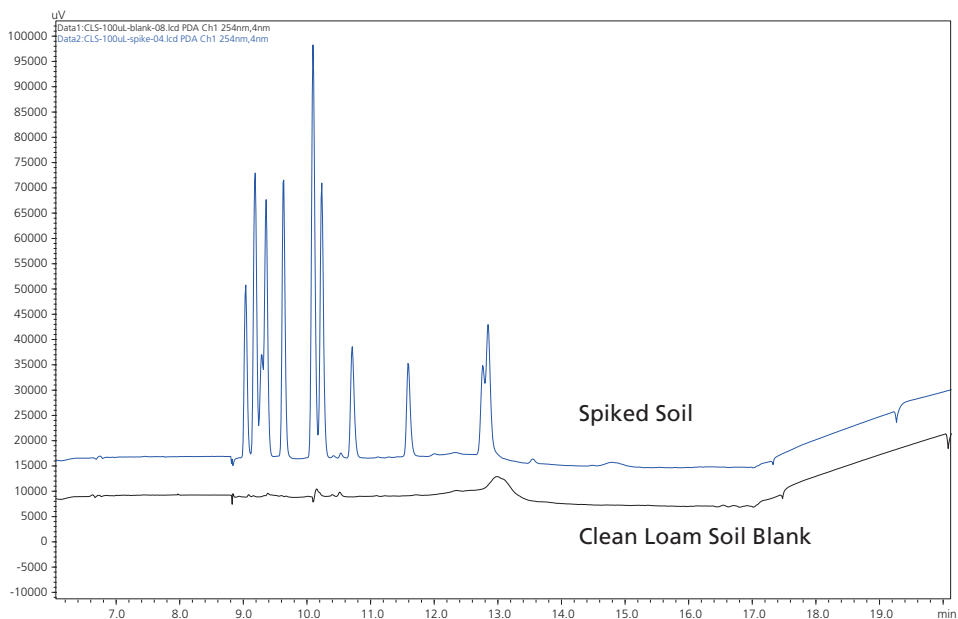


Clean Sandy Loam Extract

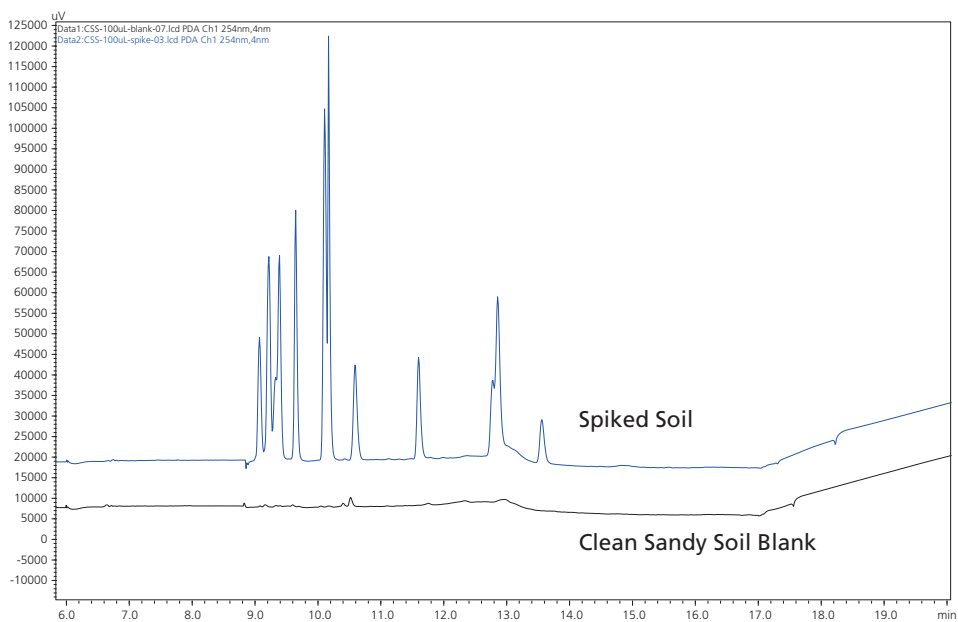


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Clean Loam Soil Extract

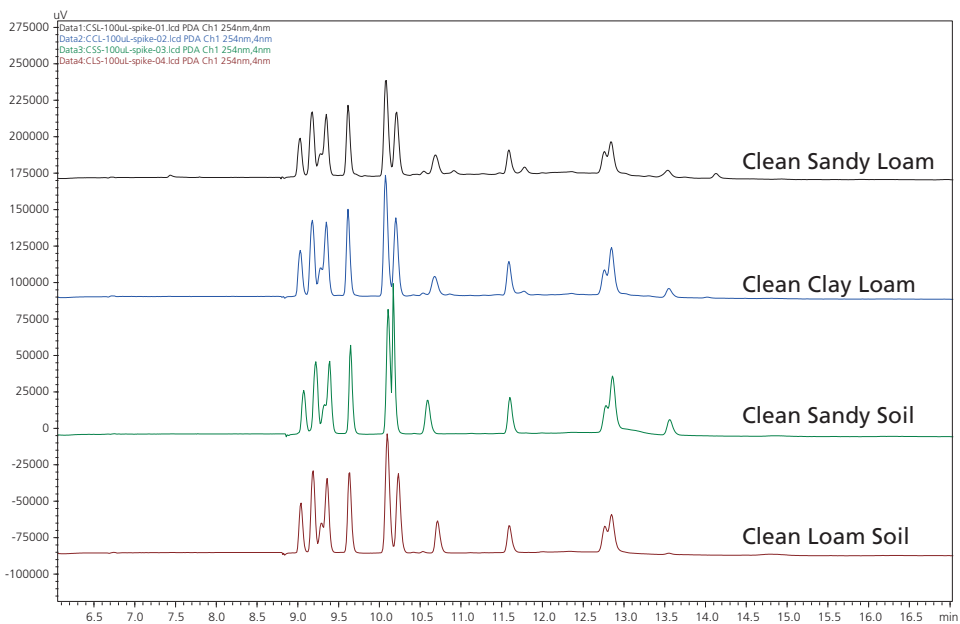


Clean Sandy Soil Extract



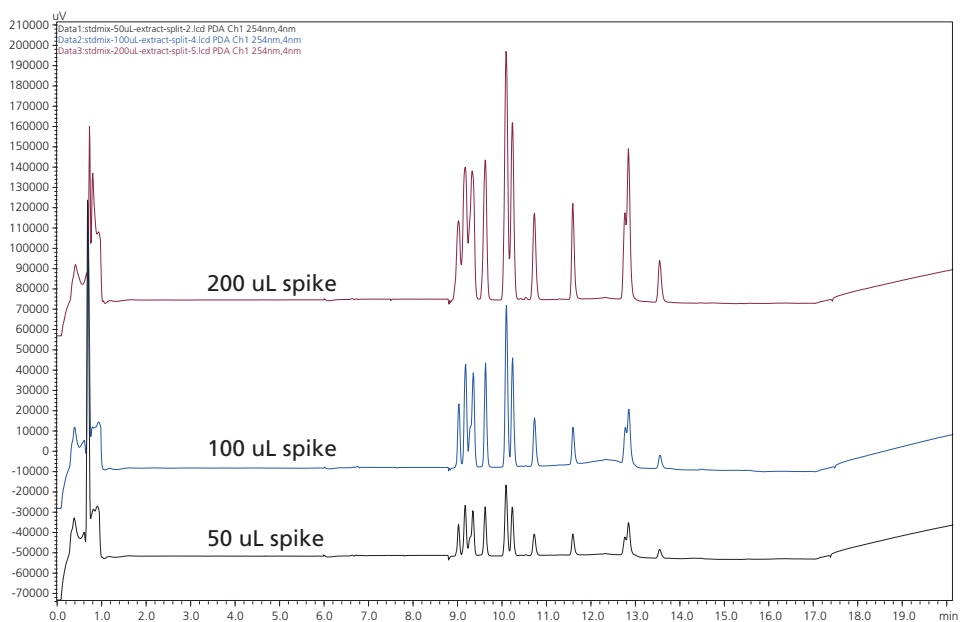
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Spiked Soil Extracts Overlay



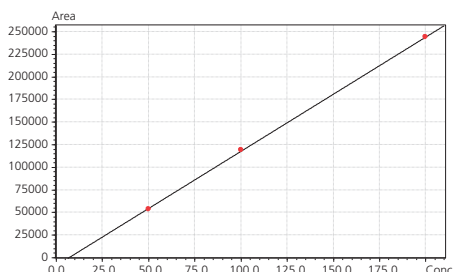
Spiked explosives standard into four different soil types

Linearity Study

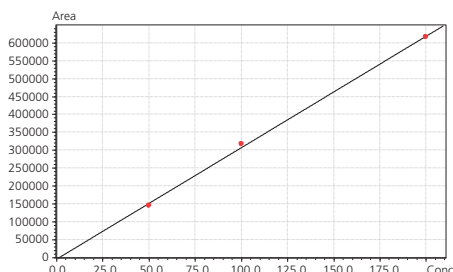


One gram of soil was spiked with 50, 100, and 200 uL of explosives mix

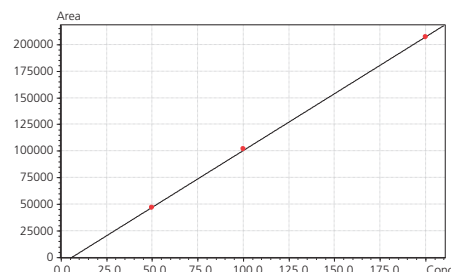
Linearity Results



2-NT
 $Y = aX + b$
 $a = 1263.65$
 $b = -8615.43$
 $R^2 = 0.9998975$
 $R = 0.9999488$



TNT
 $Y = aX + b$
 $a = 3113.98$
 $b = -3707.87$
 $R^2 = 0.9985081$
 $R = 0.9992538$



1,3,5-TNB
 $Y = aX + b$
 $a = 1066.51$
 $b = -5951.52$
 $R^2 = 0.9998268$
 $R = 0.9999134$

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.

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