Table 1 with recoveries for aliphatic fraction.

Fast Semi-Automated Total Petroleum Hydrocarbons Cleanup and Analysis

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Introduction

Soil contamination from gasoline, diesel fuel, heating oil, kerosene, or jet fuel leaks or spills is a common occurrence and a global environmental concern. Tanker transfer spills, truck transport spills and leakage from underground storage tanks continue to be sources of petroleum contamination. Recent improvements in transfer techniques, tank designs and materials have significantly reduced the danger of spills and leakage, but problems with installation or lack of operational training and maintenance procedures continue to cause environmental contamination.

These type of contaminations and spills are often analyzed for Total Petroleum Hydrocarbons. TPH is the sum of Volatile Petroleum Hydrocarbons (VPH) and Extractable Petroleum Hydrocarbons (EPH). Extracts can be analyzed for TPH using FID.

We developed a semi-automated method for fast and reliable cleanup of aliphatic and aromatic compounds from complex extracts. The extracts are fractionated using silica gel and the aliphatic and aromatic fractions are analyzed separately using GC-FID, giving a more accurate assessment of health risks.

Manual fractionation is very labor-intensive and time-consuming. The semi-automated EPH cleanup and fractionation eliminates errors associated with manual techniques and reduces glassware and solvent use. The use of certified silica columns also reduces background and interference. In this study, we present the results of the analysis with GC-FID of various TPH extracts.

Instrumentation

- FMS EZPrep123® System
- Vacuum pump
- FMS SuperVap®
- Agilent GC-FID

Consumables

- FMS, Inc. 6 g neutral silica columns
- Fisher Pesticide Grade Hexane
- Fisher Pesticide Grade Dichloromethane

Consumables

- Various aliphatic and aromatic spiking standards

Procedure

Stage 1:

- Assemble 6 g neutral silica columns with EZPrep set-up
- Syringe vial at the top is used for conditioning and sample loading.
- Condition silica column with 30 mL dichloromethane (vacuum, waste).
- Condition silica column with 30 mL hexane (vacuum, waste).

Stage 2:

- Dilute sample extract to 9 mL hexane and spike surrogate compounds (dissolved in 1 mL hexane) into sample extract.
- Load sample extract onto a silica column
- Elute column with 10 mL hexane, collecting aliphatic fraction.
- Purge aliphatic fraction line with 5 mL hexane.
- Elute column with 35 mL dichloromethane, collecting aromatic fraction.
- Purge the aromatic fraction line with 5 mL dichloromethane.

SuperVap Concentration

- Collected fractions are reduced to 1 mL final volume at ~ 5 psi nitrogen flow at 30 °C.

Analysis

- Agilent 7890 FID-ECD 30 m x 0.25 mm ID x 0.25 um film DB-5 column

Semi-automated system for TPH clean up

SuperVap-12

Results & Discussion

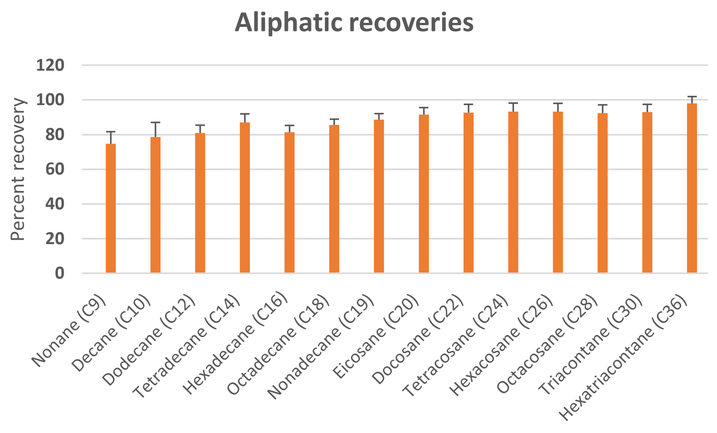

Figure 1. *Aliphatic TPH recoveries (%).*

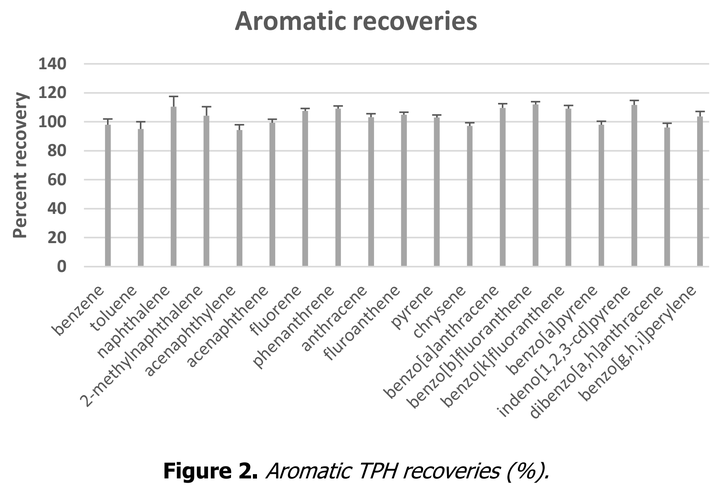

Figure 2. *Aromatic TPH recoveries (%).*

Discussion and Conclusions

The FMS EZPrep TPH semi-automated system with FMS certified 6 gm silica gel columns gives excellent and fast separation of aliphatic and aromatic hydrocarbons. Six samples can be processed with one EZPrep 123 set-up in 20 min. Excellent recoveries are seen for all analytes (Figures 1 and 2). The combination of the FMS EZPrep TPH system and FMS Teflon silica columns demonstrates consistent and reproducible data with a reliable high throughput.

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