

Fast Semi-Automated Extractable Petroleum Hydrocarbons Fractionation and Cleanup Using Pentane and Dichloromethane

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Introduction

Soil contamination from gasoline, diesel fuel, heating oil, kerosene, jet fuel leaks or spills is a common occurrence and a global environmental concern. In the United States, environmental testing labs identify fuel using the EPA Total Petroleum Hydrocarbon (TPH) method 8015B. The semi-volatile fraction is identified by the distribution pattern displayed when analyzed via GC-FID.

Petroleum products are composed of over 250 compounds, making the analysis of all of them difficult. Some states, such as Massachusetts and Washington, have created separate methods for extractable petroleum hydrocarbons (EPH) and volatile petroleum hydrocarbons (VPH). These EPH methods take a more toxicological approach and evaluate the composition of aliphatic and aromatic compounds in an extracted sample.

In this application note, we have developed a semi-automated method for fast and reliable extraction 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. Data presented here are based on the Washington State method.

Instrumentation

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

Consumables

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

Sample/Reagent Prep

- Restek EPH Surrogate Spike Mix
- Restek Fractionation Surrogate Spike Mix
- Restek Aliphatic Hydrocarbon Standard
- Restek Aromatic Hydrocarbon Standard

Procedure

Stage 1:

- Assemble silica column with EZPrep set-up.
- Syringe vial at top is used for conditioning and sample loading.
- Condition silica column with 10 mL pentane (vacuum, waste).

Stage 2:

- Spike aliphatic and aromatic compounds and surrogates into 1 mL pentane or sample extract.
- Load sample extract onto silica column
- Elute column with 15 mL pentane, collecting aliphatic fraction
- Elute column with 40 mL dichloromethane, collecting aromatic fraction.

SuperVap Concentration

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

Additional Features

- Total run time is about 20 min
- Low re-use of tubing, syringes, parts and glass ware
- No electronics and mechanical parts to fail
- No service contract or maintenance to worry about
- No repetitive motions and minimal cleaning of reusable parts

Results

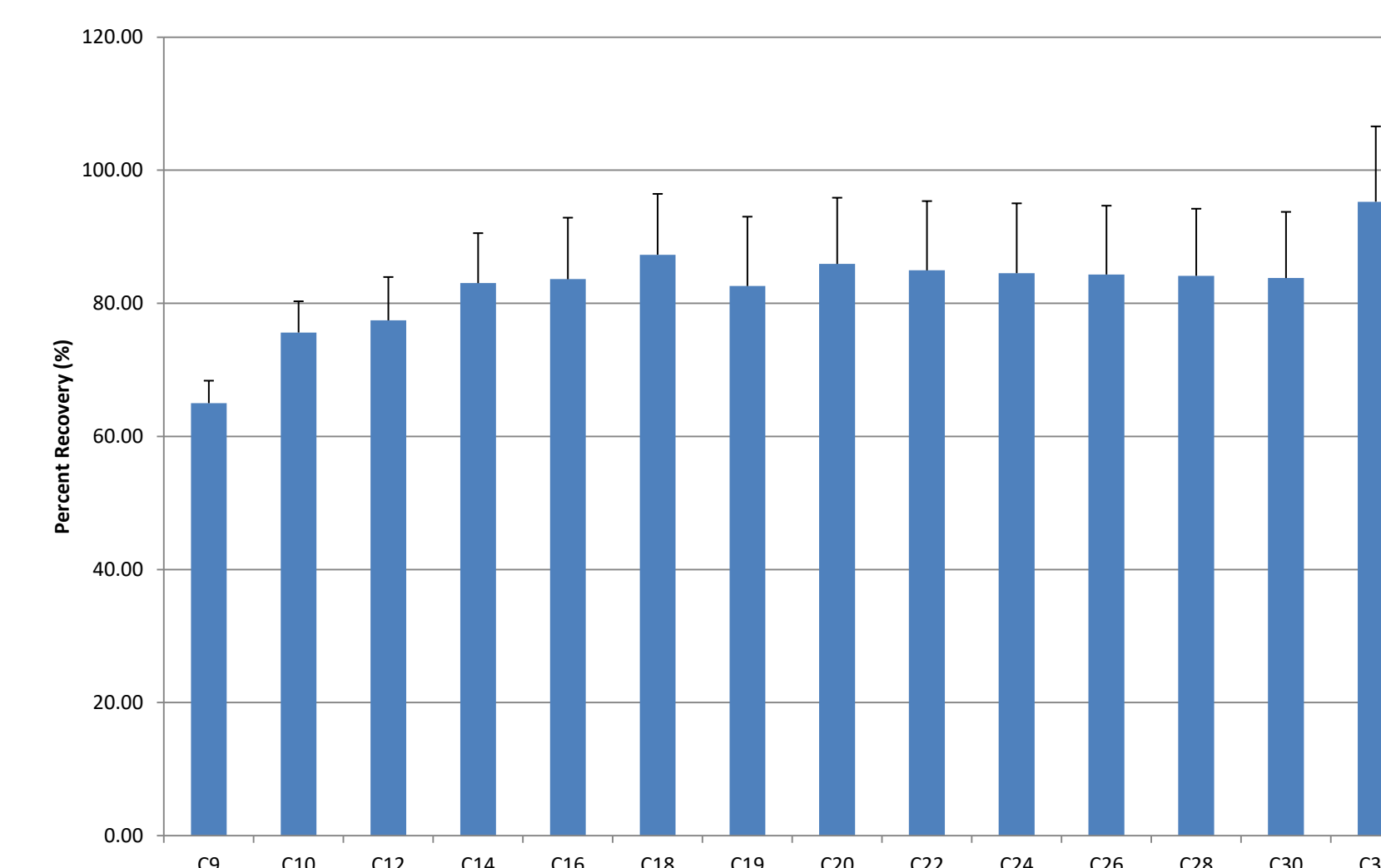


Figure 1. Recoveries for aliphatics.

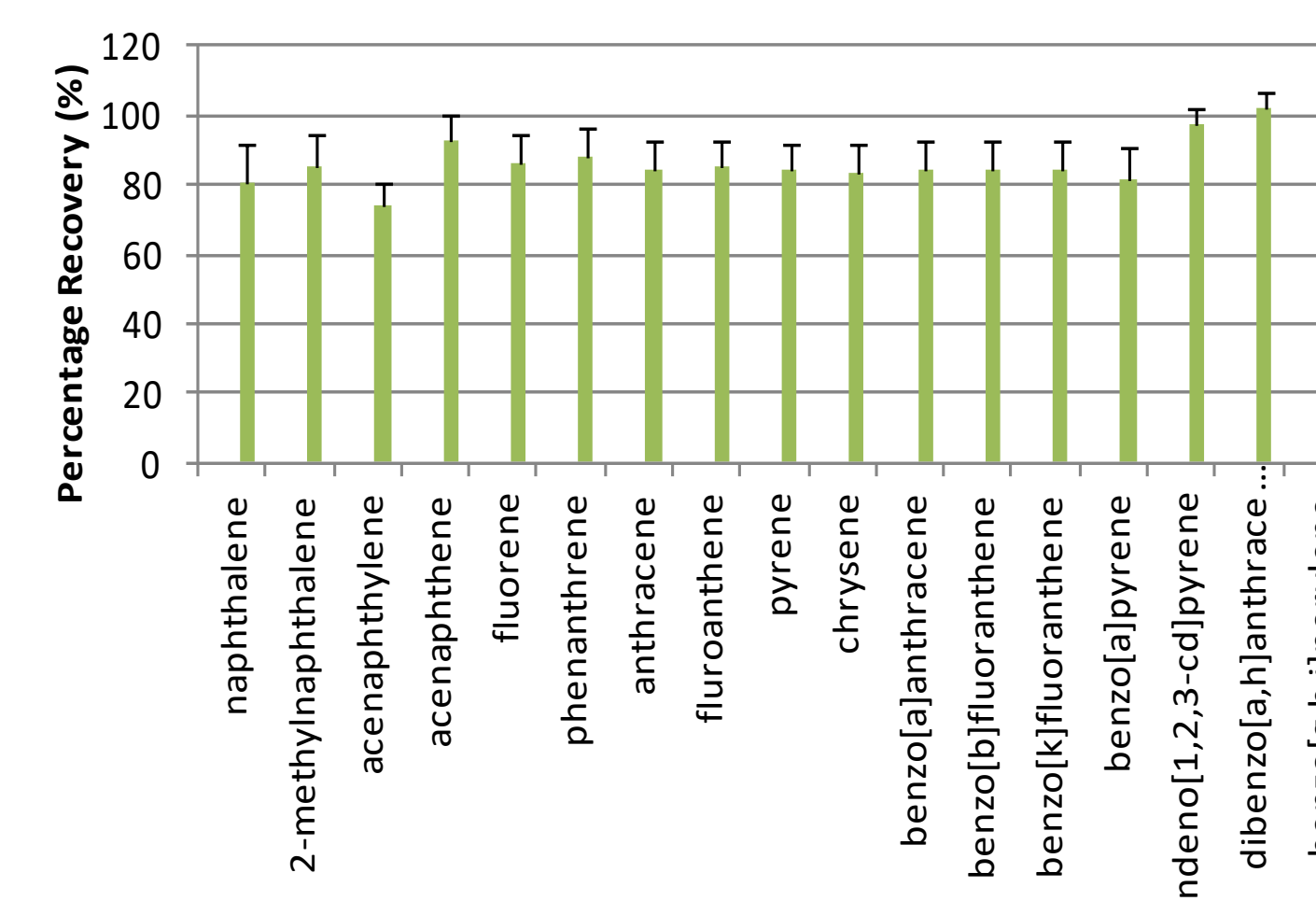
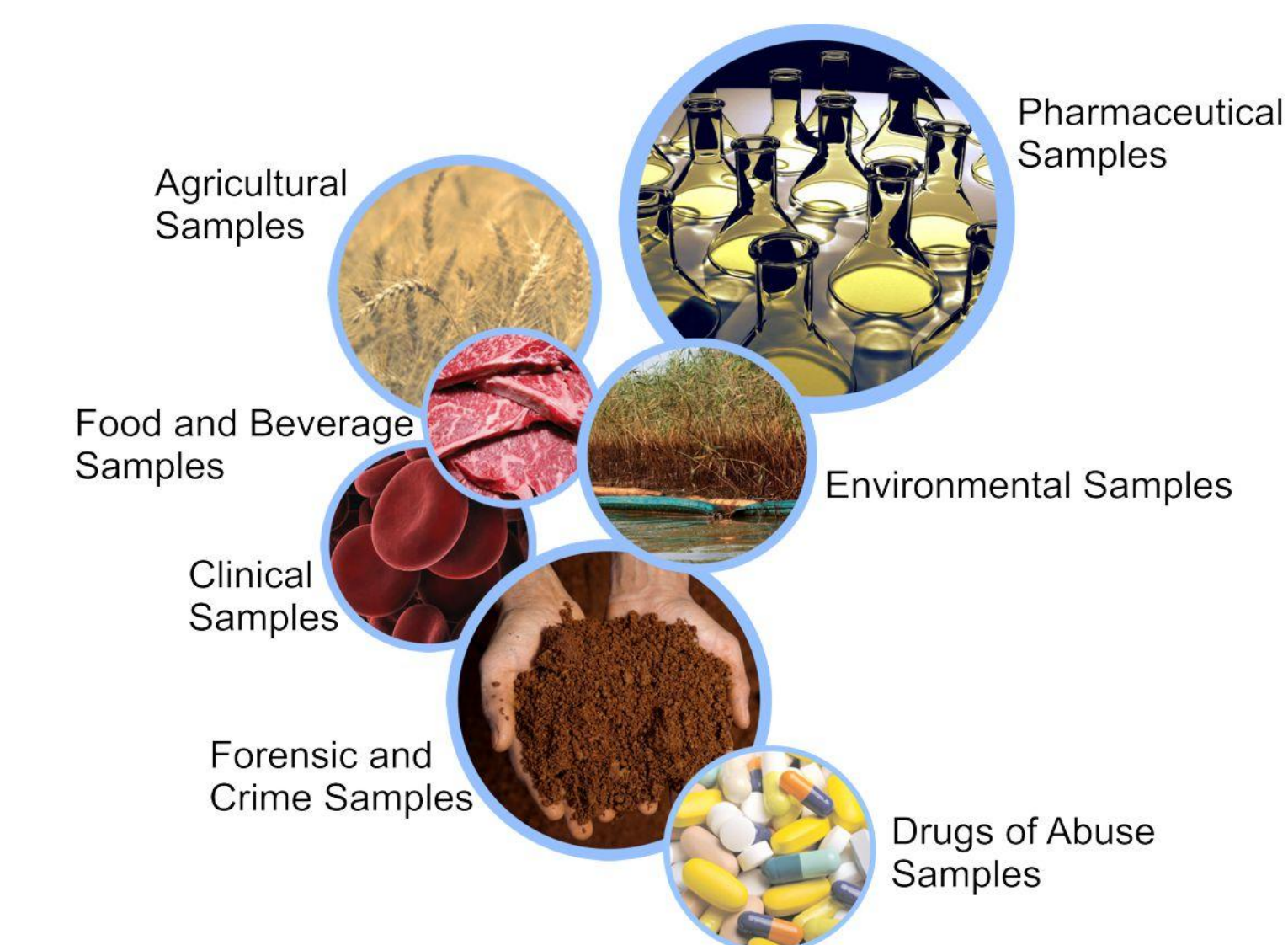


Figure 2. Recoveries for aromatics.

Discussion and Conclusions

The FMS EZPrep EPH semi-automated system with FMS certified 6 gm silica gel columns gives excellent and fast separation of Aliphatic (Alkanes) Hydrocarbons from PAHs (Aromatic) Hydrocarbons. Six samples can be processed with one EZPrep set-up in 20 min. Excellent recoveries are seen for all analytes (Figures 1 and 2). Breakthrough requirements and surrogates recoveries requirements are also easily met. The combination of the FMS EZPrep EPH system and FMS Teflon silica columns demonstrates consistent and reproducible data with a reliable high throughput.



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Sample
Extraction

Sample
Clean Up

Sample
Concentration