Application Note

Fast Semi-Automated Extractable Petroleum Hydrocarbons Fractionation and Cleanup

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 Texas, 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, FMS, Inc. has 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.

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
- ■ Restek Massachusetts EPH Surrogate Spike Mix
- ■ Restek MA Fractionation Surrogate Spike Mix
- ■ Restek MA Aliphatic Hydrocarbon Standard
- ■ Restek MA Aromatic Hydrocarbon Standard

Procedure

Stage 1:

- ■ Assemble silica column and attach to EZPrep system
- ■ A Syringe barrel at 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 into the syringe barrel onto silica column.
- ■ Elute column with 25 mL hexane, collecting the aliphatic fraction.
- ■ Purge aliphatic fraction with 5 mL hexane.
- ■ Move the EPH column to the second fraction position on the EZPrep.
- ■ Elute column with 35 mL dichloromethane, collecting the aromatic fraction.
- ■ Purge aromatic fraction with 5 mL dichloromethane.

SuperVap Concentration

- ■ Collected fractions are reduced to 1 mL final volume at ~ 5 psi nitrogen flow at 30 ^{o}C.

EZPrep123 Attributes

- ■ 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

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Table 1 Recoveries for the aliphatic fraction.

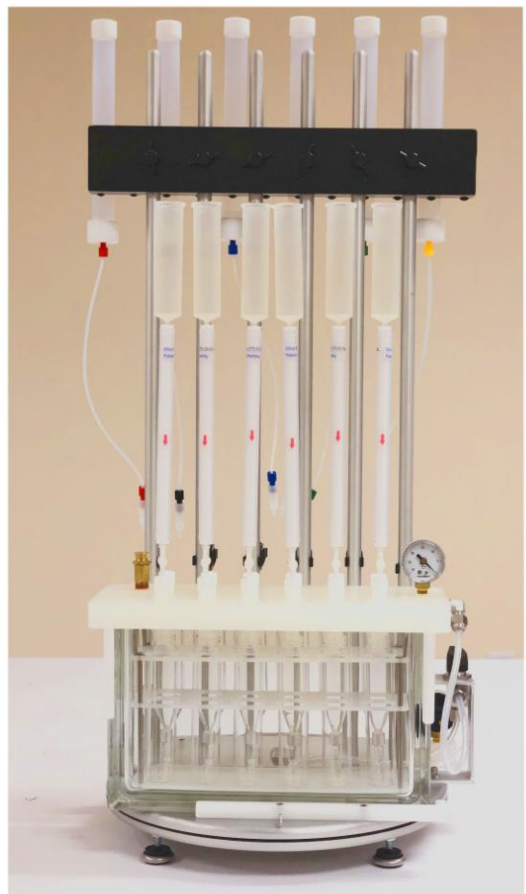
	Average Recoveries (%)	RSD (%)	Limit (%)
Nonane (C9)	74.7	7.3	30-130
Decane (C10)	78.6	8.4	40-140
Dodecane (C12)	80.9	4.5	40-140
Tetradecane (C14)	87.0	5.0	40-140
Hexadecane (C16)	81.4	3.9	40-140
Octadecane (C18)	85.6	3.3	40-140
Nonadecane (C19)	88.6	3.5	40-140
Eicosane (C20)	91.5	4.1	40-140
Docosane (C22)	92.6	4.9	40-140
Tetracosane (C24)	93.2	4.9	40-140
Hexacosane (C26)	93.2	4.8	40-140
Octacosane (C28)	92.4	4.7	40-140
Triacontane (C30)	92.9	4.5	40-140
Hexatriacontane (C36)	98.0	3.9	40-140

Table 2 Recoveries for the aromatic fraction.

	Average Recoveries (%)	RSD (%)	Limit (%)
naphthalene	110.5	6.7	40-140
2-methylnaphthalene	104.2	6.3	40-140
acenaphthylene	94.4	3.5	40-140
acenaphthene	99.3	2.5	40-140
fluorene	107.4	1.8	40-140
phenanthrene	109.0	1.9	40-140
anthracene	103.1	2.4	40-140
fluroanthene	104.8	1.8	40-140
pyrene	103.0	1.7	40-140
chrysene	97.1	2.2	40-140
benzo[a]anthracene	109.6	2.9	40-140
benzo[b]fluoranthene	111.9	1.9	40-140
benzo[k]fluoranthene	109.0	2.3	40-140
benzo[a]pyrene	98.0	2.3	40-140
indeno[1,2,3-cd]pyrene	111.6	3.1	40-140
dibenzo[a,h]anthracene	96.1	2.9	40-140
benzo[g,h,i]perylene	103.7	3.5	40-140

Conclusions

The FMS EZPrep123 EPH semi-automated system with FMS certified 6 gm EPH Silica gel columns and SuperVap for concentration gives excellent and fast separation of Aliphatic (Alkanes) Hydrocarbons from PAHs (Aromatic) Hydrocarbons. Six samples can be processed with one EZPrep123 set-up in 20 minutes. Excellent recoveries and reproducibilty are demonstrated for all analytes (Tables 1 and 2). The combination of the FMS EZPrep EPH system and FMS EPH Silica columns demonstrates consistent data with reliable high throughput and minimal reruns.



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