Fast, Automated EPH Fractionation and Cleanup



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.

In the United States, environmental testing labs identify fuel using the EPA total petroleum hydrocarbon (TPH) method 8015B. The semivolatile 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, Massachusetts and Texas among others, 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. These compounds and compound classes have very different exposure limits. The extracts are fractionated using silica gel and the aliphatic and aromatic groups analyzed separately using GC-FID and give a more accurate assessment of the risk to health. Manual fractionation is very labor intensive and time consuming. The PowerPrep EPH Cleanup and Fractionation System automates EPH fractionation, eliminates errors associated with manual techniques and reduces both glassware and solvent consumption.

Instrumentation

- FMS, Inc. PowerPrep EPH Cleanup and Fractionation System
- FMS, Inc. 6 gm Silica Column
- FMS, Inc. SuperVap Concentrator
- Thermo Fisher Scientific Polaris Q GC/MS

Method summary

PowerPrep PLE system

- 1. Pre-condition silica column with 30 mL methylene chloride.
- 2. Rinse column with 30 mL hexane.
- 3. Dilute sample extract to 9 mL hexane.
- 4. Spike fraction surrogate compounds (1 mL) into sample extract.
- 5. Load sample extract onto silica column
- 6. Elute column with 25 mL hexane, collecting aliphatic fraction.
- 7. Purge aliphatic fraction line with 5 mL hexane.
- 8. Elute column with 35 mL methylene chloride, collecting aromatic fraction.
- Purge aromatic fraction line with 5 mL methylene chloride.
- 10. Transfer a portion of the extract to a GC vial with insert.
- 11. Analyze by GC/ECD.



PowerPrep EPH Cleanup and Fractionation System





Conclusions

The FMS PowerPrep EPH system in combination with the FMS 6 gm silica gel column and FMS SuperVap concentrator is shown to automatically separate Aliphatic (Alkanes) Hydrocarbon from PAHs (Aromatic) Hydrocarbons at a high rate of speed producing excellent recoveries and reproducibilty. The combination of the FMS PowerPrep EPH system and the FMS Teflon column demonstrates consistent, reproducible, reliable high throughput automated sample fractionation of PAHs. The PowerPrep EPH system can fractionate up to 30 samples per hour.



Aliphatic Fraction 1



Aromatic Fraction 2

Results

	Aliphatics		
Compound	Conc.	Recovery	Limit
Nonane (C9)	12.547	62.74	30%-130%
Decane (C10)	13.831	69.16	40%-140%
Dodecane (C12)	14.028	70.14	40%-140%
Tetradecane (C14)	13.994	69.97	40%-140%
Hexadecane (C16)	14.944	74.72	40%-140%
Octadecane (C18)	16.131	80.66	40%-140%
Nonadecane (C19)	16.44	82.20	40%-140%
Eicosane (C20)	16.824	84.12	40%-140%
Docosane (C22)	17.374	86.87	40%-140%
Tetracosane (C24)	18.598	92.99	40%-140%
Hexacosane (C26)	19.438	97.19	40%-140%
Octacosane (C28)	19.344	96.72	40%-140%
Triacontane (C30)	18.872	94.36	40%-140%
Hexatriacontane (C36)	16.538	82.69	40%-140%

Aromatics				
Compound	Conc.	Recovery	Limit	
Naphthalene	13.633	68.17	40%-140%	
2-Methylnaphthalene	14.138	70.69	40%-140%	
Acenaphthylene	11.62	58.10	40%-140%	
Acenaphthene	14.4	72.00	40%-140%	
Fluorene	14.951	74.76	40%-140%	
Phenanthrene	15.709	78.55	40%-140%	
Anthracene	12.216	61.08	40%-140%	
Fluoranthene	15.795	78.98	40%-140%	
Pyrene	15.809	79.05	40%-140%	
Benzo[a]anthracene	18.109	90.55	40%-140%	
Chrysene	19.833	99.17	40%-140%	
Benzo[b]fluoranthene	20.33	101.65	40%-140%	
Benzo[k]fluoranthene	16.151	80.76	40%-140%	
Benzo[a]pyrene	14.46	72.30	40%-140%	
Indeno[1,2,3-cd]pyrene	18.122	90.61	40%-140%	
Dibenzo[a,h]anthracene	19.446	97.23	40%-140%	
Benzo[g,h,i]perylene	17.918	89.59	40%-140%	

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