

Pressurized Liquid Extraction (PLE®) of Soil Samples for the Analysis of Diesel Range Organics by GC/FID

Introduction

Diesel Range Organics or DRO is the cumulative analysis of extractable alkanes between the ranges C10 through C28 as measured on a Flame Ionization Detector. Within this range, specific fuel oil identifications are possible through pattern or "finger print" matching. Not regulated under RCRA, various individual US state methods exist, many expanding analysis to the Oil Range Organics (ORO) or C₂₅-C₃₅. They can also be analyzed under EPA 8015D.

The following application note focuses on the DRO constituents which due to their increased volatility can pose greater analytical difficulties than the OROs. The approach of Pressurized Liquid Extraction paired with evaporation at low temperatures was chosen for optimal recoveries.

Equipment

- FMS, Inc. PLE® system
- FMS, Inc. SuperVap® Concentrator
- FMS, Inc. concentrator tubes
- Agilent 7890 GC with FID detector

Consumables

- Fisher Optima Methylene Chloride
- Agilent Hydromatrix®
- Restek Ottawa Sand
- Aliphatic HC standard (1000µg/ml)
- O-Terphenyl Standard (2000µg/ml)
- Diesel Reference Material

Sample Prep

1. 9 X 10 grams samples were weighed out of both Ottawa sand and soil matrices
2. All Samples spiked with O-terphenyl surrogate solution.
3. 5 Replicates of each matrix were spiked with Aliphatic HC solution.
4. 3 Replicates of each matrix were spiked with Diesel reference Material.
5. Samples mixed with Hydromatrix® and transferred to 40ml extraction cells.

PLE Procedure

Cells loaded onto PLE extraction System and DRO program initiated.

Solvent: Methylene Chloride

Extraction Temp: 150 °C

Extraction Time 15 min

Flush volume: 2 X Cell volume

N₂ Purge: 1 minute

SuperVap

1. Preheat temp: 10 minutes at 35 °C
2. Evap mode w/Sensor temp: 35 °C
3. Nitrogen Pressure: 5 PSI
4. Samples reduced to 1ml
5. Samples reduced to 1ml final volume



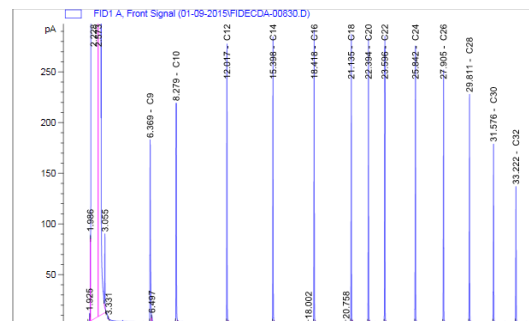
Results

Analyte	Mean Recoveries	
	Sand	Soil
Total DRO HC	82.2%	88.4%
OTP	88%	92%
Diesel Ref.	82.3%	87.9%

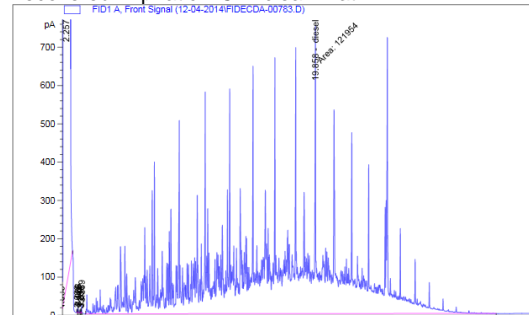
Results run in triplicate by both GC/Ion Trap and GC/FPD



FMS Inc. PLE® w/SuperVap®



Recovered Aliphatic HC in clean matrix



Recovered Diesel fuel reference material

Conclusions

Analysis of aliphatic hydrocarbon spikes showed good DRO Range recoveries for both sand and soil matrices, with all individual alkanes in the C10-C28 range recovering within 70-130%. Blank analysis not only yielded good recovery for OTP, but no detectable DRO range was measured. Lastly the analysis of a Diesel reference material resulted in calculated concentrations very close to the known reference amount (within 20% for both matrices).

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