

Improve GC Analysis of Extractable Petroleum Hydrocarbons

Using Resprep® EPH Fractionation SPE Cartridges



- Resprep® EPH fractionation SPE cartridges are manufactured for method-specific performance.
- Improved packaging and components for superior cleanliness.
- Proven quality with method-specific testing and documentation.



Concern for the environmental and health effects from exposure to materials from leaking underground petroleum storage tanks has grown over the last 20 years. The U.S. Environmental Protection Agency (EPA) and international groups have addressed some of the analytical and sample preparation techniques needed for both on-site and laboratory assessment of possible water and soil contamination. Several U.S. states also have developed their own methods to meet urgent or specific needs for analyzing samples from their own geographical areas. One of the earliest methods was developed by the Massachusetts Department of Environmental Protection (MassDEP). Introduced in 1998, the MassDEP's *Method for Determination of Extractable Petroleum Hydrocarbons (EPH)* was updated to Revision 1.1 in May 2004. This method is intended for either qualitative identification of total petroleum hydrocarbons (TPH) or for fractionation, detailed analysis, and quantification of both aliphatic and aromatic fractions from site samples of water or soil/sediment matrices [1]. This detailed quantitative analysis ensures optimal identification of suspected contaminants and supports toxicological assessment for human exposure. Because of the broad and thorough nature of the quantitative information generated by this method, many site managers and engineering firms request it to be used, even for their out of state samples. Some other states, including Texas, have adopted similarly detailed methods [2,3].

Method Challenges

Although these quantitative methods are useful, they present additional demands on laboratories, especially at the sample preparation and fractionation steps. Heavily contaminated samples require initial extraction into methylene chloride, drying with sodium sulfate, solvent exchange into hexane, and concentration, followed by fractionation and cleanup using solid phase extraction (SPE) cartridges containing large beds (5 g) of silica gel. Although these cartridges are now commercially available from many suppliers, the quality of these products must be assured for each lot, and, sometimes, even within lots. The activity level

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and capacity of the silica used in the cartridge, the compression of the bed, and the quality of the constituents and packaging used in the product all are critical to achieving good results. Because the SPE cartridges have a large diameter, voiding can occur near the upper retaining frit and within the silica bed due to settling during shipment and transport of the cartridges. Voids can be eliminated by tapping the tip of the cartridge against a hard surface, to resettle and level the bed, then inserting a clean, flat-ended rod into the cartridge and pushing firmly, to reposition the frit into contact with the silica bed.

Determining the activity level of the silica is more difficult. The volume of hexane required to fractionate the aliphatic portion of the sample, without allowing any aromatic analytes to break through into the second methylene chloride fraction, can vary, typically from 17 mL to 22 mL (± 0.5 mL), and must be determined for every lot of cartridges. Slight variations in the silica material, or in humidity levels during manufacture of the cartridges, can have a dramatic effect on the hexane fractionation results. Exposure in the lab also can affect results, so all cartridges should be left in their original packaging until needed, and unused cartridges should be resealed immediately and stored in a desiccator.

The level of extractables in commercial SPE cartridges is one of the greatest concerns with these methods. Trace levels of phthalates and other contaminants from cartridges, frits, and packaging are easily extracted along with the desired analytes, complicating low-level quantification.

Proven Performance

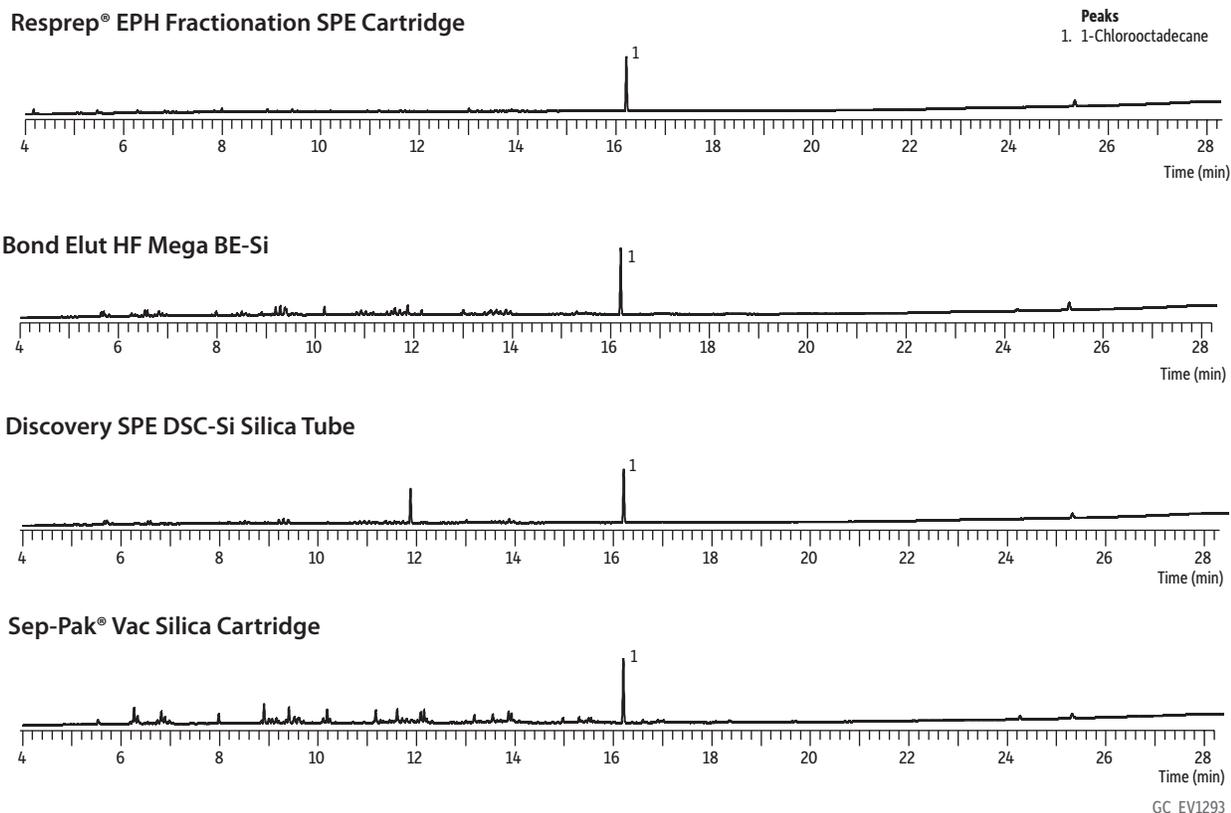
We have always specially treated our Resprep® EPH fractionation SPE cartridges (cat.# 26065) to ensure minimum background extractables and maximum silica activity. Figure 1 shows the background level of the hexane fraction from a select group of competitor cartridges compared to the significantly lower background from the new Resprep® product. We have expanded our quality testing to demonstrate method-specific performance for the fractionation of extractable petroleum hydrocarbons. We have also improved the packaging and components for superior product cleanliness. This proven quality is demonstrated with extensive documentation provided in each package. A certificate of analysis shows not only the physical characteristics of the sorbent, but also the aromatic breakthrough and the chromatographic cleanliness.

If you are conducting EPH or similar analyses and are concerned with the quality and uniformity of the SPE cartridges you have been using, try Resprep® EPH fractionation SPE cartridges. Demonstrated product quality and rigorous testing ensure minimal interference from coextracted compounds, which can improve accuracy and precision for extractable petroleum hydrocarbons.

References

- [1] Massachusetts Department of Environmental Protection, Division of Environmental Analysis, Office of Research and Standards, Bureau of Waste Site Cleanup, Method for the Determination of Extractable Petroleum Hydrocarbons (EPH) Revision 1.1, May 2004.
- [2] Texas Natural Resource Conservation Commission, TNRCC Method 1005, Total Petroleum Hydrocarbon, Revision 03, June 1, 2001.
- [3] Texas Natural Resource Conservation Commission, Draft TNRCC Method 1006, Characterization of Nc6 to Nc35 Petroleum Hydrocarbons in Environmental Samples, (May 2000).

Figure 1: New Resprep® EPH fractionation SPE cartridges have lower levels of background extractables than similar products from other vendors. Surrogate standard 1-chlorooctadecane peak is shown for reference.



Column Rtx®-5, 30 m, 0.32 mm ID, 0.25 µm (cat.# 10224)
Sample MA EPH surrogate spike mix (cat.# 31479)
Diluent: Hexane
Conc.: 20 µg/mL
Injection
Inj. Vol.: 0.5 µL splitless (hold 1 min)
Liner: 2 mm ID straight liner w/IP deactivated wool (cat.# 21718)
Inj. Temp.: 300 °C
Purge Flow: 50 mL/min

Oven
Oven Temp.: 40 °C (hold 1 min) to 300 °C at 12 °C/min to 330 °C at 6 °C/min (hold 6 min)
Carrier Gas He, constant flow
Flow Rate: 3 mL/min @ 40 °C
Detector FID @ 330 °C
Instrument PE Clarus 600 GC
Notes SPE: Condition tube with 30 mL hexane. Elute with hexane, collecting 20 mL. Concentrate to 1 mL.
Acknowledgement PerkinElmer for use of Clarus 600



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Solid Phase Extraction Cartridges from Restek
 Proven Quality • Superior Cleanliness • Method-Specific Performance

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Method Specific SPE Cartridges

These cartridges have been specifically designed to provide consistent and reproducible results for EPH fractionation.



Description	Applications	Tube Volume, Bed Weight	qty.	cat.#
EPH Fractionation	Separation of aliphatic and aromatic hydrocarbons into distinct extract fractions. Specially treated to reduce contaminants and increase capacity. Silica packing.	20 mL, 5 g	15-pk.	26065

Cartridges are manufactured using high density polypropylene and have polyethylene frits. Cartridges may be processed by any one or all of these techniques: positive pressure, sidearm flask, centrifuge, or vacuum manifold.

Reference Standards

MA EPH Surrogate Spike Mix

(2 components)

1-chlorooctadecane
o-terphenyl

4,000 µg/mL each in acetone, 1 mL/ampul
cat.# 31479 (ea.)

MA Fractionation Surrogate Spike Mix

(2 components)

2-bromonaphthalene
2-fluorobiphenyl

4,000 µg/mL each in hexane, 1 mL/ampul
cat.# 31480 (ea.)

MA Fractionation Check Mix

(31 components)

PAHs:

acenaphthene
acenaphthylene
anthracene
benzo(a)anthracene
benzo(a)pyrene
benzo(b)fluoranthene
benzo(k)fluoranthene
benzo(ghi)perylene
chrysene
dibenzo(a,h)anthracene
fluoranthene
fluorene
indeno(1,2,3-cd)pyrene
2-methylnaphthalene
naphthalene
phenanthrene
pyrene

25 µg/mL each in hexane, 1 mL/ampul
cat.# 31481 (ea.)

Hydrocarbons:

n-nonane (C9)
n-decane (C10)
n-dodecane (C12)
n-tetradecane (C14)
n-hexadecane (C16)
n-octadecane (C18)
n-nonadecane (C19)
n-eicosane (C20)
n-docosane (C22)
n-tetracosane (C24)
n-hexacosane (C26)
n-octacosane (C28)
n-triacontane (C30)
n-hexatriacontane (C36)

GC Column

Rtx®-5 Columns (fused silica)

(low polarity phase; Crossbond® diphenyl dimethyl polysiloxane)

Description	temp. limits	qty.	cat.#
30 m, 0.32 mm ID, 0.25 µm	-60 to 330/350 °C	ea.	10224

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- Proven Quality
- Superior Cleanliness
- Method-Specific Performance



Lit. Cat.# EVTS1738-UNV

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