ISOLUTE® EPH Solid Phase Extraction Columns for the Fractionation of Extractable Petroleum Hydrocarbons from Soil or Water Using Both Automated and Manual Procedures

This Chemistry Data Sheet provides procedures for both the automated and manual fractionation of EPH in soil or water extracts. Both procedures are described on page 2. Chromatograms illustrate the ability of ISOLUTE® EPH to provide efficient fractionation of hexane or pentane soil extracts into aliphatic and aromatic fractions (see Appendix).

ISOLUTE® EPH columns have been developed to fractionate pentane or hexane based soil or water extracts into aliphatic and aromatic hydrocarbon fractions (C8 – C40 aliphatics, C10 – C22 aromatics). The columns consist of a proprietary blend of silica/alumina that has been optimised to achieve a clean fractionation of the aliphatic/aromatic EPH components. ISOLUTE® EPH columns have been designed to eliminate the common problem of aromatic breakthrough into the aliphatic fraction. The approach is broadly based on procedures published by the Massachusetts Dept of Environmental Protection (MADEP) and the TPH Criteria Working Group (TPHCWG). The manual ISOLUTE® EPH column uses a similar fractionation column sorbent mass to these published methods whereas the automated ISOLUTE® EPH product and associated methodology provides reliable high throughput fractionation with substantial reductions in solvent consumption and costs, in some cases eliminating the need for evaporation of aliphatic and aromatic fractions prior to analysis.

Soil and Water Extraction Procedures

Any of the MADEP or TPHCWG approved soil or water extraction procedures are applicable to the ISOLUTE® EPH methods. The only requirements for successful fractionation are that the final solvent containing the extract should be either pentane or hexane. The presence of any polar modifier in the final extract will compromise the fractionation process. For example, if soil is extracted using dichloromethane, this solvent must be exchanged for hexane or pentane prior to fractionation. Up to 1 mL soil or water extract in hexane or pentane can be loaded onto the ISOLUTE® EPH columns.

Reduction in Solvent Consumption

ISOLUTE® EPH columns allow a significant reduction in solvent consumption compared to the existing MADEP or TPHCWG procedures. Table 1 illustrates the volume of solvents consumed using the different methods and column formats.

Table 1. Fractionation Solvent Consumption by Method.

Method	Solvent Consumed per Sample During Fractionation Step	
	Hexane/Pentane	Dichloromethane
ISOLUTE® EPH (automated)	7 mL	2 mL
ISOLUTE® EPH-M (manual)	32 mL	15 mL*
MADEP	50 mL	20 mL
TPHCWG	32 mL	30 mL

*20 mL DCM under gravity, 15 mL using vacuum manifold.

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Section 1.

Automated ISOLUTE® EPH Fractionation Methodology

The use of SPE automation provides total control of sample loading and fractionation solvent flow rates. In addition, programmed solvent switching and column solvent displacement at the end of each fractionation step ensures the use of minimum solvent volumes as well as maximising EPH recoveries and precision (see Figure 1).

The following generic method is applicable to all SPE automation platforms. Contact Biotage to request Application Notes that provide detailed set up procedures for specific automation platforms.

Automated Method Overview SPE Column:
ISOLUTE® EPH, 1.45g/3 mL P/N 928-0145-B

Generic Automated ISOLUTE® EPH SPE Procedure	
Sample	Soil or water extract in hexane or pentane.
Column Conditioning	Hexane (6 mL), flow 6 mL/min.
Sample Loading	Apply hexane or pentane extract (1 mL), flow 2 mL/min Collect column eluate (aliphatic fraction).
Aliphatic Elution	Hexane (1.5 mL), flow 3 mL/min – add to aliphatic fraction from sample loading step.
Aromatic Elution	Dichloromethane (2 mL), flow 3 mL/min Collect column eluate (aromatic fraction).

Column flow characteristics negate the use of the automation compatible 3 mL ISOLUTE® EPH column using gravity or vacuum based sample processing stations.

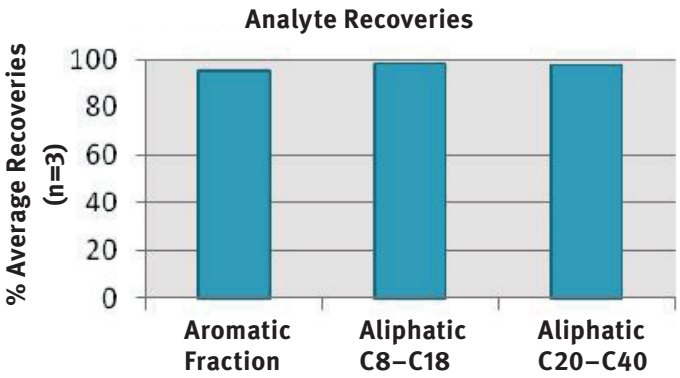

Figure 1. ISOLUTE® EPH provides high absolute recoveries (>90%) with excellent reproducibility (<2% RSD, n=3) for both Aromatic and Aliphatic fractions (Based on fractionation of 20ppm 16 aromatic/17 aliphatic component standard in Hexane).

Section 2.

Manual ISOLUTE® EPH Fractionation Methodology

Manual Method Overview SPE Column:
ISOLUTE® EPH-M, 5g/20 mL P/N 928-0500-E

Manual ISOLUTE® EPH SPE Procedure	
Sample	Soil or water extract in hexane or pentane.
Column Conditioning	Load column with 20 mL hexane[1] Allow to flow under gravity until last of the solvent reaches the top frit. At this point place aliphatic fraction collection vial under column.
Sample Loading	Apply hexane or pentane extract (up to 1 mL). Allow sample to flow under gravity until it reaches the top frit.
Aliphatic Elution	Load column with 12 mL of hexane and allow to flow under gravity. Continue to collect in the aliphatic fraction until flows cease.
Aromatic Elution	Elute column with 20 mL[2] of DCM and collect the aromatic fraction until flows cease.

[1]Pentane may be substituted for hexane in both the column conditioning and aliphatic elution steps to facilitate volume reduction at the blow down step.

[2]If a vacuum sample processing station is available, the aromatic elution solvent volume can be reduced to 15 mL. Modify aromatic elution step by allowing elution solvent to flow under gravity until it reaches the top frit, then apply vacuum, set at -5"Hg, for a maximum of 10 seconds). This is the only step where vacuum should be applied.

Section 3.

Selection of Surrogates and Internal Standard

A number of popular surrogates and internal standards are compatible with the ISOLUTE® EPH fractionation procedure. The use of o-terphenyl (aromatic surrogate), squalene (aliphatic surrogate) and 5-α-androstane (internal standard) have proved reliable. It is necessary to evaluate alternative surrogates to ensure they fall into the correct fraction.

Appendix

Reproducible Efficient Aliphatic/Aromatic Fractionation with Low Level of Extractables

Consistent, efficient fractionation without naphthalene carryover into the aliphatic fraction has been achieved by a combination of product and method optimization allied to effective manufacturing and QC procedures. Product purity, compatible with the non-selective GC-FID analysis, is defined by appropriate cleaning technology and associated QC checks. The fractionation efficiency of the automated ISOLUTE® EPH column is illustrated in Figures 2–4. The absence of naphthalene in the aliphatic fraction is one of the QC parameters used to control the activity of the ISOLUTE® EPH products.

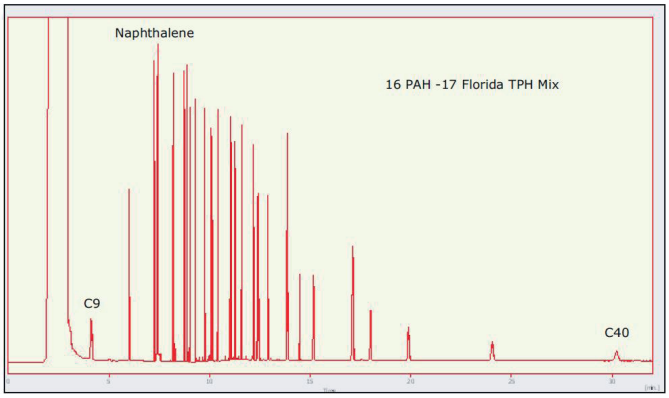

Figure 2.
Pre-fractionation chromatogram of 16 PAH, 17 Aliphatic Florida TPH mix.

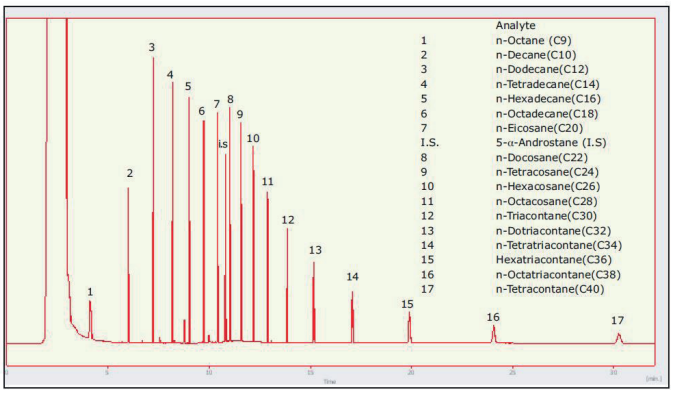

Figure 3.
Post-fractionation of PAH-TPH mix - Aliphatic Fraction.

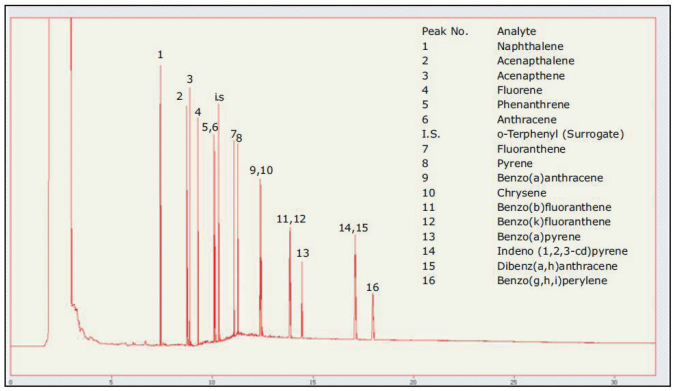

Figure 4.
Post-fractionation of PAH-TPH mix - Aromatic Fraction.

Ordering Information

Part Number	Description	Quantity
Columns for Automated EPH Fractionation		
928-0145-B	ISOLUTE® EPH 1.45 g/3 mL SPE Columns	50
Columns for Manual EPH Fractionation		
928-0500-E	ISOLUTE® EPH-M 5 g/20 mL SPE Columns	20

For more information on the Biotage® VacMaster™-10 and -20 Sample Processing Manifolds and the Biotage® Gravity Rack, please visit www.biotage.com.

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