RapidTrace Procedure for the Automated Fractionation of Extractable Petroleum Hydrocarbons (EPH) in Soil using ISOLUTE EPH SPE Columns

Application Note

Introduction

ISOLUTE EPH SPE columns and associated methodologies have been optimised to efficiently fractionate EPHs into aliphatic and polycyclic aromatic (PAH) fractions. The principle is similar to the approach taken by the Massachusetts Department of Environmental Protection (MADEP) and TPH criteria working group (TPHCWG) methods. However, compared to these methods, the ISOLUTE EPH fractionation column has been significantly reduced in size and the sorbent has been optimised in order to reduce solvent volumes and provide an automation compatible fractionation column. Automation of the fractionation process has been achieved without carryover of PAHs into the aliphatic fraction.

This Application Note describes the operating conditions for the automated fractionation of EPH into aliphatic and PAH fractions using the ISOLUTE EPH column in conjunction with the Caliper RapidTrace SPE automation system. For a more detailed description of the ISOLUTE EPH methodology, for both manual and automated fractionation methods, request Chemistry Data Sheet TN142.

Method Overview

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

SPE Procedure	
Sample	Soil extract in hexane or pentane
Column Conditioning	Hexane (6 mL)
Sample Loading	Apply hexane or pentane extract (1 mL) Collect column eluate (aliphatic fraction)
Aliphatic Elution	Hexane (1.5 mL) – add to aliphatic fractionfrom sample loading step
Aromatic Elution	Dichloromethane (2 mL)

RapidTrace settings:

- All solvent lines are first purged with appropriate solvent to prime and remove air from the system (see **Reagent Table** below for solvent details)
- Methodology is based on the fractionation of a 1ml sample extract (in pentane or hexane) which is accurately dispensed into the sample tube prior to analysis.
- Aliphatic fraction collected in Fraction 1, aromatic fraction collected in Fraction 2.

Biotage

Step	Source	Destination	Volume (mL)	Flow (mL)
Condition	Hexane	Organic waste	6.0	6.0
Rinse	Vent	Organic waste	2.0	6.0
Load	Sample	Fraction 1	1.0	2.0
Add to sample	Hexane	Cannula waste	0.3	2.0
Load	Sample	Fraction 1	0.4	2.0
Purge Cannula	Hexane	Cannula waste	2.0	6.0
Collect	Hexane	Fraction 1	1.4	3.0
Collect	Vent	Fraction 1	3.0	6.0
Purge Cannula	Hexane	Cannula waste	2.0	6.0
Collect	DCM	Fraction 2	2.0	3.0
Collect	Vent	Fraction 2	2.0	6.0
Purge Cannula	DCM	Cannula waste	4.0	30.0
Purge Cannula	Hexane	Cannula waste	6.0	30.0

Reagent Table

Line Number	
1	Hexane
2	Dichloromethane

ISOLUTE EPH Column Care and Use Guidelines

The ISOLUTE EPH columns have been designed to efficiently and consistently separate petroleum hydrocarbons into aliphatic and aromatic fractions. This has been achieved through optimisation of the EPH sorbent, sorbent activity and fractionation methodology. The ability of the column to achieve this fractionation is tested in QC.

To ensure the reliability of the fractionation process, the following points should be considered:

- ISOLUTE EPH columns should not be removed from the sealed foil bags until immediately before placing in the RapidTrace.
- Opened, partially used bags should be sealed and stored in a dessicator until required.
- The recommended Biotage methods for use with the manual and automated versions of the ISOLUTE EPH column specify column conditioning with hexane. Deviation from this recommendation, for example including a column conditioning step with dichloromethane followed by hexane, will result in incomplete aliphatic / aromatic fractionation, with breakthrough of polycyclic aromatic hydrocarbons into the aliphatic fraction.
- The Biotage EPH fractionation method specifies that the soil extract should be contained in hexane or pentane prior to loading onto the ISOLUTE EPH column. The addition or use of other more polar solvents in the final soil extract will result in a significant breakthrough of some polycyclic aromatic hydrocarbons into the aliphatic fraction.

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