Everything you ever wanted to know about EPH fractionation methods....

SPE Fractionation

Overview

- Fractionation tests must be performed on every new lot of silica gel cartridges. Accurate elution volumes must be determined and validated.
- The test mix must contain all aliphatic and aromatic hydrocarbon standards.
- Laboratory determined recoveries must be between 40-140% for aliphatic ranges and target PAH analytes except for n-nonane, which must be between 30-140%.

Method Blanks and Laboratory Control Samples (LCS)

Used to make sure that labs have a "good" extraction procedure. They measure sensitivity, contamination, and method reproducibility.

- Extracted with every batch or every 20 samples, whichever is more frequent.
- Matrix-specific (e.g., water, soil)
- Laboratory determined Relative Percent Difference (RPD) must be =25% for target PAH analytes and EPH hydrocarbon ranges.

Sample Contamination

Most SPE tubes are composed of polypropylene, a medical grade of plastic that is inert to most sample matrices. However, the tube material has been shown to leach plasticizers, specifically BHT, when exposed to halogenated solvents such as methylene chloride.

The laboratory must report the presence of this contamination in the associated range. Optionally, the laboratory may perform GC/MS analysis of the laboratory method blank extract to demonstrate that the contaminant in question is not a C11-C22 aromatic compound. Analysis of only the method blank is acceptable as long as the associated samples exhibit the same contaminant peak at the same retention time. If it is demonstrated that the contaminant is not a C11-C22 aromatic compound, it does not need to be included in the calculation. However, the laboratory must provide a discussion of the problem in the case narrative.

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GC Method

Overview

Retention windows for all compounds must be determined prior to initial calibration (ICAL) and every time a new GC column is installed.

- PAH resolution such that the valley between the two peaks is less than 25% of the average height of the two peaks.
- Resolve n-nonane (C9) from solvent front.
- Response ratio of C28 to C20 should be > 0.85.
- Surrogate and internal standards must be resolved from all aromatic and aliphatic standards.
- Naphthalene and n-dodecane in the aliphatic fraction must be adequately resolved
- Retention time windows must be updated with every CCAL

Initial Calibration (ICAL)

This is a curve that is run when a new column is first installed or when the system falls out of calibration. This usually happens after some dirty samples have been analyzed and the column deteriorates.

- Minimum of 5 point calibration curve: 1, 10, 50, 100, and 200ppm
- Low standard must be < reporting limit (RL).
- Relative Standard Deviation (RSD) should be <25% or correlation factor (r) based on a linear regression should be >0.99 for all compounds and ranges.
- Must contain all aliphatic and aromatic hydrocarbon standards.

Continuing Calibration Curve (CCAL)

This is a single injection of a mid-point level standard, which verifies quantitation still provides accurate data. Meaning, if you inject a 20ppm solution, the quantitation based on your calibration cure must confirm that value. It must be run every 24hr period or after you have run more than 20 samples, whichever comes first. Whenever the system fails to give accurate quantitation a new ICAL must be done.

- Concentration of the standard should be near the midpoint concentration of the curve
- Must contain all aliphatic and aromatic hydrocarbon standards
- Opening CCAL: %D or % drift must be <25% for all target PAH analytes and ranges except for n-nonane, which must be <30%.
- Closing CCAL: Up to four (4) compounds may exhibit a %D or % drift· >25 but < 40.
- Must meet GC performance standards.

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General Info

Internal Standards

These are used to monitor the system to make sure it is working properly. They are spiked in after the extraction procedure. If the internal standards start to look bad (area counts are high or low) then the system has changed and needs to be re-calibrated.

- Recommended internal standard for EPH analysis is 5-alphaandrostane. Alternatively, 1-Chloro-octadecane (COD) may also be as an internal standard for GC/MS analysis.
- Area counts should be between 50 and 200 % of the area counts in the associated continuing calibration (CCAL).

Surrogates

Used to measure the effectiveness of the extraction procedure. If you have low concentration of surrogates in relation to the internal standards, then you know something went wrong in the extraction procedure and it must be "re-extracted."

- Method Requires a Minimum of 2 extraction and 1 fractionation surrogate.
- Recommended extraction surrogates:
  - Chlorooctadecance
  - Ortho-terphenyl
- Recommended fractionation surrogate:
  - 2-bromonaphthalene
  - 2-fluorobiphenyl (optional).
- Recoveries must be between 40-140% for all surrogates.
- Laboratories are encouraged to develop their own in-house control limits, which should fall within the limits listed above.

What to Recommend

SPE

Primary: StrataTM EPH 5g/20mL tubes p/n 8B-S031-LEG

Secondary: If the customer would like to reduce the contamination by BHT placticizers, they may choose to try a Teflon coated tube, p/n AH0-7870.

GC

Primary: ZebronTM ZB-5 30x0.32x0.25, p/n 7HM-G002-11

Secondary: If the customer would like to improve run time, would suggest converting to a ZB-5ms column, which will improve resolution of key PAH, isomers. Suggested column: ZB-5ms 30x0.32x0.25 p/n 7HM-G010-11