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Priority Pollutant Sample Preparation, Extraction and Clean Up From Spiked Water and Solid Matrices with Internal, Volumetric and Standard Addition for Analysis by GC and GC/EI &NICI-MS

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Abstract

This manual was devised as a one semester programs of work for the Masters in Analytical Chemistry for one day per week. It was designed to demonstrate the analytical methodology and instrumentation utilized to proceed from the raw sample stage through sample preparation to the f nal analysis of reduced sample residues by capillary gas chromatography and GC/Mass Spectrometry. A variety of target analytes were employed to highlight the techniques to detect and quantify these compounds using external and internal standard addition, including selected priority pollutants , chloro-hydrocarbons (OCHs), polyaromatic hydrocarbons (PAHs) and chlorophenols. Liquid and solid matrices were employed to illustrate the analytical methodology, spiked with the target analytes to be monitored. The USEPA method 625 was adopted for liquid-liquid acid-base extraction of semi-volatile priority pollMS. Representative data analyses, calibration and quantit analytical methodology employed in the procedures for the laboratory manual are included.

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Calibration for GC/MS:

Use of internal standards:

Use of volumetric standards

(i) **PAHs:**

Note:

(ii) ***Aldrin, dieldrin and endrin***

$$Cx = \frac{A(rc).C(vs)}{RRF.A(vs).CF} \quad (3)$$

i. Recoveries

$$\% Recovery = \frac{C(x)}{C(rc)} x\ 100 \qquad \frac{A(x).A(vs)}{A(vs).A(rc)} x\ 100 = \frac{A(x)}{A(rc)} x\ 100 \quad (4)$$

Note

Important For the Analysis of Results Obtained

Experiments I and III

Introduction

Reagent, solvent and standards

A2. 20 mgL^{-1} of a mixture of naphthalene, anthracene, phenanthrene and chrysene

(NAPC) in hexane for calibration standards

Procedures for Experiments I and III

LLE Base/neutral component extraction (I)

[illegible] 500 [illegible] 1000 [illegible] (30). [illegible] - 1.0 [illegible] 2 [illegible] (1) [illegible]

b. [illegible]

c. [illegible] (i - [illegible])

d. [illegible]

e. [illegible]

[illegible] 500 [illegible] 1.0 [illegible]

[illegible] 2 [illegible] (1) [illegible] 400 [illegible]

(1) [illegible] 500 [illegible]

Preparation of reference standard solution for calibration:

a. [illegible] 1.0, 2.5, [illegible] 5.0 [illegible] 20 [illegible] (A2) [illegible] 2 [illegible] 10 [illegible] (2 [illegible] 2) [illegible] 10 [illegible]

b. [illegible] 1.0, 2.5, [illegible] 4.0 [illegible] 2 [illegible] (2) [illegible] 1.0 [illegible] 4 [illegible] (2) [illegible] 10 [illegible]

c. [illegible] 1.0, 2.0, [illegible] 4.0 [illegible] 2 [illegible] (2) [illegible] 1 [illegible] 4 [illegible] (2) [illegible] 10 [illegible]

c. [illegible] (%) [illegible]

Experiment III

Adaptation of USEPA Method 625 for the Analysis of Acidic Priority Pollutants Involving the Derivatization of Phenols Using Penta uorobenzyl Bromide

Chromatography of phenols without derivatization

Chromatography of phenols with derivatization

Method

Reaction Protocol

Prepare the following solution:

- A
- A il - 60
1. A
- **PFBBr- 10 µL**
- i - 10
1. 15
2. 15 il i 2 1
3. .

Note 0.5 200 400 $^{-1}$ 400 $^{-1}$.

Analysis

Results

GC-ECD:

a.

b. **Note:** /z 181 I- +

GC/NICI-MS: (-181)- I-

Experiment IV

Extraction of Organochlorine Compounds and Pyrethroids from Sediment/Soil samples

Introduction