

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 final 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 pollutants. Representative data analyses, calibration and quantification analytical methodology employed in the procedures for the laboratory manual are included.

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Calibration for GC/MS:

2. 10^{-1} A i P, 2

Use of internal standards:

Use of internal standards is a common method for quantification in GC/MS. It involves comparing the peak area of the analyte in the sample to the peak area of a known concentration of the same analyte in a standard solution. The internal standard is a compound that is added to the sample and standard solutions in a known amount. The ratio of the peak area of the analyte to the peak area of the internal standard is used to determine the concentration of the analyte in the sample. This method is particularly useful for samples that contain complex matrices, as the internal standard helps to compensate for any losses or variations in the sample preparation process. The internal standard should be chosen such that it is not present in the sample and has a similar chemical and physical properties to the analyte. The concentration of the internal standard should be constant in all samples and standards. The peak area of the internal standard should be measured in the same way as the peak area of the analyte. The ratio of the peak area of the analyte to the peak area of the internal standard is then used to determine the concentration of the analyte in the sample. This method is highly accurate and precise, and it is widely used in analytical chemistry.

Use of volumetric standards

(i) PAHs

Polycyclic aromatic hydrocarbons (PAHs) are a class of organic compounds consisting of two or more fused benzene rings. They are found in a wide range of natural and man-made sources, including fossil fuels, tobacco, and food. PAHs are known to be carcinogenic and mutagenic, and they are a major concern in environmental and occupational health. The analysis of PAHs in environmental samples is often done using GC/MS. The use of volumetric standards is a common method for quantification in GC/MS. It involves comparing the peak area of the analyte in the sample to the peak area of a known concentration of the same analyte in a standard solution. The concentration of the standard solution is determined by the volume of the standard solution and the amount of the analyte in the standard solution. This method is highly accurate and precise, and it is widely used in analytical chemistry.

Note:

Note: The peak area of the analyte in the sample should be compared to the peak area of the internal standard in the standard solution. The ratio of the peak area of the analyte to the peak area of the internal standard is used to determine the concentration of the analyte in the sample. This method is highly accurate and precise, and it is widely used in analytical chemistry.

(ii) Aldrin, dieldrin and endrin A P, 2

(100%) i P, 2

$$C_x = \frac{A(rc)-C(vs)}{RRF \cdot A(vs)-CF} \quad (3)$$

i. Recoveries

Internal standard (i), is added to the sample and the standard solution. The recovery of the internal standard is determined by comparing the peak area of the internal standard in the sample and standard solution. The recovery of the internal standard is calculated as follows:

$$\% \text{ Recovery} = \frac{C(x)}{C(rc)} \times 100 = \frac{A(x) \cdot A(vs)}{A(vs) \cdot A(rc)} \times 100 = \frac{A(x)}{A(rc)} \times 100 \quad (4)$$

Note

The recovery of the internal standard is determined by comparing the peak area of the internal standard in the sample and standard solution. The recovery of the internal standard is calculated as follows:

Important For the Analysis of Results Obtained

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Experiments I and III

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Introduction

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Reagent, solvent and standards

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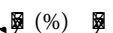

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A2. 20 mgL⁻¹ of a mixture of naphthalene, anthracene, phenanthrene and chrysene

(NAPC) in hexane for calibration standards

1. 2 mgL⁻¹ of a mixture of naphthalene, anthracene, phenanthrene and chrysene in hexane for calibration standards.

c.  i  (%)  i

... / I I- ...

Experiment III

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

I ... 5 ...

Chromatography of phenols without derivatization

- A ... 625 ...

Chromatography of phenols with derivatization

i ... / I I- ...

Method

i ...

Reaction Protocol

i ...

Prepare the following solution:

- A ... 0.5 ...

- A ... -60

1. A ...

- PFBBr- 10 µL

- i ... -10

1. ... 15 i ... 0.5 ... 0.5 ...

2. ... 15 i ... 0.5 ...

3. ...

i ...

Note ... 0.5 ... 200 ... 400 ...

Analysis

... / I I- ...

Results

... % ...

GC-ECD: ...

a. ...

b. **Note:** ...

GC/NICI-MS: ...

Experiment IV

Extraction of Organochlorine Compounds and Pyrethroids from Sediment/Soil samples

Introduction

I ...

Abstract: This paper describes the development and validation of a method for the determination of priority pollutants in spiked water and solid matrices. The method involves the use of internal, volumetric and standard addition techniques for sample preparation, extraction and clean up. The method was validated for accuracy, precision, sensitivity and specificity. The results show that the method is suitable for the determination of priority pollutants in spiked water and solid matrices.

