Effective Extraction Method for the Determination of Polycyclic Aromatic Hydrocarbons (PAHs) in Environmental Samples

SORNNARIN BANGKEDPHOL
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**Introduction**

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  - Spectrofluorometric method
  - HPLC

**Part I:** Determination of PAHs in ethanolic samples

- Qualitative analysis (synchronous scanning technique)

**Part II:** Quantitative analysis

- Optimization of extraction methods
  - Microwave extraction
  - Sonication
  - Soxhlet extraction

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**Conclusions**
Objectives

1. To study the optimum conditions for extraction of PAHs from sediment samples between microwave extraction and sonication.

2. To compare efficiency of extraction methods for extraction of PAHs from sediment samples between microwave extraction, sonication, and Soxhlet extraction.

3. To develop the spectrofluorometric method for qualitative and quantitative analysis of PAHs from extraction.
INTRODUCTION
PAHs consist of two or more fused benzene rings

PAHs are produced naturally by combustion processes, industrial processes and transport etc.

PAHs are pollutants in environment

PAHs can be polluted in many kinds of environmental samples

soil & sediment, river, plant, rain, ground water
Toxicity of PAHs

- Chronic health effect and carcinogenicity
- High bioaccumulation potential
- Low removal efficiency in treatment process
Chemical structure of the sixteen PAHs, which were identified by The Environmental Protection Agency (EPA) as priority pollutant.
Method for determination of PAHs in samples

1. Spectrofluorometric method

Jablonski diagram

- **S₀**: ground singlet state
- **S₁, S₂**: excited singlet state
- **T₁**: excited triplet state
- **IC**: internal conversion
- **ISC**: intersystem crossing
- **F**: fluorescence
- **C**: chemiluminescence
- **P**: phosphorescence

Photoexcitation or Chemiexcitation

- 10⁻¹⁵ sec.
- 10⁻⁹ - 10⁻⁷ sec.
- 10⁻³ - 10 sec.

VR
Synchronous Fluorescence Spectroscopic Technique

The excitation and emission monochromator of a spectrofluorometer are simultaneously scanned while maintaining a constant wavelength interval ($\Delta \lambda$) throughout the spectrum.
Condition of HPLC (EPA 8310)

- The injection volume: 40 µl
- Gradient elution: A = water, B = acetonitrile (50-100%B; 0-25 min, 100%B; 25-50 min)
- Analytical column: ChromSpher PAH
- Detector: photodiode array detector at 254 nm
- Column temperature: 25°C
Method for extraction

Microwave Extraction  Soxhlet Extraction  Sonication
1. Microwave Extraction

The Electromagnetic Spectrum

- Microwave oven
- Radar
- AM Radio
- FM Radio
- Microwave Oven
- X-Ray Machines

Molecular vibration
Outer-shell electron
Inner-shell electron

Molecular rotation

Common name of wave: Molecular rotation

Wavelength (in meters)

Size of a wavelength:
- Soccer Field
- House
- Baseball
- This Person
- Cell
- Bacteria
- Virus
- Protein
- Water Molecule

Outer-shell electron
Inner-shell electron
Ionic Conduction
Pressurized microwave-assisted extraction: closed-system

Localized superheating

Schematic of sample heating by microwaves
2. Soxhlet Extraction
3. Ultrasonic Extraction: Sonication

Operation frequency 30-40 KHz
Part I

Determination of PAHs in ethanolic samples by spectrofluorometric method
Part I

Synchronous Fluorescence Spectroscopic Technique: The Tool for Rapid Identification of PAHs in Liquid Samples

RESULTS
EXPERIMENT
DISCUSSION
Synthetic mixture of benzo[k]fluoranthene, fluorene and truxene

The synchronous spectrum of benzo[k]fluoranthene, fluorene and truxene at $\Delta\lambda = 20$ nm
Synthetic mixture of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene

The synchronous spectrum of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene at $\Delta \lambda = 6$ nm
Synthetic mixture of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene

The synchronous spectrum of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene at $\Delta \lambda = 20$ nm
Synthetic mixture of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene

The synchronous spectrum of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene at $\Delta \lambda = 95$ nm
Synthetic mixture of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene

The synchronous spectrum of benzo[k]fluoranthene, carbazole, chrysene, anthracene, acenaphthene and indeno[1,2,3,cd]pyrene at $\Delta \lambda = 110$ nm
Part I/II

Quantification of PAHs in synthetic sample by spectrofluorometric method
- Calibration graph: range 0.005-0.040 ppm

- Scanning: Excitation wavelength (Ex), Emission wavelength (Em)

<table>
<thead>
<tr>
<th>Compound</th>
<th>Ex (nm)</th>
<th>Em (nm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>chrysene</td>
<td>267</td>
<td>380</td>
</tr>
<tr>
<td>carbazole</td>
<td>233</td>
<td>356</td>
</tr>
<tr>
<td>acenaphthene</td>
<td>227</td>
<td>321</td>
</tr>
<tr>
<td>indeno(1,2,3-cd)pyrene</td>
<td>250</td>
<td>510</td>
</tr>
<tr>
<td>benzo(k)fluoranthene</td>
<td>307</td>
<td>407</td>
</tr>
<tr>
<td>anthracene</td>
<td>252</td>
<td>372</td>
</tr>
</tbody>
</table>
**Limit of detection (LOD):** \( y - y_B = 3S_B \)

<table>
<thead>
<tr>
<th>Concentration (ppm)</th>
<th>Intensity, ( y_i )</th>
<th>( \hat{y} ), ( y = 4381x - 0.012 )</th>
<th>( [y_i-y^\hat{}]^2 )</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.000</td>
<td>0.017</td>
<td>0.012</td>
<td>2.421X10^{-5}</td>
</tr>
<tr>
<td>0.005</td>
<td>21.626</td>
<td>21.893</td>
<td>0.071</td>
</tr>
<tr>
<td>0.010</td>
<td>41.656</td>
<td>43.798</td>
<td>4.588</td>
</tr>
<tr>
<td>0.020</td>
<td>92.319</td>
<td>87.608</td>
<td>22.1948</td>
</tr>
<tr>
<td>0.030</td>
<td>129.370</td>
<td>131.418</td>
<td>4.194</td>
</tr>
<tr>
<td>0.040</td>
<td>174.975</td>
<td>175.228</td>
<td>0.064</td>
</tr>
<tr>
<td></td>
<td></td>
<td></td>
<td>31.111</td>
</tr>
</tbody>
</table>

LOD of chrysene = 0.0019 ppm

<table>
<thead>
<tr>
<th>Concentration (ppm)</th>
<th>Intensity, ( y_i )</th>
<th>( \hat{y}, y=4381x-0.012 )</th>
<th>([y_i-\hat{y}]^2)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.000</td>
<td>0.017</td>
<td>0.012</td>
<td>2.421\times10^{-5}</td>
</tr>
<tr>
<td>0.005</td>
<td>21.626</td>
<td>21.893</td>
<td>0.071</td>
</tr>
<tr>
<td>0.010</td>
<td>41.656</td>
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<td>4.588</td>
</tr>
<tr>
<td>0.020</td>
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</tr>
<tr>
<td>0.030</td>
<td>129.370</td>
<td>131.418</td>
<td>4.194</td>
</tr>
<tr>
<td>0.040</td>
<td>174.975</td>
<td>175.228</td>
<td>0.064</td>
</tr>
</tbody>
</table>

Signal of LOD = \( a + 3S_{y/x} \)

\( a = \text{Intercept} = -0.012 \)

\( S_{y/x} = \{ \text{sum} \ (y_i - \hat{y})^2 / n-2 \}^{1/2}, \ n = 6 = 2.789 \)

Signal of LOD = \(-0.012 + 3(2.789) = 8.355\)

Therefore: LOD = \( (8.355+0.012)/4381 = 0.0019 \text{ ppm} \)
<table>
<thead>
<tr>
<th>PAHs</th>
<th>Limit of detection (LOD), ppm</th>
</tr>
</thead>
<tbody>
<tr>
<td>chrysene</td>
<td>0.0019</td>
</tr>
<tr>
<td>carbazole</td>
<td>0.0021</td>
</tr>
<tr>
<td>acenaphthene</td>
<td>0.0021</td>
</tr>
<tr>
<td>indeno(1,2,3-cd)pyrene</td>
<td>0.0017</td>
</tr>
<tr>
<td>benzo(k)fluoranthene</td>
<td>0.0012</td>
</tr>
<tr>
<td>anthracene</td>
<td>0.0024</td>
</tr>
</tbody>
</table>
### Quantitative Analysis

<table>
<thead>
<tr>
<th></th>
<th>Amount of PAHs (mg/l), 1&lt;sup&gt;st&lt;/sup&gt; replicate</th>
<th>Amount of PAHs (mg/l), 2&lt;sup&gt;nd&lt;/sup&gt; replicate</th>
<th>Amount of PAHs (mg/l), 3&lt;sup&gt;rd&lt;/sup&gt; replicate</th>
<th>Average the amount of PAHs (mg/l)</th>
<th>SD</th>
</tr>
</thead>
<tbody>
<tr>
<td>chrysene</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.000</td>
</tr>
<tr>
<td>carbazole</td>
<td>0.021</td>
<td>0.021</td>
<td>0.020</td>
<td>0.021</td>
<td>5.773X10^-4</td>
</tr>
<tr>
<td>acenaphthene</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.000</td>
</tr>
<tr>
<td>indeno[1,2,3-cd]pyrene</td>
<td>0.136</td>
<td>0.134</td>
<td>0.135</td>
<td>0.135</td>
<td>0.001</td>
</tr>
<tr>
<td>benzo[k]fluoranthene</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.020</td>
<td>0.000</td>
</tr>
<tr>
<td>anthracene</td>
<td>0.031</td>
<td>0.030</td>
<td>0.031</td>
<td>0.031</td>
<td>5.773X10^-4</td>
</tr>
</tbody>
</table>

**Hypothesis testing**

- **n < 30**: t-test
- **n ≥ 30**: Z-test
H\textsubscript{0}: \mu = 0.02
H\textsubscript{1}: \mu \neq 0.02

95% confidence limit: \alpha = 0.05

The critical values of [ t ] for a two-tailed test = 4.30

\[ t = \frac{(0.021-0.02)(3)^{1/2}}{5.773 \times 10^{-4}} = 2.000 \]

\[ t = 199.180 \quad \text{Reject } H\textsubscript{o} \]

\[ t = 31.999 \quad \text{Reject } H\textsubscript{o} \]
<table>
<thead>
<tr>
<th>Synthetic sample</th>
<th>1.0</th>
<th>1.0</th>
<th>1.0</th>
<th>1.0</th>
<th>1.0</th>
<th>1.0</th>
</tr>
</thead>
</table>

**Standard PAHs**
(5 ppm of anthracene or indeno[1,2,3,cd]pyrene)

<table>
<thead>
<tr>
<th>0.0</th>
<th>20.0</th>
<th>30.0</th>
<th>40.0</th>
<th>50.0</th>
<th>60.0</th>
</tr>
</thead>
</table>

indeno(1,2,3-cd)pyrene
anthracene

**Standard addition**
Standard addition of anthracene

Fluorescence intensity vs. Additional concentration of anthracene standard (ppm)

Equation: $y = 24675x + 265.18$

$R^2 = 0.9995$
<table>
<thead>
<tr>
<th>equation</th>
<th>$R^2$</th>
<th>Amount of PAHs, mg/l</th>
<th>real conc.</th>
</tr>
</thead>
<tbody>
<tr>
<td>$Y = 24675X + 265.180$</td>
<td>0.9995</td>
<td>0.011</td>
<td></td>
</tr>
<tr>
<td>$Y = 24727X + 264.350$</td>
<td>0.9996</td>
<td>0.010</td>
<td>0.010</td>
</tr>
<tr>
<td>$Y = 24709X + 262.770$</td>
<td>0.9996</td>
<td>0.010</td>
<td></td>
</tr>
<tr>
<td><strong>anthracene</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>$Y = 1265.2X + 25.303$</td>
<td>0.9997</td>
<td>0.020</td>
<td></td>
</tr>
<tr>
<td>$Y = 1294.6X + 25.343$</td>
<td>0.9996</td>
<td>0.020</td>
<td>0.020</td>
</tr>
<tr>
<td>$Y = 1264.3X + 25.264$</td>
<td>0.9987</td>
<td>0.021</td>
<td></td>
</tr>
<tr>
<td><strong>indeno(1,2,3-cd)pyrene</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Optimization of extraction methods using the spiked sediment sample

Part II
RESULTS
EXPERIMENT
DISCUSSION
Anthracene

Carbazole

Acenaphthene

Chrysene

Benzo(k)fluoranthene

Indeno(1,2,3,c,d)pyrene
Preparation of spiked sample

- Start with soil, grind to (100 mesh) sieve.
- Burn the soil.
- Store in a desiccator.

- Weigh 100 g of soil and add 6 PAHs.
- Shake for 2 days.
- Air-dry.

Shake at room temperature for 2 days.
Testing on PAHs adsorption on the spiked sediment sample

spiked sample 0.15 g

Shake:
- Water 30 ml, 10 min
- Ethanol 30 ml, 10 min

Filtering

Extraction: Sonication method, ethanol, 30 min
### Amount of PAHs: mg/g (n=3) equivalent of chrysene

<table>
<thead>
<tr>
<th></th>
<th>unwashed</th>
<th>water</th>
<th>ethanol</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.0045</td>
<td>0.0045</td>
<td>0.0022</td>
<td></td>
</tr>
<tr>
<td>0.0045</td>
<td>0.0044</td>
<td>0.0021</td>
<td></td>
</tr>
<tr>
<td>0.0045</td>
<td>0.0044</td>
<td>0.0021</td>
<td></td>
</tr>
<tr>
<td>0.0045</td>
<td>0.0044</td>
<td>0.0021</td>
<td></td>
</tr>
<tr>
<td>1.89X10^{-5}</td>
<td>2.15X10^{-5}</td>
<td>3.95X10^{-5}</td>
<td></td>
</tr>
<tr>
<td>0.42%</td>
<td>0.49%</td>
<td>1.86%</td>
<td></td>
</tr>
</tbody>
</table>

*Average, SD, RSD: Gaines et al. (2000)*

0.0045±0.42%  0.0044±0.49%  0.0021±1.86%
Extraction Methods

Microwave Extraction
- Extraction Solvent
- Extraction Time
- Temperature of Irradiation

Soxhlet Extraction
- Extraction Time

Sonication
- Extraction Solvent
- Extraction Time

(Conventional Method)
Microwave Extraction

- **Extraction Solvent**: methanol, acetonitrile, acetone, dichloromethane, hexane:acetone (3:2), cyclohexane:acetone (3:2)
- **Extraction Time**: 3, 5, 10, 15, 20, 25 min
- **Temperature of Irradiation**: 60%, 80%, 100%, 120%, 140%, 160% of boiling point
- **Fixed**: weight of synthetic sample ~0.25 g, extraction solvent = 20 ml

- **Procedure**:
  - Centrifuge 5,500 RPM (15 min)
  - Pipette 10 ml
  - Evaporate to 25 ml of ethanol

Quantitative analysis by spectrofluorometry
Fixed: extraction time = 10 min, temp. of irradiation = 80%BP for each solvent
Extraction solvent for microwave extraction

CH$_2$Cl$_2$

cyclohexane : acetone (3:2)
Extraction time of dichloromethane

- Fixed: extraction solvent, temp. of irradiation = 80%BP = 32 °C
Extraction time of cyclohexane:acetone (3:2)

- Fixed: extraction solvent, temp. of irradiation = 80%BP(acetone) = 45°C
Extraction solvent & time for microwave extraction

- **CH$_2$Cl$_2$**
  - Boiling point: 39.6°C
  - Time: 5 minutes

- **cyclohexane:acetone (3:2)**
  - Boiling point: 80.7°C
  - Time: 15 minutes

Boiling point
- CH$_2$Cl$_2$: 39.6°C
- cyclohexane: 80.7°C
- acetone: 56.1°C
Fixed: extraction solvent, extraction time = 5 minutes
- Fixed: extraction solvent, extraction time = 15 minutes
The optimum condition for microwave extraction

\[ \text{CH}_2\text{Cl}_2 \] 5 minutes 100% BP.

\text{cyclohexane:acetone} 15 minutes 140% BP.

<table>
<thead>
<tr>
<th>Boiling point</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>\text{CH}_2\text{Cl}_2</td>
<td>39.6°C</td>
</tr>
<tr>
<td>cyclohexane</td>
<td>80.7°C</td>
</tr>
<tr>
<td>acetone</td>
<td>56.1°C</td>
</tr>
<tr>
<td>acetone (140% BP)</td>
<td>79.0°C</td>
</tr>
</tbody>
</table>
**Sonication**

- **Extraction Solvent**: methanol, acetonitrile, acetone, dichloromethane, hexane:acetone (3:2), cyclohexane:acetone (3:2)
- **Extraction Time**: 10, 20, 30, 40, 50 min
- **Temperature of Irradiation**: No
- **Fixed**: weight of synthetic sample ~0.25 g, extraction solvent = 20 ml

- **Sonication Steps**
  - Centrifuge 5,500 RPM (15 min)
  - Pipette 10 ml
  - Evaporate to 25 ml of ethanol

**Quantitative analysis by spectrofluorometry**
- Extraction Solvent

**Fixed: extraction time = 20 minutes, temperature (measure = 41 °C)**
Fixed: extraction time = 20 min, temp. (measure = 33, 40.5, 43, 46, 51 °C)

BP of acetone = 56.1°C
The optimum condition for sonication

$\text{hexane:acetone (3:2) at 40 minutes}$
Dielectric constant

Dielectric constant = $E_0/E$

<table>
<thead>
<tr>
<th>Solvent</th>
<th>Dielectric constant at 25 °C</th>
</tr>
</thead>
<tbody>
<tr>
<td>methanol</td>
<td>32.66</td>
</tr>
<tr>
<td>acetone</td>
<td>20.56</td>
</tr>
<tr>
<td>acetonitrile</td>
<td>35.94</td>
</tr>
<tr>
<td>dichloromethane</td>
<td>8.93</td>
</tr>
<tr>
<td>hexane</td>
<td>1.88</td>
</tr>
<tr>
<td>cyclohexane</td>
<td>2.02</td>
</tr>
<tr>
<td>water</td>
<td>78.3</td>
</tr>
<tr>
<td>sulfuric acid</td>
<td>100</td>
</tr>
</tbody>
</table>

Soxhlet Extraction

- Extraction Time: 20 min, 40 min, 1, 3, 6, 12 and 24 hours
  (1 cycle time = 20 min)

- Fixed: weight of synthetic sample ~0.25 g, extraction solvent = CH₂Cl₂ 100 ml
  (Budzinski et al., 1993)

Evaporate to 25 ml of ethanol

Quantitative analysis by spectrofluorometry
The graphs depict the amount of PAHs (polycyclic aromatic hydrocarbons) in mg/g extracted over time (in hours) for different PAHs:

- Acenaphthene
- Benzo[k]fluoranthene
- Carbazole
- Chrysene
- Anthracene
- Indeno[1,2,3-cd]pyrene

The graphs illustrate how the amount of each PAH changes with time, indicating the extraction efficiency for each compound.
The optimum condition for soxhlet extraction

Type of solvent: $CH_2Cl_2$

Extraction time: 3 hr (24 hr)
Decomposition of PAHs on Soxhlet Extraction System
Part III

Recovery of the extraction methods
**Optimum Conditions**

**Microwave Extraction**
- Extraction Solvent: $\text{CH}_2\text{Cl}_2$, cyclohexane:acetone
- Extraction Time: 5 minutes, 15 minutes
- Temp of Irradiation: 100%BP, 140%BP

**Soxhlet Extraction**
- Extraction Solvent: $\text{CH}_2\text{Cl}_2$
- Extraction Time: 3 & 24 hours

**Sonication**
- Extraction Solvent: hexane:acetone
- Extraction Time: 40 minutes

CRM : LGC 6188

LGC : Laboratory of the Government Chemist
Chemical structure of PAHs, which were studied.
Conditions of HPLC (EPA method 8310)

- **Column**: Chromopher PAHs
- **Dimension**: 250 X 4.6 mm
- **Particle size**: 5 μm
- **Pore size**: 120 Å
- **Mobile phases**: solvent A = water, solvent B = acetonitrile
  - 50-100%B:0-25 min, 100%B:25-50 min
- **Temperature**: 25 °C
- **Flow rate**: 1 ml/min
- **Photodiode array detector**: 254 nm
Chromatogram of standard PAHs

- Naphthalene
- Pyrene
- Dibenz[a,h]anthracene
- Indeno[1,2,3-cd]pyrene
% Recovery of extracted PAHs in CRM of each extraction method by optimized condition

<table>
<thead>
<tr>
<th>PAHs</th>
<th>% Recovery: based on certified value of LGC6188 (average ± RSD)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>PMAE (cyclohexane:acetone [3:2])</td>
</tr>
<tr>
<td>acenaphthene</td>
<td>75.59±0.71</td>
</tr>
<tr>
<td>fluorene</td>
<td>107.52±11.01</td>
</tr>
<tr>
<td>phenanthrene</td>
<td>94.91±4.99</td>
</tr>
<tr>
<td>anthracene</td>
<td>112.89±2.50</td>
</tr>
<tr>
<td>fluoranthene</td>
<td>75.62±1.30</td>
</tr>
<tr>
<td>pyrene</td>
<td>94.35±0.67</td>
</tr>
<tr>
<td>benzo(a)anthracene</td>
<td>89.25±1.17</td>
</tr>
<tr>
<td>chrysene</td>
<td>102.95±2.01</td>
</tr>
<tr>
<td>benzo(b)fluoranthene</td>
<td>124.11±5.91</td>
</tr>
<tr>
<td>benzo(k)fluoranthene</td>
<td>176.71±1.55</td>
</tr>
<tr>
<td>benzo(a)pyrene</td>
<td>91.59±0.21</td>
</tr>
<tr>
<td>dibenzo(a,h)anthracene</td>
<td>78.22±2.69</td>
</tr>
<tr>
<td>benzo(g,h,i)perylene</td>
<td>109.92±3.39</td>
</tr>
<tr>
<td>indeno(1,2,3,cd)pyrene</td>
<td>87.81±1.50</td>
</tr>
</tbody>
</table>
Amount of individual extracted PAHs by each extraction method using selected condition.
Total amount of extracted PAHs by each extraction method using selected condition

% Recovery = 96.55 %
CONCLUSION
The synchronous scanning fluorescence technique can be used as a rapid method for qualitative analysis of PAHs. Furthermore, PAHs in samples can also be quantitatively analyzed using external calibration and standard addition method.

The suitable conditions of microwave assisted extraction, sonication and Soxhlet extraction were studied and optimized. The certified reference material was extracted by using optimum condition of each extraction method for recovery testing.

The microwave technique was a good alternative to extract PAHs in soil and sediment samples.
The optimum condition of microwave were 20 ml of cyclohexane:acetone (3:2) at 140% of BP of acetone for 15 minutes. When compared to the certified value, the total %recovery obtained by PMAE was 96.55 %.

The main advantage of PMAE were the reduction of the volume of extraction solvent, the reduction of decomposition of extracted PAHs, the reduction in extraction time.
THANK YOU
Test method for HPLC
Results

Amount of CRM extracts (mg/g) prepared by different procedures (n=3)

- **LGC6188 mg/kg**
- **MW cyclohexane:acetone**
- **MW dichloromethane**
- **sonication**
- **soxhlet 24 hr**
- **soxhlet 3 hr**

The graph shows the amount of PAHs mg/g for various procedures and samples. The x-axis represents different PAHs, and the y-axis shows the amount of PAHs in mg/g. The legend indicates the different extraction methods used.
% Recovery of CRM extracts prepared by different procedures (n=3)

- LGC6188 mg/kg
- MW cyclohexane:acetone
- MW dichloromethane
- Sonication
- Soxhlet 24 hr
- Soxhlet 3 hr

% Recovery

Compounds:
- Naphthalene
- Acenaphthene
- Fluorene
- Phenanthrene
- Anthracene
- Fluoranthe
- Pyrene
- Benzo(a)anthracene
- Fluoranthene
- Pyrene
- Benzo(b)fluoranthene
- Benzo(k)fluoranthene
- Benzo(a)pyrene
- Dibenzo(a,h)anthracene
- Benzo(g,h,i)perylene
- Indeno(1,2,3,c,d)pyrene

Extraction solvents:
- MW cyclohexane:acetone
- MW dichloromethane
- Sonication
- Soxhlet 24 hr
- Soxhlet 3 hr