The Hong Kong Polytechnic University
Department of Civil and Environmental Engineering
Water and Waste Laboratories

Laboratory Worksheet C15 : **Determination of PCB contect in different soil samples by Soxhlet Extraction**.

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Objective : To determine of PCB content in different soil samples by Soxhlet Extractor and G.C.

Introduction :

Extraction using organic solvent allows quantitative separation and determination of organic pollutants from soil. The most widely used technique is that developed by the German chemist Franz Soxhlet (1848-1927). The sample is contained in a porous cellulose or glass fiber extraction thimble. The sample is then immersed in a selected solvent that will be condensed by an overhanging condenser. The sample is refluxed for a period long enough to extract the organic contents from it. During reflux, the solvent with the extracted substances is drawn to the distillation vessel by an automatic siphon. The thimble retains the fraction of the substances not soluble in the solvent. One important point is that the target substance to be extracted must not be volatile at the boiling temperature of the selected solvent.

Organic contaminants can come from the following industries :

Oil installations.
Gas works
Power plants.
Shipyards or boatyards.
Chemical manufacturing or processing plants.
Steel mills and metal works.
Car repairing or dismantling workshops.

Apparatus :

- Thimbles.
- Glass wool.
- Balance.
- Oven at 60^{o}C or Desiccator.
- Soxhlet Extractor, semi-automatic.
- Gas Chromatography (G.C.).

Materials :

- Soil samples.
- Iso-Octane.

Procedure :

1. Air dry, grind and homogenize the soil samples.
2. Weigh 5 g of each prepared sample into extraction thimble.
3. Cover each thimble with glass wool plug.
4. Put the thimbles each into an extract collecting vessel.
5. Measure the initial dry weight of the vessel and thimble.
6. Add the solvent until most part of the thimble is immersed (in this case 50 mL).
7. Introduce the thimble into the extraction equipment
8. Close the extraction unit, start the cooling water flow and begin heating.
9. When the solvent starts to boil, immerse the thimble into the solvent by placing the slider into “Immersion” position.

10. Let the thimble to “cook” for 15-60 minutes, place the slider into “Washing” position.

11. Let it reflux for a further 30 to 60 minutes, stops heating and close the stopcock

12. Let the extractor cool to room temperature.

13. Take out the extract collecting vessel and measure its final weight.

14. Make up the volume of extraction solvent to 50 mL and measure PCB content in the extraction solvent by G.C.

Questions :

1. What are the common methods for carrying out the remediation of oil contamination?

2. What are the sampling strategy and what are the analytical and monitoring strategy for remediation projects?

3. Compare the PCB concentrations among two soil samples. (In case G.C. measurement is not available, use the enclosed data for analysis.)

Sample data :

Sample ID	Dry mass, g	Initial mass, g (soil + thimble)	Final mass, g (soil + thimble)	PCB content, µg/L
Nai Chung	5.0231	12.5561	12.3061	35.91775
Fanling	5.1366	12.5246	12.0742	59.05367

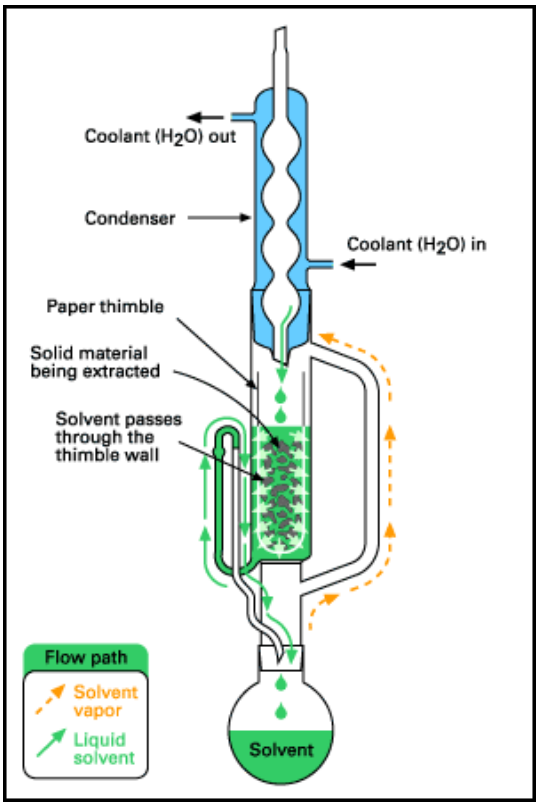

Fig. 1 Soxhlet Extractor Setup.

Data File C:\HPCHEM\2\DATA\KCY\NC2A(1).D Sample Name: NC2a(1)

Nai Chung 2a(1) Brij 35

injection= 1ul

```
==========================================================================
Injection Date    : 3/31/00 4:03:29 PM
Sample Name       : NC2a(1)
Acq. Operator     : JOANNA                          Vial :   2

Acq. Method       : C:\HPCHEM\2\METHODS\PCB.M       Inj Volume : Manually
Last changed      : 3/30/00 4:19:01 PM by JOANNA
Analysis Method   : C:\HPCHEM\2\METHODS\PCB.M
Last changed      : 4/10/00 11:28:21 AM by cmfan
                    (modified after loading)
Flow rate: 40.0 ml/min
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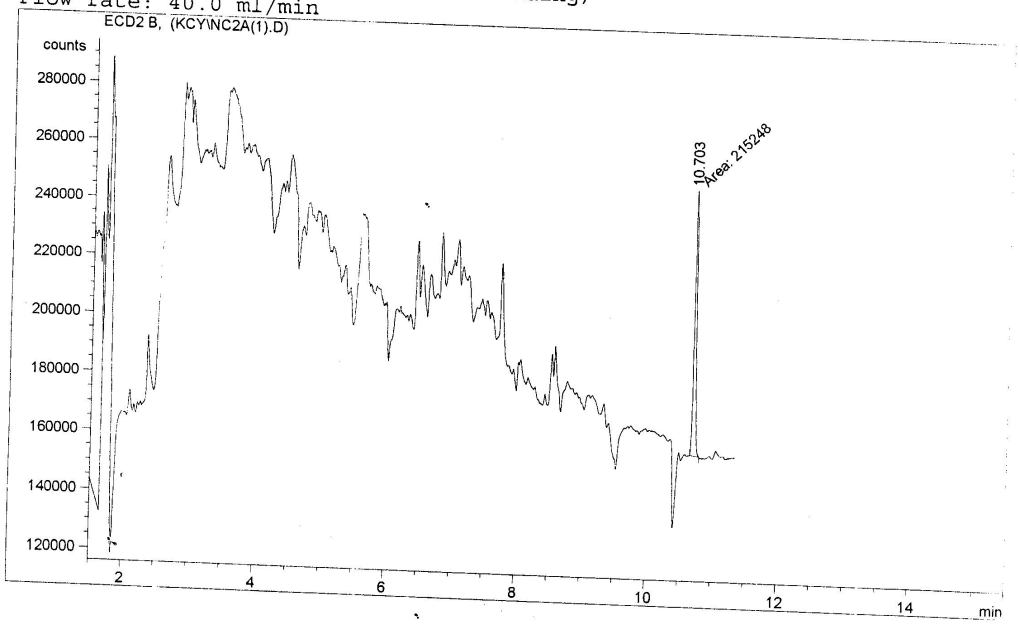


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==========================================================================
                        External Standard Report
==========================================================================

Sorted By             :      Signal
Calib. Data Modified  :      Thursday, March 30, 2000 11:16:11 AM
Multiplier            :      1.0000
Dilution              :      1.0000

Signal 1: ECD2 B,

RetTime  Type      Area       Amt/Area     Amount    Grp   Name
 [min]           counts*s                  [ug/L]
-------|------|----------|----------|----------|--|-----------------
 10.703 MM   +  2.15248e5  1.66867e-4   35.91775      Hexachloroinated Biphe

Totals :                                  35.91775

 Results obtained with enhanced integrator!
==========================================================================
                        *** End of Report ***

5890GC 4/10/00 11:31:57 AM cmfan                          Page 1 of 1
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Chart 1. Chromatograph of Nai Chung PCB analysis.

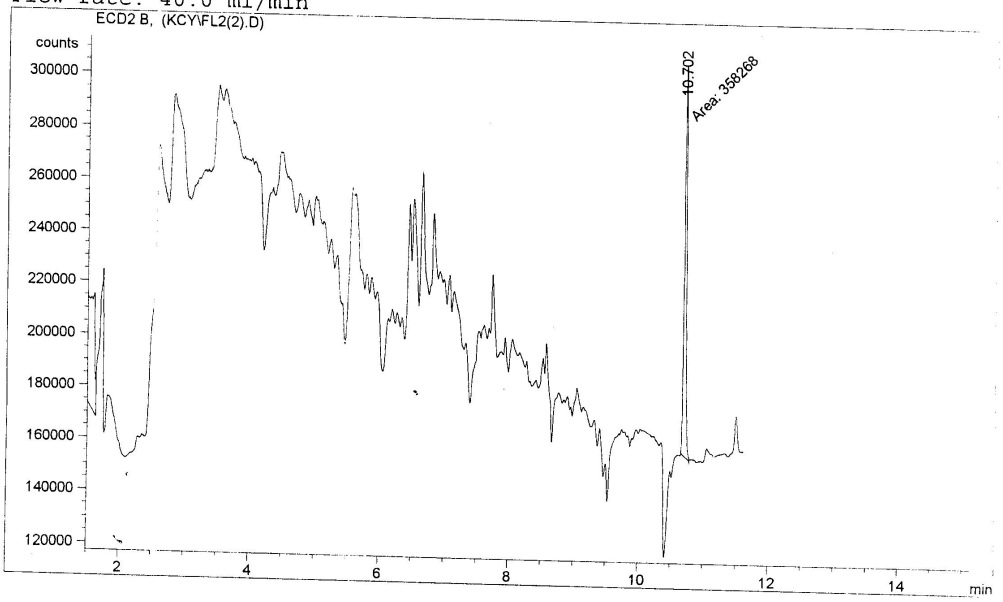
Data File C:\HPCHEM\2\DATA\KCY\FL2(2).D Sample Name: FL2(2)

Fanling2(2) Brij 35

injection= 1ul

```
==========================================================================
Injection Date   : 3/31/00 4:51:34 PM
Sample Name      : FL2(2)
Acq. Operator    : JOANNA                                  Vial :   2

                                                  Inj Volume : Manually
Acq. Method      : C:\HPCHEM\2\METHODS\PCB.M
Last changed     : 3/30/00 4:19:01 PM by JOANNA
Analysis Method  : C:\HPCHEM\2\METHODS\PCB.M
Last changed     : 4/10/00 11:28:21 AM by cmfan
                   (modified after loading)
Flow rate: 40.0 ml/min
```

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==========================================================================
                        External Standard Report
==========================================================================

Sorted By              :      Signal
Calib. Data Modified   :      Thursday, March 30, 2000 11:16:11 AM
Multiplier             :      1.0000
Dilution               :      1.0000

Signal 1: ECD2 B,

RetTime  Type      Area      Amt/Area     Amount    Grp   Name
 [min]          counts*s                 [ug/L]
-------|------|----------|----------|----------|--|------------------
 10.702 MM   +  3.58268e5  1.64831e-4   59.05367     Hexachloroinated Biphe

Totals :                                 59.05367

 Results obtained with enhanced integrator!
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                          *** End of Report ***
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Chart 2. Chromatograph of Fanling PCB analysis.