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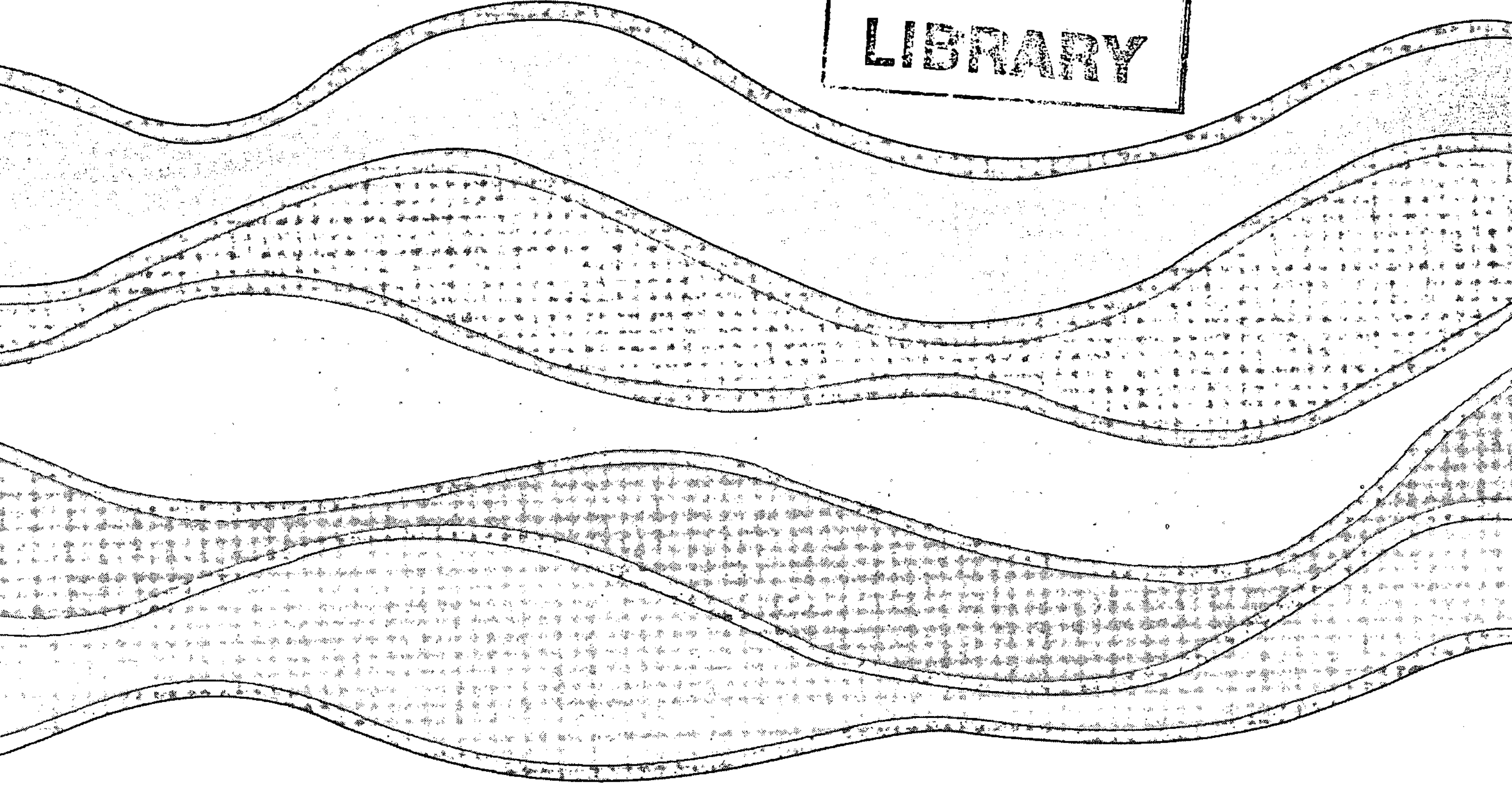

A NEW AND IMPROVED METHOD FOR THE DETERMINATION OF CHLOROBENZENES AND HEXACHLORO-1,3-BUTADIENE IN SEDIMENTS USING SUPERCRITICAL CARBON DIOXIDE EXTRACTION

H.B. Lee, T.E. Peart and R.L. Hong-You

NWRI Contribution No. 92-08

A NEW AND IMPROVED METHOD FOR THE DETERMINATION OF CHLOROBENZENES AND HEXACHLORO-1,3-BUTADIENE IN SEDIMENTS USING SUPERCRITICAL CARBON DIOXIDE EXTRACTION

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MANAGEMENT PERSPECTIVE

In many Great Lakes monitoring programs, it is required to analyze sediment samples for the determination of chlorobenzenes and hexachlorobutadiene. They are recognized as priority pollutants in Canada and the United States. The extraction procedures of these samples used to be time-consuming and produce large quantities of waste solvent which created a disposal problem. These problems can be overcome by replacing solvent extraction with the newly developed supercritical fluid extraction technique. The improved method is cost efficient and environmentally friendly because it only uses a small fraction of the time and solvent required by the traditional methods.

SOMMAIRE À L'INTENTION DE LA DIRECTION

Plusieurs programmes de surveillance des Grands Lacs nécessitent le dosage des chlorobenzènes et de l'hexachlorobutadiène des sédiments. Ces composés font partie des polluants d'intérêt prioritaire au Canada et aux États-Unis. Avec les méthodes employées jusqu'ici, l'extraction prend beaucoup de temps et génère d'importantes quantités de solvant usé, dont l'élimination pose des problèmes. Pour résoudre ceux-ci, on peut remplacer l'extraction au solvant par la nouvelle technique au fluide supercritique. La méthode améliorée est économique et écologique, car elle prend très peu de temps et ne nécessite pas beaucoup de solvant par comparaison aux méthodes classiques.

ABSTRACT

A method for the determination of chlorobenzenes and hexachlorobutadiene in sediment using a supercritical fluid extraction (SFE) technique was developed. Among the many extraction conditions evaluated, the best recovery of these chlorinated compounds was obtained by using an extraction temperature of 80°C and carbon dioxide modified by a 1:1 mixture of methanol and dichloromethane. Under the optimized conditions, an extraction time of only 25 minutes was needed to produce chlorobenzenes and HCBD results comparable to seven hours of Soxhlet extraction on a naturally contaminated sediment reference material. Cleanup of the SFE extract was performed on a miniature Florisil column with pentane as the eluting solvent. Quantitation was performed by electron capture gas chromatography with a DB-1 capillary column for all compounds except the dichlorobenzenes. The latter were analyzed by a mass selective detector. The absolute recovery for most chlorinated compounds from sediment samples fortified to 25 and 250 ng/g was between 75 and 85%.

KEY WORDS: Chlorobenzenes, hexachloro-1,3-butadiene, sediment, supercritical fluid extraction, SFE

RÉSUMÉ

On a mis au point une méthode de dosage des chlorobenzènes et de l'hexachlorobutadiène (HCBD) des sédiments dans laquelle on utilise une technique d'extraction par fluide supercritique (EFS). On a évalué de nombreuses conditions d'extraction : c'est avec l'extraction à 80°C au dioxyde de carbone modifié au moyen d'un mélange de méthanol et de dichlorométhane 1/1 qu'on a le plus récupéré de ces composés chlorés. En conditions optimales, avec des sédiments de référence naturellement contaminés, une extraction de 25 minutes seulement permet de récupérer des quantités de chlorobenzènes et de HCBD comparables à ce qu'on obtient par une extraction de 7 heures au Soxhlet. Pour purifier l'extrait EFS, on a employé une colonne de Florisil miniature et du pentane comme éluant. Pour le dosage, on a fait une chromatographie en phase gazeuse avec détection par capture d'électrons sur colonne capillaire DB-1 pour tous les composés, à l'exception des dichlorobenzènes. Pour ces derniers, on s'est servi de la spectrométrie de masse comme mode de détection. Pour la plupart des composés chlorés des échantillons de sédiments enrichis à 25 et 250 ng/g, le taux de récupération absolue se situait entre 75 et 85 %.

MOTS-CLÉS : Chlorobenzènes, hexachlorobuta-1,3-diène, sédiments, extraction par fluide supercritique, EFS.

INTRODUCTION

Chlorobenzene is manufactured by a liquid-phase chlorination of benzene in the presence of ferric chloride as a catalyst. It was used in the past to produce phenol, aniline and DDT. Nowadays, chlorobenzene is mainly used as a solvent, and to produce chloronitrobenzenes as intermediates for dyes, herbicides and insecticides. Other chlorobenzenes are produced in small quantities as side products in the chlorination process. Hexachlorobenzene has been used as a fungicide in Canada. Hexachloro-1,3-butadiene (HCBD) is primarily utilized as a solvent for elastomers, although it has also been used as a transformer fluid. Chlorobenzenes and HCBD have moderate acute toxicity to fish and mammals while HCBD is a suspected carcinogen. Many of these compounds are classified as priority pollutants in both Canada and the United States [1].

Since chlorobenzenes and HCBD are persistent in the environment, their residues are readily found in various areas of the Great Lakes Basin. For example, these compounds have been reported at environmentally significant levels in water, sediment and biota samples collected from Lake Ontario, Lake Erie and the connecting channels such as the Niagara and St. Clair Rivers [2 to 5]. Because of their high octanol/water partition coefficients, the heavier chlorobenzenes tend to bioaccumulate in fish [6,7] and they were also found in herring gull eggs along with other chlorinated insecticides [8].

Due to their low solubilities, chlorobenzenes are readily adsorbed and accumulated in lake and river sediments, producing elevated levels of these toxic compounds. In the past, the Soxhlet technique [9] and, to a smaller extent, steam distillation [10] were used for the extraction of chlorobenzenes from sediments. Both techniques are time-consuming and the Soxhlet extraction also produces a large volume of waste solvent that can create a disposal problem. Since chlorobenzenes and HCBD are relatively volatile, they can easily be lost in the concentration step of the Soxhlet extracts if the evaporation is not carried out properly.

In the last few years, there are many successful applications of supercritical fluid extraction (SFE) to environmental samples. These include the extraction of PCBs [11 to 13], PAHs [13,14], pesticide residues [15], chlorophenols [15,16], and resin and fatty acids [17] from sediment or soil and chlorinated dioxins and furans in fly ash samples [18] using supercritical carbon dioxide, nitrous oxide or freon-22. Although the most popular, carbon dioxide is the weakest solvent among the three supercritical fluids. However, the addition of suitable modifiers can greatly enhance the extractability of many polar organic compounds. In this report, we present a rapid, cost-efficient and environmentally friendly method using supercritical carbon dioxide for the determination of chlorobenzenes and HCBD in sediments.

EXPERIMENTAL

Reagents and chemicals

Chlorobenzenes, 1,3,5-tribromobenzene and HCBD were either obtained from Aldrich Chemicals Co. (Milwaukee, Wisconsin, USA) or from USEPA. 1,4-Dichlorobenzene-d_4 was a product of MSD Isotopes (Pointe Claire, Quebec, Canada). ECD grade supercritical carbon dioxide was supplied by Scott Specialty Gases (Troy, Michigan, USA). Distilled-in-glass grade solvents were purchased from Burdick and Jackson (Muskegon, Michigan, USA).

SFE module

All SFE was performed with the Hewlett-Packard (HP) 7680A SFE module which was controlled by an Intel 80386-class personal computer and dedicated software operating under the Windows-286 environment for multitasking purpose. The HP 7680A employs a thermostated, variable diameter restrictor nozzle, instead of a fixed diameter fused silica capillary restrictor as in other extractors, to depressurize the supercritical fluid [19]. In this case, the nozzle allows instant depressurization of the carbon dioxide, and at the same time permits the decoupling of flow and pressure; thus, the pressure and flow rate of the extraction fluid can both be set independently. It also eliminates the possibility of plugging the flow in the event of a frozen restrictor and enables the use of flow rates as high as 4 mL/min for quicker extractions. The SFE software was designed for multiple extraction steps, and user selectable extraction chamber temperature, density and flow rate of the extraction fluid. At the same time, it allowed for the selection of static and dynamic extraction times, and temperatures for the nozzle and trap during the extraction and elution stages, as well as the flow rate and volume of the rinse solvent. The operation of the SFE module is fully automated from the point where the sample thimble is placed inside the extraction chamber and on.

Chromatographic analysis

Chromatographic separation of chlorobenzenes and HCBD by several capillary column has been discussed before [20,21]. In this work, instrumental analysis of sediment extracts for the above chlorinated compounds with the exception of the dichlorobenzenes was carried out by a HP 5890 Series II GC equipped with an electron capture detector and a 30 m x 0.25 mm ID DB-1 fused silica capillary column (J&W Scientific). One μL splitless injections were made by the HP 7673A autosampler. Chromatographic data were processed by the HP 3365 ChemStation software. The injection port and detector temperatures were 250 and 300°C, respectively. The temperature program used was as follows: initial oven temperature, 60°C, hold for 0.75 min, programming rate, 5°C/min to 150°C, then 10°C/min to 250°C, hold at final temperature for 15 min. Splitless time was 0.75 min. Carrier gas was hydrogen and the column head pressure was 105 kPa. Detector make-up gas was argon-methane (95:5 mixture) at 30 mL/min.

Dichlorobenzenes were analyzed by a mass selective detector (MSD) interfaced to a HP 5880A GC using a 30 m x 0.25 mm ID Supelco SPB-5 column and the above temperature program. The MSD was operated in the selected ion monitoring (SIM) mode for masses m/z 146 and 148 (native dichlorobenzenes) and m/z 152 (1,4-dichlorobenzene-d_4, internal standard). Electron energy and electron multiplier voltage of 70 eV and 2000 V, respectively, were used.

Extraction procedure

Supercritical carbon dioxide extraction of the sediment was performed with the 7.0 mL stainless steel thimbles and the octadecylsilane (ODS) trap. The thimble was filled with, in the following order, 200 mg of Celite, 1.00 g of the sediment sample, and another 200 mg of Celite. Fifty μL of a 1 ppm solution of 1,3,5-tribromobenzene in methanol (surrogate standard), 100 μL of water and 500 μL of the modifier (see later Discussion) were introduced directly onto the sediment sample. The thimble was shaken for one min on a vortex mixer after addition of each liquid. A 5 min static extraction followed by a 20 min dynamic extraction were performed at a chamber temperature of 80°C with a carbon dioxide density of 0.5 g/mL (approximate pressure 168 bar or 17.1 MPa) and a flow rate of 2 mL/min. The extract was deposited onto an ODS trap which was maintained at 15°C with cryogenic CO_2 during the extraction stages. Upon completion of extraction, the chlorobenzenes were eluted from the trap by heating to 45°C and elution with 1.2 mL of iso-octane. The entire extraction procedure took approximately 40 min.

Cleanup procedure

The iso-octane extract was cleaned up on a 5 cm activated Florisil column packed in a 20 x 0.7 cm I.D. disposable Pasteur pipet. After a pre-elution with 5 mL of pentane, the SFE extract was applied to the column and the chlorobenzenes and HCBD were eluted with another 10 mL of pentane. This fraction was then evaporated in a 40°C water bath to 1.0 mL under a gentle stream of nitrogen. The extract was then shaken with a drop of mercury to remove elemental sulfur and sulfur-containing compounds. This step was repeated until the mercury remained shiny. The extract was then analysed by GC-ECD. For GC-MSD work, a 50 μL aliquot of 1,4-dichlorobenzene-d_4 was added to the extract as an internal standard prior to analysis.

RESULTS AND DISCUSSION

A freeze-dried Lake Ontario sediment reference material naturally contaminated with chlorobenzenes and HCBD was used for the development and optimization of the SFE conditions. Levels of the chlorinated contaminants in this sample, previously established by replicate Soxhlet extractions [21], were used to determine the relative percentage recoveries for the SFE results under various extraction conditions.

There are many factors that can affect the extraction efficiency in SFE extractions. The major ones include the choice of supercritical fluid, use of modifier, extraction chamber temperature, extraction fluid density (or pressure) and flow rate, extraction times, sample and extraction vessel size, the choice of trap material and eluting solvent as well as trap temperature, etc. Many of these factors are inter-related and optimal recovery of organics from a sample can be obtained from different combination of the above conditions. Since the HP 7680A is designed to be used with non-modified and modified supercritical carbon dioxide only, other supercritical fluids were not evaluated. The use of stainless steel traps were also not examined since traps with reversed-phase sorbents such as ODS have been shown to be highly efficient for the collection of a wide variety of organics in off-line SFE systems, particularly for the more volatile compounds [12]. In this work, we have evaluated the effects of the modifier, the extraction chamber temperature, the extraction fluid density and flow rate, and the extraction time, since they tend to have a greater impact on the recovery of chlorobenzenes and HCBD from sediments.

Effect of modifiers

Initially, extraction of chlorobenzenes was performed by using non-modified supercritical carbon dioxide. Since only ca. 70% of chlorobenzenes could be recovered with carbon dioxide with a density of 0.8 g/mL, the highest density available for the extractor at an extraction chamber temperature of 80°C, the use of modified carbon dioxide was deemed necessary in order to pursue quantitative recoveries. Since carbon dioxide of lower densities produced even lower recoveries, we used a density of 0.5 g/mL in the following experiments so that the effect of various modifiers on the recovery of chlorobenzenes from sediments would be amplified. For simplicity, only the recoveries of the most abundant chlorobenzene in the sediment from each homologous series are summarized in Figure 1. They are 1,4-dichloro-, 1,3,5-trichloro-, and 1,2,4,5-tetrachlorobenzenes in addition to penta- and hexa- chlorobenzenes and HCBD. Direct introduction of 500 μL of dichloromethane or methanol to the sample as a modifier for carbon dioxide improved the recovery of all chlorobenzenes from 70 to about 80% of the Soxhlet values, with methanol being the better modifier. The use of a 1:1 mixture of methanol and dichloromethane further increased the recovery of all chlorobenzenes and HCBD to ca. 90%. The highest recovery of these compounds was readily obtained by using the same mixed modifier with a 10% moisture content in the sediment sample. However, it was also noted that a further increase in moisture content of the sediment to 30% caused a 15% drop in the recovery of all chlorobenzenes and HCBD; therefore SFE of wet sediment is not recommended.

Effect of extraction chamber temperature

Using carbon dioxide at a density of 0.5 g/mL and the 1:1 mixture of methanol and dichloromethane as modifier, the recovery of chlorobenzenes and HCBD was examined at various extraction temperatures. As shown in Figure 2, a low extraction chamber temperature such as 40 or 50°C was unfavourable for the extraction of all compounds of interest, especially di- and tri- chlorobenzenes and HCBD. Since the best recovery for all compounds was invariably obtained at 80°C, the maximum operating temperature for the HP 7680A, all subsequent extractions of chlorobenzenes from sediments were carried out at this temperature.

Effect of carbon dioxide density

The effect of extraction fluid density on the recovery of chlorobenzenes and HCBD was also evaluated. Using the best modifier and a chamber temperature of 80°C, recovery of the organics obtained at carbon dioxide densities of 0.15, 0.2, 0.3, 0.5, and 0.8 g/mL is depicted in Figure 3. Within experimental errors, there was no difference in the recoveries of di- and tri- chlorobenzenes and relatively little effect on HCBD results among the various densities of supercritical carbon dioxide used. However, carbon dioxide with a density less than 0.3 g/mL was unable to provide optimal recovery for the higher molecular weight chlorobenzenes. Meanwhile, the results indicated that an extraction fluid density of either 0.5 or 0.8 g/mL produced the best recovery of all chlorobenzenes and HCBD from sediments. Since higher density carbon dioxide produced more coextractives such as humic substances, pigments, and PAHs, carbon dioxide of 0.5 g/mL was chosen for the rest of this work.

Other factors

After the modifier, extraction temperature and carbon dioxide density were optimised for the extraction of chlorobenzenes and HCBD, other factors affecting the recovery were also evaluated. The extraction time also played an important role on the recovery of chlorobenzenes. While the static extraction time had been kept constant at 5 min for the entire work, the recoveries obtained by using 5, 10, 15, 20, and 30 min of dynamic extraction times were compared. Although there was no significant difference in chlorobenzene results between 15 and 30 min of dynamic extraction, shorter times tended to produce incomplete extraction. Average recoveries of between 65 and 85% for all compounds were observed if the dynamic extraction time was reduced to 5 and 10 min, respectively. There was also no detectable difference in the chlorobenzenes and HCBD results when the extraction fluid flow rate was reduced from 2 to 1 mL/min while the dynamic extraction time was extended from 20 to 40 min, indicating that the trapping efficiency was satisfactory at the higher flow rate.

Cleanup of SFE extracts

In addition to the chlorinated compounds of interest, the SFE extract also contained other coextractives that could interfere with the GC analysis with an electron capture detector. In comparison to soxhlet extraction, SFE was more selective and the sediment extract was much lighter in colour presumably due to less coextracted pigments, humic substances, polynuclear aromatic hydrocarbons and other organics. Thus, a less stringent cleanup procedure can be used. In our work, chlorobenzenes and HCBD were eluted from a miniature (5 cm), fully activated Florisil column with pentane while the more polar coextractives in the sediment extracts remained on the column. Pentane instead of other hydrocarbon solvent was used since the former has a lower boiling point and thus will minimize losses in the evaporation step. After concentration, the sample was treated with mercury to remove sulfur and sulfur-containing compounds which were coeluted with the chlorobenzenes in the cleanup step. If the sediment is also contaminated by PCBs, they are also extracted and eluted in the same cleanup fraction as the chlorobenzenes. In this case, the analytical column must be heated to a higher temperature (e.g. 280°C) to eliminate the less volatile PCBs from causing interference in the next analysis. Octachlorostyrene, another persistent contaminant found in many Great Lakes samples, was also present in our reference material and was coextracted with the other chlorobenzenes and HCBD. However, quantitation of this compound was unsuccessful by the ECD procedure because of interference coming from the blank of the supercritical carbon dioxide which could not be removed by the Florisil cleanup. Owing to the low sensitivity of the mass spectrometer in electron impact mode toward the octachloro compound, MSD analysis of octachlorostyrene was also not attempted.

Method performance

The performance of the SFE procedure has been evaluated by recovery experiments on both spiked and naturally contaminated samples. For sediment samples spiked at 250 and 25 ng/g levels, absolute recoveries between 72 and 85% were obtained for HCBD and all chlorobenzenes except the dichloro congeners (Table I). Dichlorobenzenes were only 55 to 62% recovered by the SFE procedure. A major reason for the low recovery of dichlorobenzenes was due to losses in the solvent replacement step, which was confirmed by evaporation of a chlorobenzene solution in pentane in controlled experiments. Another possibility could be attributed to the incomplete trapping of the volatile compounds by the ODS trap, yet a lower trap temperature (5°C) during the extraction did not improve the recovery of the dichlorobenzenes. Incomplete extraction was unlikely to be the reason for low recovery since the results were similar when the chlorobenzenes were spiked into a non-active matrix such as Celite.

This new procedure has been used for the determination of HCBD and chlorobenzenes in sediments collected from the Great Lakes Basin. Replicate determinations by both SFE and Soxhlet technique were carried out on two freeze-dried, Lake Ontario samples. Our data indicated that the SFE results were generally in good agreement with the Soxhlet results and the precision for both techniques was similar (Table II). An ECD chromatogram depicting the chlorinated contaminants in a Lake Ontario sediment sample processed by the present method is given in Figure 4.

CONCLUSION

In summary, a successful extraction method for chlorobenzenes and HCBD in sediments has been developed using supercritical carbon dioxide. Under the optimized conditions, the new procedure provides recoveries for the above chlorinated pollutants similar to the Soxhlet technique. However, the SFE method is much faster and uses minimal amount of solvent. It eliminates one solvent evaporation step and thus reduces the losses of the volatile chlorobenzenes. The more efficient and selective SFE procedure is an ideal replacement for Soxhlet extraction for the determination of these chlorohydrocarbons in sediment samples.

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TABLE I

Percent recovery of chlorobenzenes and hexachloro-1,3-butadiene from spiked sediment
(Mean and standard deviation of seven replicate determinations)

Chlorobenzenes	250 ng/g	25 ng/g
1,3-Dichlorobenzene	65.3 ± 3.8	62.1 ± 6.5
1,4-Dichlorobenzene	64.9 ± 3.7	58.4 ± 6.5
1,2-Dichlorobenzene	66.8 ± 3.6	58.9 ± 5.8
1,3,5-Trichlorobenzene	76.8 ± 3.1	83.2 ± 6.0
1,2,4-Trichlorobenzene	72.0 ± 3.0	77.3 ± 7.0
1,2,3-Trichlorobenzene	73.2 ± 3.7	76.3 ± 4.3
1,2,4,5-Tetrachlorobenzene	76.7 ± 2.9	84.9 ± 7.8
1,2,3,4-Tetrachlorobenzene	74.5 ± 3.7	80.0 ± 6.9
Pentachlorobenzene	75.8 ± 3.5	84.7 ± 9.2
Hexachlorobenzene	77.6 ± 5.7	83.2 ± 7.9
Hexachloro-1,3-butadiene	78.5 ± 3.2	75.0 ± 4.9

TABLE II

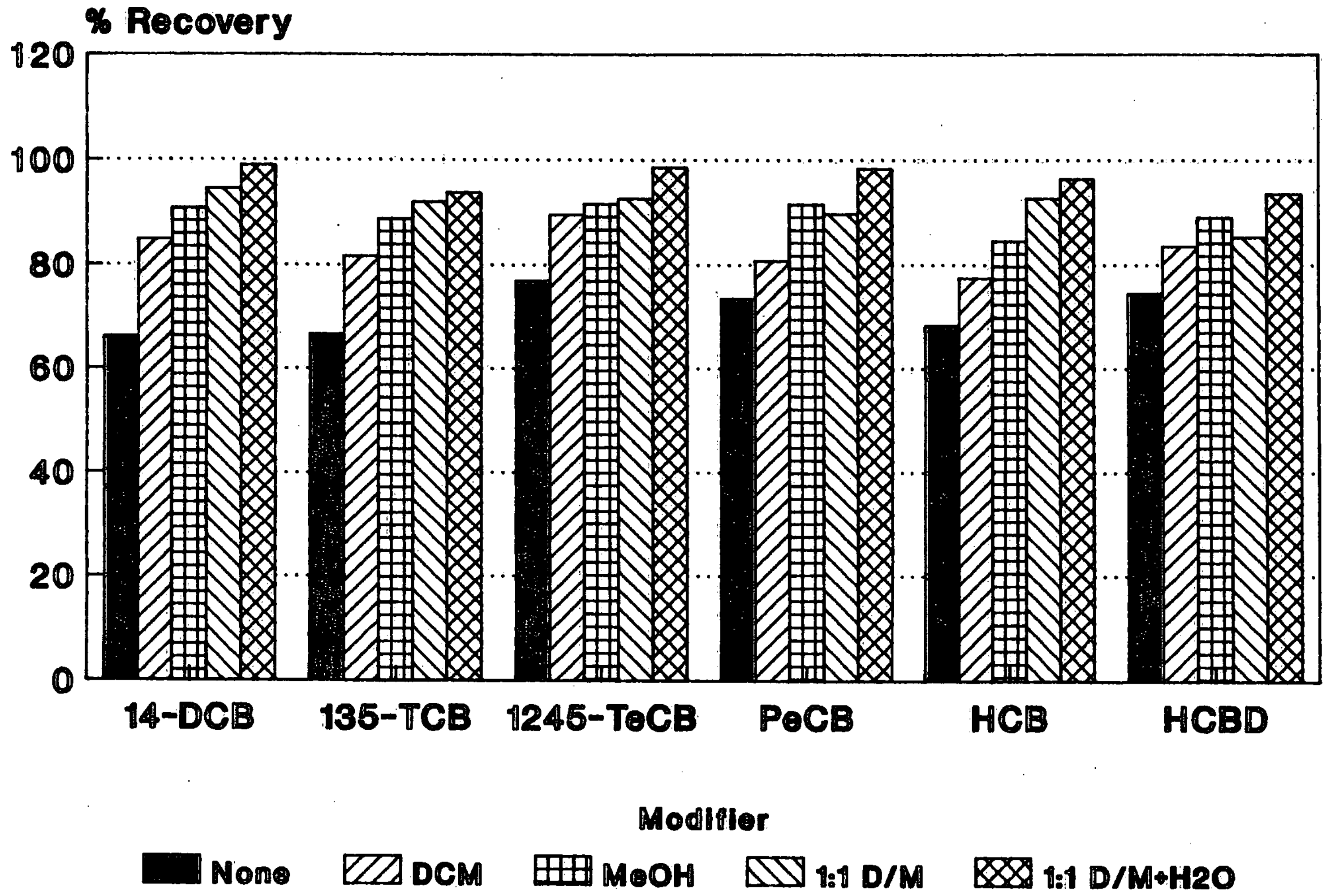
Levels of HCBD and the major chlorobenzenes (ng/g) in naturally contaminated sediments by SFE and Soxhlet extraction -- a comparison
(Mean and standard deviation of six or more replicate extraction and analyses)

Compound	Sediment 1		Sediment 2	
	SFE	Soxhlet	SFE	Soxhlet
1,4-Dichlorobenzene	57.0±5.6	58.5±2.9	87.3±8.2	98.3±6.7
1,3,5-Trichlorobenzene	37.3±2.9	34.3±2.6	89.9±7.5	99.0±8.6
1,2,4-Trichlorobenzene	89.3±6.7	80.7±5.4	146±13	127±9.0
1,2,4,5-Tetrachlorobenzene*	90.1±8.0	88.9±4.9	145±11	147±9.8
1,2,3,4-Tetrachlorobenzene	36.2±3.6	36.5±2.4	40.7±5.0	40.4±2.9
Pentachlorobenzene	50.3±4.7	48.6±2.4	61.3±6.0	62.3±5.8
Hexachlorobenzene	210±10	201±13	247±15	256±19
HCBD	19.4±2.4	21.3±1.6	59.4±7.0	63.4±5.4

* Including a small amount of 1,2,3,5-tetrachlorobenzene as this compound and 1,2,4,5-tetrachlorobenzene are coeluting under the chromatographic conditions used.

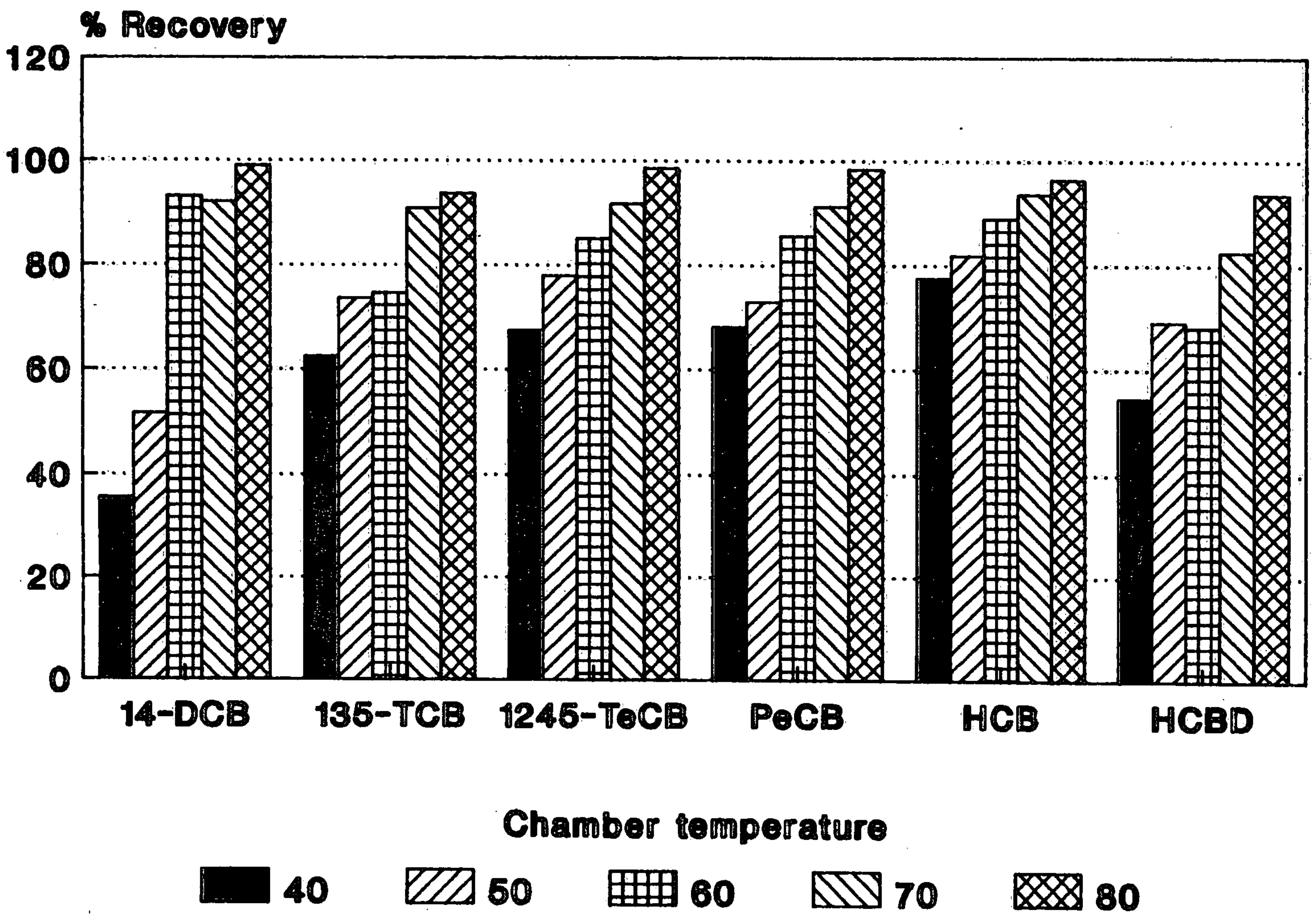
LIST OF FIGURES

Figure 1



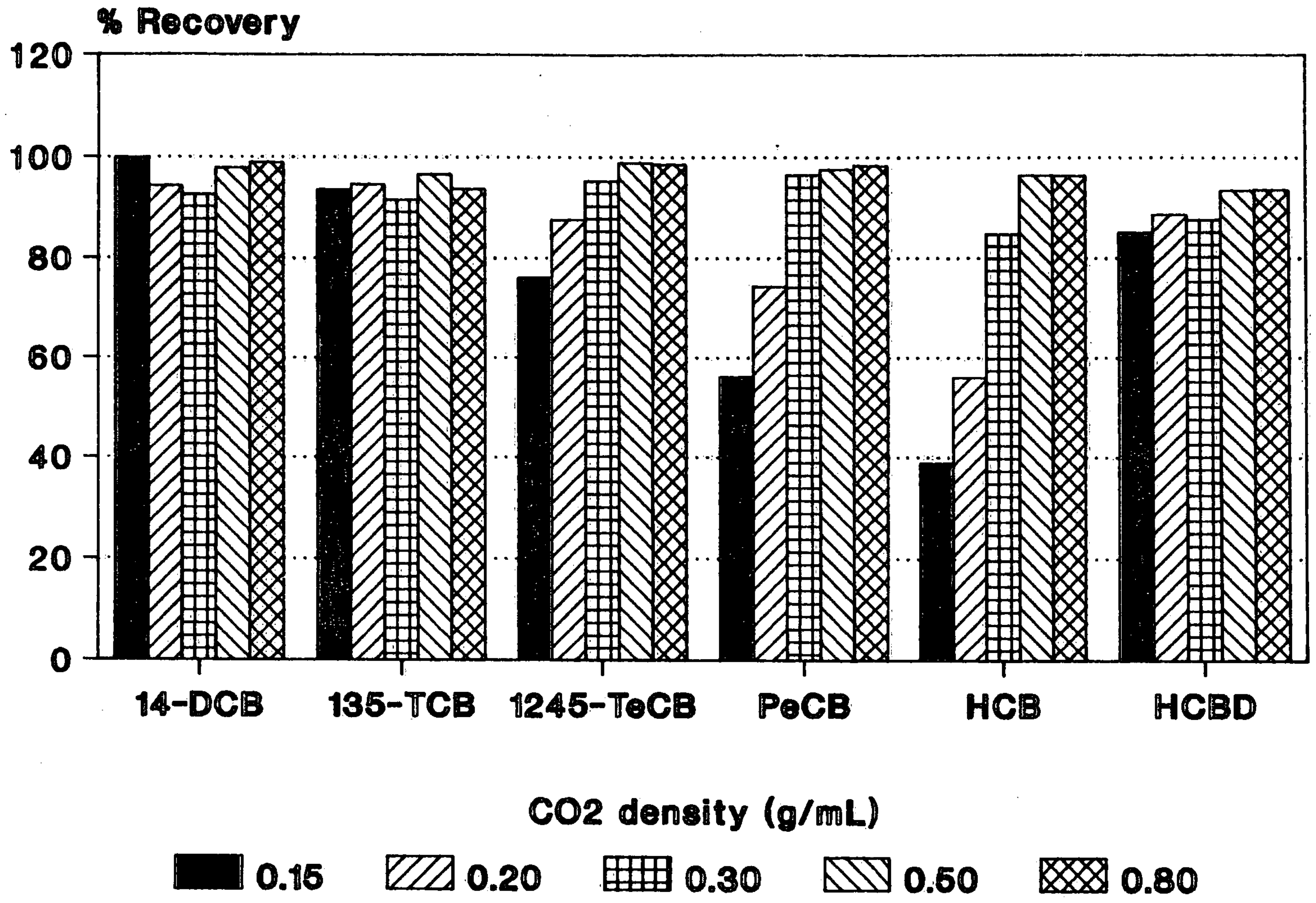
Soxhlet = 100%

Figure 2



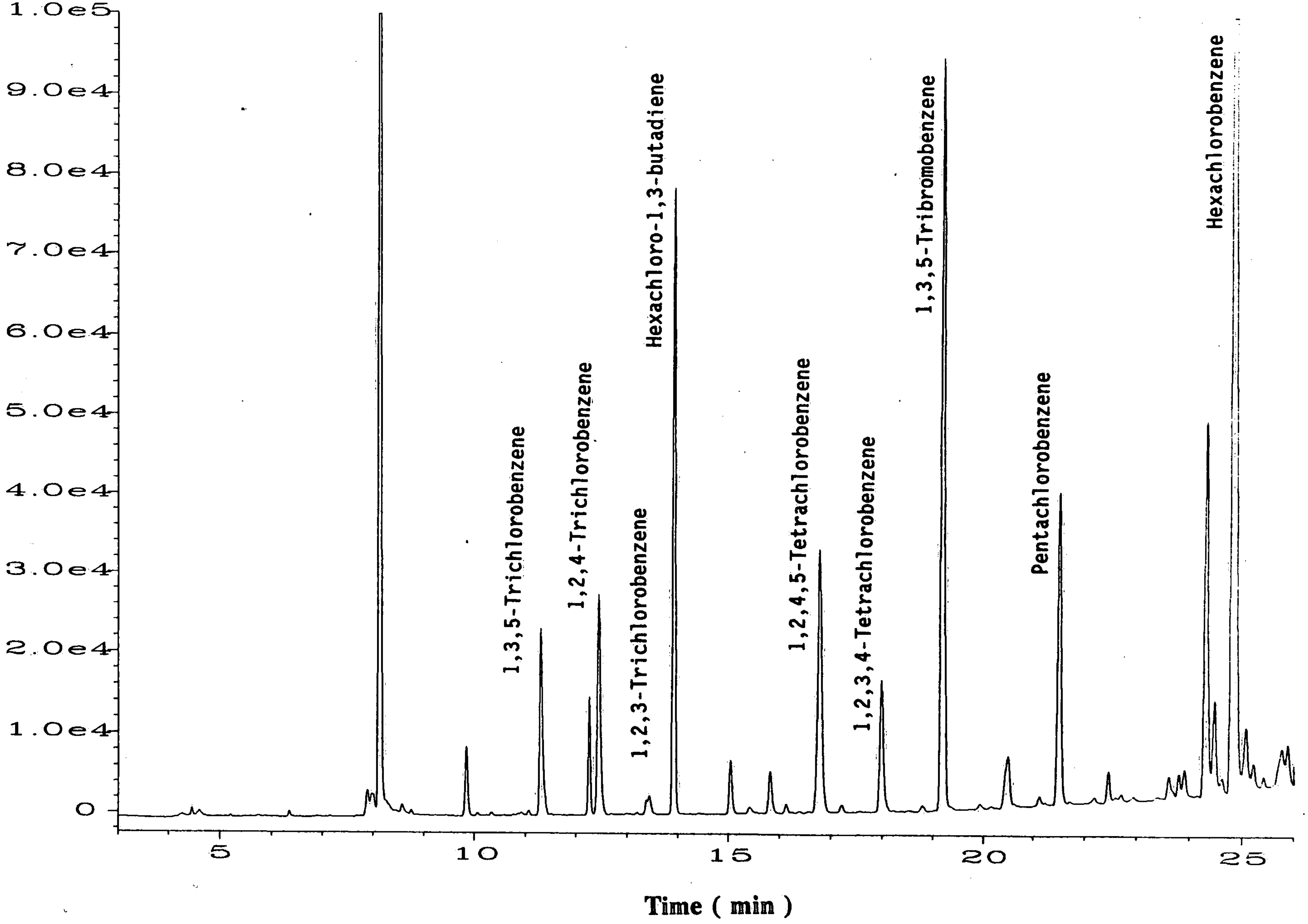
Soxhlet = 100%

Figure 3



Soxhlet = 100%

FIGURE 4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10
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20
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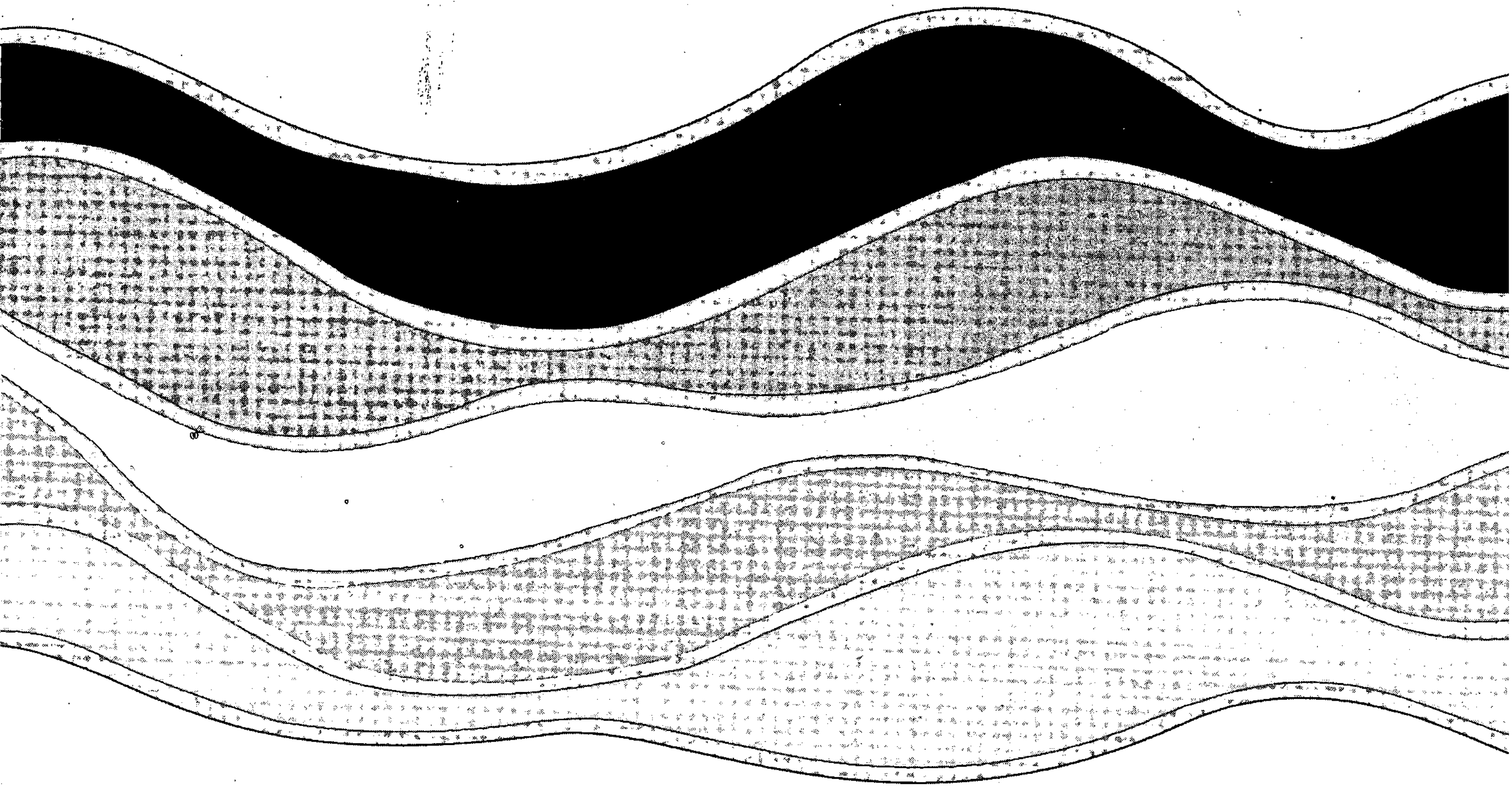
1,3,5-Tribromobenzene
Pentachlorobenzene
Hexachlorobenzene



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