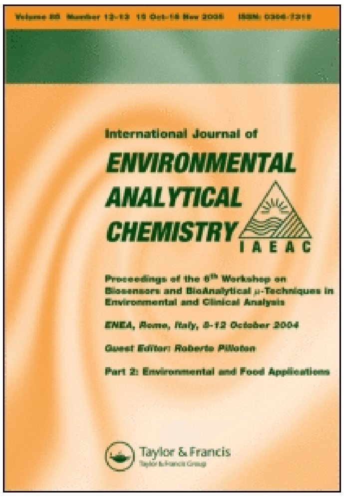

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Analysis for Phthalate Esters in Environmental Samples: Separation from PCB's and Pesticides Using Dual Column Liquid Chromatography

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A method was developed which allows phthalate esters (PE's) to be determined in environmental samples along with PCB's and several organochlorine pesticides, using a single sample extract. The extract is fractionated by dual column liquid chromatography (LC) into four fractions prior to analysis by GC/ECD. This new LC fractionation scheme for PE's involves (a) elution from an alumina column with benzene (separation from PCB's and pesticides which are eluted with hexane) followed by (b) elution from silica gel by an acetone/benzene mixture (separation from interfering moderately polar organics). Selected GC conditions enable nine different PE's to be resolved sufficiently for quantitation. The observation that PE's exhibit a decrease in ECD response as the detector temperature increases was investigated, and an optimum temperature was selected. Analysis of soil, sediment, and landfill leachate samples using the new methods gave PE results comparable to other results on environmental samples.

KEY WORDS: Phthalate esters, gas chromatography, liquid chromatographic separation, electron capture detector, environmental analysis.

INTRODUCTION

Phthalate esters (PE's) are a class of organic compounds, widely used as plasticizers, which have become ubiquitous in the environment. United States annual production has increased to over 5×10^8 kg since 1972.[1-3] A chief use of phthalates is in formulating polyvinyl chloride plastics, which typically contain 20 to 50% by weight phthalate ester.[4,5] Phthalates have been found worldwide in various environmental samples including soils,[6] sediments,[7-10] water,[7,9,11,12] biota,[6,9,13] air,[14,15] and atmospheric precipitation.[16,17] In addition, PE's have been identified in municipal wastewater effluents,[18] and in secondary sludges.[18,19] Six PE's (see Table I for acronyms) were included by the EPA in the list of priority pollutants: DMP, DEP, DBP, BBP, DEHP and DOP. In view of the worldwide distribution of PE's, their unknown environmental fate,[20] and possible adverse health effects,[21-24] together with the reported carcinogenicity of DEHP,[25] the development of an improved analytical methodology for environmental samples was undertaken.

TABLE I

Data for phthalate esters (see text)

Compound	Acronym	M.W.	GC retention time (min)	Detector response[a] area/nmol ($\times 10^{-6}$)
Dimethyl phthalate	DMP[b]	194.2	2.5	74
Diethyl phthalate	DEP[b]	222.2	3.7	51
Diallyl phthalate	DAP	246.2	6.4	67
Dibutyl phthalate	DBP[b]	278.3	10.5	13
Dihexyl phthalate	DHP	334.5	18.0	8
Butylbenzyl phthalate	BBP[b]	312.0	18.8	90
Di-2-ethylhexyl phthalate	DEHP[b]	391.0	21.0	16
Diphenyl phthalate	DPP	318.3	25.5	78
Dioctyl phthalate	DOP[b]	391.0	26.5	5

[a] ECD response at 255°C.
[b] EPA priority pollutants.

In recent published methods for PE analysis preconcentration is by extraction, using solvents of suitable polarity,[8,26] or by sorption on an HPLC column[27] or pre-column;[28] final separation and determination is by HPLC with UV detector[29-29b] or by gas chromatography (GC) with FID[6,21] or ECD.[13,30]

After the initial extraction, the separation of PE's from other environmental analytes such as organochlorine pesticides, PCB's and other interfering substances is generally necessary. Several methods have been used,[31] usually involving liquid chromatography (LC) on alumina[6,30] or Florisil.[9,13,30,32] These methods, however, have required large volumes of eluent (200–600 ml), or were designed for the analysis of only one or two PE's.

Goerlitz and Law, in determining PCB's and chlorinated hydrocarbon pesticides in sediments,[26] used solvent (acetone/hexane) extraction and dual column (alumina/silica) LC separation/clean up, and eluted with small volumes (20–40 ml) prior to GC/ECD analysis. This method has been used to analyze river sediments, soils and certain other environmental samples in this laboratory.[33-35] Further, in the course of our investigations on the distribution, transport, and fate of PE's in the environment, we have modified the Goerlitz–Law LC procedure so that PE's along with the organochlorine compounds can be determined in the extract from a single sample. With this method, the concentrations of PE's, PCB's and several pesticides have been determined in soil, sediment, water, and landfill leachate.[33-36]

Finally, a study of the temperature-dependent response of the ECD to PE's emphasizes the importance of optimizing the detector temperature to achieve an increased sensitivity for this class of compounds.[37]

EXPERIMENTAL

Reagents

a) *Double-distilled water (DDW):* Distilled water was re-distilled in a Corning borosilicate-glass still.

b) *Alumina:* Neutral, Brockman Activity I, 80–200 mesh (Fisher Scientific, Fair Lawn, N.J.); heated to 210°C for several hours, cooled and deactivated with 9% (v/w) DDW.

c) *Silica gel:* Woelm, activated, 70–150 mesh (Universal Scientific, Atlanta, GA); heated to 210°C for several hours, cooled and deactivated with 0.3% (v/w) DDW.[38]

d) *Sodium sulfate anhydrous:* Granular (Fisher Scientific); heated for several hours at 210°C, cooled and stored in a desiccator.

e) *Solvents:* Acetone, benzene and hexane (Burdick and Jackson, Muskegon, MI), pesticide-residue quality; methylene chloride (Fisher Scientific), certified A.C.S. grade.

f) *Phthalate esters:* DBP, DMP (Fisher Scientific); DEP, DEHP (Eastman Organic Chemicals, Rochester, N.Y.); DAP (Matheson, Coleman and Bell, Cincinnati, Ohio); DOP, DHP, BBP, DPP (Chem. Service, West Chester, PA).

Apparatus

a) *Glassware:* All items were detergent washed, rinsed with tap water and DDW, then with reagent grade acetone and placed in a 210°C oven overnight; upon removal, they were cooled and rinsed with hexane before use.

b) *LC columns:* 10 mm i.d. x 300 mm with a sealed-in coarse-porosity fritted glass disk. Columns are packed with a 1-cm layer of Na_2SO_4, 8 cm of alumina or silica gel, and another 1 cm of Na_2SO_4.

c) *Evaporators:* (1) Kuderna–Danish concentrator, 500 ml, with a 3-ball Snyder Column; (2) stainless steel manifold for directing stream of N_2 over liquid heated by a Fisher Infra-Radiator heat lamp.

d) *Gas chromatograph:* Hewlett–Packard Model 5750 Research GC with a ^{63}Ni electron capture dector; 6 ft x 4 mm i.d. glass column packed with 1.5% SP-2250 and 1.95% SP-2401 on 100/120 mesh Supelcoport. Operating conditions for PE analysis: Injection port—250°C, ECD—250°C; column temperature program—180°C for 4 min, increase at 4°/min to 240°C, hold for 10 min; carrier gas—5% methane in argon, at 60 ml min^{-1}. Chromatograms were traced by a Sargent Model SRG potentiometric recorder and peak areas were integrated and amounts determined using a Varian CDS-111 Chromatography Data System. The GC/ECD system was calibrated using standard solutions of PE's in hexane within the linear response range of the detector. Calibration peak areas, in arbitrary count units, were typically reproducible to ±5%.

Procedures

a) Sampling

The collection of Kishacoquillas Creek sediment and Codorus Creek and Conestoga River Basin soils, in 1979, from Pennsylvania, and a Broome Co. (N.Y.) composite soil, in 1978 are described elsewhere.[33, 34] The bottom sediment was wet-sieved and the soils, from the top 15 cm of the soil profile, were dry-sieved through a 2 mm polyethylene sieve. Sediments and some soils were stored frozen, other soils were air-dried and stored in closed containers. All samples were air-dried before use.

Leachate-sprayed soil collected on 2/17/82 was a composite of the top 10 cm of soil from each of two spraying sites at the Broome Co. Landfill.[36] Unsprayed (control) soil was collected on 6/16/82 at sites adjacent to the leachate spray areas. These soils were air-dried, sieved to <2 mm size and stored in closed containers.

Landfill leachate was collected on 6/16/82 from the Broome Co. landfill. Samples were collected in hexane-rinsed glass bottles with foil lined caps, refrigerated in ice until returned to the laboratory, and then extracted immediately to minimize loss from biodegradation.[29b, 36]

b) Extraction of samples

1) *Soils and sediments:* Approx. 20 g of material were weighed into a 250-ml erlenmeyer flask (with a ground glass stopper) and wetted with a few ml of water. Three extractions were done: First, 40 ml of acetone were added, the flask contents mixed for 20 min using a wrist-action shaker, then 80 ml of hexane were added and the flask shaken for an additional 10 min; the hexane-acetone layer was then decanted into a 500 ml separatory funnel. For the second and third extractions, the same procedure was followed except that only 20 ml of acetone were added.

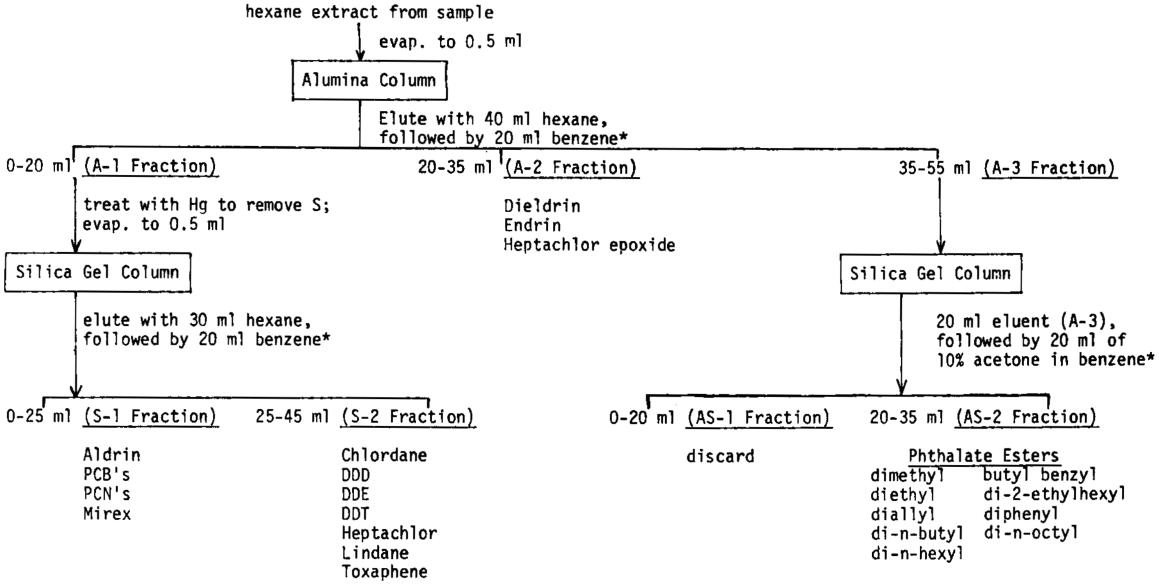
To the combined hexane-acetone extracts in the separatory funnel, 200 ml of water were added and the mixture was shaken. After the layers separated, the aqueous layer was collected in a beaker, and the organic layer was placed in a 500-ml erlenmeyer flask. The aqueous layer then was returned to the separatory funnel, back extracted with 25-ml of hexane, and discarded. The organic layer was added to the 25-ml of hexane in the separatory funnel, and washed two more times with 200 ml of water, discarding each aqueous layer. The organic layer (relatively acetone-free) then was added to a 500-ml erlenmeyer flask with enough Na_2SO_4 to remove any remaining water. This dry extract was preconcentrated to 0.5 ml using first a Kuderna–Danish evaporator and finally the N_2/heat lamp apparatus.

2) *Landfill leachate:* One-liter portions were extracted three times with 50 ml of methylene chloride in a 2 l separatory funnel. Known aliquots (25–50 ml) of this extract were evaporated to dryness with a stream of N_2, first with a heat lamp, then without heat as the sample neared dryness. The residue was then dissolved in 0.5 ml of hexane for subsequent LC clean-up and fractionation.[30]

c) LC separation procedure

1) *Development:* Various solvents or solvent mixtures were evaluated to establish a procedure for eluting PE's from alumina and silica gel columns. In these elution experiments, 0.5 ml of a standard mixture of PE's in hexane (0.5–17 ppm) was pipetted onto the top of the column and eluted with a trial solvent, solvent mixture, or a series of solvents. The eluents were collected, evaporated to 0.5 ml, and analyzed by GC/ECD. When a solvent or mixture was found which quantitatively eluted all the PE's from the column, the elution was repeated and 2-ml fractions were collected to determine the elution pattern.

2) *Procedure:* Starting with the 0.5 ml hexane concentrate from a sample, the analytes were fractionated according to the flow chart shown in Figure 1. The sample is pipetted onto the top of an alumina column and eluted with 40 ml of hexane; just as the last of the hexane enters the Na_2SO_4 layer, 20 ml of benzene are added. Fractions are collected in 25 ml graduated cylinders as follows: A-1 20 ml; A-2 15 ml; A-3 (containing the PE's) 20 ml; (void volume is 5 ml). [The A-1 fraction can be further separated on silica gel into two fractions, one containing PCB's, Aldrin, and polychlorinated naphthalenes (S-1); and one containing DDD, DDE, DDT, heptachlor, lindane, and toxaphene (S-2).][26] The A-3 fraction is treated as follows: The volume (if less than 20 ml) is adjusted with benzene to be 20 ml, and the solution is poured through a silica gel column; as the last of the solution enters the Na_2SO_4 layer, 20 ml of 10% acetone in benzene (v/v) are added. The first 20 ml of eluent (AS-1) can be discarded; the next 15 ml (AS-2) contain all nine phthalate esters included in this study, if present. The fractions of interest (i.e., A-2, S-1, S-2 and AS-2) are evaporated to 0.5 ml, and 2–10 μl are analyzed by GC/ECD.



*Approximately 5 ml of eluting liquid is retained by column.

FIGURE 1. Liquid chromatographic separation of organic pollutants.

d) Effect of temperature on electron-capture detector response

The temperature dependence of the sensitivity of the ECD to PE's was measured using a standard mix of PE's in hexane; successive 5-μl portions were injected under the conditions described above, except that the detector temperature control was varied between injections. As each phthalate peak was recorded, the detector temperature was noted. In this way, for each PE, peak areas were obtained at various detector temperatures in the range 250–340°C.

RESULTS AND DISCUSSION

Development of LC method for PE separation and clean-up

Several methods have been described for the clean-up of PE's prior to analysis; most of these methods use LC chromatography. Poole and Wibberley[21] used a series of solvent extractions to separate DEHP from interferences; this circumvented the long times involved in eluting a column with a large volume of solvent, but was not tried with other PE's which have different solubilities and solvent distribution coefficients.

Alumina has been used as a column packing to clean up several PE's by Zitko[39] and Persson *et al.*[6] Webster and Nickless[40] used an alumina column to separate several PE's (including DEHP and BBP) by eluting with portions of 0–50% diethyl ether in hexane. One procedure outlined in the EPA method 606 uses a column of alumina deactivated with 3% water from which the six EPA Priority PE's (DMP, DEP, DBP, BBP, DOP and DEHP) can be separated from interferences and eluted with hexane and 20% ethyl ether in hexane.[30] Both of these methods require large quantities (200–400) of eluent, which makes both the elution and the subsequent preconcentration time consuming.

By far the most commonly used solid phase for liquid chromatographic separation/clean-up of PE's is Florisil. Thomas[32] used Florisil to clean-up samples for PE analysis; interferences were eluted with hexane, and 25% diethyl ether in hexane, and then the PE's were eluted with diethyl ether (360 ml solvent, total). Mayer, *et al.*[9] used Florisil and recovered DBP and DEHP (90% and 50%

recovery, respectively) using a 15% diethyl ether-petroleum ether eluate. Stalling, *et al.*[41] also eluted DBP and DEHP from Florisil using 15% diethyl ether in petroleum ether, but noted that some DEHP could elute in the previous (6% diethyl ether) fraction. Giam, *et al.*[13] found that recoveries of DEHP were not reproducible when 15% diethyl ether in petroleum ether was used to elute from a Florisil column, and optimized the method to use 15% diethyl ether on Florisil deactivated with 3% water. This method was used to separate PE's into 2 fractions in the analysis of marine samples, and to separate them from PCB's, DDT and DDE.[42,43] Although this method allows for the simultaneous analyses of several organic pollutants, the PCB's, DDT and DDE, which can overlap and interfere during GC analysis,[44–46] are combined in one fraction,[13,43] DBP is divided into two separate fractions,[43] and large volumes of eluents (600 ml) are required if the samples have a high lipid content.[13,42,43]

EPA Method 606 alternatively uses a Florisil column cleanup.[30] PE's are eluted with 20% ethyl ether in hexane from 3% water-deactivated Florisil following elution with hexane with a total of 180 ml of solvent required.

In view of favorable experience with the Goerlitz and Law method, experiments were undertaken to ascertain whether or not PE's could be conveniently determined using a simple modification of that method.

Starting with a mixture of PE Standards, the A-1 and A-2 fractions obtained using the method of Goerlitz and Law showed no PE's to be present. Eluting further with 25 ml of hexane failed to remove the PE's from the alumina column, indicating a more polar solvent was necessary. Separately, 25 ml of methylene chloride, acetone, benzene, and methanol were tried; each eluted all of the PE's from the column. Benzene was chosen over the other solvents, as it is used in another part of the procedure and is most compatible with the GC/ECD method. LC chromatograms showing the elution pattern of the nine PE's tested are shown in Figure 2. 20 ml of benzene (termed the A-3 fraction) was sufficient for collecting all the PE's from the alumina column.

At this stage, several soil samples showed the need for further clean-up of the A-3 fraction to remove unwanted interferences.

Since a dual column system was being used for PCB's and

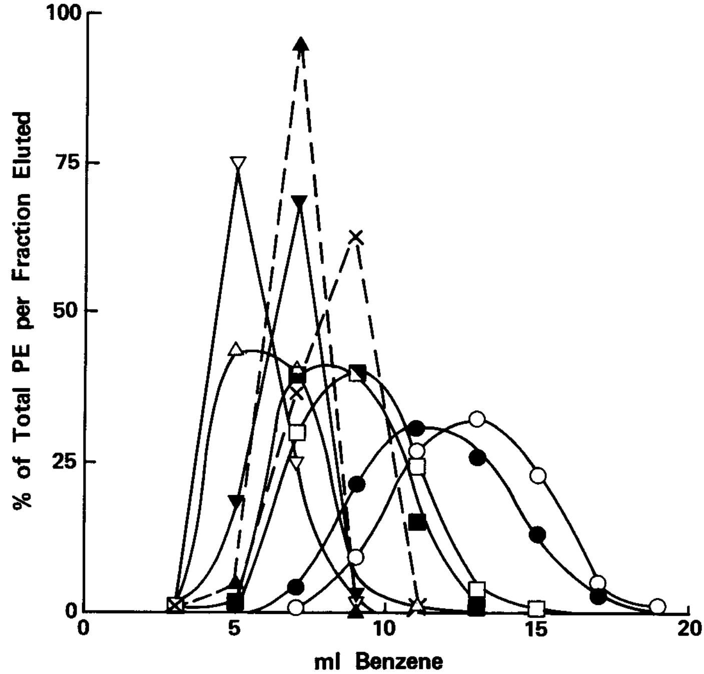


FIGURE 2. Elution of PE's from alumina with benzene (—○DMP; —●DEP; —□DAP; —■DPB; —△DEHP; ---▲DHP; —▼DPP; ---× BBP; —▽DOP).

pesticides,[26] the use of a silica gel column was evaluated for further clean-up of the A-3 fraction.

A silica gel column was eluted first with 30 ml of hexane and then with 30 ml of benzene, which were each collected as separate fractions; GC/ECD analysis showed no PE's were present in either fraction, but all were eluted with 20 ml of acetone. It was therefore hypothesized that a solvent with polarity and solvent strength between that of acetone and benzene would be sufficiently polar to remove the PE's, but would leave some more polar interfering substances on the silica gel. After considering several solvents,[47] mixtures of acetone in benzene were tried: 20–40 ml portions of 5 to 20% acetone in benzene were evaluated, and an optimum was found at 20 ml of 10% acetone in benzene. Figures 3a and b show the fractional elution of various PE's from silica gel using 10% acetone in benzene.

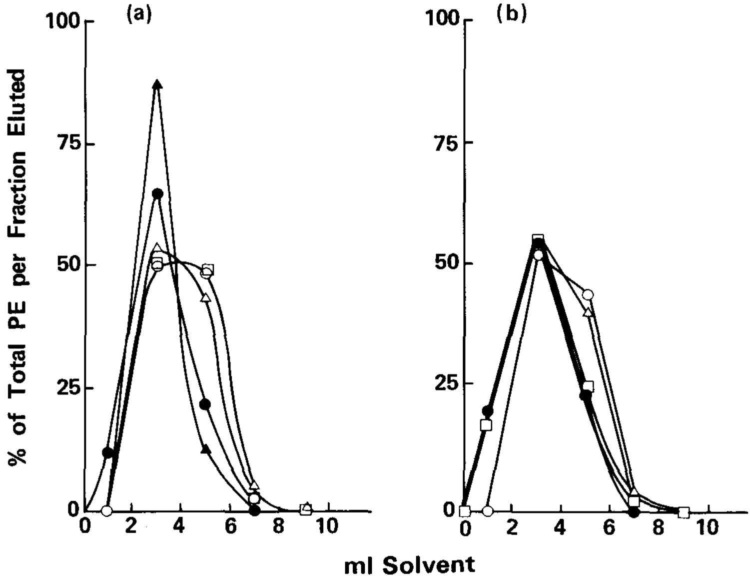


FIGURE 3. Elution of PE's from silica gel with 10% acetone in benzene.
a) —○DMP; —□DEP; —△DBP; —●DHP; —▲DOP:
b) —○DAP; —□BBP; —△DEHP; —●DPP.

In the final procedure, hexane was not used in eluting the A-3 fraction; benzene was sufficiently polar to remove any interferences which would have been removed by the hexane. Also, time was saved by not evaporating the 20 ml A-3 fraction prior to passing it through the silica gel, since the PE's are retained by silica gel with benzene as the mobile phase.

The method proposed here has three advantages over other methods used to clean up samples for PE analysis. This method allows nine different PE's to be eluted in one fraction for gas chromatographic analysis. It also allows the separation of PE's from PCB's and pesticides and using the Goerlitz and Law procedure, separates DDT and its metabolites (DDE and DDD) from the PCB's so all can be analyzed from a single extraction. Finally, although two LC columns are used, less solvent is used to elute the PE's than in other methods (80 ml total); therefore less time is needed for elution and subsequent preconcentration of the sample prior to GC/ECD analysis.

Selection of GC column conditions

The column and temperature conditions used for the GC/ECD analysis of the AS-2 fraction (see Experimental section) were chosen to give widely separated PE peaks (for all nine PE's) within a reasonable time (30 min). In this way, even though certain interfering substances might still be present in the AS-2 fraction, the probability of those substances interfering with particular PE peaks was reduced.

Further, under these conditions, the gradual loss of stationary phase (due to column bleed resulting from programming at high temperatures) did not appreciably degrade the resolution over months of use.

Optimization of detector response

The variation of sensitivity of the ECD with detector temperature for some compounds is well documented.[48–52] This makes it necessary to optimize the detector temperature when analyzing for a specific compound.[37] The slope of a plot of $\ln KT^{3/2}$ vs. $1/T$ for a compound (where T is the detector temperature in Kelvin and K is the electron-capture coefficient) gives information about its electron-capture mechanism[48, 52] and electron affinities.[49]

Phthalate esters give a good response when analyzed using ECD and show a marked temperature dependence. Vessman and Hartvig investigated DBP interference in the analysis of *p*-bromobenzophenone, and found a non-dissociative attachment mechanism and a response which decreased with increasing temperature, giving no signal above 200° in the pulsed mode.[51] Poole and Wibberley plotted $\ln AT^{3/2}$ vs. $1/T$ for DEHP (where A is the peak area, which is proportional to K[53]) and found a non-dissociative electron-capture mechanism and a response that decreased with increasing temperature.[21] Hattori, *et al.*[54] measured relative sensitivities for several phthalate esters at different detector temperatures. They found that the electron-capture coefficients (non-dissociative mechanism) decreased with increasing temperature except for DAP.

Our work with nine PE's gave results similar to those discussed above, but with some differences: All PE's (including DAP) showed a general decrease in response with an increase in temperature between about 250 and 340°C. DBP showed a slight increase between 255 and 260°C, but then decreased from 260 to 310°C, above which temperature no response was observed. Five other PE's (DMP, DEP, DHP, DPP and DOP) all exhibited this phenomenon of no peak (3–4 ng injected) above 300°–310°C.

These studies indicated that a lower detector temperature would give better sensitivity. However, the detector temperature should generally be kept above that of the column to prevent compounds from condensing in the detector. Thus, a detector temperature of 250°C was selected as an optimum, since a maximum column temperature of 240°C was used for PE analysis.

Figure 4 shows a plot of $\ln AT^{3/2}$ vs. $1/T$ for DPP, BBP and DHP. All three exhibit a positive slope, DHP having the largest.

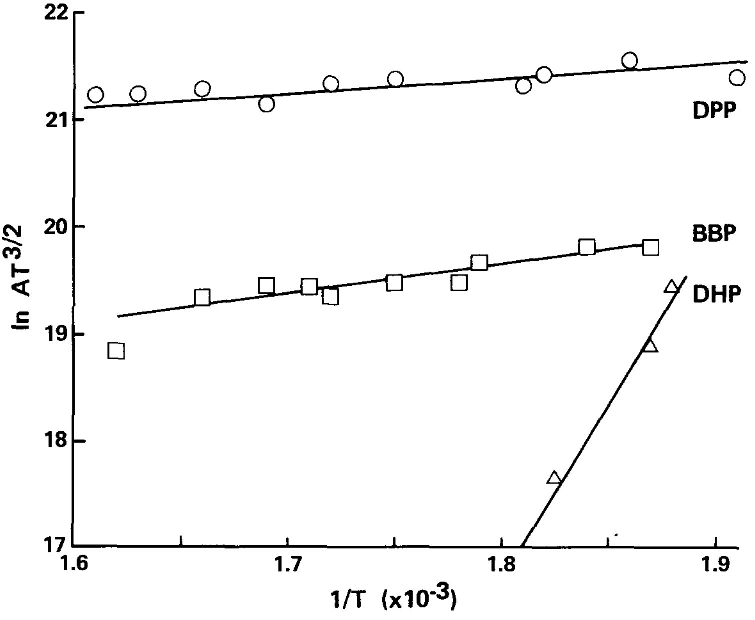


FIGURE 4. Temperature response of the ECD to PE's.

Table I compares the ECD sensitivity for the various PE's, as area per nanomole, at a detector temperature of 255°C. The order of sensitivity was found to be:

$$\text{BBP} > \text{DPP} > \text{DMP} > \text{DAP} > \text{DEP} >$$

$$\text{DEHP} > \text{DBP} > \text{DHP} > \text{DOP}.$$

For the straight chain alkyl esters, the response decreases with increasing length of chains: A plot of log area/nmol vs. molecular weight (MW) for the straight chain esters (DMP, DEP, DBP, DHP and DOP) yielded the relationship (Figure 5):

$$\log \text{area/nmol} = -0.0062\,\text{MW} + 9 \qquad (r^2 = 0.97).$$

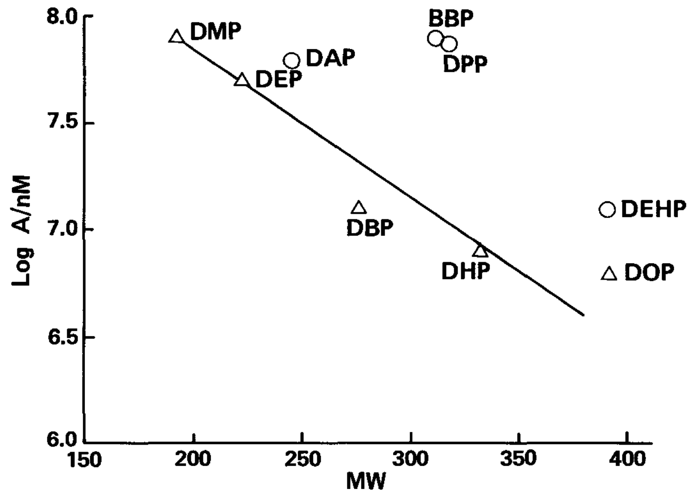


FIGURE 5. Detector response vs. molecular weight for PE's.

Results on environmental samples

This method for PE's has been used to analyze sediment, soil, landfill leachate and river water.[36] When performing environmental analysis for PE's high blanks are a severe problem,[21,30,40,55] especially for DBP[13,43] and DEHP.[13,42,43] PE's have been detected in blanks in our laboratory, therefore precautions were taken to reduce contamination and correct our analysis values. All glassware was solvent rinsed and baked in an oven at 210°C. Water was doubly distilled in an all glass still. Sodium sulfate, silica gel, and alumina were heated to 210°C for several days prior to use. Plastic tubing and containers were avoided. Finally, blanks were run with every set of samples to allow corrections for background contamination, if present.

The concentrations of some organic pollutants found in soils, sediment, and landfill leachate along with the detection limits are shown in Table II. Details of the analysis for PCB's and Chlorinated Hydrocarbon Pesticides are described elsewhere.[26,33,34]

The soil samples from the Susquehanna River Basin in Pennsylvania and New York had PCB concentrations ranging from background (35 ng/g)[56] to high, but these soils were low in most pesticides and in most PE's. The PE's found in significant concentrations in soil were DBP and DEHP. The sediment from Kishacoquillas Creek (a tributary of the Susquehanna River) contained moderate levels of several PE's, with DBP and DEHP being significantly higher. These values are in agreement with those from the top 10 cm of Chesapeake Bay sediments recently reported by Freeman and Peterson.[10] The world-wide trend of high DBP and DEHP values in soils[6] (reflected here) and sediments[8,9,10,40] are coincident with the high production of these compounds.[1,3,10] Apparently the input of these compounds to the world ecosystem overbalances their tendency to biodegrade.[1,10,36]

Details of the study of landfill leachate and leachate-sprayed soil have been reported elsewhere.[36] The fact that there are no significant differences between the Broome Co. soil composite, leachate sprayed soil, and nearby unsprayed soil is probably due to biodegradation by soil bacteria (ref. 36 and references therein). Presently this laboratory is continuing a study of the environmental chemodynamic properties of PE's. This work includes determining K_p and K_{oc} values, solubilities, biodegradation rates, and soil migration rates.

CONCLUSION

Phthalate esters are a class of organic pollutants which have a world-wide distribution and have caused concern as to human and

TABLE II

Concentrations of organic pollutants in some environmental samples (ppb)

	S-1		S-2					A-2		AS-2								
Sample	Aldrin	PCB's[a]	DDD	DDE	DDT	Heptachlor	Lindane	Dieldrin	Heptachlor epoxide	DMP	DEP	DAP	DBP	DHP	BBP	DEHP	DPP	DOP
Kishacoquillas[c] Creek sediment	0.0	112	44	15	0.9	1.1	0.0	0.8	<0.1	14	35	44	121	—[b]	—	157	—	—
Codorus Creek[c] soil no. 2	0.0	120	7	13	6	0.6	0.6	3.4	4.5	1	6	ND[e]	120	—	—	301	—	—
Conestoga River[c] soil no. 4	0.0	624	33	20	28	7	2.6	30	30	35	ND	ND	277	—	—	ND	—	—
Landfill leachate 6/16/82	—	ND	ND	ND	ND	ND	0.02	ND	ND	4	15	0.1	1.2	0.9	ND	1.0	ND	ND
Leachate-sprayed soil 6/16/82	—	20	4	4	9	ND	1	1.3	ND	24	27	6	9	ND	8.2	1026	90	ND
Nearby unsprayed soil 6/16/82	—	47	3	5	8	ND	4	1.9	ND	12	26	9	23	ND	57	1280	ND	ND
Broome County[d] soil composite	0.0	34	2.1	3.0	5.4	0.2	0.0	0.0	0.0	6	19	10	155	14	ND	1200	ND	132

[a] Total PCB's.

[b] Not analyzed for.

[c] See refs. 34 and 36.

[d] See refs. 33 and 36.

[e] Not detected. Approximate Detection Limits: (i) for soils and sediments (ng/g): PCB's and pesticides 0.3; DMP, DEP, DAP, DBP 1–3; BBP, DPP 5–10; DHP, DEHP 10–20; DOP 100. (ii) for leachate (μg/l): PCB's and pesticides 0.01; DMP, DEP, DAP, DBP 0.1; BBP, DPP 0.5; DHP, DEHP 1; DOP 5.

environmental health. Several PE's are listed as priority pollutants by the U.S. EPA.

The method of analysis discussed here combined an extraction/clean-up step which allows many organic pollutants (including PCB's, PE's and chlorinated hydrocarbon pesticides to be extracted from a single sample and fractionated to minimize interferences in the sensitive GC/ECD measurement technique. This method is economical in time and materials and has been used to analyze for these organic pollutants in soils, sediments, water and landfill leachate. The concentration of PE's reported are consistent with those found in other recent work. Further investigations into the chemodynamic properties of the PE's are in progress.

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