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# COMPARISON OF RESULTS BETWEEN PCB CONGENER METHOD AND STANDARD METHOD OBTAINED DURING VALIDATION STUDY

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#### MANAGEMENT PERSPECTIVE

This cooperative report was undertaken to illustrate the reproducibility and durability of the congener specific PCB method that also analyses particular organochlorines. The results from the congener specific and current standard method agree reasonably well. Once the columns are installed for the congener specific method, there is little need for changing them more frequently than other instances where dual This report shows that the new congener specific columns are used. methodology provides results that are comparable to the existing method at a saving of instrumentation time as only one analysis is needed for both the organochlorine analysis and PCB quantitation. Less time is required to determine the PCB concentration as this is calculated by the ancillary computer programs created to analyze the GC data, rather than the complicated subjective method now used in the standard method. Each PCB congener is treated as an individual compound and hence there are considerably more target compounds. Once the all target compounds are identified and quantified, the remaining peaks in the chromatogram can be treated by the broad spectrum techniques that are being developed.

Dr. J. Lawrence Director Research and Applications Branch

Cette étude en collaboration a été entreprise en vue d'illustrer la reproductibilité et la durabilité de la méthode de dosage des congénères spécifiques des BPC, qui permet également d'analyser les composés organochlorés particuliers. Les résultats de cette méthode et ceux de la méthode courante standard concordent passablement bien. Une fois les colonnes installées pour l'application de la méthode de dosage des congénères spécifiques, on n'a quère à les changer plus souvent que dans les autres cas où des colonnes doubles sont utilisées. Ce rapport révèle que la nouvelle méthodologie donne des résultats comparables à ceux obtenus à l'aide de la méthode actuelle tout en permettant des économies de temps d'instrumentation, étant donné qu'une seule analyse suffit pour analyser les composés organochlorés et quantifier les BPC. 11 faut également moins de temps pour déterminer les concentrations de BPC étant donné que celles-ci sont calculées par les programmes informatiques auxiliaires créés pour analyser les données CG plutôt que par la méthode subjective compliquée utilisée aujourd'hui dans la méthode standard. Chaque congénère des BPC est traité comme un composé individuel et c'est pourquoi on peut doser un nombre considérablement plus élevé de composés-cibles. Une fois l'ensemble des composés-cibles identifié et quantifié, les pics qui restent dans le chromatogramme peuvent être traités à l'aide des techniques spectrales générales qui sont actuellement mises au point.

D<sup>r</sup> J. Lawrence Directeur Direction de la recherche et des applications The results from a new congener specific gas chromatographic PCB method were compared to those derived from a standard method. The samples included replicates of a spiked sediment sample, replicates of the spike treated only with the cleanup technique, replicates of the unspiked sediment, and replicates of the spiking solution. The results derived from both methods agreed and the precision was similar. Accordingly, the PCB congener specific method provides the composition of the PCBs present but requires less analysis time.

ii.

### RÉSUMÉ

Les résultats d'une nouvelle méthode de dosage des congénères spécifiques des BPC par chromatographie gazeuse ont été comparés aux résultats obtenus par la méthode standard. Les échantillons comprenaient des échantillons multiples de sédiments enrichis, des répétitions des mêmes échantillons traités uniquement à l'aide de méthodes de lavage, des répétitions de sédiments non enrichis et des répétitions de la solution d'enrichissement. Les résultats obtenus grâce à ces méthodes concordaient et leur précision était semblable. En conséquence, la méthode de dosage des congénères spécifiques des BPC permet d'obtenir la composition des BPC en moins de temps.

### 1.0 INTRODUCTION

When a new method is introduced, there are many questions in the minds of potential users as to how the new method compares with the one presently in use. One would like to compare the precision of it as well as the durability. To answer these questions, the dual capillary column, congener method (Scott et al, 1988), was used to analyze two sets of extracts, one from water and the other from sediment. These extracts were also being analyzed by standard methods (Afghan and Forbes, 1987). The congener PCB method also analyzes for those organochlorine compounds which elute in the non-polar eluate of the cleanup step. To establish the utility of the method, the results of the analysis of these OCs must be as good as those from the standard method and the total of the PCB congener values should approximately agree with those derived by using the standard method. If there is a major difference in the results derived from the two methods, there must be an obvious explanation.

In actuality, two factors are being addressed in this study. The first is the reproducibility of the method as determined with standards. This illustrates the method does work and the instrumentation is satisfactory. The second invoves the sample processing techniques of the laboratory staff and the effect of interferences from the samples and reagents on the chromatography. This implies that some measure of the precision of the first factor can be subtracted from the overall precision to give an estimate of the second factor. However, there are many variables in that factor, and ascribing portions of the loss of precision to particular aspects would be a dubious task.

This report presents the comparison between two different analytical methods. The older method requires the use of two separate analyses to achieve the results obtained by congener method.

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#### 2.0 EXPERIMENTAL

#### 2.1 Standards and Samples

Laboratory standards of the organochlorines were obtained from the National Respository, EPA, NJ. These were made into standard solutions in isooctane, and were used as spiking solutions which were also further diluted to be used as injection standards. The OCs reported here are 1,2-, 1,3-, and 1,4-dichlorobenzene, 1,2,4-, 1,3,5-, and 1,2,3-trichlorobenzene, 1,2,3,4-tetrachlorobenzene, pentachlorobenzene, hexachlorocyclopentadiene, aldrin. hexachlorobenzene, heptachlor, octachlorostyrene, o,p'-DDT, p,p'-DDT, p,p'-DDE, photomirex and mirex. One PCB calibration solution was made from combining equal parts of Aroclors 1242, 1254 and 1260 so that a 1  $\mu$ L injection would contain 200 pg total PCB. The other calibration solution was composed of 1000 parts Aroclor 1221, 500 parts Aroclor 1016, 350 parts of Aroclor 1254 and 300 parts of Aroclor 1262. These solutions were stored in isooctane.

The sediment was from a large sample which had been collected near Niagara-on-the-Lake, Ontario, and had been characterized so that it could serve as a reference sediment for a number of studies conducted near the original collection site.

#### 3.0 METHODS

The following are summaries of the method used for preparation of samples. The more complete description can be found in Afghan and Forbes, 1987; and Lively, 1979. For the sediment cleanup, 10 g of the sediment was spiked with the OC and PCB solution and extracted immediately using the ultrasonic technique with 1:1 acetone-hexane as the extraction medium. After extraction there was no GPC step, but the sample was passed through 2 mL of Celite, concentrated, then placed on a silica gel column (deactivated with 3% water) which had been pre-rinsed with 40 mL of hexane. Then the extract was placed on the column and eluted with 40 mL of hexane (called Fraction A or OCA). Other material on the column was then eluted with 60 mL of 1:1 hexane:methylene chloride. To the eluates was added 10 mL of isooctane, and each volume was reduced on a Rotovap to about 2 mL which was transferred to a centrifuge tube and made up to 5 mL. Mercury (0.1 mL) was then added to the first fraction and shaken and if the mercury darkened another addition of mercury was made. An aliquot of the sample was placed in a microvial, capped and was ready for analysis.

The second series were water extract validation samples which were the spiking solution added to methylene chloride, concentrated to 2 mL and introduced to the top of the silica gel cleanup column and hexane then used as the eluate. 10 mL of isooctane was added to the 40 mL eluate and the volume reduced to 2 mL, the volume adjusted to 5 mL after transferring to a centrifuge tube and Hg added. This is the solvent Quality Control.

The standard method for OCs utilized an HP 5730A gas chromatograph equipped with dual capillary column capability, two Ni<sub>63</sub> detectors, two HP 3392a integrators, split/splitless injector and an HP 7671A Autosampler. The paired capillary columns were 30 m long and had an internal diameter of 0.25 mm and a liquid film thickness of 0.25  $\mu$ m. The liquid phases were SPB-1 and SPB-608. The injector was maintained at 250°C and the detectors were at 350°C. The initial temperature of 80°C was held for 2 min, then was programmed at 4°C/min to 260°C which temperature was held for 16 min. The initial purge time was 0.5 min. The carrier gas was  $H_2$  with a head pressure of 15 psi, and the make up gas was Ar/Me (95/5) with a pressure of 45 psi. For the PCB quantitation, an HP 5770A gas chromatograph, equipped with an EC detector, an HP 7672A automatic sampler, and a single packed (OV-1) glass column (3.13 mm x 1.6 m) was used. The carrier gas was Ar/Me as was the makeup The GC was operated in the isothermal mode at 190°C with each gas. determination lasting about 40 min. A solvent blank was injected as every fifth sample.

The initial analysis of the OC results were carried out on the 3392A integrators which had been preprogrammed with a calibration table. These results were then transferred manually to a computer spread-sheet

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and were scrutinized and later smoothed by removing outliers. Very little smoothing occurred before the comparisons presented in this report were undertaken.

For the standard PCB analysis, a modified Webb-McCall method (Webb-Mcall, 1967) was used which can be found in NWQL, 1987.

The new method has been reported elsewhere (Scott et al, 1988) but in brief, the method utilizes an HP 5890 gas chromatography equipped with a split/splitless injector, dual EC<sub>63</sub> detectors each connected to an HP 3392A integrator and an HP-7673A automatic sampler. The temperature program was 2 min at 80°C then the temperature increased 10°C/min to 160° then increased at a rate of 2°C/min until a maximum temperature of 260°C was reached and the run was completed and the oven cooled down. The injector was maintained at a temperature of 250°C, and a purge time of 0.8 min was used.  $H_2$  was used as carrier gas and a head pressure of 17 psi was maintained. Ar/Me (95/5) was the makeup gas at 45 psi. Isooctane was injected as every fourth sample. The GC and ancillary equipment was controlled by an HP-1000 with Real-Time-Executive (RTE) software which controlled the Laboratory Automated System (LAS) software The results were stored in the computer's memory and could be program. accessed for further analysis.

The injections of 1  $\mu$ L per column were made in the splitless mode for the capillary column work and 10  $\mu$ L for the packed column studies.

#### 4.0 RESULTS

Before presenting and discussing the results of the validation samples, the integrity of the method to produce accurate, precise results must first be shown. Table 1 lists the results obtained from a secondary PCB calibration mixture useful for determining retention times, whose concentration is 2150  $pg/\mu L$ . In the table only the sum of the individual results is given. These are from three samples injected

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randomly during the sediment and solvent extract validation. The agreement among the three sets of results are good and are similar to those found before for this solution (Scott et al, 1988). The concentrations derived from both columns are listed in the table, and there are several ways to report or present them, such as averaging the concentrations derived from the two columns. The one preferred is to use the results from one column, in this case, the DB-1 column, and confirm the presence of congeners and the approximate concentration from the results derived from the other column (DB-5). Accordingly, a mean value of 2267  $pg/\mu L$  was calculated which is 5% different from the expected concentration. This is reasonable, considering that the result is the sum of over 60 individual calcuations.

The results for the PCB calibration mixture used to calibrate the standard method using the new method are listed in Table 2(a). These were measured during both parts of the validation study, injected at random. The mean for the new method is  $182 \text{ pg/}_{\mu}$  and this is about 9% from the expected value.

In Table 2(b) the results are listed for the organo-chlorines obtained from the PCB congener method, reported as the average of the results from the two columns. These values are the mean of three analyses. The degree of precision is reasonable as is the error which is the difference between the measured value and the expected value. As the new method was calibrated on a different set of standards, this agreement is quite acceptable.

The results for the water extract validation samples are listed in Table 3. A value of 215  $pg/\mu L$  is calculated for the PCBs using the new method which is 7% higher than the expected value. Using the standard method, a value of 196.4  $pg/\mu L$  is calculated. The coefficient of variance is 11.6 for the standard method and 16.5 for the newer methodology using the DB-1 column results only. Also in this table are the results for the chlorinated hydrocarbons studied, determined using the standard and the new methods. For each analysis, the results of the same compound eluting on the two columns are averaged

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and the mean is derived from these averages. In general. the concentrations derived from the new method are greater than those obtained from using the standard method. This is most evident from the dichlorobenzenes. The concentration for tetrachlorobenzene determined by the PCB congener method is several times higher than that determined by the standard method. This will also be observed in the results for the spiked sediment extracts. When using the standard method, there is an equally high concentration obtained from the SPB-1 column but there is a doublet on the very polar SPB-608 column from which the reported value is calculated. Both columns used in the PCB congener method give rise to higher concentrations. The lower polarity liquid phases cannot resolve the tetrachlorobenzene peak from what is believed to be a chlorinated phenol peak. However, generally the two methods produce similar results and the measure of precision, the coefficient of variance (CV), for all compounds is similar. This measure, as used here, is not only related to the instrumental precision but also the preparation and the capping of the vials that contain the extract for the GC analysis.

The results from the sediment extracts obtained using the new and the standard method are listed in Table 4. The PCB result using the new method is 305 ng/g, whereas the value derived from the standard method is 236.4 ng/g. this is a difference of 70 ng/g. The organo chlorine compound results are also in Table 4. The concentrations for the dichlorobenzenes are less than found for the solvent extract, whereas these values for the most of the other compounds have increased. As was previously mentioned, the concentration of tetrachlorobenzene is a factor of five higher than expected, and this is caused by coelution with another compound believed to have been introduced in the spiking This major peak is not present in the chromatogram of the solution. unspiked sediment. The coefficients of variance for both methods are similar, indicating that the methods have approximately the same precision for these samples. Table 5 contains the concentrations of the sediment extract, unspiked, as analyzed by both methods. The new methodology produces a higher result than the standard method for the

PCB concentration and for the organochlorines that were found to be present. Also, the PCB congener methodology analyzed for more common organo-chlorine components in the sediment than the standard methodology.

#### 5.0 DISCUSSION

There are several points to be considered in comparing the new The first point is: does it produce reproducible results methodology. for the PCBs? The second is: does it produce reasonable results for the OCs? Thirdly, how does it compare with the method now employed, and lastly are there benefits to be derived from using it over methods now used? The first two questions are addressed by considering the results presented in the Tables. The results listed in Table 1 for the PCBs as well as in other reports (Scott et al., 1988; Scott and Onuska, 1989) illustrate that the method does yield reproducible results for the PCBs. The OC analyses were also presented in those reports and there is a degree of reproducibility. The third question is addressed by consideration of the results in Table 6. This contains a summary of the PCB congener method results as well as the summary for the standard method, namely the values for the standards, the unspiked sediment and the By subtracting the amount for the organo-chlorine spiked sediment. found in the sediment from the spiked sediment, a value is obtained that should be similar to the amount of the spike, or better, the amount recovered from the water extract. These results show that the values generated by the standard method are slightly preferable. However. there are three important points to consider. The standard solutions used to calibrate the standard method were the same solutions used in This gives a slight bias to the standard the verification study. method. The precision, as measured by the Coefficient of Variance, is slightly better for the standard method. The aliquots to be analyzed by both methods were prepared, vialed and capped by the same personnel. The vial caps were crimped very tightly to reduce evaporation from the microvials, but this mitigates against good reproducibility as has been shown previously (Scott and Miscunis, 1988). Indeed, in Table 1, a measure of 0.78 for the CV of the PCBs capped correctly must be compared to a CV value of 10 for tightly crimped series of another standard referred to in Table 2. Finally, in response to the fourth question, the new method required only half the time to analyze than the standard method, yet provided considerably more results, e.g., the congener makeup of the PCB fracion. In short, two separate analysis for the compounds of interest have been replaced by a single analysis, which gives results that are generally as good as each other, with the newer method giving additional information.

In addition, the columns used to analyze the samples investigated during this study were installed four months before the study commenced, and other studies were undertaken involving both sediment and water samples. Thus the method appears to be durable. This means that the calibration table needed to quantify the OCs and PCBs need not be changed on a regular basis, which would require considerable time of the operator.

#### ACKNOWLEDGEMENTS

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### ANALYSED PCB VALUES FROM STANDARDS USING PCB CONGENER METHOD (pg/uL)

	Measured Conc.		Coeff. <u>Variance</u>	Expected Conc.	
DB-1 Column	2267	17.6	0.78	2150	
DB-5 Column	(1822)	(202)	(11.1)	2150	

Note: Brackets are around the DB-5 results as the operating column is the DB-1 column

### RESULTS FROM STANDARD METHOD CALIBRATION STANDARDS USING PCB CONGENER METHOD (pg/ul)

(A) PCBs

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	Conc.	Std. C Dev. V	Coeff. Variance	Expected <u>Value</u>
Stand. Meth.	196	17.6	8.9	200
New Methodology (single column)	183	19.1	10.5	200

(B) OCs (dual column)

COMPOUND	Concentration	Expected		
	New Meth.	<u>Concentration</u>		
	54 0	50 0		
1,3-Dichloro*	54.0	50.0		
1,4-Dichloro <sup>1</sup>	51.5	50.0		
1.2-Dichloro <sup>1</sup>	47.4	50.0		
1.3.5-Trichloro <sup>2</sup>	5.1	5,.0		
1.2.4-Trichloro <sup>2</sup>	5.1	.5.0		
1.2.3-Trichloro <sup>2</sup>	5.3	5.0		
Hexachlorocyclp <sup>3</sup>	4.8	5.0		
Tetrachlorobenzene	5.7	5.0		
Pentachlorobenzene	5.7	5.0		
Hexachlorobenzene	5.7	5.0		
Heptachlor	5.5	5.0		
Aldrin	5.1	5.0		
Octachlorostyrene	9.4	10.0		
o.p'-DDT	17.4	15.0		
$\mathbf{n}, \mathbf{n'} \rightarrow \mathbf{DDT}$	16.5	15.0		
p, p' = DDF	11.4	15.0		
Photomirex	11.7	10.0		
Mirex	11.2	10.0		

<sup>1</sup> dichlorobenzene

<sup>2</sup> trichlorobenzene

<sup>3</sup> hexachlorocyclopentadiene

# COMPARISON OF RESULTS BETWEEN STANDARD AND NEW METHODS FOR WATER EXTRACT TECHNIQUE

(pg/uL)

(A) PCBs

	Conc.	<u>Std. Dev.</u>	Coeff. of Var.	<u>n</u>
Stand. Method	197.6	17.6	8.9	10
New Method	215	35.3	17	8

### (B) OCs

	CONCENTI	RATION	STD. DEV.		CV	
	_1@	2‴	<u>]@</u>	_2**	10	2"
1,3-Dichloro <sup>1</sup>	37.5	45.9	3.5	5.4	9.3	12.
1,4-Dichloro <sup>1</sup>	37.4	44.5	3.7	4.5	9.6	10.
1.2-Dichloro <sup>1</sup>	37.1	43.7	3.4	5.0	9.2	12.
1.3.5-Trichloro <sup>2</sup>	4.6	5.3	0.4	0.5	8.0	9.9
1.2.4-Trichloro <sup>2</sup>	4.9	5.3	0.4	0.5	7.7	9.8
1.2.3-Trichloro <sup>2</sup>	4.8	5.5	0.4	0.5	8.6	10.
Hexachlorocyclp <sup>3</sup>	3.7	3.7	0.5	0.7	13.	19.
Tetrachlorobenzen	e 5.2	25.7	0.4	1.9	8.3	7.6
Pentachlorobenzen	le 5.6	7.1	0.5	0.7	5.6	11.
Hexachlorobenzene	5.6	7.5	0.5	0.8	8.7	11.
Heptachlor	4.2	5.2	0.4	1.0	10.	20.
Aldrin	5.4	8.7	0.6	1.2	12.	13.
Octachlorostyrene	11.3	11.5	1.6	1.4	14.	12.
o,p'-DDT	16.6	15.6	4.0	2.8	21.	18.
p,p'-DDT	23.7	19.6	4.6	5.2	19.	26.
p,p'=DDE	14.2	16.2	3.0	2.2	21.	14.
Photomirex	11.7	15.1	2.0	1.7	17.	11.
Mirex	11.6	12.6	2.2	1.7	19.	13.

<sup>1</sup> dichlorobenzene

<sup>2</sup> trichlorobenzene

<sup>3</sup> hexachlorocyclopentadiene

results of standard methodology
results of new methodology

### COMPARISON OF RESULTS BETWEEN STANDARD AND NEW METHOD FOR SEDIMENT EXTRACT TECHNIQUE (pg/uL)

(A) PCBs	Conc.	Std. Dev.	Coeff. of Var.	<u>n</u>
Stand. Method	236.0	26.4	11	10
New Method	304	37.0	12	10

### (B) OCs

C	CONCENTRATION		STD. DEV.		<u> </u>	_
-	_1ª	2**	<u>1°</u>	2‴	_ <u>1°</u>	_2‴
1.3-Dichloro <sup>1</sup>	35.3	36.0	4.6	5.0	13.	14.
1 4-Dichloro <sup>1</sup>	36.3	35.6	4.4	5.1	12.	14.
1.2-Dichloro <sup>1</sup>	36.6	36.1	4.8	5.2	13.	14.
1.3.5-Trichloro <sup>2</sup>	4.4	5.0	0.7	1.3	15.	20.
1.2.4-Trichloro <sup>2</sup>	6.7	7.5	0.6	0.6	9.2	7.8
$1.2.3 - Trichloro^2$	5.0	4.6	0.7	0.8	18.	17.
Hexachlorocvclp <sup>3</sup>	2.5	3.1	0.6	0.8	24.	25.
Tetrachlorobenzene	8.7	23.3	0.7	1.7	8.3	7.1
Pentachlorobenzene	2 7.6	11.0	0.9	1.3	11.	12.
Hexachlorobenzene	9.2	13.4	2.9	4.6	31.	31.
Hentachlor	4.0	4.9	1.3	2.4	33.	49.
Aldrin	4.8	13.2	0.9	2.0	20.	16.
Octachlorostvrene	9.7	12.3	1.0	1.1	10.	9.2
O.P'-DDT	14.8	15.6	2.6	2.1	17.	13.
$\mathbf{p}$ , $\mathbf{p}^{\dagger}$ – $\mathbf{D}\mathbf{D}\mathbf{T}$	18.6	20.2	2.8	3.9	15.	19.
p.p'-DDE	13.9	20.5	2.0	5.0	14.	24.
Photomirex	9.7	14.3	1.5	2.4	15.	17.
Mirex	9.5	12.1	1.4	2.4	15.	20.

<sup>1</sup> dichlorobenzene

<sup>2</sup> trichlorobenzene

<sup>3</sup> hexachlorocyclopentadiene

results using standard method results using new method

# COMPARISON OF RESULTS DERIVED FROM THE UNSPIKED SEDIMENT (conc. in ng/g)

- (A) PCBs 46.5<u>+</u>3.8 71.2<u>+</u>5.0 Standard Method New Method
- (B) OC's

· · · · · · · · · · · · · · · · · · ·	CONCENTRATION			
	1 <sup>@</sup>			
1,3-Dichloro <sup>1</sup>		1.6		
1,4-Dichloro <sup>1</sup>		2.5		
1,2-Dichloro <sup>1</sup>		5.1		
1,3,5-Trichloro <sup>2</sup>		0.4		
1,2,4-Trichloro <sup>2</sup>	2.5	3.0		
1.2.3-Trichloro <sup>2</sup>		7.3		
Hexachlorocyclp <sup>3</sup>				
Tetrachlorobenzene	3.8	4.9		
Pentachlorobenzene	2.7	.3.9		
Hexachlorobenzene	3.1	5.4		
Hentachlor		Ó.5		
Aldrin		9.6		
Octachlorostyrene		1.5		
o n'-DDT	1.7			
	- • •	1.4		
p, p = DDI	2.6	3.8		
p,p -DDE Dhatomirey		0,4		
PHO LUMITEX		0.4		
MIREX		•••		

<sup>1</sup> dichlorobenzene

<sup>2</sup> trichlorobenzene

<sup>3</sup> hexachlorocyclopentadiene

• results using standard method

" results using new method

COMPOUND	SPIKED SEDIMENT		SEDIMENT ONLY		DIFFER- _ENCE		EXTRACT	
	<u>e</u>	41-	_@			#	<u>e</u>	
PCB	236	304	46.5	71.2	189	232.8	196	215
1,2-Dichloroben <sup>1</sup>	35.3	36.0		1.6	35.3	34.5	37.5	45.9
1,3-Dichloroben <sup>1</sup>	36.3	35.6		2.5	36.3	33.1	37.4	44.5
1,4-Dichloroben <sup>1</sup>	36.6	36.1		5.1	36.6	31.0	37.1	43.7
1,3,5-Trichlorb <sup>2</sup>	4.4	5.0		0.4	4.4	4.6	4.7	5.3
1,2,4-Trichlorb <sup>2</sup>	6.7	7.5	2.5	2.9	6.7	4.6	4.9	5.3
1,2,3-Trichlorb <sup>2</sup>	4.9	4.6		7.3	4.9		4.8	55
Hexachlorocyc1h <sup>3</sup>	2.5	3.1			2.5	3.1	3.7	3.7
Tetrachlorobenze	ne 8.7	23.3	3.8	4.5	.5.0	18.8	5.2	
Pentachlorobenze	ne 7.6	<u>11.0</u>	2.7	3.9	4.9	7.1	- 4.8	7.0
Hexachlorobenzen	e 9.2	16.7	3.1	5.4	6.1	11.3	5.6	7.5
Heptachlor	4.0	4.9			4.0	4.9	4.2	5.2
Aldrin	4.7	13.2		9.6	4.7	3.6	5.4	8.7
Octachlorostyren	e 9.7	12.3		1.5	9.7	10.8	11.3	11.5
o,p'-DDT	14.8	15.6	1.7		13.1	15.6	18.3	15.6
<b>p</b> , <b>p'</b> -DDT	18.6	20.2		1.4	18.6	18.8	23.7	19.6
p,p'-DDE	13.9	20.5	2.6	3.8	11.3	16.7	14.2	16.2
Photomirex	9.7	14.3		0.4	9.7	13.9	11.7	15.1
Mirex	9.5	12.1		0.4	9.5	11.7	11.6	12.6

## (A) SUMMARY OF RESULTS FOR BOTH METHODS in ng/g or equivalent

<sup>1</sup> dichlorobenzene

<sup>2</sup> trichlorobenzene

<sup>3</sup> hexachlorocyclopentadiene

• results using standard method

" results using new method