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TITLE: Analysis of Montreal LaSalle Plant Waste Samples for PCBs.

AUTHORS: B. M. Hughes, D. E. McKenzie and L. R. Minor

ABSTRACT: The Montreal LaSalle Plant requested that ESC conduct low level PCB analyses on six samples to determine proper waste disposal methodology. Samples included organic wastes primarily consisting of phthalate esters and a water/sediment aqueous waste. Two sets of samples were received for analysis. In both cases the organic matrix samples #101, #108 and #110 contained PCBs ranging from 3.7 to 7.7 ug/g. Aqueous phases and sediment from aqueous samples #102, #108B, and #110B ranges from ND to 3.9 ug/g. All analyses were conducted with a method limit of detection of 0.1 ug/g per PCB congener.

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TECHNICAL APPROVAL:

P. L. Sherman
P. L. Sherman
Environmental Sciences Center
Sr. Research Group Leader

APPROVAL BY:

J. M. Lee
J. M. Lee
Environmental Sciences Center
QA and GLP Supervisor

APPROVAL DATE:

October 21, 1987

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ANALYSIS REPORT

DATE: October 9, 1987

LOG NUMBER: U-87-09-08-02
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PROJECT NO: 760.28-7634
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TO: Christian Roy and Myra Plotnik (5090)
Montreal Plant

FROM:

Environmental Sciences Center
Monsanto Company
800 W. Lindbergh Blvd.
St. Louis, Mo. 63167

Phone No: (314) 694-1464

Title: Analysis of Montreal LaSalle Plant Waste Samples for PCBs

ABSTRACT

The Montreal LaSalle Plant requested that ESC conduct low level PCB analyses on six samples to determine proper waste disposal methodology. Samples included organic wastes primarily consisting of phthalate esters and a water/sediment aqueous waste. Two sets of samples were received for analysis. In both cases the organic matrix samples #101, #108 and #110 contained PCBs ranging from 3.7 to 7.7 ug/g. Aqueous phases and sediment from aqueous samples #102, #108B, and #110B ranges from ND to 3.9 ug/g. All analyses were conducted with a method limit of detection of 0.1 ug/g per PCB congener.

INTRODUCTION

Six samples were received from the Montreal Plant for PCB analyses. Prior to analyses it was determined that a limit of detection of 0.1 ug/g per PCB congener would be required, because the material in question was subject to a regulated level of 1 ug/g for disposal. Aqueous samples were centrifuged prior to extraction and the water and sediment were treated as separate samples. Samples were prepared using Standard Extraction Method PCX115 omitting the cleanup column step. The methodology involved weighing the samples into a Teflon capped vial and spiking the samples with carbon 13 labeled PCB congeners, and adding 10 mL 20% aqueous KOH to the vials. The vials were capped and warmed in a sandbath at approximately 60 degrees C. for 1 hour. Samples were then extracted with 25 mL Burdick and Jackson UV grade hexane. The volume of the extract was reduced in a vernal sandbath under nitrogen stream to a final volume of 1 mL if possible. The extracts were then subjected to capillary GC/MS analysis using Standard Analysis Method MSAN25. Selected ion monitoring was utilized for all analyses. Characteristic and confirming ions were monitored over appropriate retention time ranges and the ratio of characteristic to confirming response was

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calculated for all compounds detected. (See PCSC25 for more data acquisition information.) Data was accepted if the ratio matched corresponding standard congener ratios of plus or minus fifty percent. If a component was detected and the ratio was out of this range, it was assumed that the compound was not a PCB congener and the concentration was not calculated. Standard Extraction and Analysis Methods are listed in a later section of this report. A list of spiking compounds used in this study and characteristic and confirming ions monitored are listed in the Additional Information section of this report.

All concentrations were calculated using internal standard methodology, which is listed in Standard Analysis Method MSAN25. Calculations were based on a series of response factors calculated for each chlorine class from three standards each containing 24 known congeners. The standard contained the components listed in the Additional Information section of this report under the heading Native Spiking Compounds. Actual individual and average chlorine class response factors used in concentration calculations are in Tables 1b and 2b.

SUMMARY

Results of analyses are given in Tables 1 and 2. Concentrations ranged from ND (< 0.1 ug/g) per congener to 7.7 ug/g total PCB congeners in the unspiked samples. These tables list the concentration of PCBs detected per chlorine class in the extract, the summed total concentration of PCBs in the extract, the concentration of total PCBs in ug/g (corrected for sample amount and extract volume), and the percent recovery of labeled surrogates if they were added to the samples. Following Tables 1 and 2 are their a and b counterparts which list the ratio of characteristic ions to confirming ions of components detected, and response factors which were used to calculate concentrations. Samples are named with their identification number. DUP refers to duplicate analysis, SED refers to the sediment portion of an aqueous sample, + LS refers to a native spike addition to a sample of 0.1 ug each of 24 PCB congeners, and + NS indicates the addition of 1 ug each of 24 PCB congeners to the matrix. These standard materials are listed in the Additional Information Section of this report. The ratio of CL3-, CL4-, CL5-, CL6-, and CL7-biphenyl congeners indicate that the total PCBs which are present are from mixtures of Aroclor 1242 and 1254.

QC RESULTS

Samples #101 and #102 + #102-SED were spiked with 4 carbon-13 labeled PCB homologues. Recoveries ranged from 4 to 128%. After careful inspection of the data several very low spurious recoveries were attributed to chromatographic interferences, and mass spectrometer detuning during data collection. This detuning was not deemed to be serious since it only occurred for the higher molecular weight PCBs (CL8 through CL10), and samples that contained PCBs most likely contained either Aroclor 1242 or Aroclor 1254. Neither of these formulations contain significant amounts of the higher molecular weight PCBs. Some spurious results were also found in the native spiking results for lower chlorinated (CL1-CL2) biphenyls, which were due to chromatographic interferences. It is assumed that these values do not affect the conclusions of this report. In addition, low and high level native spikes (addition of 0.1 ug each for 24 native congeners and the addition of 1 ug each for 24 congeners) were performed on all matrices. Native spiking recoveries for the vast majority of reported recoveries ranged from 50 to 150% recovery. A second group of samples was extracted without the addition of carbon 13 labeled surrogates due to

lack of surrogate compounds at the time of extraction. These samples were also spiked with the native spiking standard. The recoveries from these studies indicated that the reported sample values are reliable. Individual surrogate compound recoveries are listed in Table 1, and native spiking recoveries are given in Tables 3 and 4. Some standards were also analyzed as samples in order to test the reliability of average congener response calculations. Results of analysis of LS SPK (low native spike standard) and HS SPK (high native spike standard) indicate the analysis is reliable.

DETAILS

Standard Sampling Method No(s): Samples supplied by the Montreal LaSalle Plant.

Standard Extraction Method No(s): PCX15 (See also flow sheet PCFS15)

Standard Analysis Method No(s): MSAN25 (See also flow sheet MSFS25)

Mass Spectrometer Data Acquisition Method: PCSC25

Sample Identification(s): See login information

Final Report File No(s): RA7634

Table File No(s): a:\pcb\TA7636 and a:\pcb\TA7634

Also included in this report are:

- (1) Brief descriptions and flow diagrams of Standard Extraction, Analysis and Mass Spectrometer Data Acquisition Methods used in this study.
- (2) Example of a raw data output (GC/MS) chromatogram obtained in this study.
- (3) Chain-of-Custody documentation for this study.
- (4) Analytical Request information generated at sample login.
- (5) Description of important terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis.

ADDITIONAL INFORMATION

This section describes compounds used in this study as standards and provides information on mass ranges monitored during analyses using selected ion monitoring.

Internal Standard

Anthracene-d10

Surrogate Spiking Compounds C-13 labeled

4-chlorobiphenyl 13C6
3,3',4,4'-tetrachlorobiphenyl 13C12
2,2',3,3',5,5',6,6'-octachlorobiphenyl

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decachlorobiphenyl 13C12

Native Spiking Compounds (used for native spikes)

 2-monochlorobiphenyl
 3-monochlorobiphenyl
 4-monochlorobiphenyl
 2,2'-dichlorobiphenyl
 2,5-dichlorobiphenyl
 2,4'-dichlorobiphenyl
 4,4'-dichlorobiphenyl
 2,4,6-trichlorobiphenyl
 2,3,4-trichlorobiphenyl
 2,4,4'-trichlorobiphenyl
 2,3',4,6,-tetrachlorobiphenyl
 2,3,5,6,-tetrachlorobiphenyl
 2,2',4,4',-tetrachlorobiphenyl
 2,2',3,4,5-pentachlorobiphenyl
 2,2',3,4,5'-pentachlorobiphenyl
 2,2',4,5,5'-pentachlorobiphenyl
 2,2',3,3',4,4'-hexachlorobiphenyl
 2,2',3,4,4',5'-hexachlorobiphenyl
 2,2',3,3',6,6'-hexachlorobiphenyl
 2,2',3,3',4,4',6-heptachlorobiphenyl
 2,2',3,4,4',5',6-heptachlorobiphenyl
 2,2',3,3',4,4',5,5'-octachlorobiphenyl
 2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl
 decachlorobiphenyl

Characteristic and Confirming Masses Monitored

	Characteristic (AMU)	Confirming (AMU)
anthracene-d10	188	190
4-chlorobiphenyl 6C13	194	196
3,3',4,4'-tetrachlorobiphenyl 12C13	301.8	303.8
2,2',3,3',5,5',6,6'-octachlorobiphenyl 12C13	439.7	441.7
Decachlorobiphenyl 12C13	509.6	511.6
mono-chlorobiphenyls	188	190
dichlorobiphenyls	222	224
trichlorobiphenyls	255.9	257.9
tetrachlorobiphenyls	289.8	291.8
pentachlorobiphenyls	323.9	325.9
hexachlorobiphenyls	357.8	359.8
heptachlorobiphenyls	323.9	325.9
octachlorobiphenyls	427.7	429.7
nonachlorobiphenyls	461.6	463.6
decachlorobiphenyl	497.6	499.6

ANALYST(S): B. Mason Hughes, D. E. McKenzie, and LaShawn Minor

ANALYST APPROVAL: B. Mason Hughes

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Table 1. Table of PCB Concentrations Detected in Montreal Samples Using Standard Extraction and Analysis Methods PCB175 and PCB225. (Project 7634)

FORM USED FOR CALIBRATION: +1.6405 +1.6406 +1.6413

SAMPLE ID, ...	ST01	ST01 DUP	ST02	ST02 DUP	Rech. B/L	ST02 Sed	ST02 SED DUP	Rech. B/L	NO SPE	LS SPE
ESTRACT NUMBER ...	PCB-2140 (g)	PCB-2150 (g)	PCB-2154 (g)	PCB-2155 (g)	PCB-2144 (g)	PCB-2150 (g)	PCB-2140 (g)	PCB-2145 (g)	NO SPE	LS SPE
CONCENTR. CLASS	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Monochlorobiphenyls	0	0	0	0	0	0	0	0	0	3.0
Dichlorobiphenyls	0.1	0.1	0	0	0	0	0	0	0	5.1
Trichlorobiphenyls	0.7	0.9	0	0	0	0	0.1	0.1	0	3.6
Tetrachlorobiphenyls	0.0	0.9	0	0	0	0	0.1	0	0	2.7
Pentachlorobiphenyls	2.6	2.4	0	0	0	0	0.2	0.1	0	5.4
Hexachlorobiphenyls	1.6	1.6	0	0	0	0	0.4	0.2	0	2.9
Heptachlorobiphenyls	0.2	0.4	0	0	0	0	0.1	0.1	0	2.1
Octachlorobiphenyls	0	0.1	0	0	0	0	0	0	0	1.1
Nonachlorobiphenyls	0	0	0	0	0	0	0	0	0.1	1.2
Decachlorobiphenyl	0	0	0	0	0	0	0	0	0	0.9
NOH, PCB NO ESTRACT (ug/mL)	0.4	0.6	0	0	0	0.9	0.5	0.1	0.1	27
NOH, PCB NO SAMPLE (ug/g)	4	7.7	0	0	0	1	0.8	0.1	0.1	27
CONCENTR. COMPOND	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)	(% HOC)
Monochlorobiphenyl - OC13	71	99	79	77	85	101	92	105	133	123
Tetrachlorobiphenyl - 12C13	79	100	126	100	55	96	94	75	76	70
Octachlorobiphenyl - 12C13	47	61	125	110	79	90	101	110	110	96
Decachlorobiphenyl - 12C13	70	70	110	113	66	75	90	100	90	10
NO SED FILE	+1.6405	+1.6406	+1.6407	+1.6408	+1.6411	+1.6409	+1.6410	+1.6412	+1.6413	+1.6416
EXTRACTION FACTOR	0.944	1.140	0.775	1.125	1	1.089	1.080	1	1	1
SAMPLE AMOUNT (g)	1.099	1.204	1.29	0.889	1	0.944	0.622	1	1	1
EXTRACT FINAL VOLUME (mL)	1	1.5	1	1	1	1	1	1	1	1
ADDITIONAL 950 (1030) AREA	1777553	1900900	1750000	1300501	1405147	1471011	1300591	1202057	1107912	1101000
Notes: Concentrations reported are rounded to 1 decimal place. Values of 0 should be interpreted as < 0.1 ug/g.										

Table Generation Comments:

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Table 1a

TABLE OF CHARACTERISTIC IRR RESPONSES TO CONFINEMENT TIMES FOR POB SAMPLES 1-10 FOR SAMPLES IN CONCENTRATION FOR TABLE:
 Table 1a Table of PCB Concentrations Detected in Montreal Samples Using Standard
 Extraction and Analysis Methods PCB15 and HMO275. (Project: 7834)

CONFINED ID	STANDARD USED IN CALCULATIONS										FILE NAMES										MINIMUM		MAXIMUM		AVERAGE																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																									
	S1		S2		S3		F1		F2		F3		F4		F5		F6		F7		F8		F9		F10		F11		F12		F13		F14		F15		F16		F17		F18		F19		F20		F21		F22		F23		F24		F25		F26		F27		F28		F29		F30		F31		F32		F33		F34		F35		F36		F37		F38		F39		F40		F41		F42		F43		F44		F45		F46		F47		F48		F49		F50		F51		F52		F53		F54		F55		F56		F57		F58		F59		F60		F61		F62		F63		F64		F65		F66		F67		F68		F69		F70		F71		F72		F73		F74		F75		F76		F77		F78		F79		F80		F81		F82		F83		F84		F85		F86		F87		F88		F89		F90		F91		F92		F93		F94		F95		F96		F97		F98		F99		F100		F101		F102		F103		F104		F105		F106		F107		F108		F109		F110		F111		F112		F113		F114		F115		F116		F117		F118		F119		F120		F121		F122		F123		F124		F125		F126		F127		F128		F129		F130		F131		F132		F133		F134		F135		F136		F137		F138		F139		F140		F141		F142		F143		F144		F145		F146		F147		F148		F149		F150		F151		F152		F153		F154		F155		F156		F157		F158		F159		F160		F161		F162		F163		F164		F165		F166		F167		F168		F169		F170		F171		F172		F173		F174		F175		F176		F177		F178		F179		F180		F181		F182		F183		F184		F185		F186		F187		F188		F189		F190		F191		F192		F193		F194		F195		F196		F197		F198		F199		F200		F201		F202		F203		F204		F205		F206		F207		F208		F209		F210		F211		F212		F213		F214		F215		F216		F217		F218		F219		F220		F221		F222		F223		F224		F225		F226		F227		F228		F229		F230		F231		F232		F233		F234		F235		F236		F237		F238		F239		F240		F241		F242		F243		F244		F245		F246		F247		F248		F249		F250		F251		F252		F253		F254		F255		F256		F257		F258		F259		F260		F261		F262		F263		F264		F265		F266		F267		F268		F269		F270		F271		F272		F273		F274		F275		F276		F277		F278		F279		F280		F281		F282		F283		F284		F285		F286		F287		F288		F289		F290		F291		F292		F293		F294		F295		F296		F297		F298		F299		F300		F301		F302		F303		F304		F305		F306		F307		F308		F309		F310		F311		F312		F313		F314		F315		F316		F317		F318		F319		F320		F321		F322		F323		F324		F325		F326		F327		F328		F329		F330		F331		F332		F333		F334		F335		F336		F337		F338		F339		F340		F341		F342		F343		F344		F345		F346		F347		F348		F349		F350		F351		F352		F353		F354		F355		F356		F357		F358		F359		F360		F361		F362		F363		F364		F365		F366		F367		F368		F369		F370		F371		F372		F373		F374		F375		F376		F377		F378		F379		F380		F381		F382		F383		F384		F385		F386		F387		F388		F389		F390		F391		F392		F393		F394		F395		F396		F397		F398		F399		F400		F401		F402		F403		F404		F405		F406		F407		F408		F409		F410		F411		F412		F413		F414		F415		F416		F417		F418		F419		F420		F421		F422		F423		F424		F425		F426		F427		F428		F429		F430		F431		F432		F433		F434		F435		F436		F437		F438		F439		F440		F441		F442		F443		F444		F445		F446		F447		F448		F449		F450		F451		F452		F453		F454		F455		F456		F457		F458		F459		F460		F461		F462		F463		F464		F465		F466		F467		F468		F469		F470		F471		F472		F473		F474		F475		F476		F477		F478		F479		F480		F481		F482		F483		F484		F485		F486		F487		F488		F489		F490		F491		F492		F493		F494		F495		F496		F497		F498		F499		F500		F501		F502		F503		F504		F505		F506		F507		F508		F509		F510		F511		F512		F513		F514		F515		F516		F517		F518		F519		F520		F521		F522		F523		F524		F525		F526		F527		F528		F529		F530		F531		F532		F533		F534		F535		F536		F537		F538		F539		F540		F541		F542		F543		F544		F545		F546		F547		F548		F549		F550		F551		F552		F553		F554		F555		F556		F557		F558		F559		F560		F561		F562		F563		F564		F565		F566		F567		F568		F569		F570		F571		F572		F573		F574		F575		F576		F577		F578		F579		F580		F581		F582		F583		F584		F585		F586		F587		F588		F589		F590		F591		F592		F593		F594		F595		F596		F597		F598		F599		F600		F601		F602		F603		F604		F605		F606		F607		F608		F609		F610		F611		F612		F613		F614		F615		F616		F617		F618		F619		F620		F621		F622		F623		F624		F625		F626		F627		F628		F629		F630		F631		F632		F633		F634		F635		F636		F637		F638		F639		F640		F641		F642		F643		F644		F645		F646		F647		F648		F649		F650		F651		F652		F653		F654		F655		F656		F657		F658		F659		F660		F661		F662		F663		F664		F665		F666		F667		F668		F669		F670		F671		F672		F673		F674		F675		F676		F677		F678		F679		F680		F681		F682		F683		F684		F685		F686		F687		F688		F689		F690		F691		F692		F693		F694		F695		F696		F697		F698		F699		F700		F701		F702		F703		F704		F705		F706		F707		F708		F709		F710		F711		F712		F713		F714		F715		F716		F717		F718		F719		F720		F721		F722		F723		F724		F725		F726		F727		F728		F729		F730		F731		F732		F733		F734		F735		F736		F737		F738		F739		F740		F741		F742		F743		F744		F745		F746		F747		F748		F749		F750		F751		F752		F753		F754		F755		F756		F757		F758		F759		F760		F761		F762		F763		F764		F765		F766		F767		F768		F769		F770		F771		F772		F773		F774		F775		F776		F777		F778		F779		F780		F781		F782		F783		F784		F785		F786		F787		F788		F789		F790		F791		F792		F793		F794		F795		F796		F797		F798		F799		F800		F801		F802		F803		F804		F805		F806		F807		F808		F809		F810		F811		F812		F813		F814		F815		F816		F817		F818		F819		F820		F821		F822		F823		F824		F825		F826		F827		F828		F829		F830		F831		F832		F833		F834		F835		F836		F837		F838		F839		F840		F841		F842		F843		F844		F845		F846		F847		F848		F849		F850		F851		F852		F853		F854		F855		F856		F857		F858		F859		F860		F861		F862		F863		F864		F865		F866		F867		F868		F869		F870		F871		F872		F873		F874		F875		F876		F877		F878		F879		F880		F881		F882		F883		F884		F885		F886		F887		F888		F889		F890		F891		F892		F893		F894		F895		F896		F897		F898		F899		F900		F901		F902		F903		F904		F905		F906		F907		F908		F909		F910		F911		F912		F913		F914		F915		F916		F917		F918		F919		F920		F921		F922		F923		F924		F925		F926		F927		F928		F929		F930		F931		F932		F933		F934		F935		F936		F937		F938		F939		F940		F941		F942		F943		F944		F945		F946		F947		F948		F949		F950		F951		F952		F953		F954		F955		F956		F957		F958		F959		F960		F961		F962		F963		F964		F965		F966		F967		F968		F969		F970		F971		F972		F973		F974		F975		F976		F977		F978		F979		F980		F981		F982		F983		F984		F985		F986		F987		F988		F989		F990		F991		F992		F993		F994		F995		F996		F997		F998		F999		F1000		F1001		F1002		F1003		F1004		F1005		F1006		F1007		F1008		F1009		F1010		F1011		F1012		F1013		F1014		F1015		F1016		F1017		F1018		F1019		F1020		F1021		F1022		F1023		F1024		F1025		F1026		F1027		F1028		F1029		F1	

Table 1b

Table of Average Relative Response Factors
Generated for Each Congener Class

	Characteristic Relative Response Factors		Response from standard3	Average Response Factor	Average Congener Response
	Response from standard1	Response from standard2			
Cl1 biphenyl #2	0.0272	0.0305	0.0432	0.033633	0.033633
Cl1 biphenyl #3	0.0292	0.0339	0.0405	0.0372	0.0406
Cl1 biphenyl #4	0.0619	0.0601	0.0949	0.074966	
Cl4biphenyl 4C1305	0.0302	0.0436	0.0664	0.050066	0.050066
Cl2biphenyl #6	0.0215	0.0262	0.0364	0.020053	
Cl2biphenyl #7	0.0441	0.0521	0.0734	0.050533	0.043166
Cl2biphenyl #8	0.0372	0.0351	0.0493	0.040533	
Cl2biphenyl #9	0.0453	0.0409	0.0725	0.055366	
Cl2biphenyl #10	0.0217	0.0223	0.0200	0.024366	
Cl2biphenyl #11	0.0233	0.0256	0.0320	0.027233	0.0250
Cl2biphenyl #12	0.0099	0.0266	0.0322	0.0259	
Cl4biphenyl #13	0.02	0.0161	0.016	0.017366	
Cl4biphenyl #14	0.0151	0.0129	0.0120	0.0136	0.016666
Cl4biphenyl #15	0.022	0.0171	0.0162	0.010433	
Cl4biph 12C13016	0.0353	0.0292	0.0210	0.020766	0.020766
Cl5biphenyl #17	0.00093	0.00762	0.0115	0.00935	
Cl5biphenyl #18	0.00934	0.00644	0.00919	0.008525	0.008674
Cl5biphenyl #19	0.00770	0.00667	0.0106	0.00835	
Cl4biphenyl #20	0.00710	0.00505	0.0072	0.006713	
Cl4biphenyl #21	0.00702	0.00502	0.00627	0.00637	0.00633
Cl6biphenyl #22	0.00707	0.00406	0.0049	0.005076	
Cl7biphenyl #23	0.00643	0.00553	0.0072	0.006433	0.007003
Cl7biphenyl #24	0.00000	0.00595	0.00703	0.007553	
Cl8biphenyl #25	0.0041	0.00202	0.00399	0.003636	0.003636
Cl8biph 12C13026	0.0163	0.0155	0.0103	0.0167	0.0167
Cl9biphenyl #27	0.00377	0.00339	0.00491	0.004025	0.004025
Cl10biphenyl #28	0.00465	0.00264	0.00317	0.003406	0.003406
Cl10biph 12C13029	0.00053	0.00073	0.00045	0.00057	0.00057

Table 2. Table of PCB Concentrations Detected in Riverine Sample Using Standard Extractions and Analysis Methods PCB15 and BSM25. (Project: TBM)

FORM USED FOR CALIBRATION: +1.6447 +1.6439 +1.6448

SAMPLE NO. ---	TL 0100	TL 0100 DUP	TL 0110	DL 01000	DL 01000 DUP	DL 01100	DL 01100 DUP	TL 0100 + DL	DL 01000 + DL	DL 01100 + DL
SUBSTANT NAME ---	PCB-2175	PCB-2174	PCB-2176	PCB-2180	PCB-2182	PCB-2184	PCB-2186	PCB-2175 +	PCB-2180 +	PCB-2184 +
CONCENTRATION CLASS	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)	(ug/mL)
Monochlorobiphenyl	0	0	0	0	0	0	0.3	7.3	4.9	3.3
Dibromobiphenyl	0.3	0.4	0.4	0	0	0.1	0.1	5.9	6.3	4.3
Trichlorobiphenyl	0.7	0.9	1.3	0	0.1	0.1	0.2	3.9	4.6	3.3
Tetrachlorobiphenyl	0.5	0.5	0.8	0	0.1	0	0.2	2.6	4.9	3.6
Pentachlorobiphenyl	1	1	1.5	0	0.4	0.1	0.7	2.7	5	3.6
Hexachlorobiphenyl	0.5	0.6	0.9	0	0.5	0.1	0.4	1.4	5.1	3.7
Heptachlorobiphenyl	0.2	0.2	0.4	0	0.1	0.1	0.1	0.4	4	2.7
Octachlorobiphenyl	0	0	0.3	0	0	0.1	0.1	0.2	2.1	1.5
Nonachlorobiphenyl	0	0	0	0	0	0.1	0.1	0.2	2.2	1.4
Decachlorobiphenyl	0	0	0	0	0	0	0	0.1	2	1.3
TOTAL PCBs BY EXTRACT (ug/mL)	3.2	3.4	5.6	0	1	0.7	2.2	26.9	41.3	26.7
TOTAL PCBs IN SAMPLE (ug/g)	3.7	5.9	3.7	0	2	0.7	5.9	25.4	41.4	26.3
DEBROMANE COMPOUND	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)	(% REC)
Monochlorobiphenyl - DC13	0	0	0	0	0	0	0	0	0	0
Tetrachlorobiphenyl - DC13	0	0	0	0	0	0	0	0	0	0
Hexachlorobiphenyl - DC13	0	0	0	0	0	0	0	0	0	0
Nonachlorobiphenyl - DC13	0	0	0	0	0	0	0	0	0	0
DEB FACTOR	+1.6439	+1.6440	+1.6444	+1.6448	+1.6450	+1.6452	+1.6454	+1.6461	+1.6460	+1.6453
DEBWEIGHT FACTOR	1.145	1.636	1.616	0.90	1.953	0.970	1.789	1.82	1.888	0.988
SAMPLE WEIGHT (g)	0.075	0.017	0.004	1.02	0.312	1.023	0.350	0.999	0.999	1.004
EXTRACTED PCB VOLUME (mL)	1	1.5	1	1	1	1	1	1	1	1
EXTRACTED PCB (1000) AREA	1277083	1146328	964895	9490095	1760048	1410023	1764501	1110769	1604442	1760953
Notes: Concentration reported are rounded to 1 decimal place. Values of 0 should be interpreted as < 0.1 ug/g.										

Table Generation Comments:

ALL FILES TEST OK

Note! These samples were not spiked with surrogate compounds.

MONS 024670

TABLE OF CHARACTERISTIC PEAK RESPONSES TO CONCENTRATIONS TIMES 100 FOR SAMPLES 1-10 FOR SAMPLES IN CONCENTRATION TABLE:
Table B2, Table of PEK Concentrations Detected in Neutral Samples Using Standard
Extraction and Analysis Methods PC2E15 and REAGENT. (Project: 7636)

CONCENTRATED IN	STANDARD USED IN CALCULATION										FILE NAMES										RECOVERY		
	61	62	63	71	72	73	74	75	76	77	78	79	710	711	712	713	714	715	716	717	718	719	720
	>14447	>14435	>14448	>14439	>14448	>14444	>14444	>14430	>14452	>14434	>14441	>14449	>14453										
001 = anthracene-9H	9053	9055	0970	0975	0830	0940	0860	9127	2017	0962	0610	0963	9104	0970	9080	9080							
002 = 2,3-DI-biphenyl	299	295	295	0	0	0	0	107	716	448	290	301	290	290	290	290							
003 = 2,3-DI-1,2,3,4	299	250	209	0	0	0	0	0	0	0	0	0	0	250	290	290							
004 = "	299	270	209	0	0	0	0	0	0	0	0	0	0	270	290	290							
005 = 4-CL-biphenyl-1,2,3,4	0	0	0	0	0	0	0	0	221	34	0	0	200	0	0	0							
006 = 2,3-DI-1,2,3,4	154	145	154	100	202	211	0	1236	516	161	150	150	154	145	154	154							
007 = 2,3-DI-1,2,3,4	154	161	1332	0	0	0	0	0	0	0	0	0	0	154	1332	154							
008 = "	154	145	162	0	0	0	0	0	0	0	0	0	0	154	162	154							
009 = "	159	145	167	0	0	0	0	0	0	0	0	0	0	159	167	159							
010 = 2,3-DI-1,2,3,4	94	92	90	04	96	100	0	120	577	115	90	94	94	92	94	94							
011 = 2,3-DI-1,2,3,4	94	92	90	0	0	0	0	0	0	0	0	0	0	92	96	94							
012 = "	98	95	94	0	0	0	0	0	0	0	0	0	0	92	94	94							
013 = 2,3-DI-1,2,3,4	07	05	07	06	05	01	0	51	4	70	05	04	05	05	07	05							
014 = 2,3-DI-1,2,3,4	06	05	02	0	0	0	0	0	0	0	0	0	0	02	05	04							
015 = "	07	00	91	0	0	0	0	0	0	0	0	0	0	07	91	09							
016 = 2,3-DI-1,2,3,4	0	0	0	0	0	0	0	0	757	340	0	0	574	0	0	0							
017 = 2,3-DI-1,2,3,4	66	67	66	100	101	100	0	135	80	114	05	05	06	64	67	66							
018 = 2,3-DI-1,2,3,4	65	63	64	0	0	0	0	0	0	0	0	0	0	63	65	64							
019 = "	67	61	63	0	0	0	0	0	0	0	0	0	0	61	67	64							
020 = 2,3-DI-1,2,3,4	57	50	61	63	63	64	0	87	230	01	60	57	57	57	61	59							
021 = 2,3-DI-1,2,3,4	56	50	56	0	0	0	0	0	0	0	0	0	0	55	56	56							
022 = "	57	45	92	0	0	0	0	0	0	0	0	0	0	57	92	71							
023 = 2,3-DI-1,2,3,4	175	175	199	230	164	190	0	151	59	110	143	195	156	199	175	160							
024 = 2,3-DI-1,2,3,4	173	160	145	0	0	0	0	0	0	0	0	0	0	145	173	199							
025 = 2,3-DI-1,2,3,4	09	94	90	0	0	63	0	0	43	77	97	09	91	09	94	91							
026 = 2,3-DI-1,2,3,4	0	0	0	0	0	0	0	0	522	105	0	0	1000	0	0	0							
027 = 2,3-DI-1,2,3,4	08	02	75	0	0	0	0	0	81	73	97	06	75	08	02	02							
028 = 2,3-DI-1,2,3,4	126	121	129	0	0	0	0	0	140	514	130	129	129	121	129	125							
029 = 2,3-DI-1,2,3,4	0	0	0	0	0	0	0	0	162	336	0	0	671	0	0	0							

MONS 024671

Table 2b

Table of Average Relative Response Factors
Generated for Each Congener Class

	Characteristic Relative Response Factors			Average Response Factor	Average Congener Response
	Response from standard1	Response from standard2	Response from standard3		
Cl1 biphenyl #2	0.299	0.265	0.268	0.277333	0.277333
Cl1 biphenyl #5	0.383	0.281	0.32	0.338	0.405444
Cl1 biphenyl #6	0.669	0.549	0.615	0.611	
Cl4biphenyl 6C13a5	100	0	0	100	100
Cl2biphenyl #8	0.205	0.197	0.185	0.195666	
Cl2biphenyl #7	0.409	0.39	0.378	0.392333	0.313508
Cl2biphenyl #8	0.277	0.258	0.264	0.264333	
Cl2biphenyl #9	0.44	0.37	0.39	0.4	
Cl3biphenyl #10	0.211	0.166	0.19	0.189	
Cl3biphenyl #11	0.256	0.181	0.219	0.218666	0.287777
Cl3biphenyl #12	0.25	0.182	0.215	0.215666	
Cl4biphenyl #13	0.223	0.159	0.198	0.193333	
Cl4biphenyl #14	0.184	0.116	0.16	0.153333	0.182111
Cl4biphenyl #15	0.232	0.151	0.216	0.199666	
Cl4biph 12C13a16	100	0	0	100	100
Cl5biphenyl #17	0.0094	0.0642	0.0778	0.074466	
Cl5biphenyl #18	0.0799	0.0447	0.0676	0.064066	0.069877
Cl5biphenyl #19	0.0042	0.0494	0.0797	0.0711	
Cl6biphenyl #20	0.0688	0.0426	0.062	0.057733	
Cl6biphenyl #21	0.0572	0.0345	0.0495	0.047866	0.050888
Cl6biphenyl #22	0.0491	0.0389	0.0636	0.047866	
Cl7biphenyl #23	0.0593	0.0346	0.049	0.047633	0.045983
Cl7biphenyl #24	0.056	0.0342	0.0428	0.044333	
Cl8biphenyl #25	0.028	0.0165	0.0255	0.023333	0.023333
Cl8biph 12C13a26	100	0	0	100	100
Cl9biphenyl #27	0.0385	0.021	0.0326	0.0307	0.0307
Cl10biphenyl #28	0.0271	0.0166	0.0226	0.021433	0.021433
Cl10biph 12C13a29	100	0	0	100	100

Table 3.

Recovery of Native PCB Additions to Montreal Sample Using
Standard Extraction and Analysis Methods PCX15 and SEMK25. (Project: 7034)

LS = The Addition of 2.4 ug to a Sample and MS = The Addition of 24 ug to a Sample

MS CAL10 FILE: >L4413

LS CAL10 FILE: >L4414

COMPOUND NO	#101+MS PCB-2152 (0 (% REC)	#102+MS PCB-2157 (0 (% REC)	#102 MS+MS PCB-2142 (0 (% REC)	#101+LS PCB-2151 (0 (% REC)	#102+LS PCB-2156 (0 (% REC)	#102 MS+LS PCB-2161 (0 (% REC)
000 = CL-biphenyl #1	68	100	89	526	8	147
005 = " #2	71	115	91	206	12	120
006 = " #3	69	109	86	80	3	83
006 = CL2-biphenyl #1	68	113	87	307	51	254
007 = " #2	72	100	86	15	9	15
008 = " #3	88	114	91	105	37	143
009 = " #4	86	115	99	136	63	153
010 = CL3-biphenyl #1	67	100	85	138	58	108
011 = " #2	109	106	92	428	78	178
012 = " #3	88	113	91	193	69	125
013 = CL4-biphenyl #1	66	91	79	114	30	106
014 = " #2	71	98	84	274	101	120
015 = " #3	64	86	80	141	100	124
017 = CL5-biphenyl #1	123	137	117	423	114	129
018 = " #2	103	148	126	384	112	139
019 = " #3	86	146	115	299	102	118
020 = CL6-biphenyl #1	67	114	98	384	102	89
021 = " #2	52	113	106	545	120	115
022 = " #3	116	130	100	503	115	127
023 = CL7-biphenyl #1	56	163	137	172	116	72
024 = " #2	38	144	118	135	109	99
025 = CL8-biphenyl	24	109	87	132	176	73
027 = CL9-biphenyl	30	89	82	89	133	53
028 = CL10-biphenyl	18	51	52	77	100	84
SUBSTITUTE COMPOUND						
000 = 4-CL-biphenyl-6C13	105	115	89	86	8	116
010 = CL4-BP-12C13 Sur.	59	77	69	86	100	45
026 = CL8-BP-12C13 Sur.	39	86	68	52	116	58
029 = CL10-BP-12C13 Sur	4	23	21	31	128	26
MS SEM FILE :	: >L4419	: >L4420	: >L4421	: >L4150	: >L4416	: >L4417

Note - Recoveries are rounded to the nearest whole number

Table 4.

Recoveries of Native PCB Additions to Montreal Samples Using

Standard Extraction and Analysis Methods PCEN15 and REAG25. (Project: 7636)

LS = The Addition of 2.4 ug to a Sample and RS = The Addition of 26 ug to a Sample

MS CALIB FILE: >L4447

LS CALIB FILE: >L4448

COMPOUND ID	TL #100 + RS		BL #1000 + RS		BL #1100 + RS		TL #100 + LS		LS SPK 2	
	PCB-2175 + (% REC)	PCB-2183 + (% REC)	PCB-2185 + (% REC)	PCB-2176 + (% REC)	LS SPK 1 (% REC)	LS BP1K2 (% REC)	LS SPK 2 (% REC)	LS BP1K2 (% REC)	LS BP1K2 (% REC)	LS BP1K2 (% REC)
#02 = CL-biphenyl #1	196	143	106	149	100	99				
#03 = " #2	157	149	104	170	100	88				
#04 = " #3	149	148	104	143	100	89				
#06 = CL2-biphenyl #1	158	131	96	192	100	106				
#07 = " #2	155	144	100	185	100	105				
#08 = " #3	149	143	100	275	100	90				
#09 = " #4	181	150	100	136	100	95				
#10 = CL3-biphenyl #1	119	147	102	306	100	67				
#11 = " #2	105	153	96	379	100	63				
#12 = " #3	88	156	94	199	100	85				
#13 = CL4-biphenyl #1	79	159	103	84	100	88				
#14 = " #2	65	163	102	114	100	75				
#15 = " #3	66	151	100	95	100	70				
#17 = CL5-biphenyl #1	58	160	104	140	100	72				
#18 = " #2	43	150	98	272	100	66				
#19 = " #3	38	146	100	176	100	62				
#20 = CL6-biphenyl #1	40	148	107	159	100	68				
#21 = " #2	14	128	109	153	100	70				
#22 = " #3	37	84	101	159	100	69				
#23 = CL7-biphenyl #1	7	104	94	68	100	71				
#24 = " #2	6	78	100	88	100	80				
#25 = CL8-biphenyl	12	46	111	39	100	65				
#27 = CL9-biphenyl	14	90	100	20	100	64				
#28 = CL10-biphenyl	11	43	104	24	100	65				
SURROGATE COMPOUNDS										
#08 = 4-CL-biphenyl-6C13	0	0	0	0	0	0				
#16 = CL6-BP-12C13 Surr.	0	0	0	0	0	0				
#26 = CL8-BP-12C13 Surr.	0	0	0	0	0	0				
#29 = CL10-BP-12C13 Surr.	0	0	0	0	0	0				

MS RAW FILE : : >L4441 : >L4451 : >L4453 : >L4462 : >L4468 : >L4475

Note - Recoveries are rounded to the nearest whole number

MONS 024674

The following pages describe Standard Sampling, Extraction, and/or Analysis Methods used for this project.

MONS 024675

ESC Standard Analysis Method (SAM)

ESC Standard Analysis Method No:

TITLE: Polychlorinated Compounds in Solvents MSAM25

ANALYTICAL INSTRUMENT AND MODE OF OPERATION: HP-5985/5987 Capillary GC/MS-
Selected Ion Monitoring with
RTE-VI operating system.

Typical Run Time: 15 - 40 min.
Typical Multiplier Voltage: 1800 - 2800 eV
Typical Dwell Times: 100 - 500 msec/mass
Maximum # of Masses: 20 masses x 5 ranges = 100 masses
Electron Energy: 70 eV

TYPICAL ANALYTE	TYPICAL INSTRUMENT LEVEL OF DETECTION (LOD) (ug/ml)	TYPICAL PRECISION @ 10X LOD (%)	TYPICAL ACCURACY @ 10X LOD (%)
--------------------	--	--	---

Organic Compounds

eluting between

toluene and n-C34-ane 0.01 - 0.1

30

30

INTERFERENCES AND LIMITATIONS: Analyte characteristic mass must be chromatographically resolved from other eluting compounds which produce the same mass.

CHROMATOGRAPHIC COLUMN: 30-meter fused silica J&W DB-5 (0.25 µm) with wide bore (0.32 mm)

COLUMN TEMPERATURE PROGRAM: 50(4)/8/300

INJECTION TYPE: Splitless

CALIBRATION AND STANDARDIZATION:

Quantitative: Use internal standard quantitation technique and calibrate instrument response factors from standard solutions of analytes of interest.

Qualitative: When multiple characteristic masses are monitored for a given analyte, the ratio of characteristic mass responses must be within 20% of those for the authentic standard. For PCB analyses, the characteristic masses which are monitored are the molecular ion containing all 35-CL isotopes and the molecular ion containing one 37-CL isotope. The following table summarizes the masses and approximate retention time regions monitored for the analysis of internal standard, surrogate standards, and chlorobiphenyl (CL-BF) through decachlorobiphenyl (CL10-BF) PCB congeners.

Analyte	Masses	Retention Times (minutes)	Relative Retention Times (a)
CL-BF isomers, anthracene-d10	188,190	15 - 22	0.60 - 0.84
4-CL-BF-6Cl3 surrogate	194,196	15 - 22	0.60 - 0.84
CL2-BF isomers	222,224	15 - 22	0.60 - 0.84
CL3-BF isomers	255.9,257.9	15 - 30	0.60 - 0.84
CL4-BF isomers	289.8,291.8	22 - 30.5	0.84 - 1.12
3,4,3',4'-CLA-BF-12Cl3 surr.	301.8,303.8	22 - 30.5	0.84 - 1.12
CL5-BF isomers	323.9,325.9	22 - 30.5	0.84 - 1.12
CL6-BF isomers	357.8,359.8	22 - 30.5	0.84 - 1.12
CL7-BF isomers (b)	323.9,325.9	22 - 30.5	0.84 - 1.12
	391.8,393.8	30.5 - 40	1.12 - 1.87
CL8-BF isomers	427.7,429.7	30.5 - 40	1.12 - 1.87
2,2',3,3',5,5',6,6-CL8-BF-12Cl3	439.7,441.7	30.5 - 40	1.12 - 1.87
CL9-BF isomers	461.6,463.6	30.5 - 40	1.12 - 1.87
CL10-BF	497.6,499.6	30.5 - 40	1.12 - 1.87
CL10-BF-12Cl3 surrogates	509.6,511.6	30.5 - 40	1.12 - 1.87

(a) Relative Retention Times calculated using a retention time of 26.78 minutes for 3,4,3',4'-CLA-BF-d6.

(b) CL7-BF isomers are analysed using either CL2-loss characteristic ions (m/e 323.9 and 325.9) or molecular ions (m/e 391.8 and 393.8).

 CALCULATIONS: Analyte concentration = (Analyte area)/((Analyte RRF)(IS area))

where: Analyte area is the characteristic mass area of the analyte.
 RRF is the relative response factor for the analyte.
 IS is the internal standard and is usually anthracene-d10.

$RRF = (\text{analyte area in standard}) / ((\text{analyte conc.})(IS \text{ area}))$

PCB concentrations are calculated in one of two ways:

1) If an Aroclor formulation can be identified from the patterns of PCB isomers, selected PCB isomers or classes of isomers (i. e. all CLA-biphenyl isomers) are used to quantify the Aroclor concentration. Relative Response Factors are calculated using the concentration of the total Aroclor formulation in the above RRF equation.

2) If no unique Aroclor formulation is present, then the total PCB concentration is calculated by using standard solutions containing known amounts of chlorobiphenyl through decachlorobiphenyl isomers to calculate Relative Response Factors for each chlorine class in the above RRF equation. The RRFs for the corresponding PCB isomer classes are used to calculate total PCB isomer concentrations of each class in unknown samples.
 (For more information regarding standards see Supplement to MSAN25 on the following page.)

 SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

DATE OF METHOD CREATION: 12/9/83

Time Stamp - <871012.1512>

Time Stamp - <871012.1512>

Supplemental Information to Standard Analysis Method MSAM23

Internal Standard

Anthracene-d10

Surrogate Spiking Compounds C-13 labeled

4-chlorobiphenyl 13C6
1,1',4,4'-tetrachlorobiphenyl 13C12
2,2',3,3',5,5',6,6'-octachlorobiphenyl
decachlorobiphenyl 13C12

Native Spiking Compounds (used for native spikes)

2-monochlorobiphenyl
3-monochlorobiphenyl
4-monochlorobiphenyl
2,2'-dichlorobiphenyl
2,3-dichlorobiphenyl
2,4-dichlorobiphenyl
4,4'-dichlorobiphenyl
2,4,6-trichlorobiphenyl
2,3,4-trichlorobiphenyl
2,4,4'-trichlorobiphenyl
2,3',4,6'-tetrachlorobiphenyl
2,3,5,6'-tetrachlorobiphenyl
2,2',4,4'-tetrachlorobiphenyl
2,2',3,4,5-pentachlorobiphenyl
2,2',3,4,5'-pentachlorobiphenyl
2,2',4,5,5'-pentachlorobiphenyl
2,2',3,3',4,4'-hexachlorobiphenyl
2,2',3,4,4',5'-hexachlorobiphenyl
2,2',3,3',6,6'-hexachlorobiphenyl
2,2',3,3',4,4',6-heptachlorobiphenyl
2,2',3,4,4',5',6-heptachlorobiphenyl
2,2',3,3',4,4',5,5'-octachlorobiphenyl
2,2',3,3',4,5,5',6,6'-nonachlorobiphenyl
decachlorobiphenyl

Characteristic and Confirming Masses Monitored

	Characteristic (AMU)	Confirming (AMU)
	-----	-----
anthracene-d10	188	190
4-chlorobiphenyl 6C13	194	196

dichlorobiphenyls	222	224
trichlorobiphenyls	255.9	257.9
tetrachlorobiphenyls	289.8	291.8
pentachlorobiphenyls	323.9	325.9
hexachlorobiphenyls	357.8	359.8
heptachlorobiphenyls	323.9	325.9
octachlorobiphenyls	427.7	429.7
nonachlorobiphenyls	461.6	463.6
decachlorobiphenyl	497.6	499.6

FLOW SHEET MSFS25

FOR ESC ANALYSIS METHOD MSAN25

EXTRACT ID: ESC EXTRACT #

..... LOG NO:

MS LOGBOOK PAGE NUMBER:

EXTRACT COLOR AND DESCRIPTION

CALIBRATE MASS SPECTROMETER []
WITH PFTBA AND DEMONSTRATE
CAPILLARY CHROMATOGRAPHIC
RESOLUTION WITH SYSTEM
PERFORMANCE STANDARD

PLACE IN A 1.6 mL AUTO-
SAMPLER VIAL THE SAMPLE EX-
TRACT OR STANDARD SOLUTION
CONTAINING PCB CONGENERS
WITH 150 uL INTERNAL
STANDARD SOLUTION. []

THE INTERNAL STANDARD SOLUTION
USUALLY CONTAINS 10 UG/mL ANTHRACENE-D10
IN CH2CL2 OR BENZENE.

PLACE VIAL IN AUTOSAMPLER []
TRAY OF HP-5985/87.

CALCULATE CHARACTERISTIC ION AREAS OF
ANALYTES OF INTEREST AND ANTHRACENE-D10
INTERNAL STANDARD.

RECORD MASS SPECTROMETRIC
AND GAS CHROMATOGRAPHIC
OPERATING PARAMETERS IN
INSTRUMENT LOGBOOK. []

[] CALCULATE CONCENTRATION
OF ANALYTES OF INTEREST
USING THE FOLLOWING
EQUATIONS:

(Chlorine Class Ion Area)

PCB Chlorine Class =
Concentration (Chlorine Class RRF) (Internal Standard Area)

(Characteristic Ion Area for Chlorine Class)

Chlorine Class =
RRF (Chlorine Class Concentration)(Internal Standard Area)

Total PCB = CL1-BP Concentration + CL2-BP Concentration +
Concentration CL3-BP Concentration + ... + CL10-BP Concentration

MS FILE NUMBER: DATE/TIME COMPLETED

ANALYST: ESC JOB NO:

MONS 024680

METHOD FILE LIST

Method file: PCSC25 GC type: 5840 Run Type: SIM, GC, EI
Column: Cap Splitless: Yes

Temperature: Source Isothermal Post Run Time
200.0 N 0.0

Run time: 40.00
Scan Start time: 15.00

	ON	OFF	ON	OFF
Relay #1:	327.0	327.0	327.0	327.0
Relay #2:	327.0	327.0	327.0	327.0
Triac #0:	327.0	327.0	327.0	327.0
Triac #1:	327.0	327.0	327.0	327.0

Sim Parameters: Number of groups: 3

Multiplier voltage: 2600

Emission current: 300 uA

Group 1		Group 2		Group 3		Group 4		Group 5	
---------	--	---------	--	---------	--	---------	--	---------	--

Start/Stop		Start/Stop		Start/Stop		Start/Stop		Start/Stop	
15.00	22.50	22.50	30.50	30.50	40.00	0.00	0.00	0.00	0.00

M/Z	Dwell	M/Z	Dwell	M/Z	Dwell	M/Z	Dwell	M/Z	Dwell
-188.0	100	-255.9	100	-391.8	100	0.0	0	0.0	0
-190.0	100	-257.9	100	-393.8	100	0.0	0	0.0	0
-194.0	100	-289.8	100	-427.7	100	0.0	0	0.0	0
-196.0	100	-291.8	100	-429.7	100	0.0	0	0.0	0
-222.0	100	-301.8	100	-439.7	100	0.0	0	0.0	0
-224.0	100	-303.8	100	-441.7	100	0.0	0	0.0	0
-255.9	100	-323.9	100	-441.6	100	0.0	0	0.0	0
-257.9	100	-325.9	100	-443.6	100	0.0	0	0.0	0
0.0	0	-357.8	100	-497.6	100	0.0	0	0.0	0
0.0	0	-359.8	100	-499.6	100	0.0	0	0.0	0
0.0	0	-439.7	100	-509.6	100	0.0	0	0.0	0
0.0	0	-441.7	100	-511.6	100	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0
0.0	0	0.0	0	0.0	0	0.0	0	0.0	0

ESC Standard Extraction Method (SEM)
TITLE: PCBs in base hydrolyzable or
ionizable matrices

ESC Standard Extraction Method No:
PCEX15

.....
MATRIX(X), (CES):

Chemical products which are base
hydrolyzable or ionizable, ie.
chloro acetyl chloride,
chloro phenols, formic acid, etc.

TYPICAL SAMPLE SIZE
TYPICAL EXTRACT VOLUME
(g) (ml)

0.5 - 1g 1 - 10

.....
TYPICAL ANALYTES: A1232, A1242, A1248, A1254, A1260 and A1262 and PCB isomers mono-
through deca-chlorobiphenyl.

.....
INTERFERENCES: Any electron capturing
species in the retention time region
of monochlorobiphenyl isomers through
deca-chlorobiphenyl isomers for ECAW01
and ECAW02. Any species in the
appropriate retention time region
which give the same relative charac-
teristic mass responses as PCB isomers
for NSA003.

TYPICAL SURROGATES
TYPICAL RECOVERIES (%)

pentachlorobenzene 50 - 120
2,4,6-trichloro- 50 - 120
biphenyl
3,3',4,4'-tetra- 50 - 120
chlorobiphenyl-d6

.....
APPARATUS AND MATERIALS:

Burdick and Jackson hexane for extraction solvent.
KOH solutions of various strengths for hydrolysis and ionisation of chemical
species such as ethers, acid chlorides, acids, esters, etc.
Silica gel columns impregnated with potassium permanganate, sulfuric acid,
and potassium hydroxide.
Disposable glassware.

(See flow sheet for this extraction method in PCFS15.)

.....
LIMITATIONS: Method applies only to matrices which contain hydrolyzable or
ionizable electron capturing species in the monochlorobiphenyl
through deca-chlorobiphenyl retention time region for EC dection methods.

.....
SAFETY PRECAUTIONS: See PCB handling protocols in laboratory safety manual.

.....
DATES OF METHOD CREATION AND UPDATES: 7/25/84
.....

FLOW SHEET PCFS15
FOR ESC EXTRACTION METHOD PCFX15

SAMPLE ID: MRC EXTRACT #
..... LOG NO:

EXTRACTION DATE and TIME:

WEIGH OUT SAMPLE INTO A 14
OR 40 mL VIAL

[] WEIGHT SAMPLE (1 g)

SURROGATE SPIKE OF SAMPLE

[] SPIKE

ADD ---- mL ---- KOH AND
SHAKE

[] -- RECORD AMOUNT AND CONC. OF BASE ADDED

NOTE - base should be added
slowly or gradually
to avoid violent
reactions

[] RINSE COLUMN WITH 2 X 20 mL
HEXANE

REMOVE EXTRACT QUANTITATIVELY []
WITH 3 X 4 mL HEXANE RINSES

COLUMN DESCRIPTION
25 mL DISPO PIPET

1 cm Na SO
2 4

REDUCE SAMPLE VOLUME TO
3-4 mL WITH NITROGEN AND
APPLY TO COLUMN QUANTITATIVELY []

4 cm 1% POTASSIUM
PERMANGANATE
SILICA GEL

ELUTE WITH 40 mL HEXANE []

1 cm SILICA GEL

NITROGEN BLOW DOWN TO 1 mL []

4 cm ACID SILICA
GEL

1 cm SILICA GEL

4 cm BASE SILICA
GEL



GLASS WOOL

EXTRACT NO:

DATE/TIME COMPLETED:

ANALYST: ESC JOB NO:

MONS 024683

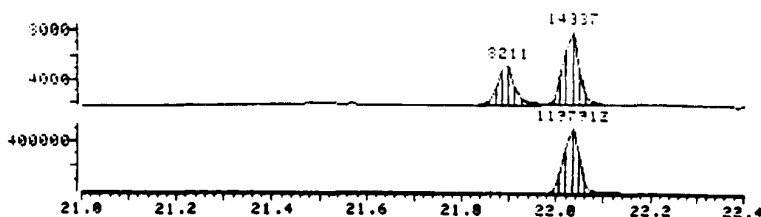
The following pages show calculations or raw data documentation of reported results.

MONS 024684

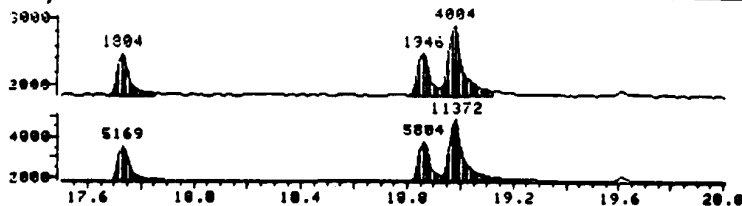
MS data file header from >L4413

Sample 1uL 150 150 D10(10UC Operator: HANSON MSG 7/12/87 0 21
Misc (ML) + HS SPIKE (DIL 1 10) #21 BTL#21
Sys # 2 MS Model: 87 SW/HW rev IA ALS # ***
Method file PCSC25 Tuning file MT8602 No. of extra records 4
Source temp.: 200 Analyzer temp.: 0 Transfer line temp 0

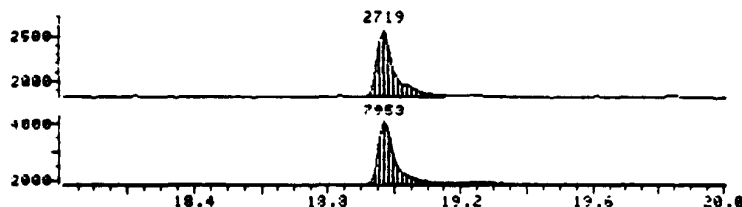
Chromatographic temperatures :	0.	0.	0	0	0
Chromatographic times, min. :	0.0	0.0	0.0	0.0	0.0
Chromatographic rate, deg/min:	0.0	0.0	0.0	0.0	0.0



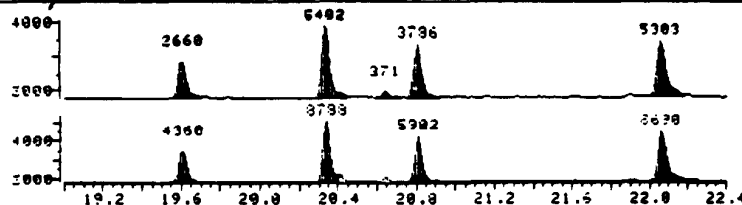
Anthracene-d10 ($m/e = 188$ and 190) #1
GC/MS FILE = >14413



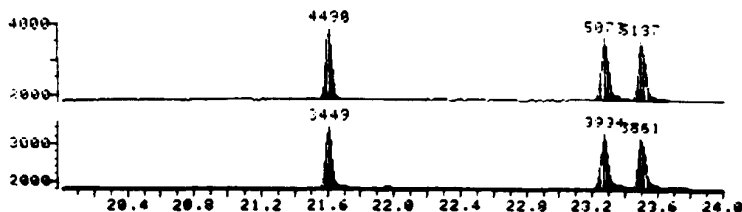
Cl1-biphenyl ($m/e = 188$ and 190) #2-#4
GC/MS FILE = >14413



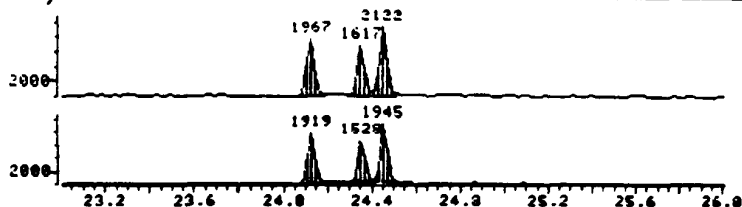
4-Cl-biphenyl-6C13 (194 and 196) #5
GC/MS FILE = >14413



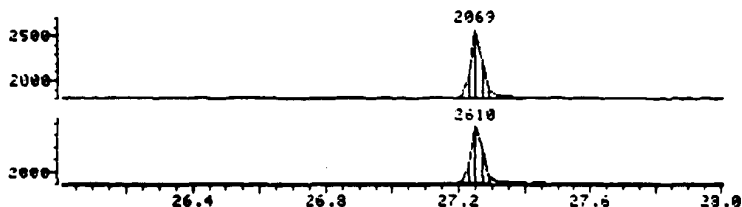
Cl2-biphenyl ($m/e = 222$ and 224) #6-#9
GC/MS FILE = >14413



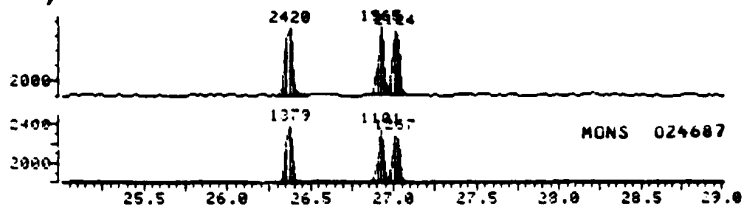
Cl₃-biphenyl (m/e 255.9 & 257.9) #10-#12
GC/MS FILE = >14413



Cl₄-biphenyl (m/e 289.8 & 291.8) #13-#15
GC/MS FILE = >14413



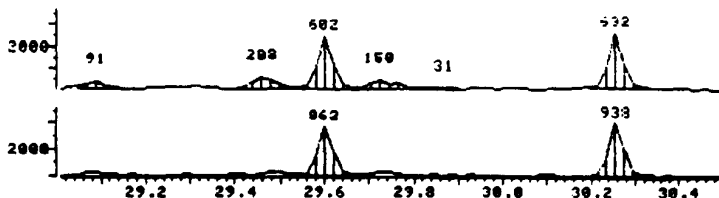
Cl₄-biphenyl-¹²C¹³ (301.8 and 303.8) #16
GC/MS FILE = >14413



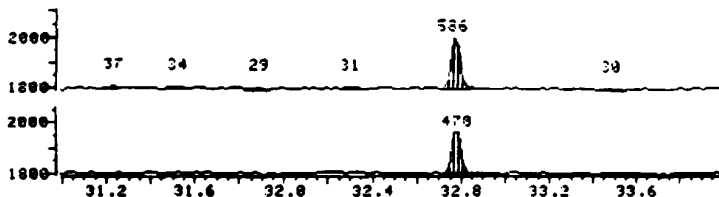
Cl₅-biphenyl (m/e 323.9 & 325.9) #17-#19
GC/MS FILE = >14413



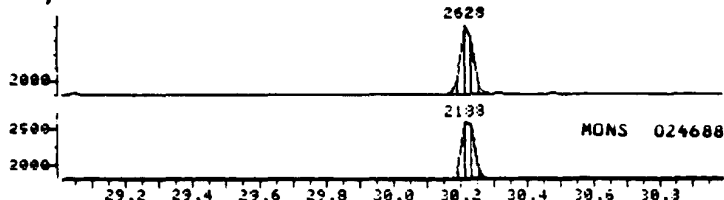
CL6-biphenyl (m/e 357.8 & 359.8) #20-#22
GC/MS FILE = >14413



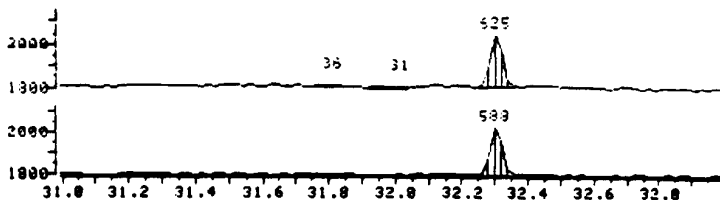
CL7-biphenyl (m/e 323.9 & 325.9) #23-#24
GC/MS FILE = >14413



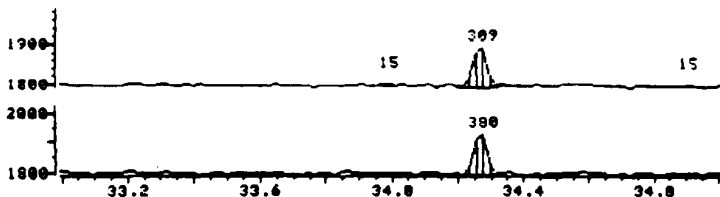
CL8-biphenyl (m/e 427.7 and 429.7) #25
GC/MS FILE = >14413



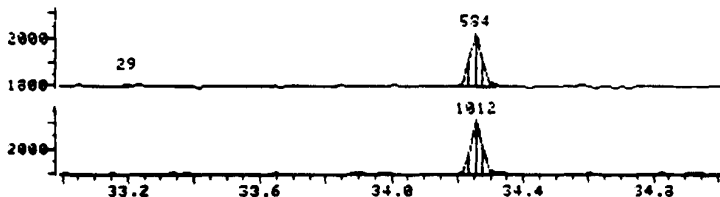
CL8-biphenyl-12C13 (439.7 and 441.7) #26
GC/MS FILE = >14413



CL9-biphenyl (m/e 461.6 and 463.6) #27
GC/MS FILE = >14413



CL10-biphenyl (m/e 497.6 and 499.6) #28
GC/MS FILE = >14413



CL10-biphenyl-12C13 (509.6 and 511.6) #29
GC/MS FILE = >14413

RU, WAIT, 15
DU, PC4413, 1

THE FOLLOWING OUTPUT IS FOR THE SAMPLE SHOWN IN THE FOLLOWING HEADER

MS data File header from DL4413

Sample: 1uL 150 150 D10(10UG Operator: MASON MS2 9/12/87 0 23
Misc : :ML) + MS SPIKE (DIL 1:10) #21 BTL#21
Sys. #: 2 MS Model: 87 SW/HW rev: IA ALS #:***
Method file: PCSC25 Tuning file: MT8602 No. of extra records 4
Source temp.: 200 Analyzer temp.: 0 Transfer line temp 0

Chromatographic temperatures 0 0 0 0 0
Chromatographic times, min. 0 0 0 0 0
Chromatographic rate, deg/min. 0 0 0 0 0

Anthracene-d10 Area = 1197912 and Std. Conc. = 1.0 ug/mL

The following output uses the following definitions for Compound Number:

#01 = anthracene-d10 (n/e 188 area and n/e 190 area)
#02 = CL-biphenyl #1 (n/e 188 area and n/e 190 area)
#03 = " #2 (n/e 188 area and n/e 190 area)
#04 = " #3 (n/e 188 area and n/e 190 area)
#05 = 4-CL-biphenyl-6C13 (n/e 194 area and n/e 196 area)
#06 = CL2-biphenyl #1 (n/e 222 area and n/e 224 area)
#07 = " #2 (n/e 222 area and n/e 224 area)
#08 = " #3 (n/e 222 area and n/e 224 area)
#09 = " #4 (n/e 222 area and n/e 224 area)
#10 = CL3-biphenyl #1 (n/e 255.9 area and n/e 257.9 area)
#11 = " #2 (n/e 255.9 area and n/e 257.9 area)
#12 = " #3 (n/e 255.9 area and n/e 257.9 area)
#13 = CL4-biphenyl #1 (n/e 289.8 area and n/e 291.8 area)
#14 = " #2 (n/e 289.8 area and n/e 291.8 area)
#15 = " #3 (n/e 289.8 area and n/e 291.8 area)
#16 = CL4-BP-12C13 Surr. (n/e 301.8 area and n/e 303.8 area)
#17 = CL5-biphenyl #1 (n/e 323.9 area and n/e 325.9 area)
#18 = " #2 (n/e 323.9 area and n/e 325.9 area)
#19 = " #3 (n/e 323.9 area and n/e 325.9 area)
#20 = CL6-biphenyl #1 (n/e 357.8 area and n/e 359.8 area)
#21 = " #2 (n/e 357.8 area and n/e 359.8 area)
#22 = " #3 (n/e 357.8 area and n/e 359.8 area)
#23 = CL7-biphenyl #1 (n/e 323.9 area and n/e 325.9 area)
#24 = " #2 (n/e 323.9 area and n/e 325.9 area)
#25 = CL8-biphenyl (n/e 427.7 area and n/e 427.7 area)
#26 = CL8-BP-12C13 Surr. (n/e 439.7 area and n/e 441.7 area)
#27 = CL9-biphenyl (n/e 461.6 area and n/e 463.6 area)
#28 = CL10-biphenyl (n/e 497.6 area and n/e 499.6 area)
#29 = CL10-BP-12C13 Surr. (n/e 509.6 area and n/e 511.6 area)

Compound Number	CHRCSTRSTC/d10 (X 10)	CNFRMNG/d10 (X 10)	Compound Delta-RT	RATIO CHRC/CNFRM (X 100)	GLCs
001	10.00	120	.00E+00	8355.39	
002	0432	0151	.00E+00	206.53 1	
003	0185	0162	.00E+00	298.25 1	
004	0949	0334	.00E+00	294.02 1	
005	0664	0227	.00E+00	292.50 1	
006	0364	0222	.00E+00	163.91 2	
007	0734	0458	- .0136	160.31 2	
000	0493	0316	.00E+00	155.89 2	
009	0725	0443	.00E+00	163.87 2	
010	0288	0375	.00E+00	76.82 3	

MONS 024690

011	0328	0423	00E+00	77.55	3
012	0322	0433	00E+00	74.44	3
013	0160	0164	00E+00	97.56	4
014	0128	0135	00E+00	94.50	4
015	0162	0177	00E+00	91.60	4
016	0218	0173	00E+00	126.15	4
017	0115	0202	00E+00	56.98	5
019	00919	0164	00E+00	56.03	5
019	0106	0177	00E+00	59.65	5
020	00720	0114	00E+00	63.15	6
021	00627	00799	- 0204	62.74	6
022	00490	00791	00E+00	61.99	6
023	00720	00503	00E+00	143.17	7
024	00783	00528	00E+00	148.42	7
025	00399	00489	- 0204	81.57	8
026	0183	0219	00E+00	83.26	8
027	00491	00522	00E+00	94.08	9
028	00317	00258	00E+00	122.98	10
029	00845	00488	00E+00	173.29	10

RU,CRT,AL,FF

MONS 024691

The following pages show chain-of-custody information for this project.

MONS 024692

Monsanto Canada Inc.
429, rue St. Patrick
Lafayette, Quebec
H6H 2M3
(514) 388-4880

September 15, 1987

Monsanto Company
800 W. Lindbergh Boulevard
St. Louis, Missouri
U.S.A. 63167

#7636

Attention: Mason Hughes

Re: Waste Plasticizer

Mr. Hughes:

Enclosed are samples 108, 108-B, 110 and 110-B for PCB analysis by GC/mass spec. As per our conversation, I have separated the organic (top layer) from the water phase (bottom layer). Sample #108 and 110 contain the top layer, and sample 108-B and 110-B contain the water layer as well as solids.

The organic phase contains long chain (C_7 - C_{11}) esters of phthalic acid (PAE). It may also contain traces of adipic acid esters. The aqueous phase is mainly water containing grey-white settleable solids. The solid phase is filter aid (diatomaceous earth) containing approximately 30% of absorbed plasticizer esters.

The charge number for this work is 04-000-1910905.

If you have any questions, please do not hesitate to contact me.

Sincerely,


Christian Roy

/la

Encl.

MONS 024693

Monsanto

CANADA INC.

DISTRICT SALES OFFICES / BUREAUX DE VENTE REGIONAUX
 2330 ARGENTIA RD. P.O. BOX 787 STREETSVILLE,
 MISSISSAUGA, ONT. L5M 2G4
 (416) 828-8222 TELE: 06-219851
 425 RUE ST-PATRICK, LASALLE, QUEBEC H5N 2H3
 (514) 388-4880 TELE: 055-88344
 484 RIVERSIDE RD. R.R. 2, ABBOTSFORD, B.C. V2S 4N2
 (604) 853-4873 TELE: 04-383832

SALES OFFICE
 BUREAU DE VENTE

REQUESTED BY / LA DEMANDE DE **Christian Roy** DATE **September 15, 198**

SHIP TO:
 EXPÉDIER À

MONSANTO COMPANY

800 N. Lindbergh Boulevard - Bldg S Receiving

St. Louis, Missouri, U.S.A. 63167

SHIPPING DATE
 DATE D'EXPÉDITION
10/7/87

FILLED BY / REMPLI PAR
10

ATTENTION
 A L'ATTENTION DE

Mason Hughes (U4E)

TELEPHONE

CUT OFF STOCK DELIVERY EXPECTED
 STOCK ÉPUISÉ LIVRAISON PRÉVUE

SHIP VIA: EXPÉDIER PAR ☐ PREP-UP ☐ SPECIAL ☐ FREIGHT ☐ EXPRESS ☐ AIR MAIL ☐ TOLERO ☐ TOLERO ☐ AIR FREIGHT ☐

ITEM ARTICLE	QUANTITY Quantité	DESCRIPTION	LOT NO. & LOT
1	4	Waste Plasticizer 011 (samples for experimental purposes)	
2	870915	BOT Layer	NO-B Greyish
	870915	TOP "	110 yellowish
	870915	BOT	108-B Greyish
	870915	TOP	108 yellowish

**Sample Order
 Commande
 d'échantillons**

**NO CHARGE
 À TITRE GRATUIT**

Monsanto

CUSTOMER'S COPY / COPIE DU CLIENT

MONS 024694

Monsanto

INDEX 101-102

Monsanto Canada Inc.
428, rue St. Patrick
Lafayette, Quebec
H3N 2M3
(514) 388-4880

#7634

September 3, 1987

Monsanto Company
800 N. Lindbergh Boulevard
St. Louis, Missouri
U.S.A. 63167

Attention: Mason Hughes

Re: Waste Plasticizer

Enclosed are samples #101 and #102 for PCB analysis by GC/mass spec. As per our conversation, I have separated the organic (top layer) from the water phase (bottom layer). Sample # 101 contains the top layer, and sample # 102 contains the water layer as well as solids.

The upper organic phase (#101) contains long chain (C_7-C_{11}) esters of phthalic acid (PAE). It may also contain traces of adipic acid esters. The lower aqueous phase (#102) is mainly water containing gray-white settleable solids. The solid phase is filter aid (diatomaceous earth) containing approximately 30% of absorbed plasticizer esters.

The charge number for this work is [REDACTED]

If you have any questions, please do not hesitate to contact me.

Sincerely,

[Handwritten signature]

Myra Plotnick
Industrial Hygiene Chemist

[Handwritten notes: 10/18/87, gm]

MOHS 024695

Monsanto
CANADA INC.

DISTRICT SALES OFFICES / BUREAUX DE VENTE REGIONAUX
2330 ARGENTIA RD., P.O. BOX 787 STREETSVILLE
MISSISSAUGA, ONT. L5M 2G4
(416) 828-9222 TELE: 06-218851
425 RUE ST-PATRICK, LASALLE, QUEBEC H8N 2H3
(514) 388-4880 TELE: 050-88344
484 RIVERSIDE RD., R.R. 2, ABBOTSFORD, B.C. V2S 4H2
(604) 853-4573 TELE: 04-383532

SALES OFFICE
BUREAU DE VENTE

REQUESTED BY / À LA DEMANDE DE
Myra Plotnick

DATE
September 3, 1987

SHIP TO:
EXPÉDIER À

MONSANTO COMPANY

800 N. Lindbergh Boulevard - Bldg S Receiving

St. Louis, Missouri, U.S.A. 63167

Shipping DATE
DATED EXPÉDITION
3/9/87

FILLED BY / REMPLI PAR
AIB

ATTENTION
À L'ATTENTION DE

Mason Hughes (U4E)

TELEPHONE 514-388-4880

OUT OF STOCK DELIVERY EXPECTED
STOCK ÉPUISÉ LIVRAISON PRÉVUE

SHIP VIA EXPÉDIER PAR ☐ BULK / en vrac ☐ PARCEL POST / colis postal ☐ FREIGHT / fret ☐ EXPRESS / express ☐ AIR MAIL / aérien ☐ SIGNED / signé ☐ PER FREIGHT / par fret ☐

ITEM ARTICLE	QUANTITY QUANTITÉ	DESCRIPTION	LOT NO. N° DE LOT
1	2	Waste Plasticizer Oil (samples for experimental purposes)	
2			

**Sample Order
Commande
d'échantillons**

**NO CHARGE
À TITRE GRATUIT**

Monsanto

CUSTOMER'S COPY / COPIE DU CLIENT

MONS 024696

The following pages describe Analytical Request Information generated for this project.

MONS 024697

Time Stamp - <871012.1621>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: September 8, 1987

LOG NO: U-87-09-08-02

REQUESTER: Myra Plotnick/Canada

PROJECT NO: 760.34-7634

LOGIN NOTEBOOK PAGE: 3759209

REPORT RESULTS TO: FILES/MEERS, HUGHES, SHERMAN, MCKENZIE, MINOR

ANALYSIS REQUESTS SENT TO: FILES MEERS, SHERMAN, HUGHES, MCKENZIE, MINOR

SAMPLE PICKUP BY: MINOR

SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 2 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
101 UPPER ORGANIC PHASE	1 X 30 mL	PCB-2149, PCB-2150-DUP PCB-2151+LS, PCB-2152+HS
102 WATER CONTAINING GREY-WHITE SETTLABLE SOLID	1 X 30 mL	WATER ONLY PCB-2154, PCB-2155-DUP PCB-2156+LS, PCB-2157+HS SEDIMENT PCB-2159, PCB-2160-DUP PCB-2161+LS, PCB-2162+HS
METHOD BLANK(S)		PCB-2165, PCB-2164

RECORDED BY: MINOR

REQUIRED ANALYSES: PCB (incidental)

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158639

ESC STANDARD EXTRACTION METHOD(S): PCCK15

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
-X- CCGC/MS ()	10-12-87	MSAN25	157533-157534

FINAL REPORT NOTEBOOK PAGE: 3440445

OTHER NOTEBOOK PAGES:

QUALITATIVE: X

SEMIQUANTITATIVE:

QUANTITATIVE: X

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

MONS 024698

.....
COMMENTS:
.....

SAMPLE DISPOSITION: HOLD FOR 90 DAYS
.....

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL
.....

ANALYSIS REQUEST FILE NAME:AA7634
.....

MONS 024699

Time Stamp - <871012.1619>

MONSANTO COMPANY/ENVIRONMENTAL SCIENCES CENTER

ANALYSIS REQUEST

DATE: September 18, 1987 LOG NO: U-87-09-18-02
REQUESTER: ROY/MONTREAL PROJECT NO: 760.28-7636
LOGIN NOTEBOOK PAGE (To be filled out at login.): 3759212
REPORT RESULTS TO: FILES/MEES, SHERMAN, HUGHES, MCKENZIE, MINOR
ANALYSIS REQUESTS SENT TO: FILES MEES, SHERMAN, HUGHES, MCKENZIE
SAMPLE PICKUP BY: MINOR
SAMPLE LOCATION: U-420

SAMPLE DESCRIPTION FOR 4 SAMPLES

SAMPLE IDENTIFICATION	NUMBER OF BOTTLES	ESC NUMBER(S)
870915 BOTTOM LAYER 110-B	1 X 40 mL	WATER ONLY PCB-2184, PCB-2185+HS SEDIMENT PCB-2186, PCB-2187+HS
870915 TOP 110	1 X 40 mL	PCB-2178, PCB-2179+HS
870915 BOTTOM LAYER 108-B	1 X 40 mL	WATER ONLY PCB-2180, PCB-2181+HS SEDIMENT PCB-2182, PCB-2183+HS
870915 TOP LAYER 108	1 X 40 mL	PCB-2173, PCB-2174-DUP PCB-2175+LS, PCB-2176+HS
METHOD BLANK		PCB-2188

RECORDED BY: MINOR

REQUIRED ANALYSES: PCBs

EXTRACTION LOGBOOK NOTEBOOK PAGE: 158640

ESC STANDARD EXTRACTION METHOD(S): PCEX15

ANALYTICAL TECHNIQUES:

TECHNIQUE (EN)	DATE COMPLETED	ESC STANDARD ANALYSIS METHOD	INSTRUMENT LOGBOOK PAGE
X CGGC/MS ()	10-12-87	MSAN25	157533-157534

FINAL REPORT NOTEBOOK PAGE: 3440446

OTHER NOTEBOOK PAGES:

MONS 024700

QUALITATIVE:

SEMIQUANTITATIVE:

QUANTITATIVE:

REQUIRED QA/QC: SEE GOOD LABORATORY PRACTICES MANUAL

COMPLETION DATE REQUESTED:

COMMENTS:

SAMPLE DISPOSITION: HOLD FOR 90 DAYS

SAFETY PRECAUTIONS: SEE SAFE LABORATORY PRACTICES MANUAL

ANALYSIS REQUEST FILE NAME: AA7636.LI

MONS 024701

The following sections describe important terminology used in Quality Assurance/Quality Control, Analyte Extraction and Instrumental Analysis. ESC and ESC/UAC Good Laboratory Practices Manuals can be obtained by contacting Janet Lee (U4D) NRTS 344-1273, GLP and QA/QC Supervisor.

MONS 024702

Quality Assurance/Quality Control Terminology

This section describes commonly used terminology and phrases which are used in conjunction with Quality Assurance/Quality Control (QA/QC). This information may be helpful in understanding some details of QA/QC procedures which have been incorporated in the present report. The following two sections also give useful information concerning terminology used in analyte extraction and instrumental analysis of samples and sample extracts.

Quality Assurance/Quality Control - Terminology used to describe activities which are designed to produce analytical results of known quality. In an effort to minimize costs of environmental analyses, specific QA/QC elements have been incorporated into ESC/UAG extraction and analysis steps which should not unduly burden analytical studies with large development and method validation costs. This approach has been taken so that sets of samples can be analyzed for various analytes without extensively validating methods over wide concentration ranges and without using methods which are designed for only one matrix.

One price which one pays for this approach is that analytical precision and accuracy are sacrificed, somewhat. However, in many cases, when clearly defined regulatory limits have been set, confirming that the analyte is present at levels well below or well above the limit does not need the type of accuracy that is required when the regulated analyte concentration is in the vicinity of the regulatory limit. In these latter cases, more extensive QA/QC procedures are required than are included in the present study.

Precision - Terminology used to evaluate the reproducibility of a certain determination. Parameters which affect analysis precision range from sample acquisition, to sample storage, to sample extraction, and finally to sample extract analysis. Normally, the major factor which affects the precision of the analysis is the sample extraction step. This step normally results in sample analysis precision on the order of $\pm$ or $\sim$ 30%.

The precision of a result can be determined in a number of ways. Replicate analyses of the same sample, followed by the calculation of a percent relative standard deviation for that determination is the most popular method of assessing precision. However, this calculation should not be made when there are less than three replicate points from which to calculate the percent relative standard deviation. In addition, replicate extractions and instrumental analyses must be performed. Therefore obtaining percent relative standard deviation data can be very expensive and is not normally obtained unless large studies are being conducted. A second method of assessing precision is with the use of duplicate analyses. The present report includes results of at least one sample which was analyzed in duplicate. NOTE!! It is the responsibility of the analysis requester to review these data and draw his own conclusions concerning the precision of the determination and, more importantly, if the precision of the determination meets his needs.

(continued)

Accuracy - Terminology used to determine the "correctness" of the data. The accuracy of most steps in the analysis of samples is typically on the order of 10%. However, when analyte concentrations in the sample extracts approach the level of detection of a determination, accuracy as well as precision can approach 100% or greater. The accuracy of a determination of an analyte which is present at concentrations which are much greater than the instrumental level of detection is most strongly affected by the homogeneity of the sample matrix and the precision of the extraction step, which results in typical accuracies in environmental analyses to be on the order of 30%. Normally, the accuracy of standard solutions which are used to calibrate analytical instrumentation is well below this amount, and therefore is normally not an important factor in producing inaccurate data. From time to time, analyte responses of standard solutions from different sources will be compared to standard solutions used for instrument calibration. This information is normally included in the section entitled "QA/QC Results".

The accuracy of a result can be estimated in a number of ways. The current study uses surrogate standard recovery and native analyte recovery information to aid in assessing the accuracy of a result. This recovery information, normally reported as percent recovery, gives you information concerning the percent of surrogate standards and native analytes which were recovered after adding to your sample matrix and performing the various extractions, clean-ups, and instrumental analyses associated with the determination. The use of surrogates also assumes that the spiked surrogates are bound in the matrix in the same way as the analytes of interest. Surrogate standards are added to every sample while native analytes are added to only 10% of each set of samples. Therefore surrogate standard recovery data could be used to correct the reported results of an analyte concentration, if the user of the data feel such corrections are warranted. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Bias - Terminology used to describe how the accuracy of a determination may be affected. A low bias means that values below the correct value are normally measured. A high bias means that values above the correct value are normally measured. The instrumental analysis step of extract analysis normally does not contribute significantly to analysis bias. Most bias in environmental analysis is introduced in the sample extraction step. A low bias is introduced when the sample is not completely extracted and the extract does not contain 100% of the analyte. A high bias is introduced when the final volume of the extract is less than the correct volume. Surrogate Standards (SS), which are discussed in the next section, can be used to determine the amount of bias which is incorporated in the reported result. NOTE!! No data reported in this report includes any correction for this bias. It is the responsibility of the person requesting the analyses to apply appropriate bias corrections, if he/she feels that such corrections are warranted.
(continued)

Page 3. Quality Assurance/Quality Control Terminology

Level of Detection (LOD) - Terminology used to indicate what analyte concentration will produce a signal-to-noise ratio of 10. For the present report, this value is not experimentally determined. Normally, a Limit of Quantitation is used to determine the threshold concentration above which an analyte can be detected.

Limit of Quantitation (LOQ) - Terminology used to indicate the lowest analyte concentration which can reliably be quantified. The precision of the determination of the analyte concentration at the LOQ is normally on the order of 100%. The LOQ is normally determined experimentally by adding a known amount of analyte to the sample matrix and measuring the percent recovery at the LOQ. In some cases, when there is insufficient sample quantity, or for convenience, an LOQ will be determined using other methods. These methods should be clearly described in this report.

Further Information

The general area of Quality Assurance/Quality Control, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding the validity of the results reported in this study.

ASTM Standard Practice D4210-83 entitled "Intralaboratory Quality Control Procedures and a Discussion on Reporting Low-Level Data".

ASTM Standard Practice D2777-77 entitled "Determination of Precision and Bias of Methods of Committee D-19 on Water".

An article in Analytical Chemistry, authored by 29 well-known environmental scientists. This article was funded by a grant from the Environmental Protection Agency and acknowledges 24 additional scientists in industry and government who were involved in the review of the article. Its citation is: "Principles of Environmental Analysis", L. H. Keith, et. al., Anal. Chem. 1983, 55, 2210 - 2218.

40 CFR Part 136 entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

QAQCTE:MH

Analyte Extraction Terminology

This section describes commonly used terminology and phrases which are used in conjunction with analyte extraction. This information may be helpful in understanding some details of how analytes in your samples were isolated for instrumental analysis. Instrumental Analysis Terminologies are given in the following section of this report.

Sample - Terminology used to reference the material which you submit for analysis. Samples may be solid, liquid, and in some cases, gases. In general, when a sample is received for analysis, it is logged into the ESC sample login book and proper chain-of-custody documents are maintained in the event this information may be required for litigation purposes.

Analyte - Terminology used to reference specific compounds or formulations of compounds. When an analysis is requested for a certain analyte within a certain sample matrix, normally some type of extraction process is required.

Extraction - This terminology refers to the step which isolates the analyte of interest into an appropriate solvent. Extraction is required for several reasons. In many cases, the concentration of the analyte is below the level of detection of the instrument which will be used for analysis. Therefore, extraction involves removing the analyte of interest from a relatively large volume of sample and placing it into a relatively small volume of solvent. This places the analyte in the solvent at a concentration which can easily be detected by the analytical instrument.

A second reason that extraction is required, is that the sample may not be appropriate for direct instrumental analysis of the analyte of interest. This can be easily understood for analytes in soils. Very few analytical instruments allow soils to be directly inserted into the instrument. Therefore some type of isolation step (i. e. extraction) is required in order to place the analyte in an appropriate matrix for instrumental analysis.

When the analyte of interest is present at sufficient concentration, and the sample matrix can be directly inserted into the analytical instrument, the sample, or the diluted sample, can be directly analyzed. However, this case represents a minority of sample analyses which ESC is asked to perform.

(continued)

Page 2, Analyte Extraction Terminology

Matrix - Terminology used to reference the major components of a sample. In most cases, major components are not of direct interest except as they affect the types of extraction steps and other isolation procedures which must be used to isolate the analyte from the sample matrix. For the majority of environmental samples which ESC is asked to analyze, the matrix is normally water or soil. However, many environmental matrices contain relatively high levels of organic compounds which may interfere with the instrumental analysis of the analyte of interest. Therefore, depending upon the analyte of interest, the required level of detection, and the amounts and types of other organic materials which may interfere with the instrumental analysis, extraction methods must be developed which are very specific for a given set of samples. In order to deal with almost a limitless set of matrices, analytes, extraction methods and instrumental methods, an organized approach to sample extraction, followed by instrumental analysis, has been developed at ESC.

Extract - Terminology used to identify the analyte-containing solvent which was obtained from the extraction of a certain matrix.

Extract Clean-up - Terminology used to identify chemical steps which are required in order to reduce the concentration of organic compounds which may be extracted with the analyte of interest. These co-extracted compounds may be at such high levels and/or produce responses so that they interfere with the instrumental analysis of the analyte in the extract.

Extractable Compounds - Terminology which refers to the ability of a compound to be isolated from the sample matrix and placed into the extract solvent. When the sample matrix is primarily soil which contains little organic matter, most organic compounds are extractable compounds. However, when the sample matrix is primarily water, only those compounds which are hydrophobic are extractable compounds.

Standard Extraction Method - Terminology used to reference an organized description of the steps involved in isolating an analyte from a given matrix. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific Standard Extraction Methods which are used in this study are listed in the final report in the section entitled "Details". In addition, copies of these Standard Extraction Methods are shown in a separate section of this report. Standard Extraction Methods also include details of sample clean-up, if required.

Extraction Flow Sheet - Terminology used to reference a graphic description of a Standard Extraction Method. This flow sheet is filled out by the analyst in the laboratory and serves as a detailed record of what occurred in the sample extraction step. Flow sheets of individual sample extractions may be included in a section of this report.

(continued)

Page 3, Analyte Extraction Terminology

Surrogate Standards (\$\$) - One important element in the extraction of an analyte from a matrix, is the determination of what percent of the analyte was actually extracted from the matrix. For methods which involve liquid/liquid extraction of water with an organic solvent, or liquid/solid extraction of soils with an organic solvent, it is imperative that data be obtained which gives the analysis requester information concerning what percentage of the analyte was isolated in the extract during the extraction step. Surrogate Standards serve this important purpose.

The philosophy on the use of Surrogate Standards is as diverse as the analysts who use them. If cost were no object, analyte recovery information could be obtained by the use of standards addition analysis methods. However, since many samples require extensive labor to isolate an analyte into an extract, standards addition analysis methods are too costly for most studies. (A standards addition analysis requires each sample to be extracted and analyzed at least three times.) Surrogate Standards give the same type of information, with only one analysis being required.

A "Surrogate" Standard is a compound which is chosen to behave exactly as the analyte. It is a surrogate for that analyte and thus information obtained for this surrogate can be assumed to also apply to the analyte of interest. For the analysis of regulated analytes, it is important that when data is generated that shows that the analyte is not present, that we can also show that if the regulated analyte were present in the sample matrix, it would have been detected. This is especially true when complex extraction and instrumental analysis steps are involved in this determination. Therefore, when an extraction is performed, a known amount of a Surrogate Standard is added to the sample before any extraction steps take place. When the extract is then analyzed for the analyte of interest, the amount of the Surrogate Standard is also determined. From this information one can document, for each sample, what recoveries were measured for this Surrogate Compound. From this, one can describe some level of confidence on the data generated for the analyte of interest.

A detailed discussion on how Surrogate Standards are chosen is beyond the scope of this section on Analyte Extraction Terminology. EPA normally uses two types of Surrogate Standards. The first is when the surrogate is exactly the same as the analyte of interest, except that the naturally occurring carbon, hydrogen, or halogen atoms in the analyte have been replaced with non-naturally occurring isotopes. These surrogates can be expected to act exactly as the native analytes. One good example of this type of surrogate is the 2,3,7,8-TCDD surrogate in which all carbon atoms are carbon-13 or all chlorine atoms are chlorine-37.

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Page 4. Analyte Extraction Terminology

The second type of Surrogate Standard is one which does not have the same chemical structure as the analyte of interest, but is known, or assumed to act chemically identical to the analyte of interest during the extraction and clean-up steps. Examples of this type of surrogates are deuterated compounds which may be added to a matrix in which any chromatographable materials are to be identified. This approach can only be applied to GC/MS analyses, since the surrogates can be differentiated from the native compounds from their different mass spectra. Since it is not known before sample extraction and extract analysis what analytes may be present, surrogates must be chosen that could not possibly be present in the sample. In addition, since only liquid/liquid or liquid/solid extraction would be involved, with no clean-up (since clean-up would remove compounds in which the requester is interested) any hydrophobic deuterated compound could serve as a surrogate. Normally, phenol-d5, 2-Cl-phenol-d4, CL1-benzene-d4, pyrene-d10 and chrysene-d12 are used as Surrogate Standards for the analysis of a wide variety of hydrophobic materials in environmental samples.

A sub-set of this second type of Surrogate Standards are the surrogates which are used in the capillary GC/EC analysis of Aroclor formulations. Since mass spectrometric detection is not used, surrogates which contain unique isotopes cannot be distinguished from their native counterparts. Therefore, other compounds are used as Surrogate Standards since they have been observed to act exactly as the polychlorinated biphenyls (PCBs) in the Aroclor formulations. Any halogenated compound can be used which acts exactly as PCBs in the extraction and clean-up step. ESC normally uses CL3-benzene and 2,4,6-CL3-biphenyl for this purpose.

Native Analytes - Terminology which references the analytes of interest which contain naturally-occurring isotopes of carbon, hydrogen, halogens, etc.

Native Spikes - Terminology which references the addition of a known amount of native analytes to the sample matrix. Normally, at least one sample will be spiked with the native analyte at the reported level of detection (low native spike) and at 10 times the level of detection (high native spike). Recoveries of the native analytes provide further documentation on the quality of the analyses performed. For sample sets which contain more than 10 samples, the frequency of low and high native spikes is 10% of the number of samples.

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Page 5. Analyte Extraction Terminology

Method Blank - Terminology which refers to a sample which is known to contain none of the analytes of interest. For water analyses, this is normally Burdick and Jackson Brand Water, de-ionized, or distilled water. For soil, it may be soil which has been previously analyzed and known to contain none of the analytes of interest. All extraction steps, using randomly selected glassware, solvents, and all surrogate spiking solutions must be used. This provides documentation that the analytes of interest were not inadvertently present in extraction glassware and solvents used in the analysis. A minimum of one method blank must be generated with each set of samples. For sample sets containing more than 10 samples, method blanks are generated at a frequency of 10% of the number of samples.

Duplicate Analyses - Terminology used to indicate the duplicate extraction and duplicate extract analysis of a sample. This provides documentation on the precision of an analysis. Normally the precision of environmental analyses of homogeneous samples is on the order of 30%. A minimum of one duplicate analysis is required for each sample set. For sample sets containing more than 10 samples, duplicate analyses are generated at a frequency of 10% of the number of samples.

Further Information

The general area of Analyte Extraction, along with much of the terminology described above, is very complex. The following references may provide further information which may be useful in understanding extraction fundamentals and terminology.

40 CFR Part 136 entitled "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule". This document includes details of EPA's 600-series analysis methods. A current copy of these methods can be found in the October 26, 1984 Federal Register.

Statement of Work for the Contract Laboratory Program which is used in conjunction with EPA's Superfund Site studies. A current copy may be obtained by contacting:

Joan F. Fisk, Project Officer
Analytical Support Branch
Hazardous Response Support Division
USEPA
Washington, D. C. 20460

ANEXTE:MM

Instrumental Analysis Terminology

This section describes commonly used terminology and phrases which are used in conjunction with extract analysis. This information may be helpful in understanding some details of how instrumental analyses of sample extracts were performed. The previous section of this report describes terminology associated with analyte extraction.

Sample Extract - Terminology used to describe the solvent containing the analyte of interest which is obtained from some type of sample extraction step. Detailed information on how this extract is prepared can be found in the Standard Extraction Methods and flow sheets associated with the methods. Summaries of these methods which were used for this project can be found in a separate section of this report.

Internal Standard (IS) - Terminology used to reference a compound which is added to the sample extract and standard solutions of the analytes of interest immediately before instrumental analysis. The purpose of the internal standard is to provide data which can be used to correct for variations in injected volumes of extract and standard solutions which may occur during instrumental analysis. The internal standard must not be present in the sample extract. Typical internal standards used in mass spectrometric analyses are benzene-d6, naphthalene-d8, anthracene-d10 and chrysene-d12. These deuterated compounds produce unique mass spectra and are not present in naturally-occurring samples. A commonly used internal standard for GC/EC analysis is hexachlorobenzene.

Internal Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of an internal standard be added to the sample extract prior to instrumental analysis. Responses of all analytes are calculated relative to the internal standard using the following relationship:

$$RR - \text{Relative Response} = \frac{(\text{Analyte Area})}{(\text{Internal Standard Area})}$$

If different volumes of the sample extracts and standard solutions are injected, these differences are reflected by differences in the internal standard area. In addition, if instrument sensitivity changes, these changes are also reflected in changes in the internal standard area. The calculation of the relative response corrects for these types of fluctuations.

In order to determine the analyte concentration-Relative Response relationship, analyte Relative Response Factors are calculated using the following relationship for data obtained from the analysis of standard solutions:

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Page 2. Instrumental Analysis Terminology

$$\text{RRF} - \text{Relative Response Factor} = \frac{\text{(Analyte Area)} \text{ (Internal Standard Concentration)}}{\text{(Internal Standard Area)} \text{ (Analyte Concentration)}}$$

When identical amounts of the internal standard are added to both extracts and standard solutions, the internal standard concentration term in the above equation can be eliminated. When this is done and Relative Response is substituted for the ratio of analyte area to internal standard area, the RRF equation becomes:

$$\text{RRF} = \text{RR}/(\text{Analyte Concentration})$$

Analyte concentrations in the extracts are calculated from the following equation:

$$\text{Analyte Extract Concentration} = \text{RR}/\text{RRF}$$

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{\text{(Analyte Extract Concentration)} \text{ (Extract Volume)}}{\text{(Amount of Sample Extracted)}}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the internal standard quantitation method is applied to specific methods are described in the various Instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\text{Analyte Sample Concentration (Corrected for Analytical Bias)} = \frac{\text{(Analyte Sample Concentration)}}{\text{(Assumed Analyte Recovery)}}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. NOTE!! No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

External Standard Quantitation Method - Terminology which refers to a method of data analysis. This method requires that a known amount of the analyte standard be analyzed under the same conditions as the sample extract. In order to determine the analyte concentration-analyte response relationship, Response Factors are obtained from the following relationship:

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Page 3. Instrumental Analysis Terminology

RF - Response Factor - (Analyte Area)/(Analyte Concentration)

Analyte concentrations in the extracts are calculated from the following equation:

Analyte Extract Concentration = (Analyte Area)/RF

In order to obtain the concentration of the analyte in the sample, the following equation is used:

$$\text{Analyte Sample Concentration} = \frac{\begin{array}{c} \text{(Analyte Extract} \\ \text{Concentration)} \end{array} \begin{array}{c} \text{(Extract Volume)} \\ \text{-----} \\ \text{(Amount of Sample Extracted)} \end{array}}$$

The area terms in the above equations may be total ion areas, FID peak areas, EC peak areas, or extracted ion areas. The details of how the external standard quantitation method is applied to specific methods are described in the various instrumental Standard Analysis Methods.

The following equation shows what calculations should be made to correct for analytical bias:

$$\begin{array}{c} \text{Analyte Sample Concentration} \\ \text{(Corrected for Analytical Bias)} \end{array} = \frac{\begin{array}{c} \text{(Analyte Sample Concentration)} \\ \text{-----} \\ \text{(Assumed Analyte Recovery)} \end{array}}$$

The Assumed Analyte Recovery can be obtained from an average of surrogate recoveries or native analyte recoveries, measured for the sample in question. **NOTE!!** No data reported in this study include any correction for surrogate standard recovery. It is the responsibility of the person requesting the analyses to apply appropriate recovery corrections, if he/she feels that such corrections are warranted.

Standard Analysis Methods - Terminology used to reference an organized description of the steps involved in the instrumental analysis of a sample or a sample extract. These methods are assigned short names which can also be used as computer file names for these methods. The names of specific instrumental Standard Analysis Methods which are used in this study are listed in the final report in the section entitled "Details". In addition, copies of these Standard Analysis Methods are shown in a separate section of this report. Flow sheets of the Standard Analysis Methods are usually also provided in this section.

Analysis Flow Sheet - Terminology used to reference a graphic description of a Standard Analysis Method. This flow sheet gives specific information on how extracts and standards are analyzed and what types of data reduction methods are used.

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Page 4. Instrumental Analysis Terminology

Further Information

The general area of Extract Analysis, along with much of the terminology described above, is very complex. References provided in the previous section entitled "Analyte Extraction Terminology" may provide further information which may be useful in understanding instrumental analysis fundamentals and terminology.

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