100242

	Versar Sample #	PP Sample #	Sample location	Received
Dec 19	TB-17	N/A	Trip Blank	
	SP-1	SP-1-1-1	Soil pile no 1	✓
	SD-2	SR-3-1-1	Soil drainage # 3; 0-6"	✓
	SD-3	SR-4-1-1	" = 4; 6-6"	✓
	SD-4	SR-5-1-1	" # 5; 12-18"	✓
	TB-16	—	Trip Blank	
	SWT-9	SW-3-0-1	Surface water, location 3	✓
	SWD-18	SW-3-0-1	Duplicate of SWT-9	✓
	SWR-8	SW-17-0-1	Surface water, location 17	✓
	BB-10	SW-4-0-3	Field blank, Surface water	✓
	SR-11	SD-8-0-1	Sediment, location 8	✓
	SR-12	SD-13-0-1	" " 13	✓
	SR-13	SD-3-0-1	" " 3	✓
	SR-14	SD-8-0-1	Duplicate of SR-11	✓
	SB-6	BS-1-0-1	Sedimentation basin Sediment	✓
	BB-5	SD-4-0-3	Equip. blank, sediment	✓
	B-19	N/A	trip blank	
	B-20	N/A	trip blank	
Jan 18	SWB-7	BZ-1-0-1	Sedimentation Basin Monitoring Zone	✓
	SWB-7F	"	"	
	DP-1	SS-3-1-1	Spill Pathway	✓
	DP-2	SS-8-2-1	Spill Pathway	✓
	DP-3	SS-13-1-1	"	✓
	DP-4	SS-16-2-1	"	✓
	DP-5	SS-17-2-1	"	✓
	EQB-1	SS-6-1-3	Equipment Blank (Soils)	✓
	TB-21	N/A	trip BLK	
	TB-22	N/A	trip BLK	
	DP-6	SS-23-2-1	Spill Pathway	✓
	DP-7	SS-27-1-1	"	✓
	DP-8	SS-29-2-1	"	✓
	DP-9	SS-33-2-1	"	✓
	DP-10	SS-38-1-1	"	✓
	DP-11	Dup of DP-10	"	✓
	TB-23	N/A	trip BLK	
	TB-24	N/A	trip BLK	
	TB-25	N/A	trip BLK	

	Versar #	RP sample #	location	Received
Mar 1	WS-1	SS-T-23-1-1	Wetlands Sediment	
	WS-2	SS-T-23-1-1	"	
	WS-3	SS-T-25-1-1	"	
	WS-4	SS-T-39-1-1	"	
	WS-5	SS-P-14-1-1	"	
	WS-6	SS-R-17-1-1	"	
	WS-7	SS-W-12-1-1	"	
	EQB	SS-T-12-1-1	Equipment blk	
	TB-26	N/A	trip blk	
	CB-1	SB-3-7-1	Catch basin Oning	
	CB-2	SB-4-5-1	"	
	EQB	SB-1-1-3	Equipment blank	
	TB-27	N/A	trip blank	
Apr 18	F-1 (Five Fillets)	F-1 (5 Fillets 5 whole body)	Down stream - Red lion creek	
	F-2 (Four Fillets)	F-2 (4 Fillets 4 whole body)	Up stream - "	
	—	F-3 (5 whole body)	Down stream "	

AR300575

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401
(301) 266-9180

DATE : May 3, 1990

SUBJECT: Data Review of Case R3-2 for Sample 45808 Re-extract
Site: Standard Chlorine

FROM : Cynthia E. Kennedy, Environmental Scientist CEK
Program Support Section (3ES23)

TO : Robert Guarni, Project Manager
DE/MD Section (3HW25)

THRU : Cindy Metzger, Chief CCM
Program Support Section (3ES23)

Overview

Case R3-2 contained seven (7) low concentration water samples and twenty (20) low concentration soil samples for analysis of the following eight extractable compounds:

1,2,3-Trichlorobenzene
1,2,4-Trichlorobenzene
1,2,3,4-Tetrachlorobenzene
1,2,4,5-Tetrachlorobenzene
Pentachlorobenzene
Hexachlorobenzene
1-Chloro-3-nitrobenzene
Nitrobenzene

This data review is for the re-extract of sample 45808 which was not included in the orginal data review report dated April 2, 1990. Analysis was performed by Versar, Inc. following Contract Laboratory Program (CLP) Statement of Work for the above parameters only.

Summary

All requested parameters were successfully analyzed for sample 45808 RE. Several compounds coeluted and therefore, had to be quantitated together. 1,2,3-Trichlorobenzene coeluted with 1,3,5-trichlorobenzene. Although 1,3,5-trichlorobenzene was not in the parameter list, the laboratory calculated 1,2,3-trichlorobenzene based on the response factor for 1,2,4-trichlorobenzene. The reported value for 1,2,3-trichlorobenzene is the combined value for both the 1,2,3- and 1,3,5-trichlorobenzene isomers. 1,2,4,5-Tetrachlorobenzene and 1,2,3,5-tetrachlorobenzene also [illegible] reported value for 1,2,4,5-tetrachlorobenzene is the combi[illegible] [illegible] isomers.

The initial calibration consisted of only three standard concentrations compared to the five required by the CLP SOW. Tentatively identified compounds were not determined.

Minor Problems

- Sample 45808 had all surrogate recovery values below 10% as mentioned in the orginal data review report. Re-extraction data was submitted to CRL on April 12, 1990. All surrogate recoveries for 45808RE were within the required recovery limits.

- Since a re-extraction was required, the analysis exceeded the technical holding time of fourteen (14) days by more than three (3) months. Sample results are qualified as biased low, "L", and detection limits are qualified as unreliable, "R".

- Two compounds failed precision criteria for the continuing calibration standards, pentachlorobenzene and hexachlorobenzene. Therefore the sample result for pentachlorobenzene was qualified as estimated, "J". The detection limit for hexachlorobenzene was previously qualified as unreliable,"R",

All data were reviewed using the National Functional Guidelines as modified for use by Region III. The text of this report has been formulated to address only those problem areas affecting data usability.

1. Appendix A - Glossary of data qualifier codes.
2. Appendix B - Data Summary Forms.
3. Appendix C - Results as reported by the laboratory.
4. Appendix D - Support Documentation.

APPENDIX A

Glossary of Data Qualifiers Codes

GLOSSARY OF DATA QUALIFIER CODES (ORGANIC)

CODES RELATING TO IDENTIFICATION
(confidence concerning presence or absence of compounds):

U - Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) - Confirmed identification.

B - Not detected substantially above the level reported in laboratory or field blanks.

R - Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N - Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

CODES RELATED TO QUANTITATION
(can be used for both positive results and sample quantitation limits):

J - Analyte present. Reported value may not be accurate or precise.

K - Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L - Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ - Not detected, quantitation limit may be inaccurate or imprecise.

UL - Not detected, quantitation limit is probably higher.

OTHER CODES

Q - No analytical result.

revised 01/90

APPENDIX B

Data Summary Forms

DATA SUMMARY FORM: Semi-volatiles

Site Name: Standard Chlorine

SOIL SAMPLES (ug/Kg)

Case #: R3-2 **Sampling Date(s):** 11/28/89

+ Result from diluted analysis
To calculate sample quantitation limit:
(CRQL * Dilution Factor) / ((100 - % moisture)/100)

	Sample No.	45808RE	
	Dilution Factor	1/5	
	% Moisture	51.2	
	Location	SR-14	
CRQL	COMPOUND		
330	1,2,3- Trichlorobenzene **	16700	L
330	1,2,4- Trichlorobenzene	54000†	L
330	1,2,3,4-Tetrachlorobenzene	32000†	L
330	1,2,4,5-Tetrachlorobenzene***	3500	L
330	Pentachlorobenzene	2906	J
330	Hexachlorobenzene		R
330	1-Chloro-3-nitrobenzene		R
330	Nitrobenzene		R

CRQL = [illegible]tract Required Quantitation Limit

SEE NARRATIVE FOR CODE DEFINITIONS

** 1,[illegible] Trichlorobenzene and 1,3,5- Trichlorobenzene coelute. Results presented are for bo[illegible]mers

*** 1,2,3,5- Tetrachlorobenzene and 1,2,4,5-Tetrachlorobenzene coelute. Results presented [illegible] for both isomers.

revised 12/88

AR300581

APPENDIX C

Results as reported by the laboratory for all target analytes

1B
SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

45808 RE

ab Name:VERSAR Contract:_____

ab Code:VERSAR Case No.:420.1 SAS No.: _____ SDG No.: 3 RE

atrix: (soil/water)SOIL (g/ml)G Lab Sample ID: __89911RE

ample wt/vol: 30.19 Lab File ID: __Z3995

evel:(low/med) LOW Date Received: ___11/30/89

Moisture: not dec._____ dec._____ Date Extracted: 01/10/90

xtraction: (SepF/Cont/Sonc) SONC Date Analyzed: 02/20/90

PC Cleanup: (Y/N) Y pH:_____ Dilution Factor: _ 1

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	
98-95-3	Nitrobenzene	1400	U
120-82-1	1,2,4-Trichlorobenzene		E
87-61-6	1,2,3-Trichlorobenzene	16700	E
121-73-3	1-Chloro-3-Nitrobenzene	1400	U
634-66-2	1,2,3,4-Tetrachlorobenzene		E
95-94-3	1,2,4,5-Tetrachlorobenzene	3500	
	Pentachlorobenzene	2900	
118-74-1	Hexachlorobenzene	1400	U

1) 1,2,3-Trichlorobenzene and 1,3,5-Trichlorobenzene coelute and cannot be quantified separately.

2) 1,2,3,5-Tetrachlorobenzene and 1,2,4,5-Tetrachlorobenzene coelute and cannot be quantified separately.

1B

SEMIVOLATILE ORGANICS ANALYSIS DATA SHEET

EPA SAMPLE NO.

45808 RE DL

Lab Name:VERSAR Contract:_____

Lab Code:VERSAR Case No.:420.1 SAS No.: _____ SDG No.: 3 RE

Matrix: (soil/water)SOIL (g/ml)G Lab Sample ID: __89911REDL

Sample wt/vol: 30.19 Lab File ID: __Z4018

Level:(low/med) LOW Date Received: ___11/30/89

% Moisture: not dec._____ dec._____ Date Extracted: 01/10/90

Extraction: (SepF/Cont/Sonc) SONC Date Analyzed: 02/22/90

GPC Cleanup: (Y/N) Y pH:_____ Dilution Factor: _ 5

CAS NO.	COMPOUND	CONCENTRATION UNITS: (ug/L or ug/Kg) ug/Kg	
98-95-3	Nitrobenzene	6800	U
120-82-1	1,2,4-Trichlorobenzene	54000	D
87-61-6	1,2,3-Trichlorobenzene	6800	U
121-73-3	1-Chloro-3-Nitrobenzene	6800	U
634-66-2	1,2,3,4-Tetrachlorobenzene	32000	D
95-94-3	1,2,4,5-Tetrachlorobenzene	6800	U
----------	Pentachlorobenzene	6800	U
118-74-1	Hexachlorobenzene	6800	U

1) 1,2,3-Trichlorobenzene and 1,3,5-Trichlorobenzene coelute and cannot be quantified separately.

2) 1,2,3,5-Tetrachlorobenzene and 1,2,4,5-Tetrachlorobenzene coelute and cannot be quantified separately.

AR300584

APPENDIX D

Support Documentation

CALIBRATION CHECK - SEMIVOLATILE HSL COMPOUNDS

CASE NO. _____

CONTRACT NO. _____

CONTRACT LAB: VERSAR

CALIBRATION DATE: 12/28/89

INSTRUMENT IDENTIFIER: Z

STANDARD FILE: Z4017

DATE: 2/22/90 TIME: 18:39

MINIMUM RF FOR SPCC IS .0500

MAXIMUM % D FOR CCC IS 25%

COMPOUND	MEAN RF(I)	RF(O)	% D
C410 NITROBENZENE	0.430	0.518	-20.590
C445 1,2,4-TRICHLOROBENZENE	0.450	0.438	2.635
C---- 1-CHLORO-3-NITROBENZENE	0.284	0.273	3.831
C---- 1235/1245 TETRACHLOROBE	0.738	0.604	18.130
C---- 1234-TETRACHLOROBENZENE	0.695	0.598	13.985
C---- PENTACHLOROBENZENE	0.760	0.529	30.374
C630 HEXACHLOROBENZENE	0.315	0.202	35.632

AR300586

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401
(301) 266-9180

DATE : January 4, 1990

SUBJECT: TSS and Alkalinity Report of Standard Chlorine

FROM : Daniel K. Donnelly (3ES21) DKD
Chief, Laboratory Branch

TO : Bob Guarni (3HW25)

Enclosed is a report for TSS and Alkalinity of the Standard Chlorine site. This report includes samples which were received at CRL on November 28 & 30, 1989. If you have any questions regarding this report, you may contact Ron Altman directly.

DKD:jr

Enclosure
a/s

cc: Virginia Pohlman
Versar

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401
(301) 266-9160

DATE : January 3, 1990

SUBJECT: TSS and Alkalinity Analyses of Standard Chlorine Samples
Superfund-Enforcement TGB03NPH6, (12/1/89 - 12/18/89), 891128-01 - 03, 891130-01

FROM : Ronald H.Altman RA
Chemist

TO : Daniel K. Donnelly
Chief, Laboratory Branch

THRU : Norman Fritsche NF
Team Leader, Inorganic Analysis Section

The results of the TSS and Alkalinity of the Standard Chlorine samples are presented below.

Additional quality control results are available upon request.

Sample Description:

Lab No.	Description
891128-01	Standard Chloride, Surface Water DO MS/MSD, Field Sample No. SWT-9
-02	Standard Chloride, Duplicate of SWT-9, Field Sample No. SWD-18
-03	Standard Chloride, Field Blank, Field Sample Number BB-10
891130-01	Standard Chloride, Surface Water Trib, Field Sample No. SWR-8

Results:

Sample Number	TSS(mg/l)	Alkalinity(mg/l)
891128-01	9 ± 1	18
-02	6	22.6 ± 0 (99%)
-03	<4	<2
891130-01	9 ± 1	29.3 ± 0 (99%)

RHA:ad

cc: Peggy Zawodny PZ
QCO

ICF INC.

CHAIN OF CUSTODY RECORD

PROJECT NO.	PROJECT NAME	PARAMETERS	INDUSTRIAL HYGIENE SAMPLE (Y / N)
5303.8	STANDARD CHLORINE		

SAMPLERS: (Signature) [signature] (Printed) DAVID SPENCER

FIELD SAMPLE NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	TSS	ALKALINITY							REMARKS TAG #S
SWT-9	11/27/89	1505		X	Surface water DO MS/MSD	4 ~~2~~	2	2	~~8911280~~						3-1063635 through 3-1063638
SWD-18	11/27/89	1505		X	Duplicate of SWT-9	2	1	1	~~8911280~~						3-1063645, 3-1063646
BB-10	11/27/89	1715		X	FIELD BLANK	2	1	1	~~8911280~~						3-1063639, 3-1063640

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[signature]	11/27/89 19:30				
(Printed) DAVID SPENCER		(Printed)	(Printed)		(Printed)

Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks
		[signature]	11/28 0909	Shipped via Federal Express Air Bill No. 4575149471
(Printed)		(Printed) NORMAN FRITSCHE		

Distribution: Original Plus One Accompanies Shipment (white and yellow); Copy to Coordinator Field Files (pink).

Versar INC.

CHAIN OF CUSTODY RECORD

PROJECT NO.	PROJECT NAME	PARAMETERS	INDUSTRIAL HYGIENE SAMPLE Y (N)
603041 5302	STANDARD CHLORINE		

SAMPLERS: (Signature) [signature] (Printed) DAVID SPENCER

FIELD SAMPLE NUMBER	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Alkalinity	TSS							REMARKS TAG NOS.
SWR-8	11/29/89	1445		X	SURFACE WATER TRIB	2	1	1							3-1063215, 3-1063216

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[signature]	11/29/89 1930				
(Printed)		(Printed)	(Printed)		(Printed)

Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks
		Norman Fritsche	11/30/89 0908	Shipped via Federal Express Airbill No. 4575149445
(Printed)		(Printed) NORMAN FRITSCHE		

Distribution: Original Plus One Accompanies Shipment (white and yellow); Copy to Coordinator Field Files (pink).

AR300590

AR300590

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
841 Chestnut Building
Philadelphia, Pennsylvania 19107

JUN 07 1990

Mr. Robert Touhey
Standard Chlorine of Delaware, Inc.
Governor Lea Road
P.O. Box 319
Delaware City, Delaware 19706

Re: Analytical Results of Split Samples

Dear Mr. Touhey:

Enclosed please find the latest sampling results provided to EPA. Although results are the most recent, they are not complete. I will forward the completed results when they become available to EPA.

Sincerely,

Robert Guarni
Remedial Project Manager
DE/MD Section

cc: Diane Wehner

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
REGION III
CENTRAL REGIONAL LABORATORY
839 BESTGATE ROAD
ANNAPOLIS, MARYLAND 21401
(301) 266-9180

DATE : May 24, 1990

SUBJECT: Organic Data Validation for the Standard Chlorine Site
SAS 5165C Tasks I, II

FROM : Theresa A. Simpson TAS
Region III ESAT DPO (3ES23)

TO : Robert Guarni
Regional Project Manager (3HW25)

THRU : Patricia J. Krantz, Chief TAS for
Quality Assurance Branch (3ES23)

Attached is the organic data review for the Standard Chlorine Site (SAS 5165C Tasks I, II) completed by the Region III Environmental Services Assistance Team (ESAT) contractor under the direction of Region III ESD.

If you have any questions regarding this review, please call me.

Attachment

cc: Dave Basko, Versar
Elaine Spiewak (3HW14) (w/o attachment)

TID File: 03900413 Task 1309

DATA SUMMARY FORM: NITROBENZENES

Site Name: STANDARD CHLORINE

Case #: 5165C-T1 Sampling Date: 01/04 - 01/10/90

SOIL SAMPLES
(ug/Kg)

To calculate sample quantit[illegible] (CRQL * Dilution Factor) / [illegible] % moisture/100)

5165C-TASK 1 - Sample No.	01	02	03	04	05	06	07	[illegible]	11
Dilution Factor	0.97 *	1.00 *	0.92 *	0.92 *	9.7 *	0.91 *	0.98 *	[illegible]	9.4 *
% Moisture	28.9	20.6	29.0	28.0	50.4	40.1	61.0	[illegible]	11.4
Location	WS-1	WS-2	WS-3	WS-4	WS-5	WS-6	WS-7	[illegible]	CB-2
	(Dup of WS-2)	(Dup. of WS-1)							

CRQL	COMPOUND	01		02		03		04		05		06		07		[illegible]		11	
3300	NITROBENZENE									6470						37400			
330	2,4-DINITROTOLUENE							<3300	J	<80000	J	<3300	J	<16500	J	144100		<33000	J

CRQL = Contract Required Quantitation Limit

* DUE TO AMOUNT OF SAMPLE ANALYZED AND FINAL EXTRACT VOLUME, THE EFFECTIVE DILUTION FACTOR VARIES SOMEWHAT (i.e. - 30.00g/31.0g = 0.967 X 2ml/2ml = 0.967)

AR300593

AR300593

WESTON

Appendix B

Data Summary Forms

GLOSSARY OF DATA QUALIFIER CODES (ORGANIC)

CODES RELATING TO IDENTIFICATION
(confidence concerning presence or absence of compounds):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N = Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

CODES RELATED TO QUANTITATION
(can be used for both positive results and sample quantitation limits):

J = Analyte present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

WESTON

Appendix A

Glossary of Data Qualifiers

All data for SAS 5165C-Task I and II were reviewed in accordance with the Functional Guidelines for Evaluating Organic Analyses with Modifications for use within Region III, and criteria set by the SAS contract. The text of this report addresses only those problems affecting usability.

ATTACHMENTS

1) Appendix A - Glossary of Data Qualifiers
2) Appendix B - Data Summary. These include:
   (a) All positive results for target compounds with qualifier codes where applicable.
   (b) All unusable detection limits (qualified "R").
3) Appendix C - Results as Reported by the Laboratory for All Target Compounds - Task I
4) Appendix D - Results as Reported by the Laboratory for All Target Compounds - Task II
5) Appendix E - Organic Regional Data Assessment Summary - Task I
6) Appendix F - Organic Regional Data Assessment Summary - Task II
7) Appendix G - Support Documentation - Task I
8) Appendix H - Support Documentation - Task II

DCN - DM005A12

- One pair of field duplicate samples were analyzed, but had no reported results for any of the multi-chlorinated-benzene target compounds. No useful comparison of the data can be made. One pair of analytical duplicate analyses were also performed on sample 5165C-Task II-09. The results of these analyses have been summarized in a form included in the Task II support documentation (Appendix H). All relative percent difference (RPD) values were within the QC limits set by the SAS contract. (See Appendix H).

- Due to the surrogate problem noted above, a matrix spike and matrix spike duplicate (MS/MSD) re-extraction was required. Since insufficient sample remained of the original sample chosen for the MS/MSD analyses, a different sample was chosen, without first being screened. Due to the high concentrations of the target analytes in the re-extracted MS and MSD samples, the MS and MSD spiking concentrations were insignificant. No spike recoveries (% R) or relative percent differences (%RPD's) have been reported for these analyses. No reported results were qualified based on this problem. (See Appendix H).

- The quantitation column external calibration check standard (the QC check standard) run for samples analyzed between 01/23 and 01/24/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,3,5-tetrachlorobenzene and 1,2,3,4-tetrachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

- The confirmation column external calibration check standard (the QC check standard) run for samples analyzed between 02/06 and 02/08/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,4-trichlorobenzene, 1,2,3,5-tetrachlorobenzene, pentachlorobenzene and hexachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

- The confirmation column continuing calibration run 02/08/90 at 04:36 had percent difference (%D) values for 1,2,3,4-tetrachlorobenzene and hexachlorobenzene greater than twenty percent (>20%), exceeding the QC limit set by the SAS contract. Since this column was used only for confirmation no reported results have been qualified. (See Appendix H).

- Due to the number of different dilution factors required to determine the various multi-chlorinated-benzene target compounds in some of the soil samples, the space set aside for the dilution factor(s) for each sample on the data summary forms was not large enough to include all the dilution factors used. Also, no indication of which dilution was used for which analyte can be made without extensive footnotes. The dilution factor(s) noted on the data summary forms represent only the dilutions performed on target compounds for which non-detect results were reported, or the lowest dilution used to determine a target compound if there were no non-detects. (See Appendix H). The analytical laboratory reported only one (1) Form I for each sample, regardless of the number of dilutions performed on that sample. Quantitation sheets for each dilution performed for each sample were included in the raw data for the case. Following is a table summarizing the dilution factor associated with each analyte for each sample:

	MULTI-CHLORINATED-BENZENE ISOMER						
SAMP.	123	124	135	1235	PENTA-	1234	HEXA-
-01	5X	5X	1X	1X	5X	5X	1X
-02	1X	1X	1X	1X	1X	1X	1X
-03	5X	100X	100X	5X	100X	100X	5X
-04	5X	100X	100X	5X	100X	100X	5X
-05	100X	4000X	4000X	100X	1000X	2000X	100X
-06	1X	100X	10X	1X	10X	100X	1X
-07	50X	5000X	250X	50X	250X	250X	50X
-08	1X	1X	1X	1X	1X	1X	1X
-09	1X	30000X	300X	600X	50X	600X	50X
-10	1X	1X	1X	1X	1X	1X	1X
-11	600X	6000X	1500X	1500X	600X	1500X	600X

- The QC-check sample had no positive result for metachloro-nitrobenzene. Since the control limits for recovery of this compound in the QC-check sample are advisory no corrective action was required. No reported results were qualified based on this problem. (See Appendix G).

- One pair of field duplicate samples, and one pair of analytical duplicate samples were analyzed, but had no reported results for nitrobenzene and metachloronitrobenzene. No useful comparison of the data can be made. (See Appendix G).

- The matrix spike/matrix spike duplicate (MS/MSD) analyses for Task I had all four (4) spike recoveries (%R) within the QC limit set by the SAS contract. Both of the relative percent difference (RPD) values were slightly outside the QC limit. Since the MSD extraction was performed two days after the MS extraction, and the MSD results are lower than the MS results, it is possible that the difference between the MS and MSD results is due to holding time related analyte loss from the sample.

  On the MS/MSD QC summary form the outliers are reported as "2 out of 6 outside limits". A more accurate representation of the data would be "zero (0) of four (4) spike recoveries, and two (2) out of two (2) RPD values outside limits". (See Appendix G).

Task II

- Due to low or no recovery of the surrogate compound 2-fluorobiphenyl chosen by the SAS contract, the soil samples were re-extracted. The re-extractions were performed using a different compound, 2-chloronaphthalene, for the surrogate spike. Since there was no sample volume remaining for the aqueous blanks associated with these samples, the blanks could not be re-extracted. No surrogate recoveries have been reported for the blanks. The extraction efficiency for these samples cannot be evaluated. (See Appendix H).

  The surrogate recoveries for the re-extractions of samples 5165C-Task II-09 dup and 5165C-Task II-11MS were below the QC limits set by the SAS contract. No reported resu[illegible] qualified based on this problem. (See Appendix H).

Task II

- Several of the soil samples were extracted eleven (11) to twelve (12) days from the date of sample collection, exceeding the ten (10) day holding time set by the SAS contract by one (1) to two (2) days. All samples were analyzed within the holding times set by the SAS contract. The reported results and quantitation limits for samples 5165C-Task II-01 through -07 were qualified "J" and "UJ", respectively. (See Appendix H).

- As noted in the report summary, the quantitation limit set for these samples was greater than the calibration range of the instrument used to analyze the samples. The calibration range of the instrument was 25 to 400 µg/L or about 8 to 133 µg/Kg. The CRQL set for the soil samples was 330 µg/Kg, about three (3) times the upper limit of the calibration range. Because of this, all reported results are less than the adjusted CRQL needed to represent dilutions performed on the sample extracts to meet the linear calibration range of the instrument. (e.g., a soil sample concentration of 1000 µg/Kg would require an eight fold (8X) dilution to fall within the calibration range of the instrument. The adjusted CRQL for a eight fold (8X) dilution is 330 µg/Kg X 8 or 2640 µg/Kg.)

  Based on this problem all soil sample reported results have been qualified "J", as an estimated result between the instrument detection limit and the CRQL. Although there may be some bias imposed on the results by the dilutions performed on the extracts (five to five thousand fold (5X - 5000X)), the data is more defensible than the qualification suggests. Most of the dilutions were performed by a serial dilution scheme, which would help to reduce the bias imposed by a large dilution. (See Appendix H).

NOTES

Task I

- The surrogate recoveries for sample 5165C-Task I-08 and the QC check sample (aqueous analyses) were slightly higher than the advisory QC limits set by the SAS contract. Also, most of the soil samples surrogate recoveries were affected by matrix interference, and were either not reported or greater than the advisory QC limits set by the SAS contract. Sin limits are advisory, no corrective action was required data has been qualified. (See Appendix G).

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DATE: 23 MAY 1990

SUBJECT: ORGANIC DATA VALIDATION FOR SAS 5165C-TASK I and II
Site: Standard Chlorine

FROM: DOUG McINNES DJM
ORGANIC DATA REVIEWER

DON O'BRIEN DJO
ORGANIC DATA REVIEWER

TO: TERRY SIMPSON
ESAT DEPUTY PROJECT OFFICER

THRU: RICHARD DRESSER
ESAT TEAM MANAGER

OVERVIEW

SAS 5165C-TASK I and II consisted of two (2) water samples and nine (9) soil samples per task, submitted to PACE for nitrobenzene and metachloronitrobenzene (Task I), and selected multi-chlorinated-benzene (Task II) analyses. The water samples included in this case were two (2) equipment blanks for each of the tasks. The samples were analyzed as a Contract Laboratory Program (CLP) Special Analytical Service (SAS).

SUMMARY

All samples were successfully analyzed for all target compounds for all samples. All instrument and method sensitivities for the nitrobenzene task (Task I) were according to the Contract Laboratory Program (CLP) Special Analytical Service (SAS) protocol. The quantitation limits set for the multi-chlorinated-benzene task (Task II) were above the calibration range of the instrument used. The instrument sensitivity was better than the required limits for Task II.

MINOR PROBLEMS

Task I

- Due to matrix interference, the quantitation limits for metachloronitrobenzene for several samples were estimated, based on the apparent concentration of the interference in each sample. Metachloronitrobenzene results for samples 5165C-Task I- 04, 05, 06, 07, and 11 were qualified J, denoting an estimated quantitation limit greater than the contract required detection limit (CRDL). The presence or absence of metachloronitrobenzene cannot be conf[illegible] for these samples. (See the analytical laboratory c[illegible] in Appendix G).

DATA SUMMARY FORM: CHLORO BENZENES

Site Name: STANDARD CHLORINE

Case #: 5165C-TII **Sampling Date:** 01/04 - 01/10/90

SOIL SAMPLES (ug/Kg)

To calculate sample quantitation: (CRQL * Dilution Factor) / ((1 - moisture/100)

5165C-TASK II - Sample No.		01		02		03		04		05		06		07		08		11	
Dilution Factor+		0.99/4.9 *		1.00 *		4.97 *		4.99 *		99 *		1/10 *		50 *		0.99/1.7 *		5990 *	
% Moisture		33.3		28.0		29.3		28.2		50.3		43.0		60.5		8.7		8.8	
Location		WS-1		WS-2		WS-3		WS-4		WS-5		WS-6		WS-7		CB-1		CB-2	
CRQL	COMPOUND	(Dup. of WS-2)		(Dup of WS-1)															
330	1,2,3-TRICHLORO BENZENE		UJ		UJ	530	J		UJ	1500	J		UJ	2700	J		UJ	2700	
330	1,2,4-TRICHLORO BENZENE					26000		13000	J	1100000		4400	J	150000		720000	J	690000	
330	1,3,5-TRICHLORO BENZENE					8400		8600		310000		1100	J	41000		21000		93000	
330	1,2,3,5-TETRACHLORO BENZENE					740		750		6500			UJ	1900		55000		15000	
330	PENTACHLORO BENZENE					2300		3400		34000				11000		3000		8100	
330	1,2,3,4-TETRACHLORO BENZENE					14000		13000		320000		2600	J	52000		85000		59000	
330	HEXACHLORO BENZENE						UJ		UJ	750			UJ	420			UJ	1900	

CRQL = Contract Required Quantitation Limit

* DUE TO AMOUNT OF SAMPLE ANALYZED AND FINAL EXTRACT VOLUME, THE EFFECTIVE DILUTION FA[illegible] VARIES [illegible]EWHAT (i.e. 30.00g/30.32g = 0.989 X [illegible]/1001)

\+ DUE TO THE NUMBER OF DILUTIONS PER SAMPLE, THE DE[illegible]

AR300603

AR300603

DATA SUMMARY FORM: NITROBENZENES

Site Name: STANDARD CHLORINE

Case #: 5165C-TI Sampling Date: 01/04-01/10/90

WATER SAMPLES
(ug/L)

To calculate sample quantitation limit:
(CRQL * Dilution Factor)

5165C-TASK I - Sample No.		08		10															
Dilution Factor		1.01 *		1.00 *															
Location		EQB-2		EQB-3															
CRQL	COMPOUND	(EQ. BLANK)		(EQ. BLANK)															
20																			
20																			

CRQL = Contract Required Quantitation Limit

* DUE TO AMOUNT OF SAMPLE ANALYZED, THE EFFECTIVE DILUTION FACTOR VARYS SLIGHTLY (i.e. - 1000ml/987ml = 1.013)

AR300604

DATA SUMMARY FORM: CHLORO BENZENES

Site Name: STANDARD CHLORINE

Case #: 5165C-TII Sampling Date: 01/04-01/10/90

WATER SAMPLES
(ug/L)

To calculate sample quantita limit:
(CRQL * Dilution Factor)

5165C - TASK II - Sample No.	08	10
Dilution Factor	1.04 *	1.00 *
Location	EQB-2	EQB-3
CRQL COMPOUND	(EQ. BLANK)	(EQ. BLANK)
10 1,2,3-TRICHLORO BENZENE		
10 1,2,4-TRICHLORO BENZENE		
10 1,3,5-TRICHLORO BENZENE		
10 1,2,3,5-TETRACHLORO BENZENE		
10 PENTACHLORO BENZENE		
10 1,2,3,4-TETRACHLORO BENZENE		
10 HEXACHLORO BENZENE		

CRQL = Contract Required Quantitation Limit

* - DUE TO AMOUNT OF SAMPLE ANALYZED THE EFFECTIVE DILUTION FACTOR VARYS SLIGHTLY (i.e. 1000 ml/960 ml = 1.042)

HR00605

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Appendix C

Results as Reported by the Laboratory
for all Target Compounds - Task I

Organics Analysis Data Sheet

EPA Sample No.
5165C-01-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58192

Sample Wt./Vol. 31.01 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 28.9 %

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/16/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 31.01 g / 2 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	ug/kg
	Metachloronitrobenzene	< 330	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: 1570 ug/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-02-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58193

Sample Wt./Vol. 30.11 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 30.6

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/16/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 30.11g / 2ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	µg/kg
	Metachloronitrobenzene	< 330	µg/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: 4200 µg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-03-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58194

Sample Wt./Vol. 32.77 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 29.0 %

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/16/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

~~Dilution~~ Concentration Factor 32.77g/2ml

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	ug/kg
	Metachlorobenzene	< 330	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interference Problems

Organics Analysis Data Sheet

EPA Sample No.
5165-04-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58195

Sample Wt./Vol. 32.59 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 28.0

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/16/90

Cleanup: Type None

Date Confirm. Anal. 1/25/90

Dilution Factor 32.59g / 2ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	ug/kg
	Metachloronitrobenzene	< 3300	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interferences Present

Organics Analysis Data Sheet

EPA Sample No.
5165C-05-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58196

Sample Wt./Vol. 30.81g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 50.4 %

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/17/90

Cleanup: Type NONE

Date Confirm. Anal. 1/25/90

Dilution Factor 30.81g / 20ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	6470	ug/kg
	Metachloronitrobenzene	< 80000	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interference Problems

Organics Analysis Data Sheet

EPA Sample No.
5165C-06-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58197

Sample Wt./Vol. 32.86 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 40.1

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/17/90

Cleanup: Type None

Date Confirm. Anal. 1/25/90

Dilution Factor 32.86g/2ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	µg/kg
	Metachlorobenzene	< 3300	µg/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interferences in Sample

Organics Analysis Data Sheet

EPA Sample No.
5165C-07-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58198

Sample Wt./Vol. 30.75 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 61.0

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/18/90

Cleanup: Type None

Date Confirm. Anal. 1/25/90

Dilution Factor 30.75g / 2ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitro Benzene	< 330	ug/kg
	Metachloro Nitro Benzene	< 16500	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interference Problems

Organics Analysis Data Sheet

EPA Sample No.
5165C-08-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Water

PACE Sample No. 58203

Sample Wt./Vol. 987 ml

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture N/A

Date Extracted 1/12/90

Extraction (SepF/Cont/Sonc) Sep F

Date Primary Anal. 1/15/90

Cleanup: Type None

Date Confirm. Anal. N/A

Dilution Factor 10ml/987 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitro Benzene	< 20	µg/l
	Meta Chloro Nitro Benzene	< 20	µg/l

Surrogate: 2-Fluorobiphenyl

Amount Found: 2385 µg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-09-Task1

Lab Name: PACE Laboratories, Inc.
Contract: 68-W8-0042

Lab Code: LANGST
SAS No.: 5165 C - Task 1

Matrix Soil
PACE Sample No. 58259

Sample Wt./Vol. 32.76 g
PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low
Date received 1/11/90

% Moisture 10.5
Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc
Date Primary Anal. 1/17/90

Cleanup: Type None
Date Confirm. Anal. 1/25/90

Dilution Factor 32.76g / 20ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	37400	ug/kg
	Metachloronitrobenzene	14400	ug/kg

Surrogate: 2-Fluorobiphenyl
Amount Found: Interference Problems

Organics Analysis Data Sheet

EPA Sample No.
5165C-10-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Water

PACE Sample No. 58260

Sample Wt./Vol. 1000 ml

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/11/90

% Moisture N/A

Date Extracted 1/12/90

Extraction (SepF/Cont/Sonc) SepF

Date Primary Anal. 1/15/90

Cleanup: Type None

Date Confirm. Anal. N/A

Dilution Factor 10ml / 1000 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 20	µg/l
	Metachloronitrobenzene	< 20	µg/l

Surrogate: 2-Fluorobiphenyl

Amount Found: 1988 µg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-11-TASK1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58261

Sample Wt./Vol. 31.84 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/11/90

% Moisture 11.4

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/18/90

Cleanup: Type None

Date Confirm. Anal. 1/25/90

Dilution Factor 31.84 g / 20 ml

Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 3300	ug/kg
	Metachloronitrobenzene	< 33000	ug/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: Interference Problems

Organics Analysis Data Sheet

EPA Sample No.
5165C-11 Dup-Task1

Lab Name: PACE Laboratories, Inc.

Lab Code: LANGST

Matrix Soil

Sample Wt./Vol. 30.22 g

Level (~~Wt./Vol.~~) Low

% Moisture 11.4

Extraction (SepF/Cont/Sonc) Sonc

Cleanup: Type None

Dilution Factor 30.22g / 20 ml
Concentration

Contract: 68-W8-0042

SAS No.: 5165 C - Task 1

PACE Sample No. 58199

PACE File No. N/A

Date received 1/11/90

Date Extracted 1/13/90

Date Primary Anal. 1/18/9

Date Confirm. Anal. 1/25/

CAS No.	Compound	Concentration	Units
	Nitro Benzene	< 3300	ug/kg
	Metachlorod Nitro Benzene	< 33000	ug/kg

Surrogate: 2-Fluoro Biphenyl

Amount Found: Interference Problems

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Appendix D

Results as Reported by the Laboratory
for all Target Compounds - Task II

EPA Sample #
[illegible]

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: [illegible]

Lab Sample ID [illegible]

Sample wt/vol: 30.3g

Date Received [illegible]

Level: [illegible]

Date Extracted [illegible]

% Moisture: 13.3%

Dry Weight [illegible]g

Extraction Type [illegible]

PH [illegible]

Instrument ID: A

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg)	Final Extract Volume
123 trichlorobenzene	<490 ug/kg	50mL
124 trichlorobenzene.	<490 ug/kg	50mL
135 trichlorobenzene	<490 ug/kg	10mL
1235 tetrachlorobenzene	<490 ug/kg	10mL
pentachlorobenzene	<490 ug/kg	50mL
1234 tetrachlorobenzene	<490 ug/kg	50mL
hexachlorobenzene	<490 ug/kg	10mL

EPA Sample #
[illegible]

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID [illegible]

Sample wt/vol: 20.00g

Date Received [illegible]

Level: Low

Date Extracted [illegible]

% Moisture: 75%

Dry Weight [illegible]

Extraction Type [illegible]

PH [illegible]

Instrument ID: A

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg)	Final Extract Volume
123 trichlorobenzene	<440 ug/Kg	10ml
124 trichlorobenzene	<440 ug/Kg	10ml
135 trichlorobenzene	<440 ug/Kg	10ml
1235 tetrachlorobenzene	<440 ug/Kg	10ml
pentachlorobenzene	<440 ug/Kg	10ml
1234 tetrachlorobenzene	<440 ug/Kg	10ml
hexachlorobenzene	<440 ug/Kg	10ml

5165C-03

EPA Sample #

[illegible]-3

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: [illegible]

Lab Sample ID [illegible]

Sample wt/vol: [illegible] g

Date Received [illegible]

Level: low

Date Extracted [illegible]

% Moisture: [illegible]

Dry Weight [illegible]

Extraction Type [illegible]

PH [illegible]

Instrument ID: a

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg) ug/kg	Final Extract Volume
123 trichlorobenzene	530 ug/kg	50 ml
124 trichlorobenzene	21,000 ug/kg	1000 ml
135 trichlorobenzene	2400 ug/kg	1000 ml
1235 tetrachlorobenzene	740 ug/kg	50 ml
pentachlorobenzene	2300 ug/kg	1000 ml
1234 tetrachlorobenzene	14,000 ug/kg	1000 ml
hexachlorobenzene	<460 ug/kg	50 ml

5165C-04

EPA Sample # [illegible]

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID [illegible]

Sample wt/vol: [illegible]

Date Received [illegible]

Level: [illegible]

Date Extracted [illegible]

% Moisture: [illegible]

Dry Weight [illegible]

Extraction Type Sonc.

PH 3.9

Instrument ID: 9

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg)	Final Extract Volume
123 trichlorobenzene	<460 ug/Kg	50ml
124 trichlorobenzene	13,000 ug/Kg	1000ml
135 trichlorobenzene	5600 ug/Kg	1000ml
1235 tetrachlorobenzene	750 ug/Kg	50ml
pentachlorobenzene	3400 ug/Kg	1000ml
1234 tetrachlorobenzene	13000 ug/Kg	1000ml
hexachlorobenzene	<460 ug/Kg	50ml

5165C-05

EPA Sample # 1505

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID 60962

Sample WT/vol: 30.2g

Date Received 01/09/90

Level: 1500

Date Extracted 01/16/90

% Moisture: 50.3%

Dry Weight 15.0g

Extraction Type Sonc.

PH 6.5

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	4500 ug/Kg	1000ml
124 trichlorobenzene	1,100,000 ug/Kg	40,000ml
135 trichlorobenzene	370,000 ug/Kg	40,000ml
1235 tetrachlorobenzene	6500 ug/Kg	1000ml
pentachlorobenzene	34,000 ug/Kg	10,000ml
1234 tetrachlorobenzene	320,000 ug/Kg	20,000ml
hexachlorobenzene	750 ug/Kg	1000ml

EPA Sample #
[illegible]

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID 60963

Sample wt/vol: 30.0g

Date Received [illegible]

Level: [illegible]

Date Extracted [illegible]

% Moisture: 43.0%

Dry Weight [illegible]

Extraction Type [illegible]

PH [illegible]

Instrument ID: [illegible]

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg) ug/kg	Final Extract Volume
123 trichlorobenzene	<580 ug/kg	10mL
124 trichlorobenzene	4400 ug/kg	1000mL
135 trichlorobenzene	1100 ug/kg	10mL
1235 tetrachlorobenzene	<580 ug/kg	10mL
pentachlorobenzene	<580 ug/kg	100mL
1234 tetrachlorobenzene	2600 ug/kg	1000mL
hexachlorobenzene	<580 ug/kg	10mL

EPA Sample # 5165C-07

WS-7

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID CC964

Sample wt/vol: 30.0g

Date Received [illegible]

Level: Low

Date Extracted [illegible]

% Moisture: 60.5%

Dry Weight 11.9g

Extraction Type Sonc.

PH 1.2g

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	2700 ug/Kg	500ml
124 trichlorobenzene	150,000ug/Kg	50,000ml
135 trichlorobenzene	41,000 ug/Kg	2500ml
1235 tetrachlorobenzene	1900ug/Kg	500ml
pentachlorobenzene	11,000ug/Kg	2500ml
1234 tetrachlorobenzene	52,000ug/Kg	2500ml
hexachlorobenzene	420ug/Kg	500ml

5165C-08

EPA Sample # EQB-2

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: water

Lab Sample ID C0965

Sample wt/vol: 960mL

Date Received 01/09/90

Level: low

Date Extracted 01/11/90

% Moisture: _____

Dry Weight _____

Extraction Type Sep F

PH 6.5

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/l	Final Extract Volume
123 trichlorobenzene	<9.9 ug/l	10mL
124 trichlorobenzene	<9.9 ug/l	10mL
135 trichlorobenzene	<9.9 ug/l	10mL
1235 tetrachlorobenzene	<9.9 ug/l	10mL
pentachlorobenzene	<9.9 ug/l	10mL
1234 tetrachlorobenzene	<9.9 ug/l	10mL
hexachlorobenzene	<9.9 ug/l	10mL

5165C-09

EPA Sample #

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID [illegible]

Sample wt/vol: [illegible]

Date Received [illegible]

Level: [illegible]

Date Extracted [illegible]

% Moisture: [illegible]

Dry Weight [illegible]

Extraction Type [illegible]

PH [illegible]

Instrument ID: [illegible]

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg) ug/kg	Final Extract Volume
123 trichlorobenzene	<360 ug/kg	[illegible]
124 trichlorobenzene	>70,000 ug/kg	60,000 ml
135 trichlorobenzene	21,000 ug/kg	3000 ml
1235 tetrachlorobenzene	55,000 ug/kg	6000 ml
pentachlorobenzene	3600 ug/kg	500 ml
1234 tetrachlorobenzene	85,000 ug/kg	6000 ml
hexachlorobenzene	<360 ug/kg	500 ml

5165-[illegible] Duplicate

EPA Sample #

[illegible]

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID [illegible] Duplicate

Sample wt/vol: 29.95g

Date Received [illegible]

Level: [illegible]

Date Extracted [illegible]

% Moisture: 8.7%

Dry Weight [illegible]g

Extraction Type [illegible]

PH [illegible]

Instrument ID: A

GC Column ID: [illegible]

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	< 360 ug/Kg	10 ml
124 trichlorobenzene	[illegible] ug/Kg	[illegible] ml
135 trichlorobenzene	24,000 ug/Kg	3000 ml
1235 tetrachlorobenzene	53,000 ug/Kg	6000 ml
pentachlorobenzene	3400 ug/Kg	500 ml
1234 tetrachlorobenzene	81,000 ug/Kg	6000 ml
hexachlorobenzene	< 360 ug/Kg	10 ml

5165C-10

EPA Sample #
EGB-3

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Water

Lab Sample ID 01492

Sample wt/vol: 1000mL

Date Received 01/11/90

Level: Low

Date Extracted 01/11/90

% Moisture: _____

Dry Weight _____

Extraction Type LLE

PH 6.5

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/L	Final Extract Volume
123 trichlorobenzene	<9.9 ug/L	10mL
124 trichlorobenzene	<9.9 ug/L	10mL
135 trichlorobenzene	<9.9 ug/L	10mL
1235 tetrachlorobenzene	<9.9 ug/L	10mL
pentachlorobenzene	<9.9 ug/L	10mL
1234 tetrachlorobenzene	<9.9 ug/L	10mL
hexachlorobenzene	<9.9 ug/L	10mL

5165-C-11

EPA Sample #
CB-2

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID C1493

Sample wt/vol: 30.03g

Date Received 01/09/90

Level: Low

Date Extracted 01/10/90

% Moisture: 9.8%

Dry Weight 27.4g

Extraction Type Sonc

PH 7.6%

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	2700 ug/Kg	6000mL
124 trichlorobenzene	690,000 ug/Kg	60,000mL
135 trichlorobenzene	93,000 ug/Kg	15,000mL
1235 tetrachlorobenzene	15,000 ug/Kg	15,000mL
pentachlorobenzene	8200 ug/Kg	6000mL
1234 tetrachlorobenzene	59,000 ug/Kg	15000mL
hexachlorobenzene	1900 ug/Kg	6000mL

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Appendix E

Organic Regional Data Assessment Summary
Task I

WESTON

DPO: [ ] ACTION [X] FYI

Region III

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY

CASE NO: 5165C-TASK I
SDG NO:
SOW:
NO. OF SAMPLES: 2

LABORATORY: PACE
DATA USER: PAT CHURILLA
REVIEW COMPLETION DATE: 04/25/90
MATRIX: WATER

REVIEWER: ESAT

	VOA	BNA	PEST	OTHER *
1. HOLDING TIMES				O
2. GC-MS TUNE/GC PERFORMANCE				O
3. INITIAL CALIBRATIONS				O
4. CONTINUING CALIBRATION				O
5. FIELD BLANKS (F=NOT APPLICABLE)				O
6. LABORATORY BLANKS				O
7. SURROGATES				O
8. MATRIX SPIKE/DUPLICATES				O
9. REGIONAL QC (F=NOT APPLICABLE)				F
10. INTERNAL STANDARDS				F
11. COMPOUND IDENTIFICATION				O
12. COMPOUND QUANTITATION				O
13. SYSTEM PERFORMANCE				O
14. OVERALL ASSESSMENT				O

O = No problems or minor problems that do not affect data usability
X = No more than about 5% of the data points are qualified as either estimated or unusable.
M = More than about 5% of the data points are qualified as estimated.
Z = More than about 5% of the data points are qualified as unusable.
A = DPO action requested; use in conjunction with one of the above codes.

DPO ACTION ITEMS:

AREAS OF CONCERN: * Other - The Task I target analytes are nitrobenzene and metachloronitrobenzene

DOCUMENTATION ATTACHED (See Following Pages)

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK I (soil samples)

Item 7A - The surrogate recoveries for sample 5165C-Task I-08 and the QC check sample (aqueous analyses) were slightly higher than the advisory QC limits set by the SAS contract. Also, most of the soil samples surrogate recoveries were affected by matrix interference, and were either not reported or greater than the advisory QC limits set by the SAS contract. Since the QC limits are advisory, no corrective action was required. (See Appendix G).

Item 8A - The matrix spike/matrix spike duplicate (MS/MSD) analyses for Task I had all four (4) spike recoveries (%R) within the QC limit set by the SAS contract. Both of the relative percent difference (RPD) values were slightly outside the QC limit. Since the MSD extraction was performed two days after the MS extraction, and the MSD results are lower than the MS results, it is possible that the difference between the MS and MSD results is due to holding time related analyte loss from the sample.

On the MS/MSD QC summary form the outliers are reported as "2 out of 6 outside limits". A more accurate representation of the data would be "zero (0) of four (4) spike recoveries, and two (2) out of two (2) RPD values outside limits". (See Appendix G).

Item 12A - Due to matrix interference, the quantitation limits for metachloronitrobenzene for several samples were estimated, based on the apparent concentration of the interference in each sample. Metachloronitrobenzene results for samples 5165C-Task I- 04, 05, 06, 07, and 11 were reported as estimated quantitation limits greater than the contract required detection limit (CRDL). The presence or absence of metachloronitrobenzene cannot be confirmed for these samples. (See the analytical laboratory case narrative in Appendix G).

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK I (soil samples)

Item 13A - The QC-check sample had no positive result for metachloronitrobenzene. Since the control limits for recovery of this compound in the QC-check sample are advisory no corrective action was required. No reported results were qualified based on this problem. (See Appendix G).

Item 14A - One pair of field duplicate samples, and one pair of analytical duplicate samples were analyzed, but had no reported results for nitrobenzene and metachloronitrobenzene. No useful comparison of the data can be made. (See Appendix G).

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Appendix F

Organic Regional Data Assessment Summary
Task II

WESTON

DPO: [ ] ACTION [X] FYI

Region III

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY

CASE NO: 5165C-TASK II
SDG NO:
SOW:
NO. OF SAMPLES: 2

LABORATORY: PACE
DATA USER: PAT CHURILLA
REVIEW COMPLETION DATE: 04/25/90
MATRIX: WATER

REVIEWER: ESAT

	VOA	BNA	PEST	OTHER *
1. HOLDING TIMES				O
2. GC-MS TUNE/GC PERFORMANCE				O
3. INITIAL CALIBRATIONS				O
4. CONTINUING CALIBRATION				O
5. FIELD BLANKS (F=NOT APPLICABLE)				O
6. LABORATORY BLANKS				O
7. SURROGATES				O
8. MATRIX SPIKE/DUPLICATES				O
9. REGIONAL QC (F=NOT APPLICABLE)				F
10. INTERNAL STANDARDS				F
11. COMPOUND IDENTIFICATION				O
12. COMPOUND QUANTITATION				O
13. SYSTEM PERFORMANCE				O
14. OVERALL ASSESSMENT				O

O = No problems or minor problems that do not affect data usability
X = No more than about 5% of the data points are qualified as either estimated or unusable.
M = More than about 5% of the data points are qualified as estimated.
Z = More than about 5% of the data points are qualified as unusable.
A = DPO action requested; use in conjunction with one of the above codes.

DPO ACTION ITEMS:

AREAS OF CONCERN: * Other - The Task II target analytes are selected multi-chlorinated-benzene isomers

DOCUMENTATION ATTACHED (See Following Pages)

WESTON

DPO: [ ] ACTION [X] FYI

Region III

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY

CASE NO: 5165C-TASK II
SDG NO:
SOW:
NO. OF SAMPLES: 9

LABORATORY: PACE
DATA USER: PAT CHURILLA
REVIEW COMPLETION DATE: 04/25/90
MATRIX: SOIL

REVIEWER: ESAT

	VOA	BNA	PEST	OTHER *
1. HOLDING TIMES				M
2. GC-MS TUNE/GC PERFORMANCE				O
3. INITIAL CALIBRATIONS				O
4. CONTINUING CALIBRATION				O
5. FIELD BLANKS (F=NOT APPLICABLE)				O
6. LABORATORY BLANKS				O
7. SURROGATES				O
8. MATRIX SPIKE/DUPLICATES				O
9. REGIONAL QC (F=NOT APPLICABLE)				F
10. INTERNAL STANDARDS				F
11. COMPOUND IDENTIFICATION				O
12. COMPOUND QUANTITATION				M
13. SYSTEM PERFORMANCE				O
14. OVERALL ASSESSMENT				M

O = No problems or minor problems that do not affect data usability
X = No more than about 5% of the data points are qualified as either estimated or unusable.
M = More than about 5% of the data points are qualified as estimated.
Z = More than about 5% of the data points are qualified as unusable.
A = DPO action requested; use in conjunction with one of the above codes.

DPO ACTION ITEMS:

AREAS OF CONCERN: * Other - The Task II target analytes are selected multi-chlorinated-benzene isomers

DOCUMENTATION ATTACHED (See Following Pages)

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK II (water samples)

Item 4A - The confirmation column continuing calibration run 02/08/90 at 04:36 had percent difference (%D) values for 1,2,3,4-tetrachlorobenzene and hexachlorobenzene greater than twenty percent (>20%), exceeding the QC limit set by the SAS contract. Since this column was used only for confirmation no reported results have been qualified. (See Appendix H).

Item 7A - Due to low or no recovery of the surrogate compound 2-fluorobiphenyl chosen by the SAS contract, the soil samples were re-extracted. The re-extractions were performed using a different compound, 2-chloronaphthalene, for the surrogate spike. Since there was no sample volume remaining for the aqueous blanks associated with these samples, the blanks could not be re-extracted. No surrogate recoveries have been reported for the blanks. The extraction efficiency for these samples cannot be evaluated. (See Appendix H).

Item 13A - The quantitation column external calibration check standard (the QC check standard) run for samples analyzed between 01/23 and 01/24/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,3,5-tetrachlorobenzene and 1,2,3,4-tetrachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

Item 13B - The confirmation column external calibration check standard (the QC check standard) run for samples analyzed between 02/06 and 02/08/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,4-trichlorobenzene,1,2,3,5-tetrachlorobenzene, pentachlorobenzene and hexachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

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ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK II (soil samples)

Item 1A - Several of the soil samples were extracted eleven (11) to twelve (12) days from the date of sample collection, exceeding the ten (10) day holding time set by the SAS contract by one (1) to two (2) days. All samples were analyzed within the holding times set by the SAS contract. (See Appendix H).

Item 4A - The confirmation column continuing calibration run 02/08/90 at 04:36 had percent difference (%D) values for 1,2,3,4-tetrachlorobenzene and hexachlorobenzene greater than twenty percent (>20%), exceeding the QC limit set by the SAS contract. Since this column was used only for confirmation no reported results have been qualified. (See Appendix H).

Item 8A - Due to the surrogate problem noted above, a matrix spike and matrix spike duplicate (MS/MSD) re-extraction was required. Since insufficient sample remained of the original sample chosen for the MS/MSD analyses, a different sample was chosen, without first being screened. Due to the high concentrations of the target analytes in the re-extracted MS and MSD samples, the MS and MSD spiking concentrations were insignificant. No spike recoveries (% R) or relative percent differences (%RPD's) have been reported for these analyses. (See Appendix H).

Item 12A - As noted in the report summary, the quantitation limit set for these samples was greater than the calibration range of the instrument used to analyze the samples. The calibration range of the instrument was 25 to 400 µg/L or about 8 to 133 µg/Kg. The CRQL set for the soil samples was 330 µg/Kg, about three (3) times the upper limit of the calibration range. Because of this, all reported results are less than the adjusted CRQL needed to represent dilutions performed on the sample extracts to meet the linear calibration range of the instrument. (e.g., a soil sample concentration of 1000 µg/Kg would require an eight fold (8X) dilution to fall within the calibration range of the instrument. The adjusted CRQL [illegible] eight fold (8X) dilution is 330 µg/Kg X 8 or [illegible]

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK II (soil samples)

The surrogate recoveries for the re-extractions of samples 5165C-Task II-09 dup and 5165C-Task II-11MS were below the QC limits set by the SAS contract. (See Appendix H).

Item 13A - The quantitation column external calibration check standard (the QC check standard) run for samples analyzed between 01/23 and 01/24/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,3,5-tetrachlorobenzene and 1,2,3,4-tetrachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

Item 13B - The confirmation column external calibration check standard (the QC check standard) run for samples analyzed between 02/06 and 02/08/90 exceeded the QC limits set by the SAS contract for the compounds 1,2,4-trichlorobenzene,1,2,3,5-tetrachlorobenzene, pentachlorobenzene and hexachlorobenzene. Since the QC limits are advisory, no results were qualified. (See Appendix H).

Item 14A - Due to the number of different dilution factors required to determine the various multi-chlorinated-benzene target compounds in some of the soil samples, the space set aside for the dilution factor(s) for each sample on the data summary forms was not large enough to include all the dilution factors used. Also, no indication of which dilution was used for which analyte can be made without extensive footnotes. The dilution factor(s) noted on the data summary forms represent only the dilutions performed on target compounds for which non-detect results were reported, or the lowest dilution used to determine a target compound if there were no non-detects. (See Appendix H).

The analytical laboratory reported only one (1) Form I for each sample, regardless of the number of dilutions performed on that sample. Quantitation sheets for each dilution performed for each sample were included in the raw dat[illegible] [illegible] the case. Following is a table summariz[illegible] factor associated with each analyte [illegible] [illegible] sample:

ORGANIC REGIONAL DATA ASSESSMENT SUMMARY NOTES
SAS 5165C-TASK II (soil samples)

	MULTI-CHLORINATED-BENZENE ISOMER						
SAMP.	123	124	135	1235	PENTA-	1234	HEXA-
-01	5X	5X	1X	1X	5X	5X	1X
-02	1X	1X	1X	1X	1X	1X	1X
-03	5X	100X	100X	5X	100X	100X	5X
-04	5X	100X	100X	5X	100X	100X	5X
-05	100X	4000X	4000X	100X	1000X	2000X	100X
-06	1X	100X	10X	1X	10X	100X	1X
-07	50X	5000X	250X	50X	250X	250X	50X
-09	1X	30000X	300X	600X	50X	600X	50X
-11	600X	6000X	1500X	1500X	600X	1500X	600X

Item 14B - One pair of field duplicate samples were analyzed, but had no reported results for any of the multi-chlorinated-benzene target compounds. No useful comparison of the data can be made. One pair of analytical duplicate analyses were also performed on sample 5165C-Task II-09. The results of these analyses have been summarized in a form included in the Task II support documentation (Appendix H). All relative percent difference (RPD) values were within the QC limits set by the SAS contract. (See Appendix H).

DCN - DM005A13

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Appendix G

Support Documentation - Task I

PACE Laboratories, Inc.

Surrogate Recovery Form

Matrix Water

Project 5165C-Task1

Component 2-Fluorobiphenyl

Sample	Amount Spiked	Amount Found	Percent Recovery	Limits Recovery
5165C-RBikw-Task1	2000 ug/L	2283 ug/L	114%	30-115
5165C-08-Task1	2000 ug/L	2385 ug/L	119%	30-115
5165C-10-Task1	2000 ug/L	1988 ug/L	99.4%	30-115
5165C-GCClack-Task1	2000 ug/L	2359 ug/L	118%	30-115

Comments No Corrective Action taken

PACE Laboratories, Inc.

Surrogate Recovery Form

Matrix Soil

Project 5165C - Task 1

Component 2-Fluorobiphenyl

Sample	Amount Spiked	Amount Found	Percent Recovery	Limits Recovery
5165C-RB1KS-Task1	2000 µg/g	990 µg/g	49.5%	
5165C-RB1KS1-Task1	2000 µg/g	728 µg/g	36.4%	
5165C-01-Task1	2000 µg/g	1570 µg/g	78.5%	
5165C-01MS-Task1	2000 µg/g	2016 µg/g	101%	
5165C-01MSD-Task1	2000 µg/g	1132 µg/g	56.6%	
5165C-02-Task1	2000 µg/g	4200 µg/g	210%	
5165C-03-Task1	*			
5165C-04-Task1	*			
5165C-05-Task1	*			
5165C-06-Task1	*			
5165C-07-Task1	*			
5165C-09-Task1	*			
5165C-11-Task1	*			
5165C-11Dup-Task1	*			

Comments * - Interferences in these samples prevented Quantitation of Surrogate Data

PACE Laboratories, Inc. GC Section

Matrix Spike & Matrix Spike Duplicate Report Forms 0046

Test 8090 Project # 500109.503

Analyst OK Reference

Date Analyzed 1/19/90

Sample # 5165C-01-TASK1 / 5165C-01MS-TASK1 / 5165C-01MSD-TASK

Matrix Spike

Component	Amount in sample	Amount Spiked	Amount in spike	% Recovery	Limits
Nitrobenzene	<330	2702	1638 µg/kg	60.6	6-118
Methylchlorobenzene	<330	2251	1561 µg/kg	69.3	6-118

Matrix Spike Duplicate

Component	Amount in sample	Amount Spiked	Amount in MSD	% Recovery	Limits	RPD	RPD Limits
Nitrobenzene	<330	2666	1102 µg/kg	41.3	6-118	37.9	2
Methylchlorobenzene	<330	2221	1082 µg/kg	48.7	6-118	34.9	2

2 out of 6 outside limits

Comments: Limits found in method 8090 for Nitrobenzene

Organics Analysis Data Sheet

EPA Sample No.
5165C-RB1kw-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Water

PACE Sample No. 58204

Sample Wt./Vol. 1000 ml

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received N/A

% Moisture N/A

Date Extracted 1/12/90

Extraction (SepF/Cont/Sonc) SepF

Date Primary Anal. 1/15/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 10ml / 1000 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 20	µg/l
	Metachloronitrobenzene	< 20	µg/l

Surrogate: 2-Fluorobiphenyl

Amount Found: 2283 µg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C - RB1KS-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58201

Sample Wt./Vol. 30.00 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received N/A

% Moisture 0

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/16/90

Cleanup: Type None

Date Confirm. Anal. 1/24/90

Dilution Factor 30.00 g / 2 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	µg/kg
	Metachloronitrobenzene	< 330	µg/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: 990 µg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165-RB1KS1-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 59987

Sample Wt./Vol. 30.00 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received N/A

% Moisture 0

Date Extracted 1/15/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/17/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 30.00 g / 2 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	< 330	µg/kg
	Metachloronitrobenzene	< 330	µg/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: 728 µg/g (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-01MS-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 58200

Sample Wt./Vol. 31.33

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 28.9

Date Extracted 1/13/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/17/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 31.33g /2ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitro Benzene	<330	µg/kg
	Metachloro Nitrobenzene	<330	µg/kg

Surrogate: 2-Fluorobiphenyl

Amount Found: 2927 mg/l (in Solution)

Organics Analysis Data Sheet

EPA Sample No.
5165C-01MS0-Task 1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Soil

PACE Sample No. 59761

Sample Wt./Vol. 31.76 g

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 1/8/90

% Moisture 28.9

Date Extracted 1/15/90

Extraction (SepF/Cont/Sonc) Sonc

Date Primary Anal. 1/17/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 31.76g / 2ml

Concentration

CAS No.	Compound	Concentration	Units
	Nitro Benzene		
	Meta Chloro Nitro Benzene		

Surrogate: 2-Fluoro Biphenyl

Amount Found: 3188 ug/kg

Organics Analysis Data Sheet

EPA Sample No.
5165C-Qccheck-Task1

Lab Name: PACE Laboratories, Inc.

Contract: 68-W8-0042

Lab Code: LANGST

SAS No.: 5165 C - Task 1

Matrix Water

PACE Sample No. 58202

Sample Wt./Vol. 1000

PACE File No. N/A

Level ~~(Wt./Vol.)~~ Low

Date received 11/3/88

% Moisture N/A

Date Extracted 1/12/90

Extraction (SepF/Cont/Sonc) SepF

Date Primary Anal. 1/15/90

Cleanup: Type None

Date Confirm. Anal. 1/19/90

Dilution Factor 10 ml / 1000 ml
Concentration

CAS No.	Compound	Concentration	Units
	Nitrobenzene	31.3	µg/l
	Metachloronitrobenzene	< 20	

Surrogate: 2-Fluorobiphenyl

Amount Found: 2359 µg/l (in Solution)

Revision

TES/VERSAR

page 1 of 1 EPA SAMPLE SHIPPING LOG FOR ALL SAMPLES SENT THROUGH THE CONTRACT LAB PROGRAM (12/85 version)

CASE# _____ SAS# 5165G-TASK 1 SITE NAME: STANDARD CHLORINE

Site Leader: DAVID SPENCER EPA Project Officer: Bob GUARNI

Phone: (215) 741-4211

SAS REQUEST: (details required)

① Nitrobenzene, metachloronitrobenzene

QC SAMPLE INFO AND/OR COMMENTS	CONC. (low/ med/ high)	SAMPLE PHASE (aq/ sol)	TYPE OF REQUEST org, dio inor, SAS	SAMPLE TRAFFIC REPORT NUMBER	ORGANICS OR INORGANICS LAB NAME	DATE SHIPPED	DATA RECEIVED XX out items not requested VOA	BNA	PEST	TCDD	METALS	CN	LAB NAME	SAS REQUEST (itemize)	DATE SHIPPED	DATA REC'D
MS/MSD	LOW	SOL	SAS	5165G01 TASK 1									LANGSTON	①	1/5/90	rec'd 2-27-90 JTS ✓
Dup of 5165C-01	↓	↓	↓	-02									↓	↓	↓	✓
				-03												✓
				-04												✓
				-05												✓
				-06												✓
				-07												✓
EQ BLANK	↓	AQ	↓	-08									↓	↓	↓	✓

AR300655

Revision

TES/UERSAR

page 1 of 1 EPA SAMPLE SHIPPING LOG FOR ALL SAMPLES SENT THROUGH THE CONTRACT LAB PROGRAM (12/85 version)

CASE# _____ SAS# 5165C-TASK 1 SITE NAME: STANDARD CHLORINE

Site Leader: DAVID SPENCER EPA Project Officer: Bob GUARNI

Phone: (215) 741-4211

QC SAMPLE INFO AND/OR COMMENTS	CONC. (low/med/high)	SAMPLE PHASE (aq/sol)	TYPE OF REQUEST org,dio inor, SAS	SAMPLE TRAFFIC REPORT NUMBER	ORGANICS OR INORGANICS: LAB NAME	DATE SHIPPED	DATA RECEIVED XX out items not requested: VOA	BNA	PEST	TCDD	METALS	CN
	Low	SOL	SAS	5165C-09 TASK								
EQ. BLANK	↓	AQ	↓	↓ -10 ↓								
	↓	SOL	↓	↓ -11 ↓								

SAS REQUEST: (details required)	LAB NAME	SAS REQUEST (itemize)	DATE SHIPPED	DATA REC'D
① Nitrobenzene, metachloronitrobenzene	LANGST	①	1/10/90	rec'd 2-24 JB ✓
	↓	↓	↓	✓
	↓	↓	↓	✓

AR300656

Office of Enforcement

CHAIN OF CUSTODY RECORD

841 Chestnut St.
Philadelphia, Pennsylvania

PROJ. NO. C030411 5303.8

PROJECT NAME SCD

SAMPLERS: (Signature) Jan Spoor

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Nitrobenzene, Monochlorobenzene						REMARks JAS# 5165C-TASK 1 TAG No.	
5165C-01 TASK 1	1/4/90	1410		X	WS-1	1	1						3-1043971	Do MS/MSD
5165C-02 TASK 1	1/4/90	1410		X	WS-2	1	1						3-1043975	
5165C-03 TASK 1	1/4/90	1310		X	WS-3	1	1						3-1043981	
5165C-04 TASK 1	1/4/90	1110		X	WS-4	1	1						3-1043977	
5165C-05 TASK 1	1/5/90	1218		X	WS-5	1	1						3-1100875	
5165C-06 TASK 1	1/5/90	1042		X	WS-6	1	1						3-1100879	
5165C-07 TASK 1	1/5/90	1350		X	WS-7	1	1						3-1100883	
5165C-08 TASK 1	1/4/90	0930		X	EQB-2	1	1						3-1100867	EQ BLANK

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Jan Spoor	1/8/90 1630	Connie A. Sandner			
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks SHIPPED VIA FEDERAL EXPRESS AIRBILL No. 4575150522	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

H300957

03911

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
841 Chestnut St.
Philadelphia, Pennsylvania 19107

CHAIN OF CUSTODY RECORD

PROJ. NO. 03041 5303.8

PROJECT NAME: SCD

SAMPLERS: (Signature) [signature]

TA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Nitrobenzene/Dichlorobenzene						REMARKS JAS No. 51105C-TASK1 TAS No
05C TASK1	1/12/90	1020		X	CB-1 soil	1	1						3-1100901
05C TASK1	1/9/90	1110		X	EQB-3 water	1	1						3-1100912
05C TASK1	1/10/90	1144		X	CB-2 soil	1	1						3-1100920
													Last Sample Received 1/11/90 Case Complete! [illegible]

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
[signature]	1/10/90 1530	Connie L. Sandner 1/11/90 1005			
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks: Shipped via Federal Express AIRBILL No. 4575149532	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field File

AR300658

Revision

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page 1 of 1 EPA SAMPLE SHIPPING LOG FOR ALL SAMPLES SENT THROUGH THE CONTRACT LAB PROGRAM (12/85 version)

CASE# _____ SAS# 5165C-TASK 2 SITE NAME: STANDARD CHLORINE

Site Leader: DAVID SPENCER EPA Project Officer: Bob GUARNI

Phone: (215) 741-4211

QC SAMPLE INFO AND/OR COMMENTS	CONC. (low/med/high)	SAMPLE PHASE (aq/sol)	TYPE OF REQUEST org, dio inor, SAS	SAMPLE TRAFFIC REPORT NUMBER	ORGANICS OR INORGANICS: LAB NAME	DATE SHIPPED	DATA RECEIVED (XX out items not requested): VOA	BNA	PEST	TCDD	METALS	CN
	Low	Sol	SAS	5165C-09 TASK 2								
EQ. BLANK	↓	AQ	↓	↓ -10 ↓								
	↓	Sol	↓	↓ -11 ↓								

SAS REQUEST: (details required)

② Trichloro-thru Hexachloro benzene

LAB NAME	SAS REQUEST (itemize)	DATE SHIPPED	DATA REC'D
			need SAS 2-12-90 ✓
PACE	②	1/10/90	✓
↓	↓	↓	✓

AR300659

Review

TES/VERSAR

JUL 1990

page 1 of 1 EPA SAMPLE SHIPPING LOG FOR ALL SAMPLES SENT THROUGH THE CONTRACT LAB PROGRAM (12/85 version)

CASE# _____ SAS# 5165C-TASK2 SITE NAME: STANDARD CHLORINE

Site Leader: DAVID SPENCER EPA Project Officer: Bob GUARNI

Phone: (215) 741-4211

SAS REQUEST: (details required)
② Trichloro- thru Hexachlorobenzene

QC SAMPLE INFO AND/OR COMMENTS	CONC. (low/med/high)	SAMPLE PHASE (aq/sol)	TYPE OF REQUEST org,dio inor, SAS	SAMPLE TRAFFIC REPORT NUMBER	ORGANICS OR INORGANICS: LAB NAME	DATE SHIPPED	DATA RECEIVED (XX out items not requested): VOA	BNA	PEST	TCDD	METALS	CN	SAS REQUEST: LAB NAME	SAS REQUEST (itemize)	DATE SHIPPED	DATA REC'D
MS/MSD	LOW	SOL	SAS	5165C-01 TASK2									PACE	②	1/8/90	rec'd 2-12-90 JDS
Dup 5165C-01				-02												
				-03												
				-04												
				-05												
				-06												
				-07												
EQB BLANK		AQ		-08												

ARD00660

5165C Task I

SAS 613

U.S. Environmental Protection Agency
CLP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal

☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request: December 15, 1989

E. Site Name: Standard Chlorine of Delaware, Delaware City, Delaware

sent to SMO 12-22-89

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:
Analysis of 10 low concentration soil/sediment samples for nitrobenzene and metachloronitrobenzene using SW-846 extraction method 3550 and SW-846 analysis method 8090 (both methods are attached)

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):
Analysis of 10 low concentration soil/sediment samples for the above includes 8 soil/sediment samples, 1 field duplicate, and 1 equipment blank. The equipment blank will be an aqueous sample.

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund Enforcement: RP RI/FS Oversight

OTGB03NP#6

SAS Approved By:

4. Estimated date(s) of collection: January 2 through February 2, 1990

5. Estimated date(s) and method of shipment: January 2 through February 2, 1990

Federal Express - Overnight delivery

6. Approximate number of days results required after lab receipt of samples:

Analysis must occur within 14 days of sample collection. Data package must be submitted within 45 days of laboratory receipt of the last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):

Test Methods for Evaluating Solid Waste (SW-846), Third Edition, 1986
Methods 3550 (Sonication Extraction) and 8090 (Nitroaromatics and Cyclic Ketones) using GC/FID,

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

See Attachment 1.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

See Attachment 2.

10. Other (use additional sheets or attach supplementary information, as needed):

None.

11. Name of sampling/shipping contact: David A. Basko

Phone: (215) 741-4211

12. Data Requirements

Parameter	Detection Limit	Precision Desired * (+ or − Concentration)
Nitrobenzene	330 ug/kg	+/- 25%
Metachloronitrobenzene	330 ug/kg	+/- 25%

* Advisory limits only, not mandatory (corrective action not required).

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
Duplicates	1/20 or 1/batch	+/- 25% RPD
Method Blanks	1/20 or 1/batch	< 330 ug/kg
Matrix Spike	1/20 or 1/batch	+/- 15% (Nitrobenzene)*
		+/- 30% (Metachloronitrobenzene)
2-Fluorobiphenyl (surrogate)	Every sample	30-115% Recovery*
QC Check Standard	1/20 or 1/batch	85-115% of true value (Nitrobenzene)*
		70-130% of true value (Metachloronitrobenzene)
Continuing Calibration Standard	1/10 or at end of batch	+/- 20% D RF

* Advisory limits only, not mandatory (Corrective action not required).

14. Action Required if Limits are Exceeded

Duplicates: Reanalyze sample/duplicate pair and report both sets of data. (Reanalyze 1 time only.)

Method Blanks: Reanalyze all associated samples after corrective action to reduce blanks containing less than 330.

Continuing Calibration Standard: Perform initial calibration and reanalyze all samples since the last acceptable continuing calibration standard.

15. Request prepared by: David A. Basko

Date: December 15, 1989

16. Request reviewed by: Laurie A. Brown

Date: 12-21-89

C. S. 12/22/89

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Regional representative at the Sample Management Office.

METHOD 3550

SONICATION EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 Method 3550 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 The method is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of $\leq$20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The high concentration method (individual organic components of >20 mg/kg) is much simpler and therefore faster.

1.3 It is highly recommended that the extracts be cleaned up prior to analysis. See Cleanup, Section 4.2.2 of Chapter Four, for applicable methods.

2.0 SUMMARY OF METHOD

2.1 Low concentration method: A 30-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using sonication. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

2.2 High concentration method: A 2-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using sonication. A portion of the extract is removed for cleanup and/or analysis.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for grinding: If the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, it should be processed into a homogeneous sample that meets these requirements. Fisher Mortar Model 155 Grinder, Fisher Scientific Co., Catalogue Number 8-323, or an equivalent brand and model, is recommended for sample processing. This grinder should handle most solid samples, except gummy, [illegible] or oily materials.

ATTACHMENT 1

Standardize instruments according to manufacturer's instructions. Analytical procedures, as described in the attached method, MUST be followed even if the text just indicates that those procedures should be followed. Report all holding times on the data sheets.

The instrumentation must be calibrated daily using five calibration standards and a calibration blank. The calibration standards must contain all target analytes at concentrations which bracket the anticipated range of measurement, and these standards must be prepared fresh daily from the stock solution. One of the calibration standards must be near, but above, the method detection limit. Continuing calibration must be performed by analyzing one calibration standard (containing all target analytes) at the mid-range of the initial calibration curve after each ten samples. The response factor of this continuing calibration standard must be +/- 20 percent of the average response factor of the initial calibration, or else initial calibration must be repeated, and all samples analyzed since the last acceptable continuing calibration standard must be reanalyzed.

All samples must be spiked with 2-fluorobiphenyl as a surrogate compound at a nominal final concentration of 100 ug/kg. All initial and continuing calibration standards must contain 2-fluorobiphenyl at the same concentration as the standard analytes. The average response factor of 2-fluorobiphenyl from the initial calibration must be used to calculate sample surrogate recoveries.

A matrix spike must be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent. The spike concentration must be 1 to 5 times the background sample concentration and must be determined by screening.

A QC check standard must also be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent. This check standard must be prepared from an independent source material of that used to prepare that calibration standards.

A method blank must also be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent.

4.2 Sonication: A horn-type sonicator equipped with a titanium tip should be used. The following sonicator, or an equivalent brand and model, is recommended:

Ultrasonic cell disrupter: Heat Systems - Ultrasonics, Inc., Model W-385 (475 watt) sonicator or equivalent (Power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapered microtip probe.

4.3 Sonabox: Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

4.4 Apparatus for determining percent moisture:

4.4.1 Oven: Drying.

4.4.2 Desiccator.

4.4.3 Crucibles: Porcelain.

4.5 Pasteur glass pipets: Disposable, 1-mL.

4.6 Beakers: 400-mL.

4.7 Vacuum filtration apparatus:

4.7.1 Buchner funnel.

4.7.2 Filter paper: Whatman No. 41 or equivalent.

4.8 Kuderna-Danish (K-D) apparatus:

4.8.1 Concentrator tube: 10-mL graduated (Kontes K-570050-1025 or equivalent).

4.8.2 Evaporator flask: 500-mL (Kontes K-570001-0500 or equivalent).

4.8.3 Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.8.4 Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.9 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.10 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

ATTACHMENT 2

Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, blank results, duplicate results, chain of custody forms, SAS request forms, SAS packing list(s) or traffic report(s), copy of airbill(s), and copies of analyst's logbooks(signed by analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labled with the complete EPA sample number as it appears on chain of custody and CLP paperwork.

The case narrative must document all problems encountered and the subsequent resolutions. List instrumentation and methods employed for analysis. Also, note whether samples were preserved or not and the procedure utilized in preservation. EPA QC reference samples, or equivalent reference samples must be identified as to source and lot number. Documentation of "true" value and associated 95 % confidence limits must be provided for any reference samples used.

4.11 Balance: Top-loading, capable of accurately weighing 0.01 g.

4.12 Vials and caps: 2-mL for GC auto-sampler.

4.13 Glass scintillation vials: At least 20-mL, with screw-cap and Teflon or aluminum foil liner.

4.14 Spatula: Stainless steel or Teflon.

4.15 Drying column: 20-mm I.D. Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.16 Syringe: 5-mL.

5.0 REAGENTS

5.1 Sodium sulfate: Anhydrous and reagent grade, heated at 400°C for 4 hr, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog #73898, or equivalent.

5.2 Extraction solvents: Methylene chloride:acetone (1:1, v:v), methylene chloride, hexane (pesticide quality or equivalent).

5.3 Exchange solvents: Hexane, 2-propanol, cyclohexane, acetonitrile (pesticide quality or equivalent).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Sample handling:

7.1.1 Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.1.2 Waste samples: Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two before extraction. This procedure is for solids only.

7.1.3 Dry waste samples amenable to grinding: Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

7.2 Determination of percent moisture: In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \%\ \text{moisture}$$

7.3 Determination of pH (if required): Transfer 50 g of sample to a 100-mL beaker. Add 50 mL of water and stir for 1 hr. Determine the pH of sample with glass electrode and pH meter while stirring. Discard this portion of sample.

7.4 Extraction method for samples expected to contain low concentrations of organics and pesticides ($\leq$20 mg/kg):

7.4.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample into a 400-mL beaker. Record the weight to the nearest 0.1 g. Non-porous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate using a spatula. The sample should be free-flowing at this point. Add 1 mL of surrogate standards to all samples, spikes, and blanks (see Method 3500 for details on the surrogate standard solution and the matrix spike solution). For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/uL of each base/neutral analyte and 200 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half of the extract is lost due to loading of the GPC column. Immediately add 100 mL of 1:1 methylene chloride:acetone.

7.4.2 Place the bottom surface of the tip of the #207 3/4 in. disruptor horn about 1/2 in. below the surface of the solvent, but above the sediment layer.

7.4.3 Sonicate for 3 min, with output control knob set at 10 and with mode switch on Pulse and percent-duty cycle knob set at 50%. Do NOT use microtip probe.

7.4.4 Decant and filter extracts through Whatman No. 41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

7.4.5 Repeat the extraction two or more times with two additional 100-mL portions of solvent. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with extraction solvent.

7.4.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask.

7.4.7 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 mL of extraction solvent to complete the quantitative transfer.

7.4.8 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.4.9 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip, and re-attach the Snyder column. Concentrate the extract as described in Paragraph 7.4.8, raising the temperature of the water bath, if necessary, to maintain proper distillation.

7.4.10 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Paragraph 7.4.11 or adjusted to 10.0 mL with the solvent last used.

7.4.11 Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5 mL of methylene chloride or exchange solvent through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature, as required, to complete the concentration in 5-10 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the liquid

TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040[a]	as received	2-propanol	hexane	1.0	1.0, 10.0[b]
8060	as received	hexane	hexane	2.0	10.0
8080	as received	hexane	hexane	10.0	10.0
8090	as received	hexane	hexane	2.0	1.0
8100	as received	none	cyclohexane	2.0	1.0
8120	as received	hexane	hexane	2.0	1.0
8140	as received	hexane	hexane	10.0	10.0
8250[a,c]	as received	none	-	-	1.0
8270[a,c]	as received	none	-	-	1.0
8310	as received	acetonitrile	-	-	1.0

[a]To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

[b]Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

[c]The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

reaches an apparent volume of approximately 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 mL of appropriate solvent. Adjust the final volume to the volume required for cleanup or for the determinative method (see Table 1).

7.4.12 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid and mark the level on the vial. Label with the sample number and fraction and store in the dark at 4°C until ready for analysis or cleanup.

7.5 Extraction method for samples expected to contain high concentrations of organics (>20 mg/kg):

7.5.1 Transfer approximately 2 g (record weight to the nearest 0.1 g) of sample to a 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

7.5.2 Add 2 g of anhydrous sodium sulfate to sample in the 20-mL vial and mix well.

7.5.3 Surrogate standards are added to all samples, spikes, and blanks (see Method 3500 for details on the surrogate standard solution and on the matrix spike solution). Add 2.0 mL of surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 2.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 200 ng/uL of each base/neutral analyte and 400 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.5.4 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0 mL considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 1/8-in. tapered microtip ultrasonic probe for 2 min at output control setting 5 and with mode switch on pulse and percent duty cycle of 50%. Extraction solvents are:

1. Nonpolar compounds, i.e., organochlorine pesticides and PCBs: hexane.

2. Extractable priority pollutants: methylene chloride.

7.5.5 Loosely pack disposable Pasteur pipets with 2- to 3-cm Pyrex glass-wool plugs. Filter the extract through the glass wool and collect

5.0 mL in a concentrator tube if further concentration is required. Follow Paragraphs 7.4.6 through 7.4.12 for details on concentration. Normally, the 5.0 mL extract is concentrated to 1.0 mL.

7.5.6 The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subject to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

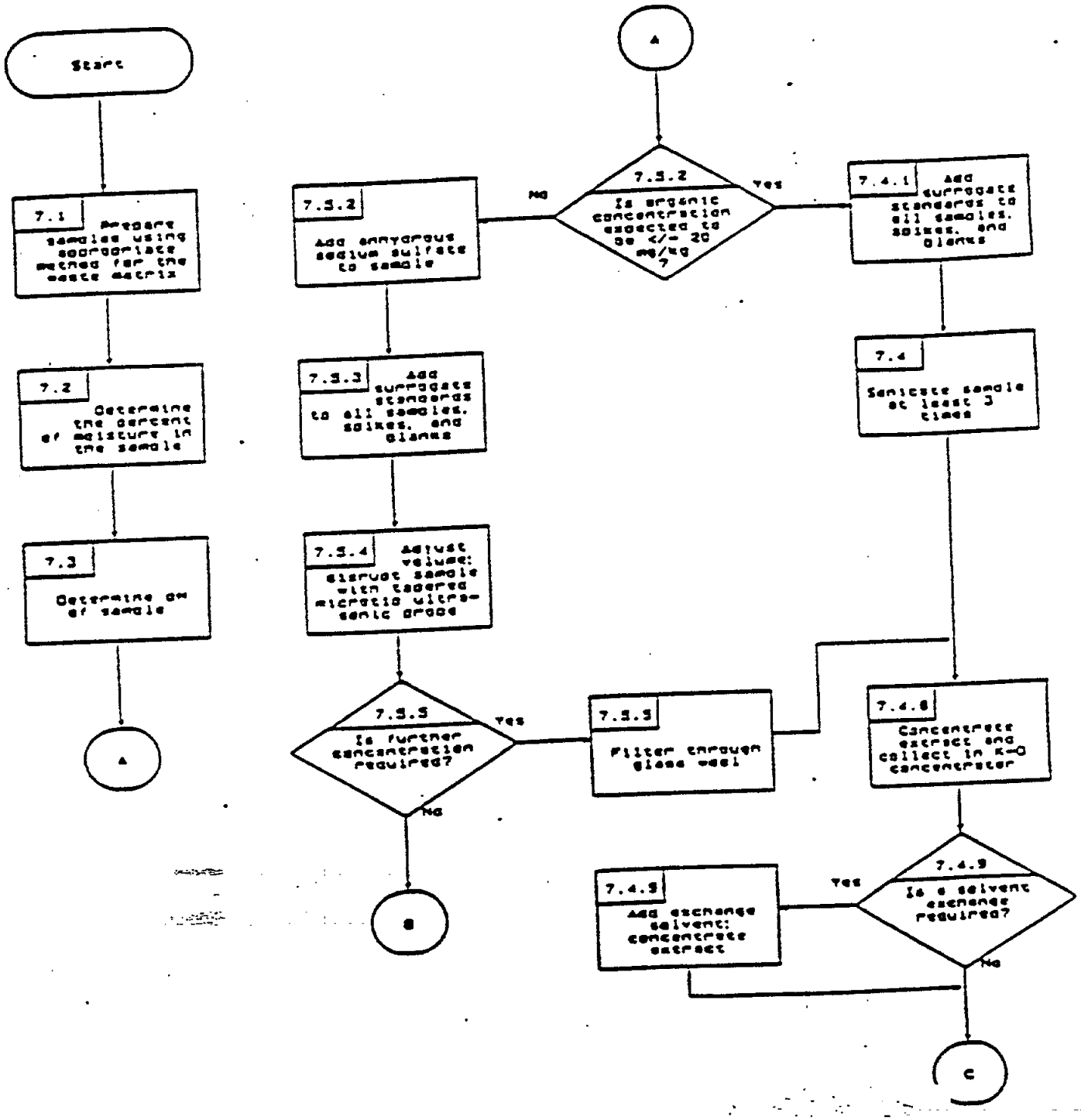
10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

2. U.S. EPA, Interlaboratory Comparison Study: Methods for Volatile and Semi-Volatile Compounds, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, EPA 600/4-84-027, 1984.

METHOD 3550

SONICATION EXTRACTION



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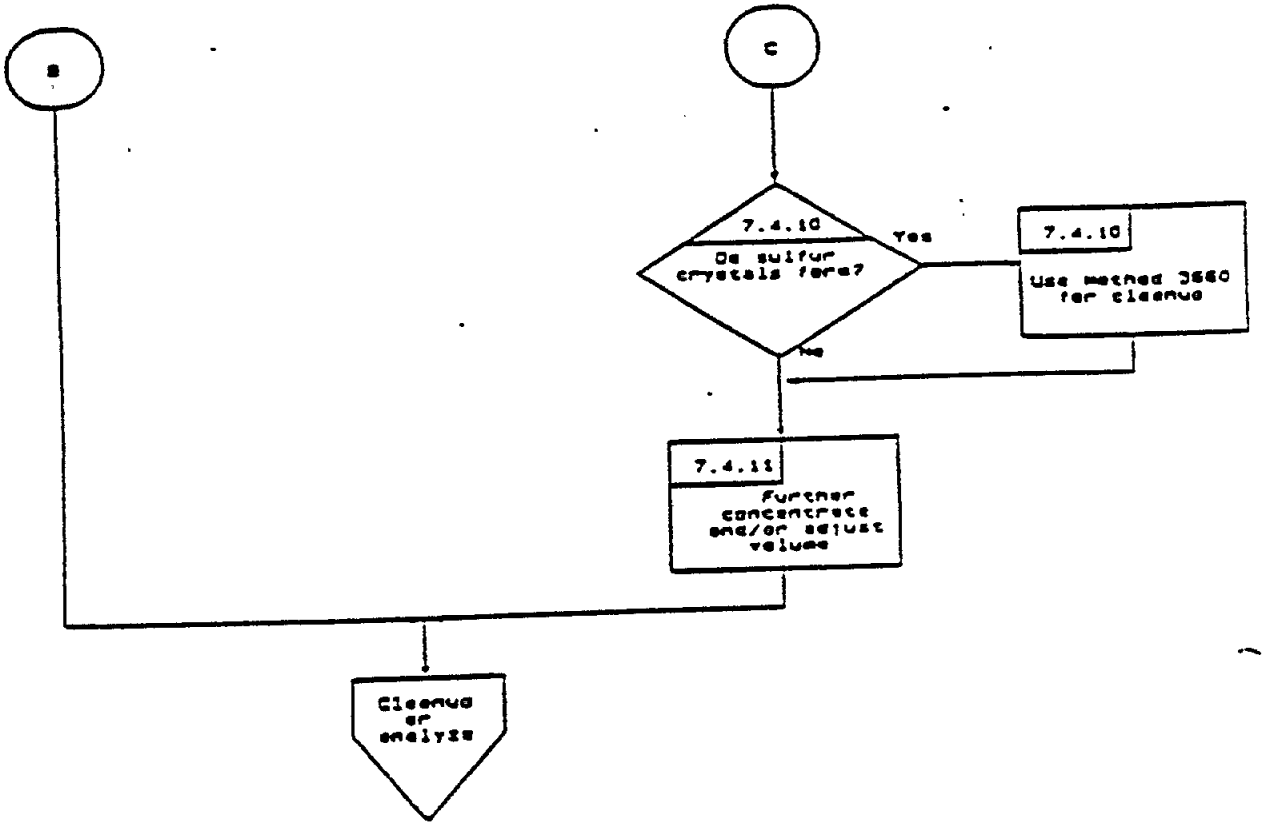
METHOD 3550

SONICATION EXTRACTION
(Continued)



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METHOD 8090

NITROAROMATICS AND CYCLIC KETONES

1.0 SCOPE AND APPLICATION

1.1 Method 8090 is used to determine the concentration of various nitroaromatic and cyclic ketone compounds. Table 1 indicates compounds that may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8090 provides gas chromatographic conditions for the detection of ppb levels of nitroaromatic and cyclic ketone compounds. Prior to use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2- to 5-uL aliquot of the extract is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD) or a flame ionization detector (FID). The dinitrotoluenes are determined using ECD, whereas the other compounds amenable to this method are determined using FID.

2.2 If interferences prevent proper detection of the analytes, the method may also be performed on extracts that have undergone cleanup.

3.0 INTERFERENCES

3.1 Refer to Method 3500, 3600, and 8000.

3.2 Solvents, reagents, glassware, and other sample-processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.3 Interferences coextracted from samples will vary considerably from source to source, depending upon the waste being sampled. Although general cleanup techniques are recommended as part of this method, unique samples may require additional cleanup.

TABLE 1. GAS CHROMATOGRAPHY OF NITROAROMATICS AND ISOPHORONE

Compound	Retention time (min)		Method detection limit (ug/L)	
	Col. 1[a]	Col. 2[b]	ECD	FID
Isophorone	4.49	5.72	15.7	5.7
Nitrobenzene	3.31	4.31	13.7	3.6
2,4-Dinitrotoluene	5.35	6.54	0.02	-
2,6-Dinitrotoluene	3.52	4.75	0.01	-
Dinitrobenzene				
Naphthoquinone				

[a]Column 1: Gas-Chrom Q (80/100 mesh) coated with 1.95% QF-1/1.5% OV-17 packed in a 1.2-m x 2-mm or 4-mm I.D. glass column. A 2-mm I.D. column and nitrogen gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by GC/FID. The column temperature was held isothermal at 85°C. A 4-mm I.D. column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used when determining the dinitrotoluenes by GC/ECD. The column temperature was held isothermal at 145°C.

[b]Column 2: Gas-Chrom Q (80/100 mesh) coated with 3% OV-101 packed in a 3.0-m x 2-mm or 4-mm I.D. glass column. A 2-mm I.D. column and nitrogen carrier gas at 44 mL/min flow rate were used when determining isophorone and nitrobenzene by GC/FID. The column temperature was held isothermal at 100°C. A 4-mm I.D. column and 10% methane/90% argon carrier gas at 44 mL/min flow rate were used to determine the dinitrotoluenes by GC/ECD. The column temperature was held isothermal at 150°C.

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]Multiply the Method Detection Limits in Table [illegible] to determine the PQL for each analyte in the matrix to[illegible]

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections and all required accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is recommended.

4.1.2 Columns:

4.1.2.1 Column 1: 1.2-m x 2- or 4-mm I.D. glass column packed with 1.95% QF-1/1.5% OV-17 on Gas-Chrom Q (80/100 mesh) or equivalent.

4.1.2.2 Column 2: 3.0-m x 2- or 4-mm I.D. glass column packed with 3% OV-101 on Gas-Chrom Q (80/100 mesh) or equivalent.

4.1.3 Detectors: Flame ionization (FID) or electron capture (ECD).

4.2 Kuderna-Danish (K-D) apparatus:

4.2.1 Concentrator tube: 10-mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts

4.2.2 Evaporation flask: 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.

4.2.3 Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.2.4 Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.3 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.4 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.5 Volumetric flasks: 10-, 50-, and 100-mL, ground-glass stopper.

4.6 Microsyringe: 10-uL.

4.7 Syringe: 5-mL.

4.8 Vials: Glass, 2-, 10-, and 20-mL capacity with Teflon-lined screw cap.

5.0 REAGENTS

5.1 Solvents: hexane, acetone (pesticide quality or equivalent.)

5.2 Stock standard solutions:

5.2.1 Prepare stock standard solutions at a concentration of 1.00 ug/uL by dissolving 0.0100 g of assayed reference material in hexane and diluting to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.2.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.3 Calibration standards: Calibration standards at a minimum of five concentration levels are prepared through dilution of the stock standards with hexane. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner if comparison with a check standard indicates a problem.

5.4 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.4.1 Prepare calibration standards at a minimum of five concentration levels for each parameter of interest as described in Paragraph 5.3.

5.4.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with hexane.

5.4.3 Analyze each calibration standard according to Section 7.0.

5.5 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each

sample, standard, and reagent water blank with one or two surrogates (e.g., 2-fluorobiphenyl) recommended to encompass the range of the temperature program used in this method. Method 3500, Section 5.3.1.1, details instructions on the preparation of base/neutral surrogates. Deuterated analogs of analytes should not be used as surrogates for gas chromatographic analysis due to coelution problems.

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a pH between 5 to 9 with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using either Method 3540 or 3550.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. The exchange is performed during the K-D procedures listed in all of the extraction methods. The exchange may be performed in one of two ways, depending on the data requirements. If the detection limits cited in Table 1 must be achieved, the exchange should be performed as described starting in Section 7.1.4. If these detection limits are not necessary, solvent exchange is performed as outlined in Section 7.1.3.

7.1.3 Solvent exchange when detection limits in Table 1 are not required:

7.1.3.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.

7.1.3.2 Momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the macro-Snyder column. Concentrate the extract using 1 mL of hexane to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the appar[illegible] reaches 1 mL, remove the K-D apparatus and allow[illegible] cool for at least 10 min. The extract will be handled differently

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at this point, depending on whether or not cleanup is needed. If cleanup is not required, proceed to Paragraph 7.1.3.3. If cleanup is needed, proceed to Paragraph 7.1.3.4.

7.1.3.3 If cleanup of the extract is not required, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane. A 5-mL syringe is recommended for this operation. Adjust the extract volume to 10.0 mL. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. Proceed with gas chromatographic analysis.

7.1.3.4 If cleanup of the extract is required, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with a minimum amount of hexane. A 5-mL syringe is recommended for this operation. Add a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K-D apparatus on the water bath (80°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.1.3.5 Remove the micro-Snyder column and rinse the flask and its lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the extract volume to 2.0 mL and proceed with Method 3620.

7.1.4 Solvent exchange when detection limits listed in Table 1 must be achieved:

7.1.4.1 Following K-D of the methylene chloride extract to 1 mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.

7.1.4.2 Remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of methylene chloride. A 5-mL syringe is recommended for this operation. Add 1-2 mL of hexane, a clean boiling chip, and attach a two-ball micro-Snyder column. Prewet the column by adding 0.5 mL of hexane to the top. Place the micro-K-D apparatus on the water bath (60-65°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the actively chatter, but the chambers will not flood. apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

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7.1.4.3 Remove the micro-Snyder column and rinse the flask and its lower joint into the concentrator tube with a minimum amount of hexane. The volume of the extract should be adjusted to 1.0 mL if the extract will be analyzed without cleanup. If the extract will require cleanup, adjust the volume to 2.0 mL with hexane. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. Proceed with either gas chromatographic analysis or with cleanup, as necessary.

7.2 Gas chromatography conditions (Recommended): The determination of dinitrotoluenes should be performed using GC/ECD. All other compounds amenable to this method are to be analyzed by GC/FID.

7.2.1 Column 1: Set 10% methane/90% argon carrier gas flow at 44 mL/min flow rate. For a 2-mm I.D. column, set the temperature at 85°C isothermal. For a 4-mm I.D. column, set the temperature at 145°C isothermal.

7.2.2 Column 2: Set 10% methane/90% argon carrier gas flow at 44 mL/min flow rate. For a 2-mm I.D. column, set the temperature at 100°C isothermal. For a 4-mm I.D. column, set the temperature at 150°C isothermal.

7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 The procedure for internal or external standard calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.3.4 If cleanup is performed on the samples, the analyst should process a series of standards through the cleanup procedure and then analyze the samples by GC. This will confirm elution patterns and the absence of interferents from the reagents.

7.4 Gas chromatographic analysis:

7.4.1 Refer to Method 8000. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to injection.

7.4.2 Follow Section 7.6 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence when using FID and after each group of 5 samples in the analysi[illegible] when using ECD.

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7.4.3 An example of a GC/FID chromatogram for nitrobenzene and isophorone is shown in Figure 1. Figure 2 is an example of a GC/ECD chromatogram of the dinitrotoluenes.

7.4.4 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).

7.4.5 Using either the internal or external calibration procedure (Method 8000), determine the identity and quantity of each analyte peak in the sample chromatogram. See Section 7.8 of Method 8000 for calculation equations.

7.4.6 If peak detection and identification are prevented due to interferences, the hexane extract may undergo cleanup using Method 3620.

7.5 Cleanup:

7.5.1 Proceed with Method 3620, using the 2-mL hexane extracts obtained from either Paragraph 7.1.3.5 or 7.1.4.3.

7.5.2 Following cleanup, the extracts should be analyzed by GC, as described in the previous paragraphs and in Method 8000.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

8.2 Procedures to check the GC system operation are found in Method 8000, Section 8.6.

8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest in acetone at a concentration of 20 ug/mL for each dinitrotoluene and 100 ug/mL for isophorone and nitrobenzene.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000, Section 8.10).

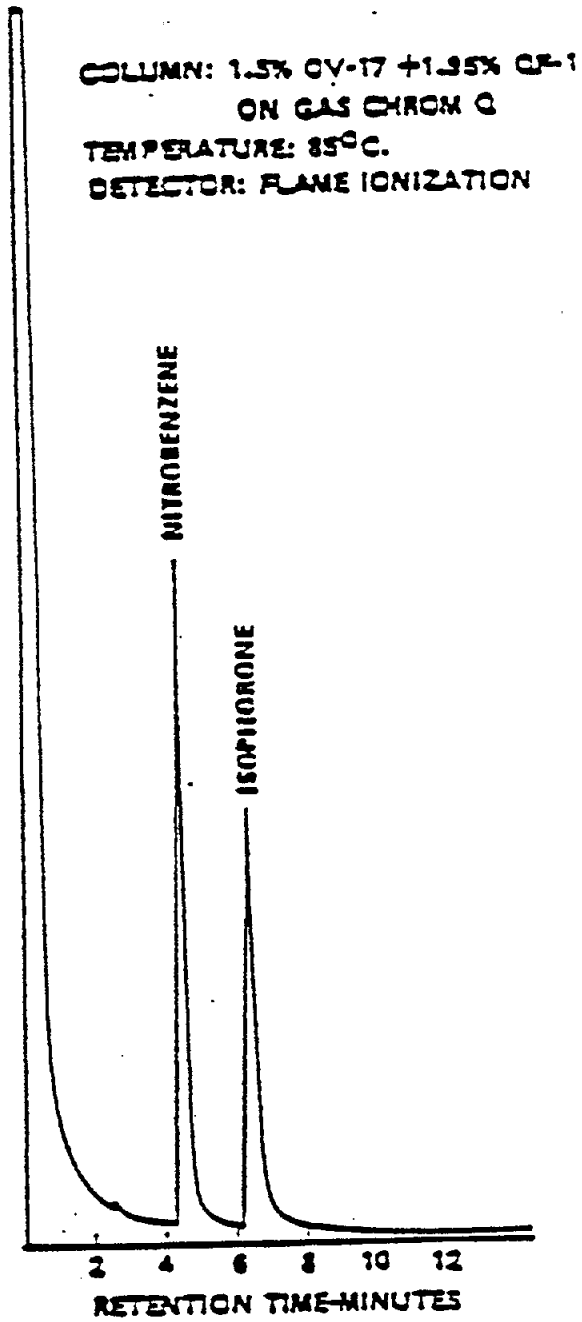

Figure 1. Gas chromatogram of nitrobenzene and isophorone.

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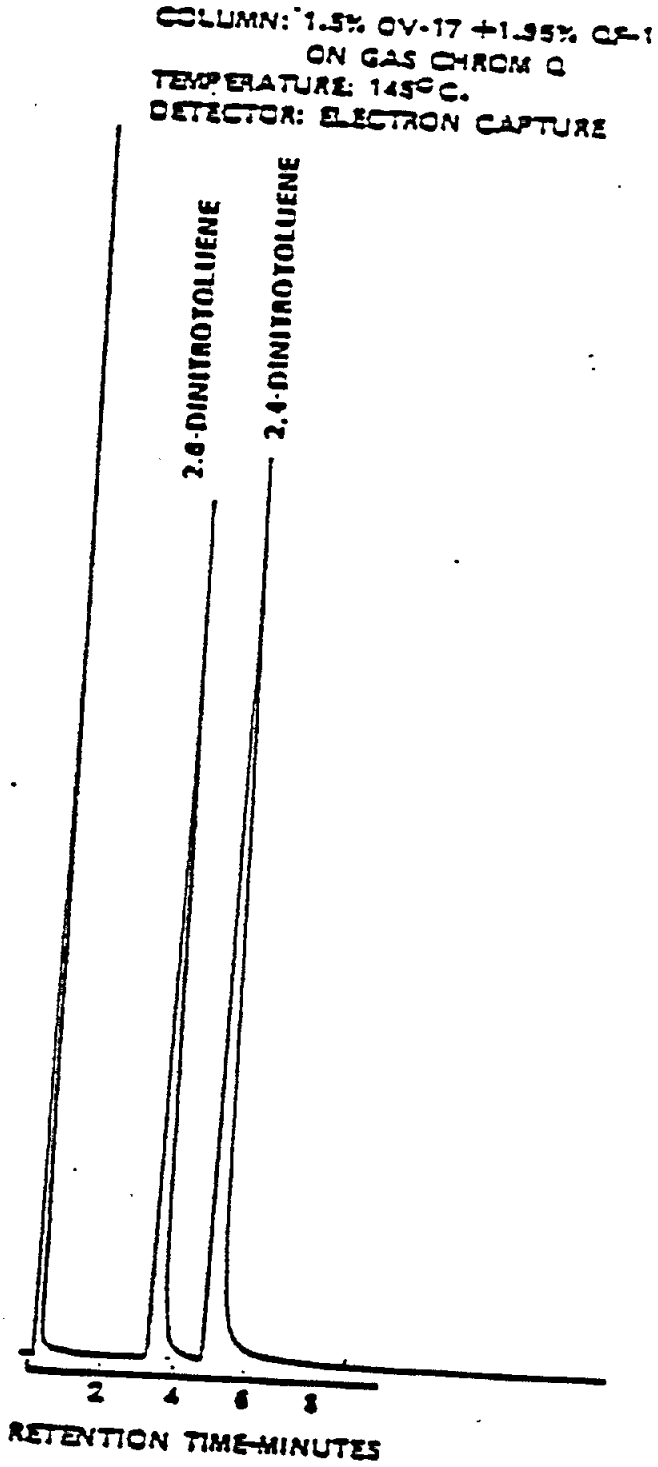

Figure 2. Gas chromatogram of dinitrotoluenes.

8.3.1 If recovery is not within limits, the following is required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The method was tested by 18 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 515 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, and calibration procedures used.

10.0 REFERENCES

1. "Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 4 - Nitroaromatics and Isophorone," Report for EPA Contract 68-03-2624 (in preparation).

2. "Determination of Nitroaromatics and Isophorone in Industrial and Municipal Wastewaters," EPA-600/4-82-024, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, June 1982.

3. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.

4. "EPA Method Validation Study 19, Method 609 (Nitroaromatics and Isophorone)," Report for EPA Contract 68-03-2624 (in preparation).

5. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

6. Provost, L.P. and R.S. Elder, "Interpretation of Percent Re American Laboratory, 15, pp. 58-63, 1983.

TABLE 3. QC ACCEPTANCE CRITERIA[a]

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
2,4-Dinitrotoluene	20	5.1	3.6-22.8	6-125
2,6-Dinitrotoluene	20	4.8	3.8-23.0	8-125
Isophorone	100	32.3	8.0-100.0	D-117
Nitrobenzene	100	33.3	25.7-100.0	6-118

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 609. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

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TABLE 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION

Parameter	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S' (ug/L)
2,4-Dinitrotoluene	0.65C+0.22	0.20$\bar{X}$+0.08	0.37$\bar{X}$-0.07
2,4-Dinitrotoluene	0.66C+0.20	0.19$\bar{X}$+0.06	0.36$\bar{X}$-0.00
Isophorene	0.49C+2.93	0.28$\bar{X}$+2.77	0.46$\bar{X}$+0.31
Nitrobenzene	0.60C+2.00	0.25$\bar{X}$+2.53	0.37$\bar{X}$-0.78

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{X}$, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in ug/L.

C = True value for the concentration, in ug/L.

$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

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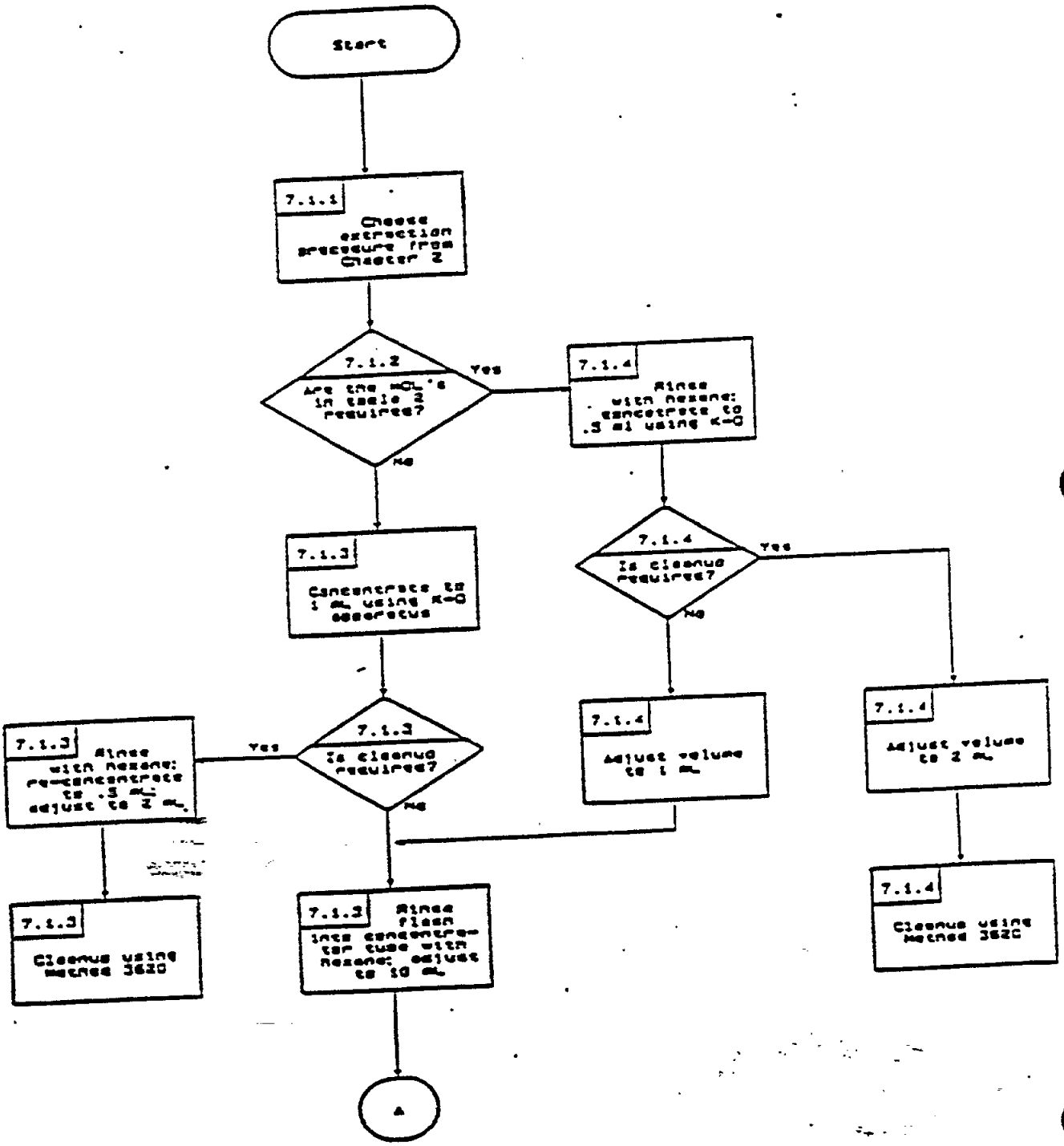
METHOD 8090

NITROAROMATICS AND CYCLIC KETONES



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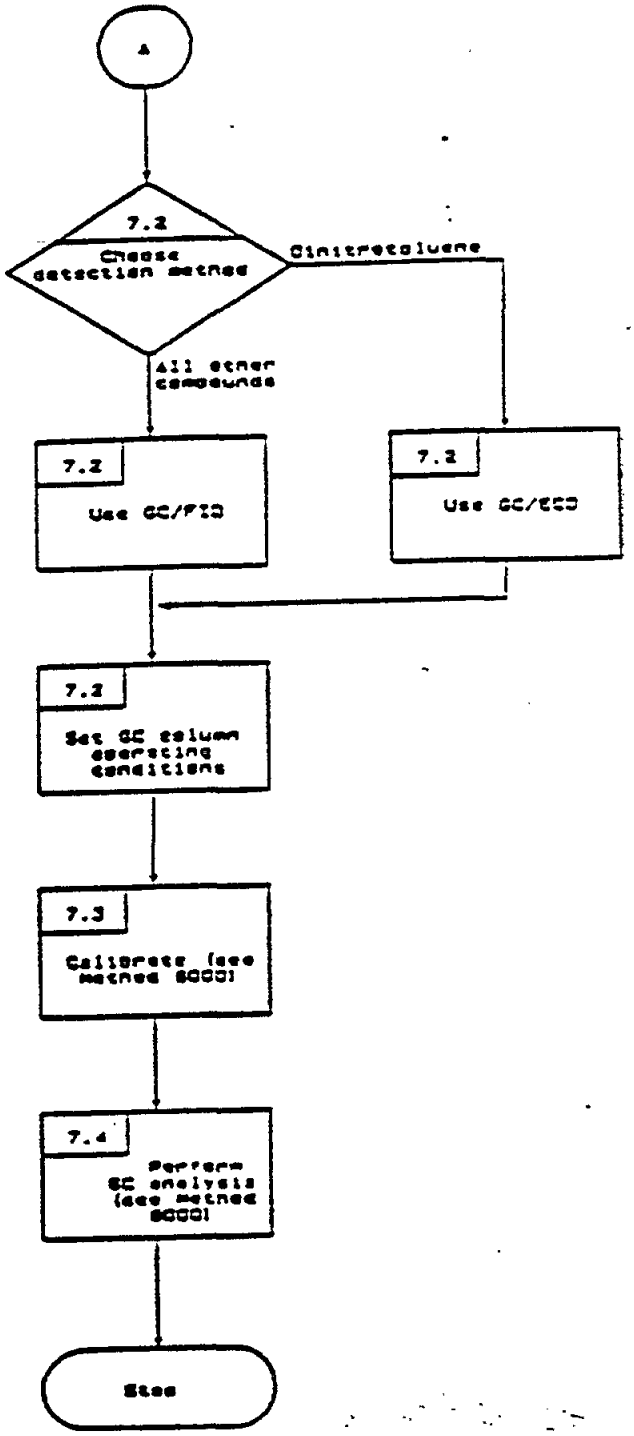
A
7.2
Choose detection method
Dinitrotoluene
All other compounds
7.2
Use GC/FID
7.2
Use GC/ECD
7.2
Set GC column operating conditions
7.3
Calibrate (see Method 8000)
7.4
Perform GC analysis (see Method 8000)
Stop

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CASE NARRATIVE

FOR

U.S. EPA SAS 5165-C-01 TASK I

PROJECT # 500109.503

SEMI-VOLATILES

SAS 5165-Task I was received in two shipments. The first was received on 1/9/90, and it consisted of seven soil samples and one water sample. The second shipment was received on 1/11/90, and it consisted of two soil samples and one water sample. These samples were assigned a PACE project number (500109.503). Each sample was assigned a unique PACE sample number for laboratory tracking purposes. After these samples were logged in they were immediately refrigerated in the PACE refrigerator R4. The samples remained there until sample extraction was started.

These samples were submitted for the analysis of nitrobenzene and metachloronitrobenzene. This analysis was to be performed utilizing EPA Method 8090 with the flame ionization detector. A surrogate standard of 2-fluorobiphenyl was to be added to all standards and samples. Standards for this analysis were obtained by PACE from reputable vendors. These vendors were the EPA Repository, Kodak, and Chem Service. Stock standards were prepared on 1/10/90. From these stock standards, working standards were prepared, as were the surrogate spiking solution, and the matrix spike solutions. These standards were evaluated on the GC before the samples were extracted.

The water samples were extracted on 1/12/90. The methodology for the water extraction was EPA Method 3510. A reagent blank, and a QC check sample were extracted along with the two water samples submitted for analysis. One liter of water was extracted, and concentrated to a ten milliliter final volume. Sample 5165-C-08-Task I contained only 987 milliliters of sample. This volume was used for the 3510 extraction.

The soil samples were extracted on 1/13/90. The methodology used for the soil extractions was EPA Method 3550, sonication of solid samples. A reagent blank, a sample duplicate, and a matrix spike were extracted along with the other soil samples. After reviewing the chain of custody form, it was noticed that a matrix spike duplicate was to be analyzed. The matrix spike duplicate, along with another soil reagent blank was extracted on 1/15/90. Sample concentration was performed using the K/D apparatus, followed by a micro-Snyder column, as is specified in EPA Method 8090. A moisture determination was performed simultaneously with the soil sample extractions. Thirty grams of soil was extracted, and concentrated to a final volume of two milliliters.

Gas chromatographic analysis was performed with a Perkin Elmer Sigma 2000 gas chromatograph, and quantitation was performed with a flame ionization detector. The GC column used for the primary analysis was a 1.5% SP-2250 & 1.95% SP-2401 on 100/120 Supelcoport. The column dimensions were 6 foot by 4mm I.D., glass. The carrier gas was helium, and the flow was 50 ml/minute. The GC oven was set at 110 degrees Centigrade for twenty minutes. For the soil samples, a temperature program was employed to ensure the passage of heavier components through the column. This program was not started until after 2-fluorobiphenyl (the last peak of interest) had eluted from the column.

Two primary analyses were required for this project. The first was the primary analysis for the water samples. This analysis was performed on 1/15/90. The second was the primary analysis for the soils. This analysis was performed from 1/16/90 to 1/18/90. Daily five point calibration were ran during each twenty-four hour period included in the run. A calibration standard was analyzed after ten sample injections. This standard was the mid-level standard. In all instances, the initial five point calibration was used for quantitation. Other daily calibrations were compared to the initial, and if the average calibration factor was within twenty percent difference of the initial, the initial was used for quantitation. There were no problems with the water primary analysis. The two water samples submitted for analysis did not require confirmation.

The primary analyses of the soil samples were not so smooth. Many of the samples showed positive hits for nitrobenzene and metachloronitrobenzene. Some of these hits were extremely high and required dilutions. These samples included: 5165-C-04, 5165-C-05, 5165-C-07, 5165-C-09, and 5165-C-11. The primary analysis were within the retention time windows developed for that day. Another problem with the primary analysis was that the amount of 2-fluorobiphenyl in some of the samples was 5 to 50 times higher than the amount added. This could be explained by an interfering peak. Sample cleanup was considered at this point, but it was not done. There were two reasons why cleanup was not done. One was that if there were high amounts of nitrobenzene in the samples, the surrogate data would be useless due to sample dilution. The second reason was that a fourteen day analysis time had been specified in the contract agreement. Most of the samples had been collected on 1/4/90. The soil primary run was not complete until 1/18/90, fourteen days from sample collection for most of the samples. At this point it was decided that sample cleanup would not be done. It was decided to go ahead and confirm the samples. The primary analysis of all samples was completed within fourteen days of the sample collection date.

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The confirmation analysis was completed using the same GC (Perkin Elmer Sigma 2000). The GC column used for the confirmation run was a 3% SP-2100 on 100/120 Supelcoport. The column dimensions were 6 foot by 2mm I.D.. The carrier was helium, and the flow was 30 ml/minute. The oven temperature was 115 degrees Centigrade, and it was held isothermal for twenty-two minutes. A temperature program was included for the analysis of the soils to eliminate the chance of heavier components interfering with the next chromatograph. Two confirmation runs were required for the samples. The first confirm run included the water samples, and some of the cleaner soil samples. The second confirm analysis was for the soils with high amounts of nitrobenzene. Most of the samples did not confirm for nitrobenzene.

The samples which had high peaks in the primary, also had similiar peaks in the confirm analysis. A comparison of the primary analyses to the confirm analysis revealed that the large peaks found in the primary analyses were also in the confirm analysis. The retention times were different then those of the components of interest. Some of the samples had to be analyzed at a 1/10 dilution. This was due to the high amounts of interferences found in the samples. Two samples were positive for nitrobenzene, sample 5165-C-05, and 5165-C-09. The retention time window had to be opened for 5165-C-05, but only by 0.004 minutes. Sample 5165-C-07 showed a positive value for nitrobenzene, but it was falsely high due to a bunching factor. This sample is a less than, and is reported as such.

Metachloronitrobenzene is a problem component in this analysis. Most of the samples showed positive for it during the primary analysis. However, the confirmation for metachloronitrobenzene did not agree with the primary analysis. It appears as if the primary analysis had an interference, and that this peak shifted away from metachloronitrobenzene in the confirmation analysis. Another peak took it's place in the confirmation analysis, but it eluted before metachloronitrobenzene. This peak is just outside the retention time window for metachloronitrobenzene in the confirm run. The interference peak is large, and interferes with the identification of metachloronitrobenzene. Samples 5165-C-04, 5165-C-05, 5165-C-06, and 5165-C-07 are all affected by this interference. The PQL for these samples was fiqured by calculating the amount found in the primary analysis, and adjusting the PQL slightly above this value, assuming that metachloronitrobenzene was the peak in question. The contaminant in the primary is of a lower concentration than the contaminant in the confirm analysis. Once again, a cleanup might have solved this problem, but there was no time to conduct a cleanup, and meet the fourteen day analysis requirement stated in the contract. Another factor was the primary analysis indicated high levels of nitrobenzene.

No surrogate data was available for a number of the soil samples. This was because a peak interfered with 2-fluorobiphenyl in the primary and in the confirm analytical runs. These were noted on the Surrogate Recovery Form. I might add that the surrogate spike level for this contract was extremely low compared to other methods. The value of the surrogate spike was three times lower than the CRQL. This low surrogate spike forced the elution of nitrobenzene to occur on the solvent front. Also included in this report were the Method Blank Summary, the MS/MSD Summary, and the results of the QC Check sample required for this contract.

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"I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hard copy data package has been authorized by the laboratory manager or his designee as verified by the following signature."

Don Wright

Don Wright
Project Officer
February 26, 1990

DCW14/et

WESTON

Appendix H

Support Documentation - Task II

External Calib Check

Paco Laboratories, Inc.

SAS #: 5155-C

Instrument ID: A

GC Column ID: DB1701

Dates of Analyses: 01-23-90 to 01-24-90

compound	Average Initial Calib Factor:	Calib Factor Reference Std	%D
123 trichlorobenzene	334.8	331.5	0.8%
124 trichlorobenzene	347.0	347.0	0%
135 trichlorobenzene	441.6	451.6	2.2%
1235 tetrachlorobenzene	821.9	525.3	44%
pentachlorobenzene	1037.4	1214.2	14%
1234 tetrachlorobenzene	551.5	924.1	51%
hexachlorobenzene	1574.4	1431.4	9.5%

External Calib Check

Paco Laboratories, Inc.

SAS #: 5165-C

Instrument ID: A

GC Column ID: DB210

Dates of Analyses: 02-08-90 to 02-08-90

compound	Average Initial Calib Factor:	Calib Factor Reference Std.	%D
123 trichlorobenzene	558.1	473.5	16%
124 trichlorobenzene	449.3	293.0	41.9%
135 trichlorobenzene	793.1	653.9	19.2%
1235 tetrachlorobenzene	1602.7	692.8	79%
pentachlorobenzene	2262.7	1819.1	22%
1234 tetrachlorobenzene	926.0	877.7	5.6%
hexachlorobenzene	3854.5	2231.9	53%

Standards Summary

Pace Laboratories, Inc. SAS # 5165-C

Instrument ID: A GC Column ID: [illegible]

	Date(s) of Analysis	From [illegible]			Date	[illegible]		
		To [illegible]						
	Time(s) of Analysis	From [illegible]			Time	[illegible]		
		To [illegible]						
compound	RT	RT Window From	RT Window To	Calib. Factor	RT	Calib. Factor	QNT Y/N	%D
123 trichlorobenzene	[illegible]	[illegible]	[illegible]	553.1	13.91	641.0	N	14.9%
124 trichlorobenzene	[illegible]	[illegible]	[illegible]	4483	16.80	528.9	N	18.0%
135 trichlorobenzene	[illegible]	[illegible]	[illegible]	293.1	17.62	290.2	N	12.2%
2-chloronaphthalene	[illegible]	23.34	23.45	24.25	23.39	27.94	N	15.2%
1235 tetrachlorobenzene	[illegible]	19.57	[illegible]	[illegible]	19.91	[illegible]	N	11.3%
pentachlorobenzene	24.55	24.51	24.94	[illegible]	24.85	[illegible]	N	14.6%
1234 tetrachlorobenzene	21.88	21.83	21.43	9.76.0	21.86	1135.0	N	22.7%
hexachlorobenzene	[illegible]	29.01	29.17	3854.5	29.05	4721.50	N	23.6%

2F

SOIL PESTICIDE SURROGATE RECOVERY

Lab Name: Pace Laboratories, Inc Contract: 68-W8-0019

Lab Code: PACE Case No.: _____ SAS No.: 5165-C SDG No.: _____

Level: (low/med) low

		EPA SAMPLE NO.	S1 (2CN) #	OTHER
5165C-01	01	WS-7	93.8%	
5165C-05	02	WS-5	45.6%	
5165C-06	03	WS-6	83.9%	
5165C-07	04	WS-7	67.9%	
5165C-09	05	CB-1	47.0	
5165C-09 Dup.	06	CB-1 Dup.	22.4 *	
5165C-11	07	CB-2	60.9	
5165C-11MS	08	CB-2 MS	16.3 *	
5165C-11MSD	09	CB-2 MSD	53.9	
	10	BLK 2	92.3	
5165C-02	11	WS-3	81.2	
5165C-03	12	WS-3	75.3	
5165C-04	13	WS-4	70.4	
	14			
	15			
	16			
	17			
	18			
	19			
	20			
	21			
	22			
	23			
	24			
	25			
	26			
	27			
	28			
	29			
	30			

S1 (2CN) = 2-CHLORONAPHTHALENE 30-115 ①

Column to be used to flag recovery values

* Values outside of QC limits

D Surrogates diluted out

① QC limit which was listed in 2-fluorobiphenyl in the SAS. 2-Chloronaphthalene used instead. See Case Narrative.

A R Gm 2-09-90

AR300698

FORM II PEST-2 1/87 Rev.

3F

SOIL PESTICIDE MATRIX SPIKE/MATRIX SPIKE DUPLICATE RECOVERY

Lab Name: [illegible] Contract: [illegible]

Lab Code: [illegible] Case No.: _____ SAS No.: 5165-C SDG No.: _____

Matrix Spike - EPA Sample No.: 51656-11 (cb 2) Level: (low/med) _____

COMPOUND	SPIKE ADDED (ug/Kg)	SAMPLE CONCENTRATION (ug/Kg)	MS CONCENTRATION (ug/Kg)	MS % REC #	QC LIMITS REC.
123 trichlorobenzene	171 ug/Kg	2300 ug/Kg	2900 ug/Kg		46-127
124 trichlorobenzene	174 ug/Kg	[illegible]	[illegible] ug/Kg		35-130
135 trichlorobenzene	173 ug/Kg	[illegible]	[illegible] ug/Kg		34-132
1235 tetrachlorobenzene	174 ug/Kg	5,300 ug/Kg	[illegible]		31-134
pentachlorobenzene	174 ug/Kg	9,400 ug/Kg	2400 ug/Kg		42-139
1234 tetrachlorobenzene	173 ug/Kg	54,000 ug/Kg	60,000 ug/Kg		23-134
hexachlorobenzene	21.4 ug/Kg	1000 ug/Kg	340 ug/Kg		

① Should be 70% - 130% as stated in scope of work. ARC 2-8-90

COMPOUND	SPIKE ADDED (ug/Kg)	MSD CONCENTRATION (ug/Kg)	MSD % REC #	% RPD #	QC LIMITS RPD	QC LIMITS REC.
123 trichlorobenzene	171 ug/Kg	3300 ug/Kg			20 ~~53~~	46-127
124 trichlorobenzene	174 ug/Kg	[illegible]			20 ~~71~~	35-130
135 trichlorobenzene	173 ug/Kg	30,000 ug/Kg			20 ~~43~~	34-132
1235 tetrachlorobenzene	174 ug/Kg	3,000 ug/Kg			20 ~~38~~	31-134
pentachlorobenzene	174 ug/Kg	4000 ug/Kg			25 ~~45~~	42-139
1234 tetrachlorobenzene	173 ug/Kg	65,000 ug/Kg			25 ~~50~~	23-134
hexachlorobenzene	21.4 ug/Kg	820 ug/Kg				

ARC 2-8-90

Column to be used to flag recovery and RPD values with an asterisk

* Values outside of QC limits

① Should be 70%-130% stated in scope of work. ARC 2-8-90

RPD: _____ out of _____ outside limits
Spike Recovery: _____ out of _____ outside limits

COMMENTS: recoveries were not calculated since the amounts spiked were insignificant when compared to the amounts already in

AR300699

FORM III PEST-2 1/87 Rev.

Pace Laboratories, Inc.

SAS # 5165-C

DUPLICATE SUMMARY

compound	Concentratio Units ug/Kg cb-1 (5165C-09)	Concentratio Units ug/Kg cb-1 DUPLICATE	RPD
123 trichlorobenzene	<360 ug/kg	<360 ug/kg	~~1.4~~
124 trichlorobenzene	730,000 ug/kg	730,000 ug/kg	1.4%
135 trichlorobenzene	21,000 ug/kg	24,000 ug/kg	13%
1235 tetrachlorobenzene	55,000 ug/kg	53,000 ug/kg	3.7%
pentachlorobenzene	3000 ug/kg	2400 ug/kg	12.5%
1234 tetrachlorobenzene	85,000 ug/kg	81,000 ug/kg	4.9%
hexachlorobenzene	<360 ug/kg	<360 ug/kg	

EPA Sample # PBLK I

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Water

Lab Sample ID MBLK H2O 01/11/90

Sample wt/(vol): 1000ml

Date Received

Level: Low

Date Extracted 01/11/90

% Moisture:

Dry Weight

Extraction Type Sep F

PH 2.5

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/l	Final Extract Volume
123 trichlorobenzene	<9.9 ug/l	10ml
124 trichlorobenzene	<9.9 ug/l	10ml
135 trichlorobenzene	<9.9 ug/l	10ml
1235 tetrachlorobenzene	<9.9 ug/l	10ml
pentachlorobenzene	<9.9 ug/l	10ml
1234 tetrachlorobenzene	<9.9 ug/l	10ml
hexachlorobenzene	<9.9 ug/l	10ml

EPA Sample # PBLK 2

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID MBLK Soil 011616L

Sample wt/vol: 29.94g

Date Received

Level: Low

Date Extracted 011616L

% Moisture:

Dry Weight

Extraction Type Sxe.

PH 10.2

Instrument ID: 2

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	<330ug/Kg	10ml
124 trichlorobenzene	<330ug/Kg	10ml
135 trichlorobenzene	<330ug/Kg	10ml
1235 tetrachlorobenzene	<330ug/Kg	10ml
pentachlorobenzene	<330ug/Kg	10ml
1234 tetrachlorobenzene	<330ug/Kg	10ml
hexachlorobenzene	<330ug/Kg	10ml

5165C-MS
EPA Sample #
GB-2-MS

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID 5140 3MS

Sample wt/vol: 30.14

Date Received [illegible]

Level: Low

Date Extracted [illegible]

% Moisture: 3.8%

Dry Weight 27.5g

Extraction Type Sonic

PH 7.6

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	2800 ug/Kg	3000 mL
124 trichlorobenzene	980,000 ug/Kg	120,000 mL
135 trichlorobenzene	110,000 ug/Kg	12,000 mL
1235 tetrachlorobenzene	10000 ug/Kg	3000 mL
pentachlorobenzene	3400 ug/Kg	3000 mL
1234 tetrachlorobenzene	50,000 ug/Kg	12,000 mL
hexachlorobenzene	740 ug/Kg	3000 mL

EPA Sample #
51654-11m30
CB-2m30

Data Sheet

Pace Laboratories, Inc.

SAS # 5165-C

Matrix: Soil

Lab Sample ID 01493m30

Sample wt/vol: 19.94

Date Received 6/11/90

Level: low

Date Extracted 6/14/90

% Moisture: 8.8%

Dry Weight 17.3g

Extraction Type Sonc.

PH 7.6

Instrument ID: A

GC Column ID: DB1701

compound	Concentratio Units (ug/L or ug/Kg) ug/Kg	Final Extract Volume
123 trichlorobenzene	3300 ug/Kg	3000 ml
124 trichlorobenzene	110,000 ug/Kg	120,000 ml
135 trichlorobenzene	130,000 ug/Kg	12,000 ml
1235 tetrachlorobenzene	13,000 ug/Kg	3000 ml
pentachlorobenzene	4900 ug/Kg	3000 ml
1234 tetrachlorobenzene	65,000 ug/Kg	12,000 ml
hexachlorobenzene	930 ug/Kg	3000 ml

Pace Laboratories, Inc.

PJS 01-22-90 ~~01-22-90~~ 01-22-90 SAS 5165-C Standards dilution

SAS Standard dilutions

	1,2,3 Trichlorobenzene	1,2,4 Trichlorobenzene	1,3,5 Trichlorobenzene	1,2,3,4 Tetrachlorobenzene	1,2,3,5 Tetrachlorobenzene	Pentachlorobenzene	Hexach.
stock conc	7.91 ug/ml	8.01 ug/ml	8.00 ug/ml	8.00 ug/ml	8.05 ug/ml	8.05 ug/ml	0.989 ~~7.91~~
Aliquot std #1	12.5 ul						
Aliquot std #2	25 ul						
Aliquot std #3	50 ul						
Aliquot std #4	100 ul						
Aliquot std #5	200 ul						
~~Surrogate~~ Aliquot 2-chlorophenol	0.4 ml						
Volume	4.0 ml						
conc std #1	24.7 ug/l	25.0 ug/l	25.0 ug/l	25.0 ug/l	25.2 ug/l	25.2 ug/l	3.09
conc std #2	49.4 ug/l	50.1 ug/l	50.0 ug/l	50.0 ug/l	50.3 ug/l	50.3 ug/l	6.18
conc std #3	98.9 ug/l	100 ug/l	100 ug/l	100 ug/l	101 ug/l	101 ug/l	12.4
conc std #4	198 ug/l	200 ug/l	200 ug/l	200 ug/l	201 ug/l	201 ug/l	24.7
conc std #5	396 ug/l	401 ug/l	400 ug/l	400 ug/l	403 ug/l	403 ug/l	49.5
~~conc Surrogate~~							

concentration of the surrogate in all samples is 3000 ug/l

SAS QC check standards

Supelco mix	conc	Aliquot	dilution	conc
1,2,4 trichlorobenzene	20 ppm	12.5 ul	1 ml	25 ug/l
1,2,3,4 tetrachlorobenzene				
1,2,3,5 tetrachlorobenzene				
Pentachlorobenzene				
hexachlorobenzene				

1,3,5 Trichlorobenzene

14630 ug/l x .017 ml ÷ 10 ml/lx = ~~37~~ 24.7 ug/ml 10 ul 24.7 ug/l

1,2,3 Trichlorobenzene =

5000 ug/ml x .005 ml ÷ 10 ml/lx = 25.0 ug/ml 10 ul 25.0 ug/l

MC 01-22-90

01-23-90 SAS 5165-C Standards dilution

made up calibration standards as in 01-22-90 for sample analysis

AR300705

PJS-01-23-90

Pace Laboratories, Inc.

01-24-90 SAS 5165-C

Sample dilution

CB 01491 CB-1 10ml extract 0.02ml to 10ml = 500ml extract

10ml extract 0.005ml to 1.5ml = 3000ml extract

01491 Dup CB-1 Dup 10ml extract 0.02ml to 10ml = 500ml extract

10ml extract 0.005ml to 1.5ml = 3000ml extract

01493 CB-2 10ml extract 0.01ml to 1.5ml = ~~750ml~~ 1500 PGS 01-24-90 extract

01493MS CB-2MS 10ml extract 0.005ml to 1.5ml = 3000ml extract

01493MSD CB-2MSD 10ml extract 0.005ml to 1.5ml = 3000ml extract

00960 WS-13 10ml extract 0.01ml to 1.0ml = 1000ml extract

10ml extract 0.2ml to 1.0ml = 500ml extract

00961 WS-4 10ml extract 0.01ml to 1.0ml = 1000ml extract

10ml extract 0.2ml to 1.0ml = 500ml extract

WS-1 00958 10ml extract 0.2ml to 1.0ml = 500ml extract

WS-5 00962 10ml extract 0.01ml to 1.0ml = 1000ml extract

WS-3 00960 1000ml extract 0.1ml to 10ml = 10,000ml extract

made up calibration standards for ~~last~~ sample analysis as on 01-22-90. PGS 01-24-90 ~~01-26-90~~

01-26-90 SAS 5165-C

the run began on 01-24-90 did not meet standard requirements.

made up the following dilutions:

CB-2 01493 1500ml extract 0.1ml to 10ml = 15000ml extract

CB-2MS 01493MS 2000ml extract 0.2ml to 0.8ml = 12,000 PGS 01-26-90 750ml extract

CB-2MSD 01493MSD 2000ml extract 0.2ml to 0.8ml = 12,000 PGS 01-26-90 ~~750~~ ml extract

WS-5 00962 1000ml extract 0.1ml to 1.0ml = 10,000ml extract

WS-6 00963 10ml extract 0.1ml to 1.0ml = 1000ml extract

~~[illegible]~~

Ran these dilutions along with some of the dilutions from 01-24-90 on the instrument + made up calibration standards as on 01-22-90. PGS 01/26/90

01-28-90 SAS 5165-C

made up the following dilutions:

CB-1 01491 PGS 01/28/90 3000ml extract 0.01ml to 10ml = 300,000 ml ex

PGS 01/28/90 3000ml extract 0.5ml to 10ml = 6000ml ml extract

CB-1 dup 01491 dup ~~01493~~ = same dilutions as above

CB-2 01493 1500ml extract 0.2ml to 0.8ml = 6,000 ml ~~6000ml~~ extract PGS 01/28/90

CB-2MS 01493MS 1200ml extract 0.1ml to 10ml = 12000ml

CB-2MSD same dilution as above.

00960 [illegible] 0.5ml to 10ml = [illegible] extract

AR300706

Pace Laboratories, Inc.

01-29-90 SAS ~~[illegible]~~ pgs 01/28/90 5165-C cont.

WS-5 00962 10,000 ml extract 0.5 ml to 1.0 ml = 20,000 ml extract.

Diluted up calibration standards as in 01-22-90. Ran dilutions on the instrument. pgs 01/28/90

01-29 SAS 5165-C

made up the following dilutions:

WS-1 00958 10 ml extract 0.2 ml to 10 ml = 50 ml extract
WS-3 00960 10 ml extract 0.2 ml to 10 ml = 50 ml extract
WS-4 00961 10 ml extract 0.2 ml to 10 ml = 50 ml extract
CB-2MS 01493MS 10 ml extract ~~0.2 ml~~ 0.02 ml pgs to 1.5 ml = 750 ml extract
CB-2MSD 01493MSD 10 ml extract 0.02 ml to 1.5 ml = 750 ml extract
WS-7 00964 10 ml extract 0.02 ml to 10 ml = 500 ml extract
WS-7 00964 10 ml extract 0.1 ml to 10 ml = 100 ml extract
WS-6 00963 10 ml extract 0.1 ml to 10 ml = 100 ml extract
CB-2 01493 10 ml extract 0.025 ml to 1.5 ml = 600 ml extract
CB-2 01493 600 ml extract 0.1 ml to 10 ml = 6000 ml extract
WS-7 00964 10 ml extract 0.004 ml to 10 ml = 2500 ml extract
WS-7 00964 2500 ml extract 0.05 ml to 10 ml = 50000 ml extract
WS-7 00964 50000 ml extract 0.1 ml to 10 ml = 500000 ml extract
~~CB-2 01493 10 ml extract~~ pgs 01/30/90
WS-5 00962 10 ml extract 0.025 ml to 10 ml = 400 ml extract
WS-5 00962 400 ml extract 0.01 ml to 10 ml = 40000 ml extract

made up calibration stds as on 1-22-90 & ran dilutions on the instrument. pgs 01/30/90

pgs ~~01-30~~ 01-31-90 SAS 5165-C

run from 01-30-90 pgs did not meet standard requirements. made up calibration standards as on 01-22-90 & ran the dilutions on the instrument. pgs 01/31/90

02-01-90 SAS 5165-C

run from 01-31-90 failed because of instrument problems. made calibration standards as on 01-22-90 & ran the dilutions on the instrument. pgs 02/01/90

Pace Laboratories, Inc.

02-02-90 5165-CSAIS

run from 02-01-90. Failed because of injector port problems. Made up calibration standards as on 01-22-90. Mean deviations from 01-30 in attachment.

PJS 02/02/90

02-05-90 5165-CSAIS

diluted the following samples:

CB-1	10ml extract	0.02ml to 1.0ml = 500ml extract	01491
CB-1 Dup	10ml extract	0.02ml to 1.0ml = 500ml extract	01491 Dup
DS-6	10ml extract	0.1ml to 1.0ml = 100ml extract	00963
DS-7	10ml extract	0.02ml to 1.0ml = 500ml extract	00964
DS-5	10ml extract	0.1ml to 1.0ml = 100ml extract	00962
DS-3	10ml extract	0.2ml to 1.0ml = 50ml extract	00960
DS-4	10ml extract	0.2ml to 1.0ml = 50ml extract	00961
DS-1	10ml extract	0.2ml to 1.0ml = 50ml extract	00958
CB-2	10ml extract	0.025 ~~0.25ml~~ to 1.5ml = 600ml extract	01493
CB-2MS	10ml extract	0.05ml to 1.5ml = 300ml extract	01493MS
CB-2MSD	10ml extract	0.05ml to 1.5ml = 300ml extract	01493MSD

PJS 02-05-90

02-06-90 5165-C

diluted the following samples

CB1	500ml extract	0.25ml to 1.5ml = 300ml extract	01491
CB1	300ml extract	0.5ml to 1.0ml = 600ml extract	01491
CB1	300ml extract	0.1ml to 1.0ml = 3000ml extract	01491
CB1 duplicate	same 3 dilutions as above		01491 Duplicate
DS-6	100ml extract	0.1ml to 1.0ml = 1000ml extract	00963
DS-7	500ml extract	0.01ml to 1.0ml = 50,000ml extract	00964
DS-7	500ml extract	0.2ml to 1.0ml = 2500ml extract	00964
DS-5	100ml extract	0.1ml to 1.0ml = 1000ml extract	00
DS-5	1000ml extract	0.1ml to 1.0ml = 10,000ml extract	00
DS-5	1000ml extract	0.05ml to 1.0ml = 20,000ml extract	00962
DS-5	1000ml extract	0.025ml to 1.0ml = 40,000ml extract	00962
DS-3	50ml extract	0.05ml to 1.0ml = 1000ml extract	00960
DS-3	100ml extract	0.1ml to 1.0ml = 10000ml extract	00960

AB300708

Acc Laboratories

02-06-90 5165-C SAS continued
dilutions of Samples:

CB-2 6000 ml extract 0.1ml to 1.0 ml = 60000 ml extract 01493
CB-2 6000 ml extract 0.4ml to 10ml = 15000 ml extract 01493
CB-2MS 3000 ml extract 0.25ml to 1.0ml = 12000 ml extract 01493MS
CB-3MS 12000 ml extract 0.1ml to 1.0ml = 120,000 ml extract 01493MS
CB-2MSD same two dilutions as above 01493MSD

pgs 02/06/90

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE No. 5165C-01

Lab Name: Pace Analytical, Inc. Contract: 68-W8-0019 | WS-1

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 00958

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,3 trichlorobenzene	Column 1	24.34	24.25	24.49	Y	–
02	Column 2	13.93	13.85	13.96	N	–
03 pentachlorobenzene	Column 1	37.45	36.85	37.09	Y	–
04	Column 2	24.84	24.52	24.94	N	–
05 1,2,3,4 tetrachlorobenzene	Column 1	33.34	33.22	33.46	Y	–
06	Column 2	21.85	21.83	21.93	N	–
07 _____	Column 1	_____	_____	_____	–	–
08	Column 2	_____	_____	_____	–	–
09 _____	Column 1	_____	_____	_____	–	–
10	Column 2	_____	_____	_____	–	–
11 _____	Column 1	_____	_____	_____	–	–
12	Column 2	_____	_____	_____	–	–

Comments: _____

10

PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO. 5165C-03

Lab Name: [illegible] Contract: 68-D8-0019 [illegible]-3

Lab Code: PACE Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 00960

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,3-trichlorobenzene	Column 1	24.36	24.25	24.49	Y	_
02	Column 2	13.92	13.88	13.96	N	_
03 1,2,4-trichlorobenzene	Column 1	26.68	26.58	26.82	Y	_
04	Column 2	16.01	15.97	16.05	N	_
05 1,3,5-trichlorobenzene	Column 1	28.36	28.26	28.50	Y	_
06	Column 2	17.63	17.58	17.68	N	_
07 1,2,3,5-tetrachlorobenzene	Column 1	31.15	31.01	31.31	Y	_
08	Column 2	19.90	19.87	19.97	N	_
09 pentachlorobenzene	Column 1	37.06	36.94	37.18	Y	_
10	Column 2	24.85	24.82	24.94	N	_
11 1,2,3,4-tetrachlorobenzene	Column 1	33.44	33.32	33.56	Y	_
12	Column 2	21.85	21.83	21.93	N	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO. 5165C-03

Lab Name: Pace Analytical, Inc. Contract: 68-W8-0019 WS-3

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB-608 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 02960

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 heptachlor epoxide	Column 1	43.00	41.87	43.17	Y	_
02	Column 2	29.05	29.01	29.17	N	_
03	Column 1				_	_
04	Column 2				_	_
05	Column 1				_	_
06	Column 2				_	_
07	Column 1				_	_
08	Column 2				_	_
09	Column 1				_	_
10	Column 2				_	_
11	Column 1				_	_
12	Column 2				_	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
5165C-04

Lab Name: [illegible] Laboratories, Inc. Contract: 68-03-0019

Lab Code: [illegible] Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1301 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 20961

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,4 trichlorobenzene	Column 1	26.63	26.58	26.82	_	_
02	Column 2	16.00	15.97	16.05	_	_
03 1,3,5,4,6 hexachlorobenzene	Column 1	25.35	28.26	28.50	_	_
04	Column 2	17.62	17.58	17.65	_	_
05 1,2,3,5,6 hexachlorobenzene	Column 1	31.15	31.01	31.31	_	_
06	Column 2	19.91	19.87	19.97	_	_
07 pentachlorobenzene	Column 1	37.05	36.94	37.18	_	_
08	Column 2	24.85	24.82	24.94	_	_
09 1,2,3,4 tetrachlorobenzene	Column 1	33.43	33.32	33.56	_	_
10	Column 2	21.85	21.83	21.93	_	_
11 _____	Column 1	_____	_____	_____	_	_
12	Column 2	_____	_____	_____	_	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
51652-05

WS-5

Lab Name: Pace Laboratories, Inc. Contract: 68-W8-0019

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB-210 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: C0962

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,3 trichlorobenzene	Column 1	24.46	24.35	24.50	Y	_
02	Column 2	13.93	13.89	13.96	N	_
03 1,2,4 trichlorobenzene	Column 1	26.55	26.48	26.72	Y	_
04	Column 2	16.01	15.97	16.05	N	_
05 1,3,5 trichlorobenzene	Column 1	28.27	28.15	28.39	Y	_
06	Column 2	17.62	17.58	17.65	N	_
07 1,2,3,5 tetrachlorobenzene	Column 1	31.26	31.11	31.41	Y	_
08	Column 2	19.91	19.87	19.97	N	_
09 Pentachlorobenzene	Column 1	37.06	36.94	37.15	Y	_
10	Column 2	24.88	24.82	24.94	N	_
11 1,2,3,4 Tetrachlorobenzene	Column 1	33.43	33.31	33.55	Y	_
12	Column 2	21.85	21.83	21.93	N	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
5165C-05

Lab Name: Pace Laboratories, Inc Contract: 68-W8-0019 LJS-5

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 00963

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 hexachlorobenzene	Column 1	42.13	41.97	42.27	Y	–
02	Column 2	29.05	29.01	29.13	N	–
03	Column 1				–	–
04	Column 2				–	–
05	Column 1				–	–
06	Column 2				–	–
07	Column 1				–	–
08	Column 2				–	–
09	Column 1				–	–
10	Column 2				–	–
11	Column 1				–	–
12	Column 2				–	–

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
51654-06

Lab Name: Pace Laboratories, Inc. Contract: 68-W8-0019 WS-6

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 10963

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,4 Trichlorobenzene	Column 1	26.71	26.58	26.82	Y	–
02	Column 2	16.01	15.97	16.05	–	–
03 1,3,5 Trichlorobenzene	Column 1	28.27	28.15	28.39	Y	–
04	Column 2	17.62	17.58	17.69	–	–
05 Pentachlorobenzene	Column 1	36.39	36.35	37.09	Y	–
06	Column 2	24.86	24.82	24.94	–	–
07 1,2,3,4 Tetrachlorobenzene	Column 1	33.45	33.32	33.56	Y	–
08	Column 2	21.86	21.83	21.93	–	–
09 _____	Column 1				–	–
10	Column 2				–	–
11 _____	Column 1				–	–
12	Column 2				–	–

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
51650-09

Lab Name: [illegible] Contract: [illegible]

Lab Code: [illegible] Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): [illegible] GC Column ID (2): [illegible]

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 100164

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,1,1-Trichloroethane	Column 1	24.37	24.25	24.49	N	_
02	Column 2	13.92	13.88	13.96	N	_
03 1,1,2-Trichloroethane	Column 1	26.60	26.48	26.72	N	_
04	Column 2	16.02	15.97	16.05	N	_
05 1,1,2,2-Tetrachloroethane	Column 1	28.23	28.15	28.39	N	_
06	Column 2	17.65	17.58	17.68	N	_
07 1,2,3-Trichloropropane	Column 1	31.17	31.01	31.31	N	_
08	Column 2	19.91	19.87	19.97	N	_
09 Pentachloroethane	Column 1	36.96	36.85	37.09	N	_
10	Column 2	24.89	24.82	24.94	N	_
11 1,2,3,4,5,6-Hexachlorobenzene	Column 1	33.34	33.22	33.46	N	_
12	Column 2	21.89	21.83	21.93	N	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO. 5165C-07

Lab Name: [illegible] Contract: [illegible] WS-7

Lab Code: [illegible] Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: [illegible]

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 heptachlorepoxide	Column 1	40.00	41.87	42.17	Y	_
02	Column 2	29.05	29.01	29.17	N	_
03	Column 1				_	_
04	Column 2				_	_
05	Column 1				_	_
06	Column 2				_	_
07	Column 1				_	_
08	Column 2				_	_
09	Column 1				_	_
10	Column 2				_	_
11	Column 1				_	_
12	Column 2				_	_

Comments: _____

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO. 5165C-09

CO-1

Lab Name: Pace Laboratories, Inc. Contract: 68-W8-0019

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB17-01 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 01401

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,3,4-tetrachlorobenzene	Column 1	26.70	26.58	26.82	Y	_
02	Column 2	16.01	15.97	16.05	N	_
03 1,2,3-trichlorobenzene	Column 1	28.37	28.26	28.50	Y	_
04	Column 2	17.63	17.58	17.68	N	_
05 1,2,3,5-tetrachlorobenzene	Column 1	31.35	31.11	31.41	Y	_
06	Column 2	19.91	19.87	19.97	N	_
07 Pentachlorobenzene	Column 1	34.06	36.94	37.18	Y	_
08	Column 2	24.85	24.82	24.94	N	_
09 1,2,3,4-tetrachlorobenzene	Column 1	33.43	33.31	33.55	Y	_
10	Column 2	21.86	21.83	21.93	N	_
11 _____	Column 1	_____	_____	_____	_	_
12	Column 2	_____	_____	_____	_	_

Comments: _____

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AR300719

00038

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
5165C-09 Duplicate

Lab Name: Pace Laboratories, Inc. Contract: 68-W8-0019

CB-1 Duplicate

Lab Code: Pace Case No.: _____ SAS No.: 5165C SDG No.: _____

GC Column ID (1): DB17C1 GC Column ID (2): DB21C

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 61463 Duplicate

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,4-trichlorobenzene	Column 1	26.70	26.58	26.82	Y	_
02	Column 2	16.01	15.97	16.05	N	_
03 1,3,5-trichlorobenzene	Column 1	28.37	28.26	28.50	Y	_
04	Column 2	17.63	17.58	17.68	N	_
05 1,2,3,5-tetrachlorobenzene	Column 1	31.35	31.11	31.41	Y	_
06	Column 2	19.91	19.87	19.97	N	_
07 Pentachlorobenzene	Column 1	37.06	36.94	37.18	Y	_
08	Column 2	24.86	24.82	24.94	N	_
09 1,2,3,4-tetrachlorobenzene	Column 1	33.43	33.31	33.55	Y	_
10	Column 2	21.86	21.83	21.93	N	_
11 _____	Column 1	_____	_____	_____	_	_
12	Column 2	_____	_____	_____	_	_

Comments: _____

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AR300720

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PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE No.
5165C-11

CB-2

Lab Name: Pace Laboratories, Inc. Contract: 68-D8-0010

Lab Code: PACE Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: 01403

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 1,2,3-trichlorobenzene	Column 1	24.36	24.25	24.46	Y	_
02	Column 2	13.93	13.88	13.96	N	_
03 1,2,4-trichlorobenzene	Column 1	26.68	26.58	26.82	Y	_
04	Column 2	16.00	15.97	16.05	N	_
05 1,3,5-trichlorobenzene	Column 1	28.38	28.26	28.50	Y	_
06	Column 2	17.62	17.58	17.68	N	_
07 1,2,3,5-tetrachlorobenzene	Column 1	31.39	31.11	31.41	Y	_
08	Column 2	19.91	19.87	19.97	N	_
09 pentachlorobenzene	Column 1	36.96	36.85	37.09	Y	_
10	Column 2	24.85	24.82	24.94	Y	_
11 1,2,3,4-tetrachlorobenzene	Column 1	33.45	33.32	33.56	Y	_
12	Column 2	21.86	21.83	21.93	N	_

Comments: _____

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FORM X PEST 1/87 Rev.

AR300721

00040

PESTICIDE/PCB IDENTIFICATION

EPA SAMPLE NO.
5165C-11

CB-2

Lab Name: Pace Laboratories, Inc. Contract: 68-W8-0019

Lab Code: Pace Case No.: _____ SAS No.: 5165-C SDG No.: _____

GC Column ID (1): DB1701 GC Column ID (2): DB210

Instrument ID (1): A Instrument ID (2): A

Lab Sample ID: CB-2

Lab File ID: _____ (only if confirmed by GC/MS)

PESTICIDE/PCB	RETENTION TIME		RT WINDOW OF STANDARD FROM	TO	QUANT? (Y/N)	GC/MS? (Y/N)
01 hexachlorobenzene	Column 1	41.99	41.57	42.17	N	–
02	Column 2	28.65	28.01	28.17	N	–
03	Column 1				–	–
04	Column 2				–	–
05	Column 1				–	–
06	Column 2				–	–
07	Column 1				–	–
08	Column 2				–	–
09	Column 1				–	–
10	Column 2				–	–
11	Column 1				–	–
12	Column 2				–	–

Comments: _____

FORM X PEST 1/87 Rev.

SAS Standard Chlori

In Reference to Case No(s):
5165-C

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: 1/16/90

Laboratory Name: PACE Laboratory, Inc (Mpls, MN)

Lab Contact: Bill Scanlon

Region: III

Regional Contact: Stevie Wilding

Call Initiated By: X Laboratory ___ Region

JAN 1990 RECEIVED

In reference to data for the following sample number(s):
WS-1 → WS-7, CB-1, CB-2

Summary of Questions/Issues Discussed:
(1) 2-Fluorobiphenyl does not have a response in the ECD and (2) the spiking level (100 ug/kg) is too low

Summary of Resolution:
(1) use 2-chloronaphthalene as surrogate
(2) spike at 1000 ug/kg
(3) re-extract all soil samples
(4) The water samples (EQB-2 and EQB-3) were already extracted using 2-Fluorobiphenyl as the surrogate and can NOT be re-extracted. We will run samples and note the problems in narrative

William H Scanlon
Signature

Date

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy

AR300723

Standard Chlorine

In Reference to Case No(s):

5165C Task 2

TES/VE

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM

Telephone Record Log

Date of Call: 1-8-90 monday

Laboratory Name: Pace Lab

Lab Contact: Sue Maf

Region: III

Regional Contact: Collen K Walling

Call Initiated By: ✓ Laboratory ___ Region

In reference to data for the following sample number(s):

SAS 5165 C Task 2 10 low soil for 1,2,4 Trichlorobenzene; 1,2,3 Trichlorobenzene; 1,3,5 Trichlorobenzene, 1,2,4,5 Tetrachlorobenzene; 1,2,3,4 Tetrachlorobenzene, pentachlorobenzene, hexachlorobenzene

Summary of Questions/Issues Discussed:

① Lab was suppose to receive shipment on Saturday 1-6-90 lab did not receive any samples then for this SAS.

② Did samples ship if so need to track.

Summary of Resolution:

Called D. Backs of VERSAR to see if samples shipped. Dave Backs said samples did not ship on Friday because lab assignment was not rec'd in time. Samples will ship today 1-8-90. Samples were collected last week (Friday ??). ~~Time~~ Lab assignments in time for sampling could have occurred if requesting contractor had submitted SAS requests earlier with sufficient lead time to process.

Collen K. Walling Region III RSCC 1-8-90
Signature Date

AR 300724

AR 300724

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy (4) Bob Guarni (3HW25) (5) Dave Backs VERSAR (PA) (6) Elaine Spiewak (3HW--) (7) trouble file (8) AFC - Diane Sima (9) Collen Walling (10) site file

In Reference to Case No(s):
5165-C Task 2
Standard Chlorine, TES/VERSAR

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: 12 January 1990

Laboratory Name: PACE

Lab Contact: Bill Scruden

Region: III

Regional Contact: Colleen Walling

Call Initiated By: X **Laboratory** **Region**

In Reference to data for the following sample number(s):
Two aqueous samples sent under this case.

Summary of Questions/Issues Discussed:
The lab received an "extra" aqueous sample with these soils. It was not labeled as a rinsate. Is it actually a rinsate which was not included in the SAS original request or is it additional volume?

Summary of Resolution:
Per the sampler(VERSAR): these were two separate rinsates not additional volume and they want both samples analyzed. Per Terri Shaughnessy of SMO the lab must analyze both samples.

Colleen Walling
Signature by L. Brown PSA

1-10-
Da

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy, (4) Dave Basko, VERSAR, Langhorne, (5) Colleen Walling, (6) Diann Sims, (7) Bob Guarni (3HW17), (8) Elaine Spiewak(3HW17), (9) trouble file, [redacted]

AR300725

In Reference to Case No(s):
5165-C Task 2, Standard Chlorine,
TES/VERSAR

Contract Laboratory Program
REGIONAL/LABORATORY COMMUNICATION SYSTEM
Telephone Record Log

Date of Call: 16 January 1990

Laboratory Name: PACE

Lab Contact: Bill Scruden

Region: III

Regional Contact: Colleen Walling/Stevie Wilding

Call Initiated By: X **Laboratory** **Region**

In Reference to data for the following sample number(s):
All samples sent under this case.

Summary of Questions/Issues Discussed:
The lab suggests that the concentration requested for the surrogates is too low. Additionally, their detector does not discern 2-fluorobiphenyl, even at higher concentrations.

Summary of Resolution:
Per Stevie Wilding: the surrogate concentration should be increased to 1000 ug/Kg. The surrogate 2-chloronaphthalene may be used instead of 2-fluorobiphenyl. All samples must be re-prepared with 2-chloronapthalene and re-extracted. The re-extraction will not be penalized for not meeting the 10 day technical holding time.

AR 300726

Stevie Wilding
Signature

1-18-90
Date

Distribution: (1) Lab Copy, (2) Region Copy, (3) SMO Copy, (4) Dave Basko, VERSAR, Langhorne, (5) Colleen Walling, (6) Diann Sims, (7) Bob Guarni (3HW17), (8) Stevie Wilding, (9) Elaine Spiewak (3HW17), (10) trouble file, [redacted]

5165C Task II

SAS 612

U.S. Environmental Protection Agency
LP Sample Management Office
209 Madison Street, Alexandria, VA 22313
PHONE: (703) 557-2490 or FTS 557-2490

SAS Number

SPECIAL ANALYTICAL SERVICES
Regional Request

☐ Regional Transmittal

☐ Telephone Request

A. EPA Region and Client: EPA Region III

B. Regional Representative: Colleen K. Walling

C. Telephone Number: (301) 266-9180

D. Date of Request: December 15, 1989

E. Site Name: Standard Chlorine of Delaware, Delaware City, Delaware

sent to SMO 12-22-89

Please provide below a description of your request for Special Analytical Services under the Contract Laboratory Program. In order to most efficiently obtain laboratory capability for your request, please address the following considerations, if applicable. Incomplete or erroneous information may result in delay in the processing of your request. Please continue response on additional sheets, or attach supplementary information as needed.

1. General description of analytical service requested:
Analysis of 10 low concentration soil/sediment samples for 1,2,4-trichlorobenzene 1,2,3-trichlorobenzene; 1,3,5-trichlorobenzene; 1,2,4,5-tetrachlorobenzene; 1,2,3,4-tetrachlorobenzene; pentachlorobenzene; and hexachlorobenzene using SW-846 extraction method 3550 and SW-846 analysis method 8120 (both methods are attached).

2. Definition and number of work units involved (specify whether whole samples or fractions; whether organics or inorganics; whether aqueous or soil and sediments; and whether low, medium, or high concentration):
Analysis of 10 low concentration soil/sediment samples for the above include 8 soil/sediment samples, 1 field duplicate, and 1 equipment blank. The equipment blank will be an aqueous sample.

AR300727

3. Program (specify whether Superfund (Remedial or Enforcement), RCRA, NPDES, etc.), Justification for analysis and Site Account Number:

Superfund Enforcement : RP RI/FS Oversight

OTGB03NPH6

SAS Approved By:

4. Estimated date(s) of collection: January 2 through February 2, 1990

5. Estimated date(s) and method of shipment: January 2 through February 2, 1990

Federal Express- Overnight delivery

6. Approximate number of days results required after lab receipt of samples:
Extraction must be performed within 10 days of sample collection, and analysis must be performed within 40 days of extraction. Data package within 30 day of analysis of last sample.

7. Analytical protocol required (attach copy if other than a protocol currently used in this program):
Test Methods for Evaluating Solid Wastes (SW-846), Third Edition, 1986
Methods 3550 (Sonication Extraction) and 8120 (Chlorinated Hydrocarbons-GC/
Both methods are attached.

8. Special technical instructions (if outside protocol requirements, specify compound names, CAS numbers, detection limits, etc.):

See Attachment 1.

9. Analytical results required (if known, specify format for data sheets, QA/QC reports, Chain-of-Custody documentation, etc.). If not completed, format of results will be left to program discretion.

See Attachment 2.

10. Other (use additional sheets or attach supplementary information, as needed):
None.

11. Name of sampling/shipping contact: David A. Basko

Phone: (215) 741-4211

12. Data Requirements

Parameter	Detection Limit	Precision Desired * (+ or − Concentration)
1,2,4-trichlorobenzene	330 ug/kg	+/- 20%
1,2,3-trichlorobenzene	330 ug/kg	+/- 20%
1,3,5-trichlorobenzene	330 ug/kg	+/- 20%
1,2,4,5-tetrachlorobenzene	330 ug/kg	+/- 20%
1,2,3,4-tetrachlorobenzene	330 ug/kg	+/- 20%
pentachlorobenzene	330 ug/kg	+/- 25%
hexachlorobenzene	330 ug/kg	+/- 25%

* Advisory limits only, not mandatory (corrective action not required).

13. QC Requirements

Audits Required	Frequency of Audits	Limits (Percent or Concentration)
LAB Duplicates	1/20 or 1/batch	+/- 25% RPD
Method Blanks	1/20 or 1/batch	< 330 ug/kg ANY target
Matrix Spikes	1/20 or 1/batch	+/- 30% Recovery*
2-fluorobiphenyl (surrogate)	Every sample	38 - 115% Recovery*
QC check standard	1/20 or 1/batch	85-115% of true value Recovery*
Continuing Calibration Standard	1/10 or at end of batch	+/- 20% D RF EACH target

* Advisory limits only, not mandatory (corrective action not required).

14. Action Required if Limits are Exceeded

Duplicates: Reanalyze sample/duplicate pair and report both sets of data. (Reanalyze 1 time only)

Method Blank: Reanalyze all associated samples after corrective action has been taken to reduce blank contamination to less than 330 ug/kg

Continuing Calibration Standard: Perform initial calibration and reanalyze all samples since last acceptable continuing calibration standard.

15. Request prepared by: David A. Basko

Date: December 15, 1989

16. Request reviewed by: Laurie L.A. Brown C.S. 12/22/89

Date: 12-21-89

Please return this request to the Sample Management Office as soon as possible to expedite processing of your request for special analytical services. Should you have any questions or need any assistance, please contact your Business Representative at the Sample Management Office.

ATTACHMENT 1

Standardize instruments according to manufacturer's instructions. Analytical procedures, as described in the attached method, MUST be followed even if the text just indicates that those procedures should be followed. Report all holding times on the data sheets.

The instrumentation must be calibrated daily using five calibration standards and a calibration blank. The calibration standards must contain all target analytes at concentrations which bracket the anticipated range of measurement, and these standards must be prepared fresh daily from the stock solution. One of the calibration standards must be near, but above, the method detection limit. Continuing calibration must be performed by analyzing one calibration standard (containing all target analytes) at the mid-range of the initial calibration curve after each ten samples. The response factor of this continuing calibration standard must be +/- 20 percent of the average response factor of the initial calibration, or else initial calibration must be repeated, and all samples analyzed since the last acceptable continuing calibration standard must be reanalyzed.

All samples must be spiked with 2-fluorobiphenyl as a surrogate compound at a nominal final concentration of 100 µg/kg. All initial and continuing calibration standards must contain 2-fluorobiphenyl at the same concentrations as the standard analytes. The average response factor for 2-fluorobiphenyl from the initial calibration must be used to calculate sample surrogate recoveries.

A matrix spike must be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent. The spike concentration must be 1 to 5 times the background sample concentration and must be determined by screening.

A QC check standard must also be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent. This check standard must be prepared from an independent source material of that used to prepare that calibration standards.

A method blank must also be analyzed at a frequency of 1/20 samples or 1/batch, whichever is more frequent.

ATTACHMENT 2

Data package must include: all raw data, all instrument and/or equipment calibration results, calculations, blank results, duplicate results, chain of custody forms, SAS request forms, SAS packing list(s) or traffic report(s), copy of airbill(s), and copies of analyst's logbooks(signed by analyst) with date and time of sample preparation and analysis.

The cover page and all sample report forms MUST be labled with the complete EPA sample number as it appears on chain of custody and CLP paperwork.

The case narrative must document all problems encountered and the subsequent resolutions. List instrumentation and methods employed for analysis. Also, note whether samples were preserved or not and the procedure utilized in preservation. EPA QC reference samples, or equivalent reference samples must be identified as to source and lot number. Documentation of "true" value and associated 95 % confidence limits must be provided for any reference samples used.

METHOD 3550

SONICATION EXTRACTION

1.0 SCOPE AND APPLICATION

1.1 Method 3550 is a procedure for extracting nonvolatile and semi-volatile organic compounds from solids such as soils, sludges, and wastes. The sonication process ensures intimate contact of the sample matrix with the extraction solvent.

1.2 The method is divided into two sections, based on the expected concentration of organics in the sample. The low concentration method (individual organic components of $\leq$20 mg/kg) uses a larger sample size and a more rigorous extraction procedure (lower concentrations are more difficult to extract). The high concentration method (individual organic components of >20 mg/kg) is much simpler and therefore faster.

1.3 It is highly recommended that the extracts be cleaned up prior to analysis. See Cleanup, Section 4.2.2 of Chapter Four, for applicable methods.

2.0 SUMMARY OF METHOD

2.1 Low concentration method: A 30-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted three times using sonication. The extract is separated from the sample by vacuum filtration or centrifugation. The extract is ready for cleanup and/or analysis following concentration.

2.2 High concentration method: A 2-g sample is mixed with anhydrous sodium sulfate to form a free-flowing powder. This is solvent extracted once using sonication. A portion of the extract is removed for cleanup and/or analysis.

3.0 INTERFERENCES

3.1 Refer to Method 3500.

4.0 APPARATUS AND MATERIALS

4.1 Apparatus for grinding: If the sample will not pass through a 1-mm standard sieve or cannot be extruded through a 1-mm opening, it should be processed into a homogeneous sample that meets these requirements. Fisher Mortar Model 155 Grinder, Fisher Scientific Co., Catalogue Number 8-323, or an equivalent brand and model, is recommended for sample processing. This grinder should handle most solid samples, except gummy, fibrous, or oily materials.

4.2 Sonication: A horn-type sonicator equipped with a titanium tip should be used. The following sonicator, or an equivalent brand and model, is recommended:

Ultrasonic cell disrupter: Heat Systems - Ultrasonics, Inc., Model W-385 (475 watt) sonicator or equivalent (Power wattage must be a minimum of 375 with pulsing capability and No. 200 1/2" Tapped Disrupter Horn) plus No. 207 3/4" Tapped Disrupter Horn, and No. 419 1/8" Standard Tapered microtip probe.

4.3 Sonabox: Recommended with above disrupters for decreasing cavitation sound (Heat Systems - Ultrasonics, Inc., Model 432B or equivalent).

4.4 Apparatus for determining percent moisture:

4.4.1 Oven: Drying.

4.4.2 Desiccator.

4.4.3 Crucibles: Porcelain.

4.5 Pasteur glass pipets: Disposable, 1-mL.

4.6 Beakers: 400-mL.

4.7 Vacuum filtration apparatus:

4.7.1 Buchner funnel.

4.7.2 Filter paper: Whatman No. 41 or equivalent.

4.8 Kuderna-Danish (K-D) apparatus:

4.8.1 Concentrator tube: 10-mL graduated (Kontes K-570050-1025 or equivalent).

4.8.2 Evaporator flask: 500-mL (Kontes K-570001-0500 or equivalent).

4.8.3 Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.8.4 Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.9 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.10 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.11 Balance: Top-loading, capable of accurately weighing 0.01 g.

4.12 Vials and caps: 2-mL for GC auto-sampler.

4.13 Glass scintillation vials: At least 20-mL, with screw-cap and Teflon or aluminum foil liner.

4.14 Spatula: Stainless steel or Teflon.

4.15 Drying column: 20-mm I.D. Pyrex chromatographic column with Pyrex glass wool at bottom and a Teflon stopcock.

NOTE: Fritted glass discs are difficult to decontaminate after highly contaminated extracts have been passed through. Columns without frits may be purchased. Use a small pad of Pyrex glass wool to retain the adsorbent. Prewash the glass wool pad with 50 mL of acetone followed by 50 mL of elution solvent prior to packing the column with adsorbent.

4.16 Syringe: 5-mL.

5.0 REAGENTS

5.1 Sodium sulfate: Anhydrous and reagent grade, heated at 400°C for 4 hr, cooled in a desiccator, and stored in a glass bottle. Baker anhydrous powder, catalog #73898, or equivalent.

5.2 Extraction solvents: Methylene chloride:acetone (1:1, v:v), methylene chloride, hexane (pesticide quality or equivalent).

5.3 Exchange solvents: Hexane, 2-propanol, cyclohexane, acetonitrile (pesticide quality or equivalent).

6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1.

7.0 PROCEDURE

7.1 Sample handling:

7.1.1 Sediment/soil samples: Decant and discard any water layer on a sediment sample. Mix sample thoroughly, especially composited samples. Discard any foreign objects such as sticks, leaves, and rocks.

7.1.2 Waste samples: Samples consisting of multiphases must be prepared by the phase separation method in Chapter Two before extraction. This procedure is for solids only.

7.1.3 Dry waste samples amenable to grinding: Grind or otherwise subdivide the waste so that it either passes through a 1-mm sieve or can be extruded through a 1-mm hole. Introduce sufficient sample into the grinding apparatus to yield at least 10 g after grinding.

7.2 Determination of percent moisture: In certain cases, sample results are desired based on a dry-weight basis. When such data is desired, a portion of sample for moisture determination should be weighed out at the same time as the portion used for analytical determination.

7.2.1 Immediately after weighing the sample for extraction, weigh 5-10 g of the sample into a tared crucible. Determine the percent moisture by drying overnight at 105°C. Allow to cool in a desiccator before weighing:

$$\frac{\text{g of sample} - \text{g of dry sample}}{\text{g of sample}} \times 100 = \%\ \text{moisture}$$

7.3 Determination of pH (if required): Transfer 50 g of sample to a 100-mL beaker. Add 50 mL of water and stir for 1 hr. Determine the pH of sample with glass electrode and pH meter while stirring. Discard this portion of sample.

7.4 Extraction method for samples expected to contain low concentrations of organics and pesticides ($\leq$20 mg/kg):

7.4.1 The following step should be performed rapidly to avoid loss of the more volatile extractables. Weigh approximately 30 g of sample into a 400-mL beaker. Record the weight to the nearest 0.1 g. Non-porous or wet samples (gummy or clay type) that do not have a free-flowing sandy texture must be mixed with 60 g of anhydrous sodium sulfate using a spatula. The sample should be free-flowing at this point. Add 1 mL of surrogate standards to all samples, spikes, and blanks (see Method 3500 for details on the surrogate standard solution and the matrix spike solution). For the sample in each analytical batch selected for spiking, add 1.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 100 ng/uL of each base/neutral analyte and 200 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half of the extract is lost due to loading of the GPC column. Immediately add 100 mL of 1:1 methylene chloride:acetone.

7.4.2 Place the bottom surface of the tip of the #207 3/4 in. disruptor horn about 1/2 in. below the surface of the solvent, but above the sediment layer.

7.4.3 Sonicate for 3 min, with output control knob set at 10 and with mode switch on Pulse and percent-duty cycle knob set at 50%. Do NOT use microtip probe.

7.4.4 Decant and filter extracts through Whatman No. 41 filter paper using vacuum filtration or centrifuge and decant extraction solvent.

7.4.5 Repeat the extraction two or more times with two additional 100-mL portions of solvent. Decant off the extraction solvent after each sonication. On the final sonication, pour the entire sample into the Buchner funnel and rinse with extraction solvent.

7.4.6 Assemble a Kuderna-Danish (K-D) concentrator by attaching a 10-mL concentrator tube to a 500-mL evaporative flask.

7.4.7 Dry the extract by passing it through a drying column containing about 10 cm of anhydrous sodium sulfate. Collect the dried extract in a K-D concentrator. Wash the extractor flask and sodium sulfate column with 100-125 mL of extraction solvent to complete the quantitative transfer.

7.4.8 Add one or two clean boiling chips to the evaporative flask and attach a three-ball Snyder column. Prewet the Snyder column by adding about 1 mL methylene chloride to the top. Place the K-D apparatus on a hot water bath (80-90°C) so that the concentrator tube is partially immersed in the hot water and the entire lower rounded surface of the flask is bathed with hot vapor. Adjust the vertical position of the apparatus and the water temperature, as required, to complete the concentration in 10-15 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood with condensed solvent. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.4.9 If a solvent exchange is required (as indicated in Table 1), momentarily remove the Snyder column, add 50 mL of the exchange solvent and a new boiling chip, and re-attach the Snyder column. Concentrate the extract as described in Paragraph 7.4.8, raising the temperature of the water bath, if necessary, to maintain proper distillation.

7.4.10 Remove the Snyder column and rinse the flask and its lower joints into the concentrator tube with 1-2 mL of methylene chloride or exchange solvent. If sulfur crystals are a problem, proceed to Method 3660 for cleanup. The extract may be further concentrated by using the technique outlined in Paragraph 7.4.11 or adjusted to 10.0 mL with the solvent last used.

7.4.11 Add a clean boiling chip and attach a two-ball micro-Snyder column to the concentrator tube. Prewet the column by adding approximately 0.5 mL of methylene chloride or exchange solvent through the top. Place the apparatus in the hot water bath. Adjust the vertical position and the water temperature, as required, to complete the concentration in 5-10 min. At the proper rate of distillation, the balls of the column will actively chatter, but the chambers will not flood. When the liquid

TABLE 1. SPECIFIC EXTRACTION CONDITIONS FOR VARIOUS DETERMINATIVE METHODS

Determinative method	Extraction pH	Exchange solvent required for analysis	Exchange solvent required for cleanup	Volume of extract required for cleanup (mL)	Final extract volume for analysis (mL)
8040[a]	as received	2-propanol	hexane	1.0	1.0, 10.0[b]
8060	as received	hexane	hexane	2.0	10.0
8080	as received	hexane	hexane	10.0	10.0
8090	as received	hexane	hexane	2.0	1.0
8100	as received	none	cyclohexane	2.0	1.0
8120	as received	hexane	hexane	2.0	1.0
8140	as received	hexane	hexane	10.0	10.0
8250[a,c]	as received	none	-	-	1.0
8270[a,c]	as received	none	-	-	1.0
8310	as received	acetonitrile	-	-	1.0

[a]To obtain separate acid and base/neutral extracts, Method 3650 should be performed following concentration of the extract to 10.0 mL.

[b]Phenols may be analyzed, by Method 8040, using a 1.0 mL 2-propanol extract by GC/FID. Method 8040 also contains an optional derivatization procedure for phenols which results in a 10 mL hexane extract to be analyzed by GC/ECD.

[c]The specificity of GC/MS may make cleanup of the extracts unnecessary. Refer to Method 3600 for guidance on the cleanup procedures available if required.

reaches an apparent volume of approximately 0.5 mL, remove the apparatus from the water bath and allow to drain and cool for at least 10 min. Remove the micro-Snyder column and rinse its lower joint into the concentrator tube with approximately 0.2 mL of appropriate solvent. Adjust the final volume to the volume required for cleanup or for the determinative method (see Table 1).

7.4.12 Transfer the concentrated extract to a clean screw-cap vial. Seal the vial with a Teflon-lined lid and mark the level on the vial. Label with the sample number and fraction and store in the dark at 4°C until ready for analysis or cleanup.

7.5 Extraction method for samples expected to contain high concentrations of organics (>20 mg/kg):

7.5.1 Transfer approximately 2 g (record weight to the nearest 0.1 g) of sample to a 20-mL vial. Wipe the mouth of the vial with a tissue to remove any sample material. Record the exact weight of sample taken. Cap the vial before proceeding with the next sample to avoid any cross contamination.

7.5.2 Add 2 g of anhydrous sodium sulfate to sample in the 20-mL vial and mix well.

7.5.3 Surrogate standards are added to all samples, spikes, and blanks (see Method 3500 for details on the surrogate standard solution and on the matrix spike solution). Add 2.0 mL of surrogate spiking solution to sample mixture. For the sample in each analytical batch selected for spiking, add 2.0 mL of the matrix spiking standard. For base/neutral-acid analysis, the amount added of the surrogates and matrix spiking compounds should result in a final concentration of 200 ng/uL of each base/neutral analyte and 400 ng/uL of each acid analyte in the extract to be analyzed (assuming a 1 uL injection). If Method 3640, Gel-permeation cleanup, is to be used, add twice the volume of surrogates and matrix spiking compounds since half the extract is lost due to loading of the GPC column.

7.5.4 Immediately add whatever volume of solvent is necessary to bring the final volume to 10.0 mL considering the added volume of surrogates and matrix spikes. Disrupt the sample with the 1/8-in. tapered microtip ultrasonic probe for 2 min at output control setting 5 and with mode switch on pulse and percent duty cycle of 50%. Extraction solvents are:

1. Nonpolar compounds, i.e., organochlorine pesticides and PCBs: hexane.

2. Extractable priority pollutants: methylene chloride.

7.5.5 Loosely pack disposable Pasteur pipets with 2- to 3-cm Pyrex glass-wool plugs. Filter the extract through the glass wool and collect

5.0 mL in a concentrator tube if further concentration is required. Follow Paragraphs 7.4.6 through 7.4.12 for details on concentration. Normally, the 5.0 mL extract is concentrated to 1.0 mL.

7.5.6 The extract is ready for cleanup or analysis, depending on the extent of interfering co-extractives.

8.0 QUALITY CONTROL

8.1 Any reagent blanks or matrix spike samples should be subject to exactly the same analytical procedures as those used on actual samples.

8.2 Refer to Chapter One for specific quality control procedures and Method 3500 for extraction and sample preparation procedures.

9.0 METHOD PERFORMANCE

9.1 Refer to the determinative methods for performance data.

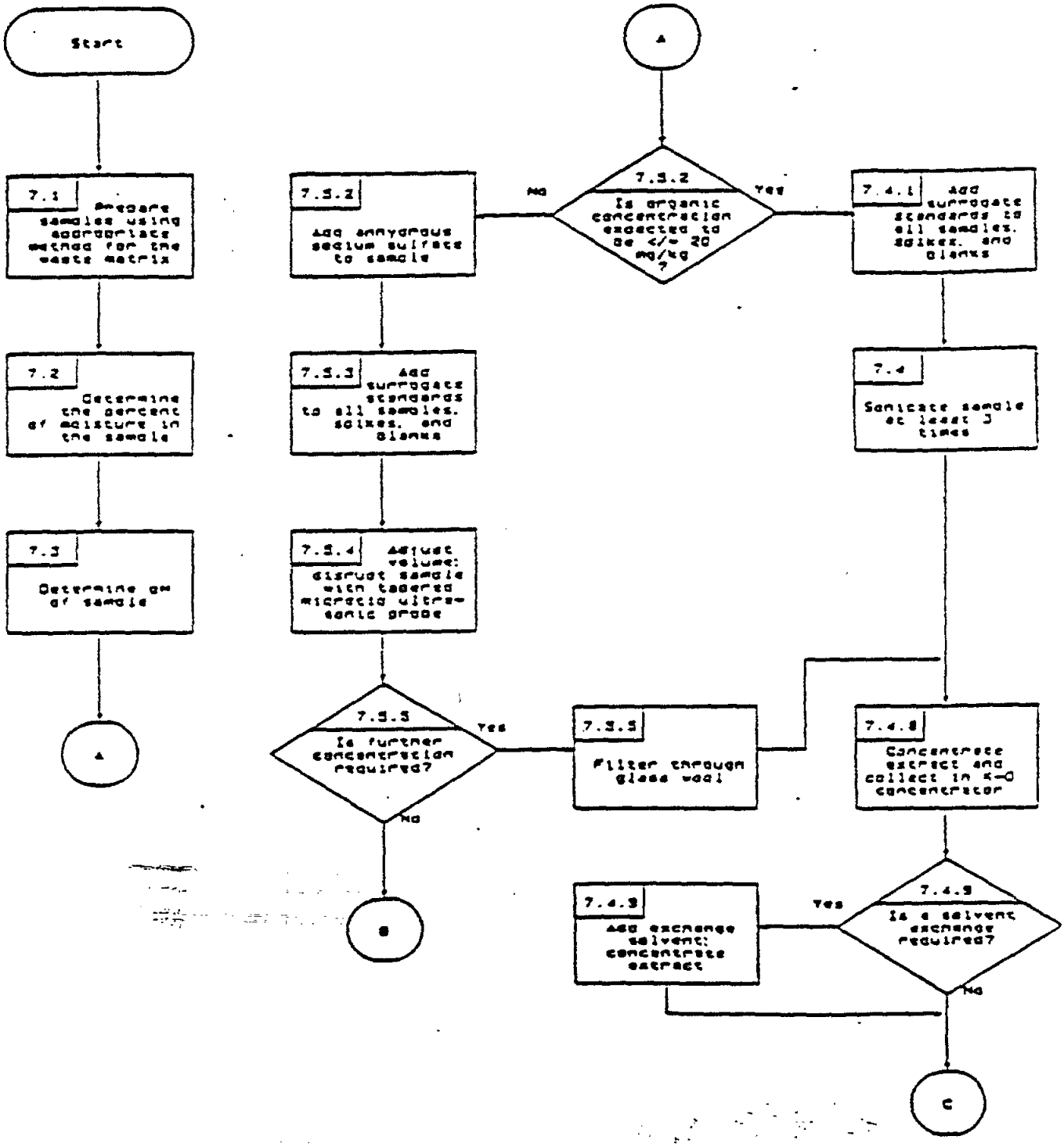
10.0 REFERENCES

1. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

2. U.S. EPA, Interlaboratory Comparison Study: Methods for Volatile and Semi-Volatile Compounds, Environmental Monitoring Systems Laboratory, Office of Research and Development, Las Vegas, NV, EPA 600/4-84-027, 1984.

METHOD 3550

SONICATION EXTRACTION



Revision 0
Date September 1986

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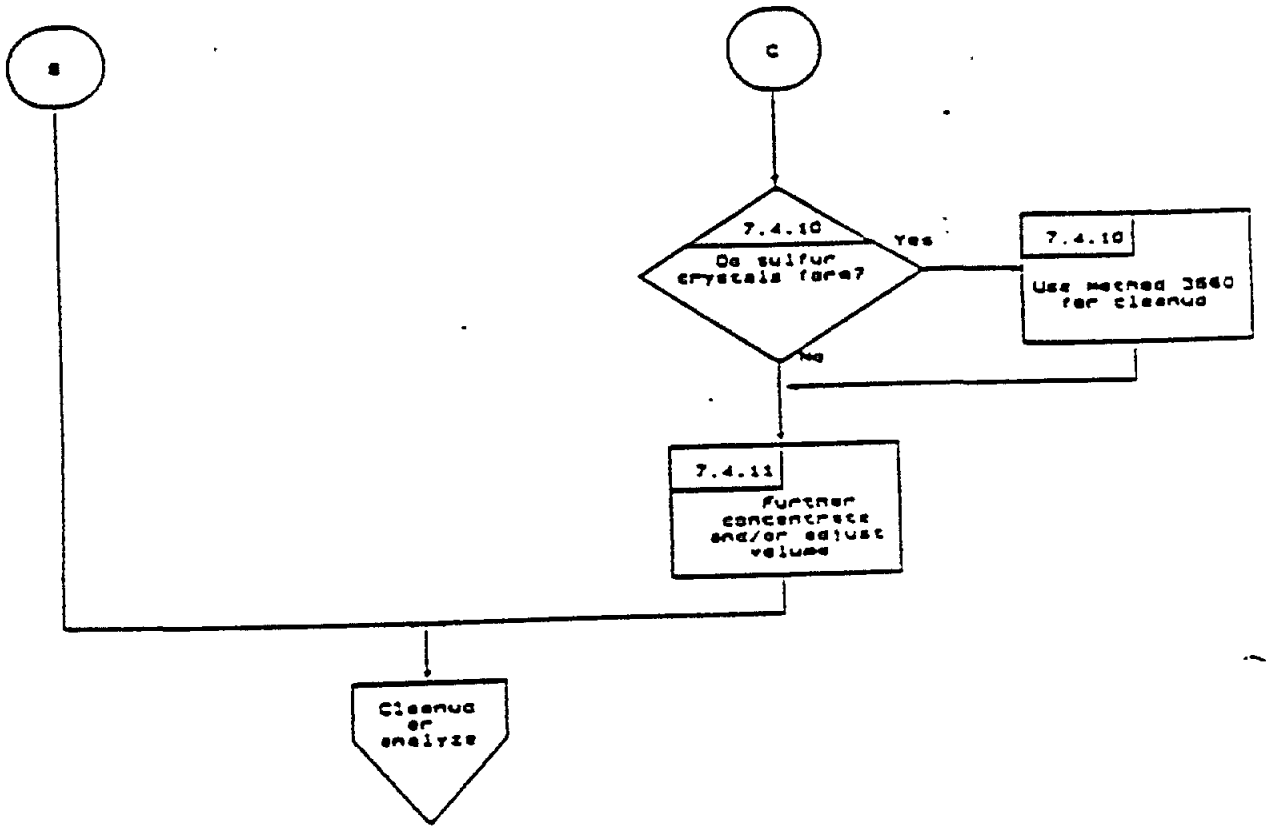
B
C
7.4.10
Do sulfur crystals form?
Yes
No
7.4.10
Use method 3660 for cleanup
7.4.11
Further concentrate and/or adjust volume
Cleanup or analyze

METHOD 8120

CHLORINATED HYDROCARBONS

1.0 SCOPE AND APPLICATION

1.1 Method 8120 is used to determine the concentration of certain chlorinated hydrocarbons. Table 1 indicates compounds that may be determined by this method and lists the method detection limit for each compound in reagent water. Table 2 lists the practical quantitation limit (PQL) for other matrices.

2.0 SUMMARY OF METHOD

2.1 Method 8120 provides gas chromatographic conditions for the detection of ppb levels of certain chlorinated hydrocarbons. Prior to use of this method, appropriate sample extraction techniques must be used. Both neat and diluted organic liquids (Method 3580, Waste Dilution) may be analyzed by direct injection. A 2- to 5-uL aliquot of the extract is injected into a gas chromatograph (GC) using the solvent flush technique, and compounds in the GC effluent are detected by an electron capture detector (ECD).

2.2 If interferences are encountered in the analysis, Method 8120 may also be performed on extracts that have undergone cleanup using Method 3620.

3.0 INTERFERENCES

3.1 Refer to Methods 3500, 3600, and 8000.

3.2 Solvents, reagents, glassware, and other sample processing hardware may yield discrete artifacts and/or elevated baselines causing misinterpretation of gas chromatograms. All of these materials must be demonstrated to be free from interferences, under the conditions of the analysis, by analyzing method blanks. Specific selection of reagents and purification of solvents by distillation in all-glass systems may be required.

3.3 Interferences coextracted from samples will vary considerably from source to source, depending upon the waste being sampled. Although general cleanup techniques are recommended as part of this method, unique samples may require additional cleanup.

4.0 APPARATUS AND MATERIALS

4.1 Gas chromatograph:

4.1.1 Gas chromatograph: Analytical system complete with gas chromatograph suitable for on-column injections

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TABLE 1. GAS CHROMATOGRAPHY OF CHLORINATED HYDROCARBONS

Compound	Retention time (min) Col. 1	Retention time (min) Col. 2	Method Detection limit (ug/L)
Benzal chloride			
Benzotrichloride			
Benzyl chloride			
2-Chloronaphthalene	2.7[a]	3.6[b]	0.94
1,2-Dichlorobenzene	6.6	9.3	1.14
1,3-Dichlorobenzene	4.5	6.8	1.19
1,4-Dichlorobenzene	5.2	7.6	1.34
Hexachlorobenzene	5.6[a]	10.1[b]	0.05
Hexachlorobutadiene	7.7	20.0	0.34
Hexachlorocyclohexane			
Hexachlorocyclopentadiene	nd	16.5[c]	0.40
Hexachloroethane	4.9	8.3	0.03
Tetrachlorobenzenes			
1,2,4-Trichlorobenzene	15.5	22.3	0.05
Pentachlorohexane			

nd = not determined.

[a]150°C column temperature.

[b]165°C column temperature.

[c]100°C column temperature.

TABLE 2. DETERMINATION OF PRACTICAL QUANTITATION LIMITS (PQL) FOR VARIOUS MATRICES[a]

Matrix	Factor[b]
Ground water	10
Low-level soil by sonication with GPC cleanup	670
High-level soil and sludges by sonication	10,000
Non-water miscible waste	100,000

[a]Sample PQLs are highly matrix-dependent. The PQLs listed herein are provided for guidance and may not always be achievable.

[b]PQL = [Method detection limit (Table 1)] X [Factor (Table 2)]. For non-aqueous samples, the factor is on a wet-weight basis.

accessories, including detectors, column supplies, recorder, gases, and syringes. A data system for measuring peak areas and/or peak heights is recommended.

4.1.2 Columns:

4.1.2.1 Column 1: 1.8-m x 2-mm I.D. glass column packed with 1% SP-1000 on Supelcoport (100/120 mesh) or equivalent.

4.1.2.2 Column 2: 1.8-m x 2-mm I.D. glass column packed with 1.5% OV-1/2.4% OV-225 on Supelcoport (80/100 mesh) or equivalent.

4.1.3 Detector: Electron capture (ECD).

4.2 Kuderna-Danish (K-D) apparatus:

4.2.1 Concentrator tube: 10-mL, graduated (Kontes K-570050-1025 or equivalent). Ground-glass stopper is used to prevent evaporation of extracts

4.2.2 Evaporation flask: 500-mL (Kontes K-570001-500 or equivalent). Attach to concentrator tube with springs.

4.2.3 Snyder column: Three-ball macro (Kontes K-503000-0121 or equivalent).

4.2.4 Snyder column: Two-ball micro (Kontes K-569001-0219 or equivalent).

4.3 Boiling chips: Solvent extracted, approximately 10/40 mesh (silicon carbide or equivalent).

4.4 Water bath: Heated, with concentric ring cover, capable of temperature control (±5°C). The bath should be used in a hood.

4.5 Volumetric flasks: 10-, 50-, and 100-mL, ground-glass stopper.

4.6 Microsyringe: 10-uL.

4.7 Syringe: 5-mL.

4.8 Vials: Glass, 2-, 10-, and 20-mL capacity with Teflon-lined screw cap.

5.0 REAGENTS

5.1 Solvents: hexane, isooctane, acetone (pesticide quality or equivalent).

5.2 Stock standard solutions:

5.2.1 Prepare stock standard solutions at a concentration of 1.00 ug/uL by dissolving 0.0100 g of assayed reference material in isooctane and diluting to volume in a 10-mL volumetric flask. Larger volumes can be used at the convenience of the analyst. When compound purity is assayed to be 96% or greater, the weight can be used without correction to calculate the concentration of the stock standard. Commercially prepared stock standards can be used at any concentration if they are certified by the manufacturer or by an independent source.

5.2.2 Transfer the stock standard solutions into Teflon-sealed screw-cap bottles. Store at 4°C and protect from light. Stock standards should be checked frequently for signs of degradation or evaporation, especially just prior to preparing calibration standards from them.

5.2.3 Stock standard solutions must be replaced after one year, or sooner if comparison with check standards indicates a problem.

5.3 Calibration standards: Calibration standards at a minimum of five concentration levels should be prepared through dilution of the stock standards with isooctane. One of the concentration levels should be at a concentration near, but above, the method detection limit. The remaining concentration levels should correspond to the expected range of concentrations found in real samples or should define the working range of the GC. Calibration solutions must be replaced after six months, or sooner if comparison with check standards indicates a problem.

5.4 Internal standards (if internal standard calibration is used): To use this approach, the analyst must select one or more internal standards that are similar in analytical behavior to the compounds of interest. The analyst must further demonstrate that the measurement of the internal standard is not affected by method or matrix interferences. Because of these limitations, no internal standard can be suggested that is applicable to all samples.

5.4.1 Prepare calibration standards at a minimum of five concentration levels for each analyte of interest as described in Paragraph 5.3.

5.4.2 To each calibration standard, add a known constant amount of one or more internal standards, and dilute to volume with isooctane.

5.4.3 Analyze each calibration standard according to Section 7.0.

5.5 Surrogate standards: The analyst should monitor the performance of the extraction, cleanup (when used), and analytical system and the effectiveness of the method in dealing with each sample matrix by spiking each sample, standard, and reagent water blank with one or two surrogates (e.g., chlorinated hydrocarbons that are not expected to be in the sample) recommended to encompass the range of the temperature program used in this method. Method 3500, Section 5.3.1.1, details instructions on the preparation of base/neutral surrogates. Deuterated analogs of analytes as surrogates for gas chromatographic analysis due to coelut

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6.0 SAMPLE COLLECTION, PRESERVATION, AND HANDLING

6.1 See the introductory material to this chapter, Organic Analytes, Section 4.1. Extracts must be stored under refrigeration and analyzed within 40 days of extraction.

7.0 PROCEDURE

7.1 Extraction:

7.1.1 Refer to Chapter Two for guidance on choosing the appropriate extraction procedure. In general, water samples are extracted at a neutral, or as is, pH with methylene chloride, using either Method 3510 or 3520. Solid samples are extracted using either Method 3540 or 3550.

7.1.2 Prior to gas chromatographic analysis, the extraction solvent must be exchanged to hexane. The exchange is performed during the K-D procedures listed in all of the extraction methods. The exchange is performed as follows.

7.1.2.1 Following K-D of the methylene chloride extract to 1-mL using the macro-Snyder column, allow the apparatus to cool and drain for at least 10 min.

7.1.2.2 Momentarily remove the Snyder column, add 50 mL of hexane, a new boiling chip, and reattach the macro-Snyder column. Concentrate the extract using 1 mL of hexane to prewet the Snyder column. Place the K-D apparatus on the water bath so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 1 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min. The extract will be handled differently at this point, depending on whether or not cleanup is needed. If cleanup is not required, proceed to Paragraph 7.1.2.3. If cleanup is needed, proceed to Paragraph 7.1.2.4.

7.1.2.3 If cleanup of the extract is not required, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with 1-2 mL of hexane. A 5-mL syringe is recommended for this operation. Adjust the extract volume to 10.0 mL. Stopper the concentrator tube and store refrigerated at 4°C if further processing will not be performed immediately. If the extract will be stored longer than two days, it should be transferred to a Teflon-sealed screw-cap vial. Proceed with gas chromatographic analysis.

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7.1.2.4 If cleanup of the extract is required, remove the Snyder column and rinse the flask and its lower joint into the concentrator tube with a minimum amount of hexane. A 5-mL syringe is recommended for this operation. Add a clean boiling chip to the concentrator tube and attach a two-ball micro-Snyder column. Prewet the column by adding about 0.5 mL of hexane to the top. Place the micro-K-D apparatus on the water bath (80°C) so that the concentrator tube is partially immersed in the hot water. Adjust the vertical position of the apparatus and the water temperature, as required, to complete concentration in 5-10 min. At the proper rate of distillation the balls of the column will actively chatter, but the chambers will not flood. When the apparent volume of liquid reaches 0.5 mL, remove the K-D apparatus and allow it to drain and cool for at least 10 min.

7.1.2.5 Remove the micro-Snyder column and rinse the flask and its lower joint into the concentrator tube with 0.2 mL of hexane. Adjust the extract volume to 2.0 mL and proceed with Method 3620.

7.2 Gas chromatography conditions (Recommended):

7.2.1 Column 1: Set 5% methane/95% argon carrier gas flow at 25 mL/min flow rate. Set column temperature at 65°C isothermal, unless otherwise specified (see Table 1).

7.2.2 Column 2: Set 5% methane/95% argon carrier gas flow at 25 mL/min flow rate. Set column temperature at 75°C isothermal, unless otherwise specified (see Table 1).

7.3 Calibration: Refer to Method 8000 for proper calibration techniques. Use Table 1 and especially Table 2 for guidance on selecting the lowest point on the calibration curve.

7.3.1 The procedure for internal or external calibration may be used. Refer to Method 8000 for a description of each of these procedures.

7.3.2 If cleanup is performed on the samples, the analyst should process a series of standards through the cleanup procedure and then analyze the samples by GC. This will validate elution patterns and the absence of interferents from the reagents.

7.4 Gas chromatographic analysis:

7.4.1 Refer to Method 8000. If the internal standard calibration technique is used, add 10 uL of internal standard to the sample prior to injecting.

7.4.2 Follow Section 7.6 in Method 8000 for instructions on the analysis sequence, appropriate dilutions, establishing daily retention time windows, and identification criteria. Include a mid-level standard after each group of 10 samples in the analysis sequence.

7.4.3 Examples of GC/ECD chromatograms for certain chlorinated hydrocarbons are shown in Figures 1 and 2.

7.4.4 Record the sample volume injected and the resulting peak sizes (in area units or peak heights).

7.4.4 Using either the internal or external calibration procedure (Method 8000), determine the identity and quantity of each component peak in the sample chromatogram which corresponds to the compounds used for calibration purposes. See Section 7.8 of Method 8000 for calculation equations.

7.4.5 If peak detection and identification are prevented due to interferences, the hexane extract may undergo cleanup using Method 3620.

7.5 Cleanup:

7.5.1 Proceed with Method 3620 using the 2-mL hexane extracts obtained from Paragraph 7.1.2.5.

7.5.2 Following cleanup, the extracts should be analyzed by GC, as described in the previous paragraphs and in Method 8000.

8.0 QUALITY CONTROL

8.1 Refer to Chapter One for specific quality control procedures. Quality control to validate sample extraction is covered in Method 3500 and in the extraction method utilized. If extract cleanup was performed, follow the QC in Method 3600 and in the specific cleanup method.

8.2 Procedures to check the GC system operation are found in Method 8000, Section 8.6.

8.2.1 The quality control check sample concentrate (Method 8000, Section 8.6) should contain each parameter of interest at the following concentrations in acetone: hexachloro-substituted hydrocarbon, 10 ug/mL; and any other chlorinated hydrocarbon, 100 ug/mL.

8.2.2 Table 3 indicates the calibration and QC acceptance criteria for this method. Table 4 gives method accuracy and precision as functions of concentration for the analytes of interest. The contents of both Tables should be used to evaluate a laboratory's ability to perform and generate acceptable data by this method.

8.3 Calculate surrogate standard recovery on all samples, blanks, and spikes. Determine if the recovery is within limits (limits established by performing QC procedures outlined in Method 8000, Section 8.10).

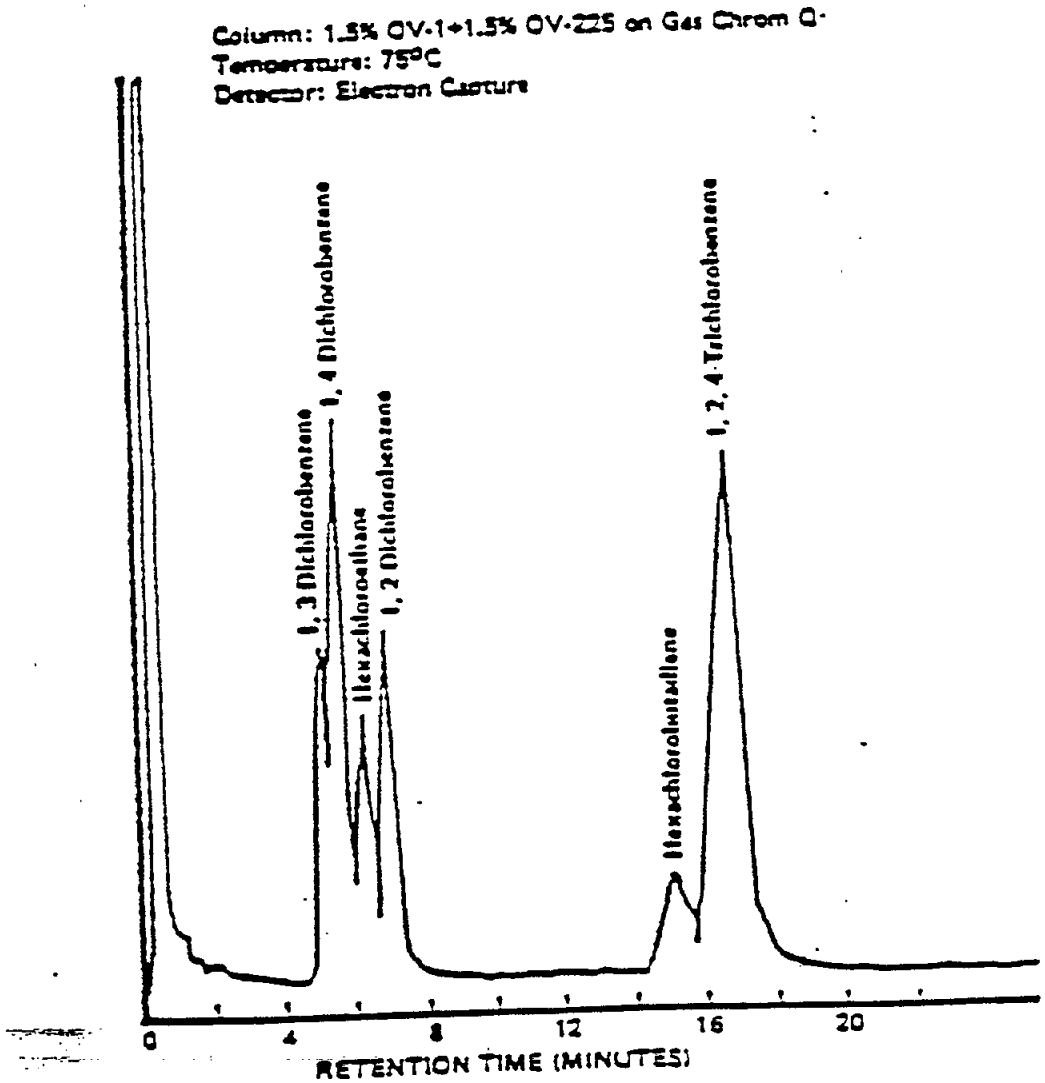

Figure 1. Gas chromatogram of chlorinated hydrocarbons (low molecular weight compounds).

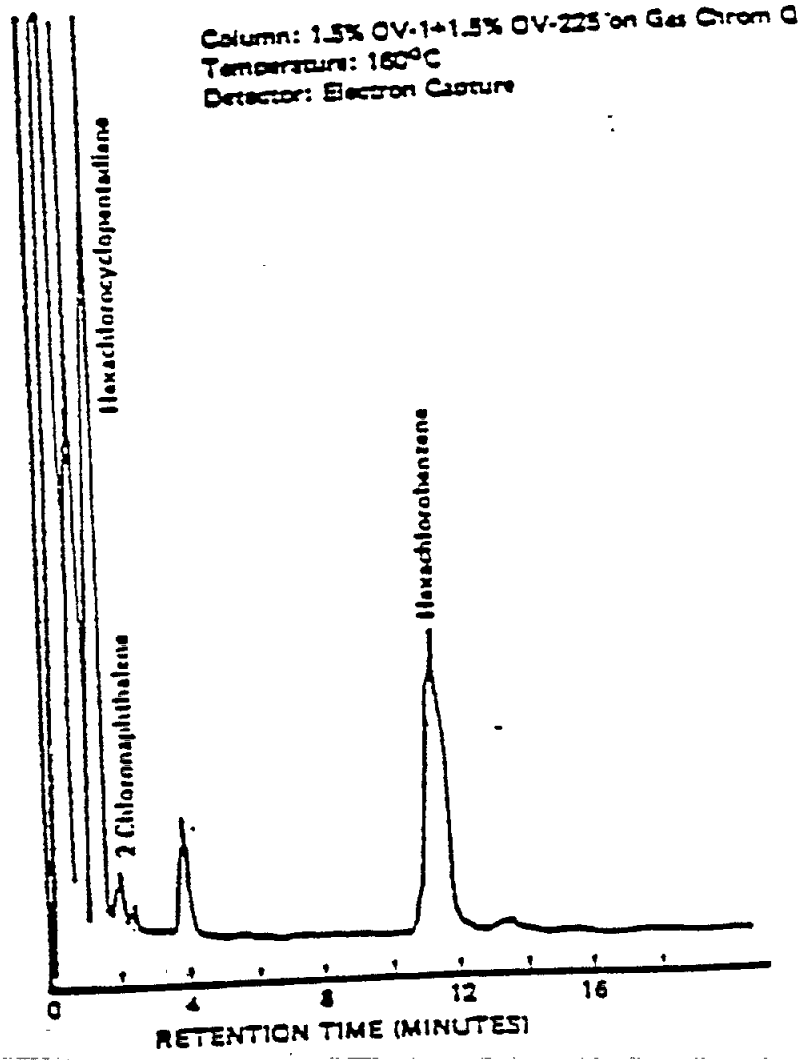

Figure 2. Gas chromatogram of chlorinated hydrocarbons (high molecular weight compounds).

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8.3.1 If recovery is not within limits, the following procedures are required.

- Check to be sure there are no errors in calculations, surrogate solutions and internal standards. Also, check instrument performance.
- Recalculate the data and/or reanalyze the extract if any of the above checks reveal a problem.
- Reextract and reanalyze the sample if none of the above are a problem or flag the data as "estimated concentration."

9.0 METHOD PERFORMANCE

9.1 The method was tested by 20 laboratories using reagent water, drinking water, surface water, and three industrial wastewaters spiked at six concentrations over the range 1.0 to 356 ug/L. Single operator precision, overall precision, and method accuracy were found to be directly related to the concentration of the parameter and essentially independent of the sample matrix. Linear equations to describe these relationships for a flame ionization detector are presented in Table 4.

9.2 The accuracy and precision obtained will be determined by the sample matrix, sample-preparation technique, and calibration procedures used.

10.0 REFERENCES

1. "Development and Application of Test Procedures for Specific Organic Toxic Substances in Wastewaters. Category 3 - Chlorinated Hydrocarbons, and Category 8 - Phenols," Report for EPA Contract 68-03-2625 (in preparation).

2. Burke, J.A. "Gas Chromatography for Pesticide Residue Analysis; Some Practical Aspects," Journal of the Association of Official Analytical Chemists, 48, 1037, 1965.

3. "EPA Method Validation Study 22, Method 612 (Chlorinated Hydrocarbons)," Report for EPA Contract 68-03-2625 (in preparation).

4. "Method Performance for Hexachlorocyclopentadiene by Method 612," Memorandum from R. Slater, U.S. Environmental Protection Agency, Environmental Monitoring and Support Laboratory, Cincinnati, Ohio 45268, December 7, 1983.

5. U.S. EPA 40 CFR Part 136, "Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act; Final Rule and Interim Final Rule and Proposed Rule," October 26, 1984.

6. Provost, L.P. and R.S. Elder, "Interpretation of Percent Recovery Data," American Laboratory, 15, pp. 58-63, 1983.

7. "Determination of Chlorinated Hydrocarbons in Indust Wastewaters," Report for EPA Contract 68-03-2625 (in preparat

TABLE 3. QC ACCEPTANCE CRITERIA[a]

Parameter	Test conc. (ug/L)	Limit for s (ug/L)	Range for $\bar{X}$ (ug/L)	Range P, P_s (%)
2-Chloronaphthalene	100	37.3	29.5-126.9	9-148
1,2-Dichlorobenzene	100	28.3	23.5-145.1	9-160
1,3-Dichlorobenzene	100	26.4	7.2-138.6	0-150
1,4-Dichlorobenzene	100	20.8	22.7-126.9	13-137
Hexachlorobenzene	10	2.4	2.6-14.8	15-159
Hexachlorobutadiene	10	2.2	0-12.7	0-139
Hexachlorocyclopentadiene	10	2.5	0-10.4	0-111
Hexachloroethane	10	3.3	2.4-12.3	8-139
1,2,4-Trichlorobenzene	100	31.6	20.2-133.7	5-149

s = Standard deviation of four recovery measurements, in ug/L.

$\bar{X}$ = Average recovery for four recovery measurements, in ug/L.

P, P_s = Percent recovery measured.

D = Detected; result must be greater than zero.

[a]Criteria from 40 CFR Part 136 for Method 612. These criteria are based directly upon the method performance data in Table 4. Where necessary, the limits for recovery have been broadened to assure applicability of the limits to concentrations below those used to develop Table 4.

Table 4. METHOD ACCURACY AND PRECISION AS FUNCTIONS OF CONCENTRATION[a]

Parameter	Accuracy, as recovery, x' (ug/L)	Single analyst precision, s_r' (ug/L)	Overall precision, S' (ug/L)
Chloronaphthalene	0.75C+3.21	0.28$\bar{X}$-1.17	0.38$\bar{X}$-1.39
1,2-Dichlorobenzene	0.85C-0.70	0.22$\bar{X}$-2.95	0.41$\bar{X}$-3.92
1,3-Dichlorobenzene	0.72C+0.87	0.21$\bar{X}$-1.03	0.49$\bar{X}$-3.98
1,4-Dichlorobenzene	0.72C+2.80	0.16$\bar{X}$-0.48	0.35$\bar{X}$-0.57
Hexachlorobenzene	0.87C-0.02	0.14$\bar{X}$+0.07	0.36$\bar{X}$-0.19
Hexachlorobutadiene	0.61C+0.03	0.18$\bar{X}$+0.08	0.53$\bar{X}$-0.12
Hexachlorocyclopentadiene[a]	0.47C	0.24$\bar{X}$	0.50$\bar{X}$
Hexachloroethane	0.74C-0.02	0.23$\bar{X}$+0.07	0.36$\bar{X}$-0.00
1,2,4-Trichlorobenzene	0.76C+0.98	0.23$\bar{X}$-0.44	0.40$\bar{X}$-1.37

x' = Expected recovery for one or more measurements of a sample containing a concentration of C, in ug/L.

s_r' = Expected single analyst standard deviation of measurements at an average concentration of $\bar{X}$, in ug/L.

S' = Expected interlaboratory standard deviation of measurements at an average concentration found of $\bar{X}$, in ug/L.

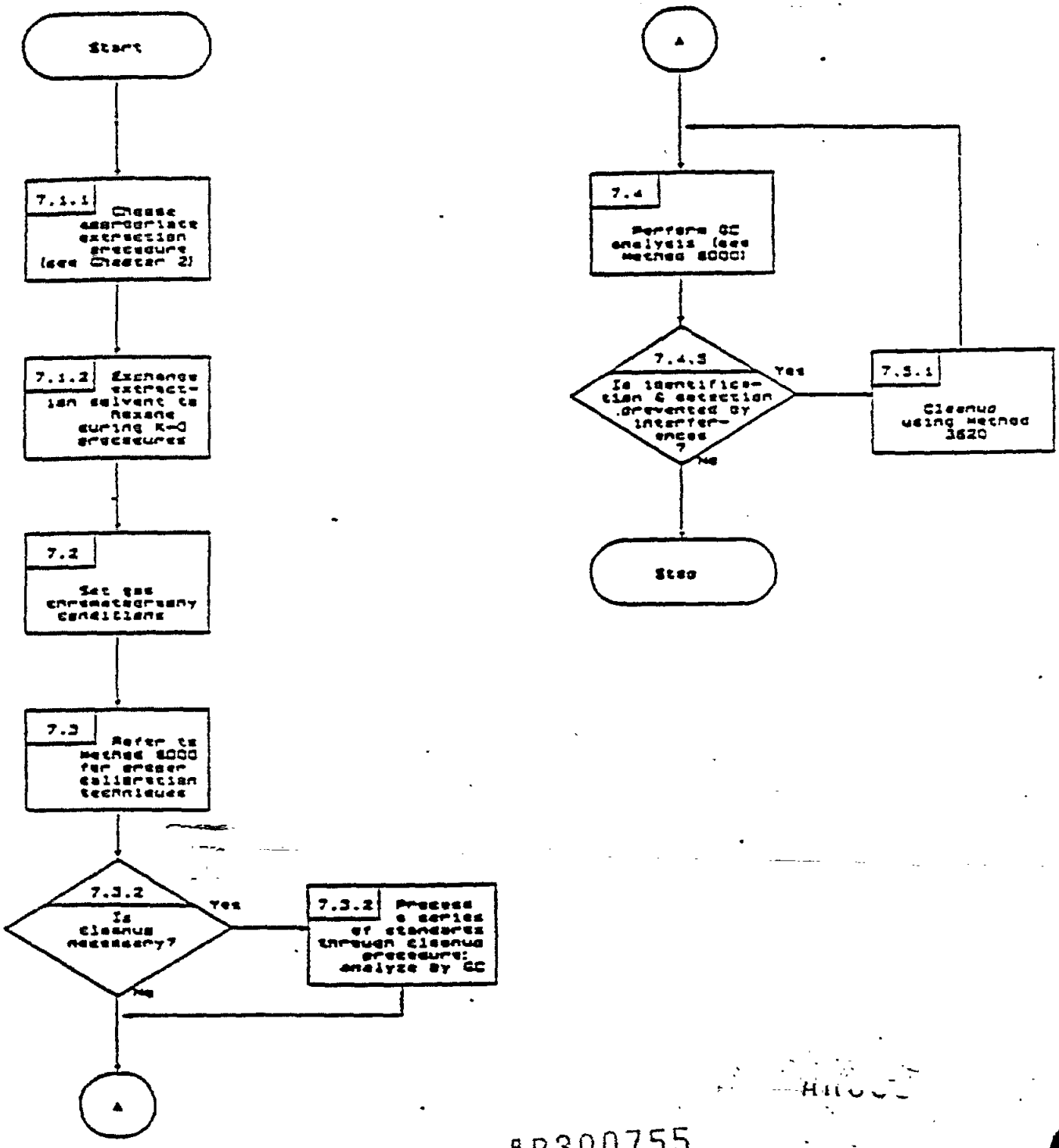
C = True value for the concentration, in ug/L.

$\bar{X}$ = Average recovery found for measurements of samples containing a concentration of C, in ug/L.

[a]Estimates based upon the performance in a single laboratory.

METHOD 8120

CHLORINATED HYDROCARBONS



AR300755

Revision 0
Date September 1986

Case Narrative
PACE Laboratories, Inc.
Contract No.: 68-W8-0019
SDG No.: 5165C-01, Task II
EPA Sample Nos.: 5165C-01 through
5165C-11

Enclosed is the data package representing the analysis of 9 soil and 2 water samples received under SAS 5165C, Task II. The samples were received January 9 and 11, 1990, and analyzed for chlorinated benzenes (EPA Method 612) by GC-ECD. Table 1 (attached) contains source and purity information for the calibration and QC check standards utilized in this SAS.

Please note the following summary comments regarding quality control:

Surrogate Recoveries All samples were initially extracted within the 10-day holding time (from sample collection) using the surrogate 2-fluorobiphenyl, as required in the SAS scope of work. However, there was no response for this compound on the GC-ECD. In a 1/16/90 phone conversation between Bill Scruton (PACE) and Stevie Wilding (Region III), Bill suggested that the samples be re-extracted (beyond the holding time) using 2-chloronaphthalene as an alternate surrogate. Additionally, since the requested soil detection limit was 330 ug/kg, he suggested that the surrogate be spiked at 1000 ug/kg rather than the 100 ug/kg stated in the scope of work. Stevie approved both requests and said there would be no penalties for the missed holding times.

The nine soils were re-extracted using 2-chloronaphthalene as the surrogate. Except for 5165C-09 Dup. and 5165C-11MS, all surrogate recoveries were within the QC Advisory Limits of 30-115%, as given for 2-fluorobiphenyl in the SAS. Only the seven soils received on 1-9-90 were beyond the holding time.

Insufficient sample remained to re-extract the two rinsates using 2-chloronaphthalene. Surrogate recoveries for these two samples were not calculated since little or no response was achieved for 2-fluorobiphenyl.

Instrumental Calibrations All initial and continuing calibration standards met the criteria required by the SAS scope of work.

For the confirmation run, the GC was only calibrated once for the 33 hour run. The % D for 1,2,3,4-tetrachlorobenzene's exceeded 20% in the check standard analyzed at the end of the run. Since this run was used for confirmation purposes only, corrective action was not taken.

Matrix Spike/Matrix Spike Duplicate (MS/MSD)

Initially, sample 5165C-01 was screened to determine spiking levels. For the re-extractions, 5165C-11 was used for the MS/MSD analyses because there was insufficient sample available to do another QC set using 5165C-01. In order to preserve the holding time of 5165C-11, this sample was not screened to determine spiking levels. Spiking was performed using levels determined in the screening of [illegible] levels proved to be insignificant when compared to the [illegible] the unspiked analysis of 5165C-11. Meaningful recoveries of the spiking compounds could not be calculated.

Case Narrative
PACE Laboratories, Inc.
Contract No.: 68-W8-0019
SDG No.: 5165C-01, Task II
EPA Sample Nos.: 5165C-01 through
5165C-11

I certify that this data package is in compliance with the terms and conditions of the contract, both technically and for completeness, for other than the conditions detailed above. Release of the data contained in this hardcopy data package and has been authorized by the Laboratory Manager or his designee, as verified by the following signature.

C Haile

Clarence L. Haile, Ph.D.
Director of Sampling and Analytical Services

9 Feb 90

Date

TABLE 1

Calibration Standards

Analyte	Supplier	Lot #	% Purity
1,2,3-Trichlorobenzene	Aldrich	JT02308PK	99
1,2,4-Trichlorobenzene	Supelco	LA 12157	99
1,3,5-Trichlorobenzene	EPA	17603	5000 ± 250 ug/ml
1,2,3,5-Tetrachlorobenzene	Riedel de Haen	8288	99
1,2,3,4-Tetrachlorobenzene	Aldrich	00330LM	90
Pentachlorobenzene	Riedel de Haen	5350	99
Hexachlorobenzene	Riedel de Haen	90790	99

QC Check Standards

Analyte	Supplier	Lot #	% Purity
1,2,3-Trichlorobenzene	EPA	17503	5000 ± 250 ug/ml
1,2,4-Trichlorobenzene	Supelco	LA23023	20 ug/ml
1,3,5-Trichlorobenzene	Aldrich	HW00718EV	99%
1,2,3,5-Tetrachlorobenzene	Supelco	LA23023	20 ug/ml
1,2,3,5-Tetrachlorobenzene	Supelco	LA23023	20 ug/ml
Pentachlorobenzene	Supelco	LA23023	20 ug/ml
Hexachlorobenzene	Supelco	LA23023	20 ug/ml

SAMPLE DELIVERY GROUP (SDG)
TRAFFIC REPORT (TR) COVER SHEET

Lab Name: PACE Laboratories Inc. Contract No.: 68-01-W8-

Lab Code: PACE Case No.: _____ SAS No.: 5165 - C

Full Sample Analysis Price in Contract: $ 136 TASK 2

SDG No./First Sample in SDG: 5165C-01-TASK2 Sample Receipt Date: 01/09/90
(Lowest EPA Sample Number in first shipment of samples received under SDG) (MM/DD/YY)

Last Sample in SDG: 5165C-11-TASK2 Sample Receipt Date: 01/11/90
(Highest EPA Sample Number in last shipment of samples received under SDG) (MM/DD/YY)

EPA Sample Numbers in the SDG (listed in alphanumeric order):

1	5165C-01-Task2	11	5165C-11-Task2
2	5165C-02-Task2	12	
3	5165C-03 Task2	13	
4	5165C-04 TASK2	14	
5	5165C-05 TASK2	15	
6	5165C-06 TASK2	16	
7	5165C-07 TASK2	17	
8	5165C-08 TASK2	18	
9	5165C-09 TASK2	19	
10	5165C-10 TASK2	20	

Note: There are a maximum of 20 field samples in an SDG.

Attach Traffic Reports to this form in alphanumeric order (i.e., the order listed on this form).

Lisa [illegible] 01/12/90
Sample Custodian Date

AR300759

Office of Enforcement

REGION 3
841 Ches[illegible] St.
Philadelphia, Penns[illegible]ania 19107

CHAIN OF CUS[illegible]DY RECORD

PROJ. NO. C03041 5303.8

PROJECT NAME: SCD

SAMPLERS: (Signature) Jax [illegible]

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION		NO. OF CONTAINERS	Tri-Through Water Chlorinated Biozetics						REMARKS JAS No. 51165C - TASK 2 TAG No.
51165C -09-TASK2	1/10/90	1029		X	CB-1	1491	1	1						3-1009092
51165C 10-TASK2	1/9/90	1110		X	EQB-3	1492	1	1						3-1009111
51165C -11-TASK2	1/10/90	1144		X	CB-2	1493	1	1						3-1009119
					SD6# = 51165C-01 TASK 2									
					Final SD6# = 51165C-11-TASK2									

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Jax [illegible]	1/10/90 1530				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature)	Date / Time	Remarks	
		[illegible]	1-11-90 10:00AM	Shipped via Federal Express AIRBILL No. 1575149321	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field Files

R300760

3 03920

ENVIRONMENTAL PROTECTION AGENCY
Office of Enforcement

REGION 3
841 Chestnut St.
Philadelphia, Pennsylvania 19107

CHAIN OF CUSTODY RECORD

PROJ. NO.: C03041 5303.8

PROJECT NAME: SCD

SAMPLERS: (Signature)

STA. NO.	DATE	TIME	COMP.	GRAB	STATION LOCATION	NO. OF CONTAINERS	Trichloroethylene Reagent						REMARKS SAS # 5165C-TASK 2 TAG NO.	
5165C-01 TASK 2	1/4/90	1410		X	WS-1 958	1	1						3-1063970	DO MS/MSD
5165C-02 TASK 2	1/4/90	1410		X	WS-2 959	1	1						3-1063974	
5165C-03 TASK 2	1/4/90	1310		X	WS-3 960	1	1						3-1063980	
5165C-04 TASK 2	1/4/90	1110		X	WS-4 961	1	1						3-1063984	
5165C-05 TASK 2	1/5/90	1218		X	WS-5 962	1	1						3-1063986	
5165C-06 TASK 2	1/5/90	1042		X	WS-6 963	1	1						3-1100878	
5165C-07 TASK 2	1/5/90	1350		X	WS-7 964	1	1						3-1100882	
5165C-08 TASK 2	1/4/90	0930		X	EQB-2 965	1	1						3-1100886	EQ BLANK
					SDG # = 5165C-01 TASK 2									
					Final SDG = 5165C-08 TASK 2									

HR300761

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
	1/8/90 1630				
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Relinquished by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)	Date / Time	Received for Laboratory by: (Signature) MJC	Date / Time 1/9/90 1000	Remarks Shipped via Federal Express Air Bill No. 4575150543	

Distribution: Original Accompanies Shipment; Copy to Coordinator Field F

U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number
5165C-T2-2

SPECIAL ANALYTICAL SERVICE

PACKING LIST

Sampling Office:	Sampling Date(s):	Ship To:	For Lab Use Only
[illegible]	[illegible]	PACE LABORATORIES, INC. 1710 Douglas Drive North Minneapolis, MN [illegible]	Date Samples Rec'd: 1/11/90
Sampling Contact: [illegible] (name)	Date Shipped: [illegible]		Received By: [illegible]
[illegible] (phone)	Site Name/Code: [illegible]	Attn: [illegible]	

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1. [illegible]	[illegible] soil [illegible]	Good
2. [illegible]	[illegible]	↓
3. [illegible]	[illegible] soil [illegible]	↓
4.		
5.		
6.		
7.		
8.	SDG# = 5165C-01 TASK2	
9.	FINAL SDG# = 5165C-11 TASK2	
10.		
11.		
12.		
13.		
14.		
15.		
16.		
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U.S. ENVIRONMENTAL PROTECTION AGENCY
CLP Sample Management Office
P.O. Box 818 - Alexandria, Virginia 22313
Phone: 703/557-2490 - FTS/557-2490

SAS Number
5165C-TASK 2

SPECIAL ANALYTICAL SERVICE
PACKING LIST

Sampling Office:	Sampling Date(s):	Ship To:	For Lab Use Only
Versar	1/4/90 - 1/5/90	PACE Laboratories, INC. 1710 Douglas Drive North Minneapolis, MN 55422	Date Samples Rec'd: 1/9/90
Sampling Contact: DAVID SPENCER (name)	Date Shipped: 1/8/90		Received By: M Connolly
(215) 741-4211 (phone)	Site Name/Code: SCD	Attn: Lisa Leither	

Sample Numbers	Sample Description i.e., Analysis, Matrix, Concentration	Sample Condition on Receipt at Lab
1. 5165C-01 TASK 2	Tri-[illegible] Hex [illegible] Soil Low	good
2. 5165C-02 TASK 2		
3. 5165C-03 TASK 2		
4. 5165C-04 TASK 2		
5. 5165C-05 TASK 2		
6. 5165C-06 TASK 2		
7. 5165C-07 TASK 2		
8. 5165C-08 TASK 2	Water	
9.		
10.		
11.	SDG # = 5165C-01 TASK2	
12.	Final SDG = 5165C-08 TASK2	
13.		
14.		
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18.		
19.		
20.		

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WESTON SM

Appendix A

Glossary of Data Qualifiers

GLOSSARY OF DATA QUALIFIER CODES (ORGANIC)

CODES RELATING TO IDENTIFICATION
(confidence concerning presence or absence of compounds):

U = Not detected. The associated number indicates approximate sample concentration necessary to be detected.

(NO CODE) = Confirmed identification.

B = Not detected substantially above the level reported in laboratory or field blanks.

R = Unreliable result. Analyte may or may not be present in the sample. Supporting data necessary to confirm result.

N = Tentative identification. Consider present. Special methods may be needed to confirm its presence or absence in future sampling efforts.

CODES RELATED TO QUANTITATION
(can be used for both positive results and sample quantitation limits):

J = Analyte present. Reported value may not be accurate or precise.

K = Analyte present. Reported value may be biased high. Actual value is expected to be lower.

L = Analyte present. Reported value may be biased low. Actual value is expected to be higher.

UJ = Not detected, quantitation limit may be inaccurate or imprecise.

UL = Not detected, quantitation limit is probably higher.

OTHER CODES

Q = No analytical result.

revised 01/90

DATA SUMMARY FORM: VOLATILES 1

Site Name: Standard Chlorine

FISH SAMPLES (ug/Kg)

Case #: SAS 5256C Task 3 Sampling Date(s): 3/5/90

To calculate sample quantitation limit: (CRQL * Dilution Factor) / ((100 - % moisture)/100)

	Sample No.	5256C-01		5256C-02																	
	Dilution Factor	1.0		1.0																	
	% Moisture	78		80																	
	Location	F-1		F-2																	
CRQL	COMPOUND																				
10	Chloromethane		UJ		UJ																
10	Bromomethane		↓		↓																
10	Vinyl Chloride		↓		↓																
10	Chloroethane		↓		↓																
5	Methylene Chloride	51	B	65	B																
10	Acetone	240	B	870	J																
5	Carbon Disulfide	24	J	130	J																
5	1,1-Dichloroethene		UJ		UJ																
5	1,1-Dichloroethane		↓		↓																
5	Total-1,2-Dichloroethene		↓		↓																
5	Chloroform		↓		↓																
5	1,2-Dichloroethane		↓		↓																
10	2-Butanone		R		R																
5	1,1,1-Trichloroethane		UJ		UJ																
5	Carbon Tetrachloride		↓		↓																
10	Vinyl Acetate		↓		↓																
5	Bromodichloromethane		↓		↓																

CRQL = Contract Required Detection Limit

SEE NARRATIVE FOR CODE DEFINITIONS

revised 12/88

AR300766