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OAK RIDGE NATIONAL LABORATORY

LOCKHEED MARTIN

Environmental Sciences Division

Laboratory Evaluation of In Situ Chemical Oxidation for Groundwater Remediation, Test Area North, Operable Unit 1-07B, Idaho National Engineering and Environmental Laboratory

Volume Three - Appendix F

S. R. Cline
J. M. Giaquinto
M. K. McCracken
D. L. Denton
R. C. Starr

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ENVIRONMENTAL SCIENCES DIVISION

LABORATORY EVALUATION OF IN SITU CHEMICAL OXIDATION FOR GROUNDWATER REMEDIATION, TEST AREA NORTH, OPERABLE UNIT 1-07B, IDAHO NATIONAL ENGINEERING AND ENVIRONMENTAL LABORATORY

Volume Three - Appendix F

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Environmental Sciences Division
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PREFACE

This appendix supports the results and discussion of the laboratory work performed to evaluate the feasibility of in situ chemical oxidation for Idaho National Environmental and Engineering Laboratory's (INEEL) Test Area North (TAN) which is contained in ORNL/TM-13711/V1. This volume contains Appendix F.

Appendix F is essentially a photocopy of the ORNL researchers' laboratory notebooks from the Environmental Sciences Division (ESD) and the Radioactive Materials Analytical Laboratory (RMAL).

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Research and Technical Notebook No. _____

Assigned to: _____ Date: _____

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Research/Technical Descriptions(s)/	Inclusive Dates	Pages

Preparation of Calibration Standards

Prepared 01/14/98 by K. McCracken

Source:
J.T. Baker PURE TCE 1.456g/mL

Prepare 1000 ppm STOCK Solution
Add 17.2 µL PURE TCE to 25mL Flask and fill to volume with hexane.
(Weigh and record syringe when full and then when empty)

STDS Preparation

STD5 actual
4000 ppb 4389 ppb
Add 100 µL STOCK to hexane in 25 mL Flask

1/28/98 KM

STD4
1000 ppb 1096 ppb
Add 2.5 mL of the STD5 into 10 mL Flask and fill to volume with hexane

STD3
200 ppb 219.2 ppb
Add 0.5 mL of STD5 into 10 mL Flask and fill to volume with hexane

STD2
100 ppb 109.6 ppb
Add 0.25 mL of STD5 into 10 mL Flask and fill to volume with hexane

STD 1
10 ppb 10.96 ppb
Add 0.025 mL (25 microliters) of STD5 into 10 mL Flask and fill to volume with hexane

Place these standards in vials for storage in the 211 Freezer

17.2 µl.
syringe + TCE = 14.3559 18.82 mL actually added
syringe = 14.3285

UCN-6201

Subject Task 2a Experiment-1

1

Date 01 Month 21 Day 19 98 Year

Task 2a Experiment Descriptions

INEEL TAN -Site Chemox Treatability Study
Task Number: 2a

KM 1/20/98

Purpose/Scope:
The following laboratory experiments will evaluate the performance of the test reactors planned for use during the treatability reaction studies. In particular, the amount of TCE expected to be lost from the reactor system will be quantified via a mass balance approach.

Experiment 2a-1. Evaluation of the rubber septum to reduce TCE losses.
(To be conducted at ORNL/ESD)
One or more of the reactor sampling ports may be fitted with a thick rubber septum for the purpose of sample collection via a wide bore needle. Thus, this experiment will assess how well the fitting between the septum and the ground glass port opening minimizes TCE losses. The effect of performing repeated aliquot sampling through the septum will also be evaluated.

To focus this experiment on septum performance only, a 500 mL reactor with multiple port openings will not be used here. Instead, a 100 mL erlenmeyer flask or equivalent having only a single 24/40 std taper opening will be used. This experiment will be conducted at room temperature.

KM 1/21/98

1. A 100 mg/L aqueous TCE solution (~~≈200~~ 660mL mL) will be prepared and allowed to equilibrate for at least 30 minutes. The solution will be prepared in a Tedlar Bag using DI water and pure TCE. Measure the mass of spiking syringe filled and then after spiking to record the total TCE mass added to the reactor (a QC check on the initial TCE analysis). Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook or equivalent (project data sheets).

2. Dispense the solution into 2 tared test containers, re-weigh and record the mass of TCE spike solution added to each. Duplicate 0.5 mL samples of the spike solution will be collected immediately before the septa is added to the test containers in order to determine the initial TCE concentration.

3. The aliquot samples will be added to a pre-weighed sample container holding a known volume of hexane. Each sample container will be weighed again after sample addition to determine the volume of sample added. (A calibration check of the gravimetric scale will be performed each day of use.) All such extracts will be placed on a horizontal shaker for 30 minutes then diluted as appropriate for G.C. analysis. Store the extracts at 4°C until G.C. analysis.

4. Record the time that these duplicate samples are collected and the septum is placed onto each container. (Complete zero headspace conditions cannot be achieved here since excess water cannot be expelled from the containers as the septa are applied.)

Stock

Tedlar bag empty 40.64 g
+
H_2O 715.35 g

H_2O only 674.71 g

To get 100 mg/L TCE, need 67.47 mg TCE or 46.3 µl TCE pure

syringe 12.1834
Syringe TCE 12.2520

∴ added ~~63.6 µg~~ SPC mg TCE to Tedlar bag.

S.R. Cline Last Updated: 01/21/98 Filename: 2a-Experimental Descriptions.doc

Continued on page ~~47~~ KM 1/28/98

K. McCracken 01/21/98
Recorded by Date

SRC 1/27/98
Read and understood by Date

Subject Task 2a Experiment -1

2

Date 01 21 1998
Month Day Year

5. After 2 hours, collect duplicate aqueous samples from one of the containers by puncturing the septum with the sampling syringe (wide bore needle desired since later experiments will be soil/sludge slurries). The septum will have to be punctured by a second needle in order to pull the samples into the syringe body. Do nothing to the other test container.

6. Extract and dilute the samples as needed (described above).

7. Repeat this sampling technique at least 5 more times over a period of 24 hours for the 1st test container.

8. Collect duplicate aqueous samples from the second test container after 24 hours. (This will be the first time that the septum of the 2nd test container is punctured.

9. Analyze the extracts via GC/ECD for TCE quantification. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks, etc. are also analyzed on each analysis day.

10. Prepare a TCE mass balance to determine total mass of TCE not accounted for in either the remaining spike solution in the vial or in the sample aliquots for each test container.

Bottle 2a-1-B1
Rubber + septum
empty 123.70
+ TCE soln. 370.25

Bottle 2a-1-B2
Rubber + septum
empty 124.08
+ TCE soln. 367.83

TCE soln 246.55 ml TCE soln. 243.75 ml

	Sample Time	GC #	wt. empty	wt. + hexane	wt. + sample	sample source & # dup (a or b)
1/21/98	1410 = T_0	2a-1-1	14.0290	16.9710	17.9169	2a-1-B1-1a
	1410	2a-1-2	14.0229	17.2968	17.7729	2a-1-B1-1b
	1410	2a-1-3	14.0835	17.3780	17.8779	2a-1-B2-1a
	1410	2a-1-4	14.1063	17.3729	17.8655	2a-1-B2-1b
	1610 = T_2	2a-1-5	14.0712	17.3352	17.8412	2a-1-B2-2a
	1610	2a-1-6	14.9576	17.1981	17.7083	2a-1-B2-2b * permanganate contamination
	1830	2a-1-7	14.0295	17.3056	17.9886	2a-1-B2-3a
	1832	2a-1-8	14.1076	17.4357	17.9975	2a-1-B2-3b
1/22/98	0850	2a-1-9	14.0783	17.1861	17.6798	2a-1-B2-4a
	0850	2a-1-10	14.0940	17.3172	17.7549	2a-1-B2-4b
	1030	2a-1-11	14.1498	17.3892	17.8422	2a-1-B2-5a
	1030	2a-1-12	14.1181	17.3491	18.0028	2a-1-B2-5b

Continued on page 8

K. McCracken 01/21/98
Recorded by Date

SRC 1/27/98
Read and understood by Date

Su .ct Task 2a Experiment-1

Date 01 Month 21 Day 19 98 Year

sample time	#	wt empty	wt + hexane	wt + sample	sample source & dup #
1250	2a-1-13	14.0530	17.2536	17.7149	2a-1-B2-6a
1250	2a-1-14	14.1005	17.3340	17.8066	2a-1-B2-6b
1415	2a-1-15	14.0660	17.2893	17.7266	2a-1-B1-2a
1415	2a-1-16	14.1735	17.4015	17.9163	2a-1-B1-2b
1415	2a-1-17	14.1089	17.3199	17.7715	2a-1-B2-7a
1415	2a-1-18	13.9603	17.1730	17.7070	2a-1-B2-7b

Samples on shaker in RM 291 — 1420 to 1450.

$\frac{100 mg}{L H_2O} \times 0.0005 L_{H_2O} = \frac{0.05 mg\ TCE}{0.005 L\ hexane} = 10 ppm$ in Hexane

dilute all samples as follows:
100 µL sample into 1 mL hexane = df 11

GC sample order
1. Blk = 1mL hexane
2. Std = 1ppm TCE in hexane
3-12 = samples 1-10
13. Blk
14-21 = samples 11-18.

NO MORE ENTRIES SRC 1/27/98

Continued on page 2a

K. McCracken — Recorded by — 01/21/98 — Date

SR Cla — Read and understood by — 1/27/98 — Date

Subject Experiment 2a-2

Date 01 (Month) 28 (Day) 19 98 (Year)

Experiment 2a-2. Evaluation of the pressure relief fitting to minimize and quantify TCE losses

(To be conducted at ORNL/ESD)

A shakedown experiment already conducted has shown that a thin layer of Parafilm M material (wax based) is easily ruptured when placed on an otherwise sealed container in less time than required to remove a rubber septum when a constant volume and rate of air is added to the container. However, a high degree of TCE diffusion is suspected through this type of material. A similar test was performed using aluminum foil as the thin film covering the bottom of the adapter. While desirable in the fact that TCE diffusion is likely minimal, the aluminum foil possessed a great deal of tensile strength in this configuration and could not be easily ruptured by pressurization of an erlenmeyer flask. For the purpose of this experiment four (4) different conditions will be evaluated: (a) no thin film, (b) Parafilm M, (c) aluminum foil punctured with a single pinhole (to reduce tensile strength), and (d) DuraSeal™, a solvent resistant LDPE film. Erlenmeyer flasks (125 mL) or equivalent containers having a 24/40 std. Taper opening will be used in this experiment. There will not be an attempt to pressurize these containers. The "no thin film" set-up will represent the ruptured film case. This experiment will be conducted at room temperature.

1. Prepare 24/40 std. Taper adapters as described above (a-d), securing the edges of each thin film by addition of a Teflon sleeve onto the ground glass fitting.

2. Assemble the ORBO-32 Tubes. Two ORBO-32 tubes will be used in series for each container in the event that the TCE adsorption capacity of the first tube is exceeded. Using Pliers, break the ends of two ORBO tubes. Smooth the broken ends with a file and connect them in series using tygon tubing. Orient them such that the "A" charcoal bed of each tube is first in line. Weigh bottles + relief fitting assemblies before & after addition of TCE soln. ["A" is the larger of the two (100 mg charcoal), "B" contains 50 mg charcoal.]
NOTE: STORE THE OPENED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

3. A 100 mg/L aqueous TCE solution (~~=100 mL~~ 1200 mL) will be prepared in a Tedlar bag and allowed to equilibrate ~~for at least 30 minutes~~ overnight prior to being distributed into each container. (Preparing only one spiking solution, allows for each test set-up to have the same initial conditions). Measure the mass of spiking syringe when filled and then after spiking to record the total TCE mass added to the reactor (a QC check on the initial TCE analysis). Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook or equivalent (project data sheets).

4. Duplicate 0.5 mL samples of the spike solution will be collected immediately before the containers are sealed in order to determine the initial TCE concentration.

5. The duplicate aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane. Each sample container will be weighed again after sample addition to determine the volume of sample added. (A calibration check of the gravimetric scale will be

KM 1/28/98

Used this one since the difference is water drops

Tedlar Bag empty ~~41.70g~~ 47.23g

Bag + 1200 ml H_2O ~~1241.85g~~ 1241.16g

syringe + 82.5 µl TCE ~~12.5656g~~ 12.3000g

syringe empty ~~12.1830g~~ 12.1828g

= ~~120~~ 118 mg TCE / ~~1200~~ 1194 mL H_2O

allow TCE to equilibrate in H_2O overnight.

6098 mg/mL

	Bottle 2a-2-B1 (no seal)	2a-2-B2 (parafilm)	2a-2-B3 (aluminum foil)	2a-2-B4 (duraseal)
wt. empty	139.21g	145.45g	145.25g	148.99g
+ H_2O	394.72	403.06	398.42	405.16
TCE				

Continued on page 10

K McCracken — Recorded by — Date 1/28/98

SRO — Read and understood by — Date 2/3/98

Subject Experiment 2a-2

Date 01 Month 28-29 Day 19 98 Year

performed each day of use.) All such extracts will be placed on a horizontal shaker for 30 minutes. Store the extracts at 4°C until G.C. analysis.

6. Record the time at which the sample is collected and the containers are closed.
6b → 24 Sample Indupl: cate after t = 4hrs (Record time) SLC 2/3/98 KM 1/28/98

7. After ~~4~~ hours, remove the adapter from each container and collect an aqueous sample (0.5 mL) Extract and dilute the samples as needed for G.C. analysis for eventual analysis of the remaining TCE concentration in the solution. Store the extracts at 4°C until G.C. analysis.

8. For each container, separate the contents of each charcoal bed into separate vials pre-weighed with a known volume of hexane then reweigh the vials to determine the mass of charcoal present.

9. Extract these samples for 30 minutes and dilute as needed for G.C. analysis as done for the aqueous aliquots.

10. Analyze the extracts via GC/ECD for TCE quantification. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks, etc. are also analyzed on each analysis day. Formal data packages, as outlined in Section 2.4.3.3 of the Chapter 2 Implementation Plan will not be prepared for this experiment.

11. Prepare a TCE mass balance on each container to determine which thin film configuration best minimized the mass of TCE that escaped from the reaction chamber. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

2a-2-B1 TCE soln. 2a-2-B2 2a-2-B3 2a-2-B4

Sample Time	GC #	wt. empty	wt. + hexane	wt. + sample	Sample source & dupl. #	GC (w/hexane) dilution
1/29 1140	19	14.1825	17.4569	17.9488	2a-2-B1-1a	101
1140	20	14.0933	17.4081	17.9004	2a-2-B1-1b	
1142	21	14.1210	17.4194	17.9159	2a-2-B2-1a	
1142	22	13.9452	17.2270	17.7236	2a-2-B2-1b	
1144	23	14.0964	17.3710	17.8675	2a-2-B3-1a	
1144	24	14.0570	17.3140	17.8110	2a-2-B3-1b	
1146	25	14.0668	17.3308	17.8269	2a-2-B4-1a	
1146	26	14.1199	17.3664	17.8642	2a-2-B4-1b	
1545	27	14.2159	17.4678	17.9660	2a-2-B1-2a	
1545	28	14.1485	17.4326	17.9282	2a-2-B1-2b	
1546	29	14.0122	17.2946	17.7933	2a-2-B2-2a	
1546	30	14.1899	17.4559	17.9500	2a-2-B2-2b	

KM 1/29

Continued on page 11

K. McCracken — Recorded by

1/28/98-1/29/98 — Date

SPC — Read and understood by

2/3/98 — Date

Subject Experiment 2a-2

Date 01 (Month) 29 (Day) 19 98 (Year)

Sample Time	GC #	wt. empty	wt. + hexane	wt. + sample	Sample source & dupl. #	GC dilution (w/hexane)
1547	31	14.0147	17.2630	17.7519	2a-2-B3-2a	101
1547	32	14.0829	17.3363	17.8315	2a-2-B3-2b	
1548	33	14.0939	17.3380	17.8334	2a-2-B4-2a	
1548	34	14.0971	17.3428	17.8394	2a-2-B4-2b	
1139 1140	35	14.1284	17.3813	17.8725	2a-2-B1-3a	
1140	36	14.1348	17.3710	17.8653	2a-2-B1-3b	
1142	37	13.9245	17.1452	17.6405	2a-2-B2-3a	
1142	38	13.9416	17.1711	17.6644	2a-2-B2-3b	
1144	39	14.0788	17.3004	17.7961	2a-2-B3-3a	
1144	40	14.0158	17.2433	17.7382	2a-2-B3-3b	
1146	41	13.9634	17.1861	17.6771	2a-2-B4-3a	
1146	42	14.0174	17.2377	17.7320	2a-2-B4-3b	
1150	43	14.0259	17.2~~3426~~ 2044	17.3414	2a-2-B1-f1-1	~~101~~ SRC Red. 1:10 to 201 2/3/98
1150	44	13.9729	17.1954	17.2400	2a-2-B1-f1-2	—
1150	45	13.9321	17.2015	17.2997	2a-2-B1-f-21	—
1152	46	14.0642	17.3043	17.3458	2a-2-B1-f2-2	—
1152	47	14.0318	17.2516	17.3474	2a-2-B2-f1-1	~~101~~ SRC Red. 1:10 to 201
1152	48	14.0499	17.2736	17.3157	2a-2-B2-f1-2	—
1152	49	14.1268	17.3608	17.4600	2a-2-B2-f2-1	—
1154	50	13.9838	17.2020	17.2446	2a-2-B2-f2-2	—
1154	51	13.9785	17.1911	17.2957	2a-2-B3-f1-1	~~101~~ SRC Red. 1:10 to 201
1154	52	13.8972	17.0954	17.~~2973~~ 1773	2a-2-B3-f1-2	—
1156	53	14.1561	17.3808	17.4206	2a-2-B3-f2-2	—
1156	54	14.1025	17.3058	17.4271	2a-2-B3-f2-1	—
1158	55	14.2177	17.4606	17.5644	2a-2-B4-f1-1	~~101~~ SRC Red. 1:10 to 201
1158	56	13.8473	17.0726	17.1169	2a-2-B4-f1-2	—
1200	57	13.9677	17.2053	17.3077	2a-2-B4-f2-1	—
1200	58	14.2637	17.4996	17.5427	2a-2-B4-f-2-2	—

GC sample order

1. Blank
2. 5ppb VOC custom Std.
3. 10 ppb " "
4. 50ppb VOC custom Std
5. 100ppb VOC custom Std
6. 500 ppb " "
7. 1000ppb " "
8.– 47. Samples 19-58

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Subject Experiment 2a-2

Date 02 (Month) 02 (Day) 19 98 (Year)

Dilutions were made on 3 samples listed below.

GC#	sample source	dilution w/hexane
43R	2a-2-B1-f1-1	201
51R	2a-2-B3-f1-1	201
55R	2a-2-B4-f1-1	201

GC sample order

1. Blank
2. 100ppb TCE custom std.
3-5 $43^R, 51^R, 55^R$, Respectively

KM 2/2/98

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Subject Preparation of Calibration Standards

Date 01 (Month) 30 (Day) 19 98 (Year)

Preparation of Calibration Standards (From VOC custom mixes)

Source:
Supelco Custom Mix Standard, 200 mg/L each Compound

DATE PREPARED 1/30/98 WH

LOT No. LA 73307

Prepare 10 mg/L working STOCK Solution
Add 0.5 mL of Custom Mix (Ampule) to 10 mL Flask filled to volume with hexane
(Weigh and record syringe when full and then when empty)

SYRINGE FULL: 18.2233
SYRINGE EMPTY: 17.8206
} 0.4027g methanol sg = 0.78
0.5163 mL added

Preparation of Calibration Standards ∴ (.5163)(200 ppm) = (10 mL)(X ppm)
STOCK = 10.326 ppm

1000 ppb
Add 2.5 mL STOCK Solution to hexane in 25 mL Flask 1033 ppb

500 ppb
Add 5 mL of 1000 PPB STD into 10 mL Flask and fill to volume with hexane 516.3 ppb

100 ppb
Add 1 mL 1000 PPB STD into 10 mL Flask and fill to volume with hexane 103.3 ppb

50 ppb
Add 0.5 mL 1000 PPB STD into 10 mL Flask and fill to volume with hexane 51.63 ppb

10 ppb
Add 0.1 mL of 1000 PPB STD into 10 mL Flask and fill to volume with hexane 10.33 ppb

5 ppb
Add 0.05 mL (50 µL) of 1000 PPB STD into 10 mL Flask and fill to volume with hexane 5.16 ppb

Place these standards in vials for storage in the 211 Freezer. cannot locate! SRC
Place the Certificate of Analysis into the Laboratory Research Notebook.

KM 1/30/98

prepared by William Holden.

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Subject Experiment 2a-3

Date 2 (Month) 13 (Day) 19 98 (Year)

Experiment 2a-3. Overall performance of the test reactor

(To be conducted at ORNL/ESD)

In this experiment a complete reactor will be set up using the most optimal component(s) from the previous Task 2a experiments described above. To be consistent, a 100 mg/L TCE spike solution will be used here as well. This experiment will be conducted at approximately 12-15°C.

1. Set up a glass Kettle reactor, ensuring that Teflon sleeves are fitted onto each of the glass adapters to be used. The top of each adapter will be sealed (Teflon stopper) during spiking and equilibration to minimize the mass of TCE lost.

2. Remove one of the stoppers and fill the reactor with distilled water, minimizing the headspace volume inside of the reactor. The volume created by the "convex" shaped lid is also filled. A record of the DI water volume added will be maintained.

3. Spike the reactor with TCE. Desired Concentration: 100 mg/L
(Add a mass of pure phase TCE (density of 1.456 g/mL) to the liquid volume inside of the reactor that will yield a TCE solution of 100 mg/L. Measure the mass of the spiking syringe when filled and then after spiking to record the total TCE mass added to the reactor (a QC check on the initial TCE analysis). Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook or equivalent (project data sheets).

4. Replace the removed stopper back onto the adapter and allow the solution to equilibrate *at least* overnight in an incubator maintained at ~~12-15°C~~ 11-13°C. (Record the equilibration period and temperature).

5. During equilibration period, prepare an ORBO-32 assembly in the standard taper adapter and cover the bottom end of the adapter with the selected thin film [parafilm]. Only one desorption tube will be used. Since this particular experiment will not be conducted under pressurization, only one adapter will need to be fitted as a pressure relief adapter.
STORE UN-SEALED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

6. After equilibration, collect duplicate aqueous samples from the reactor (from the glass adapter that is sealed with a stopper) The aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane. Each sample container will be weighed again after sample addition to determine the volume of sample added. (A calibration check of the gravimetric scale will be performed each day of use.) All such extracts will be placed on a horizontal shaker for 30 minutes. Store the extracts at 4°C until G.C. analysis.

7. Replace one of the adapters sealed with a stopper with the one fitted with the charcoal desorption tube assembly from Step 5, Record the time the samples in the step above are collected and the desorption tube is added as Start Time=0.

Kettle reactor wt. 506.69 g
Reactor + H_2O 957.42

water volume 450.73 ml

1.456 g/ml = TCE density

$\frac{100 mg}{1000 ml} \cdot 450.7 ml = 45.07 mg$

$45.07 mg \cdot \frac{1000 \mu l}{1456 mg} = 29.0 \mu l$ TCE

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Tested 2/16/98 KM

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Subject Experiment 2a-3

Date 2 (Month) 13 (Day) 19 98 (Year)

F.M. 2/13/98

8. Similarly, samples will be collected at the following time intervals: 30 min, 60 min, 2 hrs, 4 hrs, 24 hrs *(duplicate)*, 30 hrs, 48 hrs. The exact time at which each aliquot is collected will be recorded.

9. Breakdown the reactor after the last set of duplicate samples is collected. The charcoal desorption tube will then be removed from the reactor. Each charcoal bed contained in the tube will be placed into a pre-filled and pre-weighed vial containing a known volume of hexane. These extracts will be processed and analyzed in the same manner as the aqueous aliquots.

NOTE: The entire contents of the reactors will be extracted at the end of the experiments in Task 4 but is not necessary here since a solid phase is not present.

10. Analyze the samples and charcoal extracts via GC/ECD for TCE. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks etc. are also analyzed on each analysis day. Formal data packages, as outlined in Section 2.4.3.3 of the Chapter 2 Implementation Plan will not be prepared for this experiment.

11. Perform a mass balance for the reactor, taking sample aliquot volumes, initial concentrations, etc. into account to determine the overall reactor performance to maintain gas-tight conditions. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

syringe + TCE 14.3715 g

syringe after 14.3272 g

44.3 mg TCE added to Reactor on 2/13/98 at 1450.

Reactor + aqueous TCE placed in incubator in 1505, Rm 377. Incubator temp. = 11.5 °C.

KM 2/13/98

2/16/98

prepare single orBo Tube for use in Reactor
cover inside end of adapter w/ parafilm & add teflon sleeve.

End

Sample Time	GC#	WT. Empty	WT + hexane	WT. + Sample	Sample Solution Dup#	GC dilution
	59		18.2188			
	60		18.2211			
	61		18.1303		2/16/98 KM	
	62		18.2007			
	63		18.1561			
	64		18.1625			
	65		18.2471			
	66		18.1524			
	67		18.1312			
	68		18.2036			

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SLO — Read and understood by — 2/13/98 — Date

Subject Experiment 2b-1

Date 02 (Month) 17 (Day) 1998 (Year)

2.2 Determine Time Required for Hexane Extraction of TCE from Organic Sludge, Sediment, and Basalt (Task 2b)

A 30 minute extraction time period was used for the Task 2a Reactor Development experiments involving only aqueous TCE solutions; however, this task will help determine what the most effective extraction period is for each medium being evaluated: groundwater or groundwater with crushed basalt, aggregate basalt (following experiment 2c-1), sludge, or sediment at a 4:1 Liquid/Solid ratio. (Note that the hexane extraction period for aggregate basalt cannot be evaluated until after Task 2c, "TCE equilibration with aggregate basalt" is completed.) In this batch experiment, several aliquot samples will be taken with time as a TCE spiked mixture is being extracted with hexane. Samples will be taken of both the solvent and aqueous phases. The aqueous phase will be further evaluated by determining the fraction of TCE present in both the liquid and solid phases at each time interval. All media to be used in this experiment will be oven-dried, and the groundwater will be sparged to remove any initial TCE in order to conduct this test under controlled laboratory conditions. Each media type will be evaluated in duplicate. For these experiments, only a single initial TCE concentration will evaluated: 1 mg/L TCE for the reactive zone media and 100 mg/L TCE for the hot spot media. The contaminant equilibration period will be conducted at 12-15°C; however, the hexane extraction portion of the experiment will be conducted at room temperature.

Experiment 2b-1. Hexane Extraction Test for the Reactive Zone Media

(To be conducted at ORNL/ESD)

1. Prepare a STOCK (Saturated) TCE solution (2000 mg/L) by adding 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and allow to equilibrate overnight at room temperature. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.)

empty syringe 17.1757 g

+ TCE 17.3569 g

after TCE 17.1853

INTO 5 ml d.i. H_2O. R.T. overnight to equilibrate

.1716 g TCE added

∴ .118 mL added

2. Obtain the tare weight of the container to be used to prepare the spike solution (tedlar bag, flask, or equivalent). Fill with approximately 300 mL of groundwater from TAN-40 previously sparged of any residual TCE or other VOCs that may have been present. (Properly indicate usage of this media in the treatability study tracking logbook.). By gravimetric methods, determine the amount of groundwater added to the container, and then place in an environmental chamber at 12-15 °C overnight. (A calibration check of the gravimetric scale will be performed each day of use.)

3. After temperature equilibration of the groundwater, remove the container from the incubator and add the appropriate quantity of the Saturated STOCK TCE solution to produce a TCE spike concentration of 1.0 mg/L. (Reactive zone media will only be spiked at 0.1 and 1 mg/L TCE during the Task 4 degradation studies.) This solution can be mixed and equilibrated within a few minutes.

4. After mixing, obtain duplicate aqueous samples (= ~~1~~ 350 mL) of the spike solution to obtain the initial TCE concentration of the spiked groundwater. The duplicate aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (= ~~1~~ 350 mL). Each sample container will be weighed again after sample addition to determine the actual volume of

1. ~350 ml TAN 40 Groundwater into 500 ml beaker – 10:35 ↓ 13:35
   + airsparge using building air + airstone
   RT.
2. Cover beaker w/ parafilm. Place in 15°C incubator overnight
3. ~25 g sediment → drying in oven ~80°C overnight
   ~28 g CRUSHED BASALT ↗

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Subject Experiment 2b-1

Date 02 17 1998
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sample added. These extracts will be placed on a horizontal shaker for 30 minutes. Store the extracts at 4°C until G.C. analysis.

5. Obtain the tare weight of six (6) 40 mL VOA vials. Add approximately 10 g of crushed (< 1/8" size fraction) basalt to two of these vials, and approximately 10 g of sediment to two more of the vials. These materials should be previously dried to remove any residual TCE that might have been present. Re-weigh the vials to determine the exact masses added. (Properly indicate usage of this media in the treatability study tracking logbook.)

6. Completely fill the remaining volume of the 40 mL vials with the spiked groundwater, and reweigh each vial to determine the total volume of spike solution added. Record the time each vial is filled and capped. Place the vials on an orbital shaker maintained at 12-15°C and allow the spike solution and media (if applicable) to equilibrate overnight.

7. Obtain a tare weight on six empty containers to be used for the hexane extractions (125 mL VOA bottles with Teflon lined caps). Add 40 mL of hexane to each of these and reweigh.

8. Remove the 40 mL vials from the shaker/incubator. Pour the entire contents of each 40 mL vial into a separate 125 mL extraction bottle. Reweigh each extraction bottle and record the time as "extraction time=0". (The amount of TCE that may have been lost during contaminant equilibration will not be directly determined, but would be expected to be approximately the same for each 40 mL vial.)

9. Place each extraction jar on a horizontal reciprocating shaker at room temperature. Collect a 1 mL aliquot of the hexane layer from each extraction bottle at the following time periods: 5 minutes, 30 minutes, 1 hour, ~~3 hours,~~ and 24 hrs. Place these samples directly into a GC Vial (2 mL). (Calibrate the pipette used to collect the aliquot) Record the time the sample was collected. Store at 4°C until analyzed (no dilution will be necessary). KM changed sampling timepts. 2/19

10. Also collect (using a disposable syringe with a luer-lok tip) a ~~10~~ 6 mL aliquot of the aqueous layer from the extraction bottle at the same time periods used in Step 9: 5 minutes, 30 minutes, 1 hour, 3 hours, and 24 hrs. Dispense ½ of the aliquot (≈3 mL) directly into a into 12 mL VOA vial or equivalent that is pre-weighed and pre-filled with hexane (≈3 mL). Using a 0.45µm syringe filter, filter the remaining aliquot volume in the syringe (≈3 mL) into another 12 mL VOA vial or equivalent that is pre-weighed and pre-filled with hexane (≈3 mL). KM changed volumes 2/19

11. Re-weigh each of these 12 mL VOA vials in the above step to determine the total sample volume added to each. Place these samples on a reciprocating shaker for at least 30 minutes. Store these extractions at 4°C if not analyzed immediately.

12. Analyze via GC/ECD for TCE, diluting if necessary. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks, etc. are also analyzed on each analysis day. Formal data packages, as outlined in Section 2.4.3.3 of the Chapter 2 Implementation Plan will not be prepared for this experiment.

13. Using the analytical results (TCE in the hexane phase, aqueous phase, and solid phases), select the extraction time period that is most effective and efficient for each media type evaluated. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

250 mL RD flask + adapter
empty 144.870 g
+ g.w. 404.293 g
g.w. wt 259.43 g
SPIKED
TCE 260 ml
↓
+
g.w. ~ 260 ml

* Note: The same disposable syringe was used for both dups (eg. bottle 1 + 2) for each sampling period
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Subject Experiment 2b-1

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sample Time	GC #	WT. EMPTY	WT. +Hexane	WT. +sample	sample id.
1115 2/18	59	14.973	18.212	23.219	2b-1-59 To_1 for spiked g.w.
1115 2/18	60	14.969	18.212	23.202	2b-1-60 To_2 for spiked g.w.
1328	61a	14.892	~~18.152~~ 16.7214	19.709	2b-1-B1-T_5 aqueous layer
1331	62a	14.925	~~18.193~~ 16.8748	19.861	2b-1-B2-T_5 " "
1334	63a	14.901	~~18.155~~ 16.8662	20.302	2b-1-B3-T_5 " "
1341	64a	14.914	~~18.163~~ 16.8649	19.633	2b-1-B4-T_5 " "
1344	65a	14.992	~~18.230~~ 16.9392	19.808	2b-1-B5-T_5 " "
1346	66a	14.905	~~18.154~~ 16.8382	19.812	2b-1-B6-T_5 " "
1349	67a	14.890	~~18.150~~ 16.8122	19.674	2b-1-B1-T_{30} " "
1351	68a	14.960	~~18.205~~ 16.9003	20.084	2b-1-B2-T_{30} " "
1354	69a	14.9256	16.875	20.417	2b-1-B3-T_{30} " "
1356	70a	14.973	16.917	20.300	2b-1-B4-T_{30} " "
1358	71a	14.989	16.943	19.900	2b-1-B5-T_{30} " "
~~1440~~ 1400	~~72a~~	14.893	16.856	19.775	2b-1-B6-T_{30} " "
1518	73a	14.954	16.882	19.977	2b-1-B1-T_{2hr} " "
1520	74a	14.992	16.942	19.909	2b-1-B2-T_{2hr} " "
1522	75a	14.948	16.885	20.122	2b-1-B3-T_{2hr} " "
1524	76a	14.863	16.808	19.796	2b-1-B4-T_{2hr} " "
1526	77a	14.908	16.854	20.156	2b-1-B5-T_{2hr} " "
1528	78a	14.924	16.868	19.939	2b-1-B6-T_{2hr} " "
1400 2/20/98	79a	14.916	16.858	20.046	2b-1-B1-T_{24hr} " "
1400	80a	14.957	16.912	19.947	2b-1-B2-T_{24hr} " "
1401	81a	14.977	16.920	20.173	2b-1-B3-T_{24hr} " "
1402	82a	14.885	16.833	19.871	2b-1-B4-T_{24hr} " "
1402	83a	14.999	16.968	19.582	2b-1-B5-T_{24hr} " "
1403	84a	14.900	16.855	19.513	2b-1-B6-T_{24hr} " "
	~~85a~~				2b-1-B1-T_{24hr} " "
	~~86a~~				2b-1-B2-T_{24hr} " "
	~~87a~~		Removed		2b-1-B3-T_{24hr} " "
	~~88a~~		Time point		2b-1-B4-T_{24hr} " "
	~~89a~~		Km 2/19		2b-1-B5-T_{24hr} " "
	~~90a~~				2b-1-B6-T_{24hr} " "

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Subject Experiment 2b-1

Date 02-19 (Month) 18-19 (Day) 19 98 (Year)

Sample Time	VOA 40 ml Vial #	WT empty	WT + soil	WT + GW	sample id. & source
11.20	V1	24.896	35.463	72.814	sediment + spiked g.w. 2b-1-V1
1121	V2	24.866	34.960	72.985	sediment + spiked g.w. 2b-1-V2
1121	V3	25.007	35.896	74.110	crushed basalt + spiked g.w. 2b-1-V3
1121	V4	24.899	35.253	74.518	crushed basalt + spiked g.w. 2b-1-V4
1120	V5	25.136	—	67.174	spiked g.w only 2b-1-V5
1120	V6	24.849	—	66.596	spiked g.w only 2b-1-V6

Time of Extraction	VOA # 125ml Bottle	WT empty	WT. + hexane	WT. + sample	Sample I.d.
2/19/98 1320	B1	138.83	165.00	211.964	sample V1 + 40ml hexane
1323	B2	123.30	150.05	197.160	sample V2 +
1325	B3	124.33	150.97	199.584	Sample V3 +
1336	B4	124.84	150.65	199.550	Sample V4 +
1338	B5	138.63	165.07	206.534	Sample V5 +
1339	B6	138.37	165.12	206.328	Sample V6 +

Sample Time	G.C #	sample I.d. & source	
1327	85H	2b-1-B1-T5	Hexane Layer
1331	86H	2b-1-B2-T5	
1334	87H	2b-1-B3-T5	
1341 1338	88H	2b-1-B4-T5	2/20/98
1343	89H	2b-1-B5-T5	
1346	90H	2b-1-B6-T5	
1348	91H	2b-1-B1-T30	
1350	92H	2b-1-B2-T30	
1352	93H	2b-1-B3-T30	
1355	94H	2b-1-B4-T30	
1357	95H	2b-1-B5-T30	
1359	96H	2b-1-B6-T30	
1517	97H	2b-1-B1-T24hr.	
1519	98H	2b-1-B2-T24hr.	
1521	99H	2b-1-B3-T24hr.	

Sample Time	GC #	sample id & source	
1523	100H	2b-1-B4-T24HR	Hexane Layer
1525	101H	2b-1-B5-T24HR	
1527	102H	2b-1-B6-T24HR	
1400	103H	2b-1-B1-T24HR	
1400	104H	2b-1-B2-T24HR	
1401	105H	2b-1-B3-T24HR	
1402	106H	2b-1-B4-T24HR	
1402	107H	2b-1-B5-T24HR	
1403	108H	2b-1-B6-T24HR	
Removed Timepoint Km 2/19	85H	2b-1-B1-T24hr.	
	86H	2b-1-B2-T24hr.	
	87H	2b-1-B3-T24hr.	
	88H	2b-1-B4-T24hr.	
	89H	2b-1-B5-T24hr.	
	90H	2b-1-B6-T24hr.	

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Subject Experiment 2b-1

Date 02 (Month) 18-19 (Day) 19 98 (Year)

Sample ~~time~~ Time	GC#	wt. empty	wt. + hexane	wt + sample	Sample i.d.
1328	109	14.996	16.974	19.989	2b-1-B1-T5 filtered ag. layer
1331	110	14.932	16.904	19.853	2b-1-B2-T5
1334	111	14.882	16.850	19.278	2b-1-B3-T5
1341	112	14.965	16.928	20.344	2b-1-B4-T5
1344	113	14.948	16.885	19.742	2b-1-B5-T5
1346	114	14.915	16.861	19.854	2b-1-B6-T5
1349	115	14.883	16.828	19.816	2b-1-B1-T30
1351	116	14.894	16.848	20.035	2b-1-B2-T30
1353	117	14.925	16.868	19.085	2b-1-B3-T30
1356	118	14.908	16.868	19.850	2b-1-B4-T30
1358	119	14.878	16.822	19.201	2b-1-B5-T30
~~1440~~ 1400	120	14.997	16.963	20.047	2b-1-B6-T30
1518	121	14.893	16.849	19.094	2b-1-B1-T2hr
1520	122	14.986	16.940	19.892	2b-1-B2-T2hr
1522	123	14.992	16.943	19.376	2b-1-B3-T2hr
1524	124	14.960	16.915	19.896	2b-1-B4-T2hr
1526	125	14.899	16.838	19.116	2b-1-B5-T2hr
1528	126	14.946	16.896	19.837	2b-1-B6-T2hr
1400	127	14.937	16.895	19.249	2b-1-B1-T24hr
1400	128	14.906	16.862	19.775	2b-1-B2-T24hr
1401	129	14.908	16.866	19.707	2b-1-B3-T24hr
1402	130	14.931	16.889	19.600	2b-1-B4-T24hr
1402	131	14.905	16.864	~~19~~ 20.118	2b-1-B5-T24hr
1403	132	14.907	16.860	19.870	2b-1-B6-T24hr
		Removed timepoint KM 2/19			2b-1-B1-T24hr 2b-1-B2-T24hr 2b-1-B3-T24hr 2b-1-B4-T24hr 2b-1-B5-T24hr 2b-1-B6-T24hr

2/2/98

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Read and understood by _____ Date

Subject EXPERIment 2C-1
BASALT EQUILIBRATION

Date 03 (Month) 04 (Day) 19 98 (Year)

03/04/98 11:00 sparged ≈ 3100mL TANAD GW
FOR ≈ 3 hrs Then moved to 15°C incubator
for temp. equilibration.
spiked w/ 1000ppm TCE
Placed ≈ 350g Crushed Basalt + ≈ 250g Aggregate Basalt
into 80°C OVEN TO Remove Any Residual TCE overnite

03/09/98 13:47 Weigh vials & add dried basalt. 2C-1-1 to 2C-1-24 ground water. 2C-1-25 to 2C-1-48 crushed basalt

vial #	wt empty	+ basalt	vial #	wt empty	+ basalt	vial #	wt empty	+ basalt
1345 2C-1-1	25.440	—	1353 2C-1-26	25.623	35.695	1491 2C-1-51	25.352	35.239
1344 2C-1-2	25.270	—	1353 27	25.615	35.681	1401 52	25.237	35.444
1344 2C-1-3	25.405	—	1354 28	25.208	35.255	1402 53	25.394	35.309
1345 2C-1-4	25.249	—	1355 29	25.396	35.445	1402 54	25.561	35.710
1345 5	25.456	—	1355 30	25.284	35.320	1403 55	25.342	35.541
1346 6	25.640	—	1356 31	25.187	35.246	1404 56	25.490	35.316
1346 7	25.319	—	1356 32	25.284	35.421	1405 57	25.591	35.755
1347 8	25.523	—	1357 33	25.352	35.449	1405 58	25.227	35.414
1348 9	25.271	—	1358 34	25.384	35.429	1406 59	25.147	35.233
1348 10	25.471	—	1358 35	25.415	35.493	1406 60	25.153	35.373
1349 11	25.467	—	1359 36	25.204	35.220	1407 61	25.458	35.507
1350 12	25.352	—	1400 37	25.438	35.441	1408 62	25.136	35.212
1350 13	25.383	—	1400 38	25.267	35.273	1409 63	25.504	35.439
1351 14	25.283	—	1416 39	25.431	35.477	1410 64	25.549	35.826
1410 15	25.391	—	1417 40	25.386	35.415	1422 65	25.332	35.282
1411 16	25.545	—	1417 41	25.503	35.569	1423 66	25.432	35.676
1412 17	25.322	—	1418 42	25.493	35.716	1423 67	25.296	35.268
1413 18	25.414	—	1419 43	25.343	35.400	1424 68	25.182	35.475
1414 19	25.225	—	1420 44	25.310	35.351	1425 69	25.209	35.215
1414 20	25.353	—	1420 45	25.203	35.376	1425 70	25.511	35.428
1415 21	25.236	—	1421 46	25.216	35.357	1426 71	25.459	35.544
1415 22	25.494	—	1428 47	25.353	35.383	1426 72	25.292	35.429
1429 23	25.501	—	1429 48	25.287	35.367	2C-1-1 to 24 = ground water		
1430 24	25.442	—	1400 49	25.328	35.741	2C-1-25 to 48 = crushed basalt		
1353 25	25.309	35.364	1400 50	25.247	35.429	2C-1-49 to 72 = aggregate basalt		

Continued on page 27

SRCline 3/4/98
Recorded by Date

SRC 4/8/98
Read and understood by Date

Subject Experiment 2C-1
Basalt Equilibration

Date 03 (Month) 09 (Day) 19 98 (Year)

Sample Time	GC #	wt. empty	wt. + hexane	wt. + sample	sample i.d.
3/10-13:41	133	11.146	14.405	19.421	To-Tedlar bag-a-To
3/10-13:41	134	11.126	14.392	19.379	To-Tedlar bag-b-To
3/10-15:40	135	11.276	14.546	19.549	2C-1-1a-2hr
3/10-15:40	136	11.101	14.380	19.394	2C-1-1b-2hr (duplicate)
3/10-15:47	137	11.112	14.393	19.232	2C-1-2-2hr
3/10-15:44	138	11.175	14.504	19.192	2C-1-25-2hr
3/10-15:45	139	11.044	14.373	18.873	2C-1-26-2hr
3/10-15:47	140	11.146	14.413	19.369	2C-1-49-2hr
3/10-15:48	141	11.051	14.357	18.963	2C-1-50-2hr
3/10-1705	142	11.071	14.285	18.662	2C-1-3-3.5hr
3/10-1705	143	11.067	14.377	18.793	2C-1-4-3.5hr
3/10-1706	144	11.040	14.383	18.863	2C-1-27-3.5hr
3/10-1707	145	11.061	14.300	18.790	2C-1-28-3.5hr
3/10-1709	146	11.124	14.422	18.890	2C-1-51-3.5hr
3/10-1710	147	11.114	14.352	19.168	2C-1-52-3.5hr.
3/10 1843	148	11.124	14.416	18.606	2C-1-5-5hr
3/10 1845	149	11.144	14.423	18.664	2C-1-6-5hr
3/10 1846	150	11.111	14.378	18.627	2C-1-29-5hr
3/10 1847	151	11.102	14.390	18.674	2C-1-30-5hr
3/10 1849	152	11.000	14.271	19.094	2C-1-53-5hr
3/10 1850	153	10.979	14.277	18.785	2C-1-54-5hr
3/10 1849	154	10.981	14.303	19.137	2C-1-53b-5hr (dup)
3/11-0935	155	11.015	14.200	18.596	2C-1-7-20hr.
3/11-0937	156	11.129	14.470	18.804	2C-1-8-20hr
3/11-0937	157	11.110	14.573	18.947	2C-1-31-20hr
3/11-0938	158	11.064	14.321	18.716	2C-1-32-20hr
3/11-0939	159	11.107	14.409	18.855	2C-1-55-20hr
3/11-0940	160	11.217	14.454	18.887	2C-1-56-20hr.
3/11-1325	161	10.955	14.211	18.600	2C-1-9-24hr.
3/11-1327	162	10.951	14.294	18.731	2C-1-10-24hr
3/11-1328	163	11.100	14.417	18.922	2C-1-33-24hr.
3/11-1328	164	11.129	14.451	18.937	2C-1-33-24hr dup.

Results not Valid (filtered) 7/17/98

Continued on page 23

M.K. McCracken — Recorded by — Date 03/09/98

Read and understood by — Date 4/9/98

Subject Experiment 2C-1

Date 03 / 10 / 19 98
Month Day Year

Sample #	wt. + g.w.	Sample #	wt + g.w	Sample #	wt. + g.w
2C-1-1	68.164	2C-1-34	74.313	2C-1-67	74.258
2C-1-2	67.784	2C-1-35	74.052	2C-1-68	74.015
2C-1-3	68.050	2C-1-36	73.659	2C-1-69	73.913
2C-1-4	67.591	2C-1-37	74.330	2C-1-70	74.560
2C-1-5	68.232	2C-1-38	73.946	2C-1-71	73.701
2C-1-6	68.020	2C-1-39	74.327	2C-1-72	74.102
2C-1-7	67.566	2C-1-40	74.184		
2C-1-8	67.728	2C-1-41	74.230		
2C-1-9	67.682	2C-1-42	74.416		
2C-1-10	67.894	2C-1-43	74.050		
2C-1-11	67.854	2C-1-44	74.150		
2C-1-12	67.430	2C-1-45	73.895		
2C-1-13	67.193	2C-1-46	73.842		
2C-1-14	67.448	2C-1-47	73.615		
2C-1-15	67.513	2C-1-48	73.710		
2C-1-16	67.616	2C-1-49	74.198		
2C-1-17	67.621	2C-1-50	74.047		
2C-1-18	68.033	2C-1-51	74.413		
2C-1-19	67.749	2C-1-52	73.900		
2C-1-20	67.675	2C-1-53	73.546		
2C-1-21	67.955	2C-1-54	73.849		
2C-1-22	67.860	2C-1-55	74.304		
2C-1-23	67.848	2C-1-56	74.117		
2C-1-24	67.966	2C-1-57	74.403		
2C-1-25	73.940	2C-1-58	74.317		
2C-1-26	73.581	2C-1-59	73.757		
2C-1-27	74.164	2C-1-60	73.801		
2C-1-28	73.618	2C-1-61	74.417		
2C-1-29	74.329	2C-1-62	73.447		
2C-1-30	74.370	2C-1-63	74.410		
2C-1-31	74.298	2C-1-64	74.631		
2C-1-32	74.679	2C-1-65	73.983		
2C-1-33	73.978	2C-1-66	74.395		

groundwater spiking info.

weighing pan 116.92g
+ tedlar bag 168.96g
+ groundwater 3212.00g

∴ g.w = 3043.04 ml

$C_1V_1 = C_2V_2$

$\frac{1100 mg}{L} X = \frac{1 mg}{L} \cdot 3043.04 ml$

∴ 2.77 ml of saturated TCE
in 3043.04 ml g.w.

stir on rotary shaker - 10 min.

Results Not Valid
(filtered Samples)
SRC 4/17/98

Continued on page 24

M.K. McCracken — Recorded by — 03/10/98 — Date

SRC — Read and understood by — 4/17/98 — Date

Subject Experiment 2C-1
Basalt Equilibration

Date 03 (Month) 13 (Day) 19 98 (Year)

Sample time	GC#	wt. empty	wt. thexane	wt sample	sample i.d.
3/11-1329	165	11.103	14.427	18.937	2C-1-34-24hr.
3/11-1330	166	11.098	14.438	18.877	2C-1-57-24hr.
3/11-1331	167	11.241	14.526	18.963	2C-1-58-24hr.
3/15 1923	168	11.102	14.391	19.93	2C-1-~~11~~ slc -13-5d
3/15 1923	169	11.078	14.420	18.773	2C-1-~~18~~ slc -14-5d
3/15 1924	170	11.219	14.579	19.332	2C-1-~~35~~ slc -37-5d
3/15 1925	171	10.956	14.290	19.030	2C-1-~~36~~ slc -38-5d
3/15 1926	172	11.104	14.445	19.091	2C-1-~~59~~ slc -61-5d
3/15 1927	173	10.998	14.321	18.920	2C-1-~~60~~ slc -62-5d
3/16 1315	174	11.111	14.333	18.660	2C-1-11-6d
3/16 1316	175	11.178	14.516	18.843	2C-1-12-6da
3/16 1316	176	11.146	14.~~447~~ 342	18.675	2C-1-12-6db(dup)
3/16 1317	177	11.228	14.~~568~~ 497	18.833	2C-1-35-6d
3/16 1318	178	11.072	14.~~342~~ 368	19.079	2C-1-36-6d
3/16 1318	179	11.108	14.427	18.740	2C-1-59-6d
3/16 1319	180	11.126	14.454	19.063	2C-1-60-6d
3/17 1500	181	11.102	14.485	18.903	2C-1-15-7d
3/17 1501	182	11.079	14.360	18.741	2C-1-16-7d
3/17 1501	183	11.070	14.292	18.542	2C-1-39-7d
3/17 1503	184	11.129	14.472	18.844	2C-1-40-7d
3/17 1504	185	11.102	14.443	18.947	2C-1-63-7d
3/17 1505	186	11.112	14.462	18.909	2C-1-64-7d
3/18 1329	187	11.115	14.436	18.852	2C-1-17-8d
3/18 1329	188	11.023	14.337	18.666	2C-1-18-8d
3/18 1330	189	11.076	14.405	18.760	2C-1-41-8d
3/18 1331	190	11.049	14.305	18.675	2C-1-42-8d
3/18 1332	191	11.070	14.371	18.877	2C-1-65-8d
3/18 1334	192	11.~~8~~138	14.446	18.983	2C-1-66a-8d
3/18 1335	193	11.104	14.366	~~18~~ 19.557	2C-1-66b(dup)-8d
3/23 1515	212	11.018	14.223	18.792	2C-1-19-13d
3/23 1515	213	11.118	14.419	18.934	2C-1-20-13d
3/23 1516	214	11.~~266~~	14.517	19.003	2C-1-~~43~~-13d
3/23 1516	215	11.148	14.342	18.770	2C-1-~~44~~-13d

Results Not Valid
Samples Filtered
SLC 4/17/98

Continued on page 25

M K McCracken — Recorded by — 03/13/98 — Date

SLC — Read and understood by — 4/8/98 — Date

Subject Experiment 2C-1
Basalt Equilibration

Date 03 (Month) 18 (Day) 19 98 (Year)

sample time	GC#	wt. empty	wt. + hexane	wt. + sample	sample I.d.
3/23-1517	216	11.061	14.335	18.970	2C-1-67-13d
3/23-1517	217	11.163	14.439	19.107	2C-1-68-13d
3/26-1535	218	11.267	14.484	18.965	2C-1-21-16d
3/26-1535	219	11.129	14.436	18.950	2C-1-22-16d
3/26-1536	220	11.112	14.390	18.842	2C-1-45-16d-a
3/26-1537	221	11.023	14.332	18.816	2C-1-45-16d-b
3/26-1537	222	11.212	14.489	18.933	2C-1-46-16d
3/26-1538	223	11.070	14.360	18.906	2C-1-69-16d
3/26-1539	224	11.057	14.346	18.929	2C-1-70-16d
3/30-1440	225	11.178	14.469	19.063	2C-1-23-21d
3/30-	226	11.077	14.374	18.911	2C-1-24-21d
3/30-	227	11.098	14.404	18.999	2C-1-47-21d
3/30-	228	11.156	14.428	18.897	2C-1-48-21d
3/30-	229	11.020	14.258	18.827	2C-1-71-21d
3/30-	230	11.112	14.417	19.585	2C-1-72-21d

Results Not Valid
(Filtered)
SLC 4/17/98

Continued on page 26

M.K. McCracken — Recorded by
3/18/98 — Date
SL C — Read and understood by
4/17/98 — Date

Subject Experiment 2C-1
Basalt Equilibration

Date 03 (Month) 18 (Day) 1998 (Year)

Experiment 2c-1. Sorption Kinetics Test for Crushed and Aggregate Basalt.

(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt) will be prepped by sparging and/or drying to remove any residual TCE present on the media. (Properly indicate usage of this media in the treatability study tracking logbook.) Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking.

2. Prepare a STOCK (Saturated) TCE solution (2000 mg/L) by adding 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and allow to equilibrate overnight at room temperature. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.)

3. Sparge approximately 3100 mL of groundwater from TAN-40 of any residual TCE or other VOCs that may have been present and then place the groundwater into an incubator at 12-15°C. After temperature equilibration of the groundwater (*likely overnight*), remove the container from the incubator.

4. Obtain the tare weight of the container to be used to prepare the spike solution (tedlar bag). Fill with the Tedlar bag with the chilled TAN-40 GW. By gravimetric methods, determine the amount of groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.) All information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

5. Add the appropriate quantity of the Saturated STOCK TCE solution into the tedlar bag to produce a TCE spike concentration of 1.0 mg/L. ***SHOULD BE APPROXIMATELY 3 ML*** Record volume and/or weight added. (Reactive zone media will only be spiked at 0.1 and 1 mg/L TCE during the Task 4 degradation studies.) This solution can be mixed and equilibrated within a few minutes.

6. After mixing, obtain duplicate aqueous samples (≈ 5 mL) of the spike solution to obtain the initial TCE concentration of the spiked groundwater. The duplicate aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (≈ 5 mL). Each sample container will be weighed again after sample addition to determine the actual volume of sample added. These extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

Results Not Valid
SPC 4/17/98

7. Obtain the tare weights of seventy two (72) 40 mL VOA vials. Add approximately 10 g of aggregate basalt to 24 of the vials and 10 g of the crushed basalt to another 24 of the these vials. Gravimetrically determine the actual amount of solid media added.

8. Dispense the spiked groundwater from the tedlar bag into each of the 72 VOA vials that have been prepared, filling them completely with no headspace and cap them. Record the time that each vial is filled as "T=0" for that vial.

Continued on page 27

M.K. McCracken — Recorded by — 3/18/98 Date

SPC — Read and understood by — 4/8/98 Date

Subject Experiment #2C-1
Basalt Equilibration

Date 03 (Month) 18 (Day) 19 98 (Year)

9. Re-weigh each vial after it is completely filled to determine the actual volume of spiked groundwater added to it. Place the vials back into the environmental chamber at 12-15°C Slowly agitate the vials during this step.

The remaining steps will be performed at the time intervals of approximately 2 h, 4 h, 6 h, 18 h, 24h, 4d, 5d, 6d, 7d, 12 d, 14d, and 21d. The latter times may be changed depending upon results of the samples obtained for the earlier time periods.

10. Remove six (6) 40 mL vials from the incubator (two with groundwater only, two with crushed basalt, and two containing aggregate basalt.) Collect an aqueous sample (≈ 5mL) from each vial using a disposable syringe with luer lok needle. Record the time the sample is ~~collected.~~

11. Filter (0.45µm membrane) and dispense the sample into to a pre-weighed vial holding a known volume of hexane (≈5 mL). This sample vial will be weighed again after sample addition to determine the actual volume of sample added. *A new syringe and filter will be used for each sample.* All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

12. Randomly collect and process (steps 10 and 11) a duplicate VOC sample from one of the six VOA vials during every other sampling period.

13. **FOR THE LAST SAMPLING PERIOD STUDIED ONLY:** Extract the remaining contents of each vial with hexane. (The mass of any remaining TCE on the solid phase can be determined with this extraction when a mass balance is performed using data from the aqueous aliquot sample) To accomplish this, transfer the remaining sample in the six 40 mL VOA vials (including basalt material) into a 125 mL extraction jar that has been pre-weighed and holding a known volume of hexane (≈ 35 mL). The extraction jar will be weighed again after sample addition to determine the actual mass of sample (solid +Liquid) added.

14. **FOR THE LAST SAMPLING PERIOD STUDIED ONLY:** The extracts in the above step will be placed on a horizontal or reciprocating shaker for 2 hours. A 1 mL aliquot of the hexane phase will be placed into a 2 mL G.C. vial and stored at 4°C until analysis.

GC ANALYSIS of VOCs
All VOC analyses for this experiment will be conducted using a G.C./ECD detector. The instrument will calibrated for TCE using a 5 point calibration curve with standard concentrations ranging from 10-1000 µg/L (ppb)

The VOC analyses of the hexane extracts will closely follow EPA Method 8000B. Significant deviations from this method will be clearly documented.

IMPROPER Procedure
filter absorbs TCE
SLC 4/17/98

Not Valid Results

Continued on page X

M. K. McCracken 3/18/98
Recorded by Date

SLC 4/17/98
Read and understood by Date

Subject Experiment 2a-3
Test Reactor Performance.

Date 03 (Month) 18 (Day) 19 98 (Year)

Experiment 2a-3. Overall performance of the test reactor
(To be conducted at ORNL/ESD)
In this experiment a complete reactor will be set up using the most optimal component(s) from the previous Task 2a experiments described above. To be consistent, a 100 mg/L TCE spike solution will be used here as well; however, this experiment will be conducted at approximately 12-15°C.

1. Set up a reactor, ensuring that a Teflon sleeve is fitted onto the glass adapter to be used. The top of the adapter will be sealed (Teflon stopper) during spiking and equilibration to minimize the mass of TCE lost.

2. Fill the reactor with distilled water, minimizing the headspace volume inside of the reactor. A record of the DI water volume added will be maintained.

3. Spike the reactor with TCE, Desired Concentration: 100 mg/L
Add a mass of pure phase TCE (density of 1.456 g/mL) to the liquid volume inside of the reactor that will yield a TCE solution of 100 mg/L. Measure the mass of the spiking syringe when filled and then after spiking to record the total TCE mass added to the reactor (a QC check on the initial TCE analysis). Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

4. Replace the removed stopper back onto the adapter and allow the solution to equilibrate *at least* overnight in an incubator maintained at 12-15°C. (Record the equilibration period and temperature).

5. During the equilibration period, prepare an ORBO-32 assembly in the standard taper adapter and cover the bottom end of the adapter with the selected thin film [parafilm].
STORE UN-SEALED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

6. After equilibration, collect duplicate (~~=0.5~~ 5 mL) aqueous samples from the reactor (from the glass adapter that is sealed with a stopper) The aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (=5 mL). Each sample container will be weighed again after sample addition to determine the actual volume of sample added. (A calibration check of the gravimetric scale will be performed each day of use.) All such extracts will be placed on a horizontal shaker for 30 minutes. Store the extracts at 4°C until G.C. analysis.

7. Replace the adapter sealed with the stopper with one fitted with the charcoal desorption tube assembly from Step 5. Record the time the samples in the step above are collected and the desorption tube is added as Start Time=0.

Reactor + glass + teflon Stopper sleeve
WT. empty 256.16 g
WT. + H_2O 785.19
ml = 529.03

$\frac{100 mg}{1000 mg} \cdot 529.03 ml = 52.90 mg$

$52.90 mg \cdot \frac{1000 ml}{1456 mg} = 36.2 \mu l$ pure phase TCE

Syringe + TCE 13.7479
syringe after TCE 13.6939

TCE added: 3/18/98-1430 & began incubation at 12°C.
↓
Incubation ended at 12°C 3/19/98-0910

Balance Calib 3/18 X177440
wt. 50 g scale 49.96 g ✓
3/18 X177339
Balance Calib. wt. 1g scale 0.9988 ✓

Balance Calib. 3/19 X177339
wt. 1g scale 1.0000 ✓
pipet calib 3/19 SC002 P5000 5 ml 4.9998 g ✓
5 ml 5.0000 g

Continued on page 29

M.K. McCracken 03/18/98
Recorded by Date

SPC 9/8/98
Read and understood by Date

Subject Experiment 2a-3
Test Reactor Performance

Date 03 18 1998
Month Day Year

8. Collect at the following time intervals in the same manner as performed in step 6: 30 min, 60 min, 2 hrs, 4 hrs, 24 hrs *(duplicate)*, 30 hrs, 48 hrs. The exact time at which each aliquot is collected will be recorded.

9. Breakdown the reactor after the last sample is collected. The charcoal desorption tube will then be removed from the reactor. Each charcoal bed contained in the tube will be placed into a pre-filled and pre-weighed vial containing a known volume of hexane (≈5 mL). These extracts will be processed and analyzed in the same manner as the aqueous aliquots.

NOTE: The contents of such a test reactor will be extracted at the end of the experiments in Task 4 but is not necessary here since a solid phase is not present.

10. Analyze the sample and charcoal extracts via GC/ECD for TCE. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks, etc. are also analyzed on each analysis day. Formal data packages, as outlined in Section 2.4.3.3 of the Chapter 2 Implementation Plan will not be prepared for this experiment.

11. Perform a mass balance for the reactor, taking sample aliquot volumes, initial concentrations, etc. into account to determine the overall reactor performance in maintaining gas-tight conditions. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

Balance calib. 3/20
X177339.
wt. 20.0 scale 20.0001

Balance calib. 3/21
X177339
wt. 20.0 scale 20.0000

Sample Time	GC #	wt. empty	wt. + hexane	wt. + sample	Sample i.d.	dilutions
3/19-0922	194	11.222	14.475	19.504	2a-3-To-1	101
3/19-0922	195	11.083	14.475	19.448	2a-3-To-2	
3/19-0952	196	11.101	14.460	19.457	2a-3-30min-1	
3/19-0952	197	11.059	14.374	19.383	2a-3-30 min-2	
3/19-1022	198	11.139	14.456	19.476	2a-3-1hr-1	
3/19-1022	199	11.074	14.401	19.386	2a-3-1hr-2	
3/19-1122	200	10.924	14.209	19.217	2a-3-2hr-1	
3/19-1122	201	11.047	14.327	19.309	2a-3-2hr-2	
3/19-1322	202	11.159	14.414	19.417	2a-3-4hr-1	
3/19-1322	203	11.035	14.257	19.274	2a-3-4hr-2	
3/20-0925	204	10.848	14.087	18.947	2a-3-24hr-1	
3/20-0925	205	11.116	14.436	19.335	2a-3-24hr-2	
3/20-1622	206	11.010	14.305	19.249	2a-3-31hr-1	(31hrs)
3/20-1622	207	11.072	14.361	19.333	2a-3-31hr-2	(31hrs)
3/21-1634	208	11.094	14.369	19.347	2a-3-48hr-1	(55hrs)
3/21-1635	209	11.084	14.351	19.330	2a-3-48hr-2	(55hrs)
3/21-1636	210	10.986	14.201	14.293	2a-3-chrcl-1	101
3/21-1636	211	11.002	14.243	14.283	2a-3-chrcl-2	101

*chrcl = charcoal

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Subject Experiment 2a-3
Test Reactor Performance

Date 03 (Month) 30 (Day) 19 98 (Year)

Time/date	GC#	id.	dilutions.
3/30-1530	231	2a-3-chrcl-1R	201
	232	2a-3-chrcl-2R	1

M K McCracken — Recorded by — 03/30/98 — Date

Continued on page

SRC — Read and understood by — 4/7/98 — Date

Subject Permanganate Calibration Curve

Date 04 (Month) 06 (Day) 1998 (Year)

Preparation of Permanganate Calibration Curve

Instrument: HACH DR/2000 spectrophotometer

Summary: This procedure involves the production of a MnO_4^- calibration curve by the colorimetric absorbance (λ= 525 nm) of prepared solutions of $KMnO_4$ using a Hach DR/2000 spectrophotometer.

The following solutions concentrations will be prepared:

5, 10, 20, 30, and 50 mg/L MnO_4^-.

The 50 mg/L MnO_4^- std will result from the preparation of a 66.4 mg/L $KMnO_4$ solution

Reagent to be used:
CAIROX Potassium Permanganate, Free-Flowing Grade (Carus™)

STD 5 (50 mg/L MnO_4^-)
Prepare Solution by adding 3.32 grams of KMnO4 into a 50 mL volumetric flask and fill to volume with DI water. Place this solution on a magnetic stir plate for at least 15 minutes before preparing the following standard solutions.

STD 4 (30 mg/L MnO_4^-)
Add 15 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 3 (20 mg/L MnO_4^-)
Add 10 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 2 (10 mg/L MnO_4^-)
Add 5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 1 (5 mg/L MnO_4^-)
Add 2.5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

Keep these solutions in darkness until used for calibration.
Record the abs obtained for a DI water blank and for each sample
Prepare the linear calibration curve and constants.

Computation Error.

added 3.324 g KMnO4 to 50 mL volumetric + 50 mL d.i. H_2O. (foil wrapped flask) stir 1 hr, RT.

std #	abs (525 nm)	i.d.
BLK	0.000	d.i. water
std 1	3.652	5 mg/L MnO_4^-
std 2	3.714	10 mg/L MnO_4^-
std 3	3.749	20 mg/L MnO_4^-
std 4		30 mg/L MnO_4^-
std 5	3.874	50 mg/L MnO_4^-

Will Re-Perform

DATA NOT Valid

M.K. McCracken — Recorded by — 04/06/98 — Date

Continued on page

Read and understood by — Date 4/7/98

Subject Additional lab Test #1

Date 04 06 1998
Month Day Year

Additional Lab Experiment
Note: This experiment is being in conducted in addition to the tasks set forth in the statement of work.

1. All groundwater to be used in this experiment (TAN-40 groundwater) will be prepped by sparging to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking. (Properly indicate usage of this media in the treatability study tracking logbook.)

2. Prepare a STOCK (Saturated) TCE solution (2000 mg/L) by adding 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and allow to equilibrate overnight at room temperature. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.)

3. Obtain the tare weight of the 1 liter (small) Tedlar bag to be used to prepare the spike solution. Fill with approximately 520 mL of 12°C TAN-40 groundwater. By gravimetric methods, determine the amount of groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.) Place the bag into the incubator at 12°C until it is spiked.

4. Obtain the tare weight of twelve (12) 40 mL VOA vials.

5. Remove the Tedlar bag from the incubator and add the appropriate quantity of the Saturated STOCK TCE solution to produce a 1 mg/L TCE spike solution. (The volume of saturated STOCK TCE needed should be on the order of 0.5 mL). This solution can be mixed and equilibrated within 20 minutes. All Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

6. Prepare a 2660 mg/L KMnO4 solution by adding 66.44 mg (0.0664 grams) Carus™ Free Flowing Technical Grade KMnO4 into a 25 mL volumetric flask and fill to volume with DI water. (This solution should have a MnO_4^- concentration of 2000 mg/L)

7. After mixing for at least 1 hour, collect an aliquot of this solution for KMnO4 analysis. (A calibration check should not be needed since we just prepared the calibration curve). The sample will have to be diluted to less than 50 mg/L MnO_4^-. (on the order of 1.25 mL into a 100 mL flask)

8. Dispense the following volumes of the KMnO4 solution into the 40 mL vials and reweigh each:
   - Vials ~~1&2~~ 3&4: NONE
   - Vials ~~3&4~~ 1&2: 0.1 mL (will yield approximately 5 mg/L MnO_4^-)
   - Vials 5&6: 0.3 mL (will yield approximately 15 mg/L MnO_4^-)
   - Vials 7&8: 0.8 mL (will yield approximately 40 mg/L MnO_4^-)
   - Vials 9&10: 1.6 mL (will yield approximately 80 mg/L MnO_4^-)
   - Vials 11&12: 2 mL (will yield approximately ~~80~~ 100 mg/L MnO_4^-)

Tedlar bag
~~47.2g~~
39.62g
+ H_2O 562.34g
H_2O = 522.72g = mL
added 523µl TCE saturated soln.

shake on rotary shaker 30rpm. 12°C

added 0.0664g KMnO4 to 25ml d.i. H_2O in foil wrapped volumetric. stir 1hr.

Reaction Start Times

vial:	Time	vial:	Time	vial:	Time	vial:	Time
1	1200	4	1203	7	1204	10	1207
2	1202	5	1204	8	1206	11	1208
3	1202	6	1204	9	1206	12	1210

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Continued on page

Subject Additional Lab Test #1

Date 04 (Month) 07 (Day) 1998 (Year)

9. Quickly dispense the spike solution into each of the reactors to be used. Completely fill each reactor to minimize free headspace. Immediately re-seal each vial, record the "REACTION START TIME" and reweigh each to determine the actual amount of spiked groundwater added. Vigorously hand shake each vial at the beginning of the reaction time and then place the samples in the 12C incubator/shaker.

10. At approximately 1 hour from the reaction start time, remove the reactors from the incubator and collect a 10 mL aliquot (using a syringe with a Luer Tip). Record the time the sample is collected.

11. Add a 0.45 µm syringe filter unto the end of the syringe and dispense (filtered) approx. 5 mL of the sample into a pre-weighed vial holding a known volume of hexane (≈5 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

12. **This step needs to be done fairly quickly since the reaction is not yet "quenched"!!**
Dispense (filtered) the remaining portion of the sample in the syringe (≈5 mL) into a beaker for MnO_4^- analysis. (Some samples may have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO_4^-). Check standards (10 and 50 mg/L MnO_4^-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

13. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

14. GC ANALYSIS of VOCs
All VOC analyses for this experiment will be conducted using a G.C./ECD detector. The instrument will calibrated for TCE using a 5 point calibration curve with standard concentrations ranging from 5-1000 µg/L (ppb). The VOC analyses of the hexane extracts will closely follow EPA Method 8000B. Significant deviations from this method will be clearly documented. Formal data packages will not be prepared for these analyses.

Reaction Start time

Start Sample time	Vial #	wt. empty	wt. + $KMnO_4$	wt. + spike soln	sample i.d.
4/7/98 - 1200	1	25.188	25.288	67.300	~~No~~ KmAO4 - 0.1 ml
1202	2	25.273	25.374	68.046	~~No~~ KmnO4 - 0.1 ml
1202	3	25.469	25.469	67.859	No KMnO4
1203	4	25.237	25.237	67.376	No KMnO4
1204	5	25.494	25.801	68.181	KMnO4 - 0.3 ml
1204	6	25.482	25.790	67.830	KMnO4 - 0.3 ml
1204	7	25.449	26.254	67.793	KMnO4 - 0.8 ml
1206	8	25.230	26.033	67.798	KMnO4 - 0.8 ml
1206	9	25.259	26.857	67.644	KMnO4 - 1.6 ml
1207	10	25.296	26.898	67.617	KMnO4 - 1.6 ml
1208	11	25.451	27.447	68.018	KMnO4 - 2.0 ml
1210	12	25.227	27.220	67.389	KMnO4 - 2.0 ml

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Subject Additional Lab Test #1

Date 04 (Month) 07 (Day) 19 98 (Year)

Sample time	GC#	wt. empty	wt. + hexane	wt. + Sample	Sample I.d.
4/7/98 1300	231	11.128	14.367	18.800	MnO4-0
1302	232	11.063	14.462	18.845	MnO4-0
1303	233	11.042	14.407	18.834	MnO4-0.1
1304	234	10.977	14.323	18.711	MnO4-0.1
1305	235	11.123	14.474	18.804	MnO4-0.3
1306	236	11.177	14.515	18.880	MnO4-0.3
1307	237	11.063	14.405	18.619	MnO4-0.8
1308	238	10.972	14.296	18.748	MnO4-0.8
1309	239	11.042	14.356	18.656	MnO4-1.6
1310	240	11.054	14.353	19.157	MnO4-1.6 * not filtered
1311	241	11.139	14.441	18.917	MnO4-2.0
1312	242	11.035	14.329	18.742	MnO4-2.0

Spectrophotometric Results.

GC Sample #	abs. at 525 nm	sample I.d.
231	0.000	
232	-0.002	
233	0.097	
234	0.096	
235	0.305	
236	0.308	
237	0.824	
238	0.817	
239	1.653	
240	1.636	
241	2.004	
242	2.002	
50 mg/L std	3.874	
10 mg/L std	3.714	
Blank	0.000	

DF = 1

These are valid data points

Undiluted

Data not valid

samples not selected A/13/98

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Recorded by Date

SRC 4/13/98
Read and understood by Date

Subject Permanganate Calibration Curve

Date 04 (Month) 08 (Day) 19 98 (Year)

Preparation of Permanganate Calibration Curve

Instrument: HACH DR/2000 spectrophotometer

Summary: This procedure involves the production of a MnO_4^- calibration curve by the colorimetric absorbance (λ= 525 nm) of prepared solutions of $KMnO_4$ using a Hach DR/2000 spectrophotometer.

The following solutions concentrations will be prepared:

5, 10, 20, 30, and 50 mg/L MnO_4^-.

The 50 mg/L MnO_4^- std will result from the preparation of a 66.4 mg/L $KMnO_4$ solution

Reagent to be used:
CAIROX Potassium Permanganate, Free-Flowing Grade (Carus™)

STD 5 (50 mg/L MnO_4^-)
Prepare Solution by adding 33.2 mg of KMnO4 into a 500 mL volumetric flask and fill to volume with DI water. Place this solution on a magnetic stir plate for at least 15 minutes before preparing the following standard solutions. 0.03374 into 500 ml DI Water 1345 to 1400 @ 23°C

STD 4 (30 mg/L MnO_4^-)
Add 15 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

Pipette calibrated @ 5.01 g 2.50 g

STD 3 (20 mg/L MnO_4^-)
Add 10 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 2 (10 mg/L MnO_4^-)
Add 5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 1 (5 mg/L MnO_4^-)
Add 2.5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

Keep these solutions in darkness until used for calibration.
Record the abs obtained for a DI water blank and for each sample
Prepare the linear calibration curve and constants.

SRC 4/13/98

Abs Readings	Abs
Std 1	0.089
2	0.190
3	0.380
4	0.577
5	0.951

4/8/98 performed by William Holden

S.R. Cline Last Updated: 04/08/98 Filename: Preparation of Permanganate Calibration Curve.doc

Continued on page X

WH Holden — Recorded by — 4/8/98 Date

SRC — Read and understood by — 4/13/98 Date

Subject Additional Lab Test II

Date 04 (Month) 09 (Day) 19 98 (Year)

Intentionally left Blank SRC

Analysis of KMnO4 Samples ~ 48 hrs after exp. (properly diluted!) SRCline

Spectrophotometric Results

GC Sample #	abs. at 525nm	Sample id.	dil	
BLK	0.000	blank	1	
50mg/L std	1.087	50mg/L Std.	1	ok
10mg/L Std	0.195	10mg/L Std	1	ok
231	-0.001	NO KMnO4	1	
232	0.001	No KMnO4	1	
233	0.055	KMnO4 - 0.1	1	
234	0.055	MnO4 - 0.1	1	
235	0.256	MnO4 - 0.3	1	
236	0.259	MnO4 - 0.3	1	
237	0.777	MnO4 - 0.8	1	
238	0.772	MnO4 - 0.8	1	
239	0.316	MnO4 - 1.6	5	
240	0.313	MnO4 - 1.6	5	
241	0.399	MnO4 - 2.0	5	
242	0.401	MnO4 - 2.0	5	
φ	0.437	MnO4 - Tφ	100	

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SRC — Read and understood by — 4/13/98 — Date

Subject Experiment 3a-R1

Date 04 (Month) 16 (Day) 1998 (Year)

EXP. 3a-R1 (FOR RUN #1 of Table 2-3)

General Procedure for Task 3a Experiments - Oxidant Demand of Reactive Zone Media
(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with a consistent and controlled initial TCE concentration (≈ 0 mg/L). Properly indicate usage of this media in the treatability study tracking logbook.

2. Follow Table 2-3 in Chapter 2 of the Implementation Plan to obtain which test conditions to evaluate (soil type, oxidant concentration, etc.) The use of glass, wide-mouth erlenmeyer flasks, beakers, or equivalent will be used. It is currently planned to run four (4) test conditions from Table 2-3 each time this procedure is used. Thus, 8 reactors will be used during each run of this procedure.

3. Obtain the tare weight of each reactor. Add approximately 100g of solid media to the reactors in Table 2-3 that require it Re-weigh the reactors to determine the actual mass added. To each reactor, add 400 mL of TAN-40). Re-weigh each reactor to determine the actual weight of solution added. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Place the reactors in the incubator at 12°C and allow to equilibrate to that temperature. Slowly agitate the reactors during this step.

5. Once temperature equilibrium is reached, remove the reactors from the incubator and collect a 5 mL aliquot (using a disposable syringe) of the water and/or slurry from each for pH analysis.

6. The pH analyses will be conducted using conventional pH meters/electrodes, calibrated each day of use via a minimum of two standard buffer solutions (4, 7, and 10). Values obtained for each sample will be recorded. A calibration check will also be conducted following the last sample to be measured each day. Values obtained for the samples will be corrected if a significant drift (i.e., > 15%) occurred.

7. Add the appropriate mass of crystalline $KMnO_4$ (Carus™ Free Flowing Technical Grade) into each reactor according to which test condition in Table 2-3 is being evaluated. The exact mass of $KMnO_4$ added to each reactor will be determined gravimetrically and recorded. The time that $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time).
0.01% = 100 mg/L MnO_4^- @ 400 mL → 40 mg MnO_4^- needed ∴ add 53 mg $KMnO_4$ To each

8. Place each reactor back into the 12°C incubator as the oxidant consumption reaction proceeds. For each reactor, remove the reactor from the incubator at approximately 5-10 minutes from the reaction start time, and collect a 5-10 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator.

9. Add a 0.45 μm syringe filter onto the end of the syringe and filter approximately 1-2 mL into a beaker. Remove the syringe filter and dispense the remaining (unfiltered) portion of the sample

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Subject Experiment 3a-R1

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into another beaker for pH analysis. Measure and record the pH of each sample as done in Step 6.

10. Perform a MnO_4^- analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

11. Collect and analyze aliquots from each reactor for pH and $KMnO_4$ (Steps 8-10) at the following ADDITIONAL time periods: 1hr, 2 hrs, 4 hrs, 18 hrs, and 24 hrs.

12. Interpret results and clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

shaking @ ~100 RPM
Inc. to 200 RPM after t = 24 hrs
Collected = 6mL aliquot each time

Table 2-3. Oxidant consumption by reactive zone media.

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial ~~Potassium~~ Permanganate Concentration (%)
RUN 1	Groundwater	NA	NA	0	0	0.01
	Groundwater	NA	NA	0	0	0.01
	Groundwater/basalt	Crushed	4:1	0	0	0.01
	Groundwater/basalt	Crushed	4:1	0	0	0.01
	Groundwater/basalt	Aggregate	4:1	0	0	0.01
	Groundwater/basalt	Aggregate	4:1	0	0	0.01
	Groundwater/sediment	NA	4:1	0	0	0.01
	Groundwater/sediment	NA	4:1	0	0	0.01

Note: hand stirred #7 + #8 flasks after t=3.5hr 7/17 ∴ t = ~ 16 hrs (1 day)

sample#	flask wt.	flask + medium	flask + H_2O	sample i.d
1	214.56	—	610.87	groundwater
2	152.05	—	548.67	groundwater
3	151.17	251.49	650.81	crushed basalt
4	223.92	324.34	725.49	crushed basalt
5	161.24	262.04	658.38	aggregate basalt
6	213.94	313.99	714.20	aggregate basalt
7	219.68	319.79	714.20	sediment
8	161.88	262.06	657.02	sediment

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Read and understood by Date

Subject Experiment 3a-R1

Date 04 (Month) 16 (Day) 19 98 (Year)

flask #	KMnO4	Time 0 (4/16/98)	T10min (4/16/98)	T1hr (4/16/98)	T2hr (4/16/98)	T3hr (4/16/98)	T4 (4/17/98)	T24hr (4/17/98)
1	52.6 mg	1429	1450	1529	1626	1749	954	1432
2	52.6	1430	1450	1530	1628	1750	955	1433
3	53.7	1431	1451	1531	1629	1752	956	1434
4	54.1	1432	1452	1532	1631	1753	957	1435
5	54.4	1434	1453	1533	1632	1754	958	1437
6	53.0	1435	1453	1534	1634	1755	959	1438
7	54.1	1436	1454	1536	1635	1756	1000	1439
8	55.3	1438	1455	1538	1637	1757	1001	1439

↓ 5 dil ↓ 5 dil DF = 5

flask #	T0 λ	T0 pH	T10min λ	T10min pH	T1hr λ	T1hr pH	T2hr λ	T2hr pH
1		8.26	0.419	8.07	0.400	7.90	~~0.476~~	8.20 ← Recal.
2		8.22	0.416	8.20	0.372	8.04	~~0.443~~	8.08
3		8.19	0.197	8.15	0.193	8.04	~~0.275~~	8.10
4		8.21	0.220	8.15	0.208	8.10	~~0.296~~	8.11
5		8.22	0.430	8.19	0.397	8.11	~~0.450~~	8.15
6		8.20	0.386	8.19	0.375	8.12	~~0.394~~	8.15
7		7.83	0.423	7.91	0.395	7.81	~~0.471~~	7.80
8		7.81	0.421	7.87	0.410	7.78	~~0.467~~	7.75

DF = 5 * DF = 5

flask #	T3hr λ	T3hr pH	T10min λ	T10min pH	T24hr λ	T24hr pH	T2hr λ Repeats
1	0.434	8.19	0.424	8.26	0.425	8.40	0.397 OK
2	0.434	8.06	0.423	8.39	0.430	8.42	~~0.422~~ → .371
3	0.314	8.12	0.395	8.31	0.399	8.39	0.205 OK
4	.305	8.15	0.370	8.25	0.376	8.21	~~0.291~~ → .259
5	0.426	8.12	~~0.458~~ .422	8.22	0.424	8.25	~~0.437~~ → .387
6	0.418	8.19	0.392	8.29	0.419	8.28	~~0.415~~ → .393
7	0.466	7.82	0.374	8.01	0.357	7.96	~~0.485~~ → .425
8	0.497	7.77	0.365	7.96	0.319	8.00	~~0.501~~ → .463

T10min Sample	λ	T1hr sample	λ
Blank	0.000	Blank	0.000
50mg	1.084	50mg	1.080
10mg	0.216	10mg	0.203

Cal OK! 4/17/98 Blank Spec λ = 0
50mg/L 1.089
10mg/L 0.202

Continued on page —

M. K. McCracken 4/16/98 Recorded by Date

S R C 5/7/98 Read and understood by Date

* Calibrated but diff. probe used!

Subject Experiment 3a-R2

Date 04 (Month) 21 (Day) 19 98 (Year)

EXP. 3a-R2

General Procedure for Task 3a Experiments - Oxidant Demand of Reactive Zone Media

(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with a consistent and controlled initial TCE concentration (≈ 0 mg/L). Properly indicate usage of this media in the treatability study tracking logbook.

2. Follow Table 2-3 in Chapter 2 of the Implementation Plan to obtain which test conditions to evaluate (soil type, oxidant concentration, etc.) The use of glass, wide-mouth erlenmeyer flasks, beakers, or equivalent will be used. It is currently planned to run four (4) test conditions from Table 2-3 each time this procedure is used. Thus, 8 reactors will be used during each run of this procedure.

3. Obtain the tare weight of each reactor. Add approximately 100g of solid media to the reactors in Table 2-3 that require it Re-weigh the reactors to determine the actual mass added. To each reactor, add 400 mL of TAN-40). Re-weigh each reactor to determine the actual weight of solution added. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Place the reactors in the incubator at 12°C and allow to equilibrate to that temperature. Slowly agitate the reactors during this step.

5. Once temperature equilibrium is reached, remove the reactors from the incubator and collect a 5 mL aliquot (using a disposable syringe) of the water and/or slurry from each for pH analysis.

6. The pH analyses will be conducted using conventional pH meters/electrodes, calibrated each day of use via a minimum of two standard buffer solutions (4, 7, and 10). Values obtained for each sample will be recorded. A calibration check will also be conducted following the last sample to be measured each day. Values obtained for the samples will be corrected if a significant drift (i.e., > 15%) occurred.

7. Add the appropriate mass of crystalline $KMnO_4$ (Carus™ Free Flowing Technical Grade) into each reactor according to which test condition in Table 2-3 is being evaluated. The exact mass of $KMnO_4$ added to each reactor will be determined gravimetrically and recorded. The time that $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). USE A Rubber STOPPER + HAND MIX VIGOROUSLY

8. Place each reactor back into the 12°C incubator as the oxidant consumption reaction proceeds. For each reactor, remove the reactor from the incubator at approximately 5-10 minutes from the reaction start time, and collect a 5-10 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator. } @ ≈ 200 rpm

9. Add a 0.45 µm syringe filter onto the end of the syringe and filter approximately 1-2 mL into a beaker. Remove the syringe filter and dispense the remaining (unfiltered) portion of the sample

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0.1% = 1000 mg/L MnO_4^-

∴ Add $KMnO_4$ @ Conc = 1328 mg/L

For 400 mL Soln ⇒ Add 1328 * 0.4 = 530 mg $KMnO_4$ each Flask

MKA 4/27/98

sparged 1 gal TAN 40 Ground water - 2.5 hrs.

dried, ~214 g aggregate basalt, ~200 g crushed basalt, ~200 g sediment

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into another beaker for pH analysis. Measure and record the pH of each sample as done in Step 6.

10. Perform a MnO_4^- analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

11. Collect and analyze aliquots from each reactor for pH and $KMnO_4$ (Steps 8-10) at the following ADDITIONAL time periods: 1hr, 2 hrs, 4 hrs, 18 hrs, and 24 hrs.

12. Interpret results and clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

KMcCracken 4/21/98

A DF=50 should work .1mL Sample → 5mL DI Water
[Actually, DF = 51]

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial ~~Potassium~~ Permanganate Concentration (%)
RUN 2 Exp. 3A-R2	Groundwater	NA	NA	0	0	0.10
	Groundwater	NA	NA	0	0	0.10
	Groundwater/basalt	Crushed	4:1	0	0	0.10
	Groundwater/basalt	Crushed	4:1	0	0	0.10
	Groundwater/basalt	Aggregate	4:1	0	0	0.10
	Groundwater/basalt	Aggregate	4:1	0	0	0.10
	Groundwater/sediment	NA	4:1	0	0	0.10
	Groundwater/sediment	NA	4:1	0	0	0.10

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Sample #	flask wt	flask + medium	flask + H_2O	sample I.D.
1	213.89	—	608.31	ground water
2	161.21	—	558.01	ground water
3	223.88	~~330~~ 323.96	720.53	crushed basalt
4	151.13	251.28	646.09	crushed basalt
5	219.63	319.63	716.09	aggregate basalt
6	152.00	252.06	647.97	aggregate basalt
7	161.85	261.98	662.20	sediment
8	214.51	312.61	708.42	sediment

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Flask#	KMnO4 added	Time Ø	T10min	T1hr	T2hr	T3hr	T18hr	T24hr
1	0.5203	1310	1322	1410	1510	1610	0855	1325
2	0.528	1311	1324	1411	1511	1611	0855	1326
3	0.527	1312	1324	1413	1513	1612	0856	1327
4	0.532	1314	1325	1414	1514	1613	0857	1329
5	0.528	1315	1326	1414	1516	1613	0859	1330
6	0.536	1316	1328	1416	1517	1614	0900	1331
7	0.529	1318	1329	1417	1519	1615	0901	1332
8	0.530	1319	1329	1419	1520	1616	0903	1333

pH readings

Flask	TØ	T10min	T1hr	T2hr	T3hr	T18hr	T24hr
1	8.31 km	8.38	8.38	8.37	8.35	8.20	8.19
2	8.69	8.73	8.72	8.72	8.70	8.49	8.47
3	8.62	8.60	8.54	8.48	8.45	8.24	8.23
4	8.61	8.59	8.52	8.46	8.43	8.21	8.24
5	8.64	8.67	8.63	8.55	8.51	8.14	8.15
6	8.67	8.69	8.65	8.58	8.54	8.15	8.19
7	8.29	7.98	7.98	8.04	~~7.83~~ km 8.05	7.96	7.87
8	8.24	8.10	8.10	8.16	8.06	7.98	7.78

spec. readings

Flask	TØ	DF 100 T10min	DF 100 T1hr	DF 50 T2hr	DF 50 T3hr	DF 50 T18hr	DF 50 T24hr
1		0.204	0.204	0.433	0.446	0.450	0.445
2		0.216	0.224	0.427	0.456	0.430	0.430
3		0.207	0.202	0.429	0.445	0.400	0.430
4		0.208	0.221	0.438	0.507	0.45	0.420
5		0.217	0.216	0.438	0.429	0.435	0.455
6		0.202	0.197	0.456	0.424	0.490	0.450
7		0.204	0.175	0.436	0.435	0.420	0.410
8.		0.145	0.161	0.428	0.407	0.435	0.430

~~T10min~~ sample	T10min	T1hr	T2hr	T3hr	T18+24hr std curve ~~T18hr~~ km	~~T24hr~~ km
BLK	0.000	0.000	0.000	0.000	0.000	20 ~~0.515~~ 0.470 km
50	1.065	1.069	1.069	1.069	1.120	10 0.239
10	0.186	0.176	~~0.173~~ 0.173 km	0.178	0.790	0.102

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Subject Calibration Standards

Date 4 (Month) 24 (Day) 1998 (Year)

KM 4/27/98

Preparation of Calibration Standards
(From VOC custom mixes)

Source:
Supelco Custom Mix Standard, 200 mg/L each Compound

DATE PREPARED 4/22/98
LOT No. LA 73307

Prepare 10 mg/L working STOCK Solution
Add 0.5 mL of Custom Mix (Ampule) to 10 mL Flask filled to volume with hexane
(Weigh and record syringe when full and then when empty)

SYRINGE FULL: 17.5875
SYRINGE EMPTY: 17.1712

∴ 0.4163g (200 ppm) Custom Mix Added
OR 0.534 mL (assuming spg = .78 for MeOH)
∴ $C_1V_1 = C_2V_2$ (200 ppm)(.534 mL) = (x)(10 mL)
x = 10.68 ppm

Preparation of Calibration Standards

1000 ppb
Add 2.5 mL STOCK Solution to hexane in 25 mL Flask. Actual = 1068 ppb

500 ppb
Add 5 mL of 1000 PPB STD into 10 mL Flask and fill to volume with hexane Actual = 534 ppb

100 ppb
Add 5 mL of 1000 PPB STD into 50 mL Flask and fill to volume with hexane (transfer to 40 mL VOA vial) Actual = 107 ppb

50 ppb
Add 0.5 mL 1000 PPB STD into 10 mL Flask and fill to volume with hexane Actual = 53.4 ppb

10 ppb
Add 0.1 mL of 1000 PPB STD into 10 mL Flask and fill to volume with hexane Actual = 10.7 ppb

5 ppb
Add 0.05 mL (50 µL) of 1000 PPB STD into 10 mL Flask and fill to volume with hexane
Actual = 5.3 ppb

Place these standards in vials for storage in the 211 Freezer.
Place the Certificate of Analysis into the Laboratory Research Notebook.

balance calib. X177339 wt. 10.0g → 9.9998g

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Subject Additional Exp. #2

Date 04 (Month) 28 (Day) 19 98 (Year)

Additional Lab Experiment #2
Note: This experiment is being in conducted in addition to the tasks set forth in the statement of work.

1. All groundwater to be used in this experiment (TAN-40 groundwater) will be prepped by sparging to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking. (Properly indicate usage of this media in the treatability study tracking logbook.)

Already Done

2. Prepare a STOCK (Saturated) TCE solution (1100 mg/L) by adding 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and allow to equilibrate overnight at room temperature. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.)

2A-Kitty: It would be nice to measure the conc of the Saturated solution for our own information: Please extract 10 µL of this STOCK into 20 mL hexane for 2 hours, then put 1 mL of the hexane phase into a gc vial for analysis (DF=2000).

3. Obtain the tare weight of the 1 liter (small) Tedlar bag to be used to prepare the spike solution. Fill with approximately 850 mL of TAN-40 groundwater. By gravimetric methods, determine the amount of groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Add the appropriate quantity of the Saturated STOCK TCE solution to produce a 1 mg/L TCE spike solution. (The volume of saturated STOCK TCE needed should be approx. 0.78 mL for 850 mL of GW). Place the Bag in the incubator at 12C overnight. All Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

5. Obtain the tare weight of twenty (20) 40 mL VOA vials.

6. Prepare a 2660 mg/L KMnO4 solution by adding 66.44 mg (0.0664 grams) Carus™ Free Flowing Technical Grade KMnO4 into a 25 mL volumetric flask and fill to volume with DI water. (This solution should have a MnO_4^- concentration of 2000 mg/L) * We will need ≈ 20 mL of this.

7. After mixing for at least 1 hour, collect an aliquot of this solution for KMnO4 analysis. The sample will have to be diluted to less than 50 mg/L MnO_4^-. (DF=80 --.625 mL into a 50 mL flask will work great!)

NEXT DAY

8. Dispense the following volumes of the KMnO4 solution into the 40 mL vials and reweigh each:
   - Vials 1-6: 0.3 mL (will yield approximately 15 mg/L MnO_4^-)
   - Vials 7-12: 1.2 mL (will yield approximately 60 mg/L MnO_4^-)
   - Vials 13-18: 2 mL (will yield approximately 100 mg/L MnO_4^-)
   - Vials 19-20: NONE.

9. Remove spike solution from incubator and quickly dispense the spike solution into each of the reactors to be used. Completely fill each reactor to minimize free headspace. Immediately re-seal each vial, record the "REACTION START TIME" and reweigh each to determine the actual

Stock TCE (saturated)

Tedlar Bag - 1L

wt. empty - 38.090g

wt. + H_2O - 885.06

water - 845.97ml

added 77µl saturated TCE soln.

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S.R. Cline Last Updated: 04/24/98 Filename: Additional Lab Experiment #2.doc

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amount of spiked groundwater added. Vigorously hand shake each vial at the beginning of the reaction time and then place the samples in the 12C incubator/shaker. @ 200 RPM

10. At 2 hours from the reaction start time, remove vials 1,2,7,8,13,14 from the incubator and collect a 10 mL aliquot (using a syringe with a Luer Tip). Record the time the sample is collected. (We are now "done" with these 40 mL vials)

11. Dispense (UN filtered) approx. 5 mL of the sample into a pre-weighed vial holding a known volume of hexane (≈5 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis. The hexane phase of these extracts will NOT have to be diluted for analysis.

12. **This step needs to be done fairly quickly since the reaction is not yet "quenched"!!**
Filter (via 0.45 µm syringe filter) the remaining portion of the sample in the syringe (≈5 mL) into a beaker for MnO_4^- analysis. (Some samples may have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO_4^-).

VIALS 1-6 NO Dilution will be Needed
VIALS 7-18 A DF=3 will work fine

Check standards (10 and 50 mg/L MnO_4^-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

13. At 4-5 hours after the start of the reaction, repeat Steps 10-12 using Vials 3,4,9,10,15,16

14. At 24 hours after the start of the reaction, repeat Steps 10-12 using Vials 5,6,11,12,17,18.

15. After the 24 hrs samples are processed, remove the Controls (Vials 19 and 20) from the incubator and collect a 5 mL sample from each, dispensing it (UN Filtered) into a pre-weighed vial holding a known volume of hexane (≈5 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis. **The hexane phase of these two extracts will need a dilution of 101.**

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16. GC ANALYSIS of VOCs
All VOC analyses for this experiment will be conducted using a G.C./ECD detector. The instrument will calibrated for TCE using a 5 point calibration curve with standard concentrations ranging from 5-1000 µg/L (ppb). The VOC analyses of the hexane extracts will closely follow EPA Method 8000B. Significant deviations from this method will be clearly documented. Formal data packages will not be prepared for these analyses.

17. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

Flask #	Start Time	wt. empty	wt. KMnO4
1			
2			
3			
4			
5			
6			
7			
8			

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Subject Extra Exp. #2

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To	vial#	wt. empty	wt. + $KMnO_4$	wt. + spike soln.	sample i.d.
1152	1	24.857	25.154	66.657	0.3 ml – 2000ng/L MnO_4
1153	2	25.082	25.389	67.549	0.3 ml –
1154	3	25.078	25.374	68.549	0.3 ml –
1154	4	25.280	25.575	67.958	0.3 ml –
1155	5	25.047	25.345	67.371	0.3 ml –
1156	6	25.238	25.532	67.691	0.3 ml –
1156	7	25.164	26.351	67.098	1.2 ml
1157	8	24.942	26.137	66.941	1.2 ml
1157	9	25.227	26.427	67.405	1.2 ml
1158	10	25.088	26.291	67.205	1.2 ml
1159	11	25.209	26.405	67.449	1.2 ml
1159	12	24.862	26.065	67.571	1.2 ml
1200	13	23.763	25.716	66.787	2.0 ml
1200	14	24.505	26.478	66.942	2.0 ml
1201	15	24.530	26.488	66.810	2.0 ml
1201	16	25.134	27.102	67.617	2.0 ml
1202	17	25.131	27.083	67.953	2.0 ml
1202	18	25.270	27.226	67.312	2.0 ml
1203	19	24.961	—	68.004	— ml $KMnO_4$
1203	20	24.996	—	67.410	— ml $KMnO_4$

Spec. Results

Vial#	Abs. at 525	dilutions	Vial#	abs. at 525	dilutions
1	0.281	—	11	0.348	3
2	0.284	—	12	0.285	3
3	0.281	—	13	~~0.69~~ KM 0.609	3
4	0.276	—	14	0.631	3
5	0.263	—	15	0.627	3
6	0.258	—	16	0.594	3
7	0.365	3	17	0.529	3
8	0.363	3	18	0.564	3
9	0.389	3	19	0.005	—
10	0.378	3	20	0.001	—

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SRA — Read and understood by — Date 5/7/98

Subject Extra Exp #2

Date 4 (Month) 28 (Day) 19 98 (Year)

SRC

	Sample Time	GC#	wt. empty	wt. + hexane	wt. + sample	Sample I.d.
4/28/98	1352	285	11.116	14.361	19.355	MnO4-7-0.3-2hr
	1352	286	10.992	14.266	19.163	MnO4-2-0.3-2hr.
	1353	287	11.075	14.344	19.450	MnO4-7-1.2-2hr.
	1354	288	10.925	14.173	19.228	MnO4-8-1.2-2hr
	1355	289	10.977	14.205	19.014	MnO4-13-2.0-2hr.
	1356	290	11.045	14.257	19.350	MnO4-14-2.0-2hr.
	1552	291	11.028	14.248	19.461	MnO4-3-0.3-4hr.
	1553	292	11.115	14.324	19.437	MnO4-4-0.3-4hr.
	1554	293	11.130	14.304	19.271	MnO4-9-1.2-4hr.
	1555	294	11.111	14.332	19.597	MnO4-10-1.2-4hr.
	1556	295	11.123	14.318	19.414	MnO4-15-2.0-4hr.
	1556	296	11.088	14.277	19.446	MnO4-16-2.0-4hr.
4/29/98	1120	297	11.079	14.396	19.511	MnO4-5-0.3-24hr.
SRC	1121	298	11.022	14.273	19.317	MnO4-6-0.3-24hr
	1122	299	11.013	14.279	19.194	MnO4-11-1.2-24hr
	1123	300	11.083	14.327	19.404	MnO4-12-1.2-24hr.
	1124	301	10.991	14.204	19.312	MnO4-17-2.0-24hr.
	1125	302	10.969	14.149	19.283	MnO4-18-2.0-24hr.
	1127	303	11.005	14.195	19.207	~~Blank~~ Ø-19-Ø-24hr
	1128	304	11.158	14.358	19.345	~~Blank~~ Ø-20-Ø-24hr.

T2hr. Blank 0.000
Std 5ong/L 1.052
Std 10ng/L 0.198
stock 0.538 = DF80

T24hr. Blank 0.000
Std 5ong/L 1.064
std 10ng/L 0.205

T4hr. Blank 0.000
Std 5ng/L 1.056
Std 10ng/L 0.208

1128	305	11.101	14.341	19.052	Ø-19-Ø24hr-S4618
1129	306	10.990	14.256	19.000	Ø-19-Ø24hr-S4523

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SRC — Read and understood by — Date 5/7/98

Subject Exp 3a-R3

Date 04 (Month) 30 (Day) 1998 (Year)

Exp. 3a - R3

General Procedure for Task 3a Experiments - Oxidant Demand of Reactive Zone Media

(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with a consistent and controlled initial TCE concentration (≈ 0 mg/L). Properly indicate usage of this media in the treatability study tracking logbook.

2. Follow Table 2-3 in Chapter 2 of the Implementation Plan to obtain which test conditions to (Runs) evaluate (soil type, oxidant concentration, etc.) The use of glass, wide-mouth erlenmeyer flasks, beakers, or equivalent will be used. It is currently planned to run four (4) test conditions from Table 2-3 each time this procedure is used. Thus, 8 reactors will be used during each run of this procedure.

3. Obtain the tare weight of each reactor. Add approximately 100g of solid media to the reactors in Table 2-3 that require it Re-weigh the reactors to determine the actual mass added. To each reactor, add 400 mL of TAN-40). Re-weigh each reactor to determine the actual weight of solution added. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Place the reactors in the incubator at 12°C and allow to equilibrate to that temperature. ~~Slowly~~ agitate the reactors during this step. ≈ 200 RPM

5. Once temperature equilibrium is reached, remove the reactors from the incubator and collect a 5 mL aliquot (using a disposable syringe) of the water and/or slurry from each for pH analysis.

6. The pH analyses will be conducted using conventional pH meters/electrodes, calibrated each day of use via a minimum of two standard buffer solutions (4, 7, and 10). Values obtained for each sample will be recorded. A calibration check will also be conducted following the last sample to be measured each day. Values obtained for the samples will be corrected if a significant drift (i.e., > 15%) occurred.

7. Add the appropriate mass of crystalline $KMnO_4$ (Carus™ Free Flowing Technical Grade) into each reactor according to which test condition in Table 2-3 is being evaluated. The exact mass of $KMnO_4$ added to each reactor will be determined gravimetrically and recorded. The time that $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Shake w/ hand (Rubber Stopper) @ START TO Ass. st in getting it into solution.

8. Place each reactor back into the 12°C incubator as the oxidant consumption reaction proceeds. For each reactor, remove the reactor from the incubator at approximately 5-10 minutes from the reaction start time, and collect a 5-10 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator. ≈ 200 RPM

9. Add a 0.45 µm syringe filter onto the end of the syringe and filter approximately 1-2 mL into a beaker. Remove the syringe filter and dispense the remaining (unfiltered) portion of the sample

→ For 1% MnO_4^-, we need 1.32% $KMnO_4$ or 13,277 mg/L
Therefore For ≈ 400 mL soln we need 5.3 grams per flask!

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SP — Read and understood by — 5/8/98 — Date

Subject Exp. 3a - R3

Date 04 30 19 98
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into another beaker for pH analysis. Measure and record the pH of each sample as done in Step 6.

10. Perform a MnO_4^- analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%. DF needed ≈ 500 maybe best to do this in a vol. flask

11. Collect and analyze aliquots from each reactor for pH and $KMnO_4$ (Steps 8-10) at the following ADDITIONAL time periods: 1hr, 2 hrs, 4 hrs, 18 hrs, and 24 hrs.

12. Interpret results and clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial ~~Potassium~~ Permanganate Concentration (%)
Exp. 3a-R3 RUN 3	Groundwater	NA	NA	0	0	1.00
	Groundwater	NA	NA	0	0	1.00
	Groundwater/ basalt	Crushed	4:1	0	0	1.00
	Groundwater/ basalt	Crushed	4:1	0	0	1.00
	Groundwater/ basalt	Aggregate	4:1	0	0	1.00
	Groundwater/ basalt	Aggregate	4:1	0	0	1.00
	Groundwater/sediment	NA	4:1	0	0	1.00
	Groundwater/sediment	NA	4:1	0	0	1.00

K. McCracken 4/30/98

Sample #	flask wt.	flask+media	flask + H_2O	Sample id.
1	223.780	—	644.18	groundwater
2	214.424	—	640.56	groundwater
3	213.836	313.836	732.50	crushed basalt
4	161.133	261.335	688.14	crushed basalt
5	219.567	319.672	737.17	aggregate basalt
6	151.070	251.470	677.87	aggregate basalt
7	161.800	262.026	687.05	sediment
8	151.954	251.978	669.17	sediment

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Subject Exp 3a-23

Date 04 (Month) 30 (Day) 19 98 (Year)

Flask#	KMnO4(g)	T0	T15min	T1hr.	T2hr.	T4hr.	T20hr.	T24hr. (5/1)
1	5.284	1232	1248	1335	1440	1625	0835	1240
2	5.312	1233	1249	1337	1442	1626	0836	1241
3	5.274	1234	1250	1338	1443	1627	0837	1242
4	5.258	1236	1252	1338	1445	1628	0838	1243
5	5.309	1237	1253	1339	1446	1628	0839	1245
6	5.273	1239	1254	1340	1448	1629	0841	1246
7	5.304	1240	1255	1341	1449	1631	0842	1247
8	5.287	1242	1256	1342	1450	1632	0843	1247

pH Readings.

Flask	T0	T15min	T1hr	T2hr	T4hr	T20hr	T24hr
1	8.37	8.82	8.79	8.80	8.79	8.44	8.36
2	8.40	8.84	8.86	8.85	8.83	8.46	8.43
3	8.36	8.73	8.61	8.51	8.43	8.27	8.26
4	8.36	8.71	8.58	8.47	8.39	8.25	8.23
5	8.36	8.82	8.80	8.77	8.67	8.26	8.25
6	8.32	8.81	8.79	8.76	8.66	8.29	8.27
7	7.95	7.66	7.57	7.56	7.55	7.53	7.51
8	7.97	7.65	7.50	7.50	7.52	7.59	7.57

Spec. Readings

Flask	T0	T15min DF500	T1hr DF500	T2hr DF500	T4hr DF500	T20hr DF500	T24hr DF500
1		0.374	0.440	0.415	0.464	0.432	0.438
2		0.297	0.396	0.397	0.466	0.430	0.455
3		0.341	0.406	0.426	0.439	0.451	0.436
4		0.356	0.427	0.413	0.476	0.439	0.450
5		0.353	0.416	0.421	0.459	0.453	0.451
6		0.322	0.386	0.411	0.449	0.451	0.444
7		0.304	0.435	0.425	0.455	0.446	0.470
8		0.298	0.471	0.438	0.446	0.450	0.440

T15min	T1hr.	T2hr.	T4hrs	T20hr	T24hr.
BLK. 2000	BLK 0.000	BLK 0.000	BLK 0.000	BLK 0.000	BLK 0.000
50g/L 1.055	50mg/L 1.055	50mg/L 1.058	50mg/L 1.067	50mg/L 1.067	50mg/L 1.068
10mg/L 0.205	10mg/L 0.205	10mg/L 0.205	10mg/L 0.205	10mg/L 0.205	10mg/L 0.205

Continued on page

K McCracken — Recorded by — 4/30/98 Date

SRC — Read and understood by — 5/8/98 Date

Subject Repeat Experiment 2C-1B

Date 05 04-05 19 98
Month Day Year

REPEAT EXPERIMENT:

Experiment 2c-1B. Sorption Kinetics Test for Crushed and Aggregate Basalt.

(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt) will be prepped by sparging and/or drying to remove any residual TCE present on the media. (Properly indicate usage of this media in the treatability study tracking logbook.) Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking.

2. Prepare a STOCK (Saturated) TCE solution (2000 mg/L) by adding 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and allow to equilibrate overnight at room temperature. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.)

3. Sparge approximately 1600 mL of groundwater from TAN-40 of any residual TCE or other VOCs that may have been present.

4. Obtain the tare weight of the container to be used to prepare the spike solution (Tedlar bag). Fill ~~with~~ the Tedlar bag with the sparged TAN-40 GW. By gravimetric methods, determine the amount of groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.) All information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

5. (TUESDAY AFTERNOON) Add the appropriate quantity of the Saturated STOCK TCE solution into the Tedlar bag to produce a TCE spike concentration of 1.0 mg/L. ***SHOULD BE APPROXIMATELY 1.6 ML*** Record volume and/or weight added. (Reactive zone media will only be spiked at 0.1 and 1 mg/L TCE during the Task 4 degradation studies.) This solution can be mixed and equilibrated at least overnite at 12°C in the incubator.

6. After mixing, obtain duplicate aqueous samples using a glass syringe with a valve stopcock and luer needle (5mL) of the spike solution to obtain the initial TCE concentration of the spiked groundwater. Weigh the syringe when filled and then again after the sample is dispensed to the P&T. The duplicate aliquot samples will be analyzed immediately using the Lab 211 P&T (30 minutes each).

7. Obtain the tare weights of thirty six (36) 40 mL VOA vials. Add approximately 10 g of aggregate basalt to 12 of the vials and 10 g of the crushed basalt to another 12 of the these vials. Gravimetrically determine the actual amount of solid media added.

8. Dispense the spiked groundwater from the Tedlar bag into each of the 36 VOA vials that have been prepared, filling them completely with no headspace and cap them. Record the time that each vial is filled as "T=0" for that vial.

Tedlar Bag wt. = 51.87g

Tedlar Bag + H_2O wt. = 1651.65g

H_2O = 1600 ml ⟶ add 1.60 ml Saturated TCE soln.

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Read and understood by Date

Subject Repeat Experiment 2C-1B

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9. Re-weigh each vial after it is completely filled to determine the actual weight of spiked groundwater added to it. Place the vials back into the environmental chamber at 12°C Agitate the vials during this step (200 rpm or greater)

X-177440 Calibration
5/5
wt. actual
50.0g 49.98g

The remaining steps will be performed at the time intervals of approximately 4 hrs, 24h, 2d, 5d, 7d, and TBD. The latter times may be changed depending upon results of the samples obtained for the earlier time periods.

10. Remove six (6) 40 mL vials from the incubator (two with groundwater only, two with crushed basalt, and two containing aggregate basalt.) Collect an aqueous sample (≈ 5mL) [allow the crushed basalt to settle a little before sampling) from each vial using a glass syringe with valve stopcock and luer needle.

5/11 balance calib.
X-177337 wt. 50g
scale 50.0003g

11. Weigh the full syringe and Record the time the sample is collected. Reweigh the syringe after the sample is dispensed into the P&T.

12. Randomly collect and process (steps 10 and 11) a duplicate VOC sample from one of the six VOA vials during each sampling period.

KM Cracken 5/4/98

13. **FOR THE LAST SAMPLING PERIOD STUDIED ONLY:** Extract the remaining contents of each vial with hexane. (The mass of any remaining TCE on the solid phase can be determined with this extraction when a mass balance is performed using data from the aqueous aliquot sample) To accomplish this, transfer the remaining sample in the six 40 mL VOA vials (including basalt material) into a 125 mL extraction jar that has been pre-weighed and holding a known volume of hexane (≈ 35 mL). The extraction jar will be weighed again after sample addition to determine the actual mass of sample (solid +Liquid) added.

14. **FOR THE LAST SAMPLING PERIOD STUDIED ONLY:** The extracts in the above step will be placed on a horizontal or reciprocating shaker for 2 hours. A 1 mL aliquot of the hexane phase will be placed into a 2 mL G.C. vial and stored at 4°C until analysis.

Vial #	GC #	wt. empty	wt. + medium	wt. + H_2O	Sample I.D.	To	Time Sampled
34	334	25.020	35.167	74.455	2C-1B-T7d-clear	1029	16/16 5/13
35		25.205	35.294	73.933	2C-1B-T0	1030	
36		24.791	~~35.752~~ KM	74.219	2C-1B-T0	1030	

Syringe #	GC #	wt. + Sample	wt. after injection	Sample I.D.	Time Sampled (To)
1	307	70.2210	65.1106	2C-1B-T0-a	0910
2	308	70.1707	65.0550	2C-1B-T0-b	0930

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K. McCracken 5/5/98
Recorded by Date

SR 6/17/98
Read and understood by Date

Subject Repeat experiment 2C-1B

Date 05 (Month) 05 (Day) 1998 (Year)

Vial #	GC#	wt. empty	wt. medium	wt. + H_2O	Sample I.D.	T_0	Time sampled
1	309	25.047	—	67.283	2C-1b-T4hr-gwa	1010	1358 5/6
2	310	24.937	—	67.175	2C-1b-T4hr-gwb	1010	1422 5/6
3	311	24.789	~~33.314~~ 34.827 KM	72.622	2C-1b-T4hr-cba	1011	1602 5/6
4	312	24.670	34.738	73.355	2C-1b-T4hr-cbb	1012	~~1645~~ 1659 5/6
5	313	24.704	35.192	74.067	2C-1b-T4hr-aba	1012	1502 5/6
6	314	24.877	35.016	73.410	2C-1b-T4h-abb	1013	1537 5/6
7	315	25.146	—	68.177	2C-1b-T24hr-gwa	1013	1030 5/7
8	316	25.013	—	67.631	2C-1b-T24hr-gwb	1014	1110 5/7
9	317	24.781	34.874	74.079	2C-1b-T24hr-cba	1015	1140 5/7
10	318	24.764	34.872	73.632	2C-1b-T24hr-cbb	1015	1255 5/7
11	319	24.715	34.841	73.274	2C-1b-T24hr-aba	1016	1324 5/7
12	320	24.931	35.222	73.677	2C-1b-T24hr-abb	1016	1348 5/7
13	321	24.672	—	66.515	2C-1b-T2d-gwa	1017	1110 5/8
14	322	24.798	—	67.462	2C-1b-T2d-gwb	1018	1150 5/8
15	323	24.660	34.800	73.197	2C-1b-T2d-cba	1018	1225 5/8
16	324	24.790	~~34.932~~ 34.932 KM	73.214	2C-1b-T2d-cbb	1019	1250 5/8
17	325	24.827	34.934	72.833	2C-1b-T2d-aba	1019	1330 5/8
18	326	24.865	34.953	72.964	2C-1b-T2d-abb	1020	1400 5/8
19	327	24.911	—	66.829	2C-1b-T5d-gwa	1020	0937 5/11
20	328	24.816	—	67.126	2C-1b-T5d-gwb	1021	1016 5/11
21	329	24.919	34.981	74.277	2C-1b-T5d-cba	1022	1047 5/11
22	330	24.957	35.155	73.925	2C-1b-T5d-cbb	1023	1125 5/11
23	331	24.724	34.927	73.273	2C-1b-T5d-aba	1023	1210 5/11
24	332	24.706	34.962	72.749	2C-1b-T5d-abb	1024	1430 5/11
~~25~~	~~333~~	~~24.876~~	~~—~~	~~67.344~~	~~2C-1b-T7d-gwa~~	~~1024~~	~~1205 5/13~~
~~26~~	~~334~~	~~24.945~~	~~—~~	~~67.515~~	~~2C-1b-T7d-gwb~~	~~1025~~	~~1240 5/13~~
~~27~~	~~335~~	~~24.793~~	~~34.850~~	~~73.686~~	~~2C-1b-T7d-cba~~	~~1025~~	~~1315 5/13~~
28	336	24.755	34.851	74.137	2C-1b-T7d-cbb	1026	1350 5/13
29	337	24.929	34.981	74.033	2C-1b-T7d-aba	1026	1507 5/13
30	~~335~~ 338 KM	24.857	35.036	73.764	2C-1b-T7d-abb	1027	1625 5/13
~~31~~ 32 KM	333	24.707	—	67.299	2C-1b-T7d-gwa	1027	1535 5/13
~~32~~ 31 KM		24.947	—	67.939	2C-1b-T	1028	
33		25.017	35.137	74.038	2C-1b-T	1028	

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K. McCracken — Recorded by — Date 05/06/98

SRC — Read and understood by — Date 6/17/98

Subject Repeat Experiment 2C-1B

Date 05 (Month) 06 (Day) 19 98 (Year)

GC#	syringe + water	syringe	GC#	syringe + water	syringe
309	70.1696	65.0080			
310	70.0613	65.0046			
311	70.2232	64.9997			
312	70.0676	64.9942			
313	70.0803	64.9873			
314	70.0483	64.9714			
315	70.1381	64.9823			
316	70.1784	64.9903			
317	70.1954	64.9705			
318	70.1259	64.9811			
319	70.0851	65.0027			
320	70.0719	64.9783			
321	70.3203	65.1357			
322	70.2890	65.0840			
323	70.1626	65.0624			
324	70.1854	65.0231			
325	70.2442	64.9931			
326	70.3906	65.0333			
327	70.0776	65.0007			
328	70.3200	64.9969			
329	70.1435	64.9811			
330	70.1321	64.9997			
331	70.0539	64.9913			
332	70.0896	65.0019			
333	70.1616	64.9976			
334	70.2573	64.9783			
335	70.1718				
336	70.0876	64.9878			
337	70.1144	65.0001			
338					

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K. McCracken — Recorded by — 5/6/98 Date

SRC — Read and understood by — 5/7/98 Date

Subject Repeat exp. 2a-3
Reactor performance

Date 05 Month 12 Day 19 98 Year

REPEAT (May 1998)

Experiment 2a-3. Overall performance of the test reactor (100 ppb) SRC 5/15

(To be conducted at ORNL/ESD)

In this experiment a complete reactor will be set up using the most optimal component(s) from the previous Task 2a experiments. ~~To be consistent, a 100 mg/L~~ TCE spike solution will be used here as well; however, this experiment will be conducted at approximately 12-15°C.

1. Set up a 500 mL Flask (29/42 S.T.), ensuring that a Teflon sleeve is fitted onto a Standard Taper Stopper. (Will be used during spiking and equilibration to minimize the mass of TCE lost)

2. Fill the reactor with 500 mL ~~bed~~ water. A record of the DI water volume added will be maintained.

3. Spike the reactor with TCE, Desired Concentration: 100 ~~mg/L~~ ppb (SRC) 5/15/98
Add approximately 50 microliters of SATURATED TCE solution to the flask. Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

4. Replace the stopper ~~[illegible]~~ and allow the solution to equilibrate *at least* overnight in an incubator maintained at 12-15°C. (Record the equilibration period and temperature).

5. During the equilibration period, prepare an ORBO-32 assembly and connect it to the stopcock adapter.
STORE UN-SEALED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

6. After equilibration, replace the stopper with the stopcock assembly.

7. Collect duplicate (~~≈0.5 mL~~ 5 mL) aqueous samples from the reactor by opening the stopcock and inserting the long sampling needle. ***(collect one using a glass syringe and one using a plastic disposable syringe)*** The aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (≈5 mL). Each sample container will be weighed again after sample addition to determine the actual weight of sample added. (A calibration check of the gravimetric scale will be performed each day of use.) All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

8. Record the time the last sample in the step above is collected and record as Time=0.

9. Collect at the following time intervals in the same manner as performed in step 7: 1 hour, 2 hrs, 4 hrs, 18 hours, and 24 hrs ***(in duplicate-collect one using a glass syringe and one using a plastic disposable syringe)***. The exact time at which each aliquot is collected will be recorded.

10. Breakdown the reactor after the last sample is collected. The charcoal desorption tube will then be removed from the reactor. Each charcoal bed contained in the tube will be placed into a pre-filled and pre-weighed vial containing a known volume of hexane (≈5 mL). These extracts will be processed and analyzed in the same manner as the aqueous aliquots.

11. Analyze the sample and charcoal extracts via GC/ECD for TCE. G.C. analyses will be conducted closely following EPA Method 8000B, ensuring that a daily calibration check, blanks, etc. are also analyzed on each analysis day. Formal data packages, as outlined in Section 2.4.3.3 of the Chapter 2 Implementation Plan will not be prepared for this experiment.

12. Perform a mass balance for the reactor, taking sample aliquot volumes, initial concentrations, etc. into account to determine the overall reactor performance in maintaining gas-tight conditions. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

calib. X-17740 wt. 50g scale 49.95g
flask: 215.05g
flask + H_2O: 715.35g
H_2O = 500.3g
add 50 µl TCE sat. soln.
syringe + TCE sat. soln. 14.3775
syringe 14.3768
TCE = 60.7 µl
spiked at 1310 5/12/98
incubated at 12°C.

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Recorded by Date

SRC 5/15/98
Read and understood by Date

Subject Exp 2a-3
Reactor Performance

Date 05 (Month) 12 (Day) 19 98 (Year)

Sample Time	GC#	wt. empty	wt. before	wt + sample	Sample i.d.	vial #	dil.
5/13 1110	338	11.031	14.280	19.204	2a-3b-T0-p*	1	1
5/13 1112	339	10.985	14.289	19.250	2a-3b-T0-g	2	1
5/13 1207	340	11.035	14.339	19.571	2a-3b-T1hr-p	3	1
5/13 1210	341	11.135	14.465	19.294	2a-3b-T1hr-g	4	1
5/13 1316	342	10.933	14.236	19.210	2a-3b-T2hr-p	5	1
5/13 1318	343	11.093	14.403	19.630	2a-3b-T2hr-g	6	1
5/13 1510	344	11.120	14.420	19.586	2a-3b-T4hr-p	7	1
5/13 1511	345	11.046	14.324	19.535	2a-3b-T4hr-g	8	1
5/14 0815	346	10.909	14.191	19.124	2a-3b-T21hr-p	9	1
5/14 0815	347	11.015	14.261	19.165	2a-3b-T21hr-g	10	1
5/14 1110	348	11.052	14.273	19.108	2a-3b-T24hr-p	11	1
5/14 1111	349	11.033	14.276	19.007	2a-3b-T24-hr-g	12	1 (SR)

* the notations "p" = plastic syringe, and "g" = glass syringe

5/14 1112	350	11.027	14.291	14.383	2a-3b-charcoal 1	#13	1
5/14 1112	351	11.107	14.382	14.431	2a-3b-charcoal 2	#17	1

Continued on page

K. McCracken — Recorded by — 5/12/98 Date

SPC — Read and understood by — 5/15/98 Date

Subject Task 4a Experiments.

Date 05 (Month) 19 (Day) 19 98 (Year)

General Procedure for Task 4a Experiments - Measure the oxidation rate of organic contaminants in the Reactive Zone Media

(To be conducted at ORNL/ESD)

NOTE: This experiment will be used to conduct the various test conditions presented in **Table 2-5** in Chapter 2 of the Implementation Plan. Control samples will be prepared and sampled throughout the reaction period to quantify changes in concentration due to mechanisms other than oxidation. In addition to the duplicate control samples, it is currently planned to run two (2) test conditions (in duplicate) from **Table 2-5** each time this procedure is used. Thus, 6 reactors will be used during each run of this procedure, and this procedure will be conducted 8 times. The controls and the treatment reactors will all contain the same media type and initial contaminant concentrations (i.e., all conditions will be the same for each run of this procedure with the exception of the MnO4- concentration used).

1. Soil Prepping

All soil and groundwater to be used in these experiments (**TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment**) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking. (Properly indicate usage of this media in the treatability study tracking logbook.) **TAN-40 GW** will also be temperature equilibrated to 12°C in this experiment.

2. TCE Stock Solution Preparation

Prepare a STOCK (Saturated) TCE solution (≈1100 mg/L) by adding greater than 100 µL of Pure TCE into a 12 mL VOA vial filled to volume with DI Water and keep refrigerated. (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.) This can easily be stored and used again in subsequent experiments.

3. TCE Spike Solution Preparation, PART I

Obtain the tare weight of the container to be used to prepare the spike solution (Tedlar bag, flask, or equivalent). Fill with approximately 3100 mL of 12°C **TAN-40 groundwater**. By gravimetric methods, determine the amount of groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.)

4. TCE Spike Solution Preparation, PART II

Add the appropriate quantity of the Saturated STOCK TCE solution to produce a TCE spike solution with the concentration prescribed in Table 2-5 for the particular test series being ran. The **reactive zone media** will only be spiked at **0.1 and 1 mg/L TCE** during this experiment, thus the volume of saturated STOCK TCE needed should be on the order of **0.3 mL and 3.0 mL**, respectively. This solution can be mixed and equilibrated within a few minutes. All Information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook. Place the spike solution in the incubator overnight at 12C for temperature equilibration.

5. Obtain Tare Weights of Reactors

Prepare the test reactors to be used. (The gas-tight reaction vessels acquired and tested in Task 2a will be used as the reactors in this experiment.) Obtain the tare weight of each, including the weight of a ground glass standard taper stopper fitted with a Teflon sleeve.

6. Fill with Solid Phase Media

Add approximately 100-110 g of the solid phase material desired for this particular test series (See **Table 2-5** in the Implementation Plan). Re-weigh each reactor to determine the actual amount of solid media added (when applicable).

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K McCracken — Recorded by — Date 05/19/98

SRC — Read and understood by — Date 6/25/98

Subject Task 4a Experiments.

Date 05 (Month) 19 (Day) 19 98 (Year)

7. Collection of Initial Spike Solution Samples
Remove the spike solution from the incubator, and obtain duplicate aqueous samples (≈ 5 mL) of the spike solution to obtain the initial TCE concentration of the spiked groundwater. Record the time the sample is collected. The duplicate aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (≈ 5 mL). Each sample container will be weighed again after sample addition to determine the actual weight of sample added. These extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

8. Distribution of Spike Solution to Reactors
Quickly dispense the spike solution into each of the reactors to be used. Add ≈500 mL to each reactor. Immediately replace the ground glass stopper and re-weigh each to determine the actual weight of spiked groundwater.

9. Soil/Spike Equilibration — SKIPPED FOR RUN 1 + RUN 2
Place the reactors in the incubator at 12°C and allow the spike solution to equilibrate for 24 hours. (For aggregate basalt, equilibrate for the length of time determined in Exp. 2c-1., unless shorter than 24 hours) Agitate the reactors at ≈200 rpm during this step.

10. Preparation of Stock MnO_4^- Solution (4 wt%)
Add 2.66 g of crystalline KMnO4 (Carus™ Free Flowing Technical Grade) into a 50 mL Flask and fill to volume with DI water, and allow to mix for at least 2 hours.

11. Prepare Reactor Closure/Carbon Tube
During equilibration period, prepare the standard taper adapters/stopcock valve. (Only one ORBO tube will be used for each reactor, the vertical sampling port will be fitted with a small septum) STORE UN-SEALED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

12. Collection of Pre-Reaction Sample from Each Reactor
After equilibration, collect a ≈10 mL aqueous sample using a long need and disposable syringe from each of the reactors and quickly reseal. Record the time the sample is collected and note whether solids were present in the aqueous sample (particularly the groundwater/sediment reactors). If significant solids are present, a portion of this aliquot volume may be used for a total solids analysis.

13. Preparation of Pre Reaction TCE Sample
Dispense (unfiltered) ≈3 mL of this aliquot sample into to a pre-weighed vial holding a known volume of hexane (≈3 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis. Record the time this sample is taken.

14. Measurement of Pre Reaction pH Value
Tare a small beaker for pH analysis. Add a 0.45 micron filter to the sample syringe and dispense (filtered) the remaining portion of the sample in the syringe (≈7 mL) into the tared beaker. Reweigh the beaker to determine the volume of the sample (assuming that the solids fraction is negligible). The pH analyses will be conducted using conventional pH meters/electrodes, calibrated each day of use via a minimum of two standard buffer solutions. Values obtained for each sample will be recorded. A calibration check will also be conducted following the last sample to be measured each day. Values obtained for the samples will be corrected if a significant drift (i.e., > 15%) occurred.

15. Addition of Stock MnO4- Solution into Reactors
Quickly remove the ground glass stopper and add the needed volume of stock $KMnO_4$ to obtain the MnO_4^- concentrations in **Table 2-5** (approximately **1.25 mL and 12.5 mL** for the **0.01 and** 0.1 ~~0.2~~% MnO_4^- concentrations respectively). Replace the glass ground stoppers with the adapters fitted with the stopcock and charcoal desorption tubes and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. (No oxidant will be added to the control reactors). The time that the stock $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12°C incubator.

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16. 30 Minute Sampling
At approximately 30 minutes from the reaction start time, remove the reactors from the incubator and collect a 10 mL aliquot (using a long needle and syringe with a Luer Tip). Record the time the sample is collected. Replace the reactor(s) back into the incubator.

17. Preparation of 30 Minute TCE Sample
Dispense (unfiltered) ≈3 mL of the collected aliquot into a pre-weighed vial holding a known volume of hexane (≈3 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

18. pH Analysis of 30 Minute Sample
Tare a small beaker. Add a 0.45 μm syringe filter unto the end of the syringe and dispense (filtered) the remaining portion of the sample in the syringe (≈7 mL) into a tared beaker for pH analysis. Reweigh the beaker to determine the volume of the sample (assuming that the solids fraction is negligible). Measure and record the pH of the sample as done in Step 13

19. MnO_4^- Analysis of the 30 Minute Sample
Use a portion of the filtered pH sample to perform a MnO_4^- analysis. The sample may have to be diluted (**likely DF3 and DF30 for the .01 and 0.1% MnO4- loadings, respectively**) so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

20. Collection and Analysis of Additional Aliquot Samples
Collect and analyze aliquots from each reactor for TCE, pH, and $KMnO_4$ (Steps 16-19) at the following ADDITIONAL time periods: 2 hrs, 4 hrs, 6 hrs, 20 hrs, and 24 hrs.
IMPORTANT—For each reactor collect and extract a duplicate sample (Steps 16-19) from one of the 6 time intervals being evaluated.

21. Extraction of Residual TCE in Reactor
The entire contents of each reactor will be extracted with hexane. First, obtain a tare weight on six (6) empty containers to be used for the hexane extraction (1 liter VOA bottles). Measure out 400 mL of hexane into two (2) 200 mL volumetric flasks. Add 200 mL of the hexane directly into the 1 L extraction bottle. Quickly pour the contents of each test reactor into a separate extraction bottle. Use the remaining 200 mL flask of hexane to rinse any residual slurry from the test reactor into the extraction bottle. Weigh and record the mass of the filled extraction bottle to determine the mass of the slurry and hexane added (taking into account the fact that hexane has a specific gravity of 0.66).
(The mass of any remaining TCE on the solid phase can be determined with this extraction when a mass balance is performed using data from the aqueous aliquot sampling that was conducted throughout the test.)

22. Reactor Extraction Continued
Place each extraction bottle on a horizontal reciprocating shaker for a minimum of 2 hours (May be longer for aggregate basalt samples, depending upon the results of Exp. 2b-2.) then collect duplicate **1 mL hexane aliquots from each extraction bottle and place into 2 mL G.C. vials (dilution is not expected to be necessary).**

23. Extraction of Charcoal Desorption Tubes
Remove the charcoal desorption tube(s) from each reactor. Each charcoal bed contained in the tube(s) will be placed into pre-filled and pre-weighed vials containing a known volume of hexane (≈5 mL). These extracts will be processed and analyzed in the same manner as the aqueous aliquots for TCE.

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24. Clean up
Clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

25. GC ANALYSIS of VOCs
All VOC analyses for this experiment will be conducted using a G.C./ECD detector. The instrument will calibrated for TCE using a 5 point calibration curve with standard concentrations ranging from 5-1000 µg/L (ppb)

The VOC analyses of the hexane extracts will closely follow EPA Method 8000B. Significant deviations from this method will be clearly documented.

Formal data packages will be prepared for the analyses performed for all Task 3 and 4 samples will be prepared as discussed in Sections 2.4.3 and 2.4.4 in Chapter 2 of the Implementation Plan. The major QA/QC items pertaining to the VOC analyses include:

Calibration standards will be prepared from a certified, custom VOC mix stock solution containing each analyte of interest. The lot number of this custom mix will be included on each initial calibration data form.

The working calibration curve will be verified through injection of a QC check standard (100 µg/L) at least once each analysis day. If the response for any analyte varies from the predicted response by more than ± 15%, a new calibration curve will be prepared.

A blank hexane sample will be analyzed between every 10 test samples.

A sample will be diluted and reanalyzed if its GC peak area response is greater than that of the highest calibration standard being used that analysis day.

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Table 2-5. TCE degradation rate in reactive zone groundwater and basalt

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial Permanganate Concentration (%)
	CONTROL Groundwater	NA	NA	0.10	0	0
	CONTROL Groundwater	NA	NA	0.10	0	0
EXP 4a	Groundwater	NA	NA	0.10	0	0.01
RUN 1	Groundwater	NA	NA	0.10	0	0.01
	Groundwater	NA	NA	0.10	0	0.10
	Groundwater	NA	NA	0.10	0	0.10

balance calib.
X 177440
wt. 50g
scale 49.95g

Tedlar bag - 53.12 g

Tedlar bag + gw. - 3263.2 g

groundwater - 3210.08 g

TCE sat. soln. stock added to get 0.1 mg/L TCE = 0.300 ml

Calib. X - 177.440 50 - 49.95

flask	wt.	wt. + Gw.	Gw vol.
1	241.280	779.63	538.35
2	252.583	772.01	519.43
3	238.018	764.28	526.26
4	255.823	782.27	526.45
5	228.770	770.75	541.98
6	252.514	783.33	530.82

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~~Sample Time~~

Flask #	$KMnO_4$	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1 a	—	1345	1425	1524	1655	1854	5/27 0845	1345
1 b		1348	1427					
2 a	—	1348	1427	1525	1657	1859	0846	1346
2 b				1527				
3 a	1.25 ml	1350	1431	1529	1658	1902	0847	1347
3 b								
4 a	1.25 ml	1352	1433	1531	1600	1904	0848	1348
4 b					1602			
5 a	12.5 ml	1353	1435	1533	1604	1906	0849	1349
5 b						1907		
6 a	12.5 ml	1354	1437	1534	1606	1912	0850	1351
6 b							0851	

pH Readings

Flask #	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1 a	7.84	8.12	8.10	8.09	8.09	8.14	8.15 / 8.17
1 b	8.13	8.11					
2 a	8.13	8.10	8.10	8.09	8.15	8.16	8.18
2 b			8.12				
3 a	8.13	8.11	8.11	8.10	8.14	8.16	8.15
3 b							
4 a	8.13	8.10	8.10	8.11	8.09	8.14	8.13
4 b				8.10			
5 a	8.13	8.18	8.19	8.18	8.19	8.23	8.22
5 b					8.18		
6 a	8.13	8.20	8.20	8.20	?	8.24	8.21
6 b						8.23	

Spec Readings → 1 & 2 NO DIL: 3 & 4 DF 5: 5 & 6 DF 50.

Flask #	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1 a	0.003	0.003	0.002	0.002	NT	NT	0.002
1 b	0.002						
2 a	0.004	0.001	0.000	0.001	NT	NT	0.001
2 b		0.004					
3 a	0.003	0.380	0.386	0.366	0.371	0.361	0.361
3 b			0.374				
4 a	0.002	0.402	0.408	0.394	0.393	0.391	0.388
4 b				0.367			
5 a	0.010	0.398	0.406	0.380	0.392	0.423	0.415
5 b					0.393		
6 a	0.009	0.415	0.398	0.420	0.417	0.417	0.424
6 b						0.413	

Std. spec. checks.

	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr	T48hr
BLK	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50 mg/L	1.086	1.086	1.071	1.080	1.079	1.083	1.091	
10 mg/L	0.228	0.228	0.223	0.228	0.222	0.217	0.221	

Spec. Reading Stock $KMnO_4$ Soln.
0.364
DF 2500

what hour is this? - 3hr KM

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Sample Time	GC#	wt. empty	wt. + hexane	wt. + sample	sample ID
0845	352	10.979	~~19.557~~ 14.287	19.582	4a-R1-TB*-a
0845	353	11.068	~~13.810~~	19.758	4a-R1-TB-b
1345	354	11.154	13.052	16.379	4a-R1-T0-1a
1347	355	11.000	12.923	16.224	4a-R1-T0-1b
1348	356	11.064	12.948	16.308	4a-R1-T0-2
1350	357	11.011	12.897	16.461	4a-R1-T0-3
1352	358	11.117	13.000	16.402	4a-R1-T0-4
1353	359	11.027	12.824	15.838	4a-R1-T0-5
1354	360	11.115	12.871	16.213	4a-R1-T0-6
1425	361	11.105	12.893	16.335	4a-R1-T30min-1
1427	362	11.004	12.908	16.012	4a-R1-T30min-2a
1429	363	11.150	12.923	16.406	4a-R1-T30min-2b
1430	364	11.077	12.931	15.937	4a-R1-T30min-3
1431	365	11.051	12.905	15.821	4a-R1-T30min-4
1433	366	11.021	12.892	16.448	4a-R1-T30min-5
1435	367	11.011	12.909	16.614	4a-R1-T30min-6
1524	368	11.119	13.046	16.742	4a-R1-T1.5hr-1
1525	369	10.989	12.903	15.986	4a-R1-T1.5hr-2
1526	370	10.960	13.006	16.224	4a-R1-T1.5hr-3a
1528	371	11.026	12.817	15.642	4a-R1-T1.5hr-3b
1530	372	11.016	12.935	16.244	4a-R1-T1.5hr-4
1532	373	11.173	13.109	16.556	4a-R1-T1.5hr-5
~~1534~~ 1534	374	11.129	12.903	16.034	4a-R1-T1.5hr-6
~~1655~~ 1655	375	11.104	12.907	15.924	4a-R1-T3hr-1
~~1657~~ 1657	376	11.062	13.010	16.245	4a-R1-T3hr-2
~~1658~~ 1658	377	11.994	13.039	16.314	4a-R1-T3hr-3
~~1700~~ 1700	378	11.002	13.009	16.071	4a-R1-T3hr-4a
~~1702~~ 1702	379	11.056	13.090	16.223	4a-R1-T3hr-4b
~~1703~~ 1703	380	11.130	13.151	16.318	4a-R1-T3hr-5
~~1705~~ 1705	381	11.037	13.043	16.252	4a-R1-T3hr-6
1854	382	11.195	13.198	16.200	4a-R1-T5hr-1
1859	383	11.189	13.228	16.129	4a-R1-T5hr-2

*TB = Tedlar bag

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sample time	GC#	wt. empty	wt. + hexane	wt. + sample	sample ID
1902	384	11.099	13.119	16.315	4a-R1-~~T6hr-3~~ T5hr-3
1904	385	11.120	13.112	16.222	4a-R1-~~T6hr-4~~ T5hr-4
1906	386	11.137	13.072	16.489	4a-R1-~~T6hr-5a~~ T5hr-5a
1907	387	11.038	13.044	16.369	4a-R1-~~T6hr-5b~~ T5hr-5b
1912	388	11.002	13.093	16.247	4a-R1-~~T6hr-6~~ T5hr-6
5/27 0845	389	11.092	13.275	16.050	4a-R1-T20hr-1
0846	390	11.145	13.229	16.300	4a-R1-T20hr-2
0847	391	11.062	13.172	16.386	4a-R1-T20hr-3
0848	392	11.127	13.239	16.318	4a-R1-T20hr-4
0849	393	11.042	13.153	16.112	4a-R1-T20hr-5
0850	394	11.074	13.190	16.319	4a-R1-T20hr-6a
0851	395	11.053	13.153	16.687	4a-R1-T20hr-6b
1345	396	11.067	13.178	16.511	4a-R1-T24hr-1a
1346	397	11.023	13.087	16.444	4a-R1-T24hr-1b
1347	398	11.063	13.204	16.268	4a-R1-T24hr-2
1348	399	10.980	13.071	16.187	4a-R1-T24hr-3
1349	400	11.053	13.125	16.078	4a-R1-T24hr-4
1350	401	11.026	13.135	16.274	4a-R1-T24hr-5
1351	402	11.000	13.097	16.103	4a-R1-T24hr-6
1408	403	11.161	13.262	13.346	4a-R1-charcoal 1-a
1408	404	11.153	13.298	13340	4a-R1-charcoal 1-b
1409	405	11.170	13.100	13.174	4a-R1-charcoal 2a
1410	406	10.967	12.936	12.982	4a-R1-charcoal 2b
1411	407	11.122	13.078	13.176	4a-R1-charcoal 3a
1412	408	11.107	13.073	13.117	4a-R1-charcoal 3b
1413	409	11.086	13.059	13.155	4a-R1 charcoal 4a
1413	410	11.204	13.176	13.217	4a-R1 charcoal 4b
1414	411	11.048	13.013	13.110	4a-R1 charcoal 5a
1415	412	11.099	13.068	13.110	4a-R1 charcoal 5b
1416	413	11.017	12.977	13.076	4a-R1 charcoal 6a
1417	414	11.051	12.999	13.040	4a-R1 charcoal 6b
1400	415	493.11	627.04	~~1204.19~~ 1192.23	4a-R1-F3 (wt + more hexane)
1403	416	491.85	625.02	1204.19	4a-R1-F4

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Date 05 (Month) 26 (Day) 19 98 (Year)

Sample time	GC#	wt. empty	wt + hexane	wt. + sample		wt. + more hexane
1405	417	493.36	627.11	1233.74	4a-R1-F5	
1408	418	492.52	626.09	1222.45	4a-R1-F6	

5/27 calib. X177446 wt. 50g Read wt. 49.95g

repeat GC on #'s 387
368
369 !

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calib.
X177440 50g 49.95g ✓

calib.
X177558 - 50g 50.01g ✓

Tedlar bag
56.76g

Tedlar Bag + GW
3215.50g

GW
3158.74

TCE sat. soln. added
3.000ml

please NOTE: deviation from plan

* sparged groundwater was incubated overnight to bring Temp. to 12°C, Then spiked w/TCE saturated soln., mixed 2hrs. to obtain a uniform soln. & used in flasks.

	media	Result form	Liquid: solid ratio	initial TCE conc. (mg/L)	initial organic co-solvent conc. (mg/L)	initial permanganate conc. (%)
	CONTROL Groundwater	NA	NA	1	0	0
	CONTROL Groundwater	NA	NA	1	0	0
EXP 4a	Groundwater	NA	NA	1	0	0.01
RUN 2	Groundwater	NA	NA	1	0	0.01
	Groundwater	NA	NA	1	0	0.10
	Groundwater	NA	NA	1	0	0.10

KM 5/28/98

5/29 calib.
X077440
50g 49.95g ✓

flask	wt.	wt + GW	GW Vol.
1	250.12	761.57	511.45
2	250.20	753.69	503.49
3	238.26	749.64	511.38
4	247.49	743.33	495.84
5	230.24	745.69	515.45
6	251.68	769.81	518.13

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Subject Exp. 4a-R2

Date 05 (Month) 28 (Day) 19 98 (Year)

Flask #	KMnO4	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1 a	—	1120/1130	1200	1304	1437	1620	5/29 0840	5/29 1118
1 b								1118
2 a	—	1122	~~1201~~	1394	1438	1620	0840	1119
2 b			1202	~~1305~~				
3 a	1.25	1124	1203	1305/1305	1439	1622	0841	1120
3 b								
4 a	1.25	1125	1204	1306	1440	1622	0842	1121
4 b					1441			
5 a	12.50	1127	1205	1307	1441	1623	0842	1122
5 b						1624		
6 a	12.50	1129	1206	1308	1442	1625	0843	1124
6 b							0844	

pH readings

Flask #	T0/Vol	T30/Vol	T1.5/Vol	T3/Vol	T5/Vol	T20/Vol	T24/Vol
1 a	~~8.43/7.46~~	8.40/7.37	8.39/7.04	8.36/7.45	8.34/7.32	8.23/7.50	8.18/7.03
1 b	8.39/7.70	8.40/7.29					8.16/7.11
2 a	8.38/7.48	8.41/7.39	8.41/7.44	8.39/7.52	8.36/7.41	8.18/7.46	8.16/7.37
2 b			8.35/7.38				
3 a	8.38/7.36	8.34/6.09	8.32/7.02	8.33/7.06	8.32/7.14	8.21/7.35	8.22/7.26
3 b				8.33/7.06			
4 a	8.40/7.51	8.34/6.20	8.33/7.37	8.33/7.05	8.33/7.10	8.22/7.31	8.23/7.27
4 b					8.39/7.29		
5 a	8.41/7.54	8.38/7.07	8.39/7.19	8.39/7.05	8.39/7.32	8.34/7.21	8.34/7.31
5 b						8.34/7.25	
6 a	8.40/7.36	8.38/7.23	8.39/7.32	8.39/7.22	8.37/7.38	8.34/7.39	8.35/7.20
6 b							

Spec. readings → 142 NDF; 384 DF5; 546 DF50

Flask #	T0	T30	T1.5	T3	T5	T20	T24
1 a	NT/0.001	NT	0.008	0.003	0.003	0.003	0.003
1 b							
2 a	0.002	NT/0.004	0.002	0.002	0.003	0.003	0.002/0.007
2 b							
3 a	0.001	0.353	0.357/0.370	0.354	0.340	0.204	0.304
3 b							
4 a	NT	0.414	0.396	0.375/0.387	0.389	0.269	0.308
4 b							
5 a	NT	0.420	0.418	0.425	0.411/0.417	0.428	0.419
5 b							
6 a	NT	0.422	0.417	0.437	0.427	0.436/0.404	0.416
6 b							

Spec. check stds.

	T0	T30	T1.5	T3	T5	T20	T24
BLK	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.068	1.070	1.068	1.065	1.068	1.070	0.063
10mg/L	0.202	0.200	0.196	0.191	0.191	0.184	0.182

Spec. reading - stock KMnO4 soln.
Abs = 0.354 DF2500

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SR C — Read and understood by — 7/18/98 Date

Subject Exp. 4a-~~4b~~ R2

Date 05 (Month) 28 (Day) 1998 (Year)

sample time	GC#	wt. empty	wt. + hexane	wt. + sample	sample ID. ~~TB-a~~	DF
1115	487	11.130	14.325	19.479	4a-R2-TB-a	2
1115	488	11.097	14.313	19.611	4a-R2-TB-b	2
1120	489	11.092	13.041	16.000	4a-R2-Tφ-1a	2
1122	490	11.161	13.121	16.221	4a-R2-Tφ-1b	2
1124	491	11.059	13.013	16.293	4a-R2-Tφ-2	2
1125	492	11.094	13.038	16.151	4a-R2-Tφ-3	2
1127	493	11.109	13.048	16.084	4a-R2-Tφ-4	2
1129	494	11.033	13.060	16.035	4a-R2-Tφ-5	2
1130	495	11.142	13.099	16.195	4a-R2-Tφ-6	2
1200	496	11.049	13.010	16.152	4a-R2-T30min-1	2
1201	497	11.053	13.024	16.230	4a-R2-T30min-2a	2
1202	498	11.103	13.068	16.218	4a-R2-T30min-2b	2
1203	499	11.135	13.102	16.367	4a-R2-T30min-3	1
1204	500	11.070	13.048	16.392	4a-R2-T30min-4	1
1205	501	10.981	12.952	16.314	4a-R2-T30min-5	1
1206	502	11.014	13.000	16.084	4a-R2-T30min-6	1
1304	503	11.084	13.048	16.221	4a-R2-T1.5hr-1	2
1304	504	11.021	13.000	15.929	4a-R2-T1.5hr-2	2
1305	505	11.060	13.850	16.265	4a-R2-T1.5hr-3a	1
1306	506	10.877	12.962	15.740	4a-R2-T1.5hr-3b	1
1306	507	11.100	13.049	16.104	4a-R2-T1.5hr-4	1
1307	508	11.012	13.038	16.139	4a-R2-T1.5hr-5	1
1308	509	11.038	13.068	16.121	4a-R2-T1.5hr-6	1
1437	510	11.126	13.073	16.226	4a-R2-T3hr-1	2
1438	511	11.214	13.180	16.229	4a-R2-T3hr-2	2
1439	512	11.016	12.982	16.049	4a-R2-T3hr-3	1
1440	513	11.065	13.026	16.060	4a-R2-T3hr-4a	1
1441	514	11.171	13.148	16.333	4a-R2-T3hr-4b	1
1441	515	10.981	12.947	16.353	4a-R2-T3hr-5	1
1442	516	11.117	13.107	16.291	4a-R2-T3hr-6	1
1620	517	11.114	12.859	15.694	4a-R2-T5hr-1	2
1620	518	11.063	13.052	16.152	4a-R2-T5hr-2	2
1621	519	11.129	13.115	16.235	4a-R2-T5hr-3	1

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K. McCracken — Recorded by — 5/28/98 Date

SR C — Read and understood by — 7/8/98 Date

Subject Exp. 4a-R2

Date 05 (Month) 28 (Day) 19 98 (Year)

sample time	GC#	wt empty	wt. + hexane	wt. + sample	sample ID	DF
1622	520	10.959	12.939	16.066	4a-R2-T5hr-4	1
1622	521	10.976	12.953	16.082	4a-R2-T5hr-5a	1
1623	522	11.070	13.048	16.029	4a-R2-T5hr-5b	1
1624	523	10.913	12.908	15.822	4a-R2-T5hr-6	1
5/29 0840	524	~~10.992~~ 11.192	~~10.955~~ 13.132	16.183	4a-R2-T21hr-1	2
0840	525	10.995	~~11.074~~ 12.972	16.103	4a-R2-T21hr-2	2
0841	526	11.104	~~11.146~~ 13.089	16.046	4a-R2-T21hr-3	1
0842	527	10.863	~~10.947~~ 13.846	15.988	4a-R2-T21hr-4	1
0842	528	11.067	~~11.043~~ 13.069	16.301	4a-R2-T21hr-5	1
0843	529	11.135	13.109	16.211	4a-R2-T21hr-6a	1
0844	530	11.053	13.031	16.082	4a-R2-T21hr-6b	1
1118	531	11.161	13.077	16.150	4a-R2-T24hr-1	2
1119	532	~~11.047~~ 11.104	13.077	16.187	4a-R2-T24hr-2a	2
1120	533	11.061	13.002	16.227	4a-R2-T24hr-2b	2
1121	534	11.108	13.056	16.274	4a-R2-T24hr-3	1
1122	535	11.054	12.967	16.015	4a-R2-T24hr-4	1
1123	536	11.022	12.982	16.116	4a-R2-T24hr-5	1
1124	537	10.955	12.895	16.087	4a-R2-T24hr-6	1
1132	538	11.074	13.021	13.122	4a-R2-char-1a	1
1132	539	11.146	13.101	13.154	4a-R2-char-1b	1
1133	540	10.947	12.905	13.001	4a-R2-char-2a	1
1133	541	11.043	12.991	13.039	4a-R2-char-2b	1
1134	542	11.186	13.131	13.233	4a-R2-char-3a	1
1134	543	11.076	13.007	13.060	4a-R2-char-3b	1
1135	544	11.064	13.023	13.124	4a-R2-char-4a	1
1135	545	10.974	12.942	12.992	4a-R2-char-4b	1
1136	546	11.114	13.066	13.175	4a-R2-char-5a	1
1136	547	11.202	13.150	13.194	4a-R2-char-5b	1
1137	548	11.065	13.023	13.128	4a-R2-char-6a	1
1137	549	11.091	13.044	13.102	4a-R2-char-6b	1
1226	550	~~493.78~~ 493.78	624.62	1169.10	~~4a-R2~~-4a-R2-f3	1
1228	551	493.16 ~~[illegible]~~	626.60	1160.00	~~4a-R2~~-4a-R2-f4	1
1230	552	492.78 ~~11.146~~	625.80	1199.70	~~4a-R2~~-4a-R2-f5	1
1231	553	492.31 ~~11.074~~	625.42	1203.10	~~4a-R2~~-4a-R2-f6	1

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K. McCracken — Recorded by — 5/28/98 Date

SR C — Read and understood by — 7/8/98 Date

Subject Exp. 4a R3

Date 06 Month 01 Day 19 98 Year

	Media	basalt form	liquid/solid ratio	initial TCE (mg/L)	initial organic co-contaminant conc (mg/L)	initial Permanganate conc. mg/L
	CONTROL Groundwater	NA	NA	0.1	0	0
	CONTROL Groundwater	NA	NA	0.1	0	0
EXP 4a	Groundwater/basalt	Crushed	4:1	0.1	0	0.01
RUN 3	Groundwater/basalt	Crushed	4:1	0.1	0	0.01
	Groundwater/basalt	Crushed	4:1	0.1	0	0.10
	Groundwater/basalt	Crushed	4:1	0.1	0	0.10

Tedlar bag 43.960 g

Baked basalt overnight.

Tedlar bag + H_2O 3144.9

Groundwater 310.94

sat. soln. TCE stock added: 0.31 mL

Spiked 6/1/98 overnight at 12°C

flask	wt empty	wt + medium (crushed basalt)	wt. + gw	gw. vol
1	245.87	—	746.36	500.49
2	252.22	—	777.16	524.94
3	236.26	336.36	791.22	454.86
4	255.83	356.10	~~803.33~~ 789.22	431.12
5	227.93	328.63	779.66	451.03
6	248.94	349.55	803.33	453.78

Calib.
6/1 X177440 50g 49.95
6/4 X177440 50g 49.96
6/5 X177440 50g 49.97

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K. McCracken 6/1/98
Recorded by Date

Read and understood by Date 7/9/98

Subject Exp. 4a f3

Date 06 (Month) 02 (Day) 19 98 (Year)

flask#	$KMnO_4$	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr.
1	—	1130	1205	1305	1430	1630	6/4 0830	1140
		1131						
2	—	1133	1207	1306	1431	1631	0831	1141
			1209	1307	1432	1632	0832	1142
3	1.25ml	1134	1210	1308				1143
				1309	1433	1634	0833	1144
4	1.25ml	1136	1213	—	1434	1635	0834	1145
5	12.5ml	1137	1214	1310	—	1636	0835	
6	12.5ml	1138	1215	1311	1435	1637	0836	1146

pH Readings

Flask#	T0 /vol	T30m /vol	T1.5h /vol	T3h /vol	T5h /vol	T20h /vol	T24h /vol
1	8.20/7.137	8.15/7.22	8.13/7.51	8.12/6.584	8.13/7.13	8.15/7.52	8.13/7.2
	8.21/7.24	8.16/7.12					
2	8.21/7.20	8.18/7.42	8.18/7.44	8.18/7.33	8.17/7.16	8.16/7.26	8.19/7.36
			8.22/6.84				8.30/6.94
3	8.20/7.04	8.22/6.94	8.21/7.15	8.22/6.91	8.23/6.79	8.26/6.63	8.31/7.00
				8.23/6.95			
4	8.21/7.00	8.23/6.85	8.23/7.19	8.24/7.03	8.24/6.85	8.25/7.04	8.30/7.02
					8.23/6.83		
5	8.22/7.11	8.29/7.00	8.27/7.17	8.24/7.12	8.23/6.85	8.23/6.91	8.24/7.09
						8.20/6.99	
6	8.20/7.05	8.27/7.08	8.25/7.10	8.23/7.04	8.21/6.99	8.21/6.98	8.23/6.85

Spec. readings 1&2 NDF; 3 & 4 DF5; 5 & 6 DF50

Flask	T0	T30m	T1.5h	T3h	T5h	T20h	T24h
1	0.004	0.034	0.008	0.009	0.010	0.013	0.012
	0.006	0.009					
2	0.007	0.007	0.007	0.008	0.010	0.012	0.012
			0.442				0.305
3	0.002	0.463	0.451	0.436	0.422	0.374	0.358
				0.466			
4	0.004	0.465	0.466	0.467	0.459	0.380	0.447
					0.466		
5	0.004	0.487	0.472	0.480	0.482	0.464	0.454
						0.457	
6	0.004	0.494	0.462	0.486	0.477	0.470	0.477

Spec chk stds.

	T0 0.000	T30m 0.000	T1.5h 0.000	T3h 0.000	T5h 0.000	T20h 0.000	T24h 0.000
BLK							
50mg/L	1.074	1.068	1.074	1.072	1.075	1.078	1.076
10mg/L	0.212	0.212	0.212	0.212	0.212	0.212	0.212

$KMnO_4$ Stock soln = 0.369 DF 250 0

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SRC — Read and understood by — Date 7/9/98

Subject Exp. 4a-R3

Date 06 (Month) 03 (Day) 19 98 (Year)

Sample Time	GC#	wt. empty	wt. + hexane	wt. + sample	sample ID	DF
6/2 1315	629	10.991	14.224	19.520	4a-R3-TB-a	1
6/2 1315	630	11.022	14.336	19.331	4a-R3-TB-b	
6/3 1130	631	10.880	12.838	15.827	4a-R3-Tφ-1a	
1131	632	10.974	12891	16.042	4a-R3-Tφ-1b	
1133	633	10.965	12.915	16.189	4a-R3-Tφ-2	
1134	634	10.945	12.905	16.012	4a-R3-Tφ-3	
1136	635	10.992	12.847	16.024	4a-R3-Tφ-4	
1137	636	10.867	12.806	16.015	4a-R3-Tφ-5	
1138	637	10.992	12.936	15.893	4a-R3-Tφ-6	
1205	638	10.878	12.812	15.968	4a-R3-T30m-1	
1207	639	10.949	12.876	15.938	4a-R3-T30m-2a	
1209	640	10.954	12.879	15.871	4a-R3-T30m-2b	
1210	641	10.845	12.752	15.888	4a-R3-T30m-3	
1213	642	10.957	12.875	15.892	4a-R3-T30m-4	
1214	643	10.890	12.794	15.893	4a-R3-T30m-5	
1215	644	10.982	12.891	15.924	4a-R3-T30m-6	
1305	645	11.036	12.929	15.751	4a-R3-T1.5h-1	
1306	646	10.946	12.843	15.812	4a-R3-T1.5h-2	
1307	647	10.901	12.782	15.793	4a-R3-T1.5h-3a	
1308	648	10.854	12.768	15.834	4a-R3-T1.5h-3b	
1309	649	10.936	12.827	15.832	4a-R3-T1.5h-4	
1310	650	10.968	12.872	15.833	4a-R3-T1.5h-5	
1311	651	10.910	12.821	15.947	4a-R3-T1.5h-6	
1430	652	10.949	12.854	16.533	4a-R3-T3h-1	
1431	653	11.000	12.903	16.062	4a-R3-T3h-2	
1432	654	10.924	12.847	16.090	4a-R3-T3h-3	
1433	655	10.909	12.811	15.982	4a-R3-T3h-4a	
1434	656	11.005	12.909	15.993	4a-R3-T3h-4b	
1435	657	10.987	12.897	15.887	4a-R3-T3h-5	
1436	658	10.987	12.890	16.024	4a-R3-T3h-6	
1630	659	10.926	12.842	15.843	4a-R3-T5h-1	
1631	660	10.848	12.751	15.738	4a-R3-T5h-2	
1632	661	11.042	12.951	16.047	4a-R3-T5h-3	

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SRC — Read and understood by — Date 7/9/98

Subject Exp. 4a - R3

Date 06 (Month) 03 (Day) 19 98 (Year)

Sample Time	GC#	wt empty	wt. + hexane	wt. + sample	Sample ID	DF
1634	662	10.911	12.836	15.818	4a-R3-T5h-4	1
1635	663	10.926	12.835	15.998	4a-R3-T5h-5a	
1636	664	10.908	12.821	15.846	4a-R3-T5h-5b	
1637	665	10.975	12.894	15.882	4a-R3-T5h-6	
6/4 0830	666	10.911	12.799	15.789	4a-R3-T20h-1	
0831	667	10.973	12.884	15.883	4a-R3-T20h-2	
0832	668	10.947	12.853	16.030	4a-R3-T20h-3	
0832	669	10.910	12.808	15.942	4a-R3-T20h-4	
0833	670	10.896	12.796	15.966	4a-R3-T20h-5	
0834	671	10.988	12.811	15.926	4a-R3-T20h-6a	
0835	672	10.980	12.890	16.098	4a-R3-T20h-6b	
1140	673	10.894	12.816	15.939	4a-R3-T24h-1	
1141	674	10.992	12.913	15.923	4a-R3-T24h-2	
1142	675	10.949	12.851	15.990	4a-R3-T24h-3a	
1143	676	10.892	12.804	15.910	4a-R3-T24h-3b	
1144	677	11.040	12.915	16.019	4a-R3-T24h-4	
1145	678	10.961	12.876	15.881	4a-R3-T24h-5	
1146	679	10.970	12.873	16.255	4a-R3-T24h-6	
1205	680	10.899	12.800	12.883	4a-R3-char-1a	
1205	681	10.950	12.872	12.918	4a-R3-char-1b	
1207	682	11.043	12.969	13.050	4a-R3-char-2a	
1207	683	11.029	12.944	12.977	4a-R3-char-2b	
1209	684	11.002	12.917	13.006	4a-R3-char-3a	
1209	685	10.934	12.839	12.884	4a-R3-char 3b	
1211	686	10.957	12.857	12.930	4a-R3-char 4a	
1211	687	10.844	12.760	12.800	4a-R3-char 4b	
1213	688	10.886	12.800	12.895	4a-R3-char-5a	
1213	689	11.008	12.910	12.946	4a-R3-char-5b	
1214	690	11.023	12.924	13.014	4a-R3-char-6a	
1214	691	10.952	12.909	12.933	4a-R3-char-6b	
1150	692	492.16	624.44/756.44	1203.71	4a-R3-f3	
1154	693	492.75	625.09/757.09	1198.20	4a-R3-f4	
1157	694	492.85	625.24/757.24	1227.85	4a-R3-f5	
1200	695	492.43	624.54/756.54	1232.37	4a-R3-f6	

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K. McCracken — Recorded by — 6/3/98 — Date

SRC — Read and understood by — 7/9/98 — Date

Subject Prep. of NEW MnO_4^- Curve

Date 06 (Month) 10 (Day) 19 98 (Year)

Preparation of Permanganate Calibration Standards

Instrument: HACH DR/2000 spectrophotometer

The following solutions concentrations will be prepared:

5, 10, 20, 30, and 50 mg/L MnO_4^-.

The 50 mg/L MnO_4^- std will result from the preparation of a 66.4 mg/L $KMnO_4$ solution

Reagent to be used:
CAIROX Potassium Permanganate, Free-Flowing Grade (Carus™)

STD 5 (50 mg/L MnO_4^-) 33.4 mg Added per KM McCracken

Prepare Solution by adding 33.2 mg of KMnO4 into a 500 mL volumetric flask and fill to volume with DI water. Place this solution on a magnetic stir plate for at least 15 minutes before preparing the following standard solutions.

STD 4 (30 mg/L MnO_4^-)
Add 15 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 3 (20 mg/L MnO_4^-)
Add 10 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 2 (10 mg/L MnO_4^-)
Add 5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

STD 1 (5 mg/L MnO_4^-)
Add 2.5 mL of STD5 into 25 mL volumetric and fill to volume with DI water.

Keep these solutions in darkness until used for calibration.
Record the abs obtained for a DI water blank and for each sample
Prepare the linear calibration curve and constants.

Solutions made 6/10/98 Read at 525nm
ZERO - 0.000 (di. water)
Std. 5 - 1.115 (50 mg/L)
Std. 4 - 0.680 (30 mg/L)
Std. 3 - 0.455 (20 mg/L)
Std. 2 - 0.230 (10 mg/L)
Std. 1 - 0.119 (5 mg/L)

SRCline 6/10/98

S.R. Cline Last Updated: 06/10/98 Filename: Preparation of Permanganate Calibration Curve.doc

SRC 6/10/98
Recorded by Date

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SRC 6/11/98
Read and understood by Date

Subject Task3a - repeat - 1B
Measure the Oxidant Demand of Reactive Zone Media

Date 06 (Month) 10 (Day) 1998 (Year)

3.1 Measure the Oxidant Demand of Reactive Zone Media (Task 3a)

General Procedure for Task 3a Experiments - Oxidant Demand of Reactive Zone Media
(To be conducted at ORNL/ESD)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with a consistent and controlled initial TCE concentration (≈ 0 mg/L). Properly indicate usage of this media in the treatability study tracking logbook.

2. Follow the table below to obtain which test conditions to evaluate (soil type, oxidant concentration, etc.) The use of glass, wide-mouth erlenmeyer flasks, beakers, or equivalent will be used. It is currently planned to run four (4) test conditions from Table 2-3 each time this procedure is used. Thus, 8 reactors will be used during each run of this procedure.

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	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial Permanganate Concentration (%)
	DI Water	NA	NA	0	0	0.01
EXP 3a	Groundwater	NA	NA	0	0	0.01
RUN 1b	Groundwater/basalt	Crushed	4:1	0	0	0.01
	Groundwater/basalt	Crushed	4:1	0	0	0.01
	Groundwater/basalt	Aggregate	4:1	0	0	0.01
	Groundwater/basalt	Aggregate	4:1	0	0	0.01
	Groundwater/sediment	NA	4:1	0	0	0.01
	Groundwater/sediment	NA	4:1	0	0	0.01

3. Obtain the tare weight of each reactor. Add approximately 50g of solid media to the reactors in the Table that require it Re-weigh the reactors to determine the actual mass added. Except for reactor #1 (200 mL DI water), add 200 mL of TAN-40). Re-weigh each reactor to determine the actual weight of solution added. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Place the reactors in the incubator at 12°C and allow to equilibrate to that temperature. Slowly agitate the reactors during this step.

flask#	wt. empty	wt. + soil	wt. + H_2O	sample ID
1	209.200	—	409.105	DI water
2	215.999	—	415.945	groundwater
3	204.018	254.052	452.309	GW + crushed basalt
4	215.213	265.214	464.114	GW + crushed basalt
5	193.884	243.743	442.867	GW + aggregate basalt
6	214.912	264.812	464.980	GW + aggregate basalt
7	161.136	211.136	410.995	GW + sediment
8	214.428	264.515	464.128	GW + sediment

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SRC 6/12/98
Read and understood by Date

Subject Task 3a - 1b

Date 06 (Month) 10 (Day) 1998 (Year)

5. For Loadings of .01wt% MnO_4^-
Preparation of Stock MnO_4^- Solution (4 wt%)
Add 2.66 g of crystalline KMnO4 (Carus™ Free Flowing Technical Grade) into a 50 mL Flask and fill to volume with DI water, and allow to mix for at least 2 hours.

6. For Loadings of .01 wt% MnO_4^-
Addition of Stock MnO4- Solution into Reactors
Quickly remove the ground glass stopper and add the needed volume of stock $KMnO_4$ to obtain the MnO_4^- concentrations in the above Table (approximately 0.5 mL for the 0.01 % MnO_4^- concentration).

7. Replace the glass ground stoppers and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. The time that the stock $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12°C incubator and 200 rpm.

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T0	flask #
6/98 1155	1
1155	2
1156	3
1156	4
1156	5
1157	6
1157	7
1157	8

8. For each reactor, remove the reactor from the incubator at approximately 30 minutes from the reaction start time, and collect a 5 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator.

9. Add a 0.45 µm syringe filter onto the end of the syringe and filter the sample.

10. Perform a MnO_4^- analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

Spec. reading 4% std MnO_4 soln. (as $KMnO_4$) 0.514
DF 1666.7 (0.3 ml into 500 ml H_2O)

11. Collect and analyze aliquots from each reactor for ~~pH and~~ diluted $KMnO_4$ (Steps 8-10) at the following ADDITIONAL time periods: 1hr, 2 hrs, 4 hrs, 18 hrs, and 24 hrs.

12. Interpret results and clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

Continued on page 77

K. McCracken — Recorded by — Date

SRO — Read and understood by — Date 6/12/98

Subject Task 3a-1b

Date 06 (Month) 11 (Day) 19 98 (Year)

flask#	T30min	T1hr	T2hr	T4hr	T18hr	T24hr
1	6/11/98 1230	1255	1357	1556	6/12/98 0820	Ø1155
2	1231	1255	1357	1556	0820	1155
3	1231	1256	1357	1557	0820	1156
4	1232	1256	1358	1557	0821	1156
5	1233	1257	1358	1558	0821	1156
6	1233	1258	1359	1558	0822	1157
7	1234	1259	1359	1559	0823	1158
8	1235	1259	1400	1559	0824	1158

spec. readings ~~def.~~

flask#	T30min	T1hr	T2hr	T4hr	T18hr	T24hr
1	0.423	0.415	0.394	0.413	0.408	0.415
2	0.423	0.417	0.419	0.429	0.426	0.411
3	0.405	0.392	0.382	0.375	0.309	0.293
4	0.405	0.395	0.382	0.379	0.315	0.295
5	0.428	0.421	0.411	0.420	0.~~391~~ 375	0.352
6	0.431	0.424	0.414	0.426	0.391	0.366
7	0.364	0.343	0.322	0.309	0.233	0.229
8	0.378	0.365	0.348	0.341	0.293	0.271

spec check	T30min	T1hr	T2hr	T4hr	T18hr	T24hr
ZERO	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.122	1.119	1.120	1.118	1.121	1.117
10mg/L	0.236	0.237	0.235	0.232	0.229	0.228

flask#	DF (dilution factors)
1	5
2	5
3	5
4	5
5	5
6	5
7	5
8	5

Continued on page

K. McCracken — Recorded by — Date

SRC — Read and understood by — Date 6/12/98

Subject Exp. 4A-R5

Date 06 15 19 98
Month Day Year

Tedlar bag 52.033 → GW. 2990.47
Tedlar + GW 3042.50 TCE sat. soln. added - 3.00 ml

KMcCracken 6/15/98

Table 2-5 CONTINUED. TCE degradation rate in reactive zone groundwater and basalt

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial Permanganate Concentration (%)
	CONTROL Groundwater	NA	NA	1	0	0
	CONTROL Groundwater	NA	NA	1	0	0
EXP 4a	Groundwater/basalt	Crushed	4:1	1	0	0.01
RUN 5	Groundwater/basalt	Crushed	4:1	1	0	0.01
	Groundwater/basalt	Crushed	4:1	1	0	0.10
	Groundwater/basalt	Crushed	4:1	1	0	0.10

spiked & equilibrated over night at 12°C. 6/15/98

Flask	wt. empty	wt + medium	wt + gw	g.w vol.	added KMnO4
1	245.54	—	740.72		
2	250.06	—	752.88		
3	238.14	338.14	744.07	See spreadsheets	
4	251.82	351.84	771.88		
5	234.52	334.52	719.31		
6	247.15	347.22	753.48		

Continued on page 79

K. McCracken 6/15/98
Recorded by Date

SPC 7/9/98
Read and understood by Date

Subject Exp. 4A-R5

Date 06 (Month) 15 (Day) 1998 (Year)

flask#	T0	T30min	T1.5hr	T3hr.	T5hr.	T20hr.	T24hr.
1	1100 / 1101	1133 / 1133	1300	1410	1605	6/19 0830	
2	1101	1134	1301 / 1301	1410	1606	0830	
3	1102	1135	1302	1411 / 1412	1607	0831	
4	1103	1136	1302	1413	1608 / 1608	0831	
5	1103	1136	1303	1415	1609	0832 / 0833	
6	1104	1137	1303	1415	1609	0834	

pH Readings

flask#	T0	/vol.	T30m	/vol.	T1.5h	/vol.	T3h	/vol	T5h	/vol.	T20h	/vol	T24h	/vol
1	8.13 / 8.14	7.07 / 7.21	8.17 / 8.17	7.61 / 7.34	8.15	7.39	8.12	7.33	8.14	6.95	8.13	7.48	8.13	7.35
2	8.00	6.99	8.15	7.44	8.15 / 8.11	7.27 / 7.00	8.14	7.22	8.13	7.18	8.15	7.22	8.14	7.22
3	8.17	7.25	8.10	7.18	8.10	7.06	8.11 / 8.10	6.78 / 6.75	8.12	6.86	8.24	6.99	8.26 / 8.22	6.78 / 6.87
4	8.17	6.88	8.09	7.22	8.09	7.01	8.09	6.67	8.11 / 8.10	6.79 / 7.10	8.21	6.95	8.22	6.86
5	8.15	7.11	8.11	7.05	8.10	7.12	8.10	7.10	8.10	7.05	8.24 / 8.14	7.10 / 6.81	8.25	6.90
6	8.14	7.12	8.09	6.89	8.07	6.84	8.07	7.01	8.07	6.71	8.13	6.87	8.15	6.73

spec. Readings

flask#	T0	T30m	T1.5h	T3h	T5h	T20h	T24h
1	0.002 / 0.003	0.003 / 0.003	0.004	0.002	0.007	0.004	0.004
2	0.002	0.003	0.004	0.000	0.003	0.004	0.003
3	0.002	0.472	0.452 / 0.454	0.438 / 0.432	0.439	0.401	0.408 / 0.409
4	0.003	0.459	0.444	0.438	0.417 / 0.545	0.399	0.409
5	0.002	0.547	0.540	0.546	0.533	0.539 / 0.528	0.536
6	0.002	0.531	0.522	0.522	0.522	0.522	0.522

spec. check.

stds.	T0	T30m	T1.5h	T3h	T5h	T20h	T24h
Blk	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50ng/L	1.109	1.109	1.115	1.120	1.126	1.128	1.127
10ng/L	0.218	0.218	0.217	0.217	0.219	0.219	0.219

spec. Reading
KMnO4 Stock Soln. DF ~~250×5~~ KM ~~0.500~~ 1666
Abs. = ~~0.632~~ KM 0.511

Continued on page 80

K. McCracken — Recorded by — 6/16/98 Date

SRC — Read and understood by — 7/9/98 Date

Subject Exp. 4A-R5

Date 06 (Month) 17 (Day) 1998 (Year)

Sample Time	GC#	wt. empty	wt. + hex.	wt. + sample	sample ID	DF
6/17 1510	763	10.986	14.241	19.160	4A-R5-TBa	2
6/17 1510	764	10.975	14.260	19.562	4A-R5-TBb	
6/18 1100	765	11.013	12.977	16.144	4a-R5-T0-1a	
1101	766	10.892	12.869	15.990	4a-R5-T0-1b2	
1101	767	10.907	12.860	16.051	4a-R5- 3	
1102	768	11.000	12.952	16.036	4a-R5 4	
1103	769	10.850	12.795	15.902	4a-R5 5	
1103	770	10.972	12.923	15.898	4a-R5 6	
1104	771	10.883	12.812	16.261	4a-R5 1b	
1133	772	10.914	12.862	15.989	4a-R5-T30m-1	
1133	773	11.003	12.958	16.079	4a-R5 2a	
1134	774	10.889	12.838	15.729	4a-R5 2b	2
1135	775	10.984	12.932	16.064	4a-R5 3	1
1136	776	10.939	12.882	15.903	4a-R5 4	1
1136	777	10.782	12.708	15.764	4a-R5 5	1
1137	778	10.968	12.918	15.792	4a-R5 6	1
1300	779	10.911	12.844	15.916	4a-R5-T1.5h-1	2
1301	780	10.900	12.792	15.945	4a-R5 2	2
1301	781	10.925	12.893	15.922	4a-R5 3a	1
1302	782	10.801	12.745	15.675	4a-R5 3b	1
1302	783	10.958	12.890	15.909	4a-R5 4	1
1303	784	10.883	12.803	15.725	4a-R5 5	1
1303	785	10.996	12.919	15.983	4a-R5 6	1
1410	786	10.949	12.827	15.776	4a-R5-T3h-1	2
1410	787	10.990	12.906	15.773	4a- 2	2
1411	788	10.867	12.781	16.002	4a- 3	1
1412	789	10.917	12.835	15.878	4a- 4a	1
1413	790	10.937	12.845	15.976	4a- 4b	1
1415	791	10.977	12.883	15.791	4a- 5	1
1415	792	10.995	12.899	15.834	4a- 6	1
1605	793	10.923	12.820	15.949	4a-R5-T5h-1	2
1606	794	10.881	12.773	15.848	4a 2	2
1607	795	11.042	12.923	15.999	4a 3	1

Continued on page 81

K. McCracken — Recorded by — Date 06/17/98

SRC — Read and understood by — Date 7/9/98

Subject Exp. 4A-R5

Date 06 (Month) 17 (Day) 1998 (Year)

Sample Time	GC#	wt. empty	wt. + hex.	wt. + sample	Sample ID	DF
1608	796	11.020	12.900	16.071	4a-R5-T5h-4	1
1608	797	11.015	12.599	15.601	4a 5a	1
1609	798	10.981	12.878	15.860	4a 5b	1
1609	799	11.014	12.914	16.114	4a 6	1
6/19 0830	800	11.037	12.933	15.819	4a-R5-T2d-1	2
0830	801	10.888	12.780	15.894	2	2
0831	802	10.995	12.888	16.066	3	1
0831	803	10.925	12.808	15.916	4	1
0832	804	10.892	12.783	15.709	5	1
0833	805	11.000	12.880	15.993	6a	1
0834	806	10.972	12.868	16.055	6b	1
1100	807	11.014	12.906	15.909	4a-R5-T2h-1	2
1100	808	10.935	12.820	15.858	2	2
1101	809	10.956	12.842	15.960	3	1
1102	810	10.963	12.822	15.653	4a	
1102	811	10.927	12.800	16.015	4b	
1103	812	10.906	12.785	15.971	5	
1104	813	10.963	12.845	16.046	6	
1149	814	10.997	12.876	12.955	4a-R5-char 1a	
1149	815	10.924	12.805	12.843	4a-R5-char 1b	
1147	816	10.876	12.735	12.817	4a-R5-char 2a	
1147	817	10.875	12.768	12.796	4a-R5-char 2b	
1145	818	10.951	12.834	12.884	4a-R5-char 3a	
1145	819	10.913	12.806	12.836	4a-R5-char 3b	
1140	820	10.954	12.854	12.940	4a-R5-char 4a	
1140	821	10.978	12.864	12.891	4a-R5-char 4b	
no sample tube broke	~~822~~	~~10.913~~	~~12.826~~		~~4a-R5-char 5a~~	
	~~823~~	~~10.950~~	~~12.848~~		~~4a-R5-char 5b~~	
1136	824	10.911	12.797	12.891	4a-R5-char-6A	
1136	825	11.019	12.916	12.955	4a-R5-char 6b	
1130	826	~~10.995~~	~~12.620~~ 758.20	1171.19	4a-R5-f3	
1131	827	493.62	~~626.17~~ 758.14		4a-R5-f4	
1132	828	492.68	~~625.55~~ 757.53	1181.43	4a-R5-f5	
1133	829	492.28	~~626.53~~ 853	1115.12 *	4a-R5-f6	1
		492.45		1187.81		

* jar cracked, transfered to new jar.

Continued on page ___

K. McCracken 6/17/98
Recorded by Date

SRC 7/9/98
Read and understood by Date

Subject Exp. 3a - R2b

Date 06 (Month) 23 (Day) 19 98 (Year)

REPEATED EXP 3A-R2b
(No pH measurements will be made!)

1. All soil and groundwater to be used in these experiments (TAN-40 groundwater, TAN-37 crushed and aggregate basalt, and sediment) will be prepped by sparging and/or drying to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with a consistent and controlled initial TCE concentration (≈ 0 mg/L). Properly indicate usage of this media in the treatability study tracking logbook.

2. Follow the table below to obtain which test conditions to evaluate (soil type, oxidant concentration, etc.) Glass, wide-mouth erlenmeyer flasks will be used. It is currently planned to run four (4) test conditions in duplicate. Thus, 8 reactors will be used during this procedure.

	Media	Basalt Form	Liquid : Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial Permanganate Concentration (%)
	DI Water	NA	NA	0	0	0.10
EXP 3a	Groundwater	NA	NA	0	0	0.10
RUN 2b	Groundwater/basalt	Crushed	4:1	0	0	0.10
	Groundwater/basalt	Crushed	4:1	0	0	0.10
	Groundwater/basalt	Aggregate	4:1	0	0	0.10
	Groundwater/basalt	Aggregate	4:1	0	0	0.10
	Groundwater/sediment	NA	4:1	0	0	0.10
	Groundwater/sediment	NA	4:1	0	0	0.10

KM McCracken 6/23/98

3. Obtain the tare weight of each reactor. Add approximately 50g of solid media to the reactors in the Table that require it. Re-weigh the reactors to determine the actual mass added. Except for reactor #1 (200 mL DI water), add 200 mL of TAN-40). Re-weigh each reactor to determine the actual weight of solution added. (A calibration check of the gravimetric scale will be performed each day of use.)

4. Place the reactors in the incubator at 12°C and allow to equilibrate to that temperature. Slowly agitate the reactors during this step.

5. **For Loadings of 0.1wt% MnO_4^-**
Preparation of Stock MnO_4^- Solution (4 wt%)
Add **2.66 g** of crystalline KMnO4 (Carus™ Free Flowing Technical Grade) into a **50 mL** Flask and fill to volume with DI water, and allow to mix for at least 2 hours. ← Run on Spec.
[.3mL → 500mL Flask DI H_2

S.R. Cline Last Updated: 06/17/98 Filename: procedure for Repeat 3a-R2 Experiment.doc

Continued on page 83

K. McCracken 6/23/98
Recorded by Date

SRC 6/29/98
Read and understood by Date

Subject Exp.3a - R2b

Date 06 (Month) 23 (Day) 1998 (Year)

6. **For Loadings of 0.1 wt% MnO_4^-**
Addition of Stock MnO4- Solution into Reactors
Quickly remove the ground glass stopper and add the needed volume of stock $KMnO_4$ to obtain the MnO_4^- concentrations in the above **Table** (approximately **5 mL** for the **0.1 % MnO_4^-** concentration).

7. Replace the glass ground stoppers and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. The time that the stock $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12°C incubator and 200 rpm.

8. For each reactor, remove the reactor from the incubator at approximately 30 minutes from the reaction start time, and collect a 5 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator.

9. Add a 0.45 µm syringe filter onto the end of the syringe and filter the sample.

10. Perform a MnO_4^- analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

K M Cracke 6/23/98

11. Collect and analyze aliquots from each reactor for $KMnO_4$ (Steps 8-10) at the following ADDITIONAL time periods: 1hr, 2 hrs, 4 hrs, 18 hrs, and 24 hrs.

12. Interpret results and clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

#	wt.empty	wt+medium	wt.+H_2O	$KMnO_4$ added	~~To~~ DF	~~DF~~ To	Sample ID
1	243.61	—	443.61	5.0 ml	50	6/23 1121	d.i.H_2O
2	256.79	—	456.79	5.0 ml	50	1121	groundwater
3	236.37	386.87	~~436.37~~ 536.87	5.0 ml	50	1122	gw+crushed basalt
4	251.61	301.92	~~451.61~~ 501.92	5.0 ml	50	1123	gw+crushed basalt
5	239.79	289.77	~~439.79~~ 489.77	5.0 ml	50	1123	gw+agg. basalt
6	251.64	301.60	~~451.64~~ 501.60	5.0 ml	50	1124	gw+agg. basalt
7	243.68	293.75	493.75	5.0 ml	50	1124	gw+sediment
8	189.69	234.69	434.69	5.0 ml	50	1125	gw+sediment

Spec. readings: To

BLK	1	4	7
50mg	2	5	8
10mg	3	6	

Continued on page 84

K. McCracke — Recorded by — 6/23/98 Date

SFC — Read and understood by — 6/23/98 Date

Subject Exp. 3A&2b

Date 06 (Month) 23 (Day) 19 98 (Year)

Flask #	T30min	T1hr	T2hr	T4hr	T10h	T24h	
1	6/23 1155	1227	1340	1535	6/24 0830	1127	
2	1155	1227	1341	1535	0830	1127	
3	1156	1228	1341	1536	0830	1127	
4	1156	1228	1342	1536	0831	1128	
5	1157	1229	1342	1536	0831	1128	
6	1158	1230	1343	1537	0832	1129	
7	1158	1230	1343	1537	0832	1129	
8	1158	1231	1344	1538	0833	1130	

spec. readings

Flask #	T30min	T1h	T2h	T4h	T10h	T24h	
1	0.435	0.419	0.428	0.421	0.424	0.391	
2	0.435	0.419	0.407	0.414	0.422	0.402	
3	0.435	0.434	0.415	0.432	0.413	0.401	
4	0.432	0.406	0.413	0.417	0.406	0.398	
5	0.435	0.427	0.406	0.413	0.416	0.400	
6	0.430	0.411	0.406	0.426	0.415	0.410	
7	0.420	0.412	0.422	0.410	0.398	0.395	
8	0.428	0.419	0.424	0.414	0.412	0.388	

spec. check	T30min	T1h	T2h	T4h	T10h	T24h	
Blank	0.000	0.000	0.000	0.000	0.000	0.000	
50mg/L	1.124	1.120	1.124	1.120	1.125	1.122	
10mg/L	0.236	0.233	0.227	0.223	0.220	0.215	

Stock $KMnO_4$ ~~DF 250~~ SKC 6/29/98

spec. reading 0.263 → 3mL → 1000mL Total Volume

ALL DF 50

Continued on page —

K. McCracken — Recorded by — 6/23/98 Date

SKC — Read and understood by — 6/29/98 Date

Subject Exp. 3AR3b

Date 06 (Month) 25 (Day) 19 98 (Year)

flasks for this experiment will contain the following

flask#	media	liquid:solid ratio	TCE	initial co-contam conc	initial KMnO4 conc
1	d.i. water -200ml	NA	0	0	1.0%
2	TAN40 groundwater 200ml	NA			
3	gw + crushed basalt	4:1			
4	gw + crushed basalt	4:1			
5	gw + aggregate basalt	4:1			
6	gw + aggregate basalt	4:1			
7	gw + sediment	4:1			
8	gw + sediment	4:1			

□□ÑCarefully weigh out KMnO4© for each flask into weigh boats. To get 1.0% MnO4©, we will need 2.655 grams of KMnO4 weighed out. Be as careful as you can.

added. I would instead re©weigh the weigh boat (with analytical balance) to see how much DIDNÀ□□ÀT get into the flask from the value obtained in the step above.

7. Replace the glass ground stoppers and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. The time that the stock KMnOÃ□Ã4Ã□Ã is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12À$□ÀC incubator and 200 rpm.
þ□□Ú□□Ù□□□□□-□□□□□□□□ÙÑ□
□□□ÿ□□□
□□Ñ8. For each reactor, remove the reactor from the incubator at approximately Ñ□#□□î□d□ô□\□□ □□□□ P□□□6Q□□□□□□□î□P#□□ÑÃ□Ã45 minutesÃ□AÑ□#□□X□P□ô□\□□ □□□□ P□□□6Q□□□□□□□X□P#□□Ñ from the reaction start time, and collect a 5 mL aliquot (using a disposable plastic syringe with a Luer Tip). Record the time the sample was collected and replace the reactor back into the incubator.Ã□□□Ã

9. Add a 0.45 À□□Àm syringe filter onto the end of the syringe and filter the sample.

10. Perform a MnOÃ□Ã4Ã□ÃÃ□Ã©Ã□Ã analysis on the filtered sample from the previous step. (The sample is filtered to remove particulate matter that could interfere with the analysis.) The filtered sample will likely have to be diluted with DI water so that its spectrophotometric absorbance (at À□□À =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4©). Check standards (10 and 50 mg/L MnO4©) will be prepared with reagent grade KMnOÃ□Ã4Ã□Ã and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

Continued on page 86

K McCracken — Recorded by — 6/25/98 Date

SRC — Read and understood by — 6/30/98 Date

Subject Exp 3A-R36

Date 06 (Month) 25 (Day) 19 98 (Year)

11. Collect and analyze aliquots from each reactor for KMnOÃ□Ã4Ã□Ã (Steps 8©10) at the following ADDITIONAL time periods: 2 hrs, 3hrs, 4 hrs, 18 hrs, and 24 hrs.
Ù□□□□□□□□□È□□□□Ù
Ã□□□Ã12. Interpret results and clean up the reactors for re©use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.Ã□□□Ã

flask #	wt. empty	wt + medium	wt. + H_2O	$KMnO_4$ added	T0	DF	Sample ID
1	243.371	—	443.377	2.6609-.0004	1205	500	DI H_2O
2	248.323	—	447.993	2.6595-.0003	1208		gw
3	240.236	290.291	490.351	2.6601-.0000	1212		gw + cr. bas.
4	256.056	306.056	506.028	2.6605-.0000	1215		gw + cr. bas.
5	242.385	292.566	492.295	2.6608-.0000	1218		gw + agg. bas.
6	248.858	298.908	498.597	2.6601-.0001	1220		gw + agg. bas.
7	243.745	293.747	493.489	2.6603-.0007	1222		gw + sed.
8	189.757	239.777	439.419	2.6614-.0001	1225		gw + sed.

Time flask	T45min*	T2hr*	T3hr.	T4hr	T18hr	T24hr	Spec. check.
1	1300	1415	1525	1615	0745	1200	T45 BLK 0.000
2	1301	1416	1526	1616	0745	1200	50mg/L 1.119
3	1301	1417	1528	1617	0746	1201	10mg/L 0.225
4	1301	1418	1528	1619	0746	1201	T2hr BLK 0.000
5	1302	1419	1529	1620	0747	1202	50mg/L 1.119
6	1302	1420	1529	1621	0747	1204	10mg/L 0.225
7	1303	1421	1530	1622	0748	1204	T3hr BLK 0.000
8	1303	1421	1530	1624	0748	1205	50mg/L 1.123
spec.	~~0.42945~~						10mg/L 0.221
1	0.429	.450	0.454	0.452	0.391	0.408	T4hr. BLK 0.000
2	0.438	.272	0.439	0.446	0.441	0.440	50mg/L 1.121
3	0.443	.453	0.460	0.447	0.442	0.449	10mg/L 0.219
4	0.440	.451	0.446	0.452	0.443	0.438	T18hr. BLK 0.000
5	0.315	.449	0.458	0.455	0.449	0.450	50mg/L 1.124
6	0.315	.450	0.458	0.452	0.450	0.445	10mg/L 0.219
7	0.454	.467	0.455	0.457	0.442	0.456	T24hr BLK 0.000
8	0.468	.468	0.461	0.461	0.445	0.453	50mg/L 1.125
							10mg/L 0.219

*please note spec. readings to T45 & T2hr. dilutions ~~are~~ need adjustable pipet. All others done using 10µl Hamilton syringe.

Continued on page

K. McCracken — Recorded by — Date 6/25/98

SPC — Read and understood by — Date 6/30/98

Subject Exp. 4a-R7

Date 07 07 1998
Month Day Year

	media	basalt	liquid/solid ratio	Initial TCE conc.	initial chromate conc.	initial permanganate conc.
	CONTROL Groundwater	NA	NA	0.1 mg/L	0 mg/L	0 (%)
	CONTROL Groundwater	NA	NA	0.1	0	0
EXP 4a	Groundwater/sediment	NA	4:1	0.1	0	0.01
RUN 7	Groundwater/sediment	NA	4:1	0.1	0	0.01
	Groundwater/sediment	NA	4:1	0.1	0	0.10
	Groundwater/sediment	NA	4:1	0.1	0	0.10

1.25 ml

12.5 ml

Tedlar bag 47.181 g
Tedlar bag + GW 2669.19
gw 2622.009
sat. soln. TCE added 0.3 ml

flask	wt. empty	wt media	wt. + g.w	g.w. Vol.
1	243.195	243.195	732.49	
2	248.252	248.252	726.42	
3	244.571	341.678	756.91	
4	251.356	348.319	738.04	
5	242.133	339.156	744.73	
6	248.714	340.304	708.25	

Calib
7/8 X177440
50g 49.96g ✓

Calib
7/10 X177440
50g 49.95g ✓

GC#	wt. empty	wt. + hexane	wt. + sample	sample ID
1096	11.166	13.102	13.150	char-b
1097	11.106	13.039	13.135	char-a

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K. McCracken 7/7/98
Recorded by Date

SRC 7/27/98
Read and understood by Date

Subject Exp. 4a. R7

Date 07 (Month) 07 (Day) 19 98 (Year)

+ $KMnO_4$ at 1200

Flask #	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	11.37	12:30	13:32	1459	1655	7/10 0835	1206
2	11.38	12:31	13:33	1500	1656	0836	1207
3	11.39	12:32	13:34 / 13:35	1501	1657	0837	1208
4	11.40	12:33	13:36	1502 / 1503	1657	0838	1209
5	11.41	12:34	1337	1504	1658 / 1659	0839	1210 / 1211
6	11.42	12:35	1338	1505	1700	0839 / 0840	1212

pH Readings

Flask	T0 / vol.	T30 / vol	T1.5 / vol	T3hr / vol.	T5hr / vol	T20hr / vol	T24hr / vol.
1	8.07 / 6.97 / 8.06 / 6.79	8.10 / 6.98	8.10 / 6.91	8.10 / 6.93	8.10 / 6.82	8.13 / 7.31	8.12 / 7.32
2	8.10 / 7.078	8.13 / 7.24 / 8.13 / 7.50	8.12 / 7.37	8.13 / 7.01	8.12 / 7.11	8.12 / 7.34	8.19 / 7.50
3	7.89 / 6.414	7.91 / 6.80	7.94 / 7.14 / 7.86 / 6.71	7.88 / 6.71	7.95 / 7.27	7.98 / 6.76	7.95 / 5.33
4	7.90 / 6.56	7.89 / 6.76	7.86 / 6.52	7.88 / 6.45 / 7.87 / 6.50	7.92 / 6.74	7.99 / 6.85	7.97 / 6.41
5	7.91 / 6.49	7.79 / 6.78	7.72 / 6.78	7.72 / 6.75	7.74 / 6.86 / 7.74 / 6.66	7.78 / 6.61	7.81 / 6.90 / 7.81 / 6.85
6	7.91 / 6.496	7.78 / 6.65	7.72 / 6.95	7.72 / 6.74	7.74 / 6.91	7.78 / 6.98 / 7.79 / 6.94	7.81 / 6.77

Spec. readings

Flask	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	0.002 / 0.003	0.002 / 0.003	0.004	0.005	0.004	0.004	0.004
2	0.001	0.003	0.001 / 0.451	0.002	0.002	0.004	0.003
3	0.003	0.480	0.490	0.468 / 0.510	0.429	0.433	0.440
4	0.004	0.529	0.530	0.516	0.503 / 0.497	0.471	0.524
5	0.002	0.529	0.512	0.517	0.519	0.494 / 0.553	0.516 / 0.519
6	0.003	0.563	0.566	0.572	0.555	0.553	0.557

Spec. check

Stds	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
BLK	0.000	0.000	0.0000	0.0000	0.000	0.000	0.000
50mg/L	1.118	1.118	1.119	1.119	1.104	1.108	1.120
10mg/L	0.225	0.225	0.223	0.224	0.220	0.219	0.221

Spec. reading $KMnO_4$ Stock. DF $KMnO_4$ 150 µl → $DI.H_2O$ 250 ml Abs. 0.540

DF 1666

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K. McCracken — recorded by — 07/07/98 — Date

SRC — Read and understood by — 7/27/98 — Date

Subject Exp. 4a - R7

Date 07 (Month) 07 (Day) 19 98 (Year)

Sample Time	GC#	wt empty	wt + Hexane	wt + sample	Sample ID	DF
7/8 1550	1029	11.183	14.454	19.682	4a-R7-TBa	1
1550	1030	11.125	14.440	19.746	4a-R7-TBb	
7/9 1137	1031	11.081	13.001	16.110	4a-R7-Tϕ-1a	
1138	1032	11.030	12.965	16.289	1b	
1139	1033	11.004	12.979	16.199	2	
1140	1034	11.061	13.030	16.121	3	
1141	1035	11.036	12.982	16.191	4	
1142	1036	11.147	13.115	16.470	5	
1142	1037	11.049	13.015	16.477	6	
1230	1038	11.162	13.111	16.653	4a-R7-T30-1	
1231	1039	11.003	12.952	16.287	2a	
1231	1040	11.061	13.011	16.401	2b	
1232	1041	11.162	13.116	16.438	3	
1233	1042	11.210	13.173	16.394	4	
1234	1043	11.148	13.096	16.443	5	
1235	1044	11.086	13.053	16.660	6	
1332	1045	11.083	13.044	16.332	4a-R7-T15hr-1	
1333	1046	11.114	13.071	16.302	2	
1334	1047	11.121	13.080	16.506	3a	
1335	1048	10.980	12.926	16.257	3b	
1336	1049	11.064	12.958	16.325	4	
1337	1050	11.183	13.129	16.515	5	
1338	1051	11.046	13.001	16.154	6	
1459	1052	11.122	13.068	16.313	4a-R7-T3hr-1	
1500	1053	11.070	13.010	16.352	2	
1501	1054	11.137	13.075	16.286	3	
1502	1055	11.251	13.200	16.671	4a	
1503	1056	11.101	13.031	16.605	4b	
1504	1057	11.244	13.178	16.615	5	
1505	1058	11.080	13.009	16.518	6	
1655	1059	11.192	13.120	16.434	4a-R7-T5hr-1	
1656	1060	11.027	12.964	16.278	2	
1657	1061	11.116	13.050	16.426	3	

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K. McCracken — Recorded by — 07/07/98 Date

SRC — Read and understood by — 7/27/98 Date

Subject Exp. 4a-R7

Date 07 (Month) 07 (Day) 19 98 (Year)

sampletime	GC#	wt. empty	wt. + Hexane	wt. + sample	sample ID	DF
1657	1062	11.124	13.064	16.290	4a-R7-T5hr-4	1
1658	1063	11.265	13.208	16.391	5a	
1659	1064	11.163	13.107	16.451	5b	
1700	1065	11.091	13.011	16.382	6	
7/10 0835	1066	11.026	12.959	16.172	4a-R7-T20hr-1	
0836	1067	10.981	12.916	16.200	2	
0837	1068	11.180	13.128	16.104	3	
0838	1069	11.141	13.084	16.165	4	
0839	1070	11.050	13.060	16.293	5	
0839	1071	11.157	13.163	16.303	6a	
0840	1072	11.037	12.992	16.223	6b	
1206	1073	10.977	12.923	~~12.4~~ 16.909	4a-R7-T24hr-1	
1207	1074	11.036	12.973	16.068	2	
1208	1075	11.164	13.100	15.974	3	
1209	1076	11.207	13.131	16.095	4	
1210	1077	11.033	12.959	16.118	5a	
1211	1078	11.127	13.052	15.889	5b	
1212	1079	11.021	12.963	16.248	6	
1225	1080	11.256	13.192	13.291	4a-R7-char-1a	
1225	1081	11.098	13.025	13.068	1b	
1226	1082	11.202	13.126	13.227	2a	
1226	1083	11.136	13.054	13.100	2b	
1227	1084	11.121	13.048	13.147	3a	
1227	1085	11.177	13.118	13.166	3b	
1228	1086	11.076	13.025	13.126	4a	
1228	1087	11.076	13.006	13.055	4b	
1229	1088	11.174	13.099	13.196	5a	
1229	1089	11.220	13.157	13.203	5b	
1230	1090	11.085	13.022	13.106	6a	
1230	1091	11.063	13.000	13.043	6b	
1240	1092	~~11.118~~ 492.33	~~12.533~~ 624.44	1173.09	4a-R7-f3	
1243	1093	~~11.039~~ 493.23	~~12.533~~ 625.10	1150.96	4a-R7-f4	
1247	1094	~~11.062~~ 493.12	~~12.533~~ 624.86	1167.04	4a-R7-f5	
1250	1095	~~11.069~~ 492.93	~~12.533~~ 624.60	1137.02	4a-R7-f6	

Continued on page

K. McCracken — Recorded by — 07/07/98 — Date

SR — Read and understood by — 7/27/98 — Date

Subject Exp. 4a - R4

Date 07 Month 14 Day 19 98 Year

	media	basalt form	liquid/ solid ratio	Initial TCE conc.	Initial co-contam conc.	initial permanganate %
	CONTROL Groundwater	NA	NA	0.1	0	0
	CONTROL Groundwater	NA	NA	0.1	0	0
EXP 4a	Groundwater/ basalt	Aggregate	4:1	0.1	0	0.01
RUN 4	Groundwater/ basalt	Aggregate	4:1	0.1	0	0.01
	Groundwater/basalt	Aggregate	4:1	0.1	0	0.10
	Groundwater/basalt	Aggregate	4:1	0.1	0	0.10

KM 1.11/98

Tedlar Bag 52.004 g
Tedlar Bag + GW 3022.0 g
g.w. ~~2970.00~~ g 2970.00 g
get soln. TCE added 0.3 mL
stock

Calib. 7/14 wt. Scale
X 177440 50g 49.96g

flask #	wt. empty	wt+media	wt+gw	gw. vol.
1	243.216	243.216	744.18	500.96
2	252.600	252.600	748.84	496.24
3	244.596	344.650	750.46	405.81
4	249.124	349.447	758.29	409.14
5	241.710	341.707	743.17	401.46
6	246.832	346.890	748.47	401.58

7/16 Calib. X177440
wt. Scale
50g 49.97g

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K. McCracken 7/14/98
Recorded by Date

SRC 7/27/98
Read and understood by Date

Subject Exp. 4a R4

Date 07 Month 14 Day 19 98 Year

Flask#	T0	T30min.	T1.5hr.	T3hr	T5hr.	T20hr	T24hr.
1	1048	1120	1220	1356	1550	0801	1042
2	1049	1121	1221	1357	1551	0802	1043
3	1050	1123	1222	1358	1552	0803	1044
4	1050	1124	1224	1358	1553	0803	1044
5	1051	1125	1225	1400	1554	0804	1045
6	1052	1126	1226	1400	1556	0805	1047

pH reading/volume.

Flask#	T0 pH/vol	T30 /vol	T1.5h/vol	T3h /vol	T5h/vol	T20h/vol	T24h/vol
1	8.08/7.27	8.08/7.42	8.10/7.37	8.09/7.35	8.07/7.44	8.07/7.35	8.08/7.35
2	8.02/7.49	8.05/7.30	8.06/7.45	8.06/7.44	8.06/7.38	8.05/7.38	8.04/7.27
3	8.01/7.05	8.03/6.83	8.02/6.89	8.02/6.96	8.03/6.90	8.30/6.89	8.31/6.65
4	8.02/7.14	8.03/7.32	8.02/7.11	8.03/7.29	8.03/7.23	8.23/7.27	8.25/7.32
5	8.02/7.18	8.14/7.45	8.12/7.22	8.12/7.46	8.13/7.42	8.28/7.30	8.28/7.25
6	8.01/7.21	8.15/7.33	8.13/7.19	8.13/7.32	8.14/7.19	8.31/7.24	8.31/7.27

spec. reading

Flask#	T0	T30min	T1.5h	T3h	T5h	T20h	T24h
1	0.003	0.005	0.004	0.008	0.008	0.005	0.008
2	0.003	0.003	0.003	0.004	0.005	0.003	0.002
3	0.004	0.506	0.498	0.506	0.498	0.447	0.481
4	0.003	0.513	0.518	0.512	0.507	0.503	0.492
5	0.003	0.493	0.488	0.493	0.497	0.500	0.497
6	0.001	0.533	0.535	0.529	0.532	0.533	0.522

spec. Check.

Stds.	T0	T30min	T1.5h	T3h	T5h	T20h	T24h.
Blank	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.123	1.124	1.119	1.120	1.123	1.124	1.123
10mg/L	0.229	0.229	0.222	0.219	0.216	0.222	0.221

spec. reading KMnO4 stock: DF 1666 Abs. 0.517 SFC 7/28/98

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K. McCracken 7/14/98
Recorded by Date

SFC 7/28/98
Read and understood by Date

Subject Exp. 4a - R4

Date 07 (Month) 14 (Day) 19 98 (Year)

Sample time	GC#	wt. empty	Wt. + hexane	wt. + sample	Sample ID	DF
7/14 14:46	1246	11.061	14.325	19.437	4a-R4-TBa	1
14:46	1247	11.038	14.219	19.486	4a-R4-TBb	
7/15 10:48	1248	11.067	13.049	16.471	4a-R4-T0-1a	
10:48	1249	11.115	13.080	16.226	1b	
10:49	1250	11.214	13.158	16.080	2	
10:50	1251	11.128	13.050	16.110	3	
10:50	1252	11.107	13.018	16.299	4	
10:51	1253	11.115	13.018	16.041	5	
10:52	1254	11.174	13.083	16.280	6	
11:20	1255	11.146	13.056	16.241	4a-R4-T30min-	
11:21	1256	11.000	12.891	16.168	2a	
11:22	1257	11.147	13.035	16.100	2b	
11:23	1258	11.132	13.020	16.153	3	
11:24	1259	11.129	13.025	16.145	4	
11:25	1260	11.183	13.074	16.252	5	
11:26	1261	11.131	13.037	16.287	6	
12:20	1262	11.093	12.997	16.031	4a-R4-T1.5h-	
12:21	1263	10.990	12.897	15.867	2	
12:22	1264	11.122	13.034	16.228	3a	
12:23	1265	11.168	13.057	16.255	3b	
12:24	1266	11.131	13.019	16.254	4a	
12:25	1267	11.178	13.050	16.302	5	
12:26	1268	11.128	13.021	16.136	6	
13:56	1269	11.107	13.000	16.050	4a-R4-T3h- 1	
13:57	1270	11.096	12.993	16.079	2	
13:58	1271	11.063	12.968	16.247	3	
13:58	1272	11.111	13.010	16.200	4a	
13:59	1273	11.043	12.941	16.002	4b	
14:00	1274	11.000	12.894	16.117	5	
14:00	1275	11.153	13.048	16.195	~~4a-R4-T5h-~~ 6	
15:50	1276	11.167	13.060	16.113	4a-R4-T5h- 1	
15:51	1277	11.043	12.948	16.117	2	
15:52	1278	11.079	12.973	16.034	3	

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K. McCracken — Recorded by

07/14/98 — Date

7/29/98 — Read and understood by — Date

Subject Exp. 4a R4

Date 07 (Month) 14 (Day) 19 98 (Year)

Sample time	GC #	wt. empty	wt. thexane	wt. sample	Sample ID	DF
15:53	1279	11.081	12.976	16.124	4a-R4-T5h-4	1
15:54	1280	11.136	13.348	16.057	5a	
15:55	1281	11.069	12.960	16.042	5b	
15:56	1282	11.065	12.937	16.062	6	
7/16 0801	1283	11.192	13.081	16.121	4a-R4-T20h-1	
08:02	1284	11.070	12.947	16.175	2	
08:03	1285	11.051	12.948	16.041	3	
08:03	1286	11.108	13.002	15.979	4	
08:04	1287	11.104	13.000	16.325	5	
08:05	1288	11.096	13.000	16.019	6a	
08:05	1289	11.088	13.013	16.490	6b	
10:42	1290	11.131	13.025	16.231	4a-R4-T24h-1	
10:43	1291	11.106	13.018	16.471	2	
10:44	1292	11.252	13.167	16.034	3	
10:44	1293	11.083	12.985	16.021	4	
10:45	1294	11.066	12.957	16.124	5	
10:46	1295	11.100	12.976	16.193	6a	
10:47	1296	11.049	12.940	16.115	6b	
10:50	1297	11.040	12.904	12.988	4a-R4-char 1a	
10:50	1298	11.208	13.069	13.106	1b	
10:51	1299	11.150	13.025	13.118	2a	
10:51	1300	11.090	12.971	13.015	2b	
10:52	1301	11.272	13.152	13.240	3a	
10:52	1302	11.110	12.990	13.040	3b	
10:53	1303	11.138	13.020	13.041	4a	
10:53	1304	11.312	13.185	13.230	4b	
10:54	1305	11.080	12.959	13.052	5a	
10:54	1306	11.241	13.110	13.135	5b	
10:55	1307	11.056	12.958	13.061	6a	
10:55	1308	11.051	12.923	12.960	6b	
10:58	1309	493.92	758.08	1177.55	4a-R4-f3	
10:59	1310	491.96	756.50	1180.28	4a-R4-f4	
11:02	1311	493.37	757.57	1184.09	4a-R4-f5	
11:04	1312	492.44	756.44	1173.92	4a-R4-f6	

Continued on page

K. McCracken 7/14/98 — Recorded by, Date

SRC 7/27/98 — Read and understood by, Date

Subject Exp. 4b - R1 & R2

Date 08 (Month) 10 (Day) 19 98 (Year)

4.2 Measure the Oxidation Rate of Organic Contaminants in Hot Spot Media (Task 4b)

General Procedure for Task 4b Experiments - Measure the oxidation rate of organic contaminants in the hot spot groundwater

(To be conducted by both ORNL/ESD and RMAL)

NOTE: This experiment will be used to conduct the various test conditions presented in **Table 2-6** in Chapter 2 of the Implementation Plan.

Control samples (no $KMnO_4$) will be prepared and sampled throughout the reaction period to quantify changes in concentration due to mechanisms other than oxidation. In addition to the duplicate control samples, it is currently planned to run two (2) test conditions (in duplicate) from **Table 2-6** each time this procedure is used. Thus, 6 reactors will be used during each run of this procedure, and this procedure will be conducted a total of 8 times (4 times at each facility). The controls and the treatment reactors will all contain the same initial contaminant concentrations (i.e., all conditions will be the same for each run of this procedure with the exception of the $KMnO_4$ concentration used).

Note: The 2,000 mg/L TCE spike solution in Table 2-6 experiments cannot be prepared in the same manner as the spike solution for the 10 and 100 mg/L spike levels. Both spiking methods are included in this procedure.

1. Soil Prepping

All groundwater to be used in these experiments (**TSF-05 groundwater**) will be prepped by sparging to remove any residual TCE present on the media. Doing so will allow this experiment to be conducted with consistent and controlled initial TCE concentrations via contaminant spiking. (Properly indicate usage of this media in the treatability study tracking logbook.)

For the 10 and 100 mg/L Spike concentrations ONLY.

–Proceed to step 9 if spiking at DNAPL levels (2,000 mg/L TCE) in Table 2-6

2. TCE Stock Solution Preparation

Prepare a STOCK (Saturated) TCE solution (≈1100 mg/L). (Use of a STOCK solution rather than pure phase TCE to prepare the spiking solution affords better accuracy in obtaining the target concentration.) This can easily be stored and used again in subsequent experiments

* For 10 ppm TCE spiking (Runs 1 & 2): Add > 0.1 mL of Pure TCE into 65-70 mL of **TSF-05 groundwater** and keep refrigerated.

For 100 ppm TCE spiking (Runs 5&6): Add > 0.6 mL of Pure TCE into 625 mL of TSF-05 GW & keep refrigerated.

3. TCE Spike Solution Preparation, PART I

Obtain the tare weight of the container to be used to prepare the spike solution (Tedlar bag, flask, or equivalent). Fill with unspiked 12°C **TSF-05 groundwater (≈3070 mL or ≈2800 mL for the 10 and 100 mg/L spike condition, respectively).** By gravimetric methods, determine the amount of unspiked groundwater added to the container. (A calibration check of the gravimetric scale will be performed each day of use.)

4. TCE Spike Solution Preparation, PART II

Add the appropriate quantity of the Saturated STOCK TCE solution to produce a TCE spike solution with the concentration prescribed in **Table 2-6** for the particular test series being ran. The **hot spot groundwater** will only be spiked at **10 and 100 mg/L TCE via this method**, thus the volume of saturated STOCK TCE needed should be on the order of **30 mL and 300 mL**, respectively. Please record the volume of STOCK TCE added (**NOTE: will also added any contaminants required during this step.**) Place the spike solution in the incubator overnight at 12C for temperature equilibration. Ensure that all information regarding the preparation of the spiking solution will be recorded in the laboratory research notebook.

5. Obtain Tare Weights of Reactors

Prepare the test reactors to be used. (The gas-tight reaction vessels acquired and tested in Task 2a will be used as the reactors in this experiment.) Obtain the tare weight of each, including the weight of a ground glass standard taper stopper fitted with a Teflon sleeve.

Note These → are 10ppm not .1 or 1 ppm! Therefore dilutions will be different but I should be here to help determine what they should be!

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vial wt. empty 100.540g
vial + H_2O 170.556g
H_2O 70.016g

syringe wt. + TCE 18.806
syringe wt. 18.572
TCE 0.234g

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6. Collection of Initial Spike Solution Samples
Remove the spike solution from the incubator, and obtain duplicate aqueous samples (≈ 5 mL) of the spike solution to obtain the initial TCE concentration of the spiked groundwater. Record the time the sample is collected. The duplicate aliquot samples will be added to pre-weighed sample containers holding a known volume of hexane (≈ 5 mL). Each sample container will be weighed again after sample addition to determine the actual weight of sample added. These extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

7. Distribution of Spike Solution to Reactors
Quickly dispense the spike solution into each of the reactors to be used. Add ≈500 mL to each reactor. Immediately replace the ground glass stopper and re-weigh each to determine the actual weight of spiked groundwater added.

8. Keep Cool until Experiment is initiated.
Place the reactors in the incubator at 12°C until the experiment is initiated. Agitate the reactors at ≈200 rpm during this step.

For the DNAPL Spiking Level (2,000 mg/L TCE) in Table 2-6 ONLY
–Proceed to Step 13 if spiking at 10 or 100 mg/L

9. >2,000 mg/L Spiking: Obtain Tare Weights of Reactors
Prepare the test reactors to be used. (The gas-tight reaction vessels acquired and tested in Task 2a will be used as the reactors in this experiment.) Obtain the tare weight of each, including the weight of a ground glass standard taper stopper fitted with a Teflon sleeve.

10. >2,000 mg/L Spiking: Reactor Filling
Dispense **≈500 mL un-spiked TSF-05 groundwater** into each of the reactors to be used. Immediately replace the ground glass stopper/sleeve and re-weigh each to determine the actual weight of groundwater added.

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11. Reactor Spiking (>2,000 mg/L TCE)
To each of the six reactors, add enough pure phase TCE to achieve a TCE concentration of 2,000 mg/L (**should be around 700 µL pure TCE for 500 mL of groundwater**). Measure the mass of the spiking syringe when filled and then after spiking to determine the actual contaminant mass added (a QC check on the initial TCE analysis). Information regarding the preparation of each spiking solution will be recorded in the laboratory research notebook.

12. Spike Solution Equilibration (>2,000 mg/L Spike Condition)
Place the reactors into the incubator at 200 rpm or greater and allow the spike solutions to equilibrate at 12°C (likely overnight)

13. **For Loadings of .01 and 0.1 wt% MnO_4^- in Table 2-6 ONLY.**
Preparation of Stock MnO_4^- Solution (4 wt%)
Add 2.66 g of crystalline KMnO4 (Carus™ Free Flowing Technical Grade) into a 50 mL Flask and fill to volume with DI water, and allow to mix for at least 2 hours.

14. Prepare Reactor Closure/Carbon Tube
During equilibration period, prepare the standard taper adapters/stopcock valve. (**Two ORBO tubes will be used.**) The vertical sampling port will be fitted with a small septum) STORE UN-SEALED ORBO TUBES OUTSIDE OF THE LABORATORY FUME HOODS. THE HOODS LIKELY HAVE THE GREATEST POTENTIAL FOR "CONTAMINATING" THE TUBES WITH TCE AND OTHER VOCs.

15. Collection of Pre-Reaction Sample from Each Reactor
After equilibration, collect a ≈10 mL aqueous sample using a long need and disposable syringe from each of the reactors and quickly reseal. Record the time the sample is collected and note whether solids were present in the aqueous sample. If significant solids are present, a portion of this aliquot volume may be used for a total solids analysis.

16. Preparation of Pre Reaction TCE Sample
Dispense (unfiltered) ≈3 mL of this aliquot sample into to a pre-weighed vial holding a known volume of hexane (≈3 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis. Record the time this sample is taken.

17. Measurement of Pre Reaction pH Value
Tare a small beaker for pH analysis. Add a 0.45 micron filter to the sample syringe and dispense (filtered) the remaining portion of the sample in the syringe (≈7 mL) into the tared beaker. Reweigh the beaker to determine the volume of the sample (assuming that the solids fraction is negligible). The pH analyses will be conducted using conventional pH meters/electrodes, calibrated each day of use via a minimum of two standard buffer solutions. Values obtained for each sample will be recorded. A calibration check will also be conducted following the last sample to be measured each day. Values obtained for the samples will be corrected if a significant drift (i.e., > 15%) occurred.

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18. **For Loadings of .01 and 0.1 wt% MnO_4^- in Table 2-6 ONLY.**
Addition of Stock MnO4- Solution into Reactors
Quickly remove the ground glass stopper and add the needed volume of stock $KMnO_4$ to obtain the MnO_4^- concentrations in Table 2-6 (approximately 1.25 mL and 12.5 mL for the **0.01 and 0.1% MnO_4^-** concentrations respectively). Replace the glass ground stoppers with the adapters fitted with the stopcock and charcoal desorption tubes and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. (No oxidant will be added to the control reactors). The time that the stock $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12°C incubator and 200 rpm or greater.

19. **For the Oxidant Loadings of 1 and 3wt% MnO_4^- in Table 2-6 ONLY**
Addition of Crystalline KMnO4 to Reactors, Part I
Pre-weigh out (into disposable tare boats) the needed quantities of crystalline $KMnO_4$ (Carus™ Free Flowing Technical Grade) that is needed for each reactor according to which test condition in Table 2-6 is being evaluated. **For the 1 and 3 wt% MnO_4^- loadings, ≈ 6.6 g and ≈20 g of KMnO4 will be needed for each reactor, respectively.** Record the actual mass of $KMnO_4$ weighed out for each reactor.

20. **For the Oxidant Loadings of 1 and 3wt% MnO_4^- in Table 2-6 ONLY**
Addition of Crystalline KMnO4 to Reactors, Part II
Quickly remove the ground glass stopper and add these pre-weighed quantities of $KMnO_4$ into the applicable reactors. Replace the glass ground stoppers with the adapters fitted with the charcoal desorption tube assemblies and quickly hand shake each reactor to ensure rapid dissolution of the oxidant. (No $KMnO_4$ will be added to the control reactors). The time that $KMnO_4$ is added to each individual reactor shall also be recorded (reaction start time). Place each reactor back into the 12°C incubator and at 200 rpm of greater.

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21. 30 Minute Sampling
At approximately 30 minutes from the reaction start time, remove the reactors from the incubator and collect a 10 mL aliquot (using a long needle and syringe with a Luer Tip). Record the time the sample is collected. Replace the reactor(s) back into the incubator.

22. Preparation of 30 Minute TCE Sample
Dispense (unfiltered) ≈3 mL of the collected aliquot into a pre-weighed vial holding a known volume of hexane (≈3 mL). This sample vial will be weighed again after sample addition to determine the actual weight of sample added. All such extracts will be placed on a horizontal shaker for 2 hours. Store the extracts at 4°C until G.C. analysis.

23. pH Analysis of 30 Minute Sample
Tare a small beaker. Add a 0.45 µm syringe filter unto the end of the syringe and dispense (filtered) the remaining portion of the sample in the syringe (≈7 mL) into a tared beaker for pH analysis. Reweigh the beaker to determine the volume of the sample (assuming that the solids fraction is negligible). Measure and record the pH of the sample as done in Step **

24. MnO_4^- Analysis of the 30 Minute Sample
Use a portion of the filtered pH sample to perform a MnO_4^- analysis. The sample may have to be diluted so that its spectrophotometric absorbance (at λ =525 nm) is less than that of the highest standard to be prepared (50 mg/L MnO4-). Check standards (10 and 50 mg/L MnO4-) will be prepared with reagent grade $KMnO_4$ and measured each analysis day. A new 5 point calibration curve will be prepared if the absorbance of these check standards differs from the expected value by >15%.

25. Collection and Analysis of Additional Aliquot Samples
Collect and analyze aliquots from each reactor for TCE, pH, and $KMnO_4$ (Steps 21-24) at the following ADDITIONAL time periods: 2 hrs, 4 hrs, 6 hrs, 20 hrs, and 24 hrs.
IMPORTANT—For each reactor collect and extract a duplicate sample (Steps 21-24) from one of the 6 time intervals being evaluated.

26. Extraction of Charcoal Desorption Tubes
After the final aliquot samples are collected, remove the charcoal desorption tube(s) from each reactor. Each charcoal bed contained in the tube(s) will be placed into pre-filled and pre-weighed vials containing a known volume of hexane (≈5 mL). These extracts will be processed and analyzed in the same manner as the aqueous aliquots for TCE.

27. Clean up
Clean up the reactors for re-use in the next run of the experiment. Also place any waste materials generated from the experiment in the appropriate satellite accumulation area.

28. GC ANALYSIS of VOCs
All VOC analyses for this experiment will be conducted using a G.C./ECD detector. The instrument will calibrated for TCE (and also for cis-1,2 DCE, trans-1,2 DCE, and vinyl chloride for the co-contaminants experiment), using a 5 point calibration curve with standard concentrations ranging from 5-1000 µg/L (ppb)

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Table 2-6. TCE degradation rate in hot spot groundwater. (TSF05)

	Media	Basalt Form	Liquid:Solid Ratio	Initial TCE Concentration (mg/L)	Initial Organic Co-Contaminant Concentration (mg/L)	Initial Permanganate Concentration (%)
	CONTROL-Groundwater	NA	NA	10	0	0
EXP 4b	CONTROL-Groundwater	NA	NA	10	0	0
RUN 1	Groundwater	NA	NA	10	0	0.01
	Groundwater	NA	NA	10	0	0.01
	Groundwater	NA	NA	10	0	0.10
	Groundwater	NA	NA	10	0	0.10

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Tedlar bag wt. 52.117
Tedlar bag + GW 3104.1
g.w 3051.88
std TCE stock added 31.0 ml

Calib. X177440
50 49.98

flask #	wt. empty	wt + g.w	g.w. Vol.	KMnO4
1	245.383	728.83	483.45	~~6.59440~~
2	250.018	747.73	497.71	0
3	236.216	734.55	498.33	
4	251.774	764.60	512.83	6.5944
5	239.488	738.46	498.97	
6	255.346	761.49	506.14	

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pH/vol.

Flask#	T0/vol	T30min	T1.5hr/vol	T3hr./vol	T5hr/vol	T20hr/vol	T24hr/vol
1	8.62/7.28 / 8.63/7.39	8.61/7.43 / 8.63/7.39	8.61/7.74	8.60/7.30	8.61/7.56	8.59/7.46	8.60/7.48 / 8.61/7.28
2	8.62/7.50	8.64/7.43	8.61/7.38	8.63/7.37	8.60/7.45	8.60/7.40	8.67/7.31
3	8.62/7.47	8.24/6.89	8.05/6.87 / 7.96/7.29	7.89/6.95	7.89/7.24	7.85/6.66	7.80/6.96
4	8.63/6.94	8.22/7.23	7.93/7.09	7.80/7.47 / 7.80/7.40	7.76/7.38	7.70/6.79	7.72/7.50
5	8.63/7.32	7.81/7.29	7.73/7.20	7.71/7.55	7.70/7.47 / 7.68/7.49	7.67/7.25	7.72/7.49
6	8.63/7.36	7.73/7.32	7.70/7.22	7.71/7.39	7.68/7.42	7.67/7.37 / 7.67/7.28	7.69/7.46

spec. readings.

Flask#	T0	T30min	T1.5hr	T3hr.	T5hr.	T20hr.	T24hr.
		Flasks 3&4-5DF		Flasks 5&6-50DF			
1	0.000 / 0.001	0.004 / 0.005	0.004	0.002	0.005	0.004	0.004 / 0.008
2	0.001	0.003	0.004	0.002	0.003	0.004	0.005
3	0.002	0.174	0.169 / 0.164	0.150	0.168	0.162	0.135 / ~~0.136~~ DA
4	0.001	0.172	0.163	0.149 / 0.148	0.154	0.136	0.119
5	0.002	0.189	0.191	0.190	0.194 / 0.206	0.194 / 0.203	0.184
6	0.001	0.202	0.204	0.209	0.204	0.203	0.190

spec. check

stds	T0	T30min	T1.5hr.	T3hr.	T5hr	T20hr	T24hr.
BLK	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.114	1.116	1.115	1.116	1.116	1.117	1.120
10mg/L	0.215	0.217	0.215	0.213	0.213	0.214	0.211

Spec. reading on $KMnO_4$ stock. ~~DF~~ ~~Abs.~~

Abs. 0.274
DF 3333

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Sample time	GC#	wt. empty	wt. therm	wt. t.g.w.	Sample I.D.	DF
16:25	1494	10.968	12.889	16.001	4b-R1-5hr-4	1
16:26	1495	11.017	12.931	16.068	5a	1
16:27	1496	10.983	12.910	15.887	5b	1
16:28	1497	10.990	12.882	15.962	6	1
8/12 08:19	1498	11.060	12.967	15.829	4b-R1-20hr.-1	20
08:20	1499	10.912	12.895	16.175	2	20
08:21	1500	11.029	12.941	16.186	3	1
08:22	1501	10.961	12.865	16.070	4	1
08:23	1502	10.881	12.782	16.072	5	1
08:24	1503	10.927	12.814	15.766	6a	1
08:25	1504	11.071	12.955	16.063	6b	1
11:34	1505	10.977	12.887	16.135	4b-R1-24hr.-1a	20
11:35	1506	11.066	12.943	16.246	1b	20
11:35	1507	10.893	12.768	15.906	2	20
11:36	1508	11.029	12.890	16.028	3	1
11:38	1509	10.945	12.829	15.874	4	1
11:39	1510	10.820	12.653	15.784	5	1
11:40	1511	11.076	12.966	15.990	6	1
11:46	1512	10.913	12.835	12.930	4b-Richar 1a	1
11:47	1513	10.965	12.872	12.922	1b	1
11:49	1514	10.923	12.768	12.869	2a	1
11:48	1515	10.912	12.788	12.857	2b	1
11:49	1516	11.010	12.894	13.000	3a	1
11:50	1517	10.991	12.863	12.914	3b	1
11:51	1518	10.841	12.729	12.819	4a	1
11:52	1519	10.970	12.854	12.900	4b	1
11:55	1520	11.123	12.936	13.025	5a	1
11:54	1521	11.011	12.810	12.908	5b	1
11:45	1522	10.915	12.787	12.900	6a	1
11:45	1523	10.929	12.777	12.832	6b	1

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Sample time	GC#	wt. empty	wt + hexane	wt. + g.w	Sample ID.	DF.
31:11:98 10:58	1461	10.908	14.102	19.323	4b-R1-TBa	20
10:58	1462	10.931	14.149	19.339	4b-R1-TBb	20
11:20	1463	10.883	12.828	16.182	4b-R1-Tϕ-1a	20
11:20	1464	11.078	13.040	16.405	1b	20
11:21	1465	10.823	12.767	16.018	2a	20
11:22	1466	10.992	12.946	16.079	3a	20
11:23	1467	10.945	12.886	16.106	4a	20
11:23	1468	10.864	12.800	15.914	5	20
11:24	1469	10.858	12.820	15.886	6	20
12:00	1470	10.885	12.833	16.006	4b-R1-T30min-1	20
12:01	1471	10.929	12.881	16.031	2a	20
12:02	1472	11.110	13.050	16.090	2b	20
12:03	1473	11.050	12.989	16.201	3	1
12:04	1474	10.945	12.880	16.133	4	1
12:05	1475	10.928	12.854	15.908	5	1
12:06	1476	10.944	12.888	16.005	6	1
13:05	1477	10.911	12.835	15.723	4b-R1-T1.5hr-1	20
13:05	1478	10.947	12.867	16.121	2	20
13:06	1479	11.000	12.920	16.073	3a	1
13:07	1480	10.923	12.835	15.834	3b	1
13:08	1481	10.984	12.901	16.068	4	1
13:09	1482	11.045	12.935	16.018	5	1
13:10	1483	11.081	13.013	16.120	6	1
14:30	1484	10.914	12.825	16.021	4b-R1-T3hr-1	20
14:31	1485	10.995	12.913	16.639	2	20
14:32	1486	10.947	12.859	15.856	3	1
14:33	1487	10.973	12.892	15.878	4a	1
14:34	1488	10.980	12.898	15.774	4b	1
14:35	1489	10.935	12.857	16.024	5	1
14:36	1490	11.022	12.938	16.025	6	1
16:22	1491	10.853	12.755	15.940	4b-R1-T5hr-1	20
16:23	1492	10.965	12.894	16.045	2	20
16:24	1493	10.891	12.800	15.915	3	↓

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	media	basalt form	liquid solid ratio	initial TCE conc. (ug/L)	initial co-contam. conc. (ug/L)	initial permanganate conc.
	CONTROL-Groundwater	NA	NA	10	0	0
EXP 4b	CONTROL-Groundwater	NA	NA	10	0	0
RUN 2	Groundwater	NA	NA	10	0	1.00
	Groundwater	NA	NA	10	0	1.00
	Groundwater	NA	NA	10	0	3.00
	Groundwater	NA	NA	10	0	3.00

KM 8/10/98

Tedlar bag wt. 52.344
Tedlar bag + gw. 315.9
sat. soln. TCE stock added. 31.0 ml

Calib X177440
50g 49.95 ✓

flask#	wt. empty	wt. + gw	g. w vol	(g) $KMnO_4$
1	241.560	755.63	514.07	—
2	250.096	748.99	498.89	—
3	240.215	737.52	497.31	6.6059
4	251.784	751.26	499.48	6.5944
5	239.611	738.76	499.15	20.0018
6	255.390	759.56	504.17	20.0153

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pH/vol. Flask #	T0/vol	T30min/vol	T1.5hr./vol	T3hr./vol	T5hr/vol	T20hr/vol	T24hr/vol
1	8.58/7.41 8.57/7.20	8.56/7.30	8.54/7.36	8.53/7.33	8.53/7.60	8.66/7.17	8.66/6.26
2	8.57/7.22	8.57/7.39 8.58/7.08	8.55/7.45	8.53/7.25	8.53/7.40	8.67/7.07	~~8.69/7.20~~ D.
3	8.57/7.12	8.39/7.31	8.43/6.85 8.43/7.29	8.44/6.91	8.45/6.99	8.57/6.88	8.57/6.96
4	8.58/7.23	8.25/7.12	8.40/7.37	8.34/7.36 8.39/7.46	8.41/7.17	8.52/7.21	8.52/7.30
5	8.58/7.17	8.70/7.23	8.80/7.39	8.81/7.48	8.82/7.38 8.86/7.43	8.93/7.65	8.91/7.22
6	8.57/7.11	8.75/7.35	8.83/7.56	8.84/7.51	8.86/7.51	8.97/7.52 8.98/7.12	8.96/7.39 8.97/7.18

spec. readings. Flask #	T0	T30min.	T1.5hr.	T3hr.	T5hr.	T20hr.	T24hr.
1	0.001 0.001	0.022 0.003	0.005	0.013	0.005	0.001	0.003 ~~0.215~~ D.
2	0.000	0.003	0.003	0.006	0.007	0.004	0.009
3	0.002	0.604 (DF 250)	0.815 (DF 250) 0.864 (250)	0.189 (1000)	0.409 (500)	0.045	0.047
4	0.000	0.462 (DF 250)	0.847 (250)	0.199 (1000) 0.202 (1000)	0.406 (500)	0.037	0.046
5	0.000	0.322 (DF 500)	1.185 (500)	0.582 (1000)	0.598 (1000) 0.604 (1000)	0.066	0.062
6	0.000	0.408 (DF 500)	1.232 (500)	0.599 (1000)	0.540 (1000)	0.070 0.069	0.073 0.067

speck check. stds.	T0	T30min	T1.5hr.	T3hr.	T5hr.	T20hr.	T24hr.
blank	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.113	1.113	1.115	1.117	1.119	1.128	1.125
10mg/L	0.222	0.219	0.220	0.219	0.219	0.216 KM	0.215

~~spec. reading on KMnO4 Stock.~~ KM ~~BF~~ Abs. spec. Repeats.

dil. 24hr	dil 20hr.	flask #	T20hr	T24hr.
0	KM ~~0~~ 0	1	0.002	0.005
0	0	2	0.005	0.009
500	KM 500	3	0.436	0.475
500	KM 500	4	0.391	0.424
1000	1000	5	0.611	0.605
1000	1000	6a	0.642	0.629
		6b	0.656	0.635

Continued on page 104

K. McCracken — Recorded by
8/13/98 — Date
SR — Read and understood by
8/17/98 — Date

Subject Exp 4b-R2

Date 08 (Month) 13 (Day) 19 98 (Year)

Sampletime	GC#	wt. empty	wt. + hexane	wt. + gw	Sample ID	DF
8/13 11:30	1660	11.020	14.292	19.223	4b-R2-TBa	20
11:31	1661	10.926	14.201	18.980	4b-R2-TBb	
11:30	1662	10.906	12.853	16.142	4b-R2-T0-1a	
11:31	1663	10.886	12.842	16.148	1b	
11:32	1664	10.904	12.869	16.092	2	
11:33	1665	10.948	12.917	16.169	3	
11:34	1666	10.851	12.809	15.928	4	
11:35	1667	10.989	12.951	16.108	5	
11:36	1668	11.013	12.954	16.084	6	
12:10	1669	10.945	12.897	16.046	4b-R2-T30min-1	
12:11	1670	10.989	12.940	16.027	2a	
12:12	1671	10.908	12.856	16.036	2b	
12:13	1672	10.983	12.933	16.193	3	1
12:14	1673	10.888	12.832	15.907	4	1
12:15	1674	10.804	12.746	15.955	5	1
12:16	1675	10.899	12.841	15.984	6	1
13:20	1676	10.912	12.851	16.117	4b R2-T1.5hr.-1	20
13:21	1677	10.950	12.889	15.997	2	20
13:22	1678	10.916	12.850	16.159	3a	1
13:23	1679	11.066	13.006	16.047	3b	1
13:24	1680	11.021	12.964	16.095	4	1
13:25	1681	11.138	13.073	16.317	5	1
13:26	1682	11.052	12.999	16.075	6	1
14:45	1683	11.059	12.990	16.008	4b R2-T3hr.-1	20
14:46	1684	11.082	13.005	16.037	2	20
14:47	1685	10.916	12.908	16.161	3	1
14:48	1686	10.910	12.848	16.033	4a	1
14:49	1687	10.965	12.905	16.032	4b	1
14:50	1688	11.113	13.048	16.240	5	1
14:51	1689	10.925	12.863	16.096	6	1
16:50	1690	10.956	12.892	15.936	4b R2-T5hr-1	20
16:51	1691	11.052	12.980	16.060	2	20
16:52	1692	10.952	12.858	15.940	3	1

Continued on page 105

K. McCracken 8/13/98
Recorded by Date

SPC 8/17/98
Read and understood by Date

Subject Exp. 4b-R2

Date 08 (Month) 13 (Day) 19 98 (Year)

Sampletime	GC#	wt. empty	wt. Hexane	wt. tgw.	Sample ID.	DF
16:53	1693	10.914	12.840	16.126	4b-R2-T5hr.-4	1
16:54	1694	10.906	12.666	16.048	5a	1
16:55	1695	10.988	12.919	16.076	5b	1
16:56	1696	10.920	12.858	15.894	6	1
8/14 8:11	1697	10.916	12.846	16.053	4b-R2-T20hr.-1	20
8:12	1698	10.933	12.861	16.290	2	20
8:13	1699	10.934	12.855	16.140	3	1
8:14	1700	10.960	12.879	16.317	4	1
8:15	1701	11.085	13.002	16.141	5	1
8:16	1702	10.965	12.896	16.246	6a	1
8:17	1703	10.930	12.854	16.514	6b	1
11:30	1704	10.892	12.810	16.869	4b-R2-T24hr.-1	20
11:31	1705	10.978	12.891	16.087	2	20
11:32	1706	10.913	12.840	16.044	3	1
11:33	1707	11.066	12.925	16.147	4	1
11:34	1708	10.853	12.755	16.262	5	1
11:35	1709	10.990	12.886	16.255	6a	1
11:36	1710	10.993	12.899	16.210	6b	1
11:46	1711	11.022	12.935	13.029	4b-R2 Char-1a	1
11:45	1712	11.084	12.977	13.005	1b	1
11:47	1713	11.014	12.933	13.012	2a	1
11:48	1714	10.946	12.854	12.888	2b	1
11:49	1715	10.994	12.903	12.992	3a	1
11:50	1716	10.960	12.854	12.899	3b	1
11:52	1717	10.852	12.770	12.861	4a	1
11:51	1718	10.910	12.836	12.875	4b	1
11:54	1719	10.920	12.847	12.945	5a	1
11:53	1720	11.017	12.927	12.970	5b	1
11:56	1721	11.039	12.960	13.044	6a	1
11:55	1722	11.008	12.901	12.936	6b	1
11:58	1723	10.930	12.811	12.905	ORBO HOOD-A	
11:57	1724	11.013	12.904	12.951	ORBO HOOD-B	
12:00	1725	10.913	12.801	12.884	ORBO INCUB-A	
11:59	1726	10.981	12.864	12.890	ORBO INCUB-B	

Continued on page

K. McCracken 8/13/98
Recorded by Date

SRC 8/17/98
Read and understood by Date

Subject Exp. 4a - R6

Date 08 Month 17 Day 19 98 Year

	media	basalt form	liquid/solid RATIO	Initial TCE conc.	Initial Co-contam. conc.	Initial MnO4 conc.
	CONTROL Groundwater	NA	NA	1 mg/L	0	0
	CONTROL Groundwater	NA	NA	1	0	0
EXP 4a	Groundwater/basalt	Aggregate	4:1	1	0	0.01
RUN 6	Groundwater/basalt	Aggregate	4:1	1	0	0.01
	Groundwater/basalt	Aggregate	4:1	1	0	0.10
	Groundwater/basalt	Aggregate	4:1	1	0	0.10

K.M. 8/17/98

Tedlar bag — 51.943
Tedlar bag + H_2O — 285.79
g.w Vol. — 2806.0
got soln. TCE added — 2.8 ml

spike soln. incubated at 12°C for 3 hrs. to equilibrate

flask#	wt empty	wt + media	wt + g.w H_2O	g.w. vol.	
1	243.482	—	753.65	510.17	
2	252.191	—	725.00	472.81	
3	240.809	340.934	729.08	388.15	
4	255.782	356.115	762.52	406.41	
5	237.839	338.229	755.75	417.52	*
6	248.858	348.988	730.88	381.89	*

8/18 calib. x 177440 / 50. 49.93

Stock KMnO4 2.66 x 2 / 5.32 g/100ml of H_2O

8/20 calib. x 177440 / 50 49.95

* Please note: While incubating Tedlar bag spike solution, a leak developed. Contents remaining in bag were used for flasks 1, 2, 3, & 4. for flasks 5 & 6, each got 200 ml of spike solution from the bag and 200 ml spike solution which had leaked out into the tray during equilibration.

Continued on page 107

K. McCracken 8/17/98
Recorded by Date

SP 8/25/98
Read and understood by Date

Subject Exp. 4a-R6

Date 08 (Month) 17 (Day) 1998 (Year)

Vol.

flask#	T0	T30 min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	7.41 / 7.59	7.34	7.41	7.62	7.43	7.64	7.60
2	7.20	7.20 / 7.18	7.33	7.48	7.26	7.34	7.35
3	7.37	6.88	6.95 / 6.99	6.98	7.22	7.10	6.88 / 7.39
4	7.32	7.00	7.40	7.31 / 6.96	7.19	7.32	7.26
5	7.23	7.18	7.43	7.56	7.29 / 7.43	7.32	7.26
6	6.85	7.19	7.47	7.22	7.17	7.33 / 7.08	7.32

pH readings

flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	8.40 / 8.41	8.37	8.42	8.38	8.38	8.28	8.26
2	8.41	8.41 / 8.43	8.41	8.38	8.38	8.24	8.24
3	8.38	8.27	8.25 / 8.23	8.24	8.24	8.19	8.22 / 8.21
4	8.37	8.27	8.22	8.23 / 8.22	8.23	8.18	8.21
5	8.35	8.39	8.37	8.36	8.36 / 8.36	8.29	8.30
6	8.34	8.39	8.37	8.35	8.36	8.29 / 8.29	8.30

spec. readings * flasks 1 & 2 NDF; 3 & 4 DF=5 ; 5 & 6 DF=50

flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	0.004 / 0.004	0.005	0.006	0.004	0.006	0.004	0.009
2	0.004	0.004 / 0.003	0.005	0.004	0.005	0.005	0.005
3	0.009	0.508	0.472 / 0.498	0.495	0.494	0.480	0.483 / 0.530
4	0.003	0.494	0.483	0.491 / 0.490	0.487	0.476	0.552
5	0.002	0.491	0.480	0.492	0.490 / 0.525	0.497	0.507
6	0.001	0.579	0.565	0.554	0.565	0.571 / 0.581	0.564

spec stds. check

stds	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
Blank	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.120	1.120	1.116	1.116	1.118	1.121	1.121
10mg/L	0.214	0.214	0.212	0.211	0.209	0.206	0.206

std. $KMnO_4$ – 150 µl → 250 ml H_2O.

DF = 1666 Abs. = 0.539

K. McCracken — Recorded by — Date 8/20/98

Continued on page 108

SPC — Read and understood by — Date 8/25/98

Subject Exp. 4a-R6

Date 08 (Month) 18 (Day) 19 98 (Year)

Sampling time	GC#	wt. empty	wt. + hexane	wt. + sample	sample ID.	DF
8/18 13:45	1727	11.224	14.471	19.722	4a-R6-TBa	2
13:45	1728	10.968	14.181	19.101	4a-R6-TB b	
8/19 11:00	1729	11.219	13.106	16.337	4a-R6-Tϕ-1a	
11:01	1730	10.969	12.854	16.011	1b	
11:02	1731	11.150	13.047	16.073	2	
11:03	1732	11.053	12.940	16.139	3	
11:04	1733	11.090	13.011	16.133	4	
11:05	1734	11.085	13.000	16.220	5	
11:06	1735	10.916	12.793	15.826	6	
11:30	1736	10.921	12.805	15.859	4a-R6-T30min-1	
11:31	1737	10.975	12.860	16.042	2a	
11:32	1738	10.028	12.921	16.105	2b	2
11:33	1739	11.202	13.068	16.175	3	1
11:34	1740	11.049	12.921	15.963	4	1
11:35	1741	11.020	12.909	16.064	5	1
11:36	1742	11.031	12.916	16.030	6	1
12:36	1743	11.145	13.034	16.210	4a-R6-T1.5hr.-1	2
12:37	1744	11.134	13.018	16.223	2	2
12:38	1745	11.098	13.029	16.174	3a	1
12:39	1746	11.076	12.961	16.283	3b	1
12:40	1747	11.214	13.097	16.194	4	1
12:41	1748	10.919	12.813	15.864	5	1
12:42	1749	10.997	12.895	15.940	6	1
14:05	1750	11.001	12.898	15.946	4a-R6-T3hr-1	2
14:06	1751	11.169	13.040	16.148	2	2
14:07	1752	11.155	13.017	16.010	3	1
14:08	1753	11.078	12.941	15.957	4a	1
14:09	1754	11.063	12.953	16.194	4b	1
14:10	1755	11.135	13.031	16.095	5	1
14:11	1756	11.070	12.962	16.210	6	1
16:04	1757	11.106	13.028	16.112	4a-R6-T5hr-1	2
16:05	1758	11.121	13.014	16.294	2	2
16:06	1759	11.186	13.074	16.404	3	1

Continued on page 109

K.M. McCracken — Recorded by — Date 8/20/98

SPA — Read and understood by — Date 8/25/98

Subject Exp. 4a - R6

Date 08 (Month) 19 (Day) 1998 (Year)

Sampletime	GC #	wt empty	wt.+hexane	wt.+sample	Sample ID	DF
16:07	1760	11.103	12.997	16.582	4a-R6-T5hr-4	1
16:08	1761	10.952	12.810	16.181	5a	1
16:09	1762	10.971	12.867	15.981	5b	1
16:10	1763	11.041	12.913	16.052	6	1
8/20 08:10	1764	11.260	13.129	16.192	4a-R6-T20hr-1	2
08:11	1765	11.079	12.970	16.029	2	2
08:12	1766	11.071	12.940	15.906	3	1
08:13	1767	11.094	12.987	16.199	4	1
08:14	1768	11.230	13.121	16.281	5	1
08:15	1769	11.073	12.974	16.129	6a	1
08:16	1770	11.049	12.936	16.252	6b	1
11:00	1771	11.154	13.035	16.065	4a-R6-T24hr-1 km	2
11:01	1772	11.041	12.930	16.171	2 km	2
11:02	1773	11.199	13.100	16.180	3a	1
11:03	1774	10.918	12.799	15.911	3b	↓
11:04	1775	11.128	13.009	16.093	4	↓
11:05	1776	11.124	12.988	16.122	5	↓
11:06	1777	11.146	13.022	16.250	6	↓
11:17	1778	11.016	12.885	12.968	4a-R6-char-1a	↓
11:17	1779	11.178	13.066	13.096	1b	↓
11:18	1780	11.082	12.965	13.062	2a	↓
11:18	1781	11.182	13.096	13.129	2b	↓
11:19	1782	11.088	13.009	13.089	3a	↓
11:19	1783	11.059	12.941	12.977	3b	↓
11:20	1784	11.060	12.954	13.038	4a	↓
11:20	1785	11.001	12.895	12.938	4b	↓
11:21	1786	11.070	12.909	13.001	5a	↓
11:21	1787	11.186	13.049	13.087	5b	↓
11:22	1788	11.198	13.071	13.161	6a	↓
11:22	1789	11.059	13.024	13.055	6b	↓
11:24	1790	493.28	~~626.28~~ 759.28 km	1148.72	4a-R6-f3	↓
11:26	1791	492.78	~~625.82~~ 758.82 km	1179.03	4a-R6-f4	↓
11:28	1792	491.95	~~625.95~~ 758.95 km	1200.01	4a-R6-f5	1
11:30	1793	492.80	~~626.00~~ 759.00 km	1162.29	4a-R6-f6	1

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K. McCracken — Recorded by; 8/20/98 — Date; SDC — Read and understood by; 8/25/98 — Date

See page 114 for DF 5 repeats - KM.

F-109

Subject Exp. 4a-R6 repeat dilutions

Date 08 (Month) 21 (Day) 19 98 (Year)

DF	GC#	Sample ID
5	1727R	4a-R6-TBa
	1728R	4a-R6-TBb
	1729R	4a-R6-Tϕ-1a
	1730R	1b
	1731R	2
	1732R	3
	1733R	4
	1734R	5
	1735R	6
	1736R	4a-R6-T30min-1
	1737R	2a
	1738R	2b
	1743R	4a-R6-T1.5h-1
	1744R	2
	1750R	4a-R6-T3h-1
	1751R	2
	1757R	4a-R6-T5h-1
	1758R	2
	1764R	4a-R6-T20h-1
	1765R	2
	1771R	4a-R6-T24h-1
5	1772R	2

Continued on page

K. McCracken — Recorded by — 08/21/98 Date

SRC — Read and understood by — 8/25/98 Date

Subject Exp. 4a - R8

Date 08 Month 24 Day 1998 Year

	Media	Soil form	liquid/solid ratio	Initial TCE conc. (mg/L)	Initial [illegible] conc. (mg/L)	Initial MnO_4 conc.
	CONTROL Groundwater	NA	NA	1	0	0
	CONTROL Groundwater	NA	NA	1	0	0
EXP 4a	Groundwater/sediment	NA	4:1	1	0	0.01
RUN 8	Groundwater/sediment	NA	4:1	1	0	0.01
	Groundwater/sediment	NA	4:1	1	0	0.10
	Groundwater/sediment	NA	4:1	1	0	0.10

KM 8/17/98

Tedlar bag 54.063g
Tedlar bag + g.w. 2755.2
g.w. vol. 2701.137
sat. soln. TCE added 2.70 ml

8/26 Calib X177440
50g 49.95g

flask#	wt. empty	wt. + media	wt. + g.w.	g.w. vol.
1	243.225	—	737.41	494.185
2	256.640	—	755.55	498.910
3	240.168	342.228	721.94	379.712
4	251.790	351.799	735.85	384.051
5	239.573	339.522	734.32	394.798
6	246.986	351.516	756.61	405.094

KM

* Sediment for this exp. is from shipment #2, consists of a mixture of:

348'-353' (349)	84.63g
213.5'-228'	148.9g
QR (441')	190.0g
366'	10.3g
T33-CI-144'-144.2	129.0g
	562.8 Total g.

All were ground & mixed together thoroughly. 8/21/98 KM

K. McCracken 8/24/98
Recorded by Date

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8/27 SRC 8/27/98
Read and understood by Date

Subject Exp. 4a - R8

Date 08 (Month) 25 (Day) 1998 (Year)

vol.

flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	7.28/7.25	7.22	7.27	7.40	7.408	7.31	7.20
2	7.11	7.09/7.09	7.42	7.36	7.308	7.08	7.29
3	7.02	6.53	7.04/7.17	6.88	6.79	6.78	6.45
4	6.76	6.89	7.00	7.26/6.71	6.27	6.78	6.82/6.73
5	7.25	7.00	7.36	7.32	6.84/7.30	6.86	6.78
6	6.85	7.03	7.15	6.83	7.28	6.82/7.33	7.34

pH readings.

flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	8.12/8.05	8.12	8.11	8.12	8.08	8.00	8.09
2	8.04	8.06/8.04	8.05	8.06	8.08	7.98	8.04
3	8.06	8.00	8.00/7.96	8.00	8.01	8.00	8.01
4	8.03	7.92	7.94	7.98/7.97	7.97	7.96	7.98/7.98
5	8.00	7.75	7.74	7.75	7.74/7.73	7.75	7.76
6	8.00	7.83	7.73	7.75	7.73	7.74/7.74	7.76

Spec readings.

flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	0.003/0.003	0.020	0.004	0.004	0.006	0.004	0.005
2	0.004	0.004/0.004	0.003	0.004	0.004	0.003	0.005
3	0.002	0.469	0.416/0.425	0.417	0.410	0.373	0.383
4	0.004	0.518	0.443	0.433/0.442	0.425	0.396	0.400/0.401
5	0.004	0.549	0.522	0.517	0.520/0.555	0.539	0.530
6	0.003	0.573	0.538	0.544	0.556	0.531/0.533	0.529

spec. check

stds	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
Blk.	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.112	1.112	1.112	1.114	1.115	1.116	1.118
10mg/L	0.222	0.222	0.222	0.220	0.231? 0.231✓	0.229	0.230

$KMnO_4$ Std. 0.6 ml → 1 L

ABS = 0.508
DF = 1666

Continued on page 112

K. McCracken — Recorded by — 8/25/98 Date

SPCC — Read and understood by — 8/27/98 Date

Subject Exp. 4a - R8

Date 08 (Month) 25 (Day) 19 98 (Year)

sample time	GC#	wt. empty	wt. + hexane	wt. + sample	Sample ID	DF
8/24 0 16:20	1794	11.170	14.401	19.478	4a-R8-TBa	5
16:20	1795	11.240	14.436	19.444	4a-R8-TBb	
8/24 10:30	1796	11.214	13.105	16.336	4a-R8-Tφ-1a	
10:31	1797	11.208	13.115	16.342	1b	
10:32	1798	11.074	12.983	16.234	2	
10:33	1799	11.200	13.070	16.452	3	
10:34	1800	11.215	13.108	16.229	4	
10:35	1801	11.120	13.096	16.407	5	
10:36	1802	10.92	12.820	16.088	6	
11:00	1803	11.190	13.053	16.224	4a-R8-T30min.-1	
11:01	1804	11.063	12.855	16.057	2a	
11:02	1805	11.030	12.883	16.011	2b	5
11:03	1806	11.131	13.012	16.459	3	1
11:04	1807	11.230	13.125	16.295	4	1
11:05	1808	11.035	12.921	16.089	5	1
11:06	1809	11.233	13.125	16.428	6	1
12:40	1810	11.198	13.088	16.203	4a-R8-T1.5hr-1	5
12:41	1811	11.333	13.210	16.400	2	5
12:42	1812	11.280	13.157	16.289	3a	1
12:43	1813	11.100	12.969	16.207	3b	1
12:44	1814	11.117	13.003	16.005	4	1
12:45	1815	11.170	13.024	16.036	5	1
12:46	1816	11.183	13.036	16.067	6	1
14:00	1817	11.053	12.913	15.767	4a-R8-T3hr-1	5
14:01	1818	11.050	12.917	16.200	2	5
14:02	1819	11.17	13.058	16.251	3	1
14:03	1820	11.057	12.943	16.132	4a	1
14:04	1821	11.265	13.153	16.135	4b	1
14:05	1822	11.166	13.075	16.229	5	1
14:06	1823	11.193	13.071	16.382	6	1
16:00	1824	11.250	13.146	16.408	4a-R8-T5hr-1	5
16:01	1825	11.160	13.045	16.423	2	5
16:02	1826	~~11.172~~ 11.148	13.128	16.308	3	1

Continued on page 113

K.McCracken — Recorded by

8/25/98 — Date

SR C — Read and understood by

8/27/98 — Date

Subject Exp. 4a - R8

Date 08 (Month) 25 (Day) 1998 (Year)

Sample time	GC#	wt. empty	wt. + hexane	wt. + sample	Sample ID	DF
16:03	1827	10.965	12.841	16.142	4a-R8-T5hr-4	1
16:04	1828	10.916	12.764	15.872	5a	1
16:05	1829	11.982	12.835	15.914	5b	1
16:06	1830	11.063	12.947	16.116	6	
8/26 08:40	1831	11.030	13.010	16.303	4a-R8-T20hr.-1	5
08:41	1832	11.028	12.925	16.206	2	5
08:42	1833	11.270	13.172	16.316	3	1
08:43	1834	11.220	13.130	16.300	4	1
08:44	1835	11.280	13.173	16.413	5	1
08:45	1836	11.201	13.099	16.555	6a	1
08:46	1837	11.040	12.941	16.179	6b	1
10:45	1838	11.068	12.968	16.209	4a-R8-T24hr.-1	5
10:46	1839	11.167	13.029	16.300	2	5
10:47	1840	11.162	13.031	15.953	3	1
10:48	1841	11.000	12.873	16.037	4a	1
10:49	1842	10.996	12.848	15.920	4b	1
10:50	1843	11.049	12.940	16.280	5	1
10:51	1844	10.955	12.843	16.141	6	1
11:02	1845	11.290	13.201	13.276	4a-R8-char-1a	
11:02	1846	11.200	13.106	13.131	1b	
11:04	1847	11.300	13.187	13.269	2a	
11:04	1848	11.173	13.066	13.098	2b	
11:05	1849	11.132	13.022	13.100	3a	
11:06	1850	11.192	13.091	13.118	3b	
11:07	1851	11.086	14.315	14.392	4a	
11:07	1852	11.140	13.014	13.031	4b	
11:08	1853	11.046	12.893	12.969	5a	
11:08	1854	11.069	12.944	12.977	5b	
11:10	1855	11.088	13.000	13.060	6a	
11:10	1856	11.100	12.993	13.011	6b	
11:11	1857	493.60	759.34	1150.52	4a-R8-f3	
11:12	1858	493.06	758.37	1144.16	4a-R8-f4	
11:13	1859	492.29	758.12	1174.23	4a-R8-f5	
11:14	1860	492.47	758.50	1188.78	4a-R8-f6	

Continued on page

K. McCracken — Recorded by — Date 8/25/98

SPC — Read and understood by — Date 8/27/98

Subject Exp. 4b-R5

Date 08 (Month) 26 (Day) 1998 (Year)

media	basalt	liquid/solid ratio	initial TCE conc.	initial catalyst conc.	initial permanganate conc.
control-groundwater-TSF05	NA	NA	100 ppm	10 ppm	0
control-groundwater-TSF05	NA	NA			0
groundwater-TSF05	NA	NA			0.01
groundwater-TSF05	NA	NA			0.01
groundwater-TSF05	NA	NA			0.10
groundwater-TSF05	NA	NA			0.10

Tedlar bag - 52.061
Tedlar bag + g.w. - 3131.50
g.w. vol. 3079.44
TCE - 0.2 ml
~~trans~~ 1,1-DCE - 25 ml
cis 1,2-DCE - 25 ml
trans-1,2-DCE - 25 ml

Calib.
X177440
50 49.969

Calib.
X177339
20 - 20.000

flask #	wt. empty	wt. + g.w.	g.w. vol.
1	241.481	733.82	
2	250.010	749.66	
3	240.650	741.01	
4	251.410	750.12	
5	246.332	753.15	
6	248.780	750.11	

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K. McCracken — Recorded by
8/26/98 — Date
SPC — Read and understood by
9-10-98 — Date

Subject Exp. 4b-R5

Date 08 (Month) 27 (Day) 19 98 (Year)

Sample Time	GC#	wt empty	wt. Hexane	wt. + sample	sample ID	DF
8/27 11:00	1861	11.099	14.363	19.893	4b-R5-TBa	100;
11:00	1862	11.173	14.463	19.549	4b-R5-TBb	
11:01	1863	11.080	13.021	16.419	4b-R5-T4-1a	
11:02	1864	11.032	13.002	16.252	1b	
11:03	1865	11.109	13.081	16.510	2	
11:04	1866	11.092	13.060	16.339	3	
11:05	1867	11.043	13.011	16.316	4	
11:06	1868	10.965	12.933	16.282	5	
11:07	1869	11.152	13.113	16.322	6	
11:36	1870	11.065	13.047	16.363	4b-R5-T30min-1	
11:37	1871	10.956	12.937	16.115	2a	
11:38	1872	11.161	13.139	16.464	2b	
11:39	1873	10.844	12.814	16.000	3	1
11:40	1874	10.891	12.867	16.156	4	1
11:41	1875	10.939	12.918	16.174	5	1
11:42	1876	10.950	12.925	16.130	6	1
12:30	1877	10.870	12.721	15.943	4b-R5-T1.5h-1	100;
12:31	1878	10.913	12.882	16.039	2	100;
12:32	1879	10.958	12.908	16.162	3a	1
12:33	1880	11.012	12.965	16.027	3b	1
12:34	1881	11.149	13.118	16.238	4	1
12:35	1882	11.024	12.986	16.265	5	1
12:36	1883	11.038	12.905	16.235	6	1
14:02	1884	11.292	13.252	16.334	4b-R5-T3h-1	100; 20
14:03	1885	11.106	13.080	16.341	2	100; 20
14:04	1886	11.147	13.122	16.412	3	1
14:05	1887	11.233	13.202	16.488	4a	1
14:06	1888	11.004	12.978	16.096	4b	1
14:07	1889	10.971	12.937	16.295	5	1
14:08	1890	10.903	12.870	16.258	6	1
16:00	1891	11.152	13.120	16.193	4b-R5-T5h-1	100;
16:01	1892	11.103	13.063	16.247	2	100;
16:02	1893	11.148	13.115	16.223	3	1

K. McCracken — Recorded by

08/27/98 — Date

Continued on page 117

SRC — Read and understood by

9/10/98 — Date

Subject Exp. 4b-R5

Date 08 Month 27 Day 1998 Year

Sample Time	GC#	wt. empty	wt. + glass	wt. + sample	Sample ID.	DF
16:03	1894	11.137	13.100	16.347	4b-R5-T5h-4	1
16:04	1895	11.071	13.040	16.383	5a	1
16:05	1896	11.263	13.224	16.426	5b	1
16:06	1897	11.200	13.160	16.500	6	1
928090	1898	11.092	13.066	16.222	4b-R5-T20h-1	100;
0811	1899	11.265	13.222	16.504	2	100;
08:12	1900	11.341	13.313	16.465	3	1
08:13	1901	11.080	13.043	16.313	4	1
08:14	1902	10.985	12.946	16.203	5	1
08:15	1903	10.965	12.930	16.184	6a	1
08:16	1904	11.007	12.955	16.250	6b	1
11:05	1905	11.127	13.078	16.279	4b-R5-T24h-1	100;
11:06	1906	11.019	12.942	16.024	2	100;
11:07	1907	10.983	12.900	16.230	3	1
11:08	1908	11.199	13.117	16.169	4	1
11:09	1909	11.127	13.045	16.324	5a	1
11:10	1910	10.904	12.827	15.922	5b	1
11:11	1911	10.979	12.902	16.057	6	1
11:12	1912	11.066	12.991	13.073	4b-R5-char-1a	2
11:12	1913	10.926	12.851	12.878	1b	1
11:13	1914	11.110	13.042	13.125	2a	2
11:13	1915	10.976	12.905	12.934	2b	1
11:14	1916	11.010	12.930	13.019	3a	2
11:14	1917	10.930	12.859	12.885	3b	1
11:15	1918	11.319	13.256	13.333	4a	2
11:15	1919	11.042	12.971	13.000	4b	1
11:16	1920	11.154	13.076	13.159	5a	2
11:16	1921	11.108	13.028	13.052	5b	1
11:17	1922	10.999	12.923	13.006	6a	2
11:17	1923	11.099	13.034	13.063	6b	1

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K. McCracken 8/27/98
Recorded by Date

SPC 9-10-98
Read and understood by Date

Subject Exp. 46-R5

Date 08 (Month) 27 (Day) 19 98 (Year)

volume

Flask#	T0	T30min	T1.5h	T3h	T5h	T20h	T24h
1	7.32/7.28	7.20	7.32	7.35	7.48	7.40	7.45
2	7.08	7.22/7.18	7.31	7.14	7.39	7.29	7.31
3	7.15	6.90	6.75/7.38	6.93	6.85	6.59	6.67
4	7.21	7.17	7.45	7.38/7.32	7.40	7.02	7.44
5	6.99	7.28	7.23	7.25	7.30/7.38	7.11	7.20/7.17
6	7.18	7.09	7.43	7.38	7.49	7.10/7.12	7.31

pH readings

Flask#	T0	T30min	T1.5h	T3h	T5h	T20h	T24h
1	8.65/8.51	8.48	8.46	8.44	8.46	8.44	8.46
2	8.50	8.50/8.51	8.46	8.45	8.46	8.44	8.46
3	8.51	7.63	7.57/7.43	7.47	7.54	7.49	7.53
4	8.50	7.50	7.35	7.30/7.25	7.38	7.33	7.36
5	8.50	7.20	7.22	7.17	7.23/7.10	7.20	7.27/7.18
6	8.50	7.09	7.14	7.08	7.07	7.12/7.10	7.13

spec. readings DF. 1&2-01 ; 3&4-05 ; 5&6-50

Flask#	T0	T30min	T1.5h	T3h	T5h	T20h	T24h
1	0.004/0.003	0.003	0.004	0.004	0.005	0.003	0.003
2	0.003	0.004/0.007	0.003	0.003	0.005	0.004	0.004
3	0.003	0.307	0.246/0.256	0.218	0.190	0.404 DF2	0.406 DF2
4	0.003	0.323	0.260	0.248/0.220	0.199	0.426 DF2	0.430 DF2
5	0.004	0.368	0.373	0.370	0.360/0.397	0.373	0.372/0.392
6	0.004	0.390	0.404	0.400	0.399	0.395/0.398	0.397

spec. stds.	T0	T30min	T1.5h	T3h	T5h	T20h	T24h
Blank	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.119	1.119	1.120	1.113	0.114	1.119	1.116
10mg/L	0.226	0.226	0.226	0.216	0.212	0.222	0.222

stock $KMnO_4$ - Abs. 0.522
DF 1666

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K. McCracken 8/27/98
Recorded by Date

SRC 9-10-98
Read and understood by Date

Subject Exp. 46-R5

Date 08 Month 28 Day 19 98 Year

Additional dilutions

GC#	DF
1861B	20
1862B	20
1863B	
1864B	
1865B	
1866B	
1867B	
1868B	
1869B	
1870B	
1871B	
1872B	
1877B	
1878B	
1884B	
1885B	
1891B	
1892B	
1898B	
1899B	
1905B	
1906B	20

calibration check standards were made by Sue Carroll.

K McCracken 8/28/98
Recorded by Date

SRC Continued on page 9-10-98
Read and understood by Date

Subject Exp. 4b - R5 Repeat/new dilutions.

Date 09 Month 01 Day 1998 Year

Repeats

GE#	DF
1879R	2
1880R	2
1881R	2
1891R	50
1905R	50

Continued on page —

K. McCracken 9/1/98
Recorded by Date

SR Cl 9-1-98
Read and understood by Date

Subject Exp. 4b-K6

Date 08 (Month) 31 (Day) 19 98 (Year)

media	~~ha~~ baseltform	liquid/solid RATIO	initial TCE conc.	initial Co-contam. conc.	initial perman conc.
Control-TSF05 groundwater	NA	NA	100ppm	10 ppm	0
Control-TSF05 groundwater					0
TSF05 gw					1
TSF05 gw					1
TSF05 gw					3
TSF05 gw					3

Tedlar Bag 60.28
Tedlar bag+gw 3050.0
gw. vol. 2990.00

X 177440
50 4994

contaminant volumes added:
TCE – 0.2 ml
1,1 DCE – 25 µl
cis 1,2-DCE – 25 µl
trans 1,2 DCE – 25 µl

flask#	wt. empty	wt. + g.w.	g.w. vol.	$KMnO_4$ added
1	245.938	735.42	489.49	—
2	252.232	737.30	485.07	—
3	238.086	720.72	482.63	6.604
4	255.829	746.45	490.72	6.609
5	239.650	716.70	477.05	20.002
6	246.840	728.36	481.52	20.003

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K. McCracken 8/31/98
Recorded by Date

Read and understood by Date 9/1/98

Subject Exp. 4b-R6

Date 09 (Month) 01 (Day) 19 98 (Year)

volume flask#	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	7.10/6.97	7.04	7.09	7.03	7.10	7.11	6.96
2	7.22	6.98/6.99	7.13	7.07	7.07	7.18	7.12
3	7.21	7.10	6.71/7.16	6.64	6.57	6.43	6.77
4	7.00	6.46	7.33	6.97/7.12	7.08	6.91	7.12
5	7.05	7.01	7.05	6.95	6.76/7.18	6.65	6.75
6	7.11	6.99	7.46	7.19	7.19	7.09/7.20	7.12/7.21

pH flask#	T0	T30min.	T1.5hr.	T3hr	T5hr	T20hr	T24hr
1	8.42/8.42	8.43	8.37	8.37	8.37	8.42	8.40
2	8.42	8.42/8.42	8.39	8.39	8.38	8.42	8.40
3	8.43	7.40	7.44/7.29	7.47	7.48	7.38	7.34
4	8.43	7.16	7.21	7.30/7.23	7.33	7.24	7.23
5	8.42	7.10	7.26	7.30	7.32/7.29	7.27	7.26
6	8.43	7.16	7.32	7.38	7.36	7.62/7.60	7.62/7.60

spec. Readings DF: 1,2=1 ; 3&4=500 ; 5&6=1000

Flask#	T0	T30min.	T1.5hr	T3hr	T5hr	T20hr	T24hr
1	0.003/0.003	0.002	0.002	0.004	0.003	0.003	0.004
2	0.002	0.009/0.003	0.002	0.003	0.003	0.002	0.005
3	0.001	0.195	0.443/0.453	0.462	0.451	0.449	0.447
4	0.001	0.217	0.439	0.457/0.463	0.435	0.452	0.451
5	0.003	0.240	0.487	0.553	0.547/0.579	0.591	0.540
6	0.002	0.323	0.440	0.559	0.592	0.573/0.569	0.557/0.552

spec. stds.	T0	T30min	T1.5hr	T3hr	T5hr	T20hr	T24hr
Blank	0.000	0.000	0.000	0.000	0.000	0.000	0.000
50mg/L	1.120	1.120	1.120	1.115	1.117	1.114	1.114
10mg/L	0.222	0.222	0.222	0.225	0.225	0.224	0.223

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K McCracken — Recorded by — 09/01/98 Date

SRC — Read and understood by — 9/14/98 Date

Subject Exp. 4b. R6

Date 09 (Month) 01 (Day) 19 98 (Year)

Sample time	GC#	wt. empty	wt. + hexane	wt. + sample	Sample ID	DF
11:10	1924	11.001	13.957	18.800	4b-R6-TBa	100
11:10	1925	10.893	13.929	19.210	4b-R6-TBb	
11:14	1926	11.047	12.835	15.943	4b-R6-Tφ-1a	
11:15	1927	10.963	12.794	16.174	1b	
11:16	1928	11.055	12.918	16.228	2	
11:17	1929	11.110	12.962	16.416	3	
11:18	1930	11.119	13.000	16.301	4	
11:19	1931	11.120	12.982	16.128	5	
11:20	1932	11.130	12.939	16.267	6	
11:40	1933	11.200	13.148	16.265	4b-R6-T30min-1	
11:41	1934	11.140	12.951	16.275	2a	
11:42	1935	10.927	12.788	16.012	2b	100
11:43	1936	11.005	12.781	16.087	3	1
11:44	1937	11.042	12.904	16.590	4	1
11:45	1938	11.001	13.011	16.745	5	1
11:46	1939	11.037	12.932	16.532	6	1
12:37	1940	10.983	12.848	15.972	4b-R6-T1.5h-1	100
12:38	1941	11.206	13.051	16.200	2	100
12:39	1942	11.152	12.956	16.100	3a	1
12:40	1943	11.180	13.059	16.198	3b	1
12:41	1944	11.071	12.886	15.805	4	1
12:42	1945	11.097	12.916	15.887	5	1
12:43	1946	11.113	12.962	16.046	6	1
14:00	1947	11.028	12.881	15.741	4b-R6-T3h-1	100
14:01	1948	10.982	12.892	15.941	2	100
14:02	1949	10.966	12.869	16.043	3	1
14:03	1950	11.002	12.906	15.962	4a	1
14:04	1951	10.925	12.931	16.025	4b	1
14:05	1952	10.883	12.760	15.756	5	1
14:06	1953	11.103	13.000	16.208	6	1
16:07	1954	11.009	12.880	15.778	4b-R6-T5h-1	100
16:07	1955	11.070	12.937	15.923	2	100
16:09	1956	11.173	13.055	16.272	3	1

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K. McCracken — Recorded by

09-01-98 — Date

SL C — Read and understood by

9/14/98 — Date

Subject Exp. 4b-R6

Date 09 (Month) 01 (Day) 1998 (Year)

Sample Time	GC#	wt. empty	wt. thermo	wt. + sample	Sample ID	DF
16:10	1957	11.159	13.003	16.182	4b-R6-T5h-4	1
16:11	1958	11.000	12.781	16.018	5a	1
16:12	1959	11.015	12.790	15.798	5b	1
16:13	1960	10.976	12.892	16.057	6	1
9/2 09:15	1961	11.012	12.952	15.906	4b-R6-T20h-1	100
09:16	1962	10.982	12.907	15.827	2	100
09:17	1963	11.005	12.939	16.146	3	1
09:18	1964	10.919	12.850	16.034	4	1
09:19	1965	11.053	12.966	16.103	5	1
09:20	1966	11.154	13.088	16.235	6a	1
09:21	1967	11.217	13.128	16.216	6b	1
11:04	1968	11.121	13.006	16.099	4b-R6-T24h-1	100
11:05	1969	11.192	13.111	16.351	2	100
11:06	1970	11.187	13.106	16.240	3	1
11:07	1971	11.000	12.879	16.052	4	↓
11:08	1972	11.019	12.973	16.187	5	↓
11:09	1973	11.031	12.989	16.346	6a	↓
11:10	1974	11.180	13.144	16.458	6b	↓
11:12	1975	10.942	12.897	12.985	4b-R6-char-1a	↓
11:12	1976	10.968	12.922	12.965	1b	↓
11:14	1977	11.139	13.095	13.175	2a	↓
11:14	1978	11.163	13.104	12.146	2b	↓
11:15	1979	11.205	13.151	13.240	3a	↓
11:15	1980	11.057	13.011	13.054	3b	↓
11:16	1981	11.257	13.208	13.291	4a	↓
11:16	1982	11.026	13.023	13.063	4b	↓
11:17	1983	11.039	12.967	13.059	5a	↓
11:17	1984	11.066	13.013	13.052	5b	↓
11:18	1985	10.962	12.926	13.017	6a	↓
11:18	1986	10.891	12.854	12.903	6b	1

Continued on page 124

K McCracken — Recorded by — 09-01-98 Date

SP Cl — Read and understood by — 9/14/98 Date

Subject Exp. 4b - Rb

Date 09 (Month) 02 (Day) 19 98 (Year)

Additional Dilutions

GC#	DF
1924b	20
1925b	↓
1926b	↓
1927b	↓
1928b	↓
1929b	↓
1930b	↓
1931b	↓
1932b	↓
1933b	↓
1934b	↓
1935b	↓
1940b	↓
1941b	↓
1947b	↓
1948b	↓
1954b	↓
1955b	↓
1961b	↓
1962b	↓
1968b	↓
1969b	20

Continued on page

KM Gracken — Recorded by — 9/1/98 — Date

SRC — Read and understood by — 9-14-98 — Date

Subject Matrix Spike Study

Date 09 (Month) 08 (Day) 19 98 (Year)

vial #	start wt. empty	wt. + media	wt. + H_2O	Sample I.D.	spiking time	end empty wt.
1	24.996	—	67.742	DI water	14:00	24.990
2	25.232	—	68.657	DI water	14:00	25.242
3	25.224	—	68.128	TAN-40 GW	14:01	25.399
4	25.203	—	68.003	TAN-40 G.W	14:02	25.417
5	24.852	—	67.446	TSF-05 GW	14:02	24.839
6	25.065	—	67.600	TSF-05 GW	14:03	25.030
7	25.208	35.464	75.017	aggregate basalt	14:05	25.201
8	24.995	35.044	74.591	aggregate basalt	14:05	24.994
9	25.017	35.036	74.527	crushed basalt	14:06	25.050
10	25.099	35.080	74.523	crushed basalt	14:06	25.094
11	25.106	35.086	73.260	sediment	14:07	25.102
12	25.225	35.245	73.042	sediment	14:07	25.237

jar #	wt empty	wt. + hexane	wt. + sample	sample time
1	121.963	148.932	191.696	14:02
2	121.759	148.161	191.555	14:02
3	121.647	148.135	190.761	14:03
4	121.461	148.105	190.574	14:03
5	121.239	148.163	190.562	14:03
6	124.713	151.488	193.932	14:04
7	121.324	148.287	197.973	14:04
8	121.178	148.240	197.715	14:05
9	121.659	148.605	197.833	14:05
10	121.718	148.266	197.383	14:06
11	121.582	149.367	197.341	14:06
12	121.368	149.407	195.903	14:07

Continued on page 127

K. McCracken — Recorded by — Date 9/8/98

Read and understood by — Date 9/14/98

Subject Matrix Spike Study

Date 09 Month 09 Day 19 98 Year

GC#	DF	Sample ID
1987	1	distilled H_2O control
1988	↓	distilled H_2O control
1989	↓	TAN-40 GW
1990	↓	TAN-40 GW
1991	↓	TSF-05 GW
1992	↓	TSF-05 GW
1993	↓	d.i. H_2O + aggregate basalt
1994	↓	d.i. H_2O + aggregate basalt
1995	↓	d.i. H_2O + crushed basalt
1996	↓	d.i. H_2O + crushed basalt
1997	↓	d.i. H_2O + sediment
1998	↓	d.i. H_2O + sediment

run w/
2 cal. checks:
1 = 10ppb - 4/22/98
2 = 500ppb - 8-24-98

Continued on page

K McCracken 9/8/98
Recorded by Date

SRC 9/17/98
Read and understood by Date

Subject 082698 Cal. Curve Information

Date 8/26 1998
Month Day Year

New Calibration Stds Preparation. 8-26-98 SR Cline

Supelco Custom Mix ~~Lot#~~ Quote 20021702 SRC 9/14 Lot # LA-77488

syringe full = 18.9070g syringe Empty 18.5654g

10PPm "stock" solution: added 0.5176mL of Custom Mix (Assuming sg of Custom Mix = 0.66 since hexane is the solvent) to 10mL Flask (Volumetric) + filled to volume

SRC 9/14/98

Resulting Stock Conc's

TCE (200.3)(.5176mL) = (x)(10mL)
x = 10.37 mg/L

Similarly
VC = 10.36 mg/L
Cis 1,2 DCE = 10.36 mg/L
Trans 1,2 DCE = 10.37 mg/L
1,1 DCE = 10.35 mg/L

STD. Prep

STD	Tare wt(g)	Tare + Hex(g)	Vol hex(mL)	Volume "stock" added	TCE (ppb)
1	10.9409	17.2048	9.99	5mL	5.5ppb
2	11.1277	17.9669	9.60	10mL	10.8
3	11.1205	17.0613	9.00	50mL	57.3
4	11.1709	17.3177	9.31	100mL	110.2
5	11.1232	17.0905	9.03	500mL	544.1
6	11.0620	17.0495	9.07	1mL	1029.8

Continued on page 129

SRC 9/14/98
Recorded by Date

Susan L. Carroll 9/15/98
Read and understood by Date

Subject Cal Curve Info Continued

Date 8/26 Month Day 1998 Year

SRC 9/14/98

Certificate of Composition

DESCRIPTION: OAKRIDGE NAT'L LABS

QUOTE 20024702 LOT NO.: LA-77488 MFG DATE: Jul-1998

SOLVENT: HEXANE

ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
CIS-1,2-DICHLOROETHYLENE	156-59-2	99.0	200.1 +/- 1.00	LA72501
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.0	200.2 +/- 1.00	LA54963
TRICHLOROETHYLENE	79-01-6	99.0	200.3 +/- 1.00	LA53375
VINYL CHLORIDE	75-01-4	99.0 (a)	200.2 +/- 1.00	LA77423
1,1-DICHLOROETHYLENE	75-35-4	99.0	199.9 +/- 1.00	LA74470

Recorded by SRC Date 9/14/98

Read and understood by Susan L. Carroll Date 9/15/98

Continued on page

Laboratory Evaluation of In Situ Chemical Oxidation as a Technology for Groundwater Remediation at the INEEL TAN Site

Laboratory Notebook

ORNL-CASD

3/9/98 Start of project notebook.
Performed density measurements on two sludge portions with MiniDens analyzer. Results filed with Daily Sample activity log.

3/19/98 Dried 2 aliquots of wet sludge for percent moisture determination. Results filed with Daily Sample activity log.

3/20/98 Performed TOC analysis of wet and dried aliquots of sludges. Results filed with Daily Sample activity log.

3/24/98 Dried a larger aliquot of sludge for % moisture determination. This dried sample will be used as the baseline for the tests. Results filed with Daily Sample Activity log

4/2/98 Digested 0.5003g of dried sludge for radiochemical analysis. Microwave digestion data sheet filed with Daily Sample Activity Log.

4/7/98 De-gassed one 4 Liter container of TSFOS GW with Helium gas for 30 mins. Prepared a 100 ppm TCE spiking solution using 356.7g groundwater and 24.4 µl of pure phase TCE (density 1.46 g/ml). Placed spiking solution in 4°C refrigerator to equilibrate overnight.

TCE conc = (Vol (ml)) × (Density (g/ml)) / Groundwater weight (g)

.0244 ml × 1.46 g/ml / 356.7 g

3.56E+4 µg / 356.7 g

99.9 µg/g

4/8/98 Measured 5ml of TCE spiking solution into 5ml of Hexane. Repeated with duplicate sample. Aliquots were shaken at 300 rpm for 2 hours. After extraction 0.01mL of the extracts were put into 0.99 mL Hexane for a 100 fold dilution. These samples will be analyzed by ESD to determine the initial TCE concentration.

4/8/98 (cont.) Approximate TCE concentration in dilutions should be 1 ppm.
Prepared solutions for Task 2-b3 using sparged groundwater, dried sludge, and TCE spiking solution. Weight + time details recorded in Excel workbook "ISCO study ~~Test~~ Electronic Log".

4/9/98 Performed Task 2-b3 extraction tests. Weight + time details recorded in Excel workbook "ISCO study Electronic Log".

4/10/98 Completed Task 2-b3 extraction tests.

4/13/98 Hexane extractions transferred to Steve Cline of ESD.

4/14/98 Additional dilutions made on specified extracts to get within the ESD's GC calibration. Vials + specifics documented in the electronic log.

4/21/98 DILUTED TCE SPIKING SOLUTION ~~[illegible]~~ 1 DOP (100X)
D.J. Z. 4-21-98

4/29/98 DRIED 2 1 LITER JARS FOR EXPERIMENTS.
D.J. Z. 4-29-98

5/4/98 Dried 2 1 liter jars for experiments. Also weighed out 8 100 gm aliquots of dried sludge into 500 ml erlenmeyer flasks for Task 3-3. Covered with parafilm. Details in electronic log.

5/5/98 Sparged 1 gallon of groundwater with He gas for 15 mins. Performed Task 3-3 runs 1 and 2 (up to the four hour measurements). Details in electronic log.

5/6/98 Completed Task 3-3 runs 1 + 2. Removed portions of post reaction sludges to dry at 110°C. Details in electronic log.

5/7/98 Sparged 1 gallon of groundwater with He gas for 15 mins. Performed Task 3-1 runs 3 and 4 (up to four hour measurements). Details in electronic log.

5-8-98 COMPLETED RUN 3 & 4 TASK 3B. PLACED (#4) 1% + (#1+#2) 3% SLURRIES BACK IN INCUBATOR UNTIL 5-11-98. DETAILS IN ELECTRONIC LOG.

[illegible] 5-8-98

5-11-98 REMOVED #4 (1%) + #1+#2 (3%) FROM INCUBATOR & ANALYZED FOR pH + MnO4. DETAILS IN ELECTRONIC LOG.

[illegible] 5-11-98

5-13-98 ANALYZED DRY SLUDGE & INFEEL TASK 3B REMAINING SLUDGES FOR TOTAL CARBON, INORGANIC CARBON & ORGANIC CARBON. RESULTS FILED WITH TRACKING LOG.

[illegible] 5-13-98

5-26-98 DRIED 2 1 LITER JARS OF SLUDGE FOR TASK 4b EXP. PREPARED 1,100 ppm TCE SATURATED SPIKE SOLUTION 0.7ml TCE/500ml GROUND WATER. PREPARED 10 ppm TCE SPK SOLUTION USING SPARGED TSF OF GROUND WATER. SEE ELECTRONIC LOG FOR DETAILS. SPARGED 1 GALLON OF GROUND WATER W/ HE GAS FOR 15 min.

[illegible] 5-26-98

5/27/98 Extracted duplicate portions of TCE spike solution prepared 5/26/98 to establish baseline TCE concentration for Task 4b run 10. Added a 4:1 ratio of spiking solution to dried sludge into reactors. Placed slurries + two control solutions into incubator set at 12°C and shaking at 200 rpm for a 24 hr. equilibration period. Details in electronic log.

5/28/98 Commenced Task 4b run 10. Weight + time details in Excel workbook "ISCO Study Electronic Log."

5/29/98 Completed Task 4b run 10. Weight + time details in "ISCO Study Electronic Log". Delivered GC extracts to ESD for analysis.

6-1-98 PREPED 10ppm SPK TCE SOLUTION USING SPARGED TSF05 + 1100 SPK GROUND WATER. SEE ELECTRONIC LOG FOR DETAILS.
dt 2 dt 6-1-98

6-2-98 EXTRACTED DUPLICATE PORTIONS OF 12°C STABILIZED 10ppm SPK FOR BASELINE ANALYSIS. ADDED 4:1 RATIO ~~SPARGED~~ 10ppm SPK SOLUTION TO DRY SLUDGE IN REACTORS. PLACED SLURRIES, INCLUDING BASELINE, SLUDGE, CONTROL + TO GROUND WATER CONTROLS INTO 12°C INCUBATOR + LET EQUILIBRATE FOR 24 HOURS. DETAILS IN ELECTRONIC LOG
dt 2 dt 6-2-98

6-3-98 COMMENCED TASK 4b RUN 9. WEIGHT + TIMES IN ELECTRONIC LOG WORKBOOK "ISCO STUDY ELECTRONIC LOG."
dt 2. dt 6-3-98

6-4-98 COMPLETED TASK 4b RUN 9. DETAILS IN ELECTRONIC LOG WORKBOOK "ISCO STUDY ELECTRONIC LOG". DELIVERED GC VIALS TO ESD FOR ANALYSIS.
dt 2 dt 6-4-98

6-8-98 PREPED 100 ppm TCE SPK 227.5g / 1100ppm SPK 2272.7g. SEE ELECTRONIC LOG FOR DETAILS. SPARGED 1 GALLON OF GROUND WATER w/ He FOR 15 min.
dt 2 dt 6-8-98

6-9-98 DUE TO LEAK IN PREP. BAG/CONTAINER, NEW 100ppm TCE SPK PREPED w/ 2272.7g SPARGED GROUND WATER + 227.3 ml 1100 TCE STD. SEE ELECTRONIC LOG FOR DETAILS. SPARGED 1 GALLON GROUND WATER w/ He FOR 15 min.
dt 2 dt 6-9-98

6-10-98 EXTRACTED DUP. PORTIONS OF 12°C STABILIZED 100ppm SPK FOR BASELINE ANALYSIS. ADDED 4:1 RATIO 100ppm SOLUTION TO DRY SLUDGE IN REACTORS. PLACED SLURRIES INTO 12°C INCUBATOR FOR EQUILIBRATION TIME OF 24 HRS. DETAILS IN ELECTRONIC LOG
dt 2. dt 6-10-98

6-11-98 COMMENCED TASK 4b RUN 11. DETAILS IN ELECTRONIC LOG WORKBOOK "ISCO STUDY ELECTRONIC LOG."
dt 2 dt 6-11-98

6-12-98 COMPLETED TASK 4b RUN 11. DETAILS IN ELECTRONIC LOG. DELIVERED GC VIALS TO ESD FOR ANALYSIS
dt 2. dt 6-12-98

6-15-98 PREPARED 100 ppm TCE SPK USING 2222.2 g SPARGED GROUND WATER + 277.8 ml SAT. TCE SOLUTION. ANALYZED CONCENTRATION TO BE 60 ppm TCE. ADDED 375 ml MORE OF SAT. TCE SOLUTION + LET EQUILIBRATE AT 12°C OVERNIGHT. SEE ELECTRONIC LOG FOR DETAILS.

dt Z. dt 6-15-98

6-16-98 EXTRACTED DUPLICATE POTIONS OF 12°C STABILIZED 100 ppm SPK, ANALYSIS FOUND TO BE 110.3 + 98.5 ppm TCE RESPECTIVELY. ADDED 4:1 RATIO 100 ppm SPIKING SOLUTION TO DPM SLUDGE IN REACTORS. PLACED SLURRIES INTO 12°C INCUBATOR FOR EQUILIBRATION TIME OF 24 HRS, DETAILS IN ELECTRONIC LOG.

dt Z. dt 6-16-98

6-17-98 COMMENCED TASK 4b RUN 12 DETAILS IN ELECTRONIC LOG WORKBOOK "ISCO STUDY ELECTRONIC LOG."

dt Z. dt 6-17-98

6-18-98 COMPLETED TASK 4b RUN 12. DETAILS IN ELECTRONIC LOG WORKBOOK "ISCO STUDY ELECTRONIC LOG." GC VIALS TO ESD.

dt Z. dt 6-18-98

6-19-98 RE-DILUTIONS PERFORMED ON SELECT EXTRACTS

dt Z. dt 6-19-98

6/19/98 Run 10 and 11 of Task 4b must be redone due to low TCE concentrations in spike solution. New run numbers will be Run 15 and 16 respectively. The run 11 data will be used as the run 9 data (TCE concentration was ~40 ppm) which also had low TCE concentration in the spiking solution (~1 ppm vs 10 ppm).

6-22-98 PREPARED 10 ppm TCE SPK USING 2460 g SPARGED GROUND WATER + 40 ml SAT. TCE SPIKING SOLUTION. ANALYZED CONCENTRATION FOUND TO BE 8.8 ppm TCE. SEE ELECTRONIC LOG FOR DETAILS.

dt Z. dt 6-22-98

6-23-98 EXTRACTED DUPLICATE ALIQUOTS OF 12°C STABILIZED 10 ppm SPK. ADDED 4:1 RATIO SPIKING SOLUTION TO DPM SLUDGE IN REACTORS. PLACED SLURRIES INTO 12°C INCUBATOR FOR 24 HR EQUILIBRATION. SEE ELECTRONIC LOG.

dt Z. dt 6-23-98

6-24-98 REACTION VESSEL 4b-R15-4 HAD CRACK. SLURRY TRANSFERRED TO NEW REACTION VESSEL WITHOUT STOPCOCK ASSEMBLY + NO ORBOTUBE. COMMENCED TASK 4b-R15, DETAILS IN ELECTRONIC LOG WORKBOOK 'ISCO STUDY ELECTRONIC LOG'

dt 2 dt 6-24-98

6-25-98 COMPLETED TASK 4b-R15. SEE ELECTRONIC LOG.

dt 2 dt 6-25-98

6-29-98 REDILUTIONS PERFORMED ON #'s 901-904 (10X).

dt 2. dt 6-29-98

7-6-98 SPARGED 1 GALLON OF GROUND WATER w/ the FOR 15 MIN.

dt 2. dt 7-6-98

7-6-98 PREPARED 100 ppm TCE SPK USING 2100 g SPARGED GROUND WATER AND 400 ml SATURATED SPIKING SOLUTION. CONCENTRATION FOUND TO BE 80 ppm BY ANALYSIS. SEE ELECTRONIC LOG

dt 2 dt 7-6-98

7-7-98 SPIKING SOLUTION FROM 7-6-98 SPIKED WITH 25 ml PURE PHASE TCE. SEE ELECTRONIC LOG. PURGED IN 12°C INCUBATOR OVERNIGHT.

dt 2 dt 7-7-98

7-8-98 EXTRACTED DUPLICATE ALIQUOTS OF 12°C STABILIZED 7-7-98 SPIKE SOLUTION. ANALYZED & CONFIRMED 90.2 + 85.6 ppm TCE RESPECTIVELY. ADDED 4:1 RATIO SPIKING SOLUTION TO DRY SLUDGE IN REACTORS. PURGED SLURRIES INTO 12°C INCUBATOR FOR 24 HR EQUILIBRATION. SEE ELECTRONIC LOG.

dt 2 dt 7-8-98

7-9-98 COMMENCED TASK 4b-RUN16. SEE ELECTRONIC LOG FOR DETAILS. 'ISCO STUDY ELECTRONIC LOG'

dt 2. dt 7-9-98

7-10-98 COMPLETED TASK 4b-RUN16. SEE ELECTRONIC LOG FOR DETAILS.

dt 2 dt 7-10-98

7-13-98 DRIED 500 g SLUDGE (TSS-05).

dt 2 dt 7-13-98

7-14-98 SPARGED 1 GALLON OF GROUND WATER w/ the FOR 15 MIN.

dt 2 dt 7-14-98

7-14-98 ADDED 4:1 RATIO OF SPIKED GROUND WATER TO DRY SLUDGE + PURE PHASE TCE FOR TCE CONCENTRATIONS OF ≥2,000 ppm. PLACED SLURRIES INTO 12°C INCUBATOR FOR EQUILIBRATION PERIOD (24 HRS).
dt 2. dt 7-14-98

7-15-98 COMMENCED TASK 4b RUN 13 + 4b RUN 14. SEE ELECTRONIC LOG 'ISCO STUDY ELECTRONIC LOG 2' FOR DETAILS. 1% + 3% MnO_4^- PRODUCED SIGNIFICANT OFF-GAS DURING INITIAL PHASE OF STUDY.
dt 2. dt 7-15-98

7-16-98 PERFORMED 20 + 24 HR PULLS + PLACED REACTORS BACK INTO 12°C INCUBATOR.
dt 2. dt 7-16-98

7-20-98 COMPLETED TASK 4b RUN 13 + 4b-RUN 14. SEE ELECTRONIC LOG FOR DETAILS. REDILUTIONS FOR 4b-RUN 14.
dt 2 dt 7-20-98

7-29-98 SPARGED 1 GALLON OF GROUND WATER w/ He FOR 15 MIN.
dt 2 dt 7-29-98

7-29-98 PREPPED 100 ppm TCE SPIKING SOLUTION FOR TASK 4b-RUN 3 + TASK 4b - RUN 4 USING 2500 g GROUND WATER IN EACH + 280 uL PURE PHASE TCE IN EACH. PLACED BOTH 2500 ml TEDLAR BAGS IN INCUBATOR AT 12°C OVERNIGHT. BOD
dt 2. dt 7-29-98

7-30-98 COMMENCED TASK 4b-RUN 3 + RUN 4. SEE ELECTRON LOG 'ISCO STUDY ELECTRONIC LOG 2' FOR DETAILS.
dt 2 dt 7-30-98

7-31-98 PERFORMED 20 + 24 HR PULLS + PLACED REACTORS BACK INTO 12° INCUBATOR OVER WEEKEND.
dt 2 dt 7-31-98

8-3-98 COMPLETED TASK 4b-RUN 3 + RUN 4. SEE ELECTRONIC LOG FOR DETAILS. REDILUTIONS PERFORMED ON 4b-RUN 3.
dt 2 dt 8-3-98

8-12-98 SPARGED 2 GALLONS OF GROUND WATER w/ He FOR 15 MIN. ADDED GROUND WATER TO REACTION FLASKS WITH PURE PHASE TCE FOR TCE CONCENTRATIONS OF ≥ 2,000 ppm. PLACED REACTORS INTO 12°C INCUBATOR FOR EQUILIBRATION PERIOD (24 HRS)
dt 2. dt 8-12-98

8-13-98 COMMENCED TASK 4b-RUN 7 + RUN 8 SEE ELECTRONIC LOG, 'ISCO STUDY ELECTRONIC LOG 2' FOR DETAILS. I_2 + $3O_2$ MnO_4^- PRODUCE SIGNIFICANT OFF-GAS DURING INITIAL PHASE OF STUDY.
TASK 4b-RUN 8-3 DEVELOPED CRACK. CONTENTS TRANSFERRED TO EXTRA FLASK. dt 2 dt 8-13-98

8-14-98 PERFORMED 20 + 24 HR PULLS + PLACED FLASKS BACK INTO INCUBATOR. DILUTIONS PLACED INTO REFRIGERATOR UNTIL 8-17-98. dt 2 dt 8-14-98

8-17-98 COMPLETED TASK 4b-RUN 7 + RUN 8. SEE ELECTRONIC LOG FOR DETAILS.
dt 2. dt 8-17-98

9-14-98 PREPARED SAMPLES FOR TCE EXTRACTION STUDY BY ADDITION OF ~10g DRY SLUDGE, .04ml SAT. TCE SOLUTION + GROUND WATER TO EQUILIBRATION VIALS. SEE ELECTRONIC LOG 'ISCO STUDY ELECTRONIC LOG 2' FOR DETAILS.
dt 2 dt 9-14-98

9-15-98 COMMENCED EXTRACTION OF TCE FROM EQUILIBRATED SAMPLES WITH DIFFERENT TIME PULLS FOR GC ANALYSIS. *NOTE* SAMP #1 LOST DUE TO BOTTLE CAP BREAKING. SEE 'ISCO STUDY ELECTRO LOG 2' FOR DETAILS.
dt 2 dt 9-15-98

9-16-98 PERFORMED 20 HR + 24 HR PULLS ON TCE EXTRACTION STUDY SEE 'ISCO STUDY ELECTRONIC LOG 2' FOR DETAILS.
dt 2 dt 9-16-9

2

4-30-97	CALIB. 7.00 # 139506	[illegible]
	↓ 10.00 # 107503	
	CK 570 12.45 # 9608149	12.46
	W-23 SLURRY ①	12.87
	↓ ②	12.92
	DUP	12.97
	CK 570 12.45 # 9608149	12.52 dt 2 dt 4-30-97
5-4-97	CALIB. 7.00 # 139506	
	↓ 10.00 # 107503	
	CK 570 12.45 # 9608149	12.47
	5-4-97 W-23 SLURRY	12.62
	TAP 8454 DUP	12.64
	CK 570 12.45 # 9608149	12.56 dt 2 dt 5-4-97
5-5-97	CALIB. 7.00 # 139503	
	↓ 10.00 # 107503	
	CK 570 7.00 135506	2.00 dt 2 dt 5-5-97
5-6-97	CALIB 2.00 # 139506	
	10.00 # 107503	
	CK 7.00 # 135506	7.02 dt 2 dt 5-6-97
5-7-97	CALIB 7.00 # 139506	
	↓ 10.00 # 107503	
	CK 2.00 # 135506	7.00
	CK 12.45 # 9601149	12.55 dt 2 dt 5-7-97
	TAP 8457 9605097-09 =	9.58
	↓ DUP	9.80
5-8-97	CALIB. 7.00 # 139506	
	10.00 # 132503	
	CK 10.00 # 107503	9.98 dt 2 dt 5-8
5-11-97	CALIB. 7.00 # 139506	
	10.00 # 132503	
	CK 10.00 # 107503	10.07 dt 2 dt 5-11

-4-98 CAL.D. 4.00 # L16503 — CAL.B
7.00 # L39506 — ϕ
CK 4.00 # L4508 4.01
TPA 98658 980505-160 6.41
-160 DUP 6.42
CK 4.00 # L11508 4.01

Ok 2 dt 5-11-98

5-20-98 CAL.B. 4.00 # L16503 — CAL.B
ϕ 7.00 # L39506 — ϕ
CK STD 4.00 # L11508 3.99 ✓
TCLP #1 4.92
TCLP #2 2.85
CK STD 2.00 # 2.02 ✓

Ok 2. dt 5-20-98

5/21/98 Sample pH Test For TCLP # 72
Resin Cation 7.37
Resin Anion 7.67
Added appropriate volume of 1N HCL To each & stirred vigorously at 50°C for 10min. Cooled. pH:

	paper	meter
Cation Resin	<0.0	1.56
Anion Resin	<0.0	2.01

5-22-98 CAL.B. 4.00 # L11508 —
7.00 # 39506 —
CK STD 4.00 # L16503 4.02
TPA 98665 TCLP EF. #1 BIK 4.96
980522-01 CAT. EX. 3.38
980522-02 AN. EX. 5.85
CK STD 4.00 # L16503 4.02

Ok 2 dt 5-22-98

5-28-98 CAL.B. 7.00 # L39506 —
10.00 # L32503 —
CK STD 7.00 # 9801288 7.00
PEPSI BOTTLE EXP.
CK STD 7.00 # 9801288 7.00

Ok 2. dt 5-28-98

5-29-98	CALIB.	7.00 # L39506	—	
	↓	10.00 # L32503	—	
	CCS STD	7.00 # 9801288	7.00	
	INTER VARIOUS SAMPLES			
	CCS STD	7.00 # 9801288	7.00	
				dt 2 dt 5-29-98
6-1-98	CALIB	7.00 # L39506	—	
	↓	10.00 # L32503	—	
	CCS STD	12.45 # 9601149	12.39 ✓	
	IPA 4468	980525-032	12.35	
		-033	12.90	
		-034	12.94	
		-035	12.31	
		-036	12.46	
		-037	12.35	
	IPA 4470	980529-029 ~~-027~~ 3D	10.37	
	↓	-029 DUP	10.38	
	CCS STD	# 9601149	12.58	
				dt 2 dt 6-1-98
6-3-98	CALIB.	7.00 # L39506	—	
	↓	10.00 # L32503	—	
	CCS STD	7.00 # 9801288	7.00	
	INTER VARIOS SAMPLES			
	CCS STD	7.00 # 9801288	7.00	
				dt 2 dt 6-3-98
6-11-98	CALIB.	7.00 # 9801288	—	
	↓	10.00 # L32503	—	
	CCS STD	10.00 # 9801098	10.01	
	INTER VARIOS SAMPLES			
	CCS STD	10.00 # 9801098	10.02	
				dt 2 dt 6-11-98
6-12-98	CALIB	7.00 # 9801288	—	
	↓	10.00 # L32503	—	
	CCS STD	10.00 # 9801098	9.98	
	↓ (INTER VARIOUS SAMPLES)			
	↓	10.00 # 9801098	10.03	
				dt 2 dt 6-12-98

6-15-98 CALIB. 4.00 # L11509 —
7.00 # 9801299 —
10.00 # L32503 —
STOCK 4.00 # L16503 4.00
STOCK 10.00 # 9801098 10.02 AFTER 1ml ADD
980615-011 8.37 1.54
-012 8.48 1.52
-013 8.18 1.49
~~-013 DUPLICATE~~
CKSTD 4.00 # L16503 4.01

Dil 2. dt 6-15-98

6-16-98 CALIB 4.00 # L11509 —
7.00 # 9801299 —
CKSTD 4.00 # L16503 4.00
TCLP Extract 980615-011 4.93
EXTRACTS -012 5.10
-013 4.93
BLK 4.92
CKSTD 4.00 # L16503 4.00

Dil 2. dt 6-16-98

6-17-98 CALIB. 7.00 # 9801299 —
10.00 # L32503 —
CKSTD 10.00 # 9801098 10.00
(INTER VARIOUS SAMPLES)
CKSTD 10.00 # 9801098 10.03

Dil 2 dt 6-17-98

6-18-98 CALIB 7.00 # 9801299 —
10.00 # L32503 —
CKSTD 10.00 # 9801098 10.02
(INTER VARIOUS SAMPLES)
CKSTD 10.00 # 9801098 10.03

Dil 2. dt 6-18-98

6-24-98 CALIB 7.00 # 9801299 —
10.00 # L32503 —
CKSTD 10.00 # 9801098 10.05
(INTER SAMPLES)
CKSTD 10.00 # 9801098 10.02

Dil 2 dt 6-24-98

6-25-98 CAMB 7.00 # 9801288
↓ 10.00 # L32503
CKSTD 10.00 9801098 10.06
(INTER SAMPLES) (TASK 4b RUN 15)
CKSTD 10.00 # 9801098 ~~10.05~~ 9.96
DD

dl 2 dt 6-25-98

6-26-98 CAMB 7.00 # 9801288
↓ 10.00 # L32503
CKSTD 10.00 # 9801098 10.01
980625-011 12.90
-012 12.93
-013 12.99
-014 12.76
-015 12.81
-016 12.77
-016DUP 12.82
CKSTD 10.00 # 9801098 10.05

dl 2 dt 6-26-98

7-6-98 CAMB 7.00 # 9812286
↓ 10.00 # L32503
CKSTD 10.00 # 9801098 10.06
TASK 4b 55 980701-014 10.38
↓ DUP 10.44
CKSTD 10.00 # 9820098 10.10
CKSTD 12.45 # 9608149 12.59

dl 2 dt 7-6-98

7-7-98 TCLP CAMB 4.00 # L16503
↓ 7.00 # ~~L11503~~ 9801288
CKSTD 4.00 # L11503 4.01
TASK 4b 56 980707-010 ~~2.20~~ 2.20 1.017g/19.6 ml H_2O
DUP 2.13
CKSTD 4.00 # L11503 3.98

dl 2 dt 7-7-98

7-8-98 CAMB 4.00 # L16503
↓ 7.00 # 9802288
CKSTD 4.00 # L11503 4.00
EPCSTD 980707-010 4.00 # L11503 2.62 4.00

dl 2 dt 7-8-98

7-9-98 CRMB 7.00 # 9801288 —
↓ 10.00 # L32503 —
CRSTD 10.00 # 9801098 10.09
(INFEED SAMPLES) (RUN 16) (TASK 46)
CRSTD 10.00 # 9801098 10.09
dt 2 dt 7-9-98

7-10-98 CRMB 7.00 # 9801288
↓ 10.00 # L32503
CRSTD 10.00 # 9801098 10.05
(INFEED SAMPLES, TASK 46 RUN 16)
CRSTD 10.00 # 9801098 10.00
dt 2 dt 7-10-98

7-15-98 CRMB 7.00 # 9801288
↓ 10.00 # L32503
CRSTD 10.00 # 9801098 10.06 ✓
TOL8960 9801015-006 10.41
↓ ↓ -007 10.48
↓ ↓ -007DD 10.48/007
(INFEED SAMPLES 2,000 TCE)
CRSTD 10.00 # 9801098 10.00
dt 2 dt 7-15-98

7-16-98 CRMB 7.00 # 9801288
↓ 10.00 # L32503
CRSTD 10.00 # 9801098 10.07
(INFEED SAMPLES)
TCUP INFEED 9801015-037 11.46 PRE ACID
↓ -038 12.01 ↓
-039 11.92
-037 6.72 POST ACID
-038 9.23 ↓
-039 6.F. 5.30
CKSW ~~9801015-037~~ DD #2 2.87 ✓
↓ 10.00 # 9801098 9.99
dt 2 dt 7-16-98

7-17-98 CMB 4.00 L16503
7.00 M16500
CRSTD # 41508 4.00 4.04
TRIBAL 980715-037 5.92
TCLP -038 7.74
EXTRACTS -039 6.23 BIK = 2.92
CRSTD 4.00 # L1508 4.06
dt 2 dt 7-17-98

7-20-98 CMB 7.00 # M16500
10.00 # L32503
CRSTD 7.00 # 9801288 7.09 ✓
(INTER SAMPLES TOSTE 46 PAN #3 + 14)
CRSTD 7.00 # 9801288 7.08
dt 2 dt 7-20-98

7-30-98 CMB 7.00 # 9801281
10.00 # L32503
CRSTD 7.00 # M16500 7.02 ✓
INTER SAMPLES
CRSTD 7.00 # M16500 7.00
dt 2 dt 7-30-98

7-31-98 CMB 7.00 # M16500
10.00 # L32503
CRSTD 7.00 # 9801281 7.09 ✓
INTER SAMPLES
CRSTD 7.00 # 9801281 7.00
dt 2 dt 7-31-98

8-3-98 CMB 7.00 # M16500
10.00 # L32503
CRSTD 7.00 # 9801281 7.09
INTER SAMPLES
CRSTD 7.00 # 9801281 7.10
dt 2 dt 8-3-98

8-4-98 CMB 4.00 # L1508
7.00 # M16500
10.00 # L32503
CRSTD 4.00 # 9802286 4.06
CRSTD 4.00 # 9802286 4.05
QC1 TCLP 980730-012 16.6 dt 2 dt 8-4-98

8-5-98 CMB3 4.00 # L11508
↓ 7.00 # M16500
CMSTD 4.00 # 9702296 4.00
TCE 951 98073002 11.54
BIK 4.92
CMSTD 4.00 # 9702296 4.10

dil 2. dt 8-5-98

8-13-98 CMB3 7.00 # M16500
↓ 10.00 # L32503
CMSTD 7.00 # 9801288
INFEL SAMPLES
TASK 46 - RUN 7 ← RUN 8
CMSTD 7.00 # 9801288

dil 2 dt 8-13-98

8-14-98 CMB3 7.00 # M16500
↓ 10.00 # L32503
CMSTD 7.00 # 9801288
INFEL SAMPLES
CMSTD 7.00 # 9801288

dil 2. dt 8-14-98

8-17-98 CMB3 7.00 # M16500
↓ 10.00 # L32503
4.00 # L11508 4.00
CMSTD 7.00 # 9801288 7.08
CMSTD 4.00 # 9702296 4.02
INFEL SAMPLES
CMSTD 4.00 # 9702296 4.03
CMSTD 7.00 # 9801288 7.09

dil 2 dt 8-17-98

8-17-98 CMB 4.00 # L11508
7.00 # M16500
CMSTD 7.00 # 9801288 7.13

	INITIAL	.02M HCl 1:2 AFTER
TAK 9060 9808 14-019	11.2	7.45
(TCE) -020	12.1	9.38
-021	11.2	6.07

CMSTD 7.00 # 9801288 7.10

9-14-98 INITIAL TCE EXTRACTION STUDY [illegible] 9-14-98

	VIAL (wt)	VIAL + SLUDGE (wt)	VIAL + SLUDGE + .04 mTCE + GW (wt)	EMPTIED BTL
1	25.3293	35.5627	71.8603	SAMPLE LOST
2	25.4702	35.7450	72.7159	26.1015
3	25.3304	35.5617	72.9094	26.0843
4 control	25.5455	—	68.2439	25.7924

9-15-98 EXTRACTION

	E. BOTTLE	E. BTL + HEX (wt)	E. BTL + HEX + SAMPLE
1	138.1271	163.8834	SAMPLE LOST
2	124.4036	150.4791	197.0575
3	134.2925	160.3808	207.1500
4 control	138.1266	164.2141	206.6300

Radiochemistry Worksheet

Request:	See Below	Date:	6/2/98
Sample:	See Below	Analyst:	RDC
Customer:	Various	Badge #:	35993
ID:	Various	Procedure:	EPA-900.0

Gross Alpha Data

BKG	CTS	MIN	DET EFF.
PIC 1	2	10	49.9%

A	ANALIS	COUNT TIME (min)	COUNTS		CPM	Wt/Vol	Dil To:	2nd Dil	Dil to:	ALIQ.	DF	Result	Error	Units
	QC	10	8543		854					0.1	10	2.9E+2	0.1E+2	
57	980505-159	10	68		6.8	30 ML	50	5	10	1	3	7.3E-1	1.9E-1	BQ/ML
	Dup	10	62		6.2	30 ML	50	5	10	1	3	6.7E-1	1.8E-1	BQ/ML
	Spike	10	5858		585.8	30 ML	50	5	10	1	3	6.5E+1	0.2E+1	BQ/ML
58	980505-160	10	404		40.4	0.5588 G	50	5		1	89	1.2E+2	0.1E+2	BQ/G
	4M Liq	10	1213		121.3	1.004 G	5.7	1	10	1	57	2.3E+2	0.1E+2	BQ/G
	8M Liq	10	1070		107.0	1.058 G	6	1	10	1	57	2.0E+2	0.1E+2	BQ/G
	4M Solid	10	35		3.5	1.004 G	50	5	10	1	100	1.1E+1	0.4E+1	BQ/G
	8M Solid	10	18		1.8	1.058 G	50	5	10	1	95	5.1E+0	2.8E+0	BQ/G
59	980505-162	10	3083		308.3	30 ML	50	1	10	1	17	1.7E+2	0.1E+2	BQ/ML
EL	INEEL	10	7		0.7	0.5063 G	50	5	10	1	198	3.3E+0	4.0E+0	BQ/G
70	980529-029	10	1064		106.4	30 ML	50	5	10	1	3	1.2E+1	0.1E+1	BQ/ML

COPY

Original Filed With IPA 8857

trol D	Measured Value	Known Value	% Diff	Units	Instrument ID
97	2.9E+2	2.74E+2	5.8	Bq/ml	PIC 9400

Reviewed by: [signature] 6/2/98

Approved by: J.M. Kellen 6/4/98

Radioactive Materials Analytical Laboratory
Inorganic Chemistry Worksheet

Request: IPA8858; INEEL
Sample: 980505-160; See Below
Customer: Sears; INEEL
ID: W-16-S1; INEELTask 3B

Date: 5/13/98
Analyst: DD
Badge #: 33952
Procedure:

Carbon Data

ration Ranges

			STD%C
TC	J12782		
			36.33
IC	M5G9187		11.32

IPA	Sample	Total Carbon %		Inorganic Carbon %		TOC %		STD
		Result	Error	Result	Error	Result	Error	% Rec.
TC Std	J12782	36.05	3.605					99%
IC Std	M5G9187			10.79	1.079			95%
8858	980505-160	**0.95**	0.095	**0.443**	0.0443	**0.507**	0.0507	
	INEEL Dry	**8.11**	0.811	**1.44**	0.144	**6.67**	0.667	
	3b-R1-3	**4.89**	0.489	**1.47**	0.147	**3.42**	0.342	
	3b-R1-4	**3.83**	0.383	**1.19**	0.119	**2.64**	0.264	
	3b-R2-3	**3.04**	0.304	**0.673**	0.0673	**2.367**	0.2367	
	3b-R2-4	**2.42**	0.242	**1.12**	0.112	**1.3**	0.13	
	3b-R3-3	**1.75**	0.175	**0.835**	0.0835	**0.915**	0.0915	
	3b-R3-4	**2.29**	0.229	**2.26**	0.226	**0.03**	0.003	
	3b-R4-3	**1.35**	0.135	**0.474**	0.0474	**0.876**	0.0876	
	3b-R4-4	**1.51**	0.151	**0.406**	0.0406	**1.104**	0.1104	
IC Std	M5G9187			9.73	0.973			86%
TC Std	J12782	31.39	3.139					86%

Comments:

COPY
Original Filed With IPA 8858

Reviewed by: 5-14-98

Approved by: 5/19/98

Request: See Below
Sample: See Below
Customer: Various
ID: Various

Date: 4/10/98
Analyst: RDC
Badge: 35993
Procedure: RA12

Beta Scintillation Data

	IPA	AnaLIS	Wt/Vol	Dil To:	2nd Dil	Dil to:	3rd Dil	Dil to:	ALIQ.	SPIKE	Bq/vial TOT	Bq/vial Error	DIL FAC	SCINT BETA	Error (±)	
1	BLANK	N/A	N/A						0		0	0				
2	Sr STD	N/A	N/A						0.1	STD	19.866	0.225	10	1.99E+2	0.1E+2	BQ/ML
3	1B97	N/A	N/A						0.1	QC	29.259	0.266	10	2.93E+2	0.1E+2	BQ/ML
4	8558	971219-015	1.0 T	15.0					0.5		79.437	0.432	30	2.4E+3	0.1E+3	BQTOT
5	8751	980309-017	0.5671 G	50.0	0.1	10			0.1		11.528	0.178	88168	1.0E+6	0.1E+6	BQ/G
6		Dup	0.5671 G	50.0	0.1	10.0			0.1		11.569	0.178	88168	1.0E+6	0.1E+6	BQ/G
7		Spike	0.5671 G	50.0	0.1	10.0			0.1		31.267	0.274	88168	2.8E+6	0.1E+6	BQ/G
8		980309-018	0.6032 G	50.0	0.1	10.0			0.1		7.515	0.151	82891	6.2E+5	0.1E+5	BQ/G
9	8756	980309-019	0.5395 G	50.0	0.1	10.0	0.1	10.0	0.1		28.105	0.260	9267841	2.6E+8	0.1E+8	BQ/G
10	8765	980323-017	0.5097 G	50.0					0.1		936.606	1.452	981	9.2E+5	0.1E+5	BQ/G
11		980323-018	1.0 T	35.0	0.1	10.0	0.1	10.0	0.1		26.874	0.256	3500000	9.4E+7	0.1E+7	BQTOT
12	8774	980406-013	0.5512 G	50.0	0.1	10.0			0.1		137.581	0.560	90711	1.2E+7	0.1E+7	BQ/G
13	8751	980309-017-4L	1.6481 G	9.13	0.1	10.0			0.1		186.977	0.653	5540	1.0E+6	0.1E+6	BQ/G
14		980309-017-8L	1.4606 G	8.09	0.1	10.0			0.1		169.723	0.623	5539	9.4E+5	0.1E+5	BQ/G
15		980309-018-4L	2.0932 G	11.48	0.1	10.0			0.1		178.264	0.637	5484	9.8E+5	0.1E+5	BQ/G
16		980309-018-8L	1.6313 G	8.94	0.1	10.0			0.1		173.604	0.629	5480	9.5E+5	0.1E+5	BQ/G
17	8756	980309-019-4L	1.1054 G	6.05	0.1	10.0			0.1		51965.000	10.833	5473	2.8E+8	0.1E+8	BQ/G
18		980309-019-8L	1.177 G	6.44	0.1	10.0			0.1		47117.200	10.303	5472	2.6E+8	0.1E+8	BQ/G
19	8751	980309-017-4S	1.6481 G	50.0					0.1		157.142	6.000	303	4.8E+4	0.2E+4	BQ/G
20		980309-017-8S	1.4606 G	50.0					0.1		56.196	0.364	342	1.9E+4	0.1E+4	BQ/G
21		980309-018-4S	2.0932 G	50.0					0.1		9.134	0.163	239	2.2E+3	0.1E+3	BQ/G
22		980309-018-8S	1.6313 G	50.0					0.1		1.677	0.139	307	5.1E+2	0.4E+2	BQ/G
23	8756	980309-019-4S	1.1054 G	50.0					0.1		3592.590	2.829	452	1.6E+6	0.1E+6	BQ/G
24		980309-019-8S	1.177 G	50.0					0.1		2149.080	2.188	425	9.1E+5	0.1E+5	BQ/G
25	INEEL	INEELTEST	0.5063 G	50.0					0.5		22.299	0.256	198	4.4E+3	0.1E+3	BQ/G

Notes:

1) Beta scintillation using 15 ml Ultima Gold cocktail.

QC Data

Control ID	Measured Value	Known Value		% Difference	Units		Instrument ID
Sr STD	2.0E+2	2.0E+2		-0.7	Bq/ml		2700TR
1B97 QC	2.9E+2	2.8E+2		3.8	Bq/ml		2700TR

COPY
Original Filed With IPA 8558

Reviewed by: [signature] 4/13/98

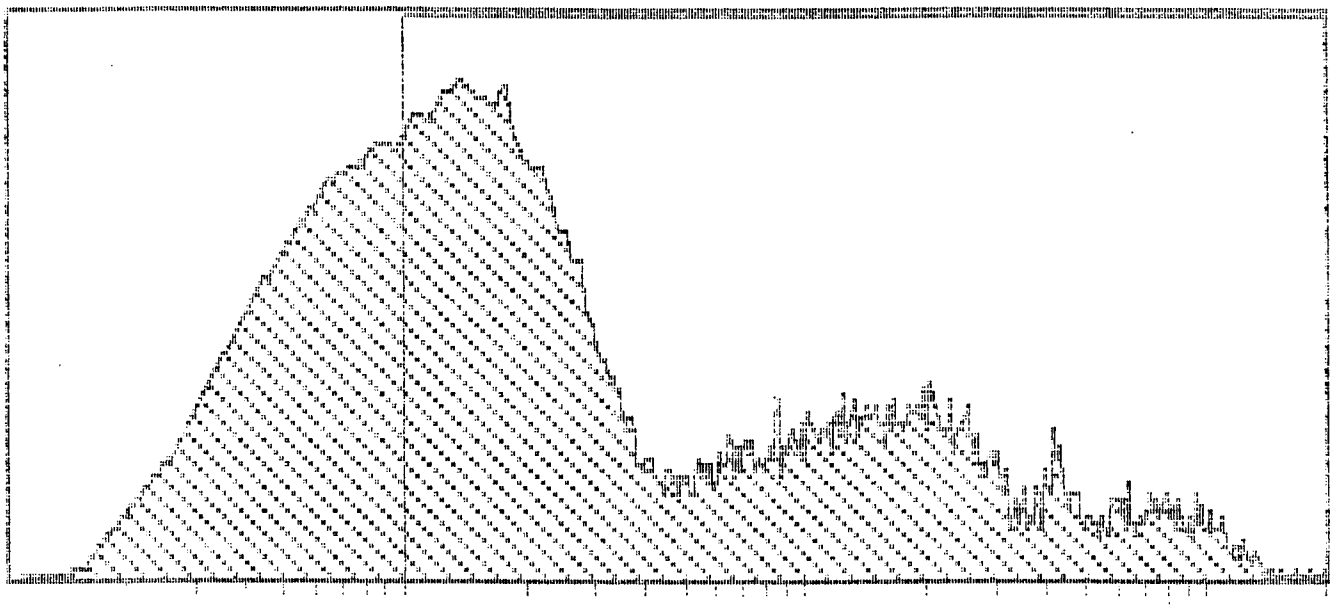
Approved by: [signature] 4/15/98

25 00.00 1088.00 1337.92 1.15 22.299 0.256 547.21

INBOL
Dx 0.5

Log SPECTRUM PLOT

971.5 Y Log maximum Protocol No. 60 Sample No. 25 Repeat 1



1 keV 2000

Region	LL	UL	CPM
A:	0.0	2000	1130.4
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

Oak Ridge National Laboratory
Radioactive Materials Analytical Laboratory
Radiochemistry Worksheet

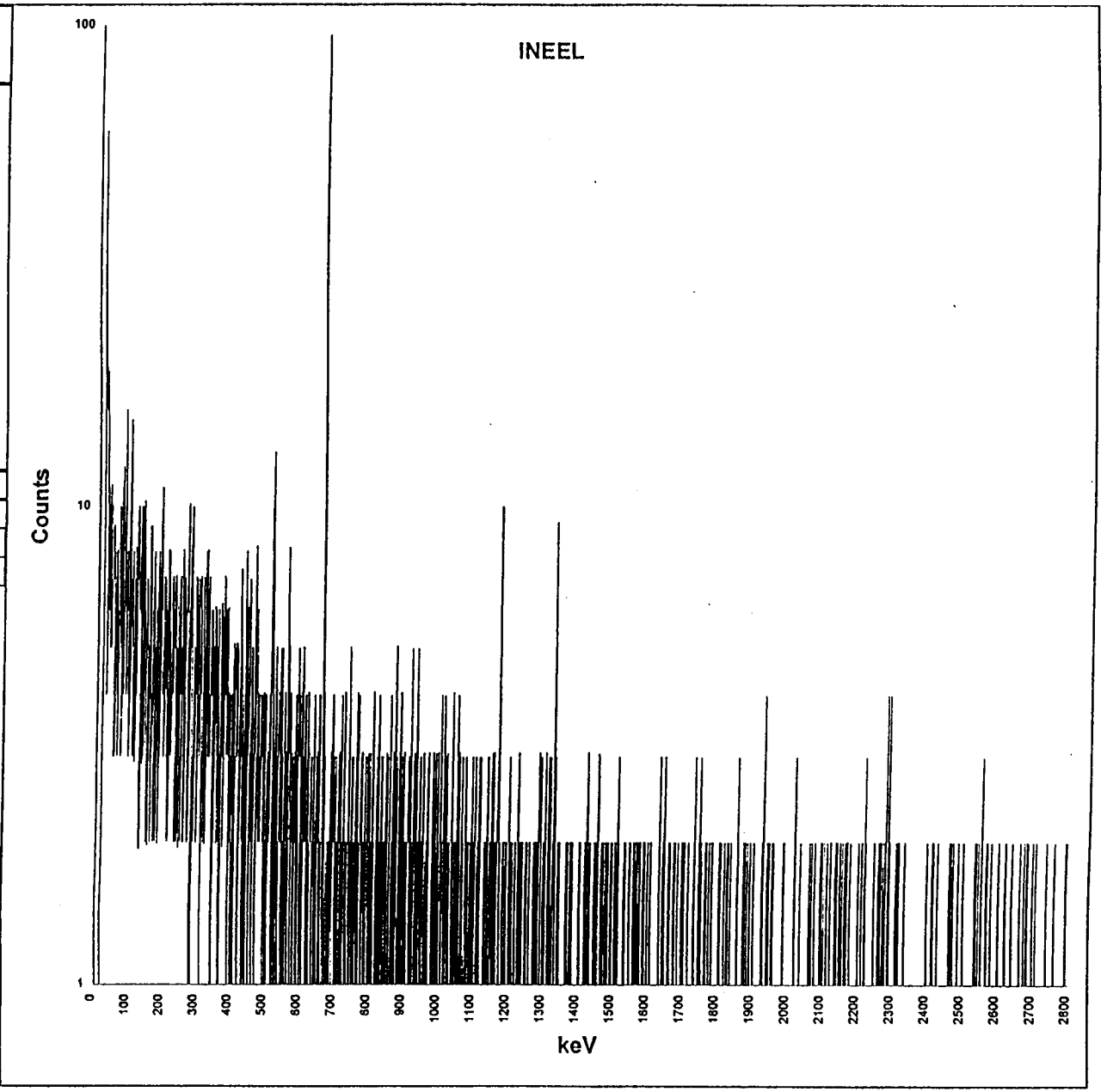
Request: NONE
Sample: NONE
Customer: GIAQUINTO
Sample ID: INEEL
Date: 4/10/98
Analyst: RDC
Badge #: 35993
Procedure: EPA-901.1
Count Time: 12:48
Duration: 30 Min
Corrected To: 4/2/98 12:00
Aliquot: 0.5063g/50 X 10
Units G

Gamma Spectrometry Data

ISOTOPE	BQ/G	± BQ/G
CO-60	1.0E+2	0.4E+2
CS-137	4.6E+2	0.8E+2

Reviewed By: 4/15/98

Approved By: 4/15/98



Initials: DJC

==========
VAX/VMS Radioactive Materials Analysis Laboratory Report 10-APR-1998 13:18:18.76
Detector GE2 Tennelec HpGe ******* Aquisition Date/Time: 10-APR-1998 12:48:03.52

Request No, Customer, Cussam: INEEL DIGEST

Sample ID : 2INEELTEST
Sample Type : VIAL @ 9 CM
Quantity : 1.01260E-01 G
Start Channel : 10
End Channel : 4096

Corre : 2-APR-1998 12:00:00.00
Geometry : GE2V9080197
BKGFILE : 2980321-BKG
Real Time : 0 00:30:00.15
Live time : 0 00:30:00.00

Pk	It	Energy	Area	Bkgnd	FWHM	Channel	%Eff	Cts/Sec	%Err	Fit
1	0	435.49	9	11	1.51	538.41	3.25E-01	5.11E-03	69.0	
2	0	440.18*	11	1	2.20	544.20	3.22E-01	5.98E-03	35.8	
3	0	465.84	7	24	3.42	575.82	3.05E-01	4.17E-03	127.5	
4	0	661.56*	158	7	1.32	817.05	2.23E-01	8.80E-02	8.7	1.66E+00
5	0	1173.30	30	0	2.31	1447.77	1.41E-01	1.67E-02	18.3	
6	0	1332.41	24	0	2.36	1643.88	1.27E-01	1.33E-02	20.4	

Flag: "*" = Peak area was modified by background subtraction

Nuclide Line Activity Report

Nuclide Type: activation

Nuclide	Energy	Area	%Abn	%Eff	Uncorrected BQ/G	Decay Corr BQ/G	2-Sigma %Error
CO-60	1173.24	30	99.90	1.409E-01	1.169E+02	1.173E+02	36.51
	1332.50	24	99.98*	1.274E-01	1.034E+02	1.037E+02	40.82

Nuclide Type: fission

Nuclide	Energy	Area	%Abn	%Eff	Uncorrected BQ/G	Decay Corr BQ/G	2-Sigma %Error
CS-137	661.66	158	85.21*	2.228E-01	4.581E+02	4.583E+02	17.34

Flag: "*" = Keyline

Summary of Nuclide Activity
Sample ID : 2INEELTEST

Acquisition date : 10-APR-1998 12:48:03

Total number of lines in spectrum	6	
Number of unidentified lines	2	
Number of lines tentatively identified by NID	4	66.67%

Nuclide Type : activation

Nuclide	Hlife	Decay	Uncorrected BQ/G	Decay Corr BQ/G	Decay Corr 2-Sigma Error	2-Sigma %Error	Flags
CO-60	5.27Y	1.00	1.034E+02	1.037E+02	0.423E+02	40.82	
			---	---			
	Total Activity :		1.034E+02	1.037E+02			

Nuclide Type : fission

Nuclide	Hlife	Decay	Uncorrected BQ/G	Decay Corr BQ/G	Decay Corr 2-Sigma Error	2-Sigma %Error	Flags
CS-137	30.00Y	1.00	4.581E+02	4.583E+02	0.795E+02	17.34	
			---	---			
	Total Activity :		4.581E+02	4.583E+02			

Grand Total Activity :	5.615E+02	5.620E+02

Flags: "K" = Keyline not found "M" = Manually accepted
"E" = Manually edited "A" = Nuclide specific abn. limit

Unidentified Energy Lines

Sample ID : 2INEELTEST

Acquisition date : 10-APR-1998 12:48:03

It	Energy	Area	Bkgnd	FWHM	Channel	Left	Pw	Cts/Sec	%Err	%Eff	Flags
0	435.49	9	11	1.51	538.41	534	9	5.11E-03	****	3.25E-01	T
0	440.18	11	1	2.20	544.20	542	5	5.98E-03	71.7	3.22E-01	
0	465.84	7	24	3.42	575.82	570	11	4.17E-03	****	3.05E-01	

Flags: "T" = Tentatively associated

Minimum Detectable Activity Report

Nuclide	Bckgnd Sum	Energy (keV)	MDA (BQ/G)
BE-7	7.	477.61	2.9078E+02
NA-22	1.	1274.53	3.0754E+01
K-40	6.	1460.83	6.1359E+02
CR-51	13.	320.08	3.0809E+02
MN-54	7.	834.83	4.4593E+01
CO-57	18.	122.06	2.0559E+01
SE-75	18.	264.66	4.1599E+01
NB-94	6.	871.10	4.3702E+01
NB-95	5.	765.79	4.2857E+01
ZR-95	3.	756.72	5.9353E+01
RU-103	4.	497.05	2.9217E+01
RU-106	4.	621.92	2.9574E+02
CD-109	9.	88.03	4.5785E+02
AG-110M	6.	657.76	3.7071E+01
TE-121	5.	573.13	4.9961E+01
TE-121M	12.	212.22	2.1486E+01
TE-123M	9.	158.99	1.6111E+01
I-125	18.	27.40	4.8672E+03
SB-125	9.	427.88	9.4621E+01
TE-127	0.	417.93	Half-Life too short
I-129	11.	39.60	3.5448E+03
I-131	12.	364.48	6.5263E+01
I-132	0.	667.73	Half-Life too short
I-133	7.	529.85	2.1935E+04
CS-134	8.	604.71	3.7827E+01
I-135	0.	1260.42	Half-Life too short
CE-144	17.	133.54	1.5129E+02
PM-147	19.	121.26	6.1660E+05
EU-152	2.	1408.00	2.0119E+02
EU-154	1.	1274.54	8.6221E+01
EU-155	14.	86.54	5.9950E+01
HG-203	6.	279.19	2.0957E+01
PB-212	0.	238.58	Half-Life too short
RA-224	11.	240.76	2.0584E+03
RA-225	13.	40.34	1.2737E+03
RA-226	8.	609.31	7.7164E+01
AC-227D	9.	235.97	1.4468E+02
TH-228	17.	84.26	1.8780E+03
TH-229	15.	193.59	3.7582E+02
U-235	12.	143.79	1.3506E+02
U-238	1.	1001.00	4.1533E+03
AM-241	9.	59.54	1.1005E+02
CM-243	4.	277.60	9.5989E+01

RADIOACTIVE MATERIALS ANALYSIS LAB
Detector GE2
VALUES WHICH ARE LESS THAN MDA

Isotope	Bkg Cts	Net Cts	Total Cts	Cts/Sec	Eff	Results BQ /G		Error
BE-7	22.	-2.	20.	-1.11E-03	2.81E-03	-5.0E+01	+/-	2.3E+02
NA-22	1.	1.	1.	5.56E-04	1.25E-03	6.0E+00	+/-	1.2E+01
CO-57	39.	6.	44.	3.06E-03	7.29E-03	6.6E+00	+/-	1.5E+01
CO-60	1.	26.	26.	1.44E-02	1.21E-03	1.6E+02	+/-	1.2E+01
CD-109	27.	-1.	26.	-5.56E-04	6.05E-03	-3.4E+01	+/-	3.6E+02
AG-110M	534.	-396.	138.	-2.20E-01	2.12E-03	-1.5E+03	+/-	1.7E+02
I-125	25.	9.	34.	5.00E-03	4.22E-05	1.3E+03	+/-	1.5E+03
CS-134	36.	-15.	21.	-8.33E-03	2.28E-03	-5.1E+01	+/-	4.0E+01
CS-137	12.	170.	182.	9.44E-02	2.11E-03	7.1E+02	+/-	2.9E+01
EU-152	1.	2.	2.	1.11E-03	1.15E-03	6.3E+01	+/-	6.3E+01
EU-154	1.	1.	1.	5.56E-04	1.25E-03	1.7E+01	+/-	3.4E+01
EU-155	32.	4.	35.	1.94E-03	5.93E-03	1.3E+01	+/-	4.2E+01
PB-212	24.	5.	29.	2.78E-03	5.35E-03	1.2E+01	+/-	2.3E+01
U-235	20.	9.	29.	5.00E-03	7.21E-03	8.9E+01	+/-	8.9E+01
U-238	20.	-11.	9.	-5.83E-03	1.52E-03	-8.0E+03	+/-	6.7E+03

Radioactive Materials Analytical Laboratory
Inorganic Chemistry Worksheet

Request: IPA8765
Sample: 980323-017; INEEL
Customer: Van Hoesen; INEEL
ID: SEE REQUEST

4/2/98
DD
33952
CASD 3051

Raw Sample Microwave Digestion Data

IPA	Sample	Vessel	Vol/Wt	Units	Conc. HNO3 (ml)				Final Volume (ml)	Final Solution % HNO3	Dilution Factor
8765	980323-017	1	0.5097	g	10				50.0	20%	98.10
	INEEL	2	0.5063	g	10				50.0	20%	98.76
	BLANK	3	0.5	ml	10				50.0	20%	100.00

COPY
Original Filed With IPA 8765

Time(M)	Power(W)	Temp(°C)
1	250	100
1	0	100
1	250	140
1	0	140
10	550	175

Digestion Comments:

Homogenization: 980323-017 asd INEEL samples were mixed to homogenize.

Reaction/Off-Gassing: 980323-017 produced a reaction and off-gassing upon nitric addition.
The INEEL sample also produced a small reaction and off-gassing upon nitric addition.

Dissolution Observations: 980320-015 formed a very light tan digest with no observable solids.
(>99% in solution)
The INEEL sample produced a cloudy-tan digest with tan, black and gray solids.

Reviewed by: [signature] 4-2-98

Approved by: [signature] 4/2/98

Inorganic Chemistry Worksheet

Request: IPA8663; 8678; INEEL
Sample: 980212-013; 980223-058
Customer: Sears; Mattus; INEEL
ID: See Request

Date: 3-19-98/3-20-98
Analyst: DD
Badge #: 33952
Procedure: SW-846-9060

Carbon Data

Calibration Ranges

			STD%C
TC	37002731		
			39.96
IC	B26346		14 3

IPA	Sample	Total Carbon % Result	Total Carbon % Error	Inorganic Carbon % Result	Inorganic Carbon % Error	TOC % Result	TOC % Error	STD % Rec.
TC Std	37002731	38.1	3.81					95%
IC Std	B26346			14.33	1.433			100%
8663	980212-013	8.49	0.849	0	0	8.49	0.849	
8678	980223-058	2.87	0.287	1.32	0.132	1.55	0.155	
INEEL1	WET	2.72	0.272	0	0	2.72	0.272	
INEEL2	WET	3.103	0.3103	0	0	3.103	0.3103	
INEEL1	DRY	5.56	0.556	0	0	5.56	0.556	
INEEL2	DRY	9.17	0.917	0	0	9.17	0.917	
IC Std	B26346			14.63	1.463			102%
TC Std	37002731	35.33	3.533					88%

Comments:

*Sample results of 0 % are reported as <0.1% on the final report.

COPY

Original Filed With IPA 8663

Reviewed by: [illegible] 3-21-98

Approved by: [illegible] 3/23/98

Radioactive Materials Analytical Laboratory

Inorganic Chemistry Worksheet

Request: IPA8751; 8756; INEEL
Sample: 980309-017,-018; 980309-019; INEEL
Customer: Sears; INEEL
ID: See Requests

Date: 3-24-98/3-25-98
Analyst: DD
Badge #: 33952
Procedure: RMAL

PERCENT MOISTURE / SOLIDS DATA

SAMPLE NO.	CONTAINER WEIGHT (g)	CONTAINER & WET WT. (g)	WET WEIGHT (g)	CONTAINER & DRY WT. (g)	DRY WEIGHT (g)
980309-017	1.1002	2.9880	1.8878	2.1009	1.0007
980309-018	1.0991	2.6945	1.5954	2.1864	1.0873
980309-019	1.0927	2.8554	1.7627	2.4754	1.3827
INEEL	189.7200	441.2600	251.5400	294.3800	104.6600

SAMPLE NO.	PERCENT SOLIDS %	PERCENT MOISTURE %
980309-017	53.0	47.0
980309-018	68.2	31.8
980309-019	78.4	21.6
INEEL	41.6	58.4

COPY
Original Filed With IPA 8751

NOTES:
1) Samples dried at an average oven temperature of 105°C, cooled in a dessicator and weighed.

Reviewed by: 3-25-98

Approved by: 3/25/98

Radioactive Materials Analytical Laboratory

Inorganic Chemistry Worksheet

Request: INEEL	Date: 3-19-98/3-20-98
Sample: 1 & 2	Analyst: DD
Customer:	Badge #: 33952
ID:	Procedure: RMAL

PERCENT MOISTURE / SOLIDS DATA

SAMPLE NO.	CONTAINER WEIGHT (g)	CONTAINER & WET WT. (g)	WET WEIGHT (g)	CONTAINER & DRY WT. (g)	DRY WEIGHT (g)
1	1.0895	3.1880	2.0985	2.2709	1.1814
2	1.0989	3.3453	2.2464	1.7306	0.6317

SAMPLE NO.	PERCENT SOLIDS %	PERCENT MOISTURE %
1	**56.3**	**43.7**
2	**28.1**	**71.9**

NOTES:

1) Samples dried at an average oven temperature of 110° F, placed in desiccator, weighed, and the process repeated until stable weight achieved.

Reviewed by: _____

Approved by: _____

Radioactive Materials Analytical Laboratory
Inorganic Chemistry Worksheet

Request: INEEL
Sample: See Below
Customer: INEEL
ID: INEEL

Date: 8/26/98
Analyst: DD
Badge #: 33952
Procedure:

Carbon Data

ration Ranges

			STD%C
TC	J12782		
			36.33
IC	M5G9187		11.32

IPA	Sample	Total Carbon % Result	Total Carbon % Error	STD % Rec.
TC Std	J12782	34.39	3.439	95%
Task4b-R10	483	**2.3**	0.23	
Task4b-R10	484	**4.62**	0.462	
Task4b-R10	485	**2.78**	0.278	
Task4b-R10	486	**2.49**	0.249	
Task4b-R9	624	**5.09**	0.509	
Task4b-R9	625	**4.2**	0.42	
Task4b-R9	626	**3.99**	0.399	
Task4b-R9	627	**5.93**	0.593	
Task4b-R9	628	**4.94**	0.494	
Task4b-R11	759	**7.11**	0.711	
Task4b-R11	760	**6.06**	0.606	
Task4b-R11	761	**5.5**	0.55	
Task4b-R11	762	**5.39**	0.539	
Task4b-R12	893	**1.74**	0.174	
Task4b-R12	894	**3.02**	0.302	
Task4b-R12	895	**2.58**	0.258	
Task4b-R12	896	**2.56**	0.256	

IPA	Sample	Total Carbon % Result	Total Carbon % Error	STD % Rec.
Task4b-R15	958	**2.74**	0.274	
Task4b-R15	959	**2.12**	0.212	
Task4b-R15	960	**1.44**	0.144	
Task4b-R15	961	**1.89**	0.189	
Task4b-R16	1025	**4.41**	0.441	
Task4b-R16	1026	**6.71**	0.671	
Task4b-R16	1027	**4.81**	0.481	
Task4b-R16	1028	**5.93**	0.593	
Task4b-R14	1236	**5.54**	0.554	
Task4b-R14	1237	**2.46**	0.246	
Task4b-R14	1238	**2.39**	0.239	
Task4b-R14	1239	**3.39**	0.339	
Task4b-R13	1242	**2.05**	0.205	
Task4b-R13	1243	**3.84**	0.384	
Task4b-R13	1244	**3.49**	0.349	
Task4b-R13	1245	**2.48**	0.248	
TC Std	J12782	33.75	3.375	93%

Comments: *Sludge dried at 105°C for ~48hrs and placed in dessicator until analyzed.

Reviewed by: 8-27-98

Approved by: 8/27/98

TOC-5000 DATA REPORT
DATE 08(AUG)-26-1998 14:50

SPL#	TC,%	RMK	IC	RMK	TOC
1	34.39	1****			
2	2.302	1****			
3	4.617	1****			
4	2.775	1****			
5	2.491	1****			
6	5.093	1****			
7	4.195	1****			
8	3.992	1****			
9	5.927	1****			
10	4.939	1****			
11	7.111	1****			
12	6.063	1****			
13	5.497	1****			
14	5.385	1****			
15	1.742	1****			
16	3.022	1****			
17	2.581	1****			
18	2.557	1****			
19	2.744	1****			
20	2.117	1****			
21	1.440	1****			
22	1.888	1****			
23	4.411	1****			
24	6.711	1****			
25	4.805	1****			
26	5.930	1****			
27	5.540	1****			
28	2.464	1****			
29	2.390	1****			
30	3.393	1****			
31	2.051	1****			
32	3.835	1****			
33	3.493	1****			
34	2.476	1****			
35	33.75	1****			

SPL#	TC	RMK	IC,%	RMK	TOC
36			19.14	2****	
37			9.755	2****	
38			0.777	2****	

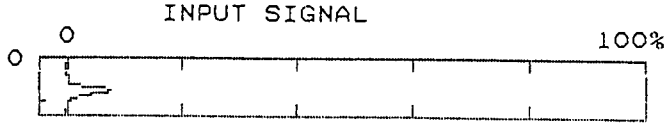
ANALYST : WJ2.dt 8-27-98
SAMPLE :

SAMPLE# 36 IC
[x30, C# 2, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	3895 19.14	26.50
MN	3895 19.14	26.50
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 14:34

INPUT SIGNAL



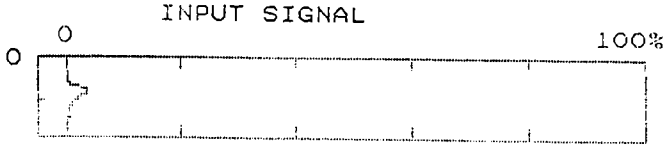
TIME [min]

SAMPLE# 37 IC
[x30, C# 2, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	1738 9.755	23.20
MN	1738 9.755	23.20
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 14:38

INPUT SIGNAL



TIME [min]

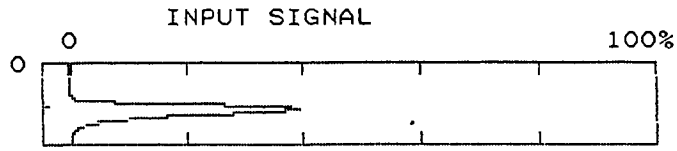
SAMPLE# 38 IC
[x30, C# 2, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	1358 0.777	227.4
MN	1358 0.777	227.4
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 14:43

```
SAMPLE#  34   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %      AMNT(mg)
    1  4034 2.476 204.2
   - - - - - - - - - - - - - - -
   MN  4034 2.476 204.2
   SD        0.000
   CV         0.00%
```

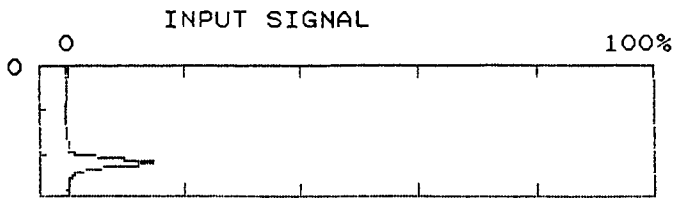
DATE 08(AUG)-26-1998 14:20



TIME [min]

```
SAMPLE#  35   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %      AMNT(mg)
    1 12603 33.75 46.80
   - - - - - - - - - - - - - - -
   MN 12603 33.75 46.80
   SD        0.000
   CV         0.00%
```

DATE 08(AUG)-26-1998 14:26

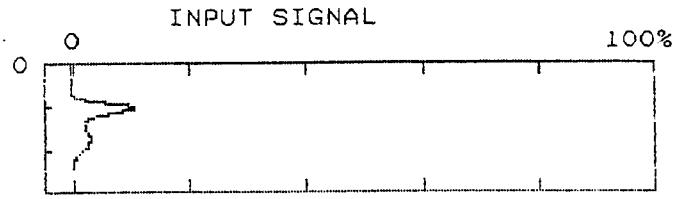


TIME [min]

SAMPLE# 32 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	7262 3.835	237.3
MN	7262 3.835	237.3
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 14:09

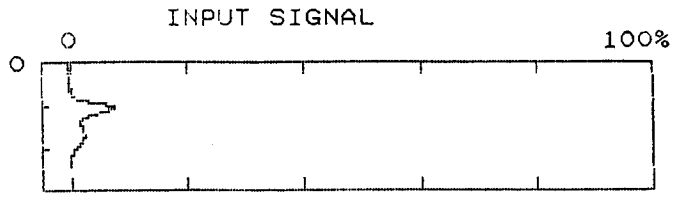


TIME [min]

SAMPLE# 33 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	5578 3.493	200.1
MN	5578 3.493	200.1
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 14:15

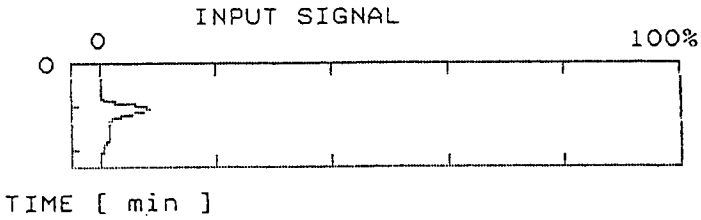


TIME [min]

SAMPLE# 30 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	5929	3.393	219.0
MN	5929	3.393	219.0
SD		0.000	
CV		0.00%	

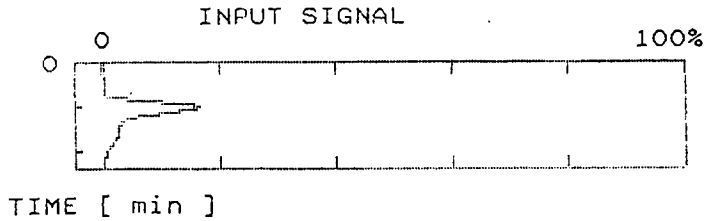
DATE 08(AUG)-26-1998 13:06



SAMPLE# 31 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	3634	2.051	222.0
MN	3634	2.051	222.0
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 14:03

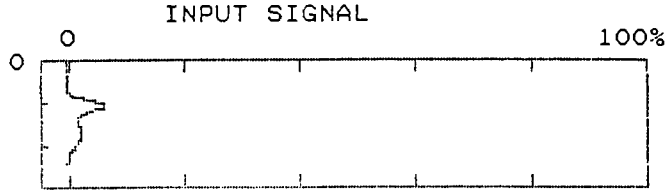


SAMPLE# 28 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	4000	2.464	203.4
MN	4000	2.464	203.4
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 12:54

INPUT SIGNAL



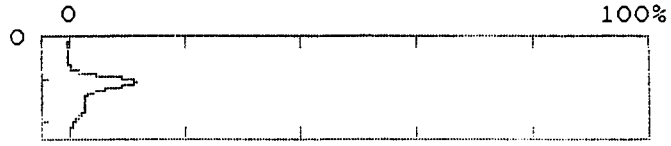
TIME [min]

SAMPLE# 29 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	3820	2.390	200.3
MN	3820	2.390	200.3
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 13:00

INPUT SIGNAL

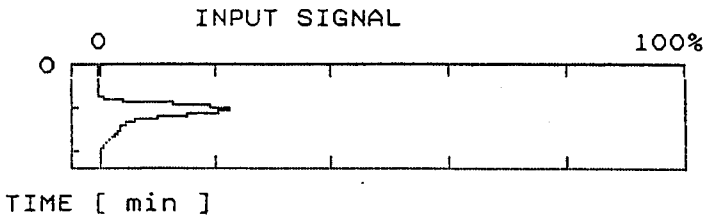


TIME [min]

SAMPLE# 26 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	9658 5.930	204.1
MN	9658 5.930	204.1
SD	0.000	
CV	0.00%	

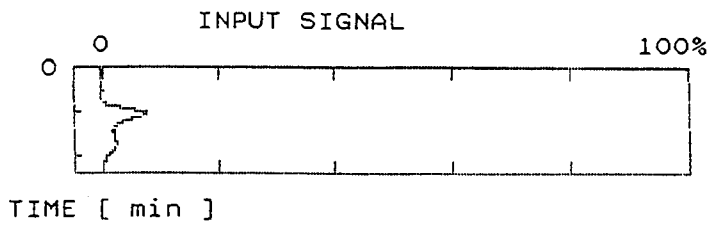
DATE 08(AUG)-26-1998 12:43



SAMPLE# 27 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	9673 5.540	218.8
MN	9673 5.540	218.8
SD	0.000	
CV	0.00%	

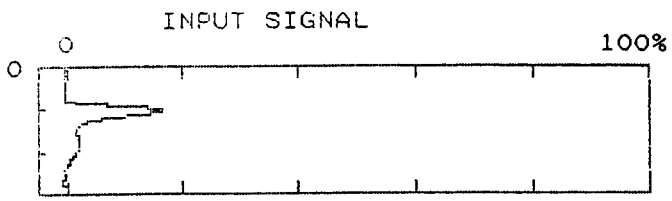
DATE 08(AUG)-26-1998 12:49



SAMPLE# 24 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	10796	6.711 201.6
MN	10796	6.711 201.6
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 12:31

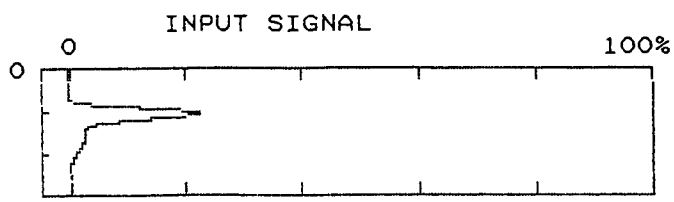


TIME [min]

SAMPLE# 25 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	8006	4.805 208.8
MN	8006	4.805 208.8
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 12:37

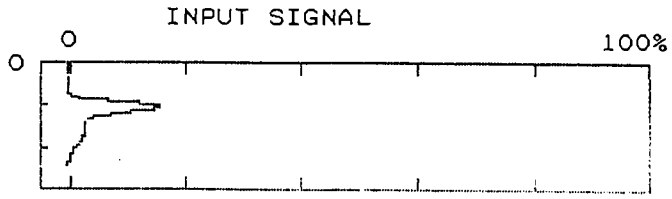


TIME [min]

SAMPLE# 22 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	3029	1.888	201.0
MN	3029	1.888	201.0
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 12:19

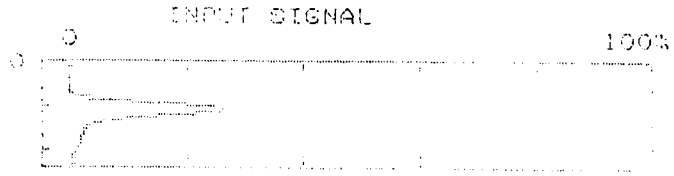


TIME [min]

SAMPLE# 23 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	7134	4.411	202.7
MN	7134	4.411	202.7
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 12:25



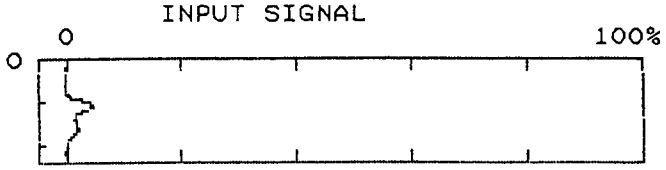
TIME [min]

SAMPLE# 20 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	3447	2.117	204.0
MN	3447	2.117	204.0
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 12:06

INPUT SIGNAL



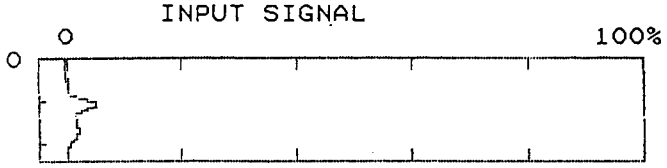
TIME [min]

SAMPLE# 21 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	2550	1.440	221.9
MN	2550	1.440	221.9
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 12:13

INPUT SIGNAL



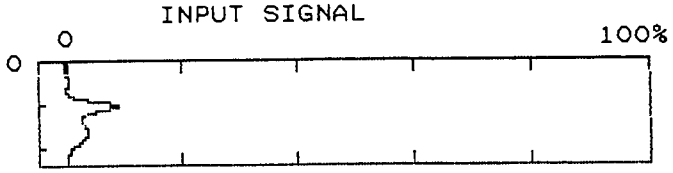
TIME [min]

SAMPLE# 18 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	4313 2.557	211.4
MN	4313 2.557	211.4
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 10:34

INPUT SIGNAL



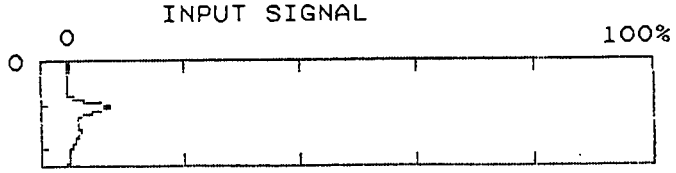
TIME [min]

SAMPLE# 19 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	5043 2.744	230.3
MN	5043 2.744	230.3
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 12:00

INPUT SIGNAL

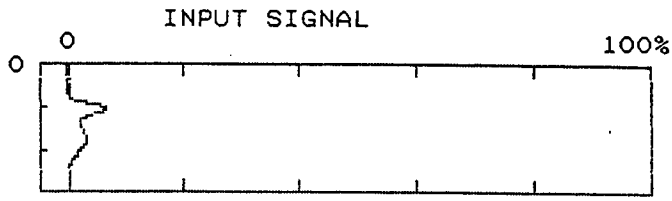


TIME [min]

SAMPLE# 16 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	4893 3.022	202.9
MN	4893 3.022	202.9
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 10:18

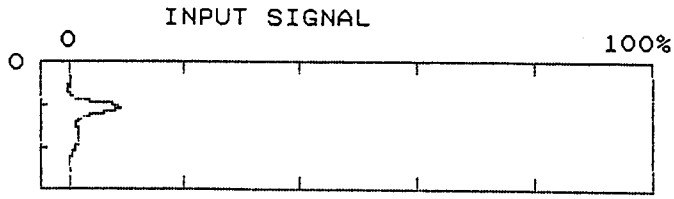


TIME [min]

SAMPLE# 17 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	4611 2.581	223.9
MN	4611 2.581	223.9
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 10:25



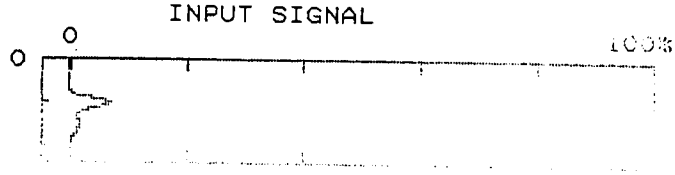
TIME [min]

SAMPLE# 14 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	9612	5.385	223.7
MN	9612	5.385	223.7
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 10:06

INPUT SIGNAL



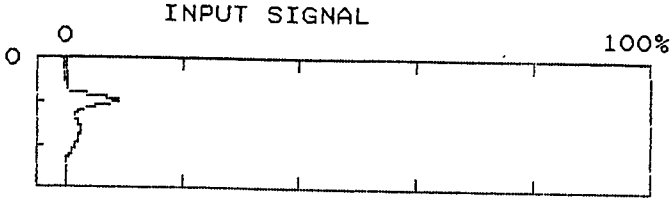
TIME [min]

SAMPLE# 15 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	[illegible]	[illegible]	[illegible]
MN	[illegible]	[illegible]	[illegible]
SD		[illegible]	
CV		[illegible]	

[illegible]

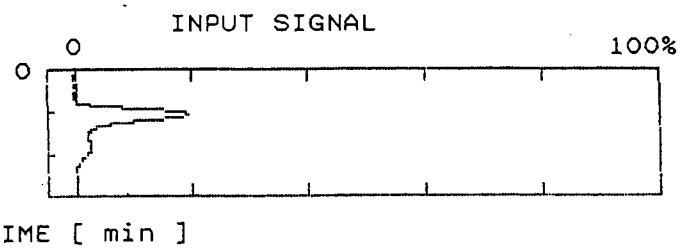
INPUT SIGNAL



TIME [min]

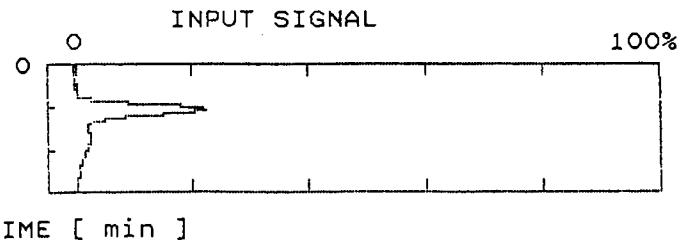
```
SAMPLE#  12   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1 10044 6.063 207.6
   - - - - - - - - - - - - - - -
   MN 10044 6.063 207.6
   SD        0.000
   CV         0.00%
```

DATE 08(AUG)-26-1998 09:51




```
SAMPLE#  13   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1  9185 5.497 209.4
   - - - - - - - - - - - - - - -
   MN  9185 5.497 209.4
   SD        0.000
   CV         0.00%
```

DATE 08(AUG)-26-1998 10:00

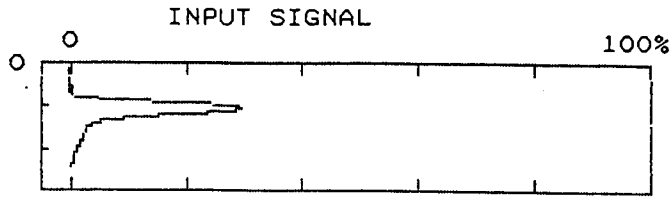


SAMPLE# 10 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	8946 4.939	227.0
MN	8946 4.939	227.0
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 09:37

INPUT SIGNAL



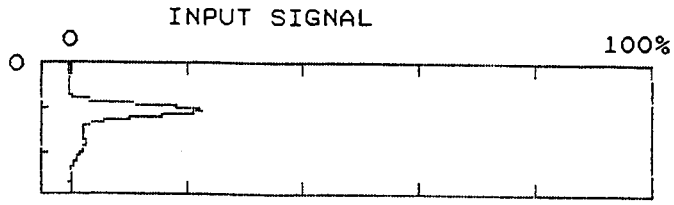
TIME [min]

SAMPLE# 11 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA %	AMNT(mg)
1	12596 7.111	222.0
MN	12596 7.111	222.0
SD	0.000	
CV	0.00%	

DATE 08(AUG)-26-1998 09:44

INPUT SIGNAL

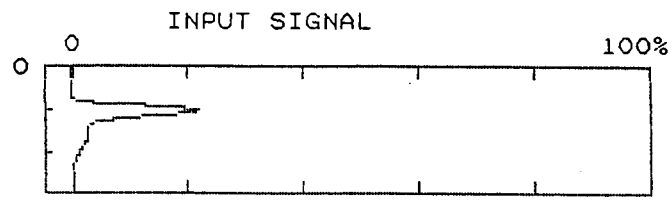


TIME [min]

SAMPLE# 8 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	6989	3.992	219.4
MN	6989	3.992	219.4
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 09:24

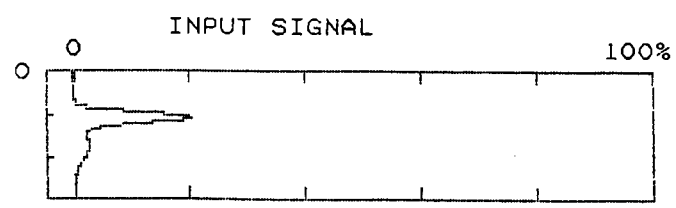


TIME [min]

SAMPLE# 9 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	9256	5.927	195.7
MN	9256	5.927	195.7
SD		0.000	
CV		0.00%	

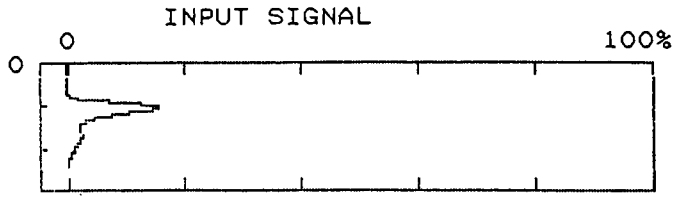
DATE 08(AUG)-26-1998 09:31



TIME [min]

```
SAMPLE#   6   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1  8762 5.093 215.6
   - - - - - - - - - - - - - - -
   MN  8762 5.093 215.6
   SD        0.000
   CV         0.00%
```

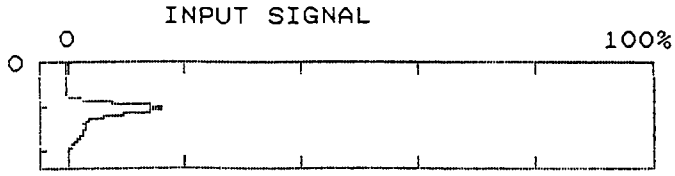
DATE 08(AUG)-26-1998 09:13



TIME [min]

```
SAMPLE#   7   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1  7103 4.195 212.2
   - - - - - - - - - - - - - - -
   MN  7103 4.195 212.2
   SD        0.000
   CV         0.00%
```

DATE 08(AUG)-26-1998 09:19



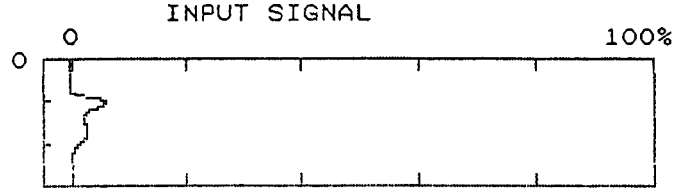
TIME [min]

SAMPLE# 4 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	4599	2.775	207.7
MN	4599	2.775	207.7
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 08:56

INPUT SIGNAL



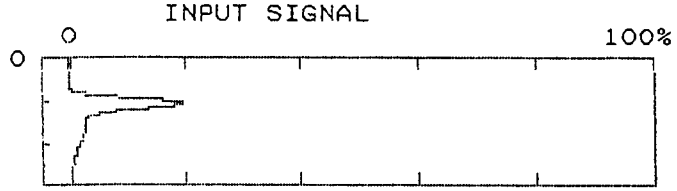
TIME [min]

SAMPLE# 5 TC
[x30, C# 1, DENSITY 1.0mg/µl]

#	AREA	%	AMNT(mg)
1	4139	2.491	208.2
MN	4139	2.491	208.2
SD		0.000	
CV		0.00%	

DATE 08(AUG)-26-1998 09:02

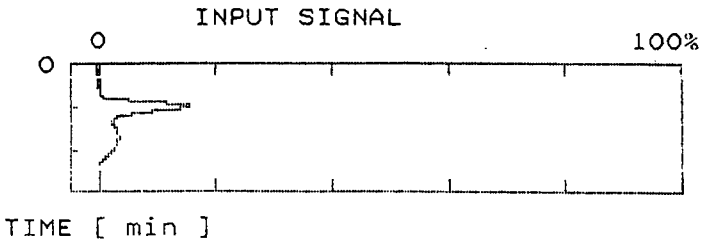
INPUT SIGNAL



TIME [min]

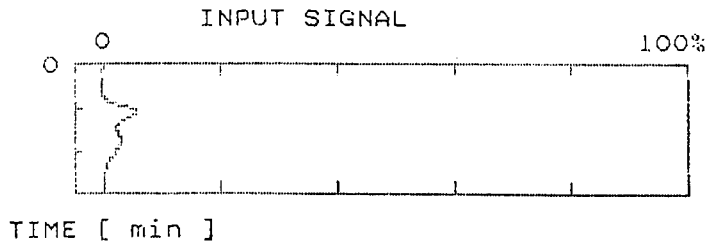
```
SAMPLE#   2   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1  3780 2.302 205.8
   - - - - - - - - - - - - - - -
   MN  3780 2.302 205.8
   SD        0.000
   CV         0.00%
```

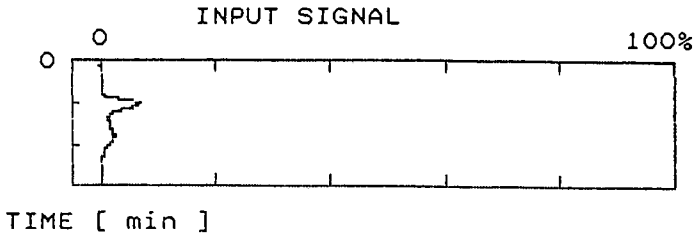
DATE 08(AUG)-26-1998 08:43



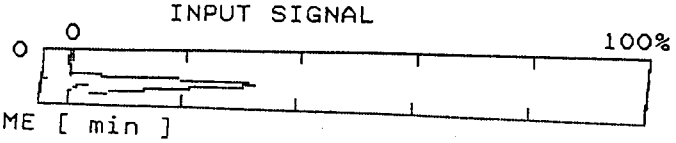

```
SAMPLE#   3   TC
 [x30, C#  1, DENSITY   1.0mg/µl]
    #  AREA %       AMNT(mg)
    1  7515 4.617 204.0
   - - - - - - - - - - - - - - -
   MN  7515 4.617 204.0
   SD        0.000
   CV         0.00%
```

DATE 08(AUG)-26-1998 08:49




INPUT SIGNAL
0
100%
0
TIME [min]

INPUT SIGNAL



TIME [min]

SAMPLE# 1 TC

[x30, C# 1, DENSITY 1.0mg/µl]

		AREA %		AMNT(mg)
1	9796	34.39	35.70	- - -
MN	9796	34.39	35.70	
SD		0.000		
CV		0.000%		

DATE (AUG)-26-1998 08:35

Run #3 Run #4

7-30-97

CONTROL WORK SHEET - 1

144 TASK 46-R3 + R4

ANALYST	
COMPLETED	
TIME	DATE
CHECKED BY (SUPERVISOR)	
CURVE FACTOR	
ANALYZED FOR	

SERIES

SAMPLE CODE	BTL	~~ORIGINAL~~ SAMPLE SIZE (BTL THEN)	~~DILUTED TO~~	~~ALIQUOT SIZE~~ (SAMPLE BTL THEN)	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1313	11.1741	14.5055	19.5211	INITIAL		200X		
1314	11.0279	14.3779	19.3687					
1315	11.1997	14.5141	19.4276					
1316	11.0279	14.3902	19.0599					
1317	11.0759	13.0885	16.3007	RUN #3 TO C1D	.01 + .12			
1318	11.0603	13.0799	16.2612	C2D				
1319	11.1693	13.1835	16.0720	4D				
1320	11.1346	13.1489	15.8934	2D				
1321	11.1712	13.1939	16.1116	3D				
1322	11.2831	13.3216	16.5613	4D				
1323	11.0886	13.1268	16.1659	C1D DUP				
1324	11.1988	13.2457	16.4071	RUN #4 C1 TO	1 + .02			
1325	11.1156	13.1480	16.5291	C2				
1326	11.1481	13.1863	16.0164	1				
1327	11.0580	13.1003	16.0446	2				
1328	11.0671	13.1017	16.2690	3				
1329	11.0902	13.1391	16.6901	4				
1330	11.0492	13.0908	15.9412	C1 DUP				
1331	11.0837	13.1415	16.0279	C1A	1 + .02	200X		
1332	11.1146	13.1736	16.1402	C2A		200X		
1333	11.1020	13.1665	16.4135	1A		200X		
1334	11.1262	13.1865	15.6990	2A				
1335	11.2739	13.3268	16.3693	3A				
1336	11.1880	13.2505	16.3014	4A				
1337	11.1592	13.2206	16.2834	C2A dup		200X		

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1338	10.9526	13.0170	16.4352		C_1 Tzn	200X		
1339	11.1001	13.1679	16.1394		C_2	200X		
1340	11.0957	13.1746	16.4797		1	1X		
1341	11.1266	13.1965	16.6931		2			
1342	11.1436	13.2042	16.4330		3			
1343	11.0701	13.1280	16.4872		4			
1344	11.0205	13.0841	16.0091		C_2 dup	200X		
1345	11.1986 ~~11.1911~~	13.2659	16.3448		C_{1A} Tzn	200X		
1346	11.1911	13.2605	16.6283		C_{2A}	200X		
1347	11.2067	13.2758	16.7230		1_A	20X		
1348	11.1994	13.2544	16.4710		2_A			
1349	11.2644	13.3263	16.1659		3_A	1X		
1350	11.1564	13.2095	16.7823		4_A	1X		
1351	11.1790	13.2295	16.3171		1_A dup	20X ~~200X~~		
1352	11.1498	13.1883	16.0310		C_1 Tzn	200X		
1353	11.2235	13.2715	16.2055		C_2	200X		
1354	11.1850	13.2135	16.2206		1	1X		
1355	11.1399	13.1972	16.2888		2			
1356	11.2940	13.3478	16.6201		3			
1357	11.2731	13.3269	16.6295		4			
1358	11.0851	13.1428	16.0862		1 dup			
1359	11.1447	13.1158	16.5637		C_{1A}	200X	Tzn	
1360	11.0996	13.0895	16.5976		C_{2A}	200X		
1361	11.1443	13.1419	16.2157		1_A	20X		
1362	11.0956	13.0888	16.2714		2_A	20X		

UCN-2133
(2 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1363	11.2437	13.2384	16.2273		3A	1X		
1364	11.1382	13.1317	15.8663		4A	1X		
1365	11.0587	13.0654	15.9770		2A dup	20X		
1366	11.2163	13.2103	16.3728		C1	200 X	1/4 LITRS	
1367	11.1891	13.1912	16.4434		C2	200 X		
1368	11.3126	13.3137	16.5873		1	1X		
1369	11.2844	13.2780	15.8999		2			
1370	11.1477	13.1636	16.5075		3			
1371	11.2446	13.2545	16.5784		4			
1372	11.1777	13.1861	16.4679		2 dup			
1373	11.2357	13.2360	16.1306		C1B	200X	1/4 LITRS	
1374	11.1190	13.1064	15.9941		C2B			
1375	11.0565	13.0567	16.4735		1B	20X		
1376	11.1456	13.1533	16.3628		2B	20X		
1377	11.0864	13.0885	16.3499		3B	1X		
1378	11.1687	13.1749	16.6210		4B	1X		
1379	11.1416	13.1495	16.3099		3B DUP	1X		
1380	11.1358	13.1460	16.1575		C1	200X	1/4 LITRS	
1381	11.1224	13.1380	16.0354		C2			
1382	11.1711	13.1861	16.4048		1	1X		
1383	11.1265	13.1491	16.4580		2			
1384	11.2918	13.3071	16.1354		3			
1385	11.0405	13.0563	16.7162		4			
1386	11.0759	13.0882	16.3184		3 DUP			
1387	11.0492	13.0824	16.7440		C1B	200X	1/20 LITRS	

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1388	11.1590	13.1749	16.1410		C_{2A}	200 X		
1389	11.1798	13.2055	16.3669		1A	2X		
1390	11.0172	13.0496	16.0531		2A	↓		
1391	11.1023	13.1023	15.8463		3A	1X		
1392	11.1403	13.1672	16.0671		4A	1X		
1393	11.1620	13.1693	16.0211		3 1A DUP	1X	~~[illegible]~~	
1394	11.1359	13.1570	16.1268		C_1	20 X	T 20 MTS	
1395	11.1619	13.1911	16.0238		C_2	↓		
1396	11.2081	13.2366	16.4995		1	1X		
1397	11.0887	13.1054	16.1390		2			
1398	11.0004	13.0219	16.3741		3			
1399	11.0215	13.0475	16.0675		4			
1400	11.1449	13.1807	16.2198		3 DUP	↓		
1401	11.1270	13.0548	15.8237		C_{1A}	200 X	T 24 HRS	
1402	11.0296	13.0422	15.7392		C_{2A}	200X		
1403	11.0643	13.0885	15.7652		1_A	20X		
1404	11.2499	13.2704	16.2208		2_A	20X		
1405	11.1048	13.1242	16.7206		3_A	1X		
1406	11.1288	13.1604	16.0629		4_A			
1407	11.1349	13.1617	15.8942		4_A dup	↓		
1408	11.0625	13.0930	15.9341		C_1	200X		
1409	11.1229	13.1572	15.9547		C_2	200X		
1410	11.0507	13.0845	16.0181		1	1X		
1411	11.1715	13.2011	16.4765		2			
1417 ~~144~~	11.0683	13.0953	16.2856		3	↓		

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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1413	11.1692	13.1889	16.4694	4	1X			
1414	11.1209	13.1683	15.9529	4 dup	1X			
1415	11.1370	13.1582	16.2060		C1 96hr	200X		
1416	11.2364	13.2672	16.0098		C2	ϕ		
1417	11.1068	13.1465	16.3239		1	40X		
1418	11.0680	13.1045	16.3845		2	40X		
1419	11.1619	13.2002	16.4585		3	1X		
1420	11.2784	13.3168	17.0012		4	1X		
1421	11.1597	13.2049	16.0246		C2 DUP	200X		
1422	11.1156	13.1655	15.9315		C1	200X		
1423	11.1151	13.1668	15.9345		C2	ϕ		
1424	11.0708	13.1295	16.0589		1	1X		
1425	11.0939	13.1559	16.4611		2			
1426	11.0814	13.1262	16.1722		3			
1427	11.1746	13.2202	16.9189		4	ϕ		
1428	11.2564	13.3090	16.1721		C2 DUP	200X		
1429	11.1543	14.5244	14.6192		Run OPS adj	1X		
1430	11.1111	14.4996	14.5311					
1431	11.1913	14.6074	14.6900					
1432	11.1505	14.5754	14.6144					
1433	11.1848	14.6067	14.7006					
1434	11.1982	14.6169	14.6537					
1435	11.1020	14.5280	14.6109					
1436	11.1536	14.5817	14.6239					
1437	11.1578	14.6034	14.6991		ϕ	ϕ		

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1438	11.1360	14.5553	14.5969	RUN #3 DILUTION	1x			
1439	11.1251	14.5317	14.6273					
1440	11.1080	14.5031	14.5418					
1441	11.1573	14.5986	14.6687	RUN #4 DILUTION				
1442	11.2408	14.6816	14.7241					
1443	11.0839	14.5209	14.6193					
1444	11.0265	14.4606	14.5041					
1445	11.1305	14.5609	14.6550					
1446	11.0727	14.5029	14.5409					
1447	11.1110	14.5349	14.6346					
1448	11.0398	14.4719	14.5104					
1449	11.1386	14.5778	14.6800					
1450	11.0791	14.5050	14.5462					
1451	11.1221	14.5586	14.6605					
1452	11.0953	14.5290	14.5757					
1453	488.39 ~~11.0352~~	271.66	1074.73	1	1			
1454	489.50 ~~11.0745~~	281.94	1085.10	2 ε	2			
1455	488.96 ~~11.1841~~	287.70	1098.16	3 ε	3			
1456	489.37 ~~11.0831~~	290.14	1100.77	4 ε	4			
1457	49.24 ~~11.1013~~	293.89	1094.56	1 ε	1A			
1458	491.27 ~~11.0944~~	281.08	1085.12	2 ε	2A			
1459	485.28	280.00	1084.80	3 ε	3A			
1460	491.36	289.80	1090.76	4 ε	4A			

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7-30-98 RUN #3 .01 F.12

400ml SM gw

0830 T.B. 9:00

CK 7.02 ✓

pH	MwOH	pH	MwOH	pH	MwOH
		0930		1100	
C1 40ml 8.07	—	C1 8.10	—	C1 8.04	—
DUP 8.07					
C2 40ml 8.17	—	C2 8.10	—	C2 8.09	—
		DUP 8.10			
1 40ml 8.17	1ml	1 6.78	29.5	1 6.03	10.5
				DUP 6.04	10.5
2 40ml 8.18		2 6.79	27.6	2 6.11	11.46
3 400g 8.23	10ml	3 6.26	49.7 0.5/8.5	3 5.93	49.6 0.5/8.
4 40ml 8.23		4 6.26	48.9	4 5.00	49.4
	0905	0935		1105	
C1 400g 8.37	—	C1 8.29	—	C1 8.25	—
DUP 8.37					
C2 400g 8.35	—	C2 8.38	—	C2 8.26	—
		DUP 8.38			
1 400g 8.36	(5.2833g) 5.24g	1 6.67	12.4 0.01/8.11	1 6.38	25.4 0.02/8.
				DUP 6.38	25.3
2 400g 8.36	(5.2818g)	2 6.61	12.4	2 6.32	25.1
3 400g 8.39	(16.00(2g)) 16.9	3 7.00	34.6	3 6.95	37.00.1/8.0
	16.0097				
4 400g 8.40		4 7.03	35.4	4 6.92	37.7

7-3-98

#3	pH 1300	MWay
C1	8.09	—
		—
C2	8.24	
1	6.31	2.37 (1x)
2	6.41	2.55
DUP	6.47	2.56
3	6.35	48.8 .5/8.5
4	6.39	48.7

#4	pH 0[illegible]5	MWay
C1	8.38	—
C2	8.40	—
1	6.71	24.7 .02/8.02
2	6.71	25.2
DUP	6.71	25.2
3	7.19	38.0 .01/8.01
4	7.22	37.7

#3	pH 1500	MWay
C1	8.01	—
		—
C2	8.11	
1	6.[illegible]	1.01 (1x)
2	6.2	1.07
		.5/8.5
3	6.11	49.4
DUP	6.11	49.3
4	6.13	49.5

#1		
C1	8.32	—
C2	8.36	—
1	6.62	25.4 .02/8.02
2	6.60	25.8
3	7.10	38.6 .01/8.01
DUP	7.1[illegible]	38.6
4	7.19	38.[illegible]
	7.00	6.1
		50.9

7/31/98

	0700	5.03
	pH	MWay
C1	7.93	—
C2	7.99	—
1	6.42	0.61 (1x)
2	6.47	.58
3	6.47	47.7 .5/8.5
4	6.21	48.0
DUP	6.21	48.0
C1	8.08	—
C2	8.09	—
1	6.73	25.3 .02/8.02
2	6.72	25.3
3	7.30	37.0 .01/8.01
4	7.32	38.0
DUP	7.31	38.1

7-31-98 0900

#3

	pH	Mass	
C1	8.01	—	
DUP	8.02	—	
C2	8.01	—	
1	6.10	0.45	
2	6.17	0.50	
3	6.04	47.5	.51/2.5
4	6.05	48.1	

#4

	pH	Mass	
C1	8.00	—	
DUP	8.00	—	
C2	8.00	—	
1	6.59	24.9	.02/8.02
2	6.61	25.1	
3	7.13	38.1	.01/8.11
4	7.19	38.1	
		10.6	
	7.00	50.5	

RUN #3 + RUN #4

8-3-98

0900 49.9

	pH	Mnout
7.9		
C1	7.77	
C2	7.83	
DUP	7.84	
1	6.67	1.10
2	6.71	1.08
3	6.44	47.2 / 1.5
4	6.41	47.4

0905

	pH	Mnout
C1	7.91	
C2	7.92	
DUP	7.92	
1	6.86	25.8 .02/2.2
2	6.73	25.2
3	7.35	37.4 .01/1.01
4	7.41	39.9
	7.10	10.3
		50.4

TASK 4b - RUN 7 & RUN 8

8-12-98

Run #7	FUM (g)	GW (g)	TCE (g)	Empty Syringe (g)	Spent TCE Syringe (g)
C1	205	400	.7975	25.3168	26.1194
C2	213	400.0	0.8151	25.3219	26.1373
1	212	400	0.8072	25.3222	26.1282
2	~~205~~	400.0	0.7945	25.3210	26.1301
3	~~205~~ 212	400	0.8055	25.3356	26.1400
4	205	400	0.8077	25.3345	26.1415
Run #8 C1	208	400	0.8117	25.3338	26.1444
C2	221	400	0.8058	25.3327	26.1385
1	209	400	0.8095	25.3327	26.1422
2	207	400	0.8171	25.3327	26.1484
3	206	400	0.8059	25.3313	26.1377
4	209	400	0.8092	25.3318	26.1400
				25.3308	

7.08 ✓ TASK 4b - RUN-7 ← RUN 8 8-13-94 4 gal = 47.4 mg/5 gal =

50.4 ✓

0830 pH	0845 MWOY	0915 pH	0915 MWOY	1045 pH	1045 MWOY
C1 8.00		C1 8.00		C1 7.00	
C1 DUP 8.00					
C2 8.16		C2 8.22		C2 8.15	
		DUP 8.22			
1 8.23	1 ml 4% ~~5.3340~~ DD	1 5.98	~~0.50~~ DD 0.49	1 6.01	0.58
				DUP 6.00	0.59
2 8.27	1 ml 4% ~~5.3336~~ DD	2 6.01	0.48	2 5.94	0.56
3 8.15	10 ml 4% ~~16.0007~~ DD	3 3.12	0.40	3 2.90	0.42
4 8.10	10 ml 4% ~~16.0004~~ DD	4 3.12	0.40	4 2.92	0.43

0830 pH	0850 MWOY	0920 pH	0920 MWOY	1050 pH	1050 MWOY
C1 8.11		C1 8.06		C1 8.05	
C1 DUP 8.11					
C2 8.12		C2 8.08		C2 8.08	
		DUP 8.07			
1 8.12	5.3340g	1 2.76	19.0 .02/8.02	1 2.78	19.4 .02/8.02
				DUP 2.79	19.4
2 8.17	5.3336g	2 2.69	18.2	2 2.71	19.4
			.01/8.01		
3 8.18	16.0007g	3 2.89	34.1	3 2.95	34.6 .01/8.01
4 8.15	16.0004g	4 2.89	33.8	4 3.02	34.4

8-13-98

1245	pH	MWO4⁻	
C1	8.00	—	
C2	8.01	—	
1	5.69	0.30	1x
2	5.81	0.22	
2 DUP	5.81	0.22	
3	2.79	0.33	
4	2.78	0.34	

1445	pH	MWO4⁻	
C1	7.79	—	
C2	7.80	—	
1	5.57	0.39	1x
2	5.70	0.37	
3	2.72	0.47	
3 DUP	2.75	0.48	
4	2.75	0.39	

1250			
C1	7.82	—	
C2	7.85	—	
1	2.65	18.8	.02/8.02
2	2.58	19.0	
2 DUP	2.58	19.0	
3	2.84	34.3	.01/8.01
4	2.93	32.9	

1450			
C1	7.83	—	
C2	7.84	—	
1	2.57	17.8	.02/8.02 ml
2	2.56	18.5	
3	2.86	34.2	.01/8.01 ml
3 DUP	2.86	34.1	
4	2.93	34.3	
	10.01 ✓		10.5 ✓
	4.02 ✓		504 ✓

8-14-98

7.08 ✓

25.2

pH 0200		mwq		pH		mwq
C1	7.51			C1	7.36	
				C1 dup	7.35	
C2	7.45			C2	7.34	
1	5.86	0.52		1	5.68	0.28
2	5.96	0.50		2	5.64	0.15
3	3.16	0.34		3	3.09	0.27
4	3.20	0.29		4	3.23	0.28
4 dup	3.20	0.28				
0205						
C1	7.32			C1	7.28	
				C1 dup	7.30	
C2	7.31			C2	7.35	
1	2.71	16.9 .02/.02		1	3.08	16.6 .02/.02
2	2.43	17.1		2	2.98	16.6
3	2.95	33.3 .01/.01		3	3.34	33.3 .01/.01
4	2.93	33.5		4	3.36	33.2
4 dup	2.94	33.5				
					4.02 ✓	10.3 ✓
					7.09 ✓	50.7 ✓

8-17-98 TABLE 4/5 - ROW 2 & ROW 8

4.02
7.08

25.2

0845

	pH	mM SO_4	
C1	7.85	—	
C2	8.04	—	
Ave	8.04	—	
1	6.15	0.21	Tx
2	5.98	0.30	
3	2.24	0.13	
4	2.77	0.26	

0930

	pH	mM SO_4	
C1	7.88	—	
C2	7.87	—	
Ave	7.88	—	
1	1.90	16.2	.02/8.0ml
2	1.83	15.8	
3	2.23	32.5	.01/8.0ml
4	2.27	33.4	
	4.03	16.3	
	7.09	50.1	

CONTROL WORK SHEET - 1

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SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1524	11.0322	13.0310	15.8988	C1	2000X		30min	
1525	11.0725	12.9330	16.0034	C2				
1526	11.1811	12.9839	15.7173	1				
1527	11.0795	12.9434	15.6728	2				
1528	11.0981	12.9533	15.5695	3				
1529	11.0910	12.9500	16.3196	4				
1530	11.0398	12.8996	15.7950	C1 DUP				
1531	11.1416	12.9808	16.0847	C1	2000X	2000X		
1532	11.1812	12.9795	15.9446	C2				
1533	11.2231	12.9529	15.7634	1	100X			
1534	11.0072	12.7371	15.5219	2	2000X			
1535	11.0748	12.8738	15.5257	3	100X			
1536	11.0851	12.9510	16.0496	4				
1537	11.0933	12.8412	15.7421	C1 DUP	2000X			
1538	11.1507	13.1605	15.7122	C4	2000X		30min	
1539	11.0464	13.0144	15.8194	C2				
1540	11.0302	13.0177	15.7964	1				
1541	11.1262	13.1166	15.9530	2				
1542	11.2401	13.2233	16.2601	3				
1543	10.9575	12.9598	16.0252	4				
1544	11.1362	13.1016	15.8733	C2 DUP	2000X			
1545	11.1245	13.1174	15.9071	C1	2000X			
1546	11.0421	13.0450	15.8270	C2				
1547	11.0892	13.0972	16.2875	1	100X			
1548	11.0818	13.0787	16.0597	2				

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SERIES

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SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1549	11.1377	13.1283	16.2289	100 x	4	T50		
1550	11.1130	13.1034	15.9053	↓	4	↓		
1551	11.0328	13.0493	15.8346	2,000 x	C2 DUP	↓		
1552	11.0248	13.0103	16.0361	2,000 x	C1	T12MGS		
1553	11.0110	13.0002	15.6281	↓	C2	↓		
1554	11.0006	13.0096	15.4744		1			
1555	11.0954	13.1041	15.9298		2			
1556	11.0674	13.0848	15.6906		3			
1557	11.0943	13.1088	16.2029		4			
1558	11.0228	13.0225	15.6594	↓	1 DUP	↓		
1559	11.0803	13.0888	15.7442	2,000 x	C1	T12MGS		
1560	11.1368	13.1493	15.8958	↓	C2			
1561	11.1013	13.1268	16.0198	1X	1			
1562	11.0591	13.0741	16.0910	↓	2			
1563	11.0962	13.1096	16.3994	1x	3			
1564	10.9436	12.9358	16.3105	↓	4			
1565	11.0523	13.0468	15.7171	1X	1 DUP	↓		
1566	11.1332	13.1232	15.8096	2,000 x	C1	T4MGS		
1567	11.0721	13.0609	15.7922		C2			
1568	11.0734	13.0577	15.8798		1			
1569	11.1275	13.1046	16.1281		2			
1570	11.0025	12.9875	15.8580		3			
1571	11.0095	12.9906	16.2930		4			
1572	11.1996	13.1876	16.1032	↓	2 DUP	↓		
1573	11.1105	13.1049	15.7391	2,000 X	C1	T4MGS		

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SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1574	11.0436	13.0356	15.4997	2,000x	C2	T4 liters		
1575	11.1346	13.1301	16.5671	5,000x	1			
1576	10.9804	12.9618	16.2437		2			
1577	11.0078	12.9944	16.5437	1x	3			
1578	11.0371	13.0204	16.6155		4			
1579	11.1603	13.1512	15.9879	5,000x	2 DUP			
1580	11.1649	13.1191	15.7722	2000x	C1	T6 liters		
1581	11.0521	13.0360	15.6195		C2			
1582	11.1637	13.1399	16.0648		1			
1583	11.0008	12.9912	15.7302		2			
1584	11.1401	13.1292	16.0236		3			
1585	11.0908	13.0830	16.4688		4			
1586	11.0144	12.9927	16.1843		3 DUP			
1587	11.2036	13.1692	16.2150	2,000x	C1	T6 liters		
1588	11.1045	13.1003	16.0992		C2			
1589	11.1241	13.1093	17.2371	1x	1			
1590	11.0453	13.0115	16.5518		2			
1591	11.1843	13.1568	16.7927		3			
1592	11.1496	13.1186	17.1110		4			
1593	11.1225	13.0923	16.2445		3 DUP			
1594	11.1618	13.1098	16.3946	2,000x	C1	T2 liters		
1595	11.0618	13.0302	16.5212		C2			
1596	11.1968	13.1569	16.4743		1			
1597	11.0272	12.9912	16.6310		2			
1598	11.0808	13.0448	16.4761		3			

UCN-2138
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1599	11.1240	13.0851	16.2475	2,000 x	4	T 20 hrs		
1600	11.1273	13.0905	16.9962	↓	4 DUP	↓		
1601	11.0837	13.0413	16.7470	2,000 x	C1	T 20 hrs		
1602	11.0785	13.0453	16.5227	↓	C2	↓		
1603	11.0881	13.0519	16.5113	1 x	1	↓		
1604	11.0349	12.9927	16.4213	↓	2	↓		
1605	11.0347	12.9864	16.5451	↓	3	↓		
1606	11.0286	12.9673	15.8678	↓	4	↓		
1607	11.1679	13.1020	17.4522	↓	4 DUP	↓		
1608	11.1082	13.0842	16.1450	2,000 x	C1 (A)	T 24 hrs		
1609	11.0398	13.0603	15.8910	↓	C2 (A)	↓		
1610	11.1438	13.1787	16.4283	↓	1 (A)	↓		
1611	11.0621	13.1016	16.1220	↓	2 (A)	↓		
1612	11.1134	13.1576	16.1571	↓	3 (A)	↓		
1613	10.9296	12.9696	15.8009	↓	4 (A)	↓		
1614	10.9888	13.0284	15.9754	↓	C1 (A) DUP	↓		
1615	11.1229	13.1590	16.1332	2,000 x	C1	T 24 hrs		
1616	11.0567	13.1376	16.5414	↓	C2	↓		
1617	11.0094	13.0952	16.0237	1 x	1	↓		
1618	11.0037	13.0788	16.1400	↓	2	↓		
1619	11.1325	13.2069	16.5726	↓	3	↓		
1620	11.1191	13.1935	16.4130	↓	4	↓		
1621	10.9702	13.0382	16.0380	2,000 x	C1 (A) DUP	↓		
1622	11.0739	13.0568	16.3349	2,000 x	C1 (A)	T 96 hrs		
1623	11.0279	13.0076	16.5358	↓	C2 (A)	↓		

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1624	11.1562	13.1490	16.4955	2000x	1	Tg 4 kts		
1625	11.0839	13.0790	16.5310		2			
1626	11.1167	13.1154	16.5875		3			
1627	11.1041	13.1090	16.6073		4			
1628	11.1597	13.1673	16.3839		C			
1629	11.0890	13.0931	16.7285	200x	C_1	Tg 4 kts		
1630	11.1328	13.1543	16.5598		C_2			
1631	11.1062	13.1274	16.6325	1x	1			
1632	11.0003	13.0107	16.3971		2			
1633	11.0522	13.0592	16.5154		3			
1634	11.2222	13.2406	16.9492		4			
1635	11.0910	13.1020	16.2317	2,000x	C			
1636	11.0419	14.3830	14.7838	500x	C	0.2130		
1637	11.1110	14.4641	14.5067	10x	C			
1638	11.0384	14.4024	14.4968	500x	C			
1639	11.0458	14.3904	14.4383	10x	C			
1640	11.0853	14.4339	14.5303	500x	1			
1641	11.0870	14.4377	14.4804	10x	1			
1642	11.1543	14.5093	14.6110	500x	2			
1643	11.0761	14.4540	14.4948	10x	2			
1644	11.0579	14.4277	14.4977	500x	3			
1645	11.1248	14.4589	14.5058	10x	3			
1646	10.9777	14.3193	14.4227	500x	4			
1647	11.0516	14.3876	14.4301	10x	4			

UCN-2133
(2 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1648	10.9596	14.2839	14.3787	10	C1 BOD	0.280 BOD		
1649	11.1279	14.4877	14.5307		C1 BOD			
1650	11.0289	14.3359	14.4290		C2 BOD			
1651	11.1690	14.5228	14.5692		C2 BOD			
~~1652~~	~~11.2210~~	~~14.2909~~			1 B1			
~~1653~~	~~11.0870~~	~~14.4384~~			1 B2			
1654	10.9547	14.2909 ~~14.4436~~	14.3919		2 B1			
1655	11.1128	14.4384 ~~14.5443~~	14.4804		2 B2			
1656	11.1010	14.4436 ~~14.2537~~	14.5300		3 B1			
1657	11.2080	14.5443	14.5886		3 B2			
1658	10.9471	14.2537	14.3558		4 B1			
1659	10.9904	14.3077	14.3468		4 B2			

UCN-2133
(3 9-60)

6/11 60

BWD

CONTROL WORK SHEET - 1

TASK 46-RUN 9

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

ANALYZED FOR

SERIES

SAMPLE CODE	g bottle + Cap	3 ml HEX ~~ORIGINAL SAMPLE SIZE~~	HEX + SAMP. ~~DILUTED TO~~	g c vials ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
554	11.2618	14.6067	19.6273	20X				
555	11.1777	14.5359	19.4582	20X				
556	11.1965	13.2522	16.7079	20X				
557	11.0571	13.0710	16.6439	20X				
558	11.0273	13.0441	16.5575	20X				
559	11.0663	13.0912	16.7512	20X				
560	10.9764	13.0134	16.6184	20X				
561	10.9569	12.9987	16.5074	20X				
562	11.0217	13.0611	16.8233	20X				
563	10.9617	13.0052	16.1326	20X				
564	11.0066	13.0581	16.5238	20X				
565	11.0209	13.0656	15.9185	20X				
566	10.9730	13.0241	16.5627	20X				
567	11.0207	13.0710	16.6749	1X				
568	10.9009	12.9405 ~~12.9914~~	16.7800	1X				
569	10.9396	12.9924	16.7784	1X				
570	11.0498	13.0990	16.5855	1X				
571	10.8957	12.9415	16.4429	20X				
572	10.9539	12.9718	16.6000 ~~12.8779~~	20X				
573	10.9771	12.8979	16.4780 ~~13.0556~~	20X				
574	11.0497	13.0771	16.1019	20X				
575	11.0121	13.0556	16.5764	1X				
576	10.9972	12.9274	16.2264	1X				
577	10.9288	12.9835	16.8290	1X				
578	10.9411	12.9902	16.8200	1X				

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE	g bottle + Cap	Hexane	+ Sample Hexane +	ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
579	10.9189			12.9664	16.4435	20X				
580	10.8781			12.8912	16.7149	20X				
581	10.9688			12.9731	16.7304	20X				
582	10.9284			12.9436	16.6823	20X				
583	10.8970			12.9170	15.7366	1X				
584	10.9041			12.9399	16.4127	1X				
585	10.9694			12.9990	16.8080	1X				
586	10.8463			12.8734	16.3652	1X				
587	10.8541			12.8785	16.5819	1X				
588	10.9397			12.9622	~~12.99~~ 16.7026	20X				
589	10.9023			12.9281	16.6582	20X				
590	10.9623			12.9912	16.3607	20X				
591	10.9734			13.0159	16.6906	1X				
592	10.9872			13.0313	16.2045	1X				
593	10.9575			12.9970	16.9021	1X				
594	10.9242			12.9753	16.6806	1X				
595	10.9388			12.9931	16.8545	1X				
596	10.9737			13.0007	16.4667	20X				
597	10.9894			13.0059	16.6537	20X				
598	10.9155			12.9335	16.6990	20X				
599	10.9655			12.9878	16.7797	1X				
600	10.9118			12.9390	16.4149	1X				
601	11.9922			13.0059	15.8916	1X				
602	10.9290			12.9746	16.7937	1X				
603	10.8623			12.9043	16.7882	1X				

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

ANALYZED FOR

SERIES g1 HEX + BR HEX + SMPLE BR

SAMPLE CODE	bottle + Cap	ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
604	10.9221	12.9539	16.7670	20X				
605	10.9806	13.0126	16.9193	20X				
606	10.9751	13.0172	16.8882	20X				
607	10.9819	13.0261	16.7745	1X				
608	10.9822	13.0281	16.6708	1X				
609	10.9735	13.0397	16.6400	1X				
610	10.8657	12.9257	16.6770	1X				
611	10.9980	13.0570	16.0013	1X				
612	10.9648	12.9844	13.0781	Sc1-1 1X				
613	10.9726	13.0131	13.0607	Sc1-2				
614	10.9378	12.9859	13.0849	#1-1				
615	10.9363	12.9793	13.0257	#1-2				
616	11.0015	13.0369	13.1242	#2-1				
617	10.9452	12.9950	13.0367	#2-2				
618	10.8546	12.8956	12.9903	#3-1				
619	10.8880	12.9259	12.9680	#3-2				
620	10.9560	13.0077	13.1034	#4-1				
621	10.9826	13.0267	13.0660	#4-2				
622	10.9493	12.9939	13.0924	C2-1				
~~623~~ 623	11.0094	13.0537	13.0985	C2-2				
C1 624	492.8	HEX + BR only 268.01	1152.55 ~~964.69~~					
1 625	491.8	272.49	1132.55					
2 626	492.4	262.07	1151.24 ~~1162.47~~					
3 627	492.4	283.00	1141.95					
4 628	492.8	284.42	1179.68					

UCN-2138
(8 9-60)

	6-1-98 Flask	Dried Sludge	6-2-98 ICE spk. soln	Added Kmno4
SC1	206.6	97.0g	(388) 388	—
C1	224.7	—	(360) 361	—
C2	220.2	—	(360) 360	—
0.012 1	208.5	92.6g	(369) 368	(4%) 0.92ml
2	214.6	97.2g	(389) 389	0.973ml
0.12 3	206.1	89.9g	(360) 353	9.5ml
4	207.9	96.6g	(384) 387	10ml

6-3-98 10 ppm

	0840 pH	KMnO4 mmol	0915 pH	KMnO4	1045 pH	MNa4
SC1	6.92	0845 START	6.88		6.86	
(SC1 DUP)	6.93					
C1	8.17		8.07		8.13	
			(C1D) 8.08			
C2	8.22		8.20		8.22	
					(C2D) 8.23	
1	6.98		6.93	+0.37 1X	7.00	+0.51 1X
2	7.00		7.02	+0.18	7.06	+0.345
3	7.04		7.11	-0.28	7.29	-0.044
4	7.23		7.22	16.6 10X 0.7/7.0	7.28	1P.C.9
	7.04					

	1245 pH	MnO_4		1445 pH	MnO_4	
C_1	6.93	—		6.94	—	
C_1	8.13	—		8.14	—	
C_2	8.22	—		8.23	—	
1	6.93	+0.61	1X	6.98	+0.78	1X
1 DUP	6.93	+0.63		~~6.98~~		
2	7.03	+0.48		7.00	+0.52	
2 DUP				7.00	+0.53	
3	7.30	+0.02		7.31	+0.16	
4	7.44	−0.26		7.49	−0.14	

pH CONTROLS	MnO_4
7.05	9.78 ✓ 49.9 ✓

6-4-98

	0510 pH	MnO_4		0845 pH	MnO_4	
SC_1	~~7.00~~ 6.93	—		6.98	—	
C_1	8.16	—		8.12	—	
C_2	8.22	—		8.23	—	
1	7.00	1.56	1X	6.99	1.50	1X
2	7.07	1.20		7.06	1.05	
3	7.35	0.89		7.34	0.90	
3 DUP	7.36	0.88				
4	7.49	+0.43		7.49	+0.43	
	7.09 ✓	50.1 ppm ✓	4 DUP	7.50	+0.42	

CONTROLS

pH	MnO_4
7.04 ✓	10.2 ✓ 50.3 ✓

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR GC

SERIES

ANALYZED FOR +sample Sample Time

SAMPLE CODE		ORIGINAL SAMPLE SIZE	~~DILUTED TO~~ Wt/g	ALIQUOT SIZE g	ABSORBANCE g	CURVE READING	DILUTION FACTOR	RESULTS /ml
			Sample Vial	+5M Hexane				
420		initial spike	11.1282	19.4892	19.4800	Initial Spk ~~45-R10-1~~		20x
421		initial spike dup	11.1643	19.5536	19.5256	Dup 2		20x
422	0 min	2	11.0274	13.043	16.483	45-R10-1	09:30	.05/ml 20x
423		~~2~~ 2 dup	11.2602	13.283	16.929	-2		
424		~~2~~ 3	10.9811	13.007	16.497	-3		
425		~~2~~ 4	11.1572	13.174	16.720	-4		
426		~~2~~ 5	11.0621	13.026	16.536	-5		
427		~~2~~ 6	11.1766	13.198	16.632	-6		
428		~~2~~ 6 dup	11.2107	13.234	15.837	C1 Dup	10:04	
429	30 min	2 dup	11.2369	13.262	16.874	45-R10-1	09:40	20x .05/ml
430		~~3~~ 2	11.0759	13.116	16.002	-2		
431		~~4~~ 2 dup	11.0880	13.105	16.935	-3		1x
432		~~5~~ 3	11.1546	13.178	16.929	-4		
433		4	11.0842	13.102	16.829	-5		
434		5	11.0989	13.123	16.868	-6		
435		6	11.1956	13.220	16.166	45-R10-2 Dup		20x .05/l
436	2 hrs.		11.2211	13.160	16.664	45-R10-1	11:15	20x
437		2	11.1812	13.195	16.686	-2		
438		3	10.9960	13.012	16.501	-3		1x
439		3 dup	11.0946	13.111	17.150	-4		
440		4	11.1649	13.188	17.130	-5		
441		5	11.0842	13.105	17.394	-6		
442		6	11.1239	13.142	16.320	45-R10-3 Dup		1x
443	4 hrs.		11.2110	13.153	16.693	45-R10-1	13:15	20x
444		2	11.0496	13.069	16.782	-2		

UCN-2133 (3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR tSample Sample Time GC DF

SAMPLE CODE		ORIGINAL SAMPLE SIZE	~~DILUTED TO~~ Vial Wt, g	tHexane ALIQUOT SIZE	ABSORBANCE	CURVE READING	~~DILUTION FACTOR~~	RESULTS /ml
445	4 hrs.	3	11.1182	13.139	17.000	45-R10-3		1x
446		4	11.1786	13.202	17.092	-4		
447		4 dup	11.1380	13.157	16.992	-5		
448		5	11.2186	13.239	17.248	-6		
449		6	11.2670	13.291	16.050	-4Dup		
450	6 ~~20~~ hrs.		11.1807	13.204	16.835	46R10-4Dup		20x
451		2	11.1689	13.204	16.904	-2		
452		3	11.0750	13.109	17.216	-3		1x
453		4	11.0821	13.115	16.932	-4		
454		5	11.0314	13.069	17.076	-5		
455		5 dup	11.2438	13.275	17.253	-6		
456		6	11.1091	13.144	16.454	-5Dup		
457	20 ~~24~~ hrs.		11.2086	13.163	16.576	46R10-1		
458		2	11.1875	13.207	16.788	2		
459		3	11.1586	13.169	16.992	3		
460		4	11.1154	13.124	16.927	4		
461		5	11.2497	13.256	17.223	5		
462		6	11.0256	13.043	16.904	6		
463		6 dup	11.0733	13.075	16.292	-6Dup		
464	24 hr		11.2040	13.209	16.802	48-R10-1		
465			11.1138	13.114	16.836	-2		
466			11.1687	13.170	17.005	-3		(R)
467			11.1839	13.178	16.920	-4		
468			11.1593	13.146	17.034	-5		
469			11.1139	13.117	17.275	-6		

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR Vial Hexane Sample Sample

SAMPLE CODE		ORIGINAL SAMPLE SIZE	~~DILUTED TO~~ Wt, g	~~ALIQUOT SIZE~~	~~ABSORBANCE~~	~~CURVE READING~~	DILUTION FACTOR	RESULTS /ml
470	24 hr		11.1090	13.121	15.957	48-210-1 POST		
471			11.1826	14.397	14.485	102301		
472			11.1264	14.463	14.502	102302		
473			11.0593	14.402	14.493	202301		
474			11.0464	14.396	14.361	202302		
475			11.1426	14.482	14.576	302301		
476			11.1836	14.540	14.580	302302		
477			11.1640	14.515	14.612	402301		
478			11.0961	14.454	14.496	402302		
479			11.0693	14.415	14.508	502301		
480			11.0940	14.441	14.484	502302		
481			11.1997	14.540	14.635	602301		
482			11.1737	14.525	14.563	602302		
483			492.3 ~~11.0945~~	847.0	1098.2	48-210-3 POST		
484			492.6 ~~11.0403~~	849.6	1074.3	48-210-4 POST		
485			492.4 ~~11.2558~~	Hex only 281.4	1147.8	48-210-5 POST		
486			491.7 ~~11.1812~~	+solvent 870.8	1098.8	48-210-6 POST		

UCN-2133
(3 9-60)

23.5 ml / 2556.4 ml G.W. = 10 ppm SPK SOLUTION

5-27-98 VIAL VIAL + HEX VIAL + HEX + EXT START 5-27-98 0925

10 ppm EXT. ①

(DUP) ②.

REACTOR	FLASK	SLUDGE	10 ppm G.W. ADDED	TRIMOX ADDED
C_1	260.5	—	360.2	—
C_2	258.4	—	363.6	—
1	245.3	90.69	361.8	4.78 g (4.78)
2	250.9	90.20	360.8	4.79 g (4.765)
3	243.3	90.02	362.6	14.5 g (14.52)
4	244.3	90.04	363.4	14.54 (14.54)

EQUIB. TIME START 5-27-98 0936

STOP 5-27-98 0930

5-28-98

T_0 = 0930

PRE MWOY TIME	pH 0930		T_{30} = 1005 30 min pH	MWOY	$T_{2\,hrs}$ 1135 pH	MWOY
1 (1)	8.13	C_1	8.08	—	C_1 8.06	—
(1) DUP	8.14					
2 (2)	8.14	C_2	8.18	—	C_2 8.16	—
		C_2 DUP	8.17	—		
3 (1)	7.11	1	7.23	22.1	7.53	13.3
					1 DUP 7.54	13.4
4 (2)	7.23	2	7.24	12.1	2 7.51	9.50
5 (3)	7.32	3	7.36	17.5	3 7.69	17.5
6 (4)	7.18	4	7.31	16.1	4 7.60	15.5
				(25.1 ✓)	OK 7.00 ✓	

$T_{4\,hrs}$ = 1335	pH	MWOY	$T_{6\,hrs}$ 1535	pH	MWOY
1 C_1	8.06	—	C_1	8.08	—
2 C_2	8.17	—	C_2	8.16	—
3 1	7.74	17.00	1	7.79	12.9
4 2	7.70	14.3	2	7.80	11.0
(2 DUP)	7.69	14.4			
5 3	7.77	14.5	3	7.81	16.5
			(3 DUP)	7.81	16.4
6 4	7.77	15.4	4	7.81	15.4
			OK 7.00 ✓		CR STDS 10.0 ✓

?

50.2 ✓

5-29-98

0710

	pH	MnO4	
C_1	8.04	—	
C_2	8.15	—	
1	7.94	-0.17	1X
2	7.96	-0.21	1X
3	7.99	14.5	.02/20.02ml
4	8.00	14.5	
4 DUP.	8.00	14.5	
CK	7.00 ✓		

0935

	pH	MnO4	
C_1	8.06	—	
C_1 DUP	8.06		
C_2	8.14	—	
1	7.95	-0.17	1X
2.	7.95	-0.21	1X
3	7.93	14.5	.02/20.02ml
4	7.94	14.0	
	-		
CK	7.00 ✓	10 ✓	49.9 ✓

6-11-98 ~~0845~~

	pH		pH	MnO4-		pH	MnO4-	
C1	7.98	C1	8.00	—	C1	8.01	—	
C1 DUP	7.99							
C2	8.08	C2	8.10	—	C2	8.10	—	
		C2 DUP	8.09	—				
1	7.21	1	7.13	0.04	1	7.10	0.50	1X
					1 DUP	7.10	0.49	
2	7.20	2	7.20	0.27	2	7.16	0.36	
3	7.11	3	7.08	45.4	3	7.25	0.04	2X
4	7.00	4	7.02	0.07	4	7.23	0.12	1X
C1 STD	10.01 ✓	MnO4 42 EK		25.0 ✓				
		50.1 (800X)						

1245 1445

6-12-98 0520

	pH	MnO4-		pH	MnO4-		pH	MnO4-
							9.98 ✓	
C1	7.99	—	C1	8.01	—	C1	8.07	50 ✓ 50.3
C2	8.10	—	C2	8.11	—	C2	8.13	—
1	7.11	0.34	1	7.13	0.30	1	7.20	0.99
2	7.19	0.31	2	7.20	0.25	2	7.26	0.84
2 DUP	7.17	0.32						
3	7.31	0.17	3	7.30	0.35	3	7.40	0.97
			3 DUP	7.31	0.35			
4	7.26	0.20	4	7.28	0.24	4	7.38	0.81
				10.02 ✓	10.04 ✓	4 DUP	7.38	0.82
					50.0 ✓			

0845

	pH	MnO4-
C1	8.03	—
C1 DUP	8.12	—
C2	8.19	—
1	7.32	0.97
2	7.39	1.03
3	7.52	0.98
4	7.55	1.14
	10.03 ✓	10.4 ✓
		50.6 ✓

6-8-98

	FLASK	SLUDGE	(TARE) GW	GW ACTUAL	SPK (0930) KMnO4
C1	208 g	—	(360)	~~361~~ 360	
C2	221.4 g	—	(360)	363	
1	210 g	94.2 g	(377)	379	0.948 ml
2	215.7 g	95.2	(381)	381	0.953 ml
3	206.5 g	93.7	(375)	376	~~9.4~~ ml 9.65 ml
4	208.4 g	90.6	(362)	365	9.4 ml

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

ANALYZED FOR

SERIES

SAMPLE CODE	BTL	BTL+ATX	SAMPLE + BTL+ATX	DIL.	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
		~~ORIGINAL SAMPLE SIZE~~	~~DILUTED TO~~	~~ALIQUOT SIZE~~				
696	10.9963	14.32~~63~~ 17	19.3207	200X				
697	10.8979	14.24~~75~~ 54	19.2450	200X				
698	11.0422	13.0580	15.9510	200X				
699	11.0783	13.0944	16.8382	↓				
700	10.9558	12.9719	16.4581	200X				
701	10.9820	13.0032	16.2211					
702	11.0121	13.0180	16.7482					
703	10.9276	12.9477	16.7017	↓				
704	10.8960	12.9190	16.5352	200X				
705	10.9743	12.9921	16.6515	200X				
706	10.9544	12.9781	15.9370	200X				
707	10.9287	12.9506	16.3368	200X				
708	11.0264	13.0456	16.5151					
709	10.9823	13.0051	16.7274					
710	10.8722	12.9014	16.4048	↓				
711	10.9635	12.9918	16.6573	200X				
712	11.0077	13.0234	16.4409	200X				
713	10.9567	12.9660	16.4767	200X				
714	10.9627	12.9646	16.6199	200X				
715	10.9892	13.0127	16.7455					
716	10.9697	12.9979	16.5563					
717	10.90~~78~~ 3	12.9354	16.3917	↓				
718	10.9302	12.9571	15.9703	200X				
719	11.0133	13.0412	16.6151	200X				
720	10.9046	12.9255	16.4863	↓				

UCN-2138
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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
721	10.9048	12.9358	16.4556	20x				
722	10.9703	13.0004	15.9719					
723	10.9310	12.9658	16.5502					
724	10.9659	13.0042	16.3987					
725	10.9878	13.0241	16.4455					
726	10.9021	~~15.9719~~ 12.8522	16.5334	200X				
727	10.9167	12.9280	16.5410	200X				
728	10.9387	12.9611	16.5739	20				
729	10.9874	13.0200	16.8709					
730	10.8692	12.9001	16.1821					
731	10.8988	12.9343	15.8348					
732	10.9061	12.9398	16.2302					
733	10.7589	12.7780	16.2775	200X				
734	11.0050	13.0167	16.6139	200X				
735	10.8926	12.9019	16.3837	20 X				
736	10.9118	12.9370	16.3664					
737	10.9662	12.9890	16.1220					
738	10.9897	13.0172	16.1672					
739	10.9138	12.9415	15.6965					
740	10.9580	12.9665	16.3624	200 X				
741	10.9864	12.9905	16.6041	200X				
742	10.9785	13.0031	16.1987	20X				
743	10.8769	12.9077	15.8403					
744	10.8671	12.8981	16.2021					
745	11.0593	13.0887	16.3354					

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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE	g	ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
746	10.8167	12.8245	15.9914	200X				
747	10.9961	14.4247	14.5258	1X				
748	10.9529	14.3839	14.4290					
749	10.9716	14.4117	14.5088					
750	10.9671	14.4017	14.4404					
751	11.0107	14.4394	14.5320					
752	10.9900	14.4219	14.4691					
753	10.7970	14.1530	14.2431					
754	10.9886	14.4097	14.4534					
755	10.9526	14.3750	14.4430					
756	11.0068	14.4244	14.4660					
757	10.9488	14.3529	14.4481					
758	10.8824	14.3022	14.3455					
#1 759	492.68	276.42	1142.33					
#2 760	492.99	278.90	1151.58					
#3 761	492.99	276.36	1163.00					
#3 762	437.31	254.85	1067.09					

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4b RUN 11

12 Jun 98 13:10 ALPHA/BETA - 1.07 Page #1
Protocol #:60 ET DPM User : RDC

Time: 30.00
Data Mode: Efficiency Tracing DPM
Background Subtract: 1st Vial

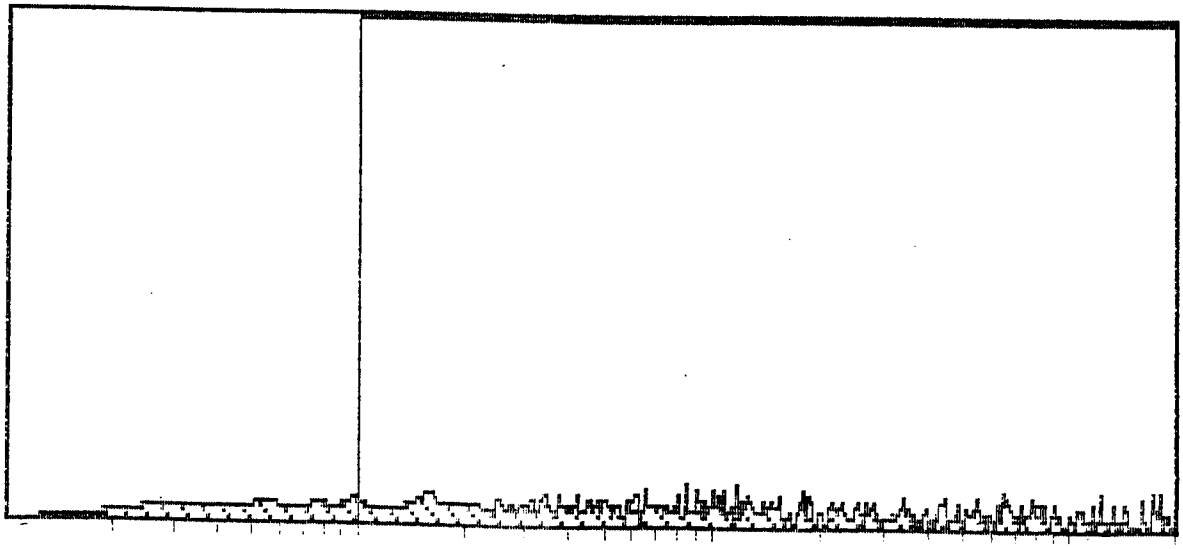
	LL	UL	LCR	2S%	BKG
Region A:	0.0 -	2000	0	0.0	40.10

Quench Indicator: tSIE
Ext Std Terminator: Count
Error = %error/100 x BQ/VIAL
Coincidence Time(ns): 18
Delay Before Burst(ns): Normal
Protocol Data Filename: C:\SPECTRA\PROT.DAT
Count Data Filename: C:\SPECTRA\SDATA60.DAT
Spectrum Data Drive & Path: C:\SPECTRA

S#	TIME	CPMA	DPM1	A:2S%	BQ/VIAL	ERROR	tSIE	FLAG
WARNING: USER HAS MODIFIED COUNT CONDITIONS								
1	30.00	40.10		5.77	0.000	0.000	625.70	B

Log SPECTRUM PLOT

369.1 Y Log maximum Protocol No. 60 Sample No. 1 Repeat 1



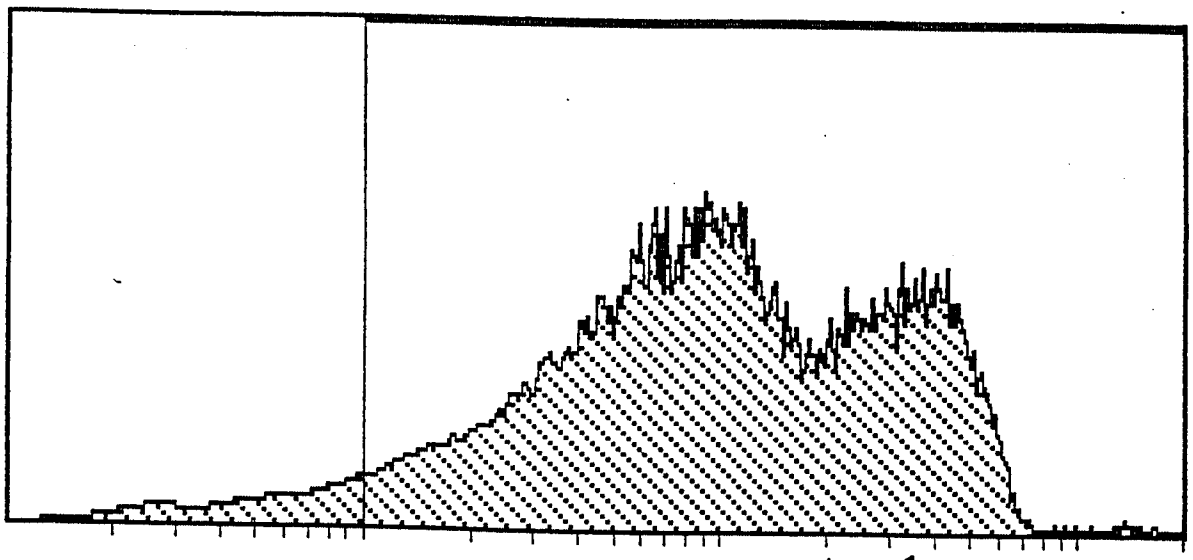
2000

Region	LL	UL	CPM
A:	0.0	2000	40.1
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

2 30.00 1095.97 1157.56 1.14 19.293 0.220 241.05

Log SPECTRUM PLOT

1424.8 Y Log maximum Protocol No. 60 Sample No. 2 Repeat 1



std RNSL 020A
186 Bq/ml

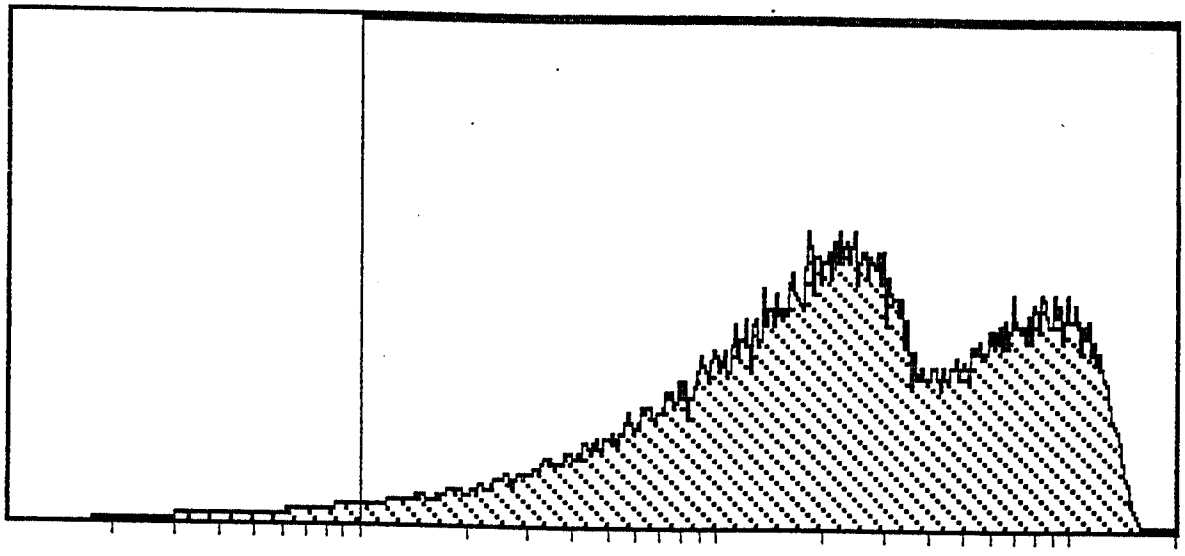
Region	LL	UL	CPM
A:	0.0	2000	1136.1
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

$\frac{19.3}{0.1} = 190$ Bq/ml
±10.

3 30.00 1682.83 1738.74 0.91 28.979 0.264 604.82

Log SPECTRUM PLOT

2524.2 Y Log maximum Protocol No. 60 Sample No. 3 Repeat 1



1 keV 1897 282 BQ/kg 2000

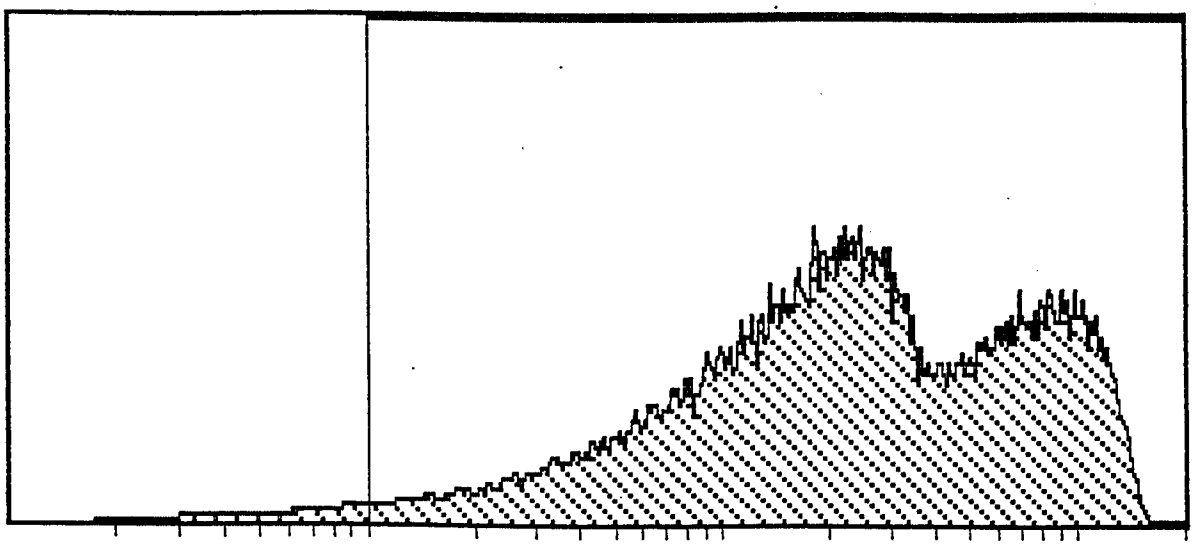
Region	LL	UL	CPM
A:	0.0	2000	1722.9
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

$\frac{29.0}{0.1}$ = 290 BQ/l ±10

3 30.00 1682.83 1738.74 0.91 28.979 0.264 604.82

Log SPECTRUM PLOT

2524.2 Y Log maximum Protocol No. 60 Sample No. 3 Repeat 1



1 keV 1897 282 BQ/kg 2000

$\frac{29.0}{0.1}$ = 290 BQ/ ±10

Region	LL	UL	CPM
A:	0.0	2000	1722.9
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

4 30.00 0.00 43.42 0.00 0.724 0.000 633.19

Log SPECTRUM PLOT

324.6 Y Log maximum Protocol No. 60 Sample No. 4 Repeat 1

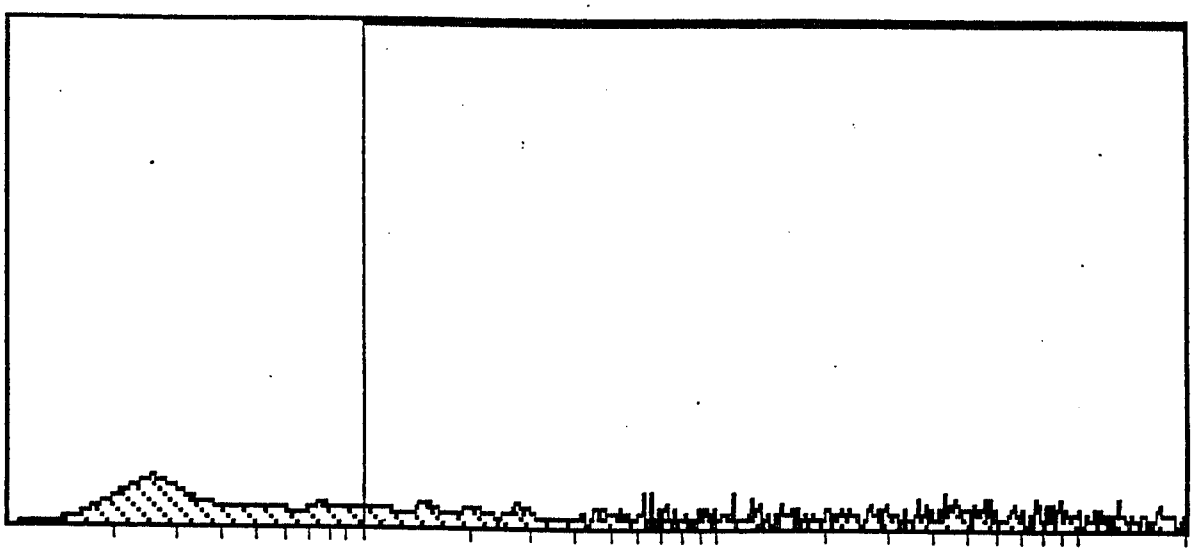
1 keV 2000

Region	LL	UL	CPM
A:	0.0	2000	39.8
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

5 30.00 20.73 73.47 17.69 1.225 0.217 629.42

Log SPECTRUM PLOT

515.4 Y Log maximum Protocol No. 60 Sample No. 5 Repeat 1



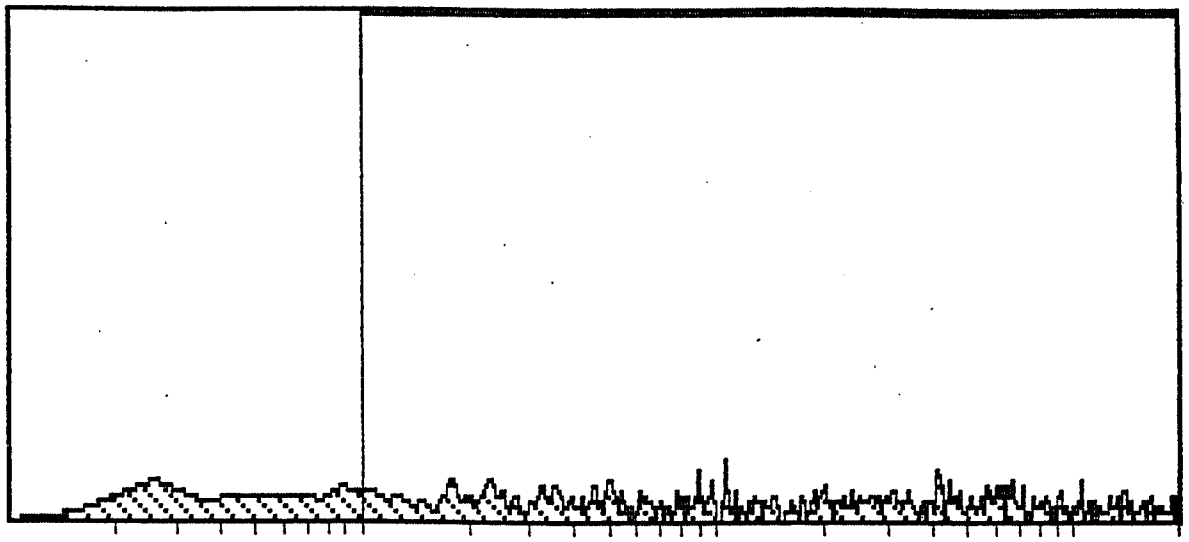
1 keV 2000

Region	LL	UL	CPM
A:	0.0	2000	60.8
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

6 30.00 12.20 60.79 28.77 1.013 0.291 636.63

Log SPECTRUM PLOT

367.0 Y Log maximum Protocol No. 60 Sample No. 6 Repeat 1



1 keV 2000

Region	LL	UL	CPM
A:	0.0	2000	52.3
B:	0.0	0.0	0.0
C:	0.0	0.0	0.0

7-15-98

TASK 4b-RUN-14

1007	pH	0845 MnO_4^-	095	pH	MnO_4^-	D.f	1045	pH	MnO_4^-	Df
C1	8.05	—	C1	8.20	—		C1	8.14	—	
C1D	8.05	—								
C2	8.24	—	C2	8.21	—		C2	8.15	—	
			C2D	8.20	—					
1	6.64	4.7929 g	1	6.37	11.91	.01/8.01 ml	1	6.51	16.4	.01/8.01
							1D	6.52	16.4	
2	6.70	4.8447 g	2	5.98	11.9		2	6.35	25.8	
3	6.73	14.6416 g	3	6.24	17.1	0.1/8.01 ml	3	6.40	35.8	02/8.01
4	6.63	14.6020 g	4	6.29	29.4		4	6.48	53.3	

	pH	0950 MnO_4^-	0920	pH	MnO_4^-	1050	pH	MnO_4^-	
C1B	8.25	—	C1B	8.10	—	C1B	8.01	—	
C1BD	8.25	—			—				
C2B	8.24	—	C2B	8.16	—	C2B	8.13	—	
			C2BD	8.16	—				
1B	6.58	.998 ml	1B	6.53	1.90	1B	6.51	2.10	1
						1BD	6.51	2.10	
2B	6.62	.905 ml	2B	6.66	1.70	2B	6.64	1.87	
3B	6.51	9.2 ml	3B	6.44	0.97	3B	6.51	1.44	
4B	7.35	9 ml	4B	6.48	0.57	4B	6.80	0.56	

TASK 4b-R13

7-15-98

	pH (Fuc)	MnO_4^-	p.f.		pH (1245)	MnO_4^-	p.f.	
C_1	8.01	—		C_1	8.21	—		
C_2	8.01	—		C_2	8.18	—		
1	6.51	8.46	.04/8.04	1	6.62	0.68		1x
2	6.48	16.3		2	6.63	22.9	1/8.1	4x
2D	6.49	16.3						
3	6.46	35.0	.02/8.02	3	6.74	38.7	0.02/8.02	4.01
				3DUP	6.74	38.6		
4	6.53	48.9		4	6.72	40.6		
	(1850)				(1450)			
C_{1B}	8.12	—		C_{1B}	8.03	—		
C_{2B}	8.14	—		C_{2B}	8.14	—		
1B	6.47	2.27	1x	1B	6.61	2.44	1x	
2B	6.65	2.00		2B	6.73	2.19		
2BDUP	6.65	2.00						
3	6.51	1.57		3B	6.66	1.65		
				3BDUP	6.65	1.65		
4	6.64	0.50		4	7.08	0.47		
				(1445)	10.06 ✓	10.1 ✓		
						50.3 ✓		

7-26-98

10.07 0700 49.5

	pH	MnO_4^-	
C1	8.13	—	
C2	8.17	—	
1	6.74	0.44	1x
2	6.73	0.71	
3	6.82	31.9	.02/8.02
4	6.83	27.7	
4 DUP	6.83	27.6	
0705			
C1	8.00	—	
C2	8.13	—	
1	6.71	2.72	1x
2	6.80	2.50	
3	6.80	2.07	
4	7.17	0.62	
4 DUP	7.17	0.62	

0845

	pH	MnO_4^-	
C1	8.06	—	
C1D	8.07	—	
C2	8.12	—	
1	6.83	0.76	1x
2	6.74	0.39	
3	6.83	31.1	.02/8.02
4	6.84	25.6	
0850			
C1	8.14	—	
C1D	8.14	—	
C2	8.15	—	
1	6.71	2.74	1x
2	6.77	2.43	
3	6.72	2.03	
4	7.13	0.64	
		10.1	
		50.4	

7-27-98

7.09 0945 49.2

	pH	MnO_4^-	
C1	8.08	—	
C2	8.23	—	
C2D	8.23	—	
1	7.00	5.87	1x
2	7.00	3.36	
3	7.14	43.4	.02/8.02
4	7.15	18.3	
0950			
C1	8.27	—	
C2	8.29	—	
C2D	8.29	—	
1	6.93	~~3.92~~ 4.92	1x
2	~~7.13~~	~~4.92~~ 3.89	
3	7.08	4.76	
4	7.08	1.84	
	7.06	10.1	
		50.0	

7-14-98

1% + 32

	FLASK	SLUDGE	THEORETICAL GW ADDED	PUMPING	ACTUAL GW	THEORETICAL TM PPTCE	PPTCE ADDITION: SYRINGE EMPTY	PPTCE ADDITION: SYRINGE PPTCE	PPTCE ADDED
C_1	209	—	360	—	360	.504	40.3446	41.0807	
C_2	221	—	↓	—	360	↓	36.8479 ~~40.3449~~	37.6089	
1	211	90.4 (4.7929)	362	4.772	363	.508	36.8707	37.6065	
2	209	91.4 (4.8447)	366	4.844	367	.514	36.8698	37.5876	
3	209	91.5 (14.4416)	366	4.64	366	.572	36.8690	37.5886	
4	208	91.3 (14.6020)	365	4.66	365	.506	36.8679	37.6019	
	.01% + .1%								
C_{1A}	204	—	360	—	360	.504	36.8552	37.6098	
C_{2A}	212	—	↓	—	362	.502	36.8663	37.5929	
1A	212	91.2	365	9.4	367	.572	36.8662	37.6026	
2A	205	90.4	362	.905	362	.508	36.8658	37.5871	
3A	210	91.8	367	9.2	368	.575	36.8648	37.5924	
4A	205	90.1	360	9	360	.504	36.8648	37.5901	

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE	Bt. g	ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1098	11.0544	13.0624	16.0796	2000X	C_1	10		
1099	11.1781	13.1809	16.1501		C_2			
1100	11.1286	13.1498	16.5438		1			
1101	11.1260	13.1443	16.8238		2			
1102	11.1658	13.1988	16.9804		3			
1103	11.1372	13.1658	16.7259		4			
1104	11.0928	13.1211	16.6416		C_1 dup			
1105	11.1399	13.1710	16.7794		C_1 A	10		
1106	11.1863	13.2134	17.0229		C_2 A			
1107	11.1273	13.1567	16.7118		1A			
1108	11.2127	13.2431	16.7968		2A			
1109	11.0731	13.0974	16.7243		3A			
1110	11.1416	13.1800	16.9710		4A			
1111	11.0921	13.1297	16.0821		C_1 A dup			
1112	11.1851	13.1409	16.9858		C_1	30		
1113	11.1714	13.1709	16.5176		C_2			
1114	11.1863	13.1999	16.5563		1			
1115	11.1678	13.1785	16.2315		2			
1116	11.2146	13.2390	16.1204		3			
1117	11.1395	13.1584	16.5786		4			
1118	11.1516	13.1762	16.3387		C_2 dup			
1119	11.1314	13.1546	16.4926		C_1 A	30		
1120	11.2194	13.2478	16.4082		C_2 A			
1121	11.1513	13.1751	16.2748		C A			
1122	11.0899	13.1087	16.1573		2 A			

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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1123	11.1637	13.1928	16.2665		3 A			
1124	11.1678	13.2082	16.3455		4 A			
1125	11.1545	13.1851	16.0972		C_2 A dup			
1126	11.1726	13.1895	16.1759		C_1	Tzm		
1127	11.1133	13.1320	15.7677		C_2			
1128	11.0514	13.0558	15.7703		1			
1129	11.1267	13.1425	15.7383		2			
1130	11.1730	13.1920	16.5306		3			
1131	11.2124	13.2249	16.8908		4			
1132	11.1845	13.2036	16.2175		1 dup			
1133	11.0830	13.1108	16.1518		C_1 A	Tzm		
1134	11.2187	13.2453	16.4517		C_2 A			
1135	11.1442	13.1805	16.1534		1 A			
1136	11.0836	13.1120	16.5368		2 A			
1137	11.2000	13.2345	16.0459		3 A			
1138	11.0809	13.1058	16.4671		4 A			
1139	11.2116	13.2457	16.1827		1 A dup			
1140	11.1253	13.1414	15.9286 ~~13.1808~~		C_1	Tu		
1141	11.1203	13.1532	16.4368		C_2			
1142	11.1419	13.1781	16.8499		1			
1143	11.1530	13.1971	16.4442		2			
1144	11.1183	13.1642	16.7746		3			
1145	11.1407	13.1845	16.9434		4			
1146	11.1519	13.2036	16.1893		2 dup			
1147	11.1228	13.1708	16.2760		C_1 A	Tu		

UCN-2133
(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1148	11.0297	13.0877	16.1463		C_2A			
1149	11.0532	13.1138	16.7562		1A			
1150	11.1567	13.2785	16.0731		2A			
1151	11.1988	13.2652	16.9933		3A			
1152	11.0868	13.1553	16.8067		4A			
1153	11.1023	13.1702	16.9347		2A dup			
1154	11.1438	13.1658	16.3577		C_1	1/6		
1155	11.1986	13.2146	16.3165		C_2			
1156	11.1415	13.1732	16.4694		1			
1157	11.1168	13.1541	16.1889		2			
1158	11.1756	13.2288	16.4935		3			
1159	11.1431	13.2106	16.4911		4			
1160	11.1562	13.2238	16.2189		3 dup			
1161	11.1624	13.2154	16.2980		C_1A	1/6		
1162	11.1732	13.2414	16.1994		C_2A			
1163	11.1671	13.2313	16.6064		1A			
1164	11.1317	13.1771	16.4494		2A			
1165	11.1042	13.1704	16.1450		3A			
1166	11.0748	13.1314	16.5581		4A			
1167	11.1069	13.1715	16.7962		3A dup			
1168	11.1929	13.2253	16.2485		C_1	1/20		
1169	11.1569	13.1934	16.2926		C_2			
1170	11.1261	13.1707	16.5542		1			
1171	11.0987	13.1416	16.4487		2			
1172	11.1448	13.1864	16.5064		3			

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(3 9-60)

CONTROL WORK SHEET - 1

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1173	11.1209	13.1679	16.7607		4			
1174	11.1375	13.1886	16.2199		4 dup			
1175	11.1959	13.2398	16.7869		C1 A	1/2		
1176	11.1075	13.1587	16.2300		C2 A			
1177	11.1433	13.1880	16.5789		1 A			
1178	11.1782	13.2232	16.6262		2 A			
1179	11.1501	13.1952	16.3166		3 A			
1180	11.1077	13.1521	16.5210		4 A			
1181	11.2269	13.2787	16.4896		4A dup			
1182	11.2081	13.2417	16.3739		C1	1/2		
1183	11.2105	13.2348	16.6416		C2			
1184	11.0927	13.1119	16.1435		1			
1185	11.1732	13.2049	16.2097		2			
1186	11.2331	13.2636	16.2757		3			
1187	11.1759	13.2175	16.5525		4			
1188	11.1045	13.1394	16.2779		C1 dup			
1189	11.1911	13.2179	16.4381		C1 A	1/4		
1190	11.2015	13.2369	16.4353		C2 A			
1191	11.1229	13.1601	16.2032		1 A			
1192	11.1469	13.1826	16.2243		2 A			
1193	11.2316	13.2714	16.2968		3 A			
1194	11.1102	13.1545	16.4699		4 A			
1195	11.1167	13.1605	16.1315		C1 A dup			
1196	11.2027	13.1896	16.6202					
1197	11.1978	13.2102	16.4617					

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(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1198	11.1905	13.2127	16.3538					
1199	11.1599	13.1799	17.6384					
1200	11.0814	13.0991	17.4425					
1201	11.1846	13.2176	17.7722					
1202	11.1444	13.1490	16.2809					
1203	11.1515	13.1844	16.4715					
1204	11.1389	13.1880	16.1138					
1205	11.1938	13.2187	16.6301					
1206	11.1912	13.2280	16.4899					
1207	11.1988	13.2444	16.8252					
1208	11.1055	13.1462	16.4059					
1209	11.1454	13.1837	16.1846					
1210	11.1953	14.5782	14.6813					
1211	11.1117	14.5180	14.5606					
1212	11.1182	14.5320	14.5326					
1213	11.0677	14.4739	14.520					
1214	11.2030	14.6144	14.7176					
1215	11.1647	14.5782	14.6148					
1216	11.1417	14.5598	14.6531					
1217	11.1728	14.5795	14.6165					
1218	11.1298	14.5472	14.6513					
1219	11.0886	14.5000	14.5444					
1220	11.1184	14.5593	14.6568					
1221	11.1614	14.5777	14.6257					
1222	11.1315	14.5275	14.6226					

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CONTROL WORK SHEET - 1

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COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1223	11.1810	14.5925	14.6351					
1224	11.0652	14.4769	14.5780					
1225	11.1256	14.5452	14.5861					
1226	10.9662	14.3835	14.4735					
1227	11.1593	14.5755	14.5938					
1228	11.2045	14.6315	14.7297					
1229	11.1099	14.5380	14.5817					
1230	11.2382	14.6621	14.746					
1231	11.0227	14.4570	14.4985					
1232	11.0993	14.5305	14.6241					
~~1233~~	~~11.1167~~	~~14.5478~~	SAMPLE WAS 7					
1234	490.09 ~~11.0058~~	297.02	1052.96					
1235	489.45 ~~11.1720~~	278.16 ~~1052.96~~	1030.35					
1236	488.82 ~~11.1341~~	276.66	1128.27					
1237	491.38 ~~11.0864~~	278.52	1138.29					
1238	490.67 ~~11.1962~~	272.26	1140.08					
1239	491.79 ~~11.0260~~	278.15	1140.12					
1240	489.95 ~~11.1688~~	287.29	1040.31					
1241	490.40 ~~11.2812~~	284.35	1041.25					
1242	489.07 ~~11.1677~~	282.20	1132.99					
1243	485.10 ~~11.1975~~	275.70	1115.91					
1244	488.72 ~~11.0758~~	277.05	1137.54					
1245	485.02 ~~11.0579~~	265.18	1108.56					

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	pH		pH	MnO4-		pH	MnO4-		pH	MnO4-	
C1	7.97	C1	7.93		C1	8.00		C1	7.98		
C1D	7.98										
C2	8.07	C2	8.07		C2	8.06		C2	8.06		
		C2D	8.06								
1	6.60	1	6.84	17.9	1	7.06	22.3	1	7.12	15.9	
					1DUP	7.03	22.4			13.5	
2	6.67	2	6.94	7.00	2	7.13	15.8	2	7.21	13.6	
							37.0	2DUP	7.22		
3	6.55	3	7.00	19.1	3	7.21		3	7.28	33.8	
				69.0			34.9			39.3	
4	6.56	4	7.00		4	7.22		4	7.22		

	pH	MnO4-		pH	MnO4-		pH	MnO4-	
C1	7.95		C1	7.96		C1	7.93		
						C1D	7.92		
C2	8.02		C2	8.01		C2	8.00		
1	7.14	23.3	1	7.24	35.2	1	7.40	49.1	
2	7.24	27.4	2	7.30	13.0	2	7.39	22.6	
3	7.33	30.8	3	7.49	31.7	3	7.62		
3DUP	7.32	30.9						31.2	
4	7.27		4	7.40	37.3	4	7.56	37.3	
	10.02	9.82 / 49.6			37.2		9.96	10.1 / 10.0	

TASK 4b- R15

10ppm TCE

1.0% MnO_4^-

3.0% MnO_4^-

	FLASK	SCUDGE	GW	KMnO4
1	208	—	(360) 363.4	—
2	221	—	(360) 359.8	—
1	210	91.9g	(368) 370.4	4.4892g
2	216	91.2g	(365) 365.2	4.8206g
3	207	91.6	(366) 365.9	14.64g
1	209	91.8	(367) 370.1	14.80g

6/23/98

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

ANALYZED FOR

SERIES

BTL HEX BTL HEX+SAMPLE

SAMPLE CODE	BTL	~~ORIGINAL SAMPLE SIZE~~	~~DILUTED TO~~	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
897	11.1041	14.4865	19.3147	20X				
898	11.0519	14.4206	19.3289					
899	11.0311	13.0540	16.9724					
900	11.0329	13.0310	16.9295					
901	10.8965	12.9032	16.3537	1X				
902	11.0154	13.0364	16.7870					
903	10.9872	13.0121	16.7616					
904	11.0045	13.0356	16.3847					
905	11.0087	13.0253	15.9826	20X				
906	11.0320	13.0108	16.7383	20X				
907	10.9248	12.9897	16.8752					
908	10.9758	12.9873	16.7544	1X				
909	11.0045	13.0219	17.1856					
910	11.1422	13.1788	17.5095					
911	10.9822	13.0197	17.0375					
912	11.0140	13.0525	16.8794	1X				
913	10.9803	12.9850	16.7006	20X				
914	11.0813	13.0816	16.7328	20X				
915	11.0080	13.0257	16.8458	1X				
916	10.9793	13.0016	16.8565					
917	10.9792	13.0071	17.0729					
918	10.9466	12.9729	17.0102					
919	11.1284	13.1455	16.6011					
920	10.9013	12.8788	16.4157	20X				
921	10.9951	13.0111	16.5590	20X				

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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
922	11.0330	13.0623	16.4571	1X				
923	11.0016	13.0346	15.9015					
924	10.9722	13.0147	16.8153					
925	11.0777	13.1145	16.9618					
926	10.9687	13.0151	16.1687					
927	11.0251	13.0393	16.6428					
928	11.1107	13.1150	16.6710					
929	11.0339	13.0507	16.6811					
930	10.9365	12.9531	16.8241					
931	10.9482	12.9791	16.7922					
932	11.0006	13.0175	16.9218					
933	11.0667	13.0943	16.0883					
934	11.0475	13.0715	16.2880					
935	10.9658	12.9991	16.4546					
936	11.1824	13.2163	17.0871					
937	11.0093	13.0419	16.4108					
938	10.9052	12.9412	16.3751					
939	11.0618	13.1016	16.6002					
940	11.0026	13.0417	16.2035					
941	11.0025	13.0329	16.3010					
942	11.1231	13.1234	16.7042					
943	11.0869	13.0921	16.4346					
944	11.0770	13.1010	16.8089					
945	11.0019	13.0515	16.8103					
946	11.0827	13.1460	17.1248					

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(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
947	11.1097	13.1564	16.3142					
948	11.1259	14.5015 ~~13.1891~~	14.5965					
949	11.1700	14.5722 ~~13.2301~~	14.6180					
950	10.9709	14.3880 ~~13.0353~~	14.4860					
951	11.0094	14.4344 ~~13.0851~~	14.4792					
952	10.9591	14.3814 ~~13.0311~~	14.4709					
953	11.0916	14.5045 ~~13.1527~~	14.5486					
954	10.8610	14.2807 ~~12.9278~~	14.3713					
955	11.0033	14.4182 ~~13.0627~~	14.4643					
956	11.0434	14.4468 13.1000	14.5423					
957	~~11.1001~~ 11.0778	14.4816 ~~13.1307~~	14.5253					
#1 958	491.01 ~~11.0619~~	285.59 ~~13.1161~~	1156.76					
#2 959	491.01 ~~10.9973~~	285.47 ~~13.0468~~	1137.66					
#3 960	488.46	280.62	1148.11					
#4 961	498.82	270.67	1142.18					
~~962~~								
~~963~~								

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(3 9-60)

6-18-98 4 DRUN 12

	pH (1.0)	MnO4 (0.02)
C1	7.88	—
C1DUP	7.91	—
C2	8.03	—
1	6.47	4.3523g
2	6.46	4.9012g
3	6.41	14.768g
4	6.43	14.868g

10.00

0930 49.4

	pH	MnO4-	
C1	7.98	—	
C2	8.02	—	
C2D	8.01	—	
1	6.79	8.70	(.02/8.02)
2	6.75	6.88	
3	6.85	21.4	(.01/8.01)
4	6.75	17.8	

1100

	pH	MnO4-	
C1	7.97	—	
C2	8.01	—	
1	6.95	5.65	(.02/8.01)
1DUP	6.95	5.65	
2	6.92	6.14	
3	7.01	17.1	(.01/8.01)
4	6.92	17.9	

1300

	pH	MnO4	
C1	7.96	—	
C2	8.02	—	
1	7.03	9.65	
2	6.99	10.9	
2DUP	6.98	10.7	
3	7.12	26.8	(.03/8.0
4	7.00	34.6	

1500

	pH	MnO4-	
C1	7.96	—	
C2	8.02	—	
1	7.11	15.4	(0.8/8.02)
2	7.00	19.9	
3	7.18	35.1	(0.8/8.02)
3DUP	7.19	35.0	
4	7.06	33.1	

10.03

9.5 49.7

6-18-98 0500 50.1

	pH	MnO4	
C1	7.94	—	
C2	8.04	—	
1	7.18	0.46	1X
2	7.13	21.2	(.2/8.2)
3	7.32	31.2	(.02/8.02)
4	7.20	33.4	
4DUP	7.20	33.2	

9.2 50.0

0900

	pH	MnO4-	
C1	7.94	—	
C1DUP	7.95	—	
C2	8.08	—	
1	7.20	0.06	1X
2	7.27	15.5	(.2/8.2)
3	7.49	24.5	(.02/8.02)
4	7.35	33.4	

6-15-98 Hbrwiz BCATCH	SLUDGE	6-16-98 G.W (0900)	KRAWOY 6-
1 208.8		(368) 360.1	
2 221.4		↓ 361	
1 208.6	91.9g	(367.6) 367.9	4.8563g
2 24.8	92.9g	(371.6) 371.3	4.9012g
3 208.0	92.2g	(368.9) 369.2	14.768g
4 209.4	91.9g	(367.6) 371.7	14.464g

CONTROL WORK SHEET - 1

ANALYST	
COMPLETED	
TIME	DATE
CHECKED BY (SUPERVISOR)	
CURVE FACTOR	
ANALYZED FOR	

SERIES 6-16-98

SAMPLE CODE	BTL	~~ORIGINAL SAMPLE SIZE~~ BTL + HEX	~~DILUTED TO~~ SAMPLE + BTL + HEX	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
830	10.8498	13.8213	18.8216	200X				
831	11.6259	14.3700	19.2974	200X				
832	10.9069	12.9287	16.3575	200X				
833	10.9998	13.0126	16.6896	200X				
834	10.9769	12.9963	16.4356	20X				
835	10.8419	12.8684	16.4440					
836	10.9557	12.9874	17.0449					
837	10.9176	12.9552	16.8438					
838	10.9891	13.0253	16.3063	200X				
839	11.1421	13.1864	16.9954	200X				
840	11.1404	13.1822	16.0182	200X				
841	10.8863	12.9290	16.5670	20V				
842	11.2066	13.2386	17.1692					
843	11.0259	13.0670	17.0265					
844	11.1221	13.1673	17.0867					
845	11.2048	13.2559	17.1401	200 X				
846	11.1785	13.1908	16.8635	200X				
847	11.1825	13.1861	16.7784	200X				
848	11.1438	13.1623	17.0177	20X				
849	11.1287	13.1477	16.8940					
850	11.1687	13.2008	17.2179					
851	11.1259	13.1481	17.2499					
852	11.1206	13.1402	16.0202					
853	11.1530	13.1697	16.5669	200 X				
854	11.1288	13.1405	16.7678	200X				

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(3 9-60)

CONTROL WORK SHEET - 1

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
855	11.2262	13.2434	16.9427	20X				
856	11.1460	13.1728	17.1881					
857	11.1877	13.2109	17.1683					
858	11.1454	13.1703	17.0895					
859	11.1567	13.1793	15.8617					
860	11.1655	13.1782	17.0928	200X				
861	11.1936	13.1902	17.0154	200X				
862	11.2100	13.2311	17.6343	20X				
863	11.0850	13.1196	17.4888					
864	11.0083	13.0340	15.9364					
865	11.0646	13.0814	17.5687					
866	11.0111	13.0317	17.7237					
867	11.0475	13.0576	17.0355	200X				
868	10.9983	12.9918	17.0123	200X				
869	11.0314	13.0400	16.8984	20X				
870	11.0294	13.0773	17.0421					
871	11.0404	13.0843	17.4252					
872	11.0147	13.0527	17.3757					
873	10.9435	12.9729	15.5910					
874	11.1855	13.1962	16.4800					
875	11.0171	13.0281	16.6144					
876	11.0140	13.0497	16.7357					
877	10.9193	12.9899	16.5336					
878	10.9667	13.0415	16.5366					
879	10.9721	13.0400	16.6525					

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(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

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CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
880	10.9796	13.0449	16.5866					
881	11.0969	14.5609 ~~13.1750~~	14.6511					
882	10.9659	14.4065 ~~13.0253~~	14.4470					
883	10.9928	14.4255 ~~13.0541~~	14.5225					
884	11.0444	14.4838 ~~13.1140~~	14.5233					
885	11.0991	14.5309 ~~13.1586~~	14.6306					
886	10.9921	14.4295 ~~13.0528~~	14.4728					
887	10.9172	14.3560 ~~12.9773~~	14.4476					
888	10.8915	14.3238 ~~12.9508~~	14.3461					
889	11.0525	14.4853	14.5796					
890	10.9999	14.4152	14.4592					
891	11.0123	14.4253	14.5141					
892	10.9901	14.4096	14.4500					
#1 893	485.38	296.84	1131.79					
#2 894	474.88	275.56	1126.54					
#3 895	473.25	290.38	1144.75					
#4 896	474.51	279.59	1138.37					

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7-9-98 CKS10 10.09 CKS10 49.5 16

0830 pH	0845 MnO_4^- add'd	0915 pH	0915 MnO_4^-	1045 pH	1045 MnO_4^-	1245 pH	1245 MnO_4^-
C_1 8.00	—	C_1 8.01	—	C_1 8.03	—	C_1 8.07	—
C_{1DP} 8.0		8.09					
C_2 8.09	—	C_2 8.09	—	C_2 8.10	—	C_2 8.14	—
		C_{2DP}					
1 6.56	.913 ml	1 6.60	1.31 (1x)	1 6.61	1.52 (1x)	1 6.59	1.91 (1x)
				1_{DP} 6.62	1.52		
2 6.53	.915 ml	2 6.63	1.88	2 6.65	2.14	2 6.62	2.20
						2_{DP} 6.62	2.21
3 6.57	.915 ml	3 6.74	0.28	3 6.82	0.54	3 6.87	0.63
4 6.53	9.00 ml	4 6.72	0.59	4 6.79	0.96	4 6.84	1.10

7-10-98

1445 pH	1445 MnO_4^-	0855 (CKS10 = 10.05) pH	0855 MnO_4^-	0845 pH	0845 MnO_4^-
		50.2			
C_1 8.04	—	C_1 8.03	—	C_1 8.04	—
	—		—	C_{1DP} 8.04	—
C_2 8.11	—	C_2 8.12	—	C_2 8.12	—
1 6.60	1.91 (1x)	1 6.62	2.52 (1x)	1 6.63	2.63 (1x)
2 6.63	2.36	2 6.63	3.06	2 6.64	3.26
3 6.87	0.84	3 6.90	1.72	3 6.90	1.95
3_{DP} 6.78	0.84				
4 6.86	1.31	4 6.91	2.14	4 6.88	2.39
pH cal = 10.07	10 = 9.94	4_{DP} 6.91	2.15	6.00	50.6
	50 = 50.0				10.2

CONTROL WORK SHEET - 1

7-8-48 STATA

ANALYST	
COMPLETED	
TIME	DATE
CHECKED BY (SUPERVISOR)	
CURVE FACTOR	
ANALYZED FOR	

SERIES

SAMPLE CODE	BTL	BTL + HEX ~~ORIGINAL SAMPLE SIZE~~	SAMPLE + BTL + HEX ~~DILUTED TO~~	D.F. ~~ALIQUOT SIZE~~	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
962	11.1469	14.5145	19.5150	200X				
963	11.0081	14.3727	19.2781	↓				
964	11.1481	13.1382	16.1166	200X				
965	11.2160	13.2215	16.6123	↓				
966	11.0497	13.0700	16.6403	↓				
967	10.9629	12.9847	16.4087	↓				
968	10.9677	12.9914	16.6663	↓				
969	11.0730	13.0956	16.6924	↓				
970	11.0394	13.0530	16.6577	↓				
971	11.0221	13.0403	16.4702	200X				
972	10.9883	12.9998	16.5847	200X				
973	11.0658	13.0901	16.3556	20X				
974	11.0214	13.0487	16.7034	↓				
975	10.9456	12.9684	16.5083	↓				
976	11.0516	13.0823	16.7820	↓				
977	10.9738	13.0045	16.0577	200X				
978	11.0182	13.0552	16.5022	200X				
979	10.9921	13.0329	16.3616	200X				
980	11.0748	13.1074	16.2231	↓				
981	11.0033	13.0417	16.4484	↓				
982	10.9803	13.0144	16.2577	↓				
983	10.9567	12.9947	16.7879	↓				
984	10.9345	12.9719	16.0312	↓				
985	11.0458	13.0593	16.7714					
986	10.9881	13.0058	16.4435					

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(3 9-60)

CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
987	10.9718	13.0044	16.5678					
988	10.9558	12.9912	15.9477					
989	11.0429	13.0818	16.5761					
990	11.0281	13.0719	16.5818					
991	11.0397	13.0852	16.3016					
992	10.9981	13.0451	16.4762					
993	11.0510	13.0786	16.0992					
994	11.0757	13.1077	16.5181					
995	11.0325	13.0701	16.6844					
996	10.9629	13.0045	16.1587					
997	11.1323	13.1748	16.2657					
998	11.0088	13.0476	16.3238					
999	11.0511	~~13.0945~~ 13.0875	16.7989					
1000	11.0553	~~16.5678~~ grp 13.0673	16.5001					
1001	11.0984	13.1068	16.3655					
1002	10.9329	12.9546	16.2018					
1003	11.0164	13.0294	16.4488					
1004	11.1015	13.1264	16.1289					
1005	11.1299	13.1571	16.6167					
1006	10.9748	13.0069	16.4999					
1007	10.9095	12.9323	15.7212					
1008	10.9407	12.9684	16.4159					
1009	10.9559	12.9825	16.3655					
1010	11.0904	13.1234	16.4875					
1011	11.1807	13.2182	16.7684					

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CONTROL WORK SHEET - 1

ANALYST

COMPLETED

TIME DATE

CHECKED BY (SUPERVISOR)

CURVE FACTOR

SERIES

ANALYZED FOR

SAMPLE CODE		ORIGINAL SAMPLE SIZE	DILUTED TO	ALIQUOT SIZE	ABSORBANCE	CURVE READING	DILUTION FACTOR	RESULTS /ml
1012	11.0928	13.1210	16.0863					
1013	11.0671	14.4197	14.5153					
1014	11.1340	14.5357	14.5777					
1015	11.0187	14.4466	14.5457					
1016	11.0961	14.5346	14.5734					
1017	11.1249	14.5577	14.6527					
1018	11.1350	14.5699	14.6100					
1019	11.1902	14.6216	14.7207					
1020	11.0374	14.4984	14.5480					
1021	11.0950	14.5224	14.6244					
1022	11.2127	14.6378	14.6928					
1023	11.1169	14.5445	14.6403					
1024	11.1224	14.5435	14.5900					
H1 1025	490.80	272.51	1131.33					
#2 1026	489.86	291.98	1152.44					
#3 1027	489.87	275.64	1144.25					
#4 1028	487.80	274.54	1133.23					

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(9-60)

7-7-89.

	FLASK	SLUDGE	GW.	GW ACTUAL	MOLY ml
C_1	207	—	360 ml	(363.6 ml)	—
C_2	220	—	↓	(361.9 ml)	—
1	208	92.0 g	368 ml	(365 ml)	.9125
2	208	91.5 g	366 ml	(366.2 ml)	0.915
3	208	91.2 g	365 ml	(365.6 ml)	9.15
4	208	90.1 g	360 ml	(360.1 ml)	9.00 ml

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5. Laboratory Records, ORNL-RC

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