

*Appendix D*  
**Data Validation Report**

# MEMPHIS ENVIRONMENTAL CENTER, INC.

## MEMORANDUM

DATE: April 4, 2001  
FROM: Joe Ricker JR  
TO: Norm Kennel  
SUBJECT: Analytical Data Quality Assessment and Validation  
Cypress Creek Investigation (Report Nos. R-210289 and 210311)

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The following details a data quality assessment and validation for 42 soil samples and four liquid samples collected during the period of March 5 through March 9, 2001 from the above referenced site. All samples were analyzed for base/neutral/acid compounds (BNAs), non-volatile organic compounds (NVOs), and metal compounds. The samples were analyzed by GTW Analytical Services, LLC (GTW). The quality assurance criteria were established by the associated Sampling and Analysis Procedures (SAP) for Velsicol Chemical Corporation – Memphis, Tennessee, as revised in January 2001. The sample identification numbers are shown on the attached chain-of-custody forms. The analytical methods used for the sample analysis are presented in Table 1. Copies of the QA/QC summary provided by the laboratory and the chain-of-custody forms are also attached.

### Sample Delivery

The samples were collected, transported, handled, and analyzed maintaining chain-of-custody protocols. Documentation relative to the collection of samples and laboratory analyses were listed on the chain-of-custody form that accompanied the samples to the laboratory. Upon review of the chain-of-custody form, it is noted that the samples were received at the laboratory in good condition and properly preserved with ice.

### Holding Time Periods

The allowable holding time periods are listed in Table 2. On the basis of the sample collection and analysis dates referenced on the chain-of-custody form and the analytical report provided by GTW, the extractions and analyses were performed within the allowable holding times.

### Laboratory Blank Analyses

Sample contamination contributed by laboratory conditions or procedures was monitored by the concurrent preparation and analysis of method blank samples. The method blank samples for all analyses yielded non-detectable concentrations of analytes of interest, indicating that no laboratory contamination occurred.

### Surrogate Compound Percent Recovery

The recoveries of surrogate compounds are used to assess the individual sample performance achieved by the laboratory for organic analyses. Surrogate recoveries for BNAs and NVOs in all samples were within control limits. Some surrogate recoveries for NVOs were unavailable due to dilution. However, because the surrogate recoveries for the blank and blank spike analyses were within control limits, no qualification of the data was necessary.

### Blank Spike Analyses

The recoveries of blank spike analyses are used to assess the analytical accuracy achieved by the laboratory. As the blank spike analyses are independent of potential matrix effects, they give a true indication of the analytical accuracy achieved by the laboratory for the respective analyses performed. Blank spike analyses were performed for BNAs, NVOs, and metals.

The blank spike recoveries for all analyses indicated that acceptable levels of accuracy were achieved for these analytical methodologies.

### Matrix Spike/Matrix Spike Duplicate (MS/MSD) Analyses

The recoveries of MS/MSD analyses are used to assess the analytical accuracy on an individual sample basis, while the relative percent difference (RPD) between the MS and the MSD indicates the analytical precision achieved for that sample. The RPD data provided were within the control limits for all BNAs and NVOs, with a few exceptions. However, because the blank spike recovery for both analyses were within control limits, no qualification of the data was necessary.

The MS/MSD data provided were unavailable or outside of control limits for numerous BNAs and NVOs due to high concentrations of the analytes in the sample and matrix interference. Again, due to the fact that the blank spike recoveries for all BNAs and NVOs were within control limits, no qualification of the data was necessary due to these circumstances.

The MS/MSD data provided for metals analyses were unacceptable in most cases. There were three samples that were spiked, and in all three samples, more than 75 percent of the recoveries were unacceptable. Three blank spike samples resulted in all recoveries within control limits, so the unacceptable matrix spike recoveries appear to be due to matrix effects. Because every metal analyte failed at least one of the three spike recoveries, all detected results should be qualified as estimated, and flagged "J". Further, all recoveries for selenium and thallium were less than 30%. All non-detect results for these analytes should be qualified as not usable and flagged "R".

### Field Blank Analysis

Because only soil samples were collected as part of this sampling event (with the exception of QA/QC rinse samples), trip blanks were not required. Two field blanks and rinse blanks were collected. The field blanks and rinse blanks resulted in no detected values, thus indicating that field decontamination procedures were adequate.

## Field Duplicate Analysis

In order to monitor the precision of the field sample collection procedures and the laboratory analytical methods, two field duplicate samples were collected and analyzed. Presented below are the field duplicate results for detected compounds.

	<u>Duplicate 1</u>		RPD	RPD Control Limit
	030501-T6-NB2	030501-DUP1		
<b>BNA (µg/kg)</b>				
Acenaphthylene	460	229	67	[40]
Anthracene	379	<330	14	[40]
Benzo[a]anthracene	2,070	<330	145	[40]
Benzo[a]pyrene	612	345	56	[40]
Benzo[b]fluoranthene	675	267	87	[40]
Benzo[g,h,i]perylene	495	219	77	[40]
Benzo[k]fluoranthene	735	518	35	[40]
Chrysene	864	376	79	[40]
Dibenzo[a,h]anthracene	337	<330	2	[40]
Fluoranthene	1,390	611	78	[40]
Hexachlorobutadiene	2,500	502	133	[40]
Indeno(1,2,3-cd)pyrene	515	<330	44	[40]
Pentachlorobenzene	913	336	92	[40]
Phenanthrene	773	309	86	[40]
Pyrene	1,720	836	69	[40]
<b>Pesticides (µg/kg)</b>				
4,4'-DDT	3,700	3,630	2	[40]
Aldrin	6,160	4,800	25	[40]
Chlordene	118,000	97,100	19	[40]
Dieldrin	31,600	42,000	28	[40]
Endrin	182,000	173,000	5	[40]
Endrin Ketone	263,000	289,000	9	[40]
Gamma-Chlordane	13,300	9,020	38	[40]
Heptachlor	8,660	7,870	10	[40]
Hex VCL	220,000	261,000	17	[40]
Hexachloronorborene	210,000	192,000	9	[40]
Isodrin	200,000	137,000	37	[40]
Octachlorocyclopentene	6,430	4,160	43	[40]
<b>Metals (mg/l)</b>				
Arsenic	2.31	2.69	15	[25]
Barium	104	135	26	[25]
Cadmium	0.34	0.36	6	[25]
Chromium	9.4	11.2	17	[25]
Cobalt	3.76	3.95	5	[25]
Copper	19.3	22.7	16	[25]
Lead	84.9	101	17	[25]
Nickel	3.68	3.59	2	[25]
Vanadium	15.8	17.3	9	[25]
Zinc	65.3	85.8	27	[25]

Duplicate 2

	030601-T5-F1	030601-DUP2	RPD	RPD Control Limit
BNA ( $\mu\text{g}/\text{kg}$ )				
Acenaphthene	1,190	498	<b>82</b>	[40]
Anthracene	451	765	<b>52</b>	[40]
Benzo[a]anthracene	1,680	858	<b>65</b>	[40]
Benzo[a]pyrene	1,650	637	<b>89</b>	[40]
Benzo[b]fluoranthene	1,480	623	<b>82</b>	[40]
Benzo[g,h,i]perylene	1,110	< 330	<b>108</b>	[40]
Benzo[k]fluoranthene	1,960	965	<b>68</b>	[40]
Chrysene	1,880	863	<b>74</b>	[40]
Dibenzo[a,h]anthracene	544	< 330	<b>49</b>	[40]
Fluoranthene	4,080	2,030	<b>67</b>	[40]
Fluorene	878	394	<b>76</b>	[40]
Indeno(1,2,3-cd)pyrene	1,030	407	<b>87</b>	[40]
Pentachlorobenzene	439	< 330	<b>28</b>	[40]
Phenanthrene	3,000	1,570	<b>63</b>	[40]
Pyrene	3,210	1,840	<b>54</b>	[40]
Pesticides ( $\mu\text{g}/\text{kg}$ )				
4,4'-DDD	< 2,500	7,240	<b>97</b>	[40]
Aldrin	22,700	16,300	<b>33</b>	[40]
Chlordene	70,000	< 2,500	<b>186</b>	[40]
Endrin	5,270	< 2,500	<b>71</b>	[40]
Endrin Ketone	2,620	< 2,500	<b>5</b>	[40]
Gamma-Chlordane	3,770	2,720	<b>32</b>	[40]
Hex VCL	58,400	42,100	<b>32</b>	[40]
Hexachloronorbomadiene	25,000	19,600	<b>24</b>	[40]
Isodrin	280,000	209,000	<b>29</b>	[40]
Octachlorocyclopentene	3,530	3,950	<b>11</b>	[40]
Metals (mg/l)				
Arsenic	3.07	0.5	<b>144</b>	[25]
Barium	140	148	<b>6</b>	[25]
Beryllium	0.54	0.2	<b>92</b>	[25]
Chromium	9.49	8.6	<b>10</b>	[25]
Cobalt	8.32	4.05	<b>69</b>	[25]
Copper	22.8	9.43	<b>83</b>	[25]
Lead	32.6	42.3	<b>26</b>	[25]
Nickel	5.24	2.73	<b>63</b>	[25]
Vanadium	18.9	15.1	<b>22</b>	[25]
Zinc	43.2	35.9	<b>18</b>	[25]

As indicated above (bold RPD value), some of the RPD values were outside control limits. The results that are outside control limits for both the primary sample and the duplicate should be qualified as estimated and flagged "J" for these analytes.

Miscellaneous Quality Control

Several pesticide samples required qualification due to non-confirmation of results due to matrix interference. Pertinent data should be qualified as estimated and flagged "J".

## Completeness

Completeness is a measure of the amount of valid data compared to the expected amount under normal conditions. In most cases, the reported concentration and associated quality control results indicate that the data are of sufficient quality. Some analytical results were qualified as estimated due to MS/MSD failures or reporting below the detection limit. The estimated data are usable as qualified. The completeness of the data reported in GTW Laboratory Report Nos. R-210289 and 210311 is 98%.

**Table 1**  
**Analytical Methods Associated With**  
**Cypress Creek Investigation**  
**Report Nos. R-210289 and 210311**

<i>Analytical Parameter</i>	<i>Method Citation</i>
Base/Neutral/Acid Compounds (BNAs)	SW-846 3510C/8270C
Pesticides	SW-846 3510C/8081A
Metals	SW-846 3050B/6010B
Mercury	SW-846 7471A
Moisture content	SW-846 3550B

**Table 2**

**Holding Time Periods  
Cypress Creek Investigation  
Report Nos. R-210289 and 210311**

<i>Parameters</i>	<i>Holding Time Period</i>
BNAs, Pesticides	- 7 days from sample collection to extraction - 40 days from extraction to completion of analysis
Metals, Mercury	- 6 months from sample collection to completion of analysis



# GTW ANALYTICAL SERVICES, LLC

3715 S. Perkins, Suite 7  
Memphis, Tennessee 38118  
(901) 323-5554

## LABORATORY REPORT

Client Contact: Norm Kennel  
Project: MEC - Cypress Creek  
Sample(s) Type: Soil

Report Date: 03/29/01  
Report No: R-210289

All sample results reported on an "as-received" basis unless otherwise indicated.

### Quality Assurance Summary (Page 1 of 2)

<u>Type of Analysis</u>	<u>Method</u>	<u>Holding Time</u>	<u>Surrogate Recoveries</u>	<u>Matrix Spike Recoveries</u>	<u>Blanks</u>	<u>Overall Summary</u>
BNA	SW-846 3550B/8270C	A	A (N-1)	A (N-1)	A	A (See N-1)
PESTICIDES	SW-846 3550B/8081A	A	A (N-2)	A (N-3)	A	A (See N-2 and N-3)
METALS	SW-846 3050B/6010B	A	NA	A (N-4)	A	A (See N-4)
MERCURY	SW-846 7471A	A	NA	A	A	A
MOISTURE CONTENT	SW-846 3550B	NA	NA	NA	NA	A

N-1: As noted in the report, some recoveries were unacceptable due to matrix interferences.

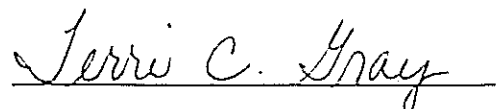
N-2: As noted in the report, some recoveries were unavailable due to dilution.

N-3: As noted in the report, some recoveries were either unacceptable due to matrix interferences, unavailable due to dilution, or unavailable due to the level of contamination that was present in the sample that was spiked.

N-4: As noted in the report, some of the recoveries were unacceptable.

A = Requirements by method were met  
NA = Not applicable

  
\_\_\_\_\_  
QA Officer

  
\_\_\_\_\_  
Technical Manager

# GTW ANALYTICAL SERVICES, LLC

3715 S. Perkins, Suite 7  
Memphis, Tennessee 38118  
(901) 323-5554

## LABORATORY REPORT

Client Contact: Norm Kennel  
Project: MEC - Cypress Creek  
Sample(s) Type: Water

Report Date: 03/29/01  
Report No: R-210289

All sample results reported on an "as-received" basis unless otherwise indicated.

### Quality Assurance Summary (Page 2 of 2)

<u>Type of Analysis</u>	<u>Method</u>	<u>Holding Time</u>	<u>Surrogate Recoveries</u>	<u>Matrix Spike Recoveries</u>	<u>Blanks</u>	<u>Overall Summary</u>
BNA	SW-846 3510C/8270C	A	A	A (N-5)	A	A (See N-5)
PESTICIDES	SW-846 3510C/8081A	A	A	N-6	A	A (See N-6)
METALS	SW-846 3005A/6010B	A	NA	A (N-5)	A	A (See N-5)
MERCURY	SW-846 7470A	A	NA	A (N-5)	A	A (See N-5)

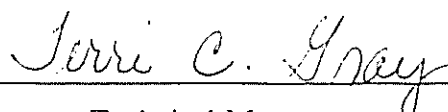
N-5: These samples were analyzed as part of a larger set which included matrix spikes that had acceptable recoveries.

N-6: Insufficient sample volume was received to perform matrix spikes.

A = Requirements by method were met  
NA = Not applicable



QA Officer



Technical Manager

GTW ANALYTICAL SERVICES, LLC  
 3715 S. Perkins, Suite 7, Memphis, TN 38118  
 Telephone (901) 323-5554; FAX (901) 323-5573

SUBMIT REPORT TO  
 COMPANY:  
 CONTACT:

210289

CHAIN OF CUSTODY  
 RECORD

PROJECT NO:

PROJECT NAME:

003

Cypress Creeds

1/2

SAMPLER'S SIGNATURE

*[Signature]*  
 (sign)

MATRIX  
 NO. OF CONTAINERS

REMARKS

SEQ. NO.	SAMPLE NO.	DATE	TIME	SAMPLE LOCATION	MATRIX	NO. OF CONTAINERS	REMARKS
1	030501-T1-NB1	3/5/01	Am	T1 - <sup>0-12"</sup> north bank	S	2	metals, heavy Pesticides
2	" - T1 - NBZ	"	Am	T1 north bank	S	2	"
3	" - T1 - SB1	"	Am	T1 south bank	S	2	"
4	" - T1 - SBZ	"	Am	T1 south bank	S	2	"
5	" - T2 - EB1	"	Am	T2 east bank	S	2	"
6	" - T2 - EBZ	"	Am	"	S	2	"
7	" - T2 - WB1	"	Am	T2 west bank	S	2	"
8	" - T2 - WBZ	"	Am	"	S	2	"
9	" - T3 - SB1	"	Am	T3 south bank	S	2	"
10	" - T3 - SBZ	"	Am	"	S	2	"
11	" - T3 - NB1	"	Am	T3 north bank	S	2	"
12	" - T3 - NBZ	"	Am	"	S	2	"
13	" - H/A1	"	Pm	H/A - 1	S	2	"
14	" - H/AZ	"	Pm	H/AZ	S	2	"
15	" - T5 - NB1	"	Pm	T5 north bank	S	2	"
16	" - T5 - NBZ	"	Pm	"	S	2	"
17	" - T5 - SB1	"	Pm	T5 south bank	S	2	"
18	" - T5 - SBZ	"	Pm	"	S	2	"
19	" - T6 - SB1	"	Pm	T6 south bank	S	2	"
20	" - T6 - SBZ	"	Pm	"	S	2	"
21	" - T6 - SB3	"	Pm	"	S	2	"
22	" - T6 - NB1	"	Pm	T6 north bank	S	2	"
23	" - T6 - NBZ	"	Pm	"	S	2	"
24	" - T6 - NB3	"	Pm	"	S	2	"
25	- Dup1	"	Pm	Duplicate	S	2	"

TOTAL NO. OF CONTAINERS - 50

RELINQUISHED BY: *[Signature]*  
 (sign)

DATE/TIME  
 3/6/01 2:20

RECEIVED BY: 2 \_\_\_\_\_  
 (sign)

RELINQUISHED BY: 2 \_\_\_\_\_  
 (sign)

DATE/TIME

RECEIVED BY: 3 \_\_\_\_\_  
 (sign)

MODE OF SHIPMENT:

SHIPPED BY:

RECEIVED FOR LABORATORY BY:  
 (sign) *[Signature]*

DATE/TIME  
 3-6-01/2:20 PM

CONDITION OF SEAL UPON RECEIPT:  
 GENERAL CONDITION OF COOLER: On Ice

COOLER OPENED BY:  
 (sign) *[Signature]*

DATE/TIME  
 3-6-01/2:20 PM

GTW ANALYTICAL SERVICES, LLC  
 3715 S. Perkins, Suite 7, Memphis, TN 38118  
 Telephone (901) 323-5554; FAX (901) 323-5573

SUBMIT REPORT TO  
 COMPANY:  
 CONTACT:

210289

CHAIN OF CUSTODY  
 RECORD

PROJECT NO:

PROJECT NAME:

003

Cypress Creek

2/2

SAMPLER'S SIGNATURE

*[Signature]*  
 (sign)

M  
A  
T  
R  
I  
X

NO. OF  
CONTAINERS

REMARKS

SEQ. NO.	SAMPLE NO.	DATE	TIME	SAMPLE LOCATION	MATRIX	NO. OF CONTAINERS	REMARKS
6	030501-HA4	3/5/01	PM	HA4	S	2	metals, SWC, Pesticides
7	" - T4-SB1	"	PM	T4 south bank	S	2	" " "
8	" - T4-SB2	"	PM	"	S	2	" " "
9	" - HA3	"	PM	HA3	S	2	" " "
0	030601-RB	3/6/01	AM	Rinse Blank	W	5	" " "
1	030601-FB	3/6/01	AM	Field Blank	W	5	" " "
2	030601-T1-F1	3/6/01	AM	T1 floor	S	2	" " "
3	" - T2-F1	"	AM	<del>T2</del> T2 floor	S	2	" " "
4	" - T3-F1	"	AM	T3 floor	S	2	" " "
5	" - T4-F1	"	AM	T4 floor	S	2	" " "
6	" - T5-F1	"	AM	T5 floor	S	2	" " "
7	" - DUP2	"	AM	Duplicate 2	S	2	" " "

TOTAL NO. OF CONTAINERS -

RELINQUISHED BY:

1 *[Signature]*  
 (sign)

DATE/TIME

3/6/01 2:20

RECEIVED BY:

2 \_\_\_\_\_  
 (sign)

RELINQUISHED BY:

2 \_\_\_\_\_  
 (sign)

DATE/TIME

\_\_\_\_\_

RECEIVED BY:

3 \_\_\_\_\_  
 (sign)

METHOD OF SHIPMENT:

SHIPPED BY:

RECEIVED FOR LABORATORY BY:

DATE/TIME

(sign) *[Signature]*

3-6-01/2:20PM

CONDITION OF SEAL UPON RECEIPT:

COOLER OPENED BY:

DATE/TIME

GENERAL CONDITION OF COOLER: On Ice

(sign) *[Signature]*

3-6-01/2:20PM

# GTW ANALYTICAL SERVICES, LLC

3715 S. Perkins, Suite 7  
Memphis, Tennessee 38118  
(901) 323-5554

## LABORATORY REPORT

Client Contact: Norm Kennel  
Project: MEC - Cypress Creek  
Sample(s) Type: Soil

Report Date: 03/30/01  
Report No: R-210311

All sample results reported on an "as-received" basis unless otherwise indicated.

### Quality Assurance Summary (Page 1 of 2):

<u>Type of Analysis</u>	<u>Method</u>	<u>Holding Time</u>	<u>Surrogate Recoveries</u>	<u>Matrix Spike Recoveries</u>	<u>Blanks</u>	<u>Overall Summary</u>
BNA	SW-846 3550B/8270C	A	A (N-1)	A (N-2)	A	A (See N-1 and N-2)
PESTICIDES	SW-846 3550B/8081A	A	A (N-3)	A (N-4)	A	A (See N-3 and N-4)
METALS	SW-846 3050B/6010B	A	NA	A (N-5)	A	A (See N-5)
MERCURY	SW-846 7471A	A	NA	A	A	A
MOISTURE CONTENT	SW-846 3550B	NA	NA	NA	NA	A

N-1: As noted in the report, one of the recoveries for sample #2100774 was unacceptable.

N-2: These samples were analyzed as part of a larger set which included matrix spikes that had acceptable recoveries.

N-3: The recovery for sample #2100773 was unavailable due to dilution.

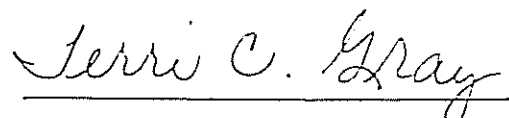
N-4: These samples were analyzed as part of a larger set which included matrix spikes. Recoveries were unavailable due to either dilution or due to the level of contamination that was present in the sample that was spiked.

N-5: As noted in the report, some of the recoveries were unacceptable.

A = Requirements by method were met  
NA = Not applicable



QA Officer



Technical Manager

MEMPHIS ENVIRONMENTAL CENTER  
 2603 Corporate Ave., Suite 100, Memphis, TN 38132  
 Telephone (901) 345-1788

SHIPPED TO (Laboratory Name):

210311

GTW

CHAIN OF CUSTODY RECORD

PROJECT NO:

003

PROJECT NAME:

Cypress Creek

SAMPLER'S SIGNATURE

*Norman Kel*  
 (sign)

MATRIX  
 NO. OF CONTAINERS

REMARKS

SEQ. NO.	SAMPLE NO.	DATE	TIME	SAMPLE LOCATION	MATRIX	NO. OF CONTAINERS	REMARKS
1	030901-T3-NB1	3/9/01	AM	T3-North Bank 1	S	2	SUOCs, Pesticides, Metals
2	030901-T3-SB1	3/9/01	AM	T3-South Bank 1	S	2	" " "
3	030901-RB	3/9/01	AM	Rinse Slank	W	5	" " "
4	030901-FB	3/9/01	AM	Field Slank	W	5	" " "
5	030901-T5-NB1	3/9/01	AM	T5-North Bank 1	S	2	" " "
6	030901-T4-NB1	3/9/01	AM	T4-North Bank 1	S	2	" " "
7	030901-T4-NB2	3/9/01	AM	T4-North Bank 2	S	2	" " "
8	030901-T6-F1	3/9/01	AM	T6-Floor	S	2	" " "
9	030901-F7	3/9/01	AM	Floor - 7	S	2	" " "

pH @ lab

Unpres. | HNO<sub>3</sub>

775, 776

*JK*

TOTAL NO. OF CONTAINERS -

24

RELINQUISHED BY:

1 *Norman Kel*  
 (sign)

DATE/TIME

3/9/01 12:10

RECEIVED BY:

2 \_\_\_\_\_  
 (sign)

RELINQUISHED BY:

2 \_\_\_\_\_  
 (sign)

DATE/TIME

\_\_\_\_\_

RECEIVED BY:

3 \_\_\_\_\_  
 (sign)

METHOD OF SHIPMENT:

SHIPPED BY:

RECEIVED FOR LABORATORY BY:

DATE/TIME

(sign) *Keith Hoover*

3-9-01/12:10PM

CONDITION OF SEAL UPON RECEIPT:

COOLER OPENED BY:

DATE/TIME

GENERAL CONDITION OF COOLER: *On Ice*

(sign) *Keith Hoover*

3-9-01/12:10PM