000.002

Truesdail Laboratories , Inc.

Shimadzu HPLC #1

DAILY SAMPLE LOG

Date Analyzed: 8/17/99

Start Time: 1:00 PM

Stop Time: 11:00 PM

Inst. Batch No.: Z

Sample ID	Dil. Factor	Method No.	Notes (see below)*
MP Blank 1	1	8315M	MDL Study
602248-Std#1			Column 018912AC
602248-Std#2			λ=360nm
602248-Std#3			Absorbance=0.01
602248-Std#4			Mobile Phase A=60:30:10 H_2O:ACN:THF
602248-Std#5			Mobile Phase B=60 40 ACN:H_2O
MP Blank 2			Time Program
602248-MDL #1			Time MP Value
" #2			5 B 0
" #3			22 B 100
" #4			25 B 100
" #5			26 B 0
" #6			33 Stop
" #7			
" #8			
" #9			
Check Std #4			
MP Blank 3			
			DH 8/19/99

Daily Instrument Maintenance Log

Analyst (Print Name): Yvonne/Der-Hawl

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)

Truesdail Laboratories , Inc.

Shimadzu HPLC #1

DAILY SAMPLE LOG

Date Analyzed: 8/20/99

Start Time: 8/20/99 3:35 PM
Stop Time: 8/21/99 10:00 AM

Inst. Batch No.: 3

Sample ID	Dil. Factor	Method No.	Notes (see below)*
MP BLANK 1	1	8315-W	Column: 018912A0
602251-SCD#1			Column Heater SP5792
#2			λ=360nm, T=30.0°C
#3			Absorbance = 0.01
#4			Mobile Phase A: 60:30:10 ~~ACN~~ DH H2O/ACN:T
#5			Mobile Phase B: 60:40 ACN: H2O
MP BLANK 2			Time Program.
602253-LCS } Soil			Time MP Value
-LCSD } Soil			5 MPB. 0
602254-LCS } Water			22 MPB 100
-LCSD } Water			25 MPB 100
~~MP BLANK 3~~			26 MPB 0
~~CHK STD #4~~			~~33 MPB~~ Stop
~~MP BLANK 4~~			
602254.3MS } Soil			Earth Tech
-3MSD } Soil			
MP BLANK 3			
CHK STD #4			
MP BLANK 4			8/21/99
602251-MB			
-1			
-2			
~~CHK STD #~~			

5/24/99

Daily Instrument Maintenance Log

The Spectra-Physics column heater SP5792 was installed 8/20/99 Am. Return to service 8/20/99 P.M. 8/20/99

Analyst (Print Name): Arvinder / Der-Haw

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)

000004

Truesdail Laboratories , Inc.

Shimadzu HPLC #1

DAILY SAMPLE LOG

Date Analyzed: 8/20/99

Start Time: 8/20/99 3:35PM

Stop Time: 8/21/99 10:00 AM

Inst. Batch No.: 3

Sample ID	Dil. Factor	Method No.	Notes (see below)*
602251-3 MB	1	8315-m	EARTH TECH
-3			
CHK STD #4			
MP BLANK 5			
602254-MB			
-1			
-2			
-3			
-4			
-5			
CHK STD #4			
MP BLANK 5			

8/20/99

Daily Instrument Maintenance Log

Analyst (Print Name): Arvinder/Der-How

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)

Truesdail Laboratories , Inc.
Shimadzu HPLC #1
DAILY SAMPLE LOG

Date Analyzed: 8/24/99

Start Time: 4:05 pm
Stop Time: 9:05 pm

Inst. Batch No.: 4

Sample ID	Dil. Factor	Method No.	Notes (see below)*
W090582103	1	1004 CARBS	Column: 018499AM
602262-A	↓	↓	Column Heater SP8792
-B			λ = 360 nm, T = 30.3°C
-C			Absorbance = ~~0.000~~ 0.01 DH 8/25/99
-D			Mobile Phase A: 60:30:10 H_2O:ACN
MP Blank			Mobile Phase B: 60:40 ACN: H_2O
			Time Program:
			Time MP value
			Client CEE
			~~8/24/99~~ YN
			8/24/99

Daily Instrument Maintenance Log

Analyst (Print Name): Yvonne Nguyen

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)

Truesdail Laboratories , Inc.

Shimadzu HPLC #1

DAILY SAMPLE LOG

000006

Date Analyzed: 8/25/99

Start Time: 3:30 pm 8/25/99

Stop Time: 12:30 ~~pm~~ (N.M) 8/26/99

CLIENT:- EARTH TECH

Inst. Batch No.: 5

Sample ID	Dil. Factor	Method No.	Notes (see below)*
MP Blank 1	1	8315M	Column: 018912A0
602251 ~~Check~~ Std 1 (KN 8/27/99)			Column Heater SP8792
602251 - 2			λ = 360 nm
- 3			Absorbence = 0.01
- 4			Mobile Phase A = 60 30 10 H_2O ACN THF
- 5			Mobile Phase B = 60 40 ACN H_2O
MP Blank 2			Time programme
602260-LCS			Time MP Value
602260-LCSD			5 B 0
MP Blank 3			22 B 100
Chk Std #4			25 B 100
MP Blank 4			26 B 100
602257-MB			33 STOP
- 1			
Chk Std #4			
MP Blank 5			

8/25/99

Daily Instrument Maintenance Log

Analyst (Print Name): Arvinder / Yvonne

*Enter what applies: Client, Standard ID., Quality Control (LCS/MS)

Truesdail Laboratories, Inc.
14201 Franklin Avenue
Tustin, CA 92780
(714) 730-6239
Fax (714) 730-6462

Pending Invoice

Laboratory Record
Invoice 00602248

8315M

MDL Study

Bill To:

Quality Control Tests
14201 Franklin Ave.
Tustin, CA 92780

Ship To:

Quality Control Tests
14201 Franklin Ave.
Tustin, CA 92780

PROJECT MANAGER	YOUR NO.	SHIP VIA	COL	PPD	SHIP DATE	TERMS	DATE	PG
Joe Bramblett						Net 30	8/16/99	1

QTY	ITEM NO.	DESCRIPTION	PRICE	UNIT	DISC %	EXTENDED PRICE
1	Void	8315M MDL Study	$1.00			$1.00
-1	Void	8315M MDL Study	$1.00			($1.00)

SALE AMOUNT	$0.00
FREIGHT	$0.00
SALES TAX	$0.00
TOTAL	$0.00
PAID TO DATE	$0.00
BALANCE DUE	$0.00

MDL Study Report
Method 8315M - Hydrazine
Client: QC

Date Prepared: 8/16/99
Date Analyzed: 8/17/99
Date Reported: 8/18/99

Multiple-Point Calibration

Area Count

	Std #1	Std#2	Std #3	Std #4	Std #5
Conc. (ug/mL)	5	10	25	50	100
mmH	5414	13373	36176	65274	162158
UDMH	4544	9805	30361	61835	138083

	Std #1	Std#2	Std #3	Std #4	Std #5
Conc. (ug/mL)	1	2	5	10	20
Hydrazine	6311	9460	27338	54693	100659

Response Factor

	Std #1	Std#2	Std #3	Std #4	Std #5	Avg. RF	Std Dev	%RSD
Conc (ug/mL)	5	10	25	50	100			
Compound	Response Factor							
mmH	1082.80	1337.30	1447.04	1305.48	1621.58	1358.84	197.69	14.55
UDMH	908.80	980.50	1214.44	1236.70	1380.83	1144.25	194.74	17.02

	Std #1	Std#2	Std #3	Std #4	Std #5	Avg. RF	Std Dev	%RSD
Conc (ug/mL)	1	2	5	10	20			
Compound	Response Factor							
Hydrazine	6311	4730	5467.6	5469.3	5032.95	5402.17	596.43	11.04

Continuous Calibration Check

Compound	Area	Theoretical Amt (ug/mL)	Actual Amt	%Diff
mmH	76801	50	56.52	113.04
UDMH	66943	50	58.50	117.01
Hydrazine	57273	10	10.60	106.02

MDL Study

Area Count

Compound	#1	#2	#3	#4	#5	#6	#7
mmH	4800	7863	8302	5373	6535	9380	9497
UDMH	3509	5120	5250	3951	5035	4854	3972
Hydrazine	5112	5494	5549	5417	5358	6778	5293

Concentration (ug/mL)

Compound	#1	#2	#3	#4	#5	#6	#7	Average	Std Dev.	MDL
mmH	3.53	5.79	6.11	3.95	4.81	6.90	6.99	5.44	1.38	4.32
UDMH	3.07	4.47	4.59	3.45	4.40	4.24	3.47	3.96	0.61	1.91
Hydrazine	0.95	1.02	1.03	1.00	0.99	1.25	0.98	1.03	0.10	0.32

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315M

Batch No.: 530

Client : QC.

Lab No.: 602248-std #1

Extr. Chem. : Arvinder / Der-Haw / Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30 PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/17/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090816D3
LCS Duplicate : Hydrazine/MMH / UDMH
20.44 mg 99.59 mg 102.8 mg
Expiration Date : @ 5 ml
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315M

Batch No.: 530

Client : QC.

Lab No.: 602248-std #2

Extr. Chem. : Arvinder / Der-Haw / Yvonne

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30 PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rev'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/17/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH / UDMH
20 44 99.59 102.5
Expiration Date : @ 70 mL
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315M

Batch No.: 530

Client : QC.

Lab No.: 602248-std #3

Extr. Chem. : Arwinder / Der-Haw / Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30 PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/17/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH/UDMH 99.59 102.5
Expiration Date : @ 25 ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315M

Batch No.: 530

Client : QC

Lab No.: 602248-Std #4

Extr. Chem. : Arvinder / Der-Haw / Yvonne

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30 PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/17/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent: ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090816D3
LCS Duplicate : Hydrazine/MMH / UDMH
Expiration Date : 20 44 % 99.59 % 102.5 % @ 50 µL
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315M

Batch No.: 530

Client : QC

Lab No.: 602248-Std #5

Extr. Chem. : Arwinder / Der-Haw / Yvonne

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30 PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/17/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH / UDMH
Expiration Date : 20 44 30 99.59 21 102.5 22
@ 100 ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#1

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30 AM	3:30 PM
pH: 5	for Fraction II:	3:30 PM	9:30 AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rev'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516D3
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : @ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#2

Extr. Chem. : Arwinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30 AM	3:30 PM
pH: 5	for Fraction II:	3:30 PM	9:30 AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090816D3
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : @ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#3

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH/UDMH
20 µg/ml 99.59% 102.5 µg/ml
Expiration Date : @ 5 µL
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#4

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip. : water [X] wastewater [ ] soil/solid (type: _____) [ ]

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : 20 ug/ml 99.55% 102.5 mg/ml @5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

[ ] Batch QC, Lab No.: _____

[ ] Acid cleanup, Date: _____

[ ] Florisil, Date: _____

[ ] Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL #5

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water [X] wastewater [ ] soil/solid (type: _____) [ ]

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30PM	9:30AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : @ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

[ ] Batch QC, Lab No.: _____

[ ] Acid cleanup, Date: _____

[ ] Florisil, Date: _____

[ ] Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 83/5m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL #6

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30 PM
pH: 5	for Fraction II:	3:30 PM	9:30 AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E0905160 03
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : @ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 83/5m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#7

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	11:30AM	3:30PM
pH: 5	for Fraction II:	3:30PM	9:30AM.

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rev'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516Q3
LCS Duplicate : Hydrazine/MMH / UDMH
Expiration Date : 20 [illegible] 99.59% 102.5 [illegible]
@ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL#8

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	3:30 PM	7:30 PM
pH: 5	for Fraction II:	7:30 PM	1:30 AM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rec'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E090516D3
LCS Duplicate : Hydrazine/MMH/UDMH
20 ug/ml 99.5% 102.5 ug/ml
Expiration Date : @ 5ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

TRUESDAIL LABORATORIES INC.

EXTRACTION CHRONICLE

Method No. : 8315m

Batch No.: 530

Client : QC

Lab No.: 602248-MDL #9

Extr. Chem. : Arvinder/Der-Haw/Yvonne.

Sample Descrip : water ☒ wastewater ☐ soil/solid (type: _____) ☐

		Start Time	Stop Time
pH: 5	for Fraction I:	3:30 PM	7:30 PM
pH: 5	for Fraction II:	7:30 PM	1:30 PM

	Weight (g)	Init. Volume (ml)	Final Volume (ml)
Sample Size :		100 ml	5 ml

Date Rev'd : 8/16/99

Date Extr. : 8/16/99

Date Finished : 8/16/99

Extraction Solvent : DCM

Lot ID. : 3023KXRP

Final Solvent : ACN

Lot ID. : 994021

Surrogate Spike 1 :
Lot ID. :
Expiration Date :

Surrogate Spike 2 :
Lot ID. :
Expiration Date :

Matrix Spike :
MS Duplicate :
Expiration Date :

Lab. Control Spike : E099516Q3
LCS Duplicate : Hydrazine/MMH/UDMH
Expiration Date : 20 ug/ml 99.59% 102.3 mg/ml @ 5 ML
Exp. 9/16/99

Method Blank ID :
Date :

Surrogate Added By: Der-Haw

Comments/Problems

Please initial in box all that apply:

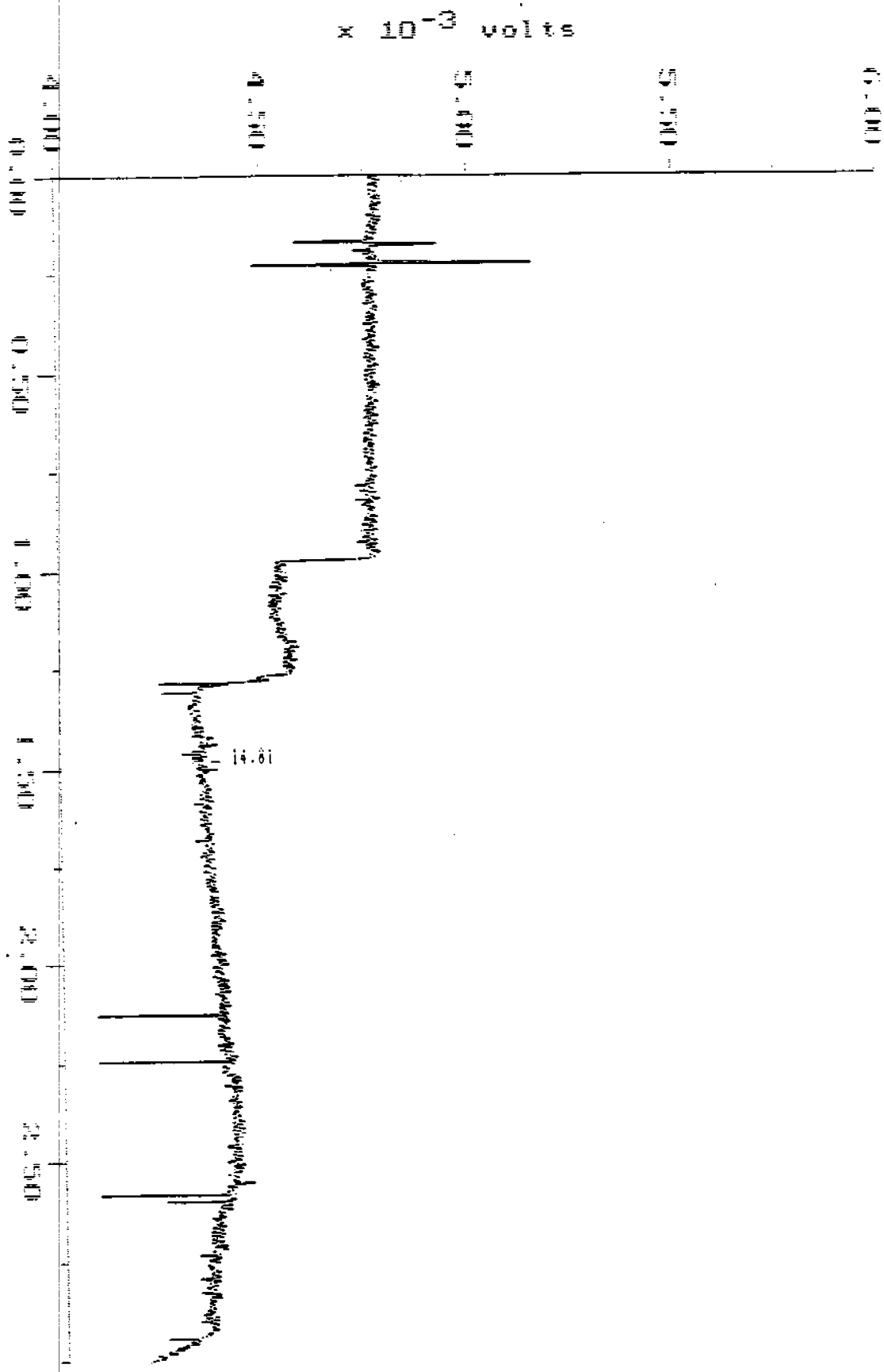
☐ Batch QC, Lab No.: _____

☐ Acid cleanup, Date: _____

☐ Florisil, Date: _____

☐ Herb. Deriv., Date: _____

Sample: MP BLANK 1 Channel: Ultraviolet Filename: AG991701
Acquired: 17-AUG-99 12:45 Method: C:\MAX\DATA1\8PA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 6:21:27

SAMPLE: MP BLANK 1

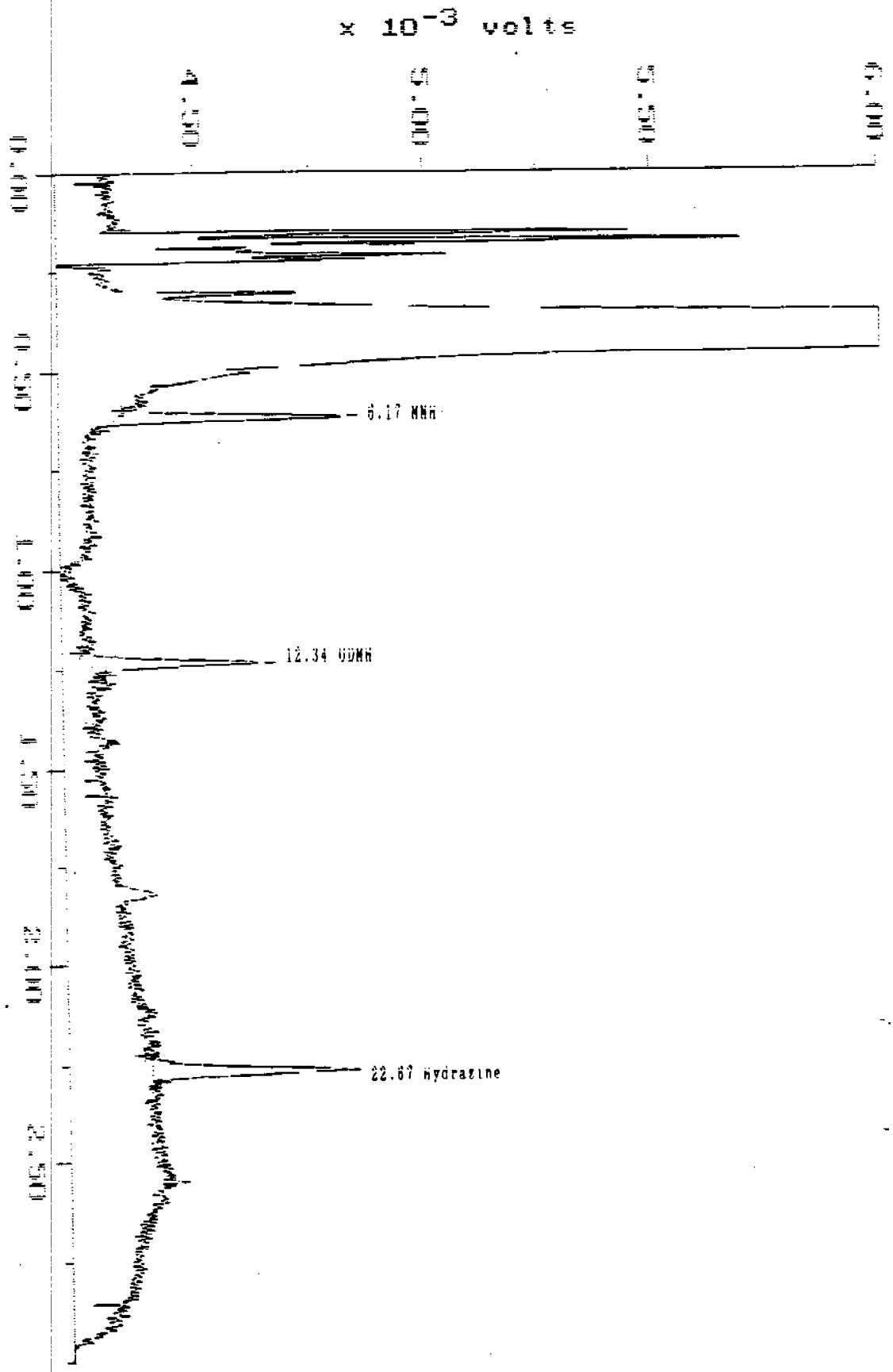
#1 in Method: EPA8315M,005 COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 12:45
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991701
Index: 1

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1			14.810	-25	-5
TOTAL				25	5

Sample: 602248 STD1 **Channel:** Ultraviolet **Filename:** AG991702
Acquired: 17-AUG-99 13:18 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:33:12

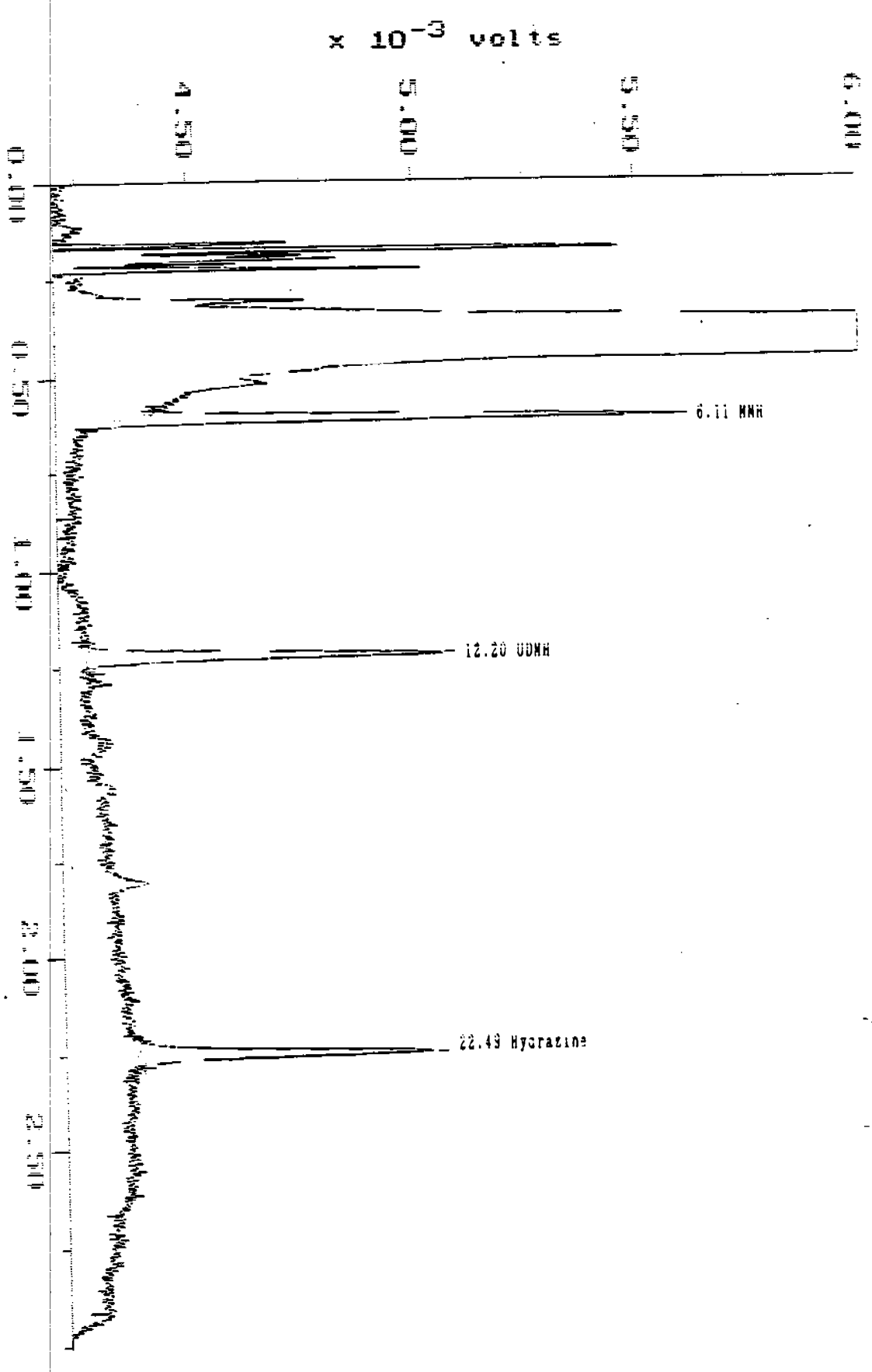
SAMPLE: 602248 STD1
#2 in Method: EPA3315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 15:18
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu 6A
Filename: AU991702
Index: 2

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1	1	MMH	6.171	5414	491
2	2	UDMH	12.338	4544	357
3	3	Hydrazine	22.666	6311	407
TOTAL				16268	1255

Sample: 602248 STD2 **Channel:** Ultraviolet **Filename:** AG991703
Acquired: 17-AUG-99 13:52 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 16-AUG-1999 5:34:15

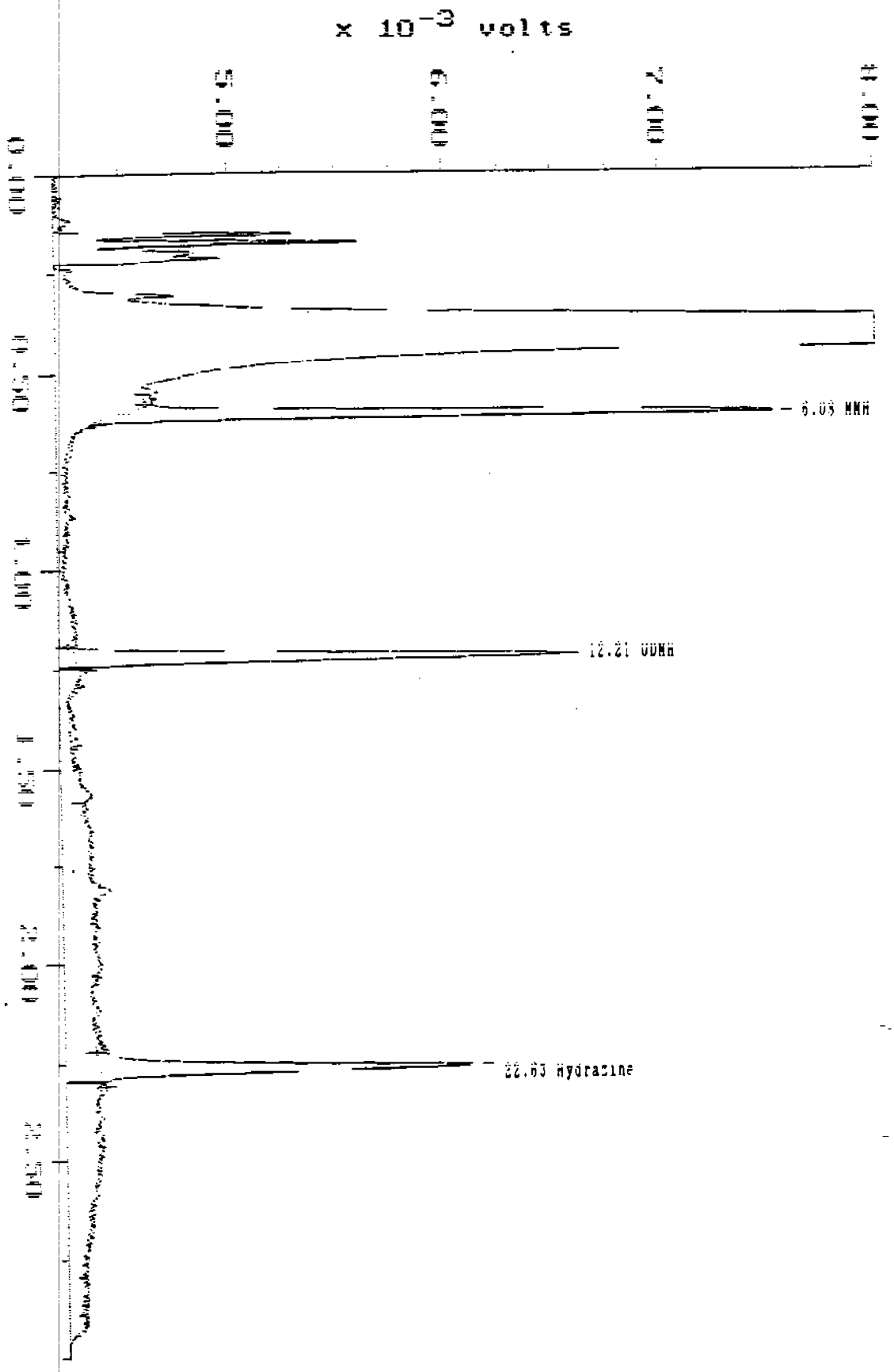
SAMPLE: 602948 STD2
#3 in Method: EPA8315M,005 COL,SHIMADZU LC/07
Acquired: 17-AUG-1999 13:52
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu 6A
Filename: AG991703
Index: 3

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.106	13373	1234
2	2	UDMH	12.199	9805	782
3	3	Hydrazine	22.485	9460	647
TOTAL				32637	2663

Sample: 602248 STD3 **Channel:** Ultraviolet **Filename:** AG991704
Acquired: 17-AUG-99 14:26 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:34:59

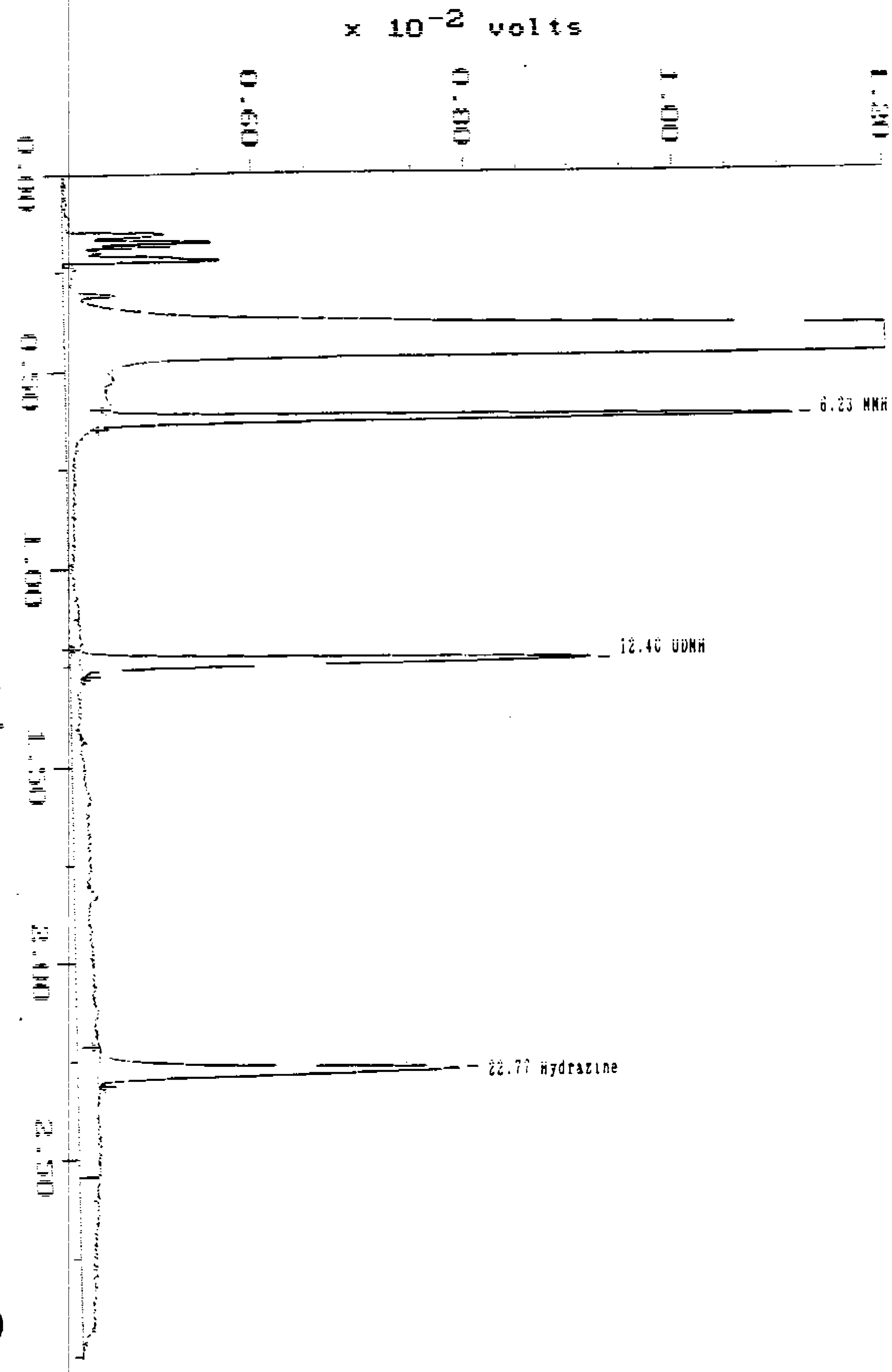
SAMPLE: 602248 STD3
#4 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 14:26
Rate: 20.0 points/sec
Duration: 50.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu 6A
Filename: AG991704
Index: 4

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.080	36176	3057
2	2	UDMH	12.212	30361	2230
3	3	Hydrazine	22.625	27338	1729
TOTAL				93874	7017

Sample: 602248 STD4 **Channel:** Ultraviolet **Filename:** AG991705
Acquired: 17-AUG-99 15:00 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:40:34

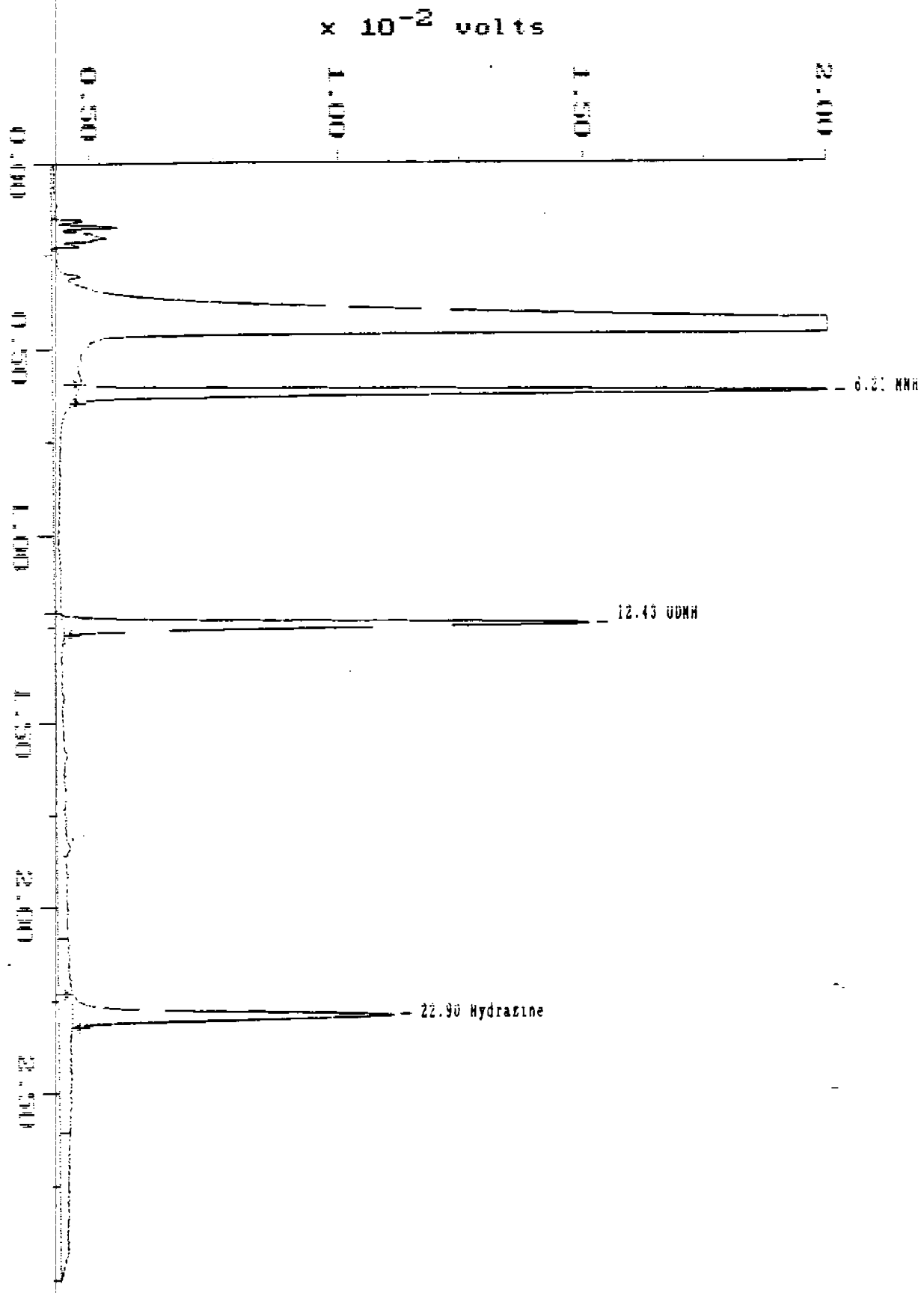
SAMPLE: 602248 STD4
#5 in Method: EPA8315M,DDS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 15:00
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu 6A
Filename: AG991705
Index: 5

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.229	65274	6567
2	2	UDMH	12.398	61835	4822
3	3	Hydrazine	22.768	54693	3403
TOTAL				181802	14793

Sample: 602248 STD5 **Channel:** Ultraviolet **Filename:** AG991706
Acquired: 17-AUG-99 15:33 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:45:23

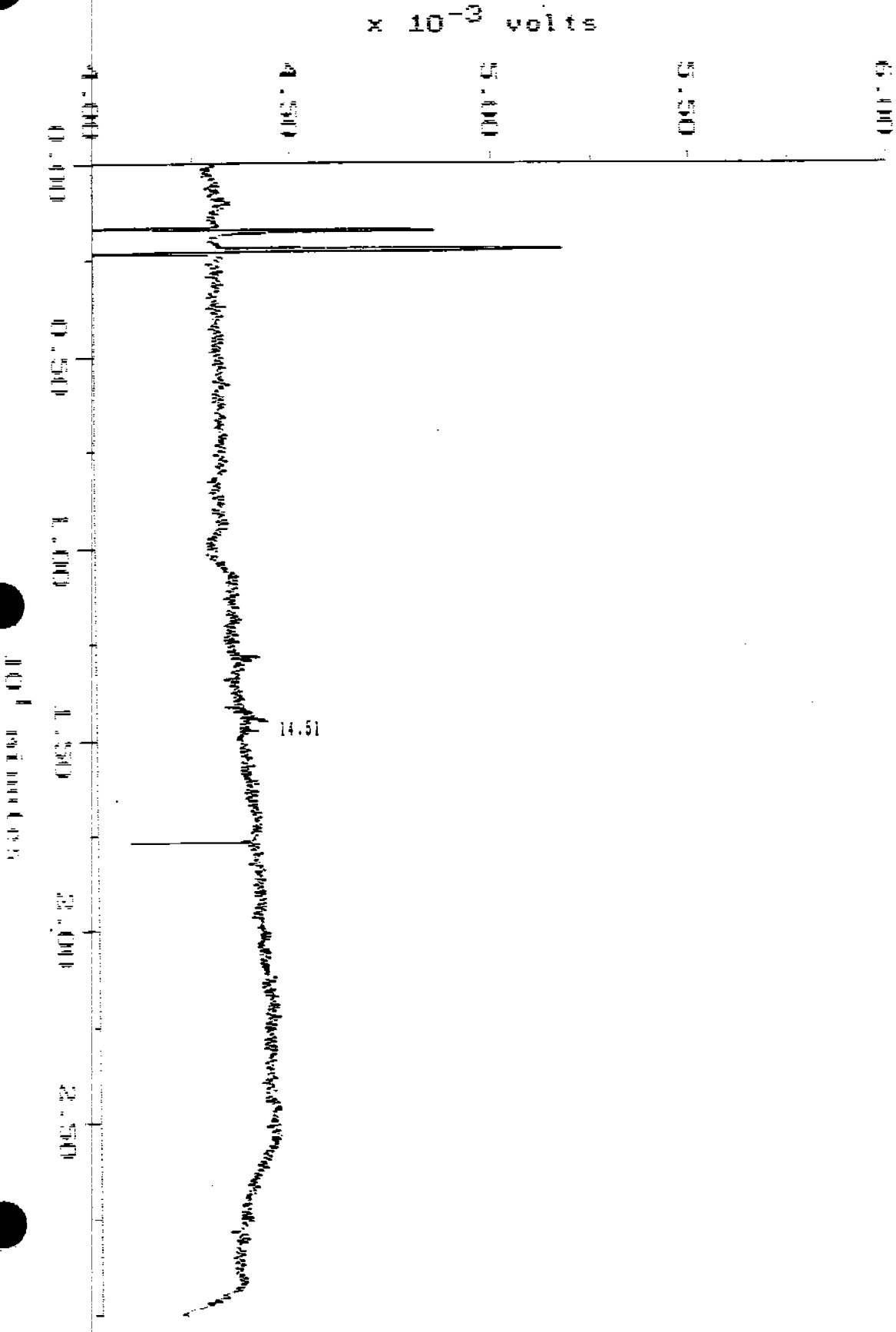
SAMPLE: 602248 STD5
#5 in Method: EPA8315M,ODS COL,SHIMADZU DC/UV
Acquired: 17-AUG-1999 15:33
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu 6A
Filename: AG991706
Index: 6

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.211	162158	15510
2	2	UDMH	12.429	138083	10692
3	3	Hydrazine	22.898	100659	6510
TOTAL				400900	32712

Sample: MP BLANK 2 **Channel:** Ultraviolet **Filename:** AG991707
Acquired: 17-AUG-99 16:07 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 6:21:46

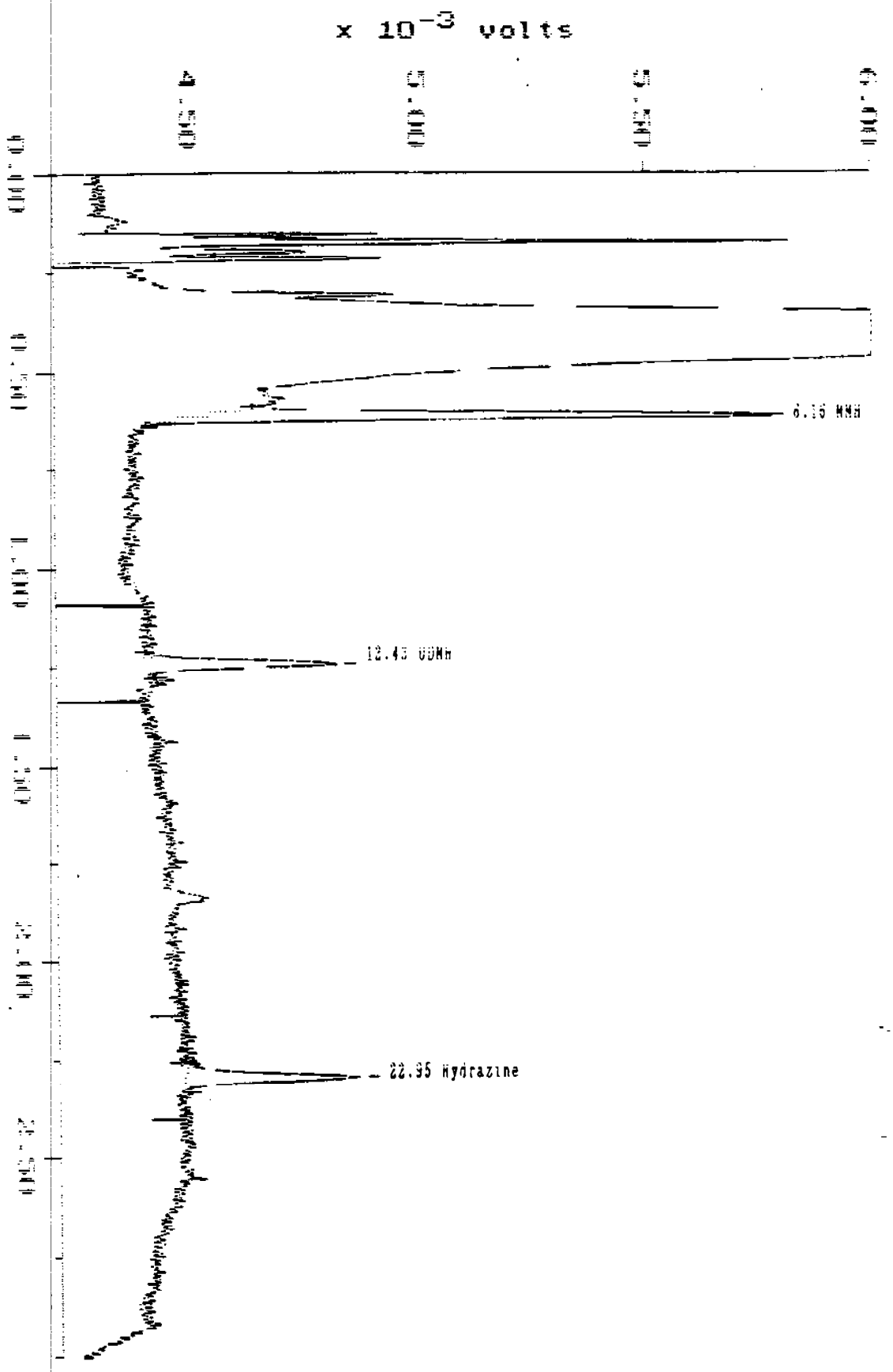
SAMPLE: MP BLANK 2
#7 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 16:07
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DM

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991707
Index: 7

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1			14.514	105	8
TOTAL				105	8

Sample: 602248 MDL#1 Channel: Ultraviolet Filename: AG991708
Acquired: 17-AUG-99 16:41 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:14:04

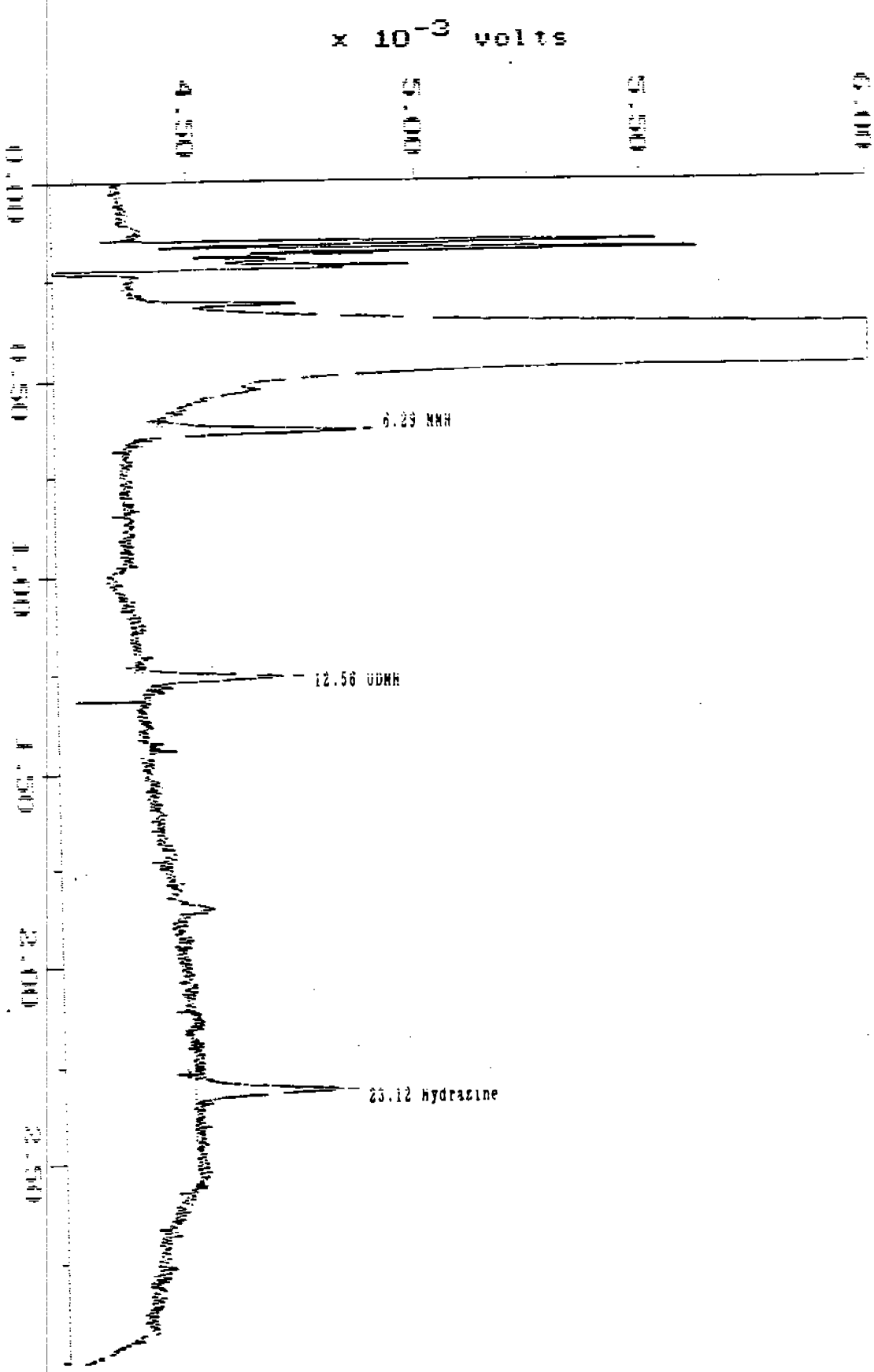
SAMPLE: 802346 MDL#1
#5 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 16:41
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu SA
Filename: AU991703
Index: 6

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.158	14886	1253
2	2	UDMH	12.425	5193	403
3	3	Hydrazine	22.951	5968	385
TOTAL				26047	2045

Sample: 602248 MDL#2 Channel: Ultraviolet Filename: AG991709
Acquired: 17-AUG-99 17:15 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:14:21

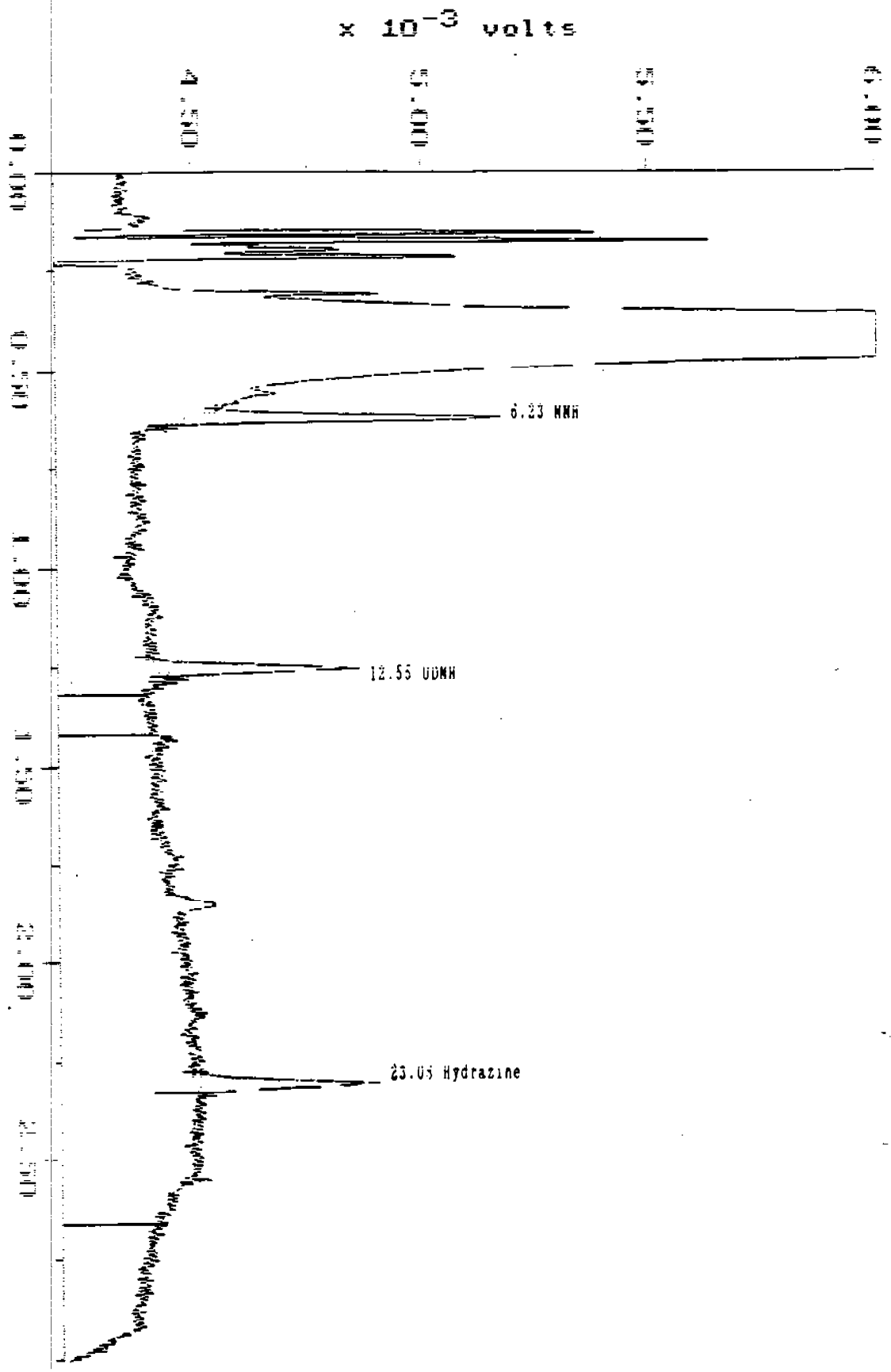
SAMPLE: CW2248 MOD#3
#9 in Method: EPA8315M,005 COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 17:15
Rate: 20.0 points/sec
Duration: 30.006 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991709
Index: 3

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.291	4800	450
2	2	DDMH	12.556	3509	297
3	3	Hydrazine	23.124	5112	315
TOTAL				13421	1062

Sample: 602248 NDL#3 **Channel:** Ultraviolet **Filename:** AG991710
Acquired: 17-AUG-99 17:48 **Method:** C:\MAX\DATA1\EPA3315M **Operator:** DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1993 5:14:40

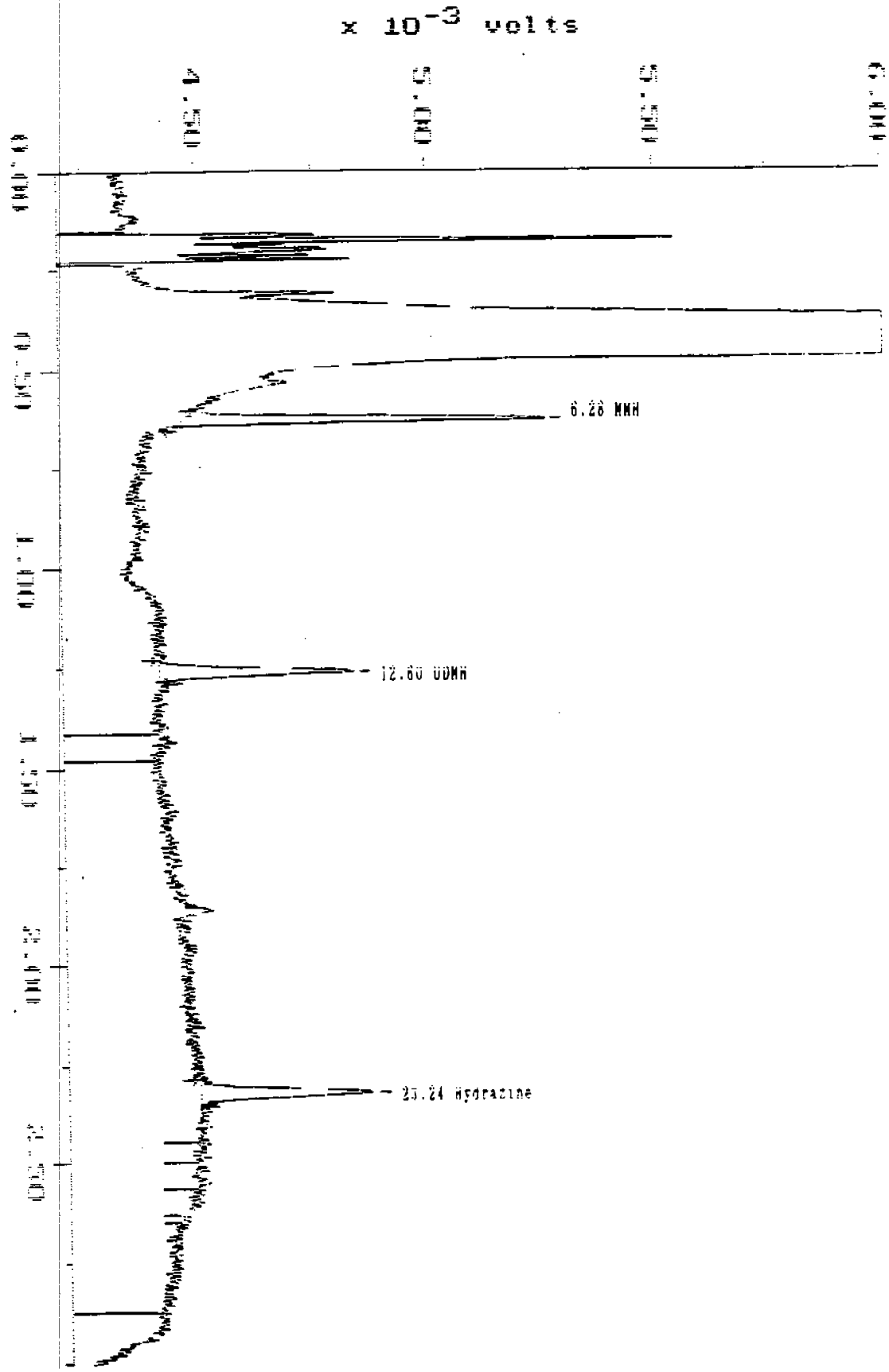
SAMPLE: 602246 MOL#3
#10 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1993 17:48
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: 4093170
Index: 10

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.232	7883	635
2	2	UDMH	12.546	5120	406
3	3	Hydrazine	23.079	5494	352
TOTAL				18477	1393

Sample: 602248 MDL#5 Channel: Ultraviolet Filename: AG991712
Acquired: 17-AUG-99 18:56 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:14:58

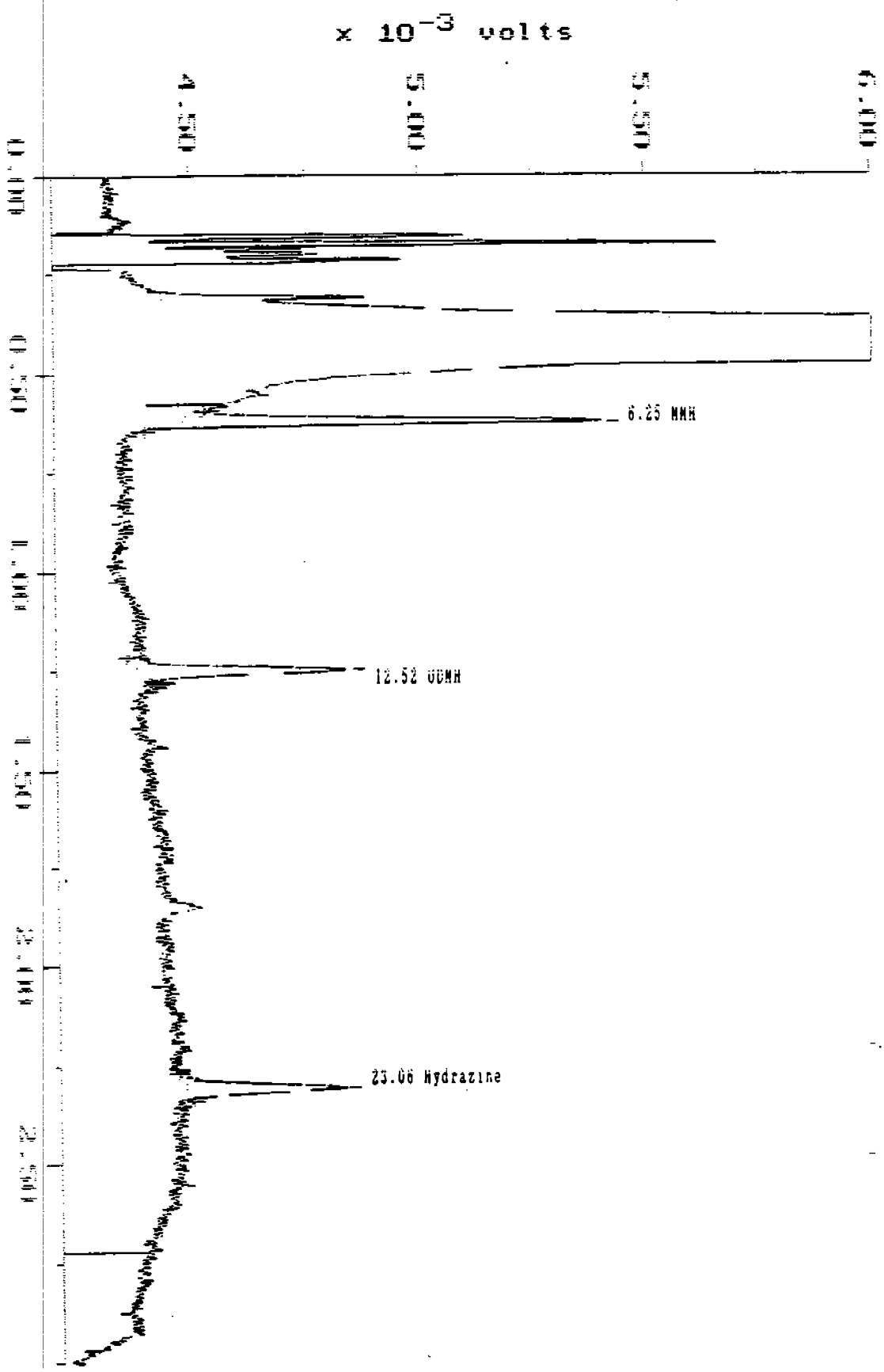
SAMPLE: 602248 MDL#4
#11 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 18:22
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991711
Index: 11

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.248	10267	938
2	2	UDMH	12.516	6127	446
3	3	Hydrazine	23.064	5156	340
TOTAL				21551	1722

Sample: 602248 MDL#4 **Channel:** Ultraviolet **Filename:** AG991711
Acquired: 17-AUG-99 18:22 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DB



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:15:16

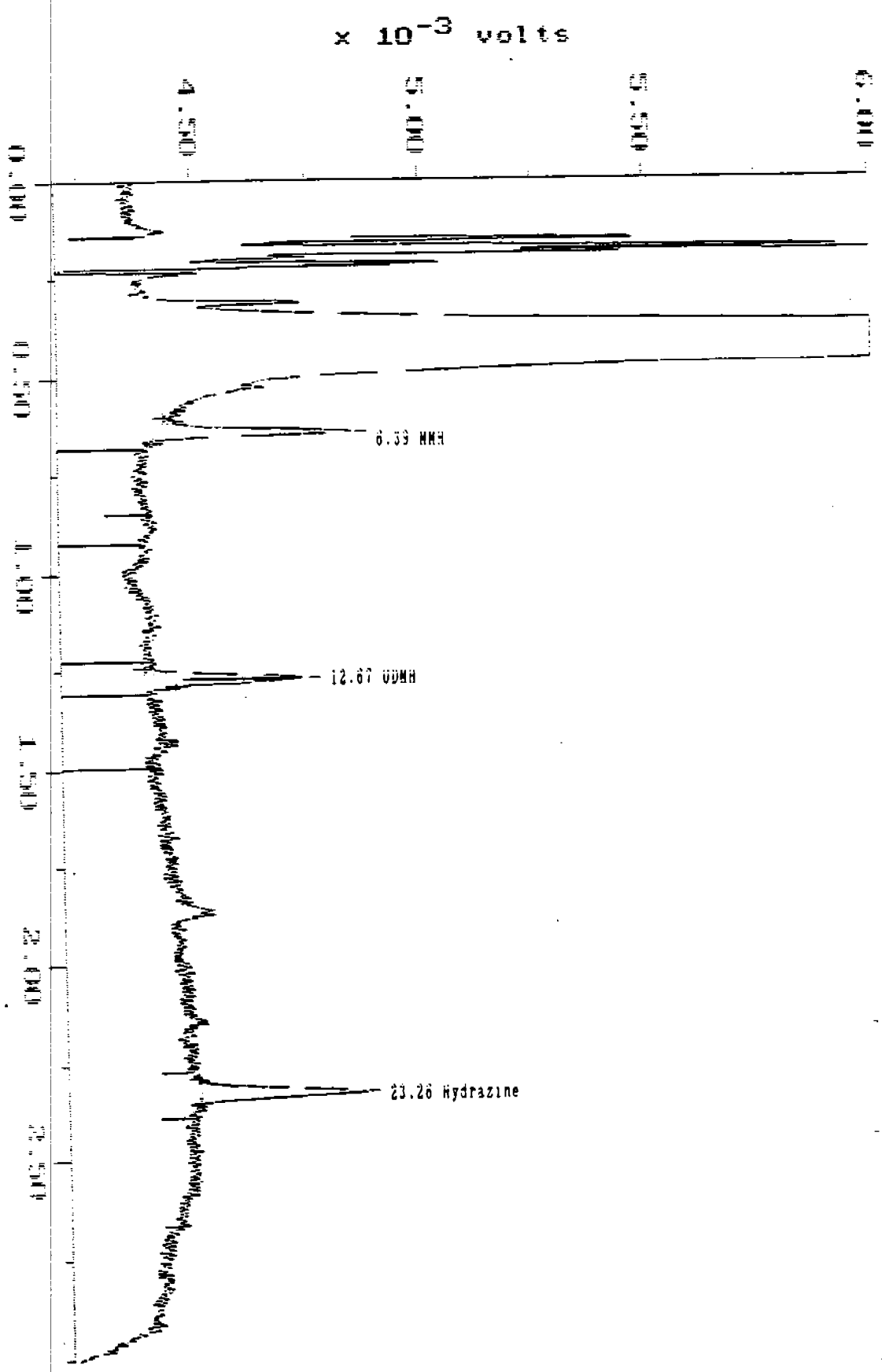
SAMPLE: 602248 MOL#5
#12 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 18:58
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: On

Type: UNKN
Instrument: Shimadzu 01
Filename: AU991712
Index: 12

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.273	8302	795
2	2	UDMH	12.604	5250	415
3	3	Hydrazine	23.237	5549	362
TOTAL				19101	1571

Sample: 602248 MDL#6 Channel: Ultraviolet Filename: AG991713
Acquired: 17-AUG-99 19:29 Method: C:\MAI\DATAI\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:15:35

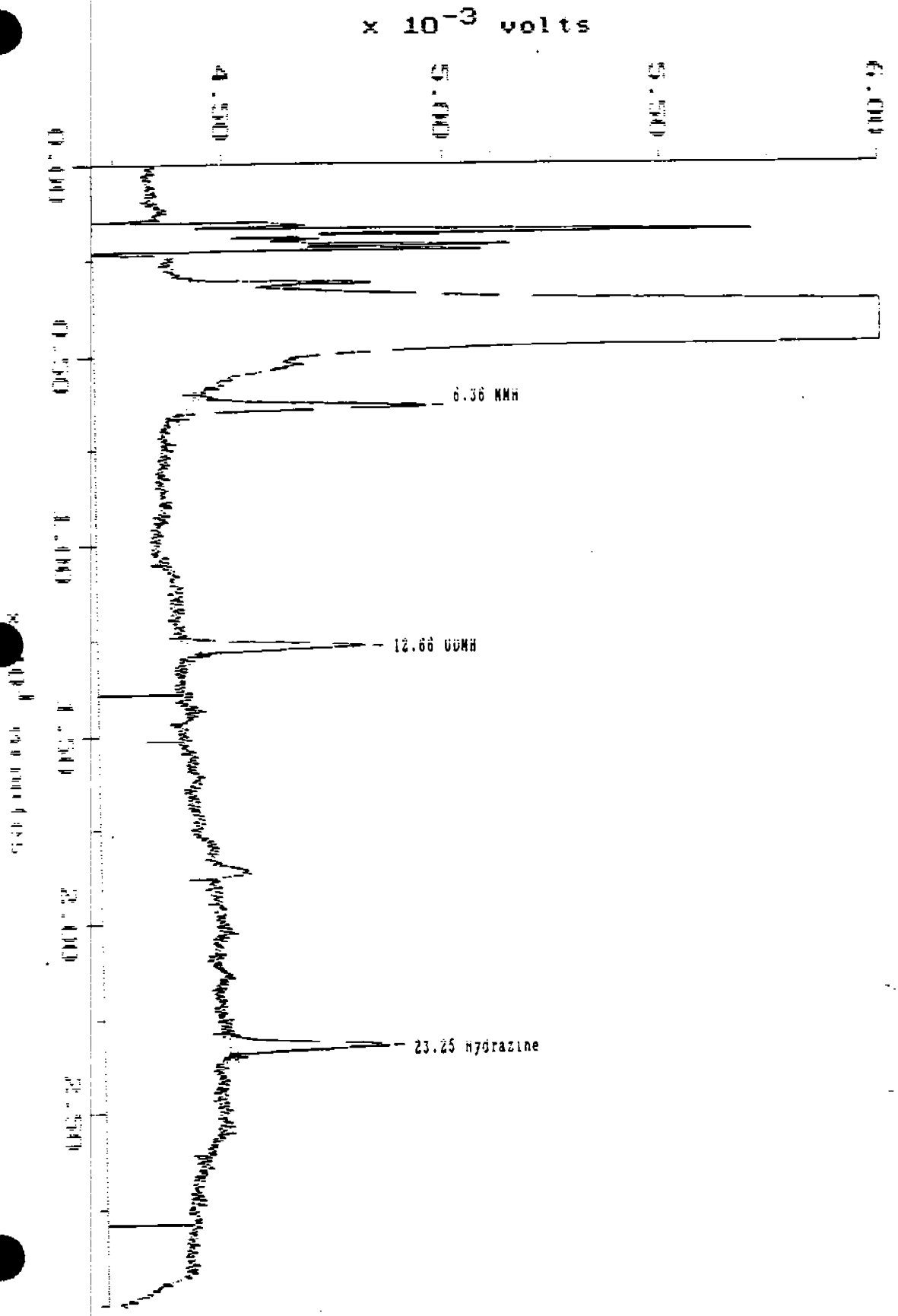
SAMPLE: 602246 MDL#6
#13 in Method: EPA8315M,DDS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 19:29
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991713
Index: 13

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.393	5573	417
2	2	UDMH	12.671	3951	310
3	3	Hydrazine	23.263	5417	352
TOTAL				14741	1079

Sample: 602248 MDL#7 Channel: Ultraviolet Filename: AG991714
Acquired: 17-AUG-99 20:03 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:15:53

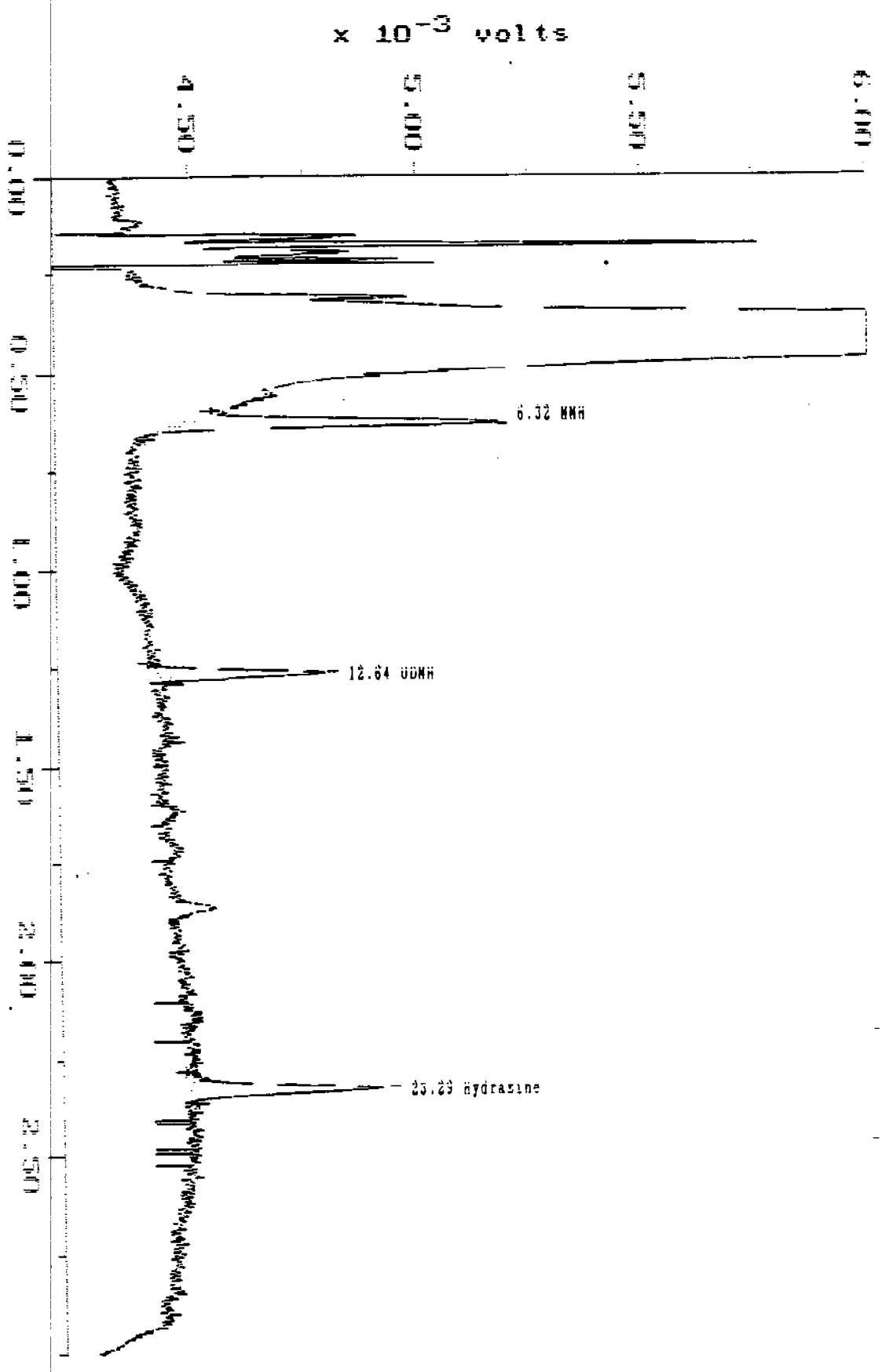
SAMPLE: 603248 MDL#7
#14 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 20:03
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991714
Index: 14

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1	1	MMH	6.360	6535	533
2	2	UDMH	12.663	5035	397
3	3	Hydrazine	23.247	5358	355
TOTAL				16928	1285

Sample: 602248 MDL88 Channel: Ultraviolet Filename: AG951715
Acquired: 17-AUG-99 20:37 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:16:11

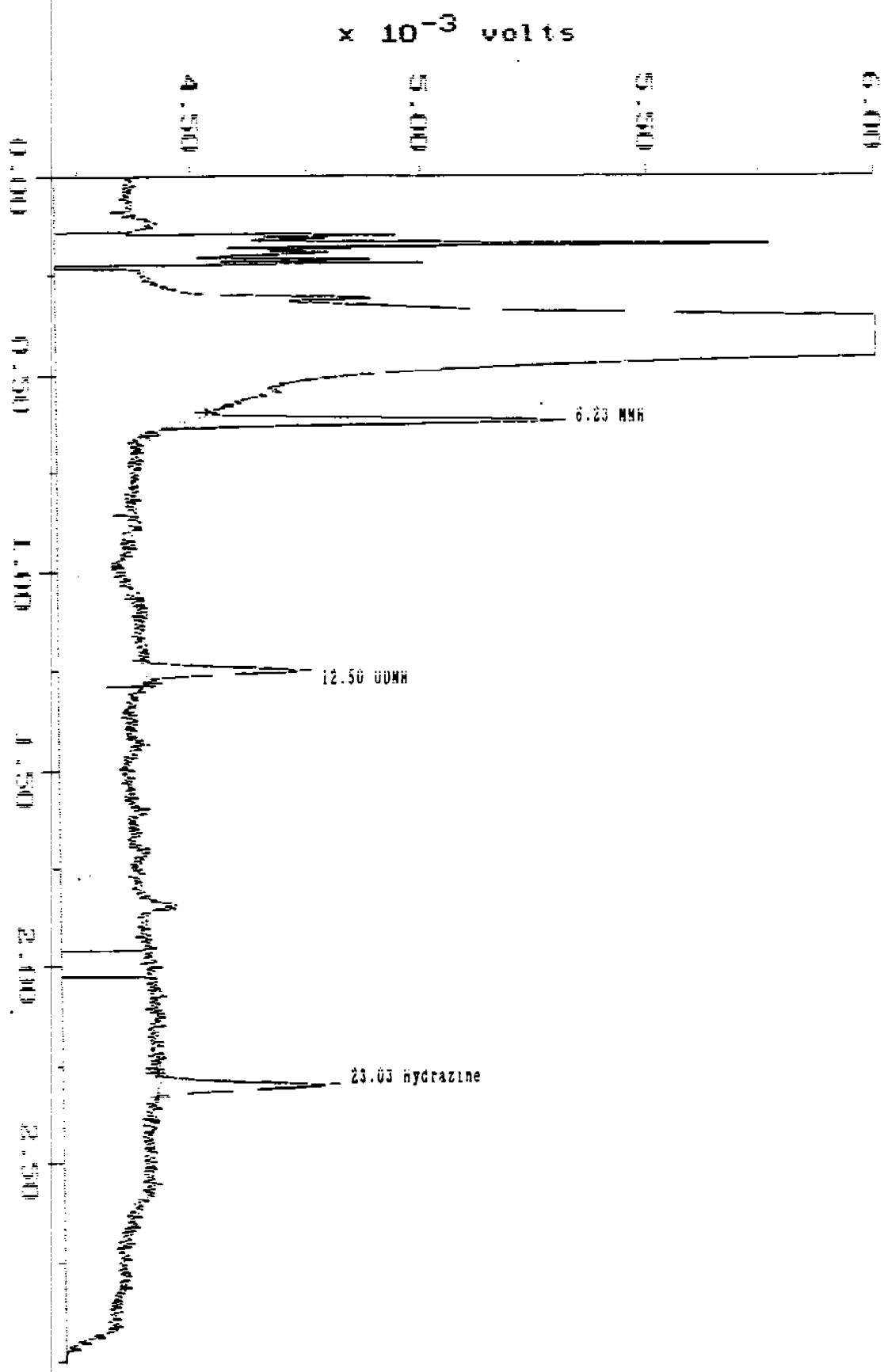
SAMPLE: 602248 MDL#8
#15 in Method: EPA8315M,ODS COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 20:37
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991715
Index: 15

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.318	9380	691
2	2	UDMH	12.643	4854	352
3	3	Hydrazine	23.287	6778	418
TOTAL				21012	1461

Sample: 602248 MDL89 Channel: Ultraviolet Filename: AG331716
Acquired: 17-AUG-99 21:11 Method: C:\MAX\DATA1\EPA8315M Operator: DH



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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:16:29

SAMPLE: 602845 MDL#3
#16 in Method: EPA8315M,ODS COL,SHIMADZU LC-10V
Acquired: 17-AUG-1999 21:11
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 9A
Filename: A0991716
Index: 16

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
1	1	MMH	6.231	9497	811
2	2	UDMH	12.503	3972	315
3	3	Hydrazine	23.025	5293	358
TOTAL				18762	1485

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BASELINE 810 CUSTOM REPORT

Printed: 18-AUG-1999 5:40:55

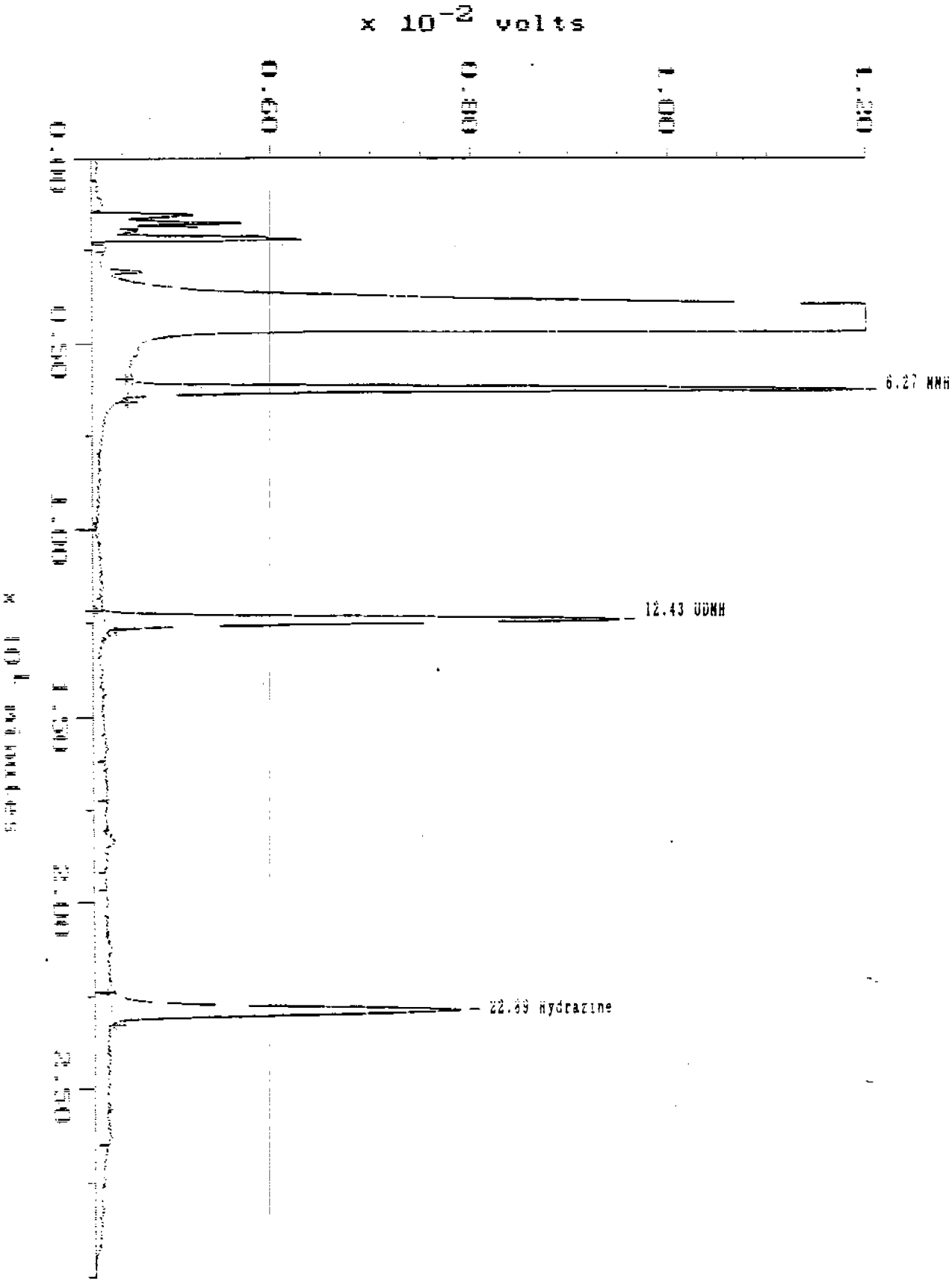
SAMPLE: CHECK STD#4
#17 in Method: EPA8315M,ODS COL.SHIMADZU GC/UV
Acquired: 17-AUG-1999 21:44
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: STND
Instrument: Shimadzu GA
Filename: AG991717
Index: 17

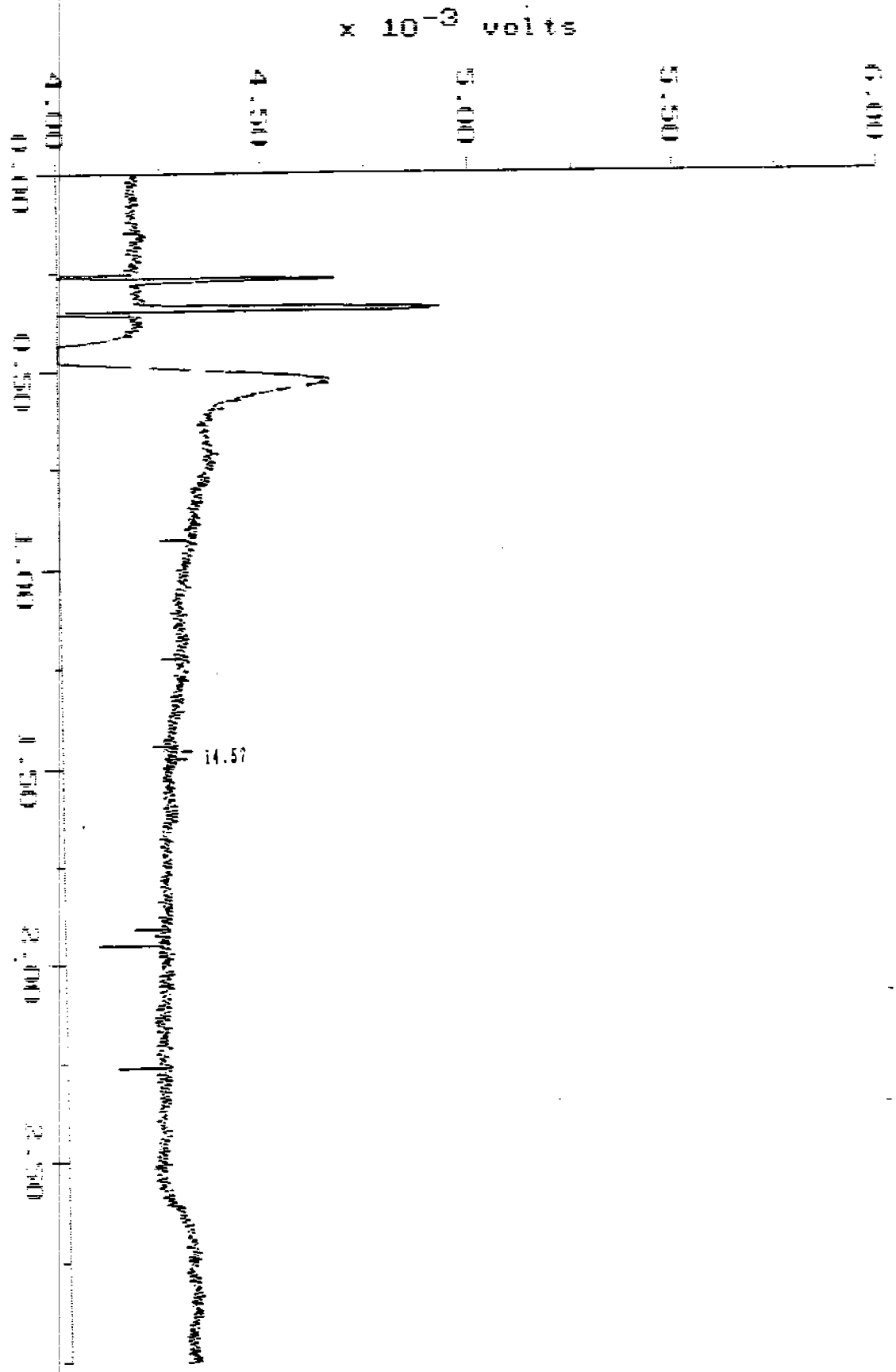
DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1	1	MNH	6.271	76801	7381
2	2	UDMH	12.429	66943	5139
3	3	Hydrazine	22.888	57273	3523
TOTAL				201016	16043

Sample: CHECK STD#4 **Channel:** Ultraviolet **Filename:** AG991717
Acquired: 17-AUG-99 21:44 **Method:** C:\MAX\DATA1\EPA8315M **Operator:** DH



Sample: MP BLANK 3 Channel: Ultraviolet Filename: AG991718
Acquired: 17-AUG-99 22:18 Method: C:\MAX\DATA1\8PA8315M Operator: DH



BASELINE (c)1990 Dynamic Solutions, Division of Millipore

BASELINE 810 CUSTOM REPORT

Printed: 16-AUG-1999 6:22:06

SAMPLE: MP BLANK 3
#18 in Method: EPA8315M.005 COL,SHIMADZU LC/UV
Acquired: 17-AUG-1999 22:16
Rate: 20.0 points/sec
Duration: 30.000 minutes
Operator: DH

Type: UNKN
Instrument: Shimadzu 6A
Filename: AG991718
Index: 18

DETECTOR: Ultraviolet

PK#	ID#	Component Name	Retention Time (minutes)	Peak Area	Peak Height
---	---	---	---	---	---
1			14.568	14	3
TOTAL				14	3

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

October 6, 1999

Charles Hamilton
Truesdail Laboratories, Inc.
14201 Franklin Ave.
Tustin, CA 92780-7008

Subject: AUDIT REPORT/RESPONSE

Dear Mr. Hamilton,

I have reviewed the response to Mr. Cirillo's audit report that you and Mr. Joe Bramblett provided to Earth Tech.

All responses are acceptable except responses to General Comments 1. and 2., and Finding 1 in the HPLC section. Since Mr. Cirillo left Earth Tech shortly after performing the audit, I will comment on these responses.

Telephone 562.951.2000
Facsimile 562.951.2100

1. A draft SOP dated 12/98 was provided with the response. This SOP is extremely incomplete, as was the case with the 12/97 SOP. Although it does address the preparation/extraction of samples for hydrazine analysis (with proprietary information omitted) and gives the HPLC operation conditions, it does not contain any information regarding calibration frequency, number of standards, acceptance criteria, use of QC samples, calculations of results, etc. It also does not contain any literature references, except a statement that it is a modification to EPA method 8315.

   This SOP must contain a proper reference to the appropriate EPA method. If there are, assuming that I understand your statement correctly, minimal changes from the "old" 8315 (1994) to the updated 8315A (1996) why does Truesdail not use the 8315A as a basis for the modification? Since the updated 8315A also contains references to the updated 8000B, the laboratory should reference the more recent update.

   Since there are no calibration or QC requirements listed in the SOP, it is imperative that the SOP include this information or reference the appropriate sections of the EPA procedure so that it is clear which requirements need to be followed. If the EPA method is referenced for the information, a copy of the EPA method must be attached to the SOP.

   As Mr. Cirillo commented, it is not apparent that the laboratory is following the requirements specified in the OU4 Addendum II QAPP. Normally, these requirements would supersede your SOP. However, if you have updated the protocol substantially since the QAPP went final, please submit a corrected version of the requirements in the same format as the QAPP Addendum II tables.

   Although you described the modifications to the method in your letter to us, these modifications need to be described in a section in the SOP titled "Variances" or "Modifications", as suggested by Mr. Cirillo.

2. Although we recently received revised reports, we have not yet received any response to Mr. Cirillo's letter of 7/29/99. Responses to the items in this letter would help clarify the

protocol at Truesdail laboratories. Furthermore, your response did not address the entire audit comment.

3. According to Mr. Cirillo's statement, the 9/94 (apparently EPA version) was available in the lab, but not the 12/97. Per your statement this is in revision but the lab has the "current version" – what is the current version? Is it the 12/98 or the version that you are currently working on? The response is confusing. An approved version should always be available in the lab. Even if a SOP is being updated, the previously approved version should be available.

4. Please provide a copy of the complete, updated SOP (with proprietary information omitted) when your revision is complete.

Please provide a response to these comments by October 20, 1999. The response to Mr. Cirillo's letter should be provided also since it is much overdue.

Very truly yours,

Earth Tech

Elisabeth Fruth

Elisabeth Fruth
QA Manager

E.S. BABCOCK & SONS LABORATORIES

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

March 24, 2000

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court
Riverside, CA 90503

Attn. Sean Jenkins

Subject: AUDIT REPORT

Audit Date: March 16, 2000

Audit Location: E.S. Babcock & Sons, Inc., Riverside, CA

Audit Scope: The laboratory had not received any Earth Tech project plans. Audit emphasis was on compliance with State of California Department of Health Services "Determination of Perchlorate by Ion Chromatography", Rev 0, laboratory SOPs, and the laboratory's Quality Assurance Manual. Attention was also given to the ability and necessity of the laboratory to follow Earth Tech project plans. Earth Tech project plans for Edwards AFB Operable Unit No. 4 Addendum II and Benicia TCUP Project were delivered to Sean Jenkins, Quality Assurance Manager.

Telephone
562.951.2000
Facsimile
562.951.2100

Auditor: Angela Wranic

Personnel Contacted: Lawrence Chrystal, Sean Jenkins, Allison Mackenzie, Karen Tracy, Alanna Wilkinson, Cindy Waddell, and Stacey Fry.

Audit Summary: The audit's focus was the perchlorate analysis. Interviews were conducted with laboratory personnel in the shipping and receiving area and the analyst who performs the perchlorate analysis. The QA program was discussed with Sean Jenkins the Quality Assurance Manager. A debriefing was held where the audit findings, concerns and recommendations from the laboratory audit were summarized. The debriefing was attended by Lawrence Chrystal, Laboratory Technical Director and Sean Jenkins.

Major Findings (Perchlorate Analysis)

1. The laboratory is not following their SOP for perchlorate analysis, which is based on the CADHS method. The laboratory SOP requires the ICV to be within ± 10 % of the true value. The analyst is using a criteria of ± 30%. This deficiency was confirmed by the laboratory internal QC audit conducted by Susie Thomas in April 1999. This deficiency was supposedly corrected per the documentation of the internal audit, but has not been resolved per our audit findings. The criteria of ± 10 % should be followed.
2. It is understandable that the laboratory did not follow Earth Tech QAPP requirements because they had not received copies of the Earth Tech project plans. This was a deficiency by Earth Tech personnel. Two project plans were supplied to laboratory at the time of the audit. The laboratory is now expected to follow the control limits in the Project Plans. Earth Tech Project Plans require a criteria of ± 10% for the ICV and CCV. Currently the laboratory is using a criteria of ±30% for the ICV and CCV. Earth Tech project plan control limits for the LCS (laboratory control sample) and MS/MSD (matrix spike and matrix spike duplicate) differ from laboratory limits. Earth Tech project plan limits are more stringent than the laboratory limits. If the control limits in the Earth Tech Project Plans cannot be followed the laboratory needs to supply Earth Tech with evidence that supports the proposed control limits, i.e., control charts. A variance can then be requested from AFCEE.

The following sections constitute the rest of the findings, concerns and recommendations grouped by area or subject.

Sample Receiving and Control *(Karen Tracy and Alanna Wilkinson)*

The following items and systems were reviewed: sample receipt, log-in, storage, tracking and anomalies; refrigerator monitoring; and sample security. Samples received are checked against the Chain-of-Custody for any discrepancies between sample labels and the Chain-of Custody. If discrepancies are found they are documented on the COC. If necessary the client is contacted by laboratory personnel. Temperature is generally not checked at the time of receipt. Samples are immediately logged into the computer system. Laboratory sample labels are generated and affixed to the samples. A computer printout of log-in information is generated and stored in a notebook. This information is permanently bound at a later date. Sample storage refrigerator temperatures are monitored and recorded daily.

Concerns

1. Internal chain-of-custodies are not used.
2. Temperature is not recorded for samples at the time of receipt. If a temperature blank is included in the cooler it is recommended the temperature be recorded. If a temperature blank is not in the shipment the temperature of the cooler should be measured and recorded. EPA Method 314.0, "Determination of Perchlorate In Drinking Water Using Ion Chromatography", Revision 1.0, November 1999 does not require perchlorate samples to be chilled to 4°C, but it does require samples be "protected from temperature

extremes." Recording the cooler temperature at time of laboratory receipt satisfies this requirement.

General Chemistry (Perchlorate Analysis) *(Cindy Waddell, Stacey Fry)*

The following information was reviewed: system for preparation of reagent water, calibration requirements, frequency and criteria of calibration checks, corrective action procedures, standards traceability, and a review of the maintenance logbook and SOP.

Finding:

1. The balance used for perchlorate analysis is checked daily with a 100 gram and a 0.1 gram weights. The measurement is recorded in a logbook. But the logbook does not include information on the tolerance range for the weights. The logbook should include a tolerance range and the analyst should check the measurements against the tolerance range.
2. Per the California Department of Health Services IC, Rev 0 a linear range study for the ion chromatograph should be conducted every 6 months. The laboratory has not met this requirement.

Concerns:

1. The maintenance logbook does not include any routine maintenance information such as maintenance performed on a daily, weekly, or monthly basis.
2. The maintenance logbook does not document return to control.

Balance and Thermometer Calibration *(Sean Jenkins)*

Documentation for calibration of balances, weights and thermometers were reviewed.

Concern:

1. The record of thermometer calibration and correction factors needs to be updated. Updated correction factors were entered on the sheet but were handwritten not typed. These changes to the typewritten text were not initialed and dated.
2. The yearly calibration for balances should be performed this month. It is scheduled by the laboratory to occur the week of March 20, 2000.
3. Documentation of yearly calibration for weights are available but poorly organized. It is recommended this documentation be organized so information can be easily tracked. Calibration for the weights was performed by an outside source in 1999. Outside calibration of weights should be performed at a minimum of once every 3 years.

Type II Water/Reagent Water *(Sean Jenkins)*

A Barnstead Nanopure water system is used in the inorganic sections. The resistance reading is monitored daily. The filter cartridge is replaced when the resistance reading is below 17.0.

Performance Evaluation Samples *(Sean Jenkins)*

PE sample results from Environmental Resource Associates (ERA) were reviewed. Results for the last year were reviewed. Results for method 300.0 were acceptable.

Finding:

1. No Performance evaluation sample was run for the perchlorate analysis. ERA has a low level perchlorate source available.

Data Review *(Sean Jenkins)*

Data review is performed by the analyst, supervisor, and the quality assurance manager. The supervisor reviews analyst calculations from the raw data. The quality assurance manager reviews the quality control for every analysis in the laboratory on a daily basis. Initials of the supervisor and quality assurance manager are documented in each project. The laboratory director signature is required for all final reports.

Finding

1. Checklists that outline what each person reviews are not used. It is important that the laboratory document exactly what each person has reviewed.

Audits *(Sean Jenkins)*

Laboratory audits are scheduled to be performed once a year. An audit checklist is used. Last year was the first year an internal audit was performed. Susie Thomas conducted the audit. A written letter is given to the analyst and supervisor summarizing corrective action that needs to be performed. Areas that need improvement and dates when this improvement, takes place are tracked on the audit checklist.

Finding

1. The laboratory SOP requires the ICV to be within ± 10 % of the true value. The analyst is using a criteria of ± 30%. This deficiency was confirmed by the laboratory internal QC audit conducted by Susie Thomas in April 1999. This deficiency was supposedly corrected per the documentation of the internal audit but has not been resolved per our audit findings. The criteria of ± 10 % should be followed.

Nonconformance/Corrective Action Reporting *(Sean Jenkins)*

Nonconformance (anomaly) reports are kept on record in project folders. A method of tracking nonconformances has just started. The reports are kept on file in the computer and the Quality Assurance Manager reviews these reports.

Concern:

1. No formal system exists for investigating and correcting systematic deficiencies.

Method Detection Limit Studies and ELAP certification *(Sean Jenkins)*

MDL studies for the perchlorate method are valid for one year. MDL studies were performed for water samples on 7/14/99 and soil on 8/31/99.

Documentation for extension of ELAP certification was received by the laboratory.

Standard Operating Procedures and Document Control *(Sean Jenkins)*

The Quality Assurance Manager distributes method SOPs to the analyst. A revision date is printed on each document that is issued.

Finding:

1. SOPs do not have a signature page. All SOPs need a signature page to indicate the SOP is approved by management. SOPs do not contain a revision number. All SOPs should have a revision number.

Concern:

1. Distribution of method SOP's to laboratory personnel is not tracked. It is recommended that a log be created that summarizes revisions of SOP's and date of issuance to laboratory personnel.
2. The SOP for perchlorate does not summarize standard preparation. It is recommended this information be included in the SOP.

Control Charts and Personnel Training *(Sean Jenkins)*

Personnel training files are maintained by departmental supervisors. New employees are trained by a co-worker or supervisor. The new analyst performs a demonstration of capability by analysis of unknown solutions. The work of a new employee is reviewed in depth for 3 weeks. Training is documented using a standardized checklist. Temporary personnel are not used frequently. When they are used they are subject to the same training procedures as a new employee.

Finding:

1. Control charts do not exist for perchlorate.

Concern:

1. Personnel training files exists for new employees but no documentation exists for

employees that have been employed by the laboratory for a number of years.

Data Review & Earth Tech Project Plans *(Sean Jenkins)*

Finding:

1. We could not distinguish which set of QC belonged to our sample in Attachment 1. How can this situation be avoided in future reports?
2. Laboratory reports have been received at the Long Beach office with cover sheets addressed to Chris Davis of the San Jose office.

Concern/Recommendation:

1. There was confusion at the laboratory distinguishing between different Earth Tech projects. It is important for the laboratory to be able to distinguish different projects because this determines which Earth Tech project plan to consult. Currently the laboratory is analyzing all Earth Tech Long Beach samples under the Project plan titled, Air Force Research Laboratory Operable Unit No. 4 Final Addendum II to Final Field Sampling Plan June 1999. This Project Plan was delivered to the laboratory on the day of the audit. The COC should indicate the project name. If the samples are received from Quanterra, Santa Ana and the project name is not on the COC contact them. Project numbers 32703, 35263, and 35261 are associated with the Edwards JPL project which is run out of the San Jose office by Chris Davis. The project plan used for these samples is titled, Quality Assurance Project Plan for Operable Units 5, 6, 8, and 10. The laboratory did not have this project plan. A telephone call was made to Chris Davis at the time of the audit. He was asked to provide this project plan to the laboratory. If the laboratory does not receive this plan they should contact Chris Davis at (408) 232-2800

I want to express my appreciation to all laboratory personnel for their interest, courteous assistance and time spent during this audit.

Please respond to the findings, concerns and recommendations enumerated in the audit report by April 7, 2000.

Very truly yours,

Earth Tech
Angela Wranic

Angela Wranic
Staff Professional VI

cc: Elisabeth Fruth
Chris Davis

E.S. BABCOCK & SONS, INC.
ESTABLISHED 1909

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e-mail: esbsales@aol.com
www.babcocklabs.com

Wet Chemistry - Batch QC

Log #: L64276
For Client: Quanterra Environmental Services
Date Rec'd: 01/14/2000
Lab Number: L64276-001 to -001

Analyte: Perchlorate WG47212 DHS-IC-Rev0

Reference	Ref Value	Units	QC Type	Result	Units	RPD	%Rec	Batch Date	LCL UCL
Blank	0.000	ug/L	MB	0.0	ug/L	---	---	000118CW	< RL
Spike Value	20.0	ug/L	LCS	21.9	ug/L	---	110.	000118CW	70% - 130%
L64348-001	0.0	ug/L	DUP	0.0	ug/L	N/A	---	000118CW	Max 20 RPD (b)
Spike Value	18.0	ug/L	MS	21.0	ug/L	---	117.	000118CW	(a)
WG47212-004	21.0	ug/L	MSD	19.7	ug/L	6.4	---	000118CW	Max 20 RPD (b)
Sample Spiked:	L64348-001	0	ug/L						
Blank	0.000	ug/L	MB	0.0	ug/L	---	---	000118CW	< RL
Spike Value	20.0	ug/L	LCS	21.7	ug/L	---	109.	000118CW	70% - 130%
L64216-001	0.0	ug/L	DUP	0.0	ug/L	N/A	---	000118CW	Max 20 RPD (b)
Spike Value	72.0	ug/L	MS	92.6	ug/L	---	129.	000118CW	(a)
WG47212-009	92.6	ug/L	MSD	92.1	ug/L	0.54	---	000118CW	Max 20 RPD (b)
Sample Spiked:	L64216-001	0	ug/L						
Spike Value	20.0	ug/L	LCS	21.9	ug/L	---	110.	000118CW	70% - 130%

Attachment 1

(a) Target range same as LCS Control Limits. If outside range, matrix interference suspected.
(b) Exceptions: Cyanide and Kjeldahl-Nitrogen: Max 30 RPD
Oil & Grease, Suspended Solids and Organic Compounds: Max 40 RPD

Susan K Thomas
E.S. Babcock & Sons, Inc.

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www.babcocklabs.com

05/03/00

Earth Tech
Attn: Angela Wranic
100 West Broadway, Suite 5000
Long Beach, CA 90802-4443

Angela:

This letter is in response to the findings from your audit conducted on March 16, 2000 at our laboratory.

1) The Perchlorate analysis now follows the QAPPs' acceptance criteria for ICV and CCV analysis. We are changing the ICV concentration to 25 µg/L. The CCV will alternate between a midpoint standard (125 µg/L) and a high standard (250 µg/L). The recovery for all of these standards must be between 90-110 %. (Note: EPA Method 314.0 criteria is 85-115%.) The SOP is currently being amended to reflect this change. This, I believe will also satisfy the requirements of your QAPPs.

2) The LCS concentration has been changed to 25 µg/L and has an acceptance criterion of 80-120% as per your QAPPs. The MS/MSD concentration has also been changed to 12.5 µg/L (multiplied by any prep/dilution factor). The acceptance range for MS/MSD is 80-120 % for waters with a maximum RPD of 20, and 75-125 % for soils with a maximum RPD of 35. This should meet the most stringent requirements of your QAPPs.

3) Internal Chain of Custody documentation is not a requirement for most of our work. We will utilize internal chain of custody for any samples received from Earth Tech in accordance with your QAPP requirements.

4) Currently, temperatures are recorded when a temperature blank is included with the ice chest. The laboratory is in the process of purchasing an infrared temperature probe to enable us to measure the temperature of samples directly when they arrive. (Please note: EPA Method 314.0 does NOT require the samples to be shipped on ice.)

5) The tolerance range is now indicated on the balance logbook.

6) The linear range study for the ion chromatograph is being performed. (Note: EPA Method 314.0 allows for second order fits.)

7) Any maintenance performed on the ion chromatograph is indicated in the maintenance log. The analyst has also been requested to indicate a return to service after the instrument has been taken off-line for repairs.

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05/03/00

8) A new record sheet has been created for thermometer calibration and correction factors. The daily temperature logbook has also been updated with the current information. The analyst has initialed the corrections in the logbook.

9) The yearly calibration for balances was completed on schedule.

10) The documentation for calibration of weights is now organized more clearly.

11) A PT sample from the EPA for perchlorate is currently in house. A PT sample from ERA was analyzed and reported last week. We should have the result of that analysis within three weeks.

12) We are currently reorganizing our review process with the addition of 1 more review level. We will take under consideration your recommendations regarding a checklist for reviews.

13) As noted in paragraph 1, the ICV acceptance range in our SOP is now being followed.

14) A computer tracking system is now in operation to investigate and correct systematic deficiencies.

15) We are currently amending our SOPs. The amended SOPs will have an approval/signature section and revision numbers. Our goal is to have this project completed by the end of this month.

16) We are developing a method for tracking the distribution of SOPs to laboratory personnel.

17) We are amending the SOP for Perchlorate to include the calibration standard and LCS preparation instructions.

18) A computer program is in development to generate control charts for all analytes in the laboratory. It is our goal to have this program in place by mid summer.

19) The Initial Demonstration of Capability that is required by NELAP is now being generated by all laboratory analysts. Documentation will be kept on file of the training of all analysts – both personally and by workcells.

20) All the samples in attachment 1 were processed together and analyzed in the sample analytical run. Currently, all samples from the analytical run are reported on the QC report. I am unable to change the format of the report. It can be determined which batch QC samples were analyzed before and after your sample by the checking the run order in the sequence (run) log. No sample QC (MS/MSD or DUP) was run on your sample. Samples chosen in-house for MS/MSDs and DUPs are of a similar matrix and at a frequency of 5% within the analytical run.

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05/03/00

21) Our laboratory reports for your projects are now being sent to the office and person indicated on the Chain of Custody.

22) We received the QAPP from Chris Davis for Operable Units 5,6,8 and 10.

Please feel free to contact Larry Chrystal or me if there are any questions.

Sincerely,

Sean H. Jenkins
QA Manager

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

May 12, 2000

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court
Riverside, CA 90503

Attn. Sean Jenkins

Subject: RESPONSE TO 05/03/2000 LETTER

Audit Date: March 16, 2000

Audit Location: E.S. Babcock & Sons, Inc., Riverside, CA

Comment: Please note my comments are indicated in bold within your text.

Telephone 562.951.2000
Facsimile 562.951.2100

1. The Perchlorate analysis now follows the QAPPs' acceptance criteria for ICV and CCV analysis. We are changing the ICV concentration to 25 µg/L. The CCV will alternate between a midpoint standard (125 µg/L) and a high standard (250 µg/L). The recovery for all of these standards must be between 90-110 %. (Note: EPA Method 314.0 criteria is 85-115%.) **Since the method specified in Earth Tech's QAPP and your SOP is DHS Modified 300.0, requirements in method 314.0 are not relevant at this time. You must follow what is outlined in the QAPP.** The SOP is currently being amended to reflect this change. **We need hard copy confirmation of this. When the SOP is changed please make a xerox copy and forward it to me. You response is considered inadequate until I receive this hard copy information.** This, I believe will also satisfy the requirements of your QAPPs.

2. The LCS concentration has been changed to 25 µg/L and has an acceptance criterion of 80-120% as per your QAPPs. The MS/MSD concentration has also been changed to 12.5 µg/L (multiplied by any prep/dilution factor). The acceptance range for MS/MSD is 80-120 % for waters with a maximum RPD of 20, and 75-125 % for soils with a maximum RPD of 35. This should meet the most stringent requirements of your QAPPs. **Please update your SOP and send us a xerox copy of this update.**

3. Internal Chain of Custody documentation is not a requirement for most of our work. We will utilize internal chain of custody for any samples received from Earth Tech in accordance with your QAPP requirements. **Please send hard copy verification of your internal COC for an EARTH TECH project.**

4. Currently, temperatures are recorded when a temperature blank is included with the ice chest. The laboratory is in the process of purchasing an infrared temperature probe to enable us to measure the temperature of samples directly when they arrive. (Please note: EPA Method 314.0 does NOT require the samples to be shipped on ice.) **It is recommended that the temperature of the cooler be recorded until you do obtain an infrared temperature probe. The method currently used is Modified 300.0 which does not require cooling.**

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

5. The tolerance range is now indicated on the balance logbook. **Send hard copy verification of this data.**

6. The linear range study for the ion chromatograph is being performed. (Note: EPA Method 314.0 allows for second order fits.) **Send hard copy verification of this data.**

7. Any maintenance performed on the ion chromatograph is indicated in the maintenance log. The analyst has also been requested to indicate a return to service after the instrument has been taken off-line for repairs. **Response is adequate.**

8. A new record sheet has been created for thermometer calibration and correction factors. The daily temperature logbook has also been updated with the current information. The analyst has initialed the corrections in the logbook. **Send hard copy verification of this data.**

9. The yearly calibration for balances was completed on schedule. **Send hard copy verification of this data.**

Telephone
562.951.2000

10. The documentation for calibration of weights is now organized more clearly. **Response is adequate.**

Facsimile
562.951.2100

11. A PT sample from the EPA for perchlorate is currently in house. A PT sample from ERA was analyzed and reported last week. We should have the result of that analysis within three weeks. **Please provide the results of these analyses where available.**

12. We are currently reorganizing our review process with the addition of 1 more review level. We will take under consideration your recommendations regarding a checklist for reviews. **Response inadequate. Please indicate your review process in more detail.**

13. As noted in paragraph 1, the ICV acceptance range in our SOP is now being followed. **See response item number 1.**

14. A computer tracking system is now in operation to investigate and correct systematic deficiencies. **If a specific deficiency were to appear multiple times on your computer tracking system what action would result?**

15. We are currently amending our SOPs. The amended SOPs will have an approval/signature section and revision numbers. Our goal is to have this project completed by the end of this month. **Send hard copy verification of this data.**

16. We are developing a method for tracking the distribution of SOPs to laboratory personnel. **Send hard copy verification of this data when it is complete.**

17. We are amending the SOP for Perchlorate to include the calibration standard and LCS preparation instructions. **Send hard copy verification of this data when it is complete.**

18. A computer program is in development to generate control charts for all analytes in the laboratory. It is our goal to have this program in place by mid summer. **Send hard copy verification of this data when it is complete.**

19. The Initial Demonstration of Capability that is required by NELAP is now being generated by all laboratory analysts. Documentation will be kept on file of the training of all analysts – both personally and by workcells. **Send hard copy verification of the Initial Demonstration of Capability for the analyst and supervisor of the perchlorate analysis.**

20. All the samples in attachment 1 were processed together and analyzed in the sample analytical run. Currently, all samples from the analytical run are reported on the QC report. I am unable to change the format of the report. It can be determined which batch QC samples were analyzed before and after your sample by the checking the run order in the sequence (run) log. **Would samples be associated with the QC run before or after? Could you include a copy of the run log in each job?** No sample QC (MS/MSD or DUP) was run on your sample. Samples chosen in-house for MS/MSDs and DUPs are of a similar matrix and at a frequency of 5% within the analytical run. **Whenever EARTH TECH has indicated on the COC which sample has been chosen as our MS/MSD for our projects, you should spike this sample. This is an Air Force requirement.**

21. Our laboratory reports for your projects are now being sent to the office and person indicated on the Chain of Custody. **Response adequate.**

22. We received the QAPP from Chris Davis for Operable Units 5,6,8 and 10. **Good**

Please respond to the findings, concerns and recommendations enumerated in the audit report by May 25, 2000.

Very truly yours,

Earth Tech

Angela Wranic

Angela Wranic
Staff Professional VI

cc: Elisabeth Fruth
Chris Davis

E.S. BABCOCK & SONS, INC.
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06/06/00

Earth Tech
Attn: Angela Wranic
100 West Broadway, Suite 5000
Long Beach, CA 90802-4443

Angela:

This letter is in response to the findings from your audit conducted on March 16, 2000 at our laboratory.

1) The Perchlorate analysis now follows the QAPPs' acceptance criteria for ICV and CCV analysis. We are changing the ICV concentration to 25 µg/L. The CCV will alternate between a midpoint standard (125 µg/L) and a high standard (250 µg/L). The recovery for all of these standards must be between 90-110 %. (Note: EPA Method 314.0 criteria is 85-115%.) The SOP is currently being amended to reflect this change. This, I believe will also satisfy the requirements of your QAPPs. (Enclosure 1)

2) The LCS concentration has been changed to 25 µg/L and has an acceptance criterion of 80-120% as per your QAPPs. The MS/MSD concentration has also been changed to 12.5 µg/L (multiplied by any prep/dilution factor). The acceptance range for MS/MSD is 80-120 % for waters with a maximum RPD of 20, and 75-125 % for soils with a maximum RPD of 35. This should meet the most stringent requirements of your QAPPs. (Enclosure 1)

3) Internal Chain of Custody documentation is not a requirement for most of our work. We will utilize internal chain of custody for any samples received from Earth Tech in accordance with your QAPP requirements. (Enclosure 2)

4) Currently, temperatures are recorded when a temperature blank is included with the ice chest. The laboratory is in the process of purchasing an infrared temperature probe to enable us to measure the temperature of samples directly when they arrive.

5) The tolerance range is now indicated on the balance logbook. (Enclosure 3)

6) The linear range study for the ion chromatograph is being performed. (Note: EPA Method 314.0 allows for second order fits.)(Enclosure 4)

7) Any maintenance performed on the ion chromatograph is indicated in the maintenance log. The analyst has also been requested to indicate a return to service after the instrument has been taken off-line for repairs.

E.S. BABCOCK & SONS, INC.
ESTABLISHED 1906

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06/06/00

8) A new record sheet has been created for thermometer calibration and correction factors. The daily temperature logbook has also been updated with the current information. The analyst has initialed the corrections in the logbook. (Enclosure 5)

9) The yearly calibration for balances was completed on schedule. (Enclosure 6)

10) The documentation for calibration of weights is now organized more clearly.

11) A PT sample from the EPA for perchlorate is currently in house. A PT sample from ERA was analyzed and reported last week. We should have the result of that analysis within three weeks. *We still haven't received the results of the ERA PT sample. We do have the results of the EPA PT sample.* (Enclosure 7)

12) We are currently reorganizing our review process with the addition of 1 more review level. We will take under consideration your recommendations regarding a checklist for reviews. For more information regarding our review process, see the Data Review and Validation SOP. (Enclosure 8)

13) As noted in paragraph 1, the ICV acceptance range in our SOP is now being followed. (Enclosure 1)

14) A computer tracking system is now in operation to investigate and correct systematic deficiencies. Client complaints, Quality Assurance Followups, and Amended Reports are tracked – including a statement of the problem, possible solutions, and results of followups. Investigations into multiple replications of problems would include an investigation by the analyst, supervisor, and QA Manager to determine the cause and solution to the problem. If needed, advice may be sought from other in-house management and/or expert outside consultation.

15) We are currently amending our SOPs. The amended SOPs will have an approval/signature section and revision numbers. Our goal is to have this project completed by the end of this month. (Enclosures 1 and 8)

16) We are developing a method for tracking the distribution of SOPs to laboratory personnel. (Enclosure 9)

17) We are amending the SOP for Perchlorate to include the calibration standard and LCS preparation instructions. (Enclosure 1)

18) A computer program is in development to generate control charts for all analytes in the laboratory. It is our goal to have this program in place by mid summer.

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19) The Initial Demonstration of Capability that is required by NELAP is now being generated by all laboratory analysts. Documentation will be kept on file of the training of all analysts – both personally and by workcells. (Enclosure 10)

20) All the samples in attachment 1 were processed together and analyzed in the sample analytical run. Currently, all samples from the analytical run are reported on the QC report. I am unable to change the format of the report. It can be determined which batch QC samples were analyzed before and after your sample by the checking the run order in the sequence (run) log. Both sets of QC must pass before we accept the analysis. A copy of the sequence log is included in each data package submitted. No sample QC (MS/MSD or DUP) was run on your sample. Samples chosen in-house for MS/MSDs and DUPs are of a similar matrix and at a frequency of 5% within the analytical run. When the client indicates a sample for QC, that sample is chosen for duplicate and/or MS/MSD analysis whenever sufficient sample is provided.

21) Our laboratory reports for your projects are now being sent to the office and person indicated on the Chain of Custody.

22) We received the QAPP from Chris Davis for Operable Units 5,6,8 and 10.

Please feel free to contact Larry Chrystal or me if there are any questions.

Sincerely,

Sean H. Jenkins
QA Manager

100 West Broadway, Suite 3000, Long Beach, California 90802

July 06, 2000

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court
Riverside, CA 90503

Attn. Sean Jenkins

Subject: Response to 6/30/2000 audit response

Audit Location: E.S. Babcock & Sons, Inc., Riverside, CA

1. Item number 1 was discussed with Sean Jenkins on 7/5/2000. She agreed to supply the page in the SOP that specifies Susann Thomas and Sean Jenkins as Larry Chrystal's (the Laboratory Director) designee. The SOP is currently being written and the needed page should be supplied by 7/10/2000.
2. Response to item number 2 is adequate.
3. Per conversation with Sean Jenkins she will supply the page in NELAP which supports the response to item number 3.
4. Response to item number 4 is adequate.

Please respond to the comments above by July 10, 2000.

Very truly yours,

Earth Tech

Angela Wranic
Staff Professional VI

cc: Elisabeth Fruth
Chris Davis

Wscpm9

100 West Broadway, Suite 5000, Long Beach, California 90802-4443

RECEIVED

July 12, 2000

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court
Riverside, CA 90503

Attn. Sean Jenkins

Telephone
562.951.2000
Facsimile
562.951.2100

Subject: Response to audit response

Audit Location: E.S. Babcock & Sons, Inc., Riverside, CA

Comments: I have received your last audit response on July 11, 2000. The responses to the audit deficiencies are acceptable and the audit is closed.

Very truly yours,

Earth Tech

Angela Wranic

Angela Wranic
Staff Professional VI

cc: Elisabeth Fruth
Chris Davis

E.S. BABCOCK & SONS, INC.
ESTABLISHED 1906

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06/30/00

Earth Tech
Attn: Angela Wranic
100 West Broadway, Suite 5000
Long Beach, CA 90802-4443

Angela:

This letter is in response to your letter of June 15, 2000 (copy enclosed).

1) NELAP requires that the signature on the SOP's are the Laboratory Director or his designee(s). All SOP's currently are signed by Susann Thomas (usually), Larry Chrystal (the Laboratory Director) or me.
2) The temperature at receipt will be recorded for your samples.
3) NELAP allows Initial Demonstration of Capability by Work Cell. The Ion Chromatography Work Cell includes Russell Kikkert, Kevin Sudds, Stacey Fry, and Cindy Waddell. Cindy Waddell will be the primary chemist performing the Perchlorate analysis. The other analysts also have Initial Demonstration of Capability of file for other IC analyses.
4) The sequence run log is included in each data package.

Please feel free to contact me if there are any furthere questions.

Sincerely,

Sean H. Jenkins
QA Manager

Enclosure #1

METHOD #: EPA 300.0 and 9056
SM 4110 B
CA DHS IC Rev 0

TITLE: The Determination of Inorganic Anions in Water by Ion Chromatography

ANALYTE:	CAS #
Bromate (BrO_3)	
Bromide Br	7726-95-6
Chlorate (ClO_3)	
Chloride Cl	7782-50-5
Chlorite (ClO_2)	
Fluoride F	7782-41-4
Nitrate (NO_3)	
Nitrite (NO_2)	
Phosphate (PO_4)	
Sulfate (SO_4)	
Perchlorate (ClO_4)	
Para-Chlorobenzene Sulfonic Acid (PCBSA)	

INSTRUMENTATION:

IC: Dionex 500DX and 120DX (see sec. 6.2)
Data Handling: Pentium Processor with Peak-Net software on Windows NT platform.
Printer: HP Laser Jet 2100
Autosampler: Alcott Micromeritics 728

1.0 Scope and Application

1.1. This method covers the determination of the following inorganic anions.

		RL, mg/L
1.1.1.	Method A.	
1.1.1.1.	Chloride	1
1.1.1.2.	Nitrate-N	0.2
1.1.1.3.	Nitrite-N	0.1
1.1.1.4.	Sulfate	0.5
1.1.1.5.	PCBSA	10
1.1.2.	Method B.	
1.1.2.1.	Bromide	0.02
1.1.2.2.	Chlorite	0.02
1.1.2.3.	Chlorate	0.02
1.1.2.4.	Bromate	0.005
1.1.3.	Method C	
1.1.3.1.	Perchlorate	0.004

Note: RL = Reporting Limit

1.2. The matrices applicable to each method are shown below:
   1.2.1. Drinking water, surface water, mixed domestic and industrial wastewaters, groundwater, reagent waters, solids (after extraction 2.3),
   1.2.2. Drinking water and reagent waters.
   1.2.3. Drinking water, groundwater and reagent waters.

1.3. The Single Laboratory Method Detection Limit (MDL, defined in section 13.1) for the above analytes is listed in Tables 1A through 1C. The MDL for a specific matrix may differ from those listed, depending upon the nature of the sample.

1.4. The working range for these analytes is as follows:

1.4.1.	Chloride	1-250 mg/L
1.4.2.	Nitrate-N	1-250 mg/L
1.4.3.	Sulfate	1-400 mg/L
1.4.4.	Bromide	0.02-0.3 mg/L
1.4.5.	Bromate	0.005-0.03 mg/L
1.4.6.	Chlorate	0.02-0.75 mg/L
1.4.7.	Chlorite	0.02-0.75 mg/L
1.4.8.	Perchlorate	0.004-0.25 mg/L
1.4.9.	PCBSA	1-500 mg/L

1.5. This method is recommended for use only by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram. Each analyst must demonstrate the ability to generate acceptable results with this method, using the procedure described in Section 10.2.

1.6. When this method is used to analyze unfamiliar samples for any of the above anions, anion identification should be supported by the use of fortified sample matrix covering the anions of interest. The fortification procedure is described in Section 11.9.

2. Summary of Method

2.1. An aliquot Sample (25 µL for Method A, 200 µL for Method B, and 740 µL for Method C)of sample is injected into an eluent stream and passed through a series of ion exchangers. The system is comprised of a guard column, separator column, and suppressor device. These separate the ions based on their affinity for a low capacity, strongly basic ion exchanger. They are then directed onto a strongly acidic cation exchanger where they

are converted to their highly conductive acidic forms. The conductivity of these acid forms is measured. Identification is based on retention time. Quantitation is based on peak height or peak area.

2.2. The main differences between Method A , B and C are the separator columns, guard columns and eluents. Sections 6 and 7 will elicit the differences.

2.3. In order to use this method for solids an extraction procedure must be performed (See Sec 11.10).

3. Definitions (see SOP Q15 for further definitions)

3.1. Stock standard solution - a concentrated solution containing a single certified standard that is a method analyte. Stock standard solutions are used to prepare calibration standards.

3.2. Calibration standards (CAL) - a solution of analytes prepared in the laboratory from stock standard solutions and diluted as needed and used to calibrate the instrument response with respect to analytic concentration.

3.3. Quality control sample (QCS) - a solution containing known concentrations of analytes, received quarterly from an outside vender (such as ERA). The analyzing laboratory uses this solution to demonstrate that it can obtain acceptable identifications and measurements with a method.

3.4. Performance evaluation sample (PE) - a solution of method analytes acquired from an outside source. A volume of the solution is added to a known volume of reagent water and analyzed with procedures used for samples. Analyte true values are unknown to the analyst.

3.5. Initial Calibration Check ICC (or Calibration Check standard) - a solution of analytes prepared in the laboratory by adding appropriate volumes of the stock standard solutions to reagent water used to evaluate the performance of the instrument system right after a calibration is performed. The low-level calibration standard is reinjected as well as the LCS to satisfy this requirement.

3.6. Laboratory duplicates (DUP) - two aliquots of the same sample that are treated exactly the same throughout laboratory analytical procedures. Analyses of laboratory duplicates indicate precision associated with laboratory procedures but not the sample collection, preservation, or storage procedures.

3.7. Laboratory fortified sample matrix (LFM) or Matrix Spike (MS) - An aliquot of an environmental sample to which known quantities of the method analytes are added in the laboratory. The LFM (or MS) is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentrations of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM (or MS) corrected for background concentrations.

3.8. Laboratory Control Sample (LCS) referenced in the method for oxyhalides as the Continuing Calibration Check and in the method for perchlorates as the Laboratory Fortified Blank and Instrument Performance Check. An aliquot of reagent water to which known quantities of the method analytes are added in the laboratory. The LCS is analyzed exactly like a sample, and its purpose is to determine whether the methodology is in control.

3.9. Reporting Level Check A standard is run daily at the reporting limit to demonstrate that the laboratory is capable of making accurate and precise measurements at the required reporting detection limit (a). Once a year this standard is run seven times in a row as part of a detection limit study (b).

3.10. Method Blank (MB) An aliquot of D.I. water is analyzed at the beginning of a run and every ten samples.

4. Interferences

4.1. Interferences can be caused by substances with retention times that are similar to and overlap those of the anion of interest. Large amounts of an anion can interfere with the peak resolution of an adjacent anion. Sample dilution and/or spiking can be used to solve most interference problems.

4.2. The water dip or negative peak that elutes near and can interfere with the fluoride peak can usually be eliminated by the addition of the equivalent of 1 mL of concentrated eluent (7.3 100X) to 100 mL of each standard and sample.

4.3. Method interferences may be caused by contaminants in the reagent water, reagents, glassware, and other sample processing apparatus that lead to discrete artifacts or elevated baseline in ion chromatograms.

4.4. Samples that contain particles larger than 0.45 microns and reagent solutions that contain particles larger than 0.20 microns require filtration to prevent damage to instrument columns and flow systems. Caution: filtration may remove perchlorate.

4.5. Any anion that is not retained by the column or only slightly retained will elute in the area of fluoride and interfere. Known coelution is caused by carbonate and other small organic anions. At concentrations of fluoride above 1.5 mg/L this interference may not be significant, however, it is the responsibility of the user to generate precision and accuracy information in each sample matrix.

4.6. The acetate anion elutes early during the chromatographic run. The retention times of the anions also seem to differ when large amounts of acetate are present. Therefore, this method is not recommended for leachates of solid samples when acetic acid is used for pH adjustment.

4.7. The quantitation of unretained peaks should be avoided, such as low molecular weight organic acids (formate, acetate, propionate, etc.) which are conductive and coelute with or near fluoride and would bias the fluoride quantitation in some drinking and most waste waters.

5. Safety

5.1. Normal, accepted laboratory safety practices should be followed during reagent preparation and instrument operation. No known carcinogenic materials are used in this method.

5.2. See SOP S01 , Concentrated Acids and Bases
SOP S02 – Compressed Gas Cylinder Handling
SOP S03 – Spill Control Policy

6. Apparatus and Materials

6.1. Balance - Analytical, capable of accurately weighing to the nearest 0.0001 g.

6.2. Ion chromatograph - Analytical system complete with ion chromatograph and all required accessories including syringes, analytical columns, compressed gasses and detectors.

6.2.1. Anion guard column: A protector of the separator column. If omitted from the system the retention limes will be shorter. Usually packed with a substrate the same as that in the separator column.

6.2.2. Anion separator column:
- 6.2.2.1. Anion separator column (Method A):
  - 6.2.2.1.1. AS-4A 4mm Dionex Column
  - 6.2.2.1.2. AG4A 4mm Dionex Guard Column
- 6.2.2.2. Anion separator column (Method B):
  - 6.2.2.2.1. AS-9HC 4mm Dionex Column
  - 6.2.2.2.2. AG-9HC 4mm Dionex Column
- 6.2.2.3. Anion separator column (Method C):
  - 6.2.2.3.1. AS-5 Dionex Column
  - 6.2.2.3.2. AG-5 Dionex Guard Column

6.2.3. Anion suppressor column:
- 6.2.3.1. Anion suppressor column (Method A): Anion self-regenerating ASRS-11.
- 6.2.3.2. Anion suppressor column (Method B and C): Anion micromembrane suppressor AMMS-11

6.2.4. Detector – CD20 conductivity cell.

7. Reagents and Consumable Materials

7.1. Sample bottles: Glass or polyethylene of sufficient volume to allow replicate analyses of anions of interest.

7.2. Reagent water: Nanopure, free of the anions of interest. Water should contain particles no larger than 0.20 microns with a conductance of <0.1uS/cm.

7.3. Eluent solution:
- 7.3.1. Method A: Dissolve 0.571 g sodium bicarbonate ($NaHCO^3$) and 0.763 g of sodium carbonate (Na^2CO^3) in 1 liter of nanopure water (7.2) and dilute to 4 liters.
- 7.3.2. Method B: Dissolve 5.298g Na_2CO_3 in 100 mL nanopure water. Add 36 mL of this solution to 1 liter of nanopure water and dilute to 2 liters.
- 7.3.3. Method C: Add 19.2 mL of 50% NaOH and 0.4765 g of 4-cyanophenol to 1 liter of nanopure water (degassed by Nanopure process DHS-IC-Rev 0 7.2). Dilute to 2 liters.

7.4. Regeneration solution (MicroMembrane Suppressor) Concentrated Sulfuric Acid:
- 7.4.1.1. 7.4.1 Method B: 0.7 mL per liter of nanopure water.
- 7.4.1.2. 7.4.2 Method C: 3.9 mL per 4 liters nanopure water.

7.5. Stock standard solutions, 1000 mg/L (1 mg/mL): Stock standard solutions are purchased as certified solutions.

Note: Stability of standards: Stock standards (7.5) are stable for at least one month when stored at 4-C. The bottle expiration dates are used as a guideline. Dilute working standards should be prepared each time a calibration is performed. LCS solutions are prepared weekly except method B solutions or those that contain phosphate which are prepared fresh daily (c).

8. Sample Collection, Preservation and Storage

8.1. Samples should be collected in scrupulously clean glass or polyethylene bottles.

8.2. Sample preservation and holding times for the anions that can be determined by this method are as follows.

Analyte	Preservation	Holding Time
Bromate	None required	28 days
Bromide	None required	28 days
Chlorate	None required	28 days
Chloride	None required	28 days
Chlorite	Cool to 4-C	immed.
	With EDA	14 days
Fluoride	None required	28 days
Nitrate-N		
chlorinated	Cool to 4°C	28 days
nonchlorinated	conc. H_2SO_4 pH < 2	14 days
o-Phosphate-P	Cool to 4°C	48 hours
Sulfate	Cool to 4°C	28 days
Perchlorate	Cool to 4°C	28 days
PCBSA	Cool to 4°C	28 days

8.3 The method of preservation and the holding time for samples analyzed by this method are determined by the anions of interest. In a given sample, the anion that requires the most preservation treatment and the shortest holding time will determine the preservation treatment. It is recommended that all samples be cooled to 4-C and held no longer than 28 days for Method A or Method C and analyzed immediately for Method B.

9. Calibration and Standardization
(See Attachment I for recipes of all standards.)

9.1. Establish ion chromatographic operating parameters equivalent to those indicated in Table 1A or 1B.

9.2. For each analyte of interest, prepare calibration standards at a minimum of three concentration levels (five for method C) by adding accurately measured volumes of one or more stock standards (7.5) to a volumetric flask and diluting to volume with reagent water. The curve is forced through the 0 point. An acceptable curve has a $r^2 \geq 0.995$. A method blank is analyzed after the calibration to verify this point (d). If a sample analyte concentration exceeds the calibration range the sample may be diluted to fall within the range. If this is not possible then three new calibration concentrations must be chosen, two of which must bracket the concentration of the sample analyte of interest. Each attenuation range of the instrument used to analyze a sample must be calibrated individually.

9.3. Using injections of 0.1 to 1.0 mL (determined by injection loop volume) of each calibration standard, tabulate peak height or area responses against the concentration. The results are used to prepare a calibration curve for each analyte. During this procedure, retention times must be recorded.

9.4. The calibration curve must be verified on each working day, or whenever the anion eluent is changed, and after every 20 samples. If the response or retention time for any analyte varies from the expected values by more than the range indicated under CCV standards, the test must be repeated, using fresh calibration standards. If the results are still out of range, a new calibration curve must be prepared for that analyte.

<u>ICV Standards</u>

	Analyte	Conc.	Acceptance Range %
Method A:	Cl	150ppm	90-110
	NO_3	111ppm	90-110
	SO_4	250ppm	90-110
Method B:	Br	20ppb	50-150
	ClO_2	20ppb	75-125
	ClO_3	20ppb	75-125
	BrO_3	5ppb	50-150
Method C:	ClO_4	25ppb	90-110

CCV Standards

	Analyte	Conc. Mid	Conc. High	Acceptance Range %
Method A:	Cl	10	150ppm	90-110
	NO_3	44.3	111ppm	90-110
	SO_4	30	250ppm	90-110
Method B: (ICR criteria)	Br	100	300ppb	90-110
	ClO_2	250	750ppb	90-110
	ClO_3	250	750ppb	90-110
	BrO_3	10	30ppb	80-120 90-110
Method C:	ClO_4	125	250ppb	90-110

Calibration Standards

Method A:

Std #1	Cl	1ppm	Std #4	Cl	100ppm
	NO_3	1ppm		NO_3	150ppm
	SO_4	1ppm		SO_4	150ppm
Std #2	Cl	10ppm	Std #5	Cl	200ppm
	NO_3	25ppm		NO_3	200ppm
	SO_4	30ppm		SO_4	350ppm
Std #3	Cl	20ppm	Std #6	Cl	250ppm
	NO_3	50ppm		NO_3	250ppm
	SO_4	60ppm		SO_4	400ppm

PCBSA Std #1 1ppm, Std #2 5ppm, Std #3 10ppm
Std #4 50ppm, Std #5 100ppm

Method B:

Std #1	ClO_3	20ppb	Std #3	ClO_3	750ppb
	ClO_2	20ppb		ClO_2	750ppb
	Br	20ppb		Br	300ppb
	BrO_3	5ppb		BrO_3	30ppb
Std #2	ClO_3	250ppb			
	ClO_2	250ppb			
	Br	100ppb			
	BrO_3	10ppb			

Method C:

ok

ClO_4: Std #1 4ppb, Std #2 10ppb, Std #3 50ppb
Std #4 100ppb, Std #5 250ppb

Lab Controls

Method A:

Acceptance range 90% - 110%

Cl	High	150 ppm	Low	10ppm
NO_3	High	111 ppm	Low	44.3ppm
SO_4	High	250 ppm	Low	30ppm

PCBSA 25ppm Acceptance range 80% - 120%

Method B:

Acceptance range 80% - 120%

ClO_3	400ppb
ClO_2	400ppb
Br	150ppb
BrO_3	15ppb

Method C:

Acceptance range 80% - 120% (e)

ClO_4	25ppb

Matrix Spikes

Method B:

Acceptance range 80% - 120%

ClO_3	400ppb
ClO_2	400ppb
Br	150ppb
BrO_3	15ppb

Method C:

Acceptance range: waters 80% - 120% max RPD 20
Soils 75% - 125% max RPD 35

ClO_4 12.5ppb X any prep or dilution factor

10. Quality Control

10.1. Our laboratory has a formal quality control program. The minimum requirements of this program consist of an initial demonstration of laboratory capability (10.2) and the analysis of control samples as a continuing check on performance. The laboratory maintains performance records to define and document the quality of data that are generated.

10.1.1. In recognition of the rapid advances occurring in chromatography, the analyst is permitted certain options to improve the separations or lower the cost of measurements. Each time such modifications to the method are made, the analyst is required to repeat the procedure in Section 10.2.

10.1.2. 5 to 10% of all samples are run in duplicate.

10.2. Before performing any analyses, the analyst demonstrates the ability to generate acceptable accuracy and precision with this method using a laboratory performance standard. Each analyst will analyze four replicates of a standard that is ten times their most recently proven MDL. Method C perchlorate requires four replicates at 25ppb. The acceptance criteria for this study is 90 – 110% recovery for water matrices and 80 – 120% recovery for solid matrices (f).

10.3. The laboratory develops and maintains accuracy statements of laboratory performance for each matrix being analyzed by the laboratory

10.4. Before processing any samples, the analyst demonstrates through the analysis of an aliquot of D.I. water (MB) that all glassware and reagent interferences are under control. Each time there is a change in reagents, the MB is monitored for the appearance of negative peaks as a safeguard against laboratory contamination (g).

10.5. When doubt exists over the identification of a peak in the chromatogram, confirmatory techniques such as sample dilution and fortification must be used.

10.6. Quality control check samples are analyzed concurrently with those performance evaluation sample studies required to maintain state certification.

10.7. In order to verify that standards have been prepared correctly a LCS is performed using a standard of known concentration from an independent source.

10.8. With each batch of samples processed analyze a single laboratory control sample containing each analyte of concern. If more than 20 samples are run in a batch analyze one LCS for every 20 samples (10 for drinking water). Evaluate the accuracy by comparing to laboratory acceptance criteria. If acceptable data cannot be obtained, locate the problem and correct it. If during the course of a run a LCS is out of range, if possible it is rerun on the spot. If this is not possible the analyst may reevaluate the data based on peak height rather than peak area. If the data still does not fall within the acceptance criteria, the analyst may choose to use the six point calibration curve (for method A) to interpret the data rather than the three point lower level curve. If all the LCS' are in range under these conditions, the data is accepted. Otherwise a fresh calibration is performed and all samples are rerun starting from the last acceptable LCS.

11. Procedure

11.1. Set-up:

11.1.1. Prepare Eluant. Turn He valve to 5psi for method A 65psi for method B, and 30 psi for Method C. Check that the He line is connected to the eluant bottle. Set pump rate as per table 1.

11.1.2. On peaknet program – click on run icon. Under file click on load method. Method A – anion 300; Method B – ICR.300HC99.met; Method C – a-clo4.met.

11.1.3. Wait for conductivity and pressure to stabilize.

11.2. Standardization and Calibration:

11.2.1. Using a clean syringe, fill one vial with the Method Blank.

11.2.1.1. Place vial in position #1 of autosampler.

11.2.1.2. Press <START> enter.

11.2.1.3. Init V <1> enter.

11.2.1.4. Rinse <0> enter.

11.2.1.5. Last V <1> enter.

11.2.2. Using a clean syringe, fill one vial with an initial calibration verification standard.

11.2.2.1. Place vial in position #2 of autosampler.

11.2.2.2. Press <START> enter.

11.2.2.3. Init V <1> enter.

11.2.2.4. Rinse <0> enter.

11.2.2.5. Last V <2> enter.

11.2.3. The initial calibration verification standard should read within the established control limits. If it does not, reinject it, if it still does not work, recalibrate.

11.2.3.1. Load calibration standards on the autosampler
11.2.3.2. Inject six calibration standards.
11.2.4. Check an initial calibration verification standard again.

11.3. Analysis:
11.3.1. Fill vials with sample, filtering (for Method A) through a 0.2 μm disc filter. For methods B & C, samples containing suspended material may be centrifuged or decanted.
11.3.2. Start the autosampler on vial 1 through 64.
11.3.2.1. Press <START> enter.
11.3.2.2. Init V <1> enter.
11.3.2.3. Rinse <0> enter.
11.3.2.4. Last V < # of last vial> enter.

Note: for method C, 2 vials per sample are loaded onto the autosampler.

11.3.3. Run continuing calibration verification standards every 10 samples. Run a lab control, method blank, and duplicate every 20 samples. Run matrix spikes every 20 samples for method B and C. Run a check standard at the end.
11.3.4. If a sample is above the high standard, dilute with D.I. water, according to the thickness and height of the peak. Make sure the peaknet software is calculating appropriately by observing peak heights and retention times.

11.4. Shutdown
11.4.1. Under Run – load stop method.
11.4.2. Turn pressure valve to 0 psi.

Note: Tables 1A and 1B summarize the recommended operating conditions for the ion chromatograph. Included in this table are estimated retention times that can be achieved by this method. Other columns, chromatographic conditions, or detectors may be used if the requirements of Section 10.2 are met.

11.5. Check system calibration daily and, if required, recalibrate as described in Section 9.

11.6. Load and inject a fixed amount of well mixed sample. Flush injection loop thoroughly, using each new sample. Use the same size loop for standards and samples. Record the resulting peak size in area or peak height units. An automated constant volume injection system may also be used.

11.7. The width of the retention time window used to make identifications should be based upon measurements of actual retention time variations of standards over the course of a day. The suggested peak window size for each analyte is $\pm$ 20% the standard deviation of the most recent low and high

standard (h). However, the experience of the analyst should weigh heavily in the interpretation of chromatograms.

11.8. If the response for the peak exceeds the working range of the system, dilute the sample with an appropriate amount of reagent water and reanalyze.

11.9. If the resulting chromatogram fails to produce adequate resolution, or if identification of specific anions is questionable, fortify the sample with an appropriate amount of standard and reanalyze.

Note: Retention time is inversely proportional to concentration. Nitrate and sulfate exhibit the greatest amount of change, although all anions are affected to some degree. In some cases this peak migration may produce poor resolution or identification.

11.10. The following extraction should be used for solid materials. Add an amount of reagent water equal to ten times the weight of dry solid material taken as a sample. This mixture is agitated for sixty minutes by shaking intermittently. Filter the resulting slurry before injecting using a 0.45 micron membrane type filter. With the exception of method C, this can be the type that attaches directly to the end of the syringe. Two samples per batch are spiked prior to extraction. These spikes are used to demonstrate that good recovery and identification of peaks is obtained with the users matrix.

12. Calculation

12.1. Prepare separate calibration curves for each anion of interest by plotting peak size in area, or peak height units of standards against concentration values. The system will then compute sample concentration by comparing sample peak response with the standard curve.

12.2. Report results in mg/L.

12.3. Report:

NO_2^- as N
NO_3^- as N or as NO_3 if desired by the client
$H(PO_4)_2^-$ as P

13. Precision and Accuracy - Method Detection Limit

13.1. The method detection limit (MDL) is defined as the minimum concentration of a substance that can be measured and reported with 99% confidence that the value is above zero. The MDL concentrations listed in Table 1A and 1B were obtained using reagent waters.

14. Calculations associated with this method:

14.1. Total Anions (TA)

mequiv. of OH + CO3 + HCO3 + SO4 + Cl + NO3 = TA

14.2. Electrochemical Balance (ECB)

Total Cations (TC) – Total Anions (TA)

14.3. Total Dissolved Solids by Summation (TDSSUM)

mg/L of 0.6(Total Alkalinity) + Na + K + Ca + Mg + SO4 + Cl + NO3 + F + SiO3 = TDSSUM

Table 1A. Chromatographic Conditions and Detection Limits In Reagent Water (Method A)

Analyte	Peak #	MDL (mg/L)
Fluoride	1	0.01
Chloride	2	0.792
Bromide	4	0.0015
Nitrate-N	5	0.0115
o-Phosphate-P	6	0.003
Sulfate	7	0.028
PCBSA	8	<1 (EDL)

Standard Conditions:

Unit:	DX 120
Columns:	as specified in 6.2.2.1
Detector:	as specified in 6.2.4
Pump Rate:	2.0 mL/min.
Eluent:	as specified in 7.3.1
Sample Loop:	25 uL

Table 1B. Chromatographic Conditions and Detection Limits In Reagent Water (Method B)

Analyte	Peak #	MDL (mg/L)
Chlorite	1	0.003
Bromate	2	0.0016
Bromide	3	0.0015
Chlorate	4	0.0011

Standard Conditions:

Unit:	DX500
Column:	as specified in 6.2.2.2
Detector:	as specified in 6.2.4
Pump Rate:	1.0 mL/min.
Eluent:	as specified in 7.3
Sample Loop:	200 uL

Table 1C. Chromatographic Conditions and Detection Limits In Reagent Water (Method C)

Analyte	MDL (mg/L)
Perchlorate	0.0018

Standard Conditions:

Unit:	DX500
Column:	as specified in 6.2.2.3
Detector:	as specified in 6.2.4
Pump Rate:	1.0 mL/min.
Eluent:	as specified in 7.3
Sample Loop:	740 uL

15.0 Corrective Action For Out of Control Or Unacceptable Data:

See SOP Q06 , Corrective Action

16.0 Pollution Prevention and Waste Management:

See SOP S05 , Neutralization Procedure for Acid and Alkaline Wastes
SOP S07 , Pollution Prevention

Method Variations

(a). Low Level Check Frequency EPA Method 300.0 section 10.8.
(b). Low Level Check Duplicates EPA Method 300.0 section 10.9.
(c). Stability of Standards - EPA Method 300.0 section 7.5.
(d). Blank in Calibration – EPA Method 300.0 section 9.2. and DHS-IC-Rev 0 section 10.2.
(e) LCS Acceptance Limit - California Department of Health Services IC Rev 0, section 9.3.2, 9.3.3.
(f). Demonstration of Capability EPA Method 300.0 section 10.2.
(g). Reagent Water Monitoring EPA Method 300.0 section 10.5.
(h). Retention Time Window – EPA Method 300.0 section 11.4.

References:

EPA SW846 method 9056
EPA 600/4-79/020 Methods for Chemical Analysis of Water and Wastes Method 300.0
California Department of Health Services IC Rev 0

Approved by Susann K Thomas 5/30/00

ATTACHMENT 1

Clo4-std

INTERMEDIATE STANDARDS 1																	
Analyte	Supplier	Lot No.	Stock	Std I.D.	grams used of Stock	Final Vol.	Final Conc	Date	Initials	Date-2	Initials-2	Date-3	Initials-3	Date-4	Initials-4	Date-5	Initials-5
KCLO4	Spectrum	KC054	salt	Std Int 1	0.0697g	50ml	1000 ppm	1/10/00	CAW								
KCLO4	Spectrum	KC054	salt	Spk Int high	0.3485g	50ml	5000 ppm	1/3/00	CAW								
Mg(CLO4)2	Baker	H01700	salt	LCS Int 1	0.056g	50ml	1000 ppm	1/10/00	CAW								
INTERMEDIATE STANDARDS 2																	
Analyte	Supplier	Lot No.	Stock mg/L	Std I.D.	mLs Used of Stock	Final Vol.	Final Conc	Date	Initials	Date-2	Initials-2	Date-3	Initials-3	Date-4	Initials-4	Date-5	Initials-5
KCLO4			Std Int 1	Std Int 2	50uL	50ml	1000 ppb	5/30/00	CAW								
Mg(CLO4)2			LCS Int 1	Spike Int 1	50uL	40ml	1250 ppb	5/1/00	CAW								
Mg(CLO4)2			LCS Int 1	LCS Int 2	50uL	50ml	1000 ppb	5/30/00	CAW								
WORKING STANDARDS																	
Analyte	Supplier	Lot No.	Stock mg/L	Std I.D.	mLs Used of Stock	Final Vol.	Final Conc	Date	Initials	Date-2	Initials-2	Date-3	Initials-3	Date-4	Initials-4	Date-5	Initials-5
KCLO4			Std Int 2	Cal 1	200uL	50ml	4ppb*	5/30/00	CAW								
KCLO4			Std Int 2	Cal 2	0.5ml	50ml	10ppb	5/30/00	CAW								
KCLO4			Std Int 2	Cal 3	2ml	40ml	50ppb	5/30/00	CAW								
KCLO4			Std Int 2	Cal 4	5ml	50ml	100ppb	5/30/00	CAW								
KCLO4			Std Int 2	Cal 5	10ml	40ml	250ppb**	5/30/00	CAW								
KCLO4			Cal 5	Mid CCV	10ml	20ml	125ppb	5/30/00	CAW								
Mg(CLO4)2			Spike Int 1	CLO4 Spk	0.5ml	50ml	12.5ppb	5/30/00	CAW								
KCLO4			Spk Int high	Soil Spk	50uL	50ml	5000ppb	5/30/00	CAW								
Mg(CLO4)2			LCS Int 2	CLO4 LCS	1ml	40ml	25ppb	5/30/00	CAW								

*Also used as low-level check.

**Also used as high CCV

NOTE: ALL INTERMED.(2) and WORKING STANDARDS EXPIRE SIX MONTHS FROM DATE MADE; ALL INTERMED.(1) STANDARDS EXPIRE 12 MONTHS FROM DATE MADE.

Analyte	Supplier	Lot No.	Stock mg/L	Std I.D.	mLs Used of Stock	Final Vol.	Final mg/L	Date	Initials	Notes	Date-2	Initials-2	Notes	Date-3	Initials-3	Notes	Date-4	Initials-4	Notes
Cl	EM	39320 (exp 7/30/02)	1000	Cal 1	50uL	50ml	1	3/10/00	KOS		4/21/00	KOS		5/22/00	KOS				
NO3	EM	39252 (exp 1/31/02)	1000		50uL	50ml	1												
SO4	EM	39261 (7/31/02)	1000		50uL	50ml	1												
Cl	EM	39130 (exp 3/31/02)	1000	Cal 2			10	3/10/00	KOS	Dilute Cal 3 (1:1)**	4/21/00	KOS	**	5/22/00	KOS	**			
NO3	EM	39252 (exp 1/31/02)	1000				25												
SO4	EM	39261 (7/31/02)	1000				30												
Cl	EM	39130 (exp 3/31/02)	1000	Cal 3	1	50ml	20	3/10/00	KOS		4/21/00	KOS		5/22/00	KOS				
NO3	EM	39252 (exp 1/31/02)	1000		2.5		50												
SO4	EM	39261 (7/31/02)	1000		3		60												
Cl	EM	39130 (exp 3/31/02)	1000	Cal 4	5	50ml	100	3/10/00	KOS		4/21/00	KOS		5/22/00	KOS				
NO3	EM	39252 (exp 1/31/02)	1000		7.5		150												
SO4	EM	39261 (7/31/02)	1000		7.5		150												
Cl	EM	39130 (exp 3/31/02)	1000	Cal 5	10	50ml	200	3/10/00	KOS		4/21/00	KOS		5/22/00	KOS				
NO3	EM	39252 (exp 1/31/02)	1000		10		200												
SO4	EM	39261 (7/31/02)	1000		17.5		350												
Cl	EM	39130 (exp 3/31/02)	1000	Cal 6	12.5	50ml	250	3/10/00	KOS		4/21/00	KOS		5/22/00	KOS				
NO3	EM	39252 (exp 1/31/02)	1000		12.5		250												
SO4	EM	39261 (7/31/02)	1000		20		400												

NOTE: Expiration date is 6 months from date made

ICR
INTERMEDIATE STANDARDS

Initial std. #2

ANALYTE	LOT #	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
ClO_2/ClO_3	969	EPA	12-1997	1000ppb	50 µL of each ClO_2 and ClO_3 1000 ppm stock std. Bring the volume up to 50 mls w/ Nano Pure.
Br	969	EPA	12-1997	1000ppb	50 µL of Br 10000 ppm stock std. Bring the volume up to500 mls w/ Nano Pure
BrO_3	969	EPA	12-1997	1000ppb	50 µL of Br O_3 1000 ppm stock std. Bring the volume up to50 mls w/ Nano Pure

Initial std. #2A

ANALYTE	LOT #	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
ClO_2/ClO_3	071599	ABSOLUTE	110299	1000ppb	50 µL of each ClO_2 and ClO_3 1000 ppm stock std. Bring the volume up to 50 mls w/ Nano Pure.
Br	071599	ABSOLUTE	110299	1000ppb	50 µL of Br 1000 ppm stock std. Bring the volume up to50 mls w/ Nano Pure
BrO_3	071599	ABSOLUTE	110299	1000ppb	50 µL of Br O_3 1000 ppm stock std. Bring the volume up to50 mls w/ Nano Pure

ICR
WORKING STANDARDS

IA. LOW LEVEL STD. #1

ANALYTE	LOT#	DATE RECEIVED	FINAL CONC.	RECIPE	
ClO_2/ClO_3	969	12-1997	20 ppb	1 ml of initial std. #2	Standard brought up to 50 mls of 1ppm Cl, NO_3, SO_4
BrO_3	969	12-1997	20 ppb	1 ml of initial std. #2	
Br	969	12-1997	5 ppb	250 µLs of initial std. #2	

IB. MID LEVEL STD. #2

ANALYTE	LOT#	DATE RECEIVED	FINAL CONC.	RECIPE	
ClO_2/ClO_3	969	12-1997	250ppb	12.5 mls of initial std. #2	Standard brought up to 50 mls of 1ppm Cl, NO_3, SO_4
BrO_3	969	12-1997	100ppb	5.0 mls of initial std. #2	
Br	969	12-1997	10 ppb	500 µLs of initial std. #2	

IC. HIGH LEVEL STD. #3

ANALYTE	LOT#	DATE RECEIVED	FINAL CONC.	RECIPE	Final Volume
ClO_2/ClO_3	969	12-1997	750ppb	18.75 mls of initial std. #2	27 mls
BrO_3	969	12-1997	300ppb	7.5 mls of initial std. #2	
Br	969	12-1997	30 ppb	750 µLs of initial std. #2	

ICR
INTERMEDIATE LAB CONTROL

LAB CONTROL INTERMEDIATE A

ANALYTE	LOT#	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
$NaClO_2$ $NaClO_3$	93451 G39750	EM Baker	102291 070494	1000PPM	0.0671g/ 50mls of nano pure 0.0638g/
Br	F183	RICCA	052798	1000ppb	50 µL and bring up w/ 50 mls of nano pure
$NaBrO_3$	31347317	EM	072794	1000PPM	.059g/50mls of nano pure

LAB CONTROL INTERMEDIATE A1

ANALYTE	LOT#	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
$NaClO_2$ $NaClO_3$	93451 G39750	EM Baker	102291 070494	1000ppb	50µL of intermediate A/ 50mls of nano pure
$NaBrO_3$	31347317	EM	072794	1000ppb	.50µL of intermediate A/50mls of nano pure

LAB CONTROL INTERMEDIATE B

ANALYTE	LOT#	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
$NaClO_2$ $NaClO_3$	93451 G39750	EM Baker	102291 070494	1000PPM	0.0671g/ 50mls of nano pure 0.0638g/
Br	1908617	RICCA	091599	1000ppb	50 µL and bring up w/ 50 mls of nano pure
$NaBrO_3$	31347317	EM	072794	1000PPM	.059g/50mls of nano pure

LAB CONTROL INTERMEDIATE B1

ANALYTE	LOT#	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
$NaClO_2$ $NaClO_3$	93451 G39750	EM Baker	102291 070494	1000ppb	50µL of intermediate A/ 50mls of nano pure
$NaBrO_3$	31347317	EM	072794	1000ppb	50µL of intermediate A/50mls of nano pure

LAB CONTROL INTERMEDIATE C

ANALYTE	LOT#	VENDOR	DATE RECIEVED	FINAL CONC.	RECIPE
ClO_2/ClO_3	969	EPA	12-1997	1000 ppb	50 µL of each ClO_2/ClO_3 1000 ppm stock lcs. Bring the volume up to 50 mls w/ nano
Br	969	EPA	12-1997	1000 ppb	50 µL of 10000 ppm stock lcs. Bring the volume up to 500 mls w/ nano
BrO_3	969	EPA	12-1997	1000 ppb	50 µL of BrO_3 1000 ppm stock lcs. Bring the volume up to 50 mls w/ nano

LAB CONTROL INTERMEDIATE D

ANALYTE	LOT #	VENDOR	DATE RECIEVED	FINAL CONC	RECIPE
ClO_2 ClO_3	012700 011800	ABSOLUTE	021600	1000 ppb	50µL of each ClO_2 and ClO_3 1000 ppm stock lab Control. Bring volume up to 50 mls w/ nano pure.
Br	120999	ABSOLUTE	021600	1000 ppb	50µL of Br 1000 ppm stock lab Control. Bring volume up to 50 mls w/ nano pure
BrO_3	012700	ABSOLUTE	021600	1000 ppb	50µL of Br 1000 ppm stock lab Control. Bring volume up to 50 mls w/ nano pure

WORKING LAB CONTROLS

WORKING LAB CONTROL 1

ANALYTE	LCS INTERMEDIATE	FINAL CONC. PPB	RECIPE	
ClO_2/ClO_3	A	400/400	10 mls of inter. A1	LCS brought up to a final volume of 25 mls w/ nano pure
Br		150	3.75 mls of inter. A	
BrO_3		50	.375 mls of inter. A1	

WORKING LAB CONTROL 2

ANALYTE	LCS INTERMEDIATE	FINAL CONC. PPB	RECIPE	
ClO_2/ClO_3	B	400/400	10 mls of inter. B1	LCS brought up to a final volume of 25 mls w/ nano pure
Br		150	3.75 mls of inter. B	
BrO_3		50	.375 mls of inter. B1	

WORKING LAB CONTROL 3

ANALYTE	LCS INTERMEDIATE	FINAL CONC. PPB	RECIPE	
ClO_2/ClO_3	C	400/400	10 mls of inter. C	LCS brought up to a final volume of 25 mls w/ nano pure
Br		150	3.75 mls of inter. C	
BrO_3		50	.375 mls of inter. C	

WORKING LAB CONTROL 4

ANALYTE	LCS INTERMEDIATE	FINAL CONC. PPB	RECIPE	
ClO_2/ClO_3	D	400/400	10 mls of inter. D	LCS brought up to a final volume of 25 mls w/ nano pure
Br		150	3.75 mls of inter. D	
BrO_3		50	.375 mls of inter. D	

Enclosure 2

Perchlorate Sample Checkout

Log No:	Sample No:	Date/Time out (MMDDYYHHMM)	Date/Time In (MMDDYYHHMM)	Initials
L68699	-007	050100 0800	050100 1400	CAW
	-008			
	-009			
L68629	-001			
L68880	-001	050800 0630	050800 1300	CAW
	-002	050800 0630	050800 1300	CAW
L69122	-001	051000 1400	051000 1500	CAW
L69317	-001	051600 0900	051600 1200	CAW
	-002	051600 0900	051600 1200	CAW
	-003	051600 0900	051600 1200	CAW

Enclosure 3

Date	Init.	Balance #1 100 ± 0.01g 0.1 ± 0.0001g	Balance #2 100 ± 0.01g 0.1 ± 0.0001g	Balance #5 100 ± 0.01g 0.1 ± 0.0001g

Enclosure 3

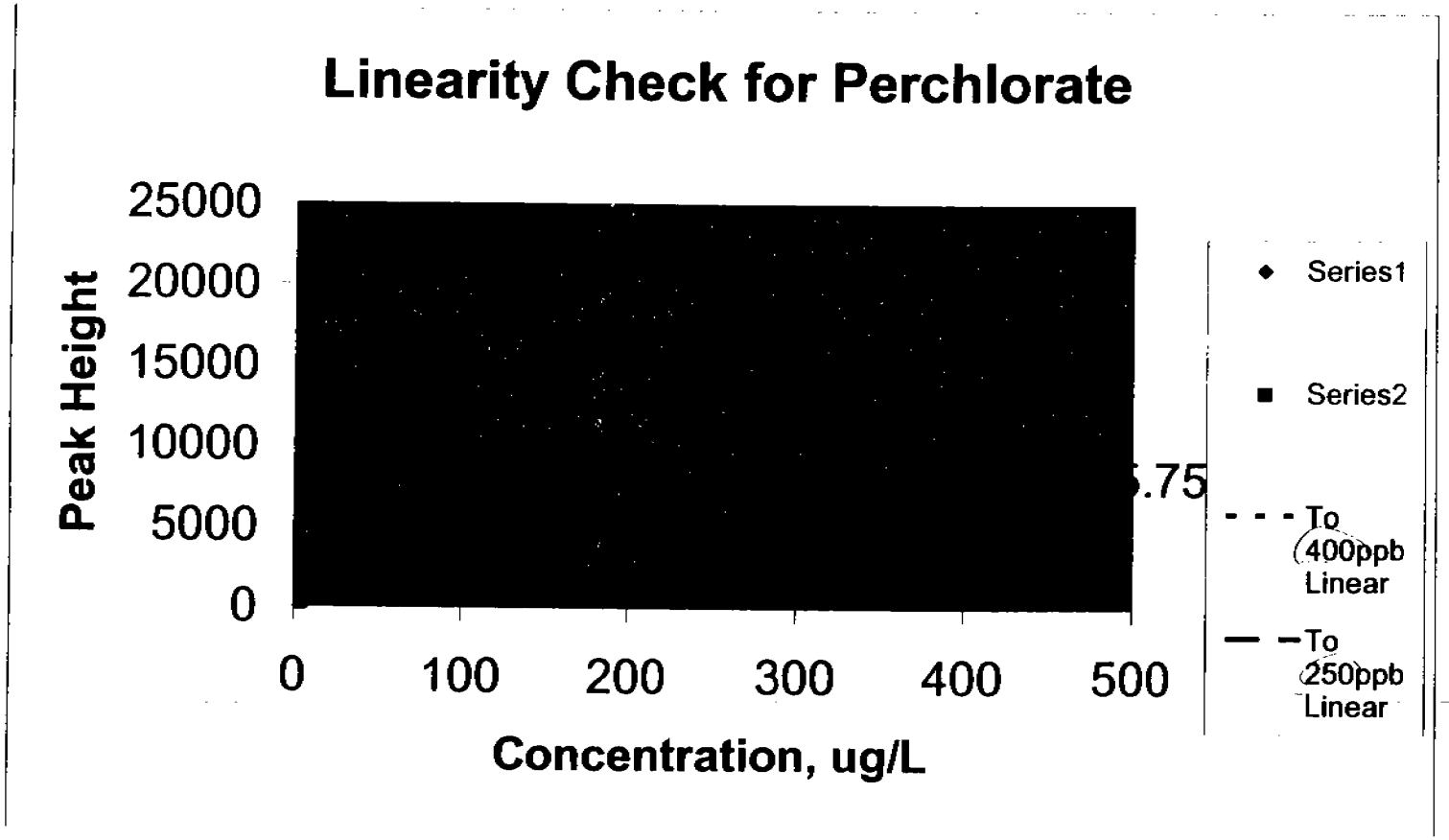
Date	Init.	Balance #3 50 ± 0.1g	Balance #4 50 ± 0.01g	

Enclosure 4

Conc. µg/L	Pk Height
4	204
10	579
50	2980
100	6187
250	13960
400	20081

Analysis Date: May 30, 2000

Linearity Criteria: $R \geq 0.995$ or $R^2 \geq 0.99$



Enclosure 5

Thermometer Calibration Log

Annual Thermometer Calibrations

ID	Reading, °C Reference Value*	Lab Thermometer	Correction	Date/Initials
I1				
I2				
O1				
O2				
F2				
R3				
R2b				
R2a				
R1				
R7				
R6				
F1				
R4				
F3				
R5				

Additional Calibrations

Temperature Log

Date/Init	Nanopure	I1 °C	I2 °C	O1 °C	O2 °C	F2 °C	R3 °C	R2b (°F)	R2a (°F)	R1 (°C)	R7 (°C)	R6 °C	F1 °C	R4 °C	F3 °C	R5 °C
	M Ohm	Corr 0°	Corr -1°	Corr +0.5°	Corr -2°	Corr 0°	Corr 0°	Corr -5°	Corr -5°	Corr +0.5	Corr +1°	Corr +0°	Corr 0°	Corr -0.5°	Corr 0°	Corr +0.5°
Range:	>17	19-21 °C	19-21 °C	103-105 °C	178-180 °C	< -5 °C	2-6 °C	35-43 °F	35-43 °F	2-6 °C	2-6 °C	2-6 °C	< -5 °C	2-6 °C	< -5 °C	2-6 °C

Enclosure 5

Enclosure 6

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC. PAGE 1
DEPARTMENT: Q.C.
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE ST: CA ZIP: 92507
MAKE: SARTROIUS MODEL: 2462
SERIAL#: 143007 CUSTOMER#: #2
CAPACITY: 200 GM
RESOLUTION: .1 MG
MANUFACTURES TOLERANCE: +/- .1 MG

DATE	INDICATED		CORRECTED		TECHNICIAN	DUE
3/20/00	0 - 200 GM	.1 MG	0 - 200 GM +/-	.1 MG	CHRIS MACE	MARCH
	0 - 100 GM	.1 MG	0 - 100 GM +/-	.1 MG		2001
	100- 200 GM	.1 MG	100-200 GM +/-	.1 MG		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
STATE OF CALIFORNIA DIVISION OF MEASUREMENT STANDARDS CERT. NUMBER: 0778.

PROCEDURES USED COMPLY WITH ONE OR MORE OF THE FOLLOWING PROCEDURES: N.I.S.T. HANDBOOK 145, HANDBOOK 44, A.S.T.M. CIRCULAR E 898, FDA CFR TITLE 21 PARTS: 210 TO 211, ISO 10012-1, ANSI/NCSL Z540-1. SNS FORM 1196.

BY: _____
SY NIELSON SERVICE, INC.

FORM: CALRPT

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC. PAGE 2
DEPARTMENT: Q.C.
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE ST: CA ZIP: 92507
MAKE: METTLER/TOLEDO MODEL: AB104
SERIAL#: 1116283705 CUSTOMER#:
CAPACITY: 100 GM
RESOLUTION: .1 MG
MANUFACTURES TOLERANCE: +/- .1 MG

DATE	INDICATED		CORRECTED		TECHNICIAN	DUE
3/20/00	0 - 100 GM	-6.4 MG	0 - 100 GM	+/- .1 MG	CHRIS MACE	MARCH 2001
	0 - 50 GM	-3.2 MG	0 - 50 GM	+/- .1 MG		
	50 - 100 GM	-3.2 MG	50- 100 GM	+/- .1 MG		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
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BY: _____
SY NIELSON SERVICE, INC.

FORM: CALRPT

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC. PAGE 3
DEPARTMENT: Q.C.
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE ST: CA ZIP: 92507
MAKE: TORBAL MODEL: PL3
SERIAL#: 506007 CUSTOMER#: #3
CAPACITY: 3 KG
RESOLUTION: .1 GM
MANUFACTURES TOLERANCE: +/- .1 GM

DATE	INDICATED		CORRECTED		TECHNICIAN	DUE
3/20/00	0 - 3 KG	-.1 GM	0 - 3 KG +/-	.1 GM	CHRIS MACE	MARCH
	0 - 1.5 KG	-.1 GM	0 - 1.5 KG +/-	.1 GM		2001
	1.5- 3 KG	-.1 GM	1.5-3 KG +/-	.1 GM		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
STATE OF CALIFORNIA DIVISION OF MEASUREMENT STANDARDS CERT. NUMBER: 0778.

PROCEDURES USED COMPLY WITH ONE OR MORE OF THE FOLLOWING PROCEDURES: N.I.S.T. HANDBOOK 145, HANDBOOK 44, A.S.T.M. CIRCULAR E 898, FDA CFR TITLE 21 PARTS: 210 TO 211, ISO 10012-1, ANSI/NCSL Z540-1. SNS FORM 1196.

BY S. Nielson
SY NIELSON SERVICE, INC.

FORM: CALRPT

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC.
DEPARTMENT: BACTERIA
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE
MAKE: SARTORIUS
SERIAL#: 3402089
CAPACITY: 110 GM
RESOLUTION: .1 MG
MANUFACTURES TOLERANCE: +/- .1 MG

PAGE 4

ST: CA ZIP: 92507
MODEL: 1601
CUSTOMER#: # 1

DATE	INDICATED	CORRECTED	TECHNICIAN	DUE
3/20/00	0 - 110 GM + 1.0 MG	0 - 110 GM +/- .1 MG	CHRIS MACE	MARCH 2001
	0 - 55 GM + .5 MG	0 - 55 GM +/- .1 MG		
	55 - 110 GM + .5 MG	55 -110 GM +/- .1 MG		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
STATE OF CALIFORNIA DIVISION OF MEASUREMENT STANDARDS CERT. NUMBER: 0778.

PROCEDURES USED COMPLY WITH ONE OR MORE OF THE FOLLOWING PROCEDURES: N.I.S.T. HANDBOOK 145, HANDBOOK 44, A.S.T.M. CIRCULAR E 898, FDA CFR TITLE 21 PARTS: 210 TO 211, ISO 10012-1, ANSI/NCSL Z540-1. SNS FORM 1196.

BY: _____
SY NIELSON SERVICE, INC.

FORM: CALRPT

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC. PAGE 5
DEPARTMENT: BACTERIA
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE ST: CA ZIP: 92507
MAKE: METTLER/TOLEDO MODEL: BD1201
SERIAL#: 09514 CUSTOMER#:
CAPACITY: 1200 GM
RESOLUTION: .1 GM
MANUFACTURES TOLERANCE: +/- .1 GM

DATE	INDICATED		CORRECTED		TECHNICIAN	DUE
3/20/00	0 - 1200 GM	- .3 GM	0 - 1200 GM	+/- .1 GM	CHRIS MACE	MARCH
	0 - 600 GM	- .2 GM	0 - 600 GM	+/- .1 GM		2001
	600-1200 GM	- .1 GM	600-1200 GM	+/- .1 GM		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
STATE OF CALIFORNIA DIVISION OF MEASUREMENT STANDARDS CERT. NUMBER: 0778.

PROCEDURES USED COMPLY WITH ONE OR MORE OF THE FOLLOWING PROCEDURES: N.I.S.T. HANDBOOK 145, HANDBOOK 44, A.S.T.M. CIRCULAR E 898, FDA CFR TITLE 21 PARTS: 210 TO 211, ISO 10012-1, ANSI/NCSL Z540-1. SNS FORM 1196.

BY: _____
SY NIELSON SERVICE, INC.

FORM: CALRPT

SY NIELSON SERVICE, INC.
2900 ADAMS ST. SUITE B-4
RIVERSIDE, CA. 92504
909 688-5445

CALIBRATION REPORT

CUSTOMER: E.S. BABCOCK & SONS, INC.
DEPARTMENT: Q.C.
ADDRESS: 6100 QUAIL VALLEY COURT
CITY: RIVERSIDE
MAKE: OHAUS
SERIAL#: 2662
CAPACITY: 120 GM
RESOLUTION: .001 GM
MANUFACTURES TOLERANCE: +/- .001 GM

PAGE 6

ST: CA ZIP: 92507
MODEL: E120
CUSTOMER#:

DATE	INDICATED		CORRECTED		TECHNICIAN	DUE
3/20/00	0 - 120 GM	+.002 GM	0 - 120 GM	+/- .001 GM	CHRIS MACE	MARCH 2001
	0 - 60 GM	+.001 GM	0 - 60 GM	+/- .001 GM		
	60 - 120 GM	+.001 GM	60 -120 GM	+/- .001 GM		

THE INSTRUMENT LISTED ABOVE WAS CALIBRATED AT AN AMBIENT TEMPERATURE AND AN AMBIENT RELATIVE HUMIDITY.

STANDARDS USED ARE TRACEABLE THROUGH:
NATIONAL INSTITUTE OF STANDARDS AND TECHNOLOGY TEST I.D. NUMBER: MMAP 822/258291-97.
LOS ANGELES COUNTY DEPARTMENT OF WEIGHTS AND MEASURES CERT. MS 13733 DATED 10/6/98 DUE 4/30/00.
STATE OF CALIFORNIA DIVISION OF MEASUREMENT STANDARDS CERT. NUMBER: 0778.

PROCEDURES USED COMPLY WITH ONE OR MORE OF THE FOLLOWING PROCEDURES: N.I.S.T. HANDBOOK 145, HANDBOOK 44, A.S.T.M. CIRCULAR E 898, FDA CFR TITLE 21 PARTS: 210 TO 211, ISO 10012-1, ANSI/NCSL Z540-1. SNS FORM 1196.

BY: _____
SY NIELSON SERVICE, INC.

FORM: CALRPT

Enclosure 7

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY
CINCINNATI, OHIO 45268

Office of Ground Water and Drinking Water
Technical Support Center

May 23, 2000

Sean H. Jenkins
E.S. Babcock & Sons, Inc.
P.O. Box 432
Riverside, CA 92502-0432

This letter is to advise you that the laboratory named above has **PASSED** the Spring 2000 Perchlorate PT Study and has been granted **APPROVAL** to monitor for perchlorate as an assessment monitoring parameter under the Unregulated Contaminant Monitoring Rule (UCMR) *[Federal Register, Volume 64, Number 180, September 17, 1999, pages 50556-50620]*. Laboratory approval is contingent upon maintaining certification to perform drinking water compliance monitoring of any inorganic parameter using an approved ion chromatographic method. If a laboratory maintains this certification, the approval to support the assessment monitoring of perchlorate under the UCMR remains active. This letter may be presented to any Public Water System (PWS) as evidence of laboratory approval for perchlorate analysis supporting the UCMR.

The data reported by your laboratory are presented below in Table 1 along with acceptable performance ranges. Only those laboratories which submitted acceptable results for both matrix conductance and perchlorate concentration passed the Spring 2000 Perchlorate PT study. See Table 2 for a summary of the performance of all laboratories.

Since you have passed the Spring 2000 Perchlorate PT study you do not have to participate in the Fall 2000 Perchlorate PT study and a PT sample will not automatically be sent to the laboratory.

Table 1. Spring 2000 Perchlorate PT Study Performance

E.S. Babcock & Sons, Inc.

Parameter	Laboratory Reported Result	Spring 2000 Study True Value (TV)	Acceptance Limits (75% - 125% of TV)
Conductivity	**445** uS/cm	471 uS/cm	353 uS/cm - 589 uS/cm
Perchlorate	**18.4** ug/L	20.3 ug/L	15.2 ug/L - 25.4 ug/L

Internet Address (URL) • http://www.epa.gov
Recycled/Recyclable • Printed with Vegetable Oil Based Inks on Recycled Paper (Minimum 25% Postconsumer)

Enclosure 7

Table 2. Summary of Laboratory Performance for the Spring 2000 Perchlorate PT Study

Participation/Performance	# of labs	Misc. Information	
Reported PT results	73		
Passed for both conductance and perchlorate	59	Representing 81% passing	
Failed for conductivity only(1)	2	1 was <75% of TV 1 was >125% of TV	
Failed for perchlorate concentration only(1)	12	6 were <75% of TV 6 were >125% of TV	
Data Summary	**Median**	**Average**	**%RSD**
Conductivity value (PT study true value = 471 uS/cm)	465 uS/cm	468 uS/cm(2)	5 %
Perchlorate concentration (PT study true value = 20.3 ug/L)	20.1 ug/L	20.2 ug/L(3)	19 %

(1) No laboratory failed both conductivity and perchlorate concentration.
(2) Conductivity average based upon 71 data results; two results of 0.461 uS/cm and 4650 uS/cm were rejected as outliers.
(3) Perchlorate average based upon 72 data results; one result at 214 ug/L was rejected as an outlier.

If you have any questions you can contact Daniel P. Hautman, Perchlorate PT Program Coordinator at 513-569-7274 or e-mail at hautman.dan@epa.gov.

Sincerely,

James J. Westrick, Chief
Technical Support Center

Enclosure 8

Q10

Standard Operating Procedure
Data Review and Validation

1.0 Analyst Review:

Analysts check to see:

1.1 If the results are calculated correctly.

1.2 If the batch QC meet acceptance criteria.

1.3 That dilution or concentration factors were correctly applied and chosen appropriately.

1.4 That the proper units were used.

1.5 That all results for that sample were reported accurately.

1.6 That any additional reporting requirements were met (i.e. QC report).

1.7 Once the analyst feels the data is acceptable, he/she enters the data and prints out a Workgroup Results Report and QC Review Report.

2.0 Peer Review:

2.1 Analyst results are verified by a designated peer analyst prior to release of data to the supervisor.

2.2 This analyst compares the raw data with the Workgroup Results Report and QC Review Report.

2.3 Any major problems are brought to the supervisor's attention immediately.

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The following checklist is used as a guide.

QC Review Report

_____MB < RL
_____LCS % recovery acceptable
_____MS % recovery acceptable
_____MSD RPD acceptable
_____DUP RPD acceptable
_____No BAD-O's
_____No QC missing

Raw Data

_____Date and initials
_____Documentation complete (i.e. acceptance limits, standard log info, etc)
_____Proper dilution choices (over range or over diluted)
_____Spot check calculations
_____Transcription check

2.4 Once the data is verified, analysts change the status of all peer reviewed results from VRFY to Done.
2.5 In the computer click on Sample Data then Data Review.
2.6 Type ANREV. Press Enter
2.7 Imput the workgroup. Type in workgroup number followed by a %. Press Enter.
2.8 Click on Approve Sample and arrow down to the next line. Repeat this process until every line is approved.
2.9 Click on Cancel to close.
2.10 Is something needs to be edited see 3.1.4
2.11 If something need to be redone see 3.1.5
2.12 If part of the samples need an approval status of NO see 3.1.6

3.0 Supervisory Review

3.1 Inorganics:

3.1.1. Pick up review sheets from the printer.

3.1.1.1. For each particular login number, make sure sample pages are in order.

3.1.2. Review pages by examining the following:
3.1.2.1. If notes on the bottom of the page are appropriate
3.1.2.2. That all tests have answers.
3.1.2.3. That the correct units and detection limits are entered.
3.1.2.4. That the customers name and sample ID make sense with the answers.

3.1.2.5. That the answers relate to each other properly. Use the following criteria: For intra-sample quality control the following procedures for checking correctness of analyses are applicable to water samples for which relatively complete analyses are made. These include pH, conductivity, total dissolved solids (TDS), major cationic and anionic constituents, demand parameters, nutrients and trace organic contaminants.

Mineral Balances

Equation (1)
me/L (Ca+Mg+Na+K+NH3-N) = total cations

Equation (2)
me/L (CO3+HCO3+OH+SO4+Cl+NO3+F) = total anions

Equation (3)
Calculated TDS = mg/L (Ca+Mg+Na+K+Cl+SO4+NO3+F+SiO2)+ (0.6)(Total Alkalinity as CaCO3)

The measured TDS may be higher than the calculated TDS because a significant contributor may not be in the calculation.

Equation (4)
Acceptance Criteria: % Difference =

$$100 \times \frac{(\text{total cations} - \text{total anions})}{(\text{total cations} + \text{total anions})/2}$$

% Difference should be μ 5%

Equation (5)
EC(Calc)= (total cations + total anions) x 50

(Both the total anion and total cations should be 1/100 of the measured EC value.) The calculated EC is expected to be within 10% of the measured EC. Thus:

Equation (6)

0.9 μ calculated EC μ 1.1

If the ratio of TDS to conductivity falls below 0.55, the lower ion sum is suspect and reanalyzed. If the ration is above 0.7, the higher ion sum is suspect and reanalyzed. If reanalysis causes no change in the lower ion sums, an unmeasured constituent, such as nitrite or organic acids may

be present at significant levels. If poorly disassociated calcium and sulfate ions are present, the TDS may be higher than the EC. The acceptance criterion is as follows:

Equation (7)

$$\frac{\text{Measured TDS}}{\text{Measured EC}} = 0.55 \text{ to } 0.7 \text{ and/or } \frac{\text{Calculated TDS}}{\text{Calculated EC}} = 0.55 \text{ to } 0.7$$

Reference: Standard Methods for the Examination of Water and Wastewater, APHA, AWWA, WEF, 18th edition

Demand Ratios
A general rule of thumb is:
BOD = 0.4 to 0.6 of COD
TOC = 0.4 (approx) of COD
TOC = 0.6 (approx) of BOD

Nutrient Relationships
Total Nitrogen = Organic Nitrogen + Inorganic Nitrogen
Inorganic Nitrogen = NO3-N + NO2-N + NH3-N
Kjeldahl Nitrogen = Organic Nitrogen + NH3-N
Organic Nitrogen = Kjeldahl Nitrogen - NH3-N
The above nitrogen relationships are checked to ensure proper calculations have been performed.

Trace Organic Contaminants:
TOX = Volatile Organic Halogens + Non-volatile Organic Halogens
Volatile Organic Halogens = Polar + Non-polar Volatile Organic Halogens
Non-volatile Organic Halogens = Polar + Non-polar Non-volatile Organic Halogens
Non-polar Volatile Organic Halogens are measured from EPA Methods 502.2, 601, 8010, 524, 624, 8240, or 8260 analysis.
Non-polar Non-volatile Organic Halogens are measured from EPA Method 525, 625, or 8270 analysis.

From the above relationships, the following is performed:
TOX · Non-polar (Volatile + Non-volatile) Organic Halides

3.1.3. If everything appears to be acceptable, date and initial the bottom of the review sheet.
3.1.3.1. Log on to TERM.
3.1.3.1.1. Pick SAMPLE DATA then approval.
3.1.3.1.2. Type ANREV. Press Enter.
3.1.3.1.3. Imput sample. Type in login number followed by a %. Press Enter.

3.1.3.2. Click on Approve Sample and arrow down to the next line. Repeat this process until every line is approved.
3.1.3.3. Click on Cancel to close.

3.1.4. If something needs to be edited:
3.1.4.1. Pick SAMPLE DATA, then Data, then edit data, then with approval.
3.1.4.2. Query by run ID, sample, or login. Fill in one of these fields then press Enter.
3.1.4.3. Move the curser to the entry list you wish to edit, and press Enter.
3.1.4.4. Type over whatever it is you are editing, then press Enter to save.
3.1.4.5. Click on Close to exit.
3.1.4.6. Change the review sheet to reflect the edit (in ink).

3.1.5. If something needs to be redone:
3.1.5.1. Pick Sample Management.
3.1.5.2. Pick workstat then update status.
3.1.5.3. Click on Status Update Only.
3.1.5.4. Fill in the fields for department number and sample, and enter the new status as "REDO". Press Enter.
3.1.5.5. Mark the test to be redone with an "X". Save by clicking on Save.
3.1.5.6. Click on cancel to exit.
3.1.5.7. Draw an arrow next to this test on the review sheet.
3.1.5.8. Go to seedpak 3 and approve everything except the redo test.

3.1.6. When approving an invoice that has redos, follow the steps in 3.1.3 except do not mark all analytes.
3.1.6.1. Approve only the entry lists that are acceptable.
3.1.6.2. If part of an entry is acceptable, set the curser on that entry list, press Enter.
3.1.6.3. Tab over to "Appl Stat". Change the YES to NO and under Rpt Flg the Y to N on the analytes that are not acceptable.

Note: If you are on the Ion Chromatograph entry list, all milequivalences must be changed to N.

3.1.6.4. If an entire entry list is unacceptable use the mouse to change the Approval Status in the upper right hand corner to NO.
3.1.6.5. Click on Approve Sample.
3.1.6.6. Click on Close to save.

Q10

3.1.7. Turn in the review sheets to the front office.

4. Quality Assurance Review

4.1. Analyte review (LIMS):

4.1.1. Check to see that all the analytes from the review sheet are reported on the final report.

4.1.1.1. Count the number of analytes on each sheet to see if they are equal.

4.1.1.1. Check to see that no analytes are duplicated.

4.1.1.2. Check to see that there is no "Approval Status Of Need or VRFY" note on the final report.

4.1.1. If there are any discrepancies, bring them to the attention of the supervisor.

4.2. Analyte review (Bablab or Word):

4.2.1. Check each individual analyte for possible transcription error.

4.2.2. Check for possible spelling errors on Word reports.

4.3. Check the comments section in the LIMS review report. Have all the conditions been met (i.e. special detection limits or QC requirements)?

4.1. Check the sample header:

4.1.1. Does the LIMS report header have any obvious errors?

4.1.2. Do Bablab or Word reports have any transcription errors? (Check Client ID, Sample ID, Lab Numbers, Sampled and Submitted information.)

4.2. View results to see if any errors are apparent. View balances: EC-TDS, pH-Alkalinity, Cations-Anions, and especially BOD-COD-TOC-O&G-TPH.

Note: This may be the first review including both organic and inorganic tests on the same report.

4.3. Check to see that all required QC is attached.

5. Final Review

5.1. The final reviewer checks the reports for any apparent mistakes prior to signing.

Approved by Susan K Thomas 5/30/00

Cynthia A. Waddell

Enclosure 9

Edward S. Babcock & Sons, Inc.
6100 Quail Valley Ct.
Riverside, CA 92507

Standard Operating Procedure/Method Statement

I have read and understand the Standard Operating Procedure/Method (circle one) and agree to perform testing in accordance with the requirements therein.

SOP/Method (circle one) Title:

The Determination of Inorganic Anions in Water by Ion Chromatography

SOP No. and Revision: I19/3.1

Signed: Cynthia A. Waddell
Employee Name

Date: 06/02/00

Enclosure 10

Demonstration of Capability
Certification Statement

Date: May 12, 2000
Laboratory Name: Edward S. Babcock & Sons, Inc.
Laboratory Address: 6100 Quail Valley Ct., Riverside, CA 92507
Analyst (or Cell) Name: ~~Cindy Waddell~~ IC Cell ~~SOS~~

Matrix: DW
Method Number: EPA 314 / DOHS Rev 0 ~~SOS~~
SOP/Revision No: I19/2.0
Analysis: Perchlorate

We, the undersigned, CERTIFY that:

1. The analysts identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Demonstration of Capability.

2. The test method(s) was perfomed by the analyst(s) identified on this certification.

3. A copy of the test method(s) and laboratory-specific SOPs are available for all personnel on-site.

4. The data associated with the demonstration of capability are true, accurate, complete, and self explanatory(1).

5. All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is well organized and available for review by authorized assessors.

Lawrence J. Chrystal Laboratory Technical Director	Lawrence Chrystal Signature	12 MAY 2000 Date
Sean H. Jenkins Quality Assurance Manager	Sean H. Jenkins Signature	5/12/2000 Date

This certification form must be completed each time a demonstration of capability study is completed.

(1) True: Consistent with supporting data.
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.
Complete: Includes the results of all supporting performance testing
Self-Explanatory: Data properly labeled and stored so that the results are clear and require no additional explanation.

Cindy Waddell
Russell Kikkert
Kevin Sudds
Stacey Fry

Initial Demonstration of Capability

(Enter data in blue spaces only. If needed, click on the Data 2 tab at the bottom to enter more analytes. Press done button when finished)

Analyst: ~~Cindy Waddell~~ IC Cell (Use full name or Cell name)

Method No: EPA314 / DoHS Rev 0

Analysis: Perchlorate

SOP No/Rev No: I19/1.0

Matrix: DW (DW, WW, or SW)

Date of Analysis: 4/4/00

Analytes	Conc.	Units	Read1	Read2	Read3	Read4	Avg	Avg %Rec	%RSD	Acceptance Criteria	P or F
Perchlorate	25	ug/L	27.48	25.73	26.79	26.34	26.59	106.3	2.776	%Rec 90-110, <10%RSD	P
							#####	#####	#####		
							#####	#####	#####		
							#####	#####	#####		
							#####	#####	#####		
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							#####	#####	#####		
							#####	#####	#####		

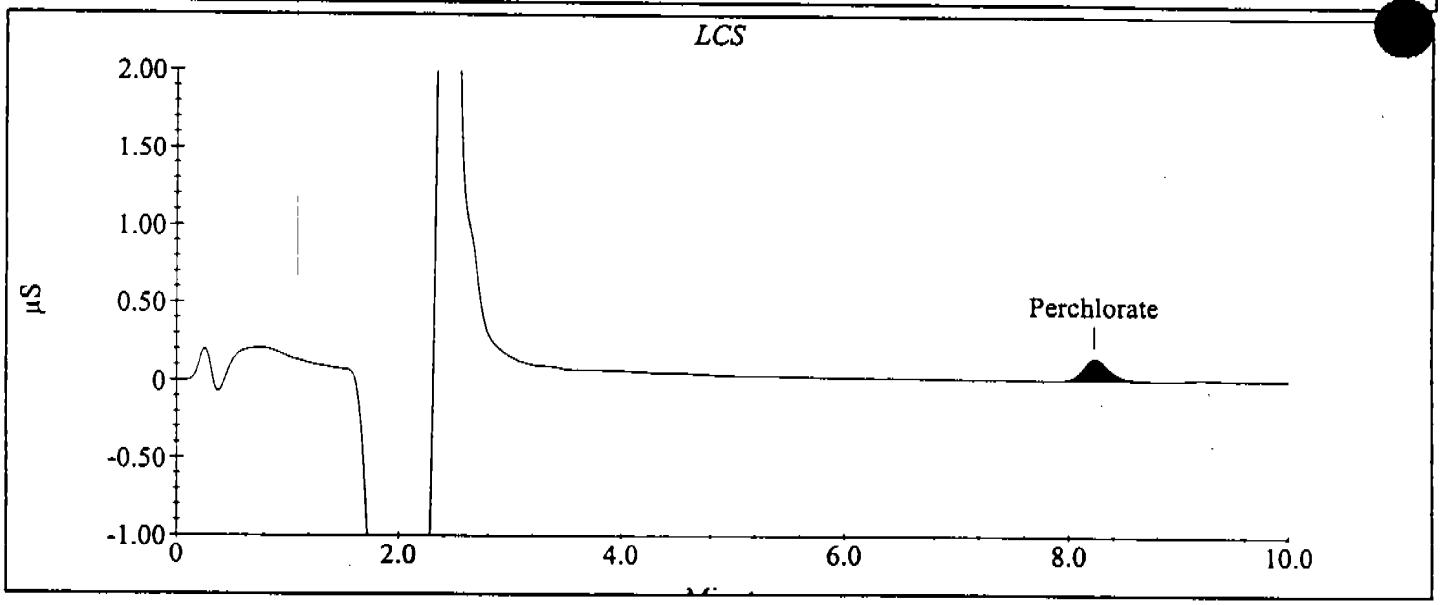
Sample Analysis Report

Sample Name : LCS
Data File Name : C:\PEAKNET\DATA\0403A_034.DXD

Schedule File Name : c:\peaknet\schedule\wg49733.sch
Injection Number : 34
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/3/00 7:25:16 PM
Calibration Date : 4/3/00 12:47:41 PM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
8.22	Perchlorate	27.48	24100	1434



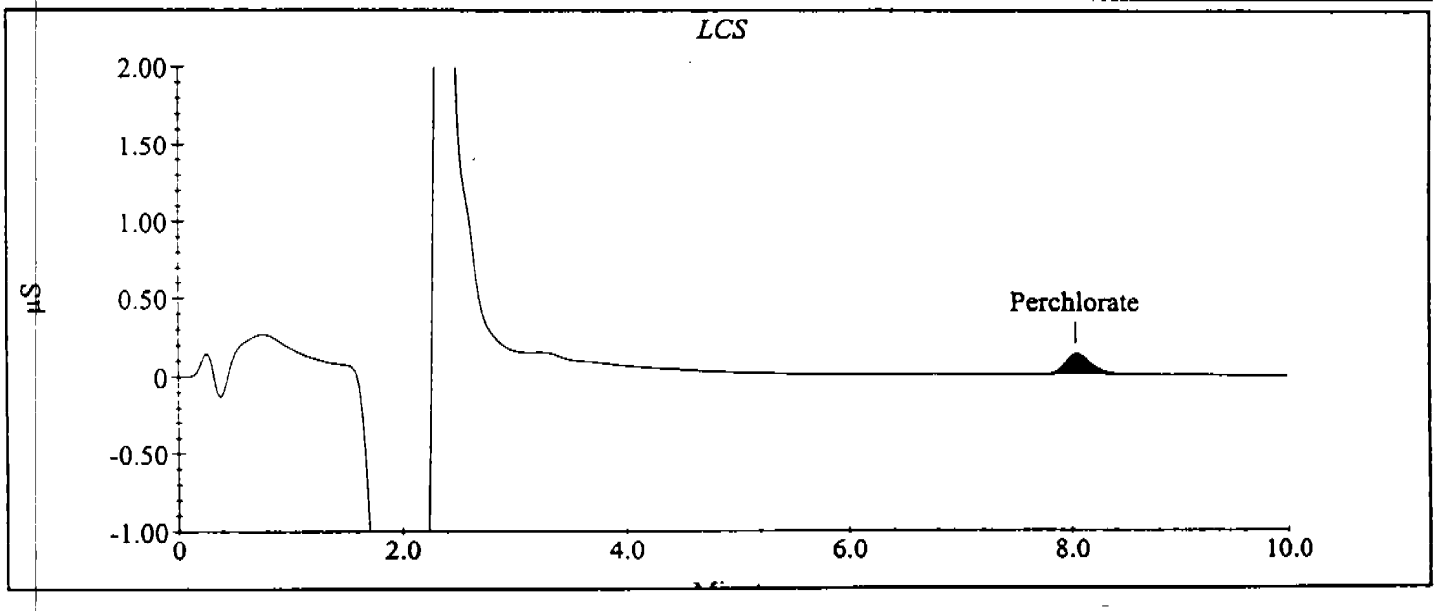
Sample Analysis Report

Sample Name : LCS
Data File Name : C:\PEAKNET\DATA\0403A_062.DXD

Schedule File Name : c:\peaknet\schedule\wg49733.sch
Injection Number : 62
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/4/00 1:12:00 AM
Calibration Date : 4/3/00 12:47:41 PM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
8.03	Perchlorate	25.73	22325	1343



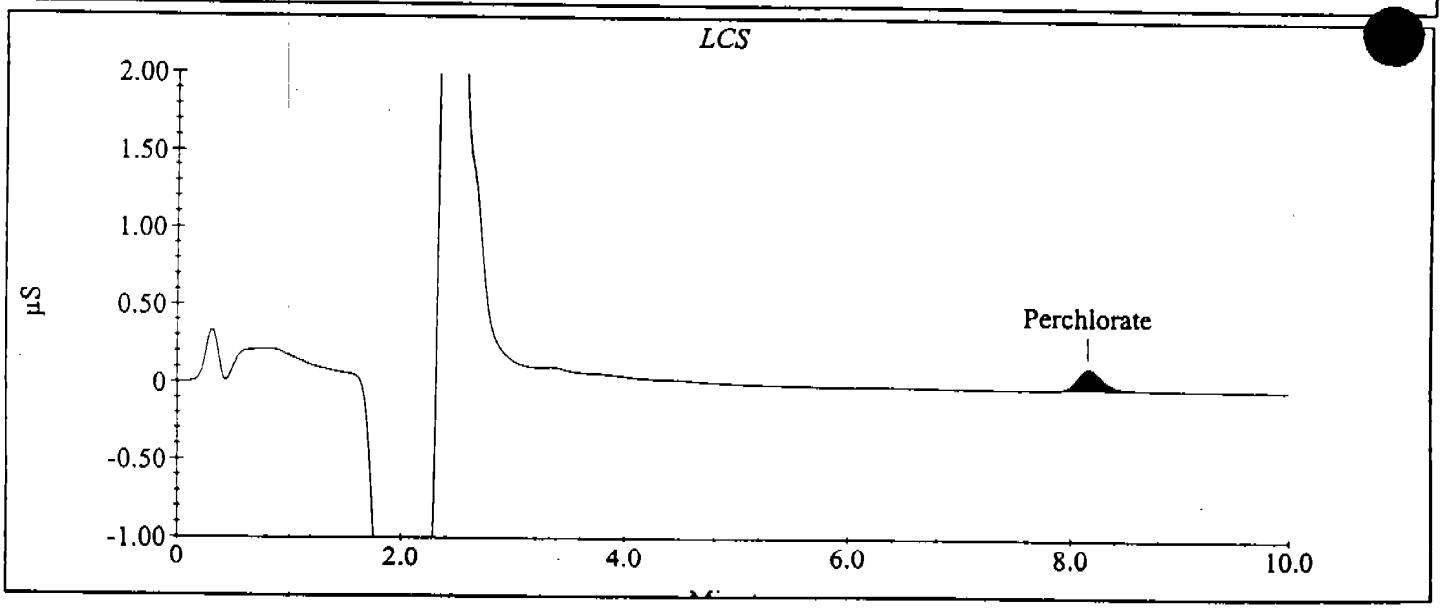
Sample Analysis Report

Sample Name : LCS
Data File Name : C:\PEAKNET\DATA\0403A_067.DXD

Schedule File Name : c:\peaknet\schedule\wg49733.sch
Injection Number : 67
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/4/00 2:13:48 AM
Calibration Date : 4/3/00 12:47:41 PM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
8.15	Perchlorate	26.79	23042	1398



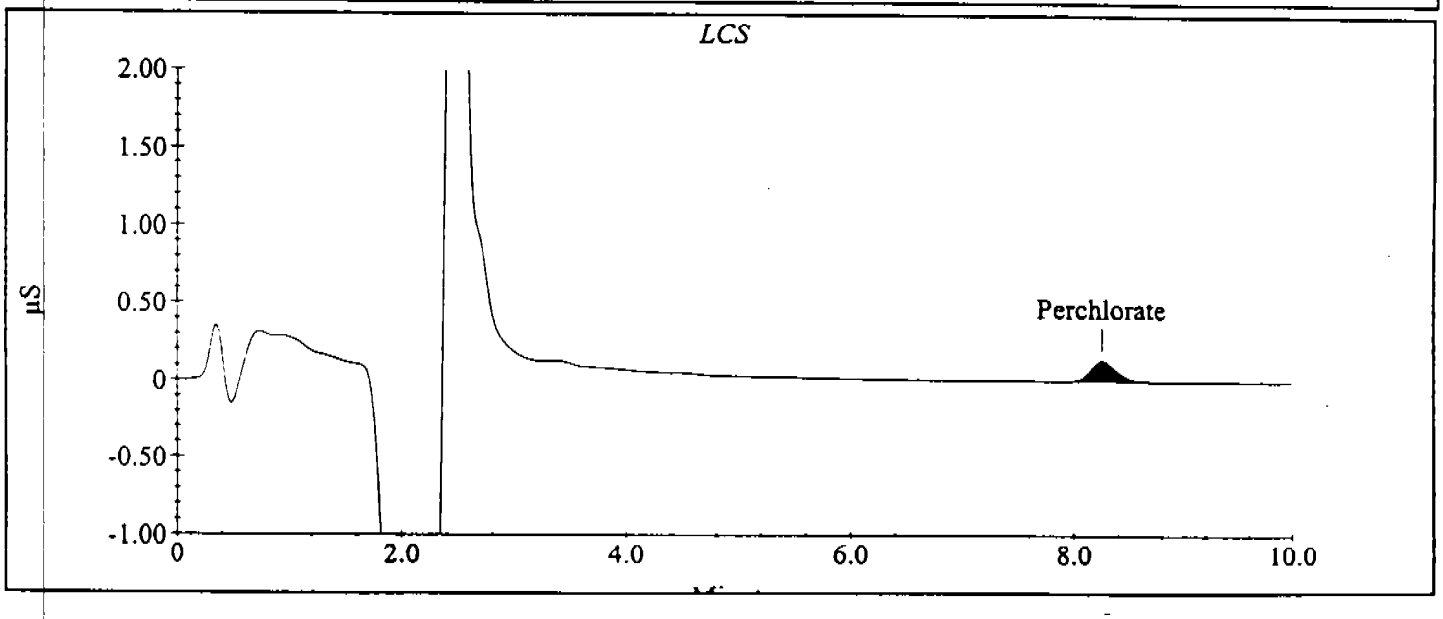
Sample Analysis Report

Sample Name : LCS
Data File Name : C:\PEAKNET\DATA\0403A_008.DXD

Schedule File Name : c:\peaknet\schedule\wg49733.sch
Injection Number : 8
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/3/00 1:06:59 PM
Calibration Date : 4/3/00 12:47:41 PM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
8.25	Perchlorate	26.34	23347	1375

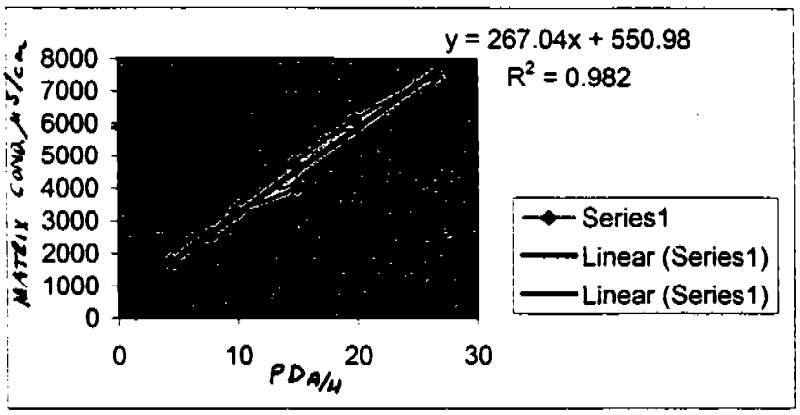


042600
DAW

Perchlorate MCT

	EC	ClO4
LFB w/ 25 µg/L ClO4	1.94	26.41 ppb
200mg Anion Stock w/ 25 µg/L ClO4	1721	~~25.16~~ 24.53 ppb
300 mg/L A.S. w/ 25 µg/L ClO4	2.55 x1000	24.15 ppb
400 mg/L A.S. w/ 25 µg/L ClO4	3.26 x1000	23.83 ppb
500 mg/L A.S. w/ 25 µg/L ClO4	4.04 x1000	23.71 ppb
600 mg/L A.S. w/ 25 µg/L ClO4	4.75 x1000	24.49 ppb
800 mg/L A.S. w/ 25 µg/L ClO4	6.13 x1000	21.86 ppb
1000 mg/L A.S. w/ 25 µg/L ClO4	7.50 x1000	20.30 ppb

Salt Level (mg/L)	Cl04 Conc (ug/L)	EC (umho/cm)	Cl04 Reading (ug/L)	Area	PkHt	A/H	Diff (A/H lfb -A/H ma)	%Diff	
0	25	1.94	26.41	25745	1656	15.5465			
200	25	1721	24.53	24949	1537	16.23227	0.685773	4.4	
300	25	2550	24.15	25339	1514	16.73646	1.189962	7.7	
400	25	3260	23.83	25484	1493	17.06899	1.522491	9.8	
500	25	4040	23.71	26555	1486	17.87012	2.323624	15	
600	25	4750	24.49	27373	1535	17.83257	2.286076	15	XXXX
800	25	6130	21.86	25487	1369	18.61724	3.070741	20	
1000	25	7500	20.3	24967	1270	19.65906	4.112558	26	



Use 5000 µS/cm as MCT. (as conservative value below 5892 µS/cm).

$y = 267.04(20) + 550.98$

$y = 5892\ \mu S/cm$

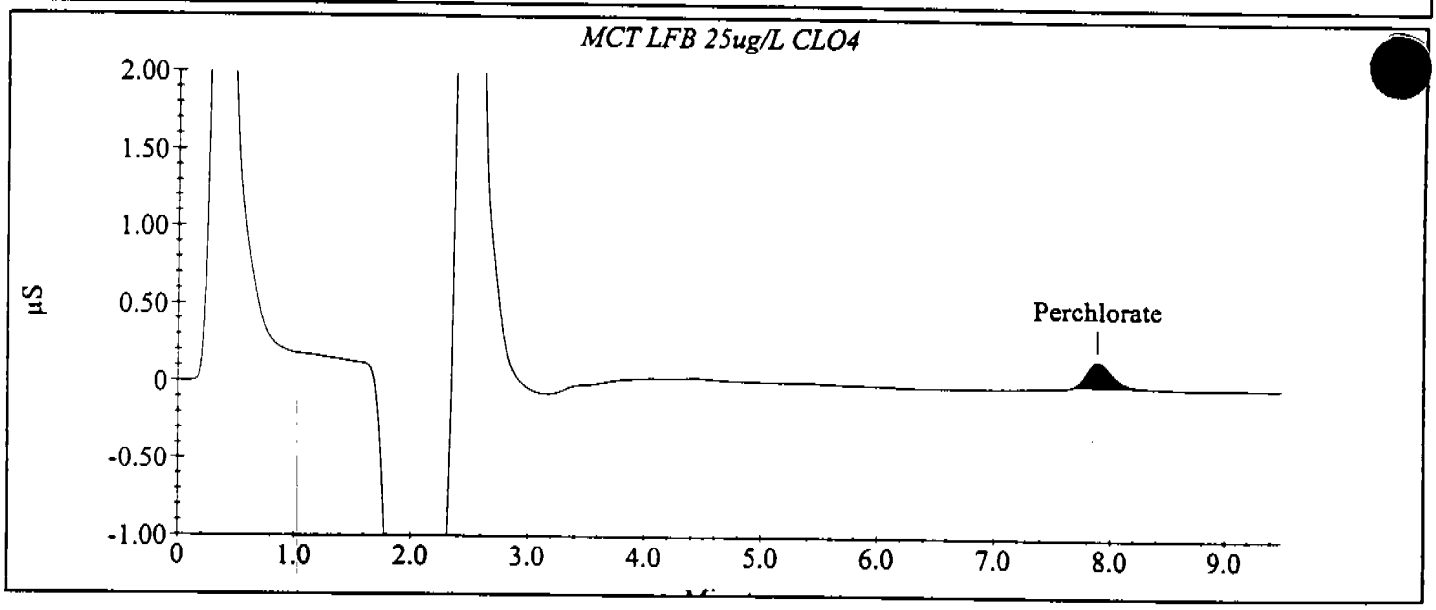
Sample Analysis Report

Sample Name : MCT LFB 25ug/L CLO4
Data File Name : C:\PEAKNET\DATA\0426A_049.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 49
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 8:49:16 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.88	Perchlorate	26.41	25745	1656



Sample Analysis Report

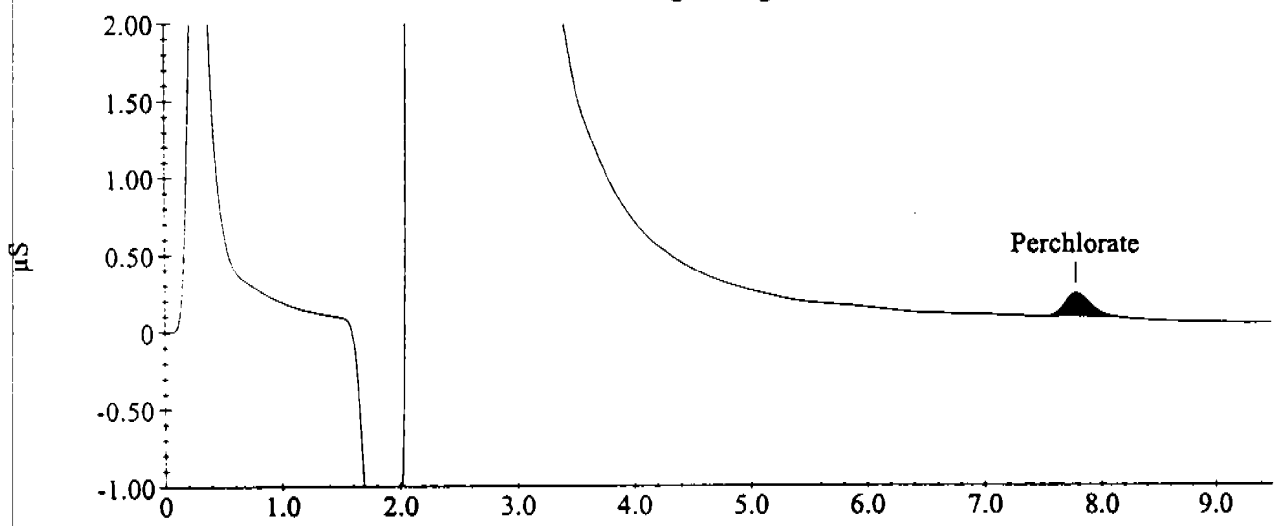
Sample Name : MCT 200mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_050.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 50
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 9:01:09 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.78	Perchlorate	24.53	24949	1537

MCT 200mg/L 25ug/LCLO4



Sample Analysis Report

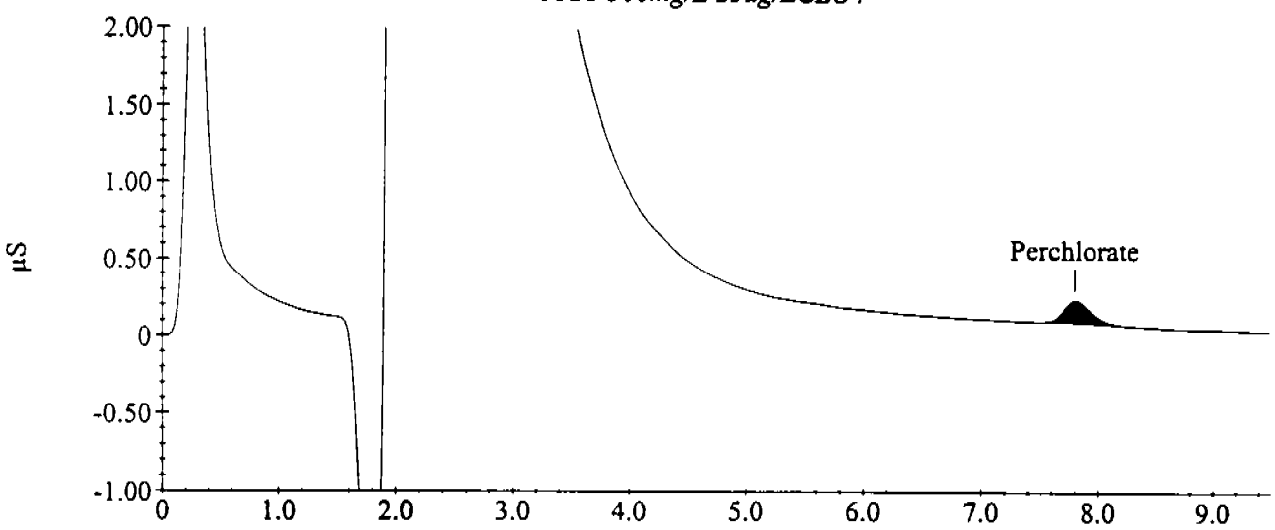
Sample Name : MCT 300mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_051.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 51
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 9:13:04 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.80	Perchlorate	24.15	25339	1514

MCT 300mg/L 25ug/LCLO4



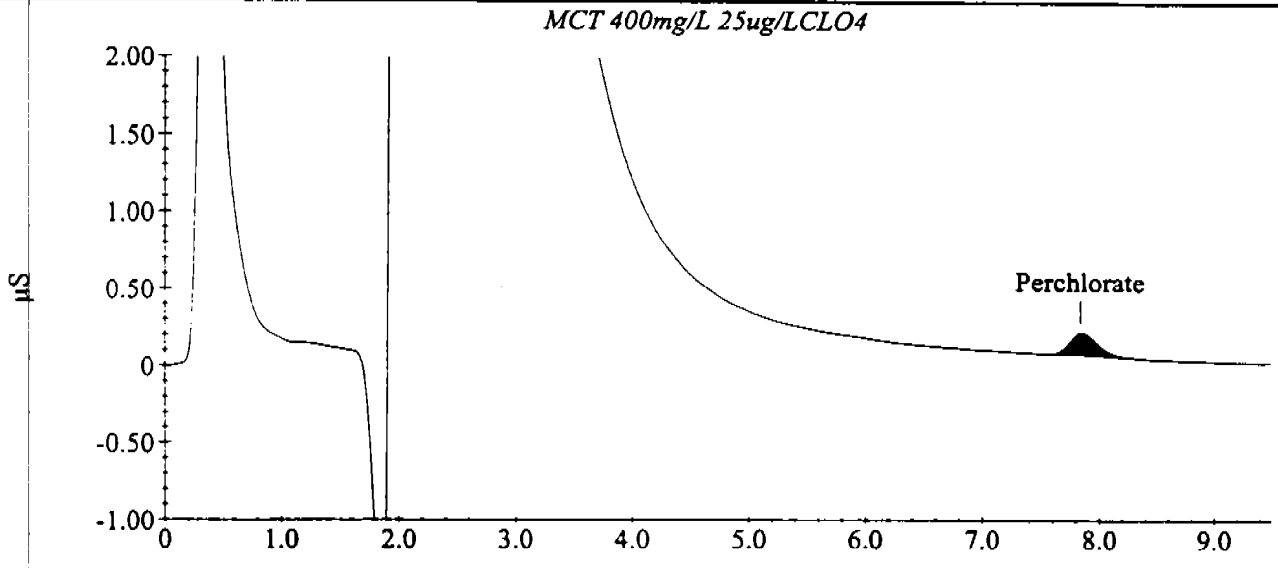
Sample Analysis Report

Sample Name : MCT 400mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_052.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 52
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 9:24:53 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.83	Perchlorate	23.83	25484	1493



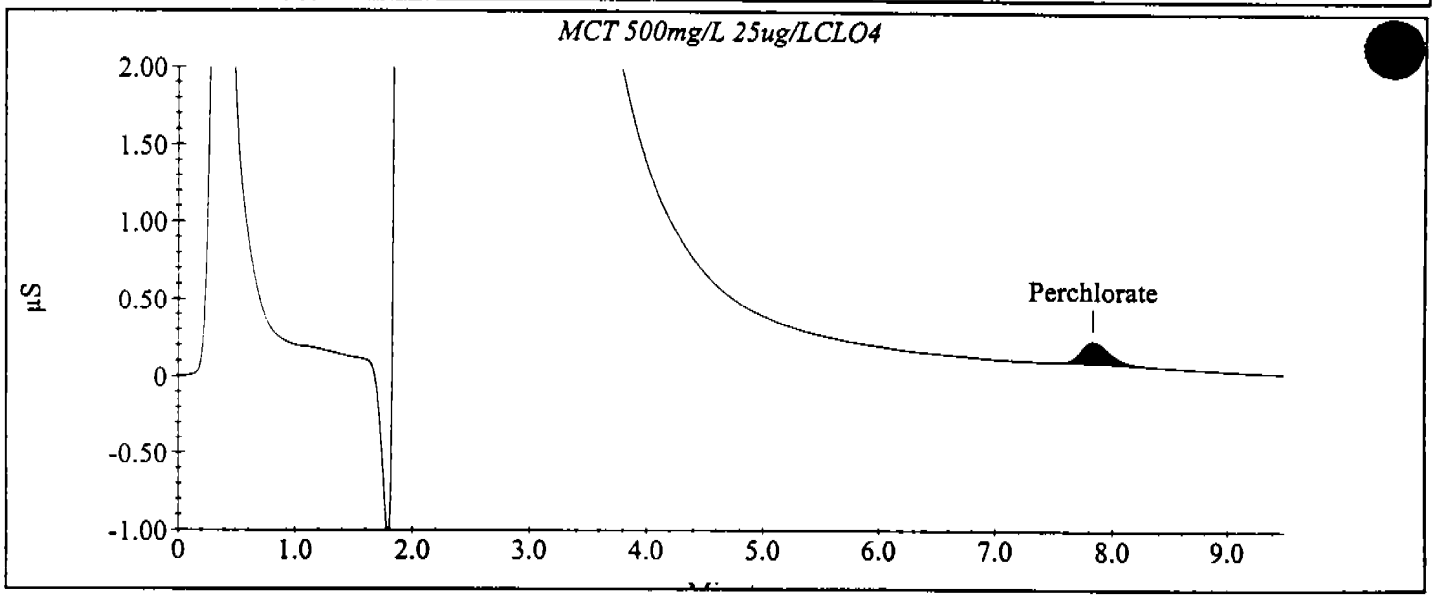
Sample Analysis Report

Sample Name : MCT 500mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_053.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 53
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 9:36:48 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.83	Perchlorate	23.71	26555	1486



Sample Analysis Report

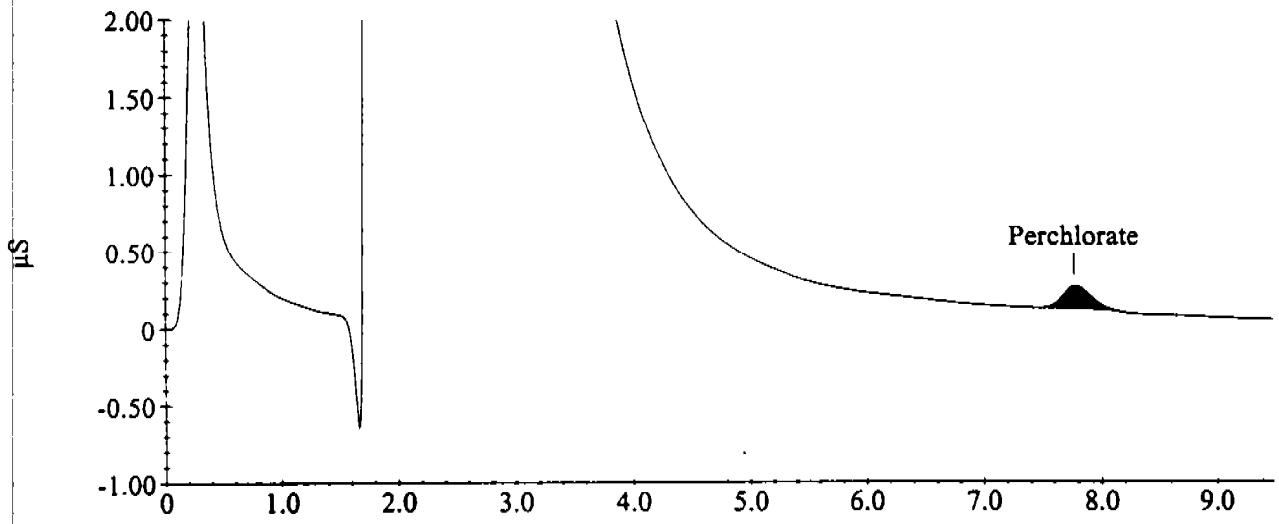
Sample Name : MCT 600mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_054.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 54
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 9:48:44 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.77	Perchlorate	24.49	27373	1535

MCT 600mg/L 25ug/LCLO4



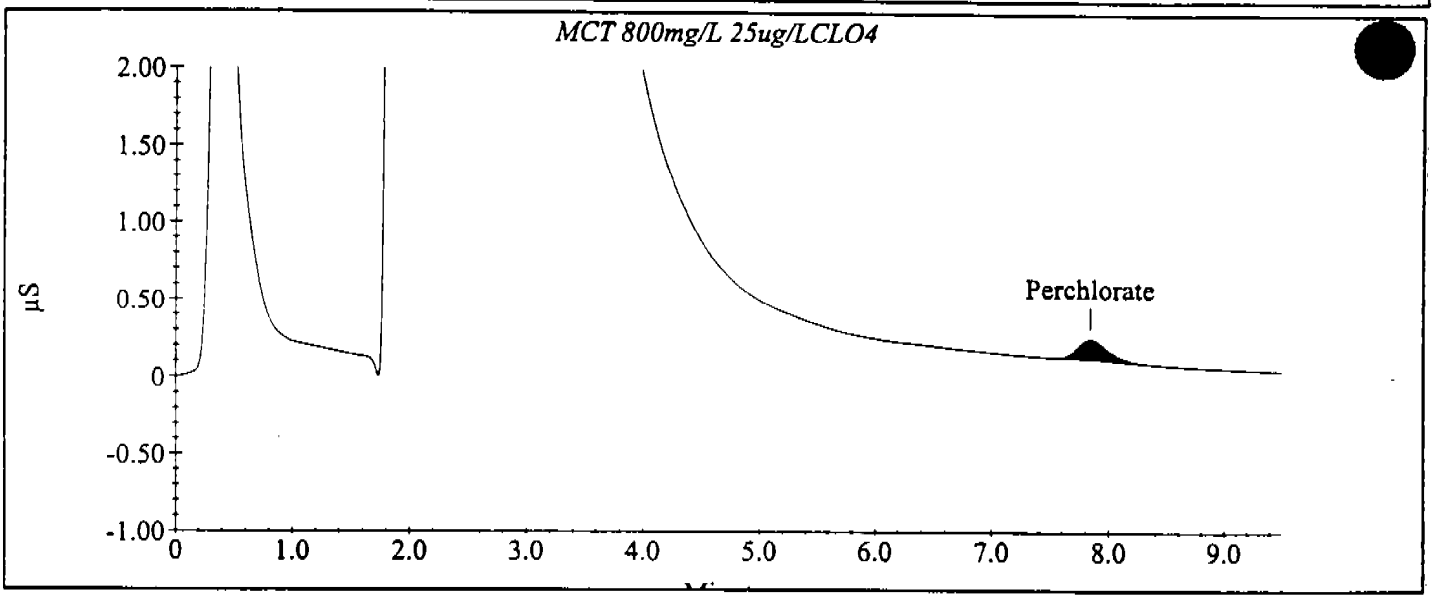
Sample Analysis Report

Sample Name : MCT 800mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_055.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 55
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 10:00:35 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.83	Perchlorate	21.86	25487	1369



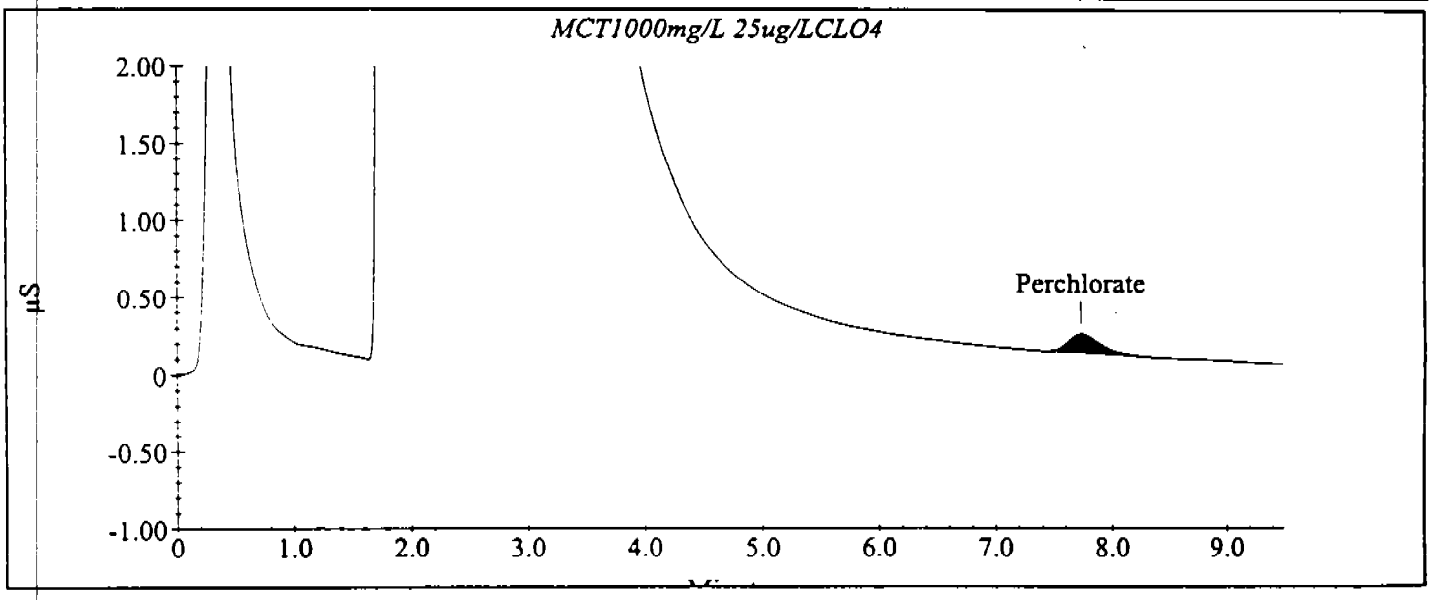
Sample Analysis Report

Sample Name : MCT1000mg/L 25ug/LCLO4
Data File Name : C:\PEAKNET\DATA\0426A_056.DXD

Schedule File Name : c:\peaknet\schedule\wg50433.sch
Injection Number : 56
Method File Name : c:\peaknet\method\a1-clo499.met
System Operator : CWADDELL
Date Time Collected : 4/26/00 10:12:28 PM
Calibration Date : 4/26/00 11:35:46 AM
Dilution Factor : 1.00

Peak Information : All Components

Retention Time	Component Name	Amount (ug/L)	Peak Area	Peak Height
7.73	Perchlorate	20.30	24967	1270



June 15, 2000

E.S. Babcock & Sons, Inc.
6100 Quail Valley Court
Riverside, CA 90503

Attn. Sean Jenkins

Subject: RESPONSE TO 06/06/2000 LETTER

Audit Date: March 16, 2000

Audit Location: E.S. Babcock & Sons, Inc., Riverside, CA

1. The signature pages for the SOP's submitted should include the signature of your Laboratory Technical Director.
2. Item # 4 in your letter was not addressed. Please address this comment.
3. Response to item #19. It appears from your enclosure #10 that Cindy Waddell performed the Demonstration of Capability but this qualified the entire workcell (Russell Kikkert, Kevin Sudds, and Stacey Fry) to perform the test. Each analyst should perform the Demonstration of Capability. Please clarify this issue.
4. Response to item #20. Please make sure each job has a sequence run log included in the package so that we may link QC batches to our samples. Past jobs have not included a sequence run log.

Please respond to the comments above by June 29, 2000.

Very truly yours,

Earth Tech

Angela Wranic

Angela Wranic
Staff Professional VI

cc: Elisabeth Fruth
Chris Davis

Memorandum

To: Chris Davis

From: Angela Wranic

Subject: Status of 06/06/2000 LETTER sent to E.S. Babcock & Sons Inc.

Date: June 28, 2000

Comments: I have left a message with Sean Jenkins the Quality Assurance officer at the laboratory informing her that we need a response to our letter. She is currently on vacation and will return on July 2, 2000.

EARTH TECH AUDIT CHECKLIST

LABORATORY AUDIT CHECKLIST

LABORATORY _____

DATE _____

AUDITORS _____

_____

_____

SAMPLE RECEIVING

Laboratory personnel: _____

1. Are sample coolers checked for proper seal and temperature upon receipt in the laboratory?
   Documented? Where?

   Are temperature blanks included in cooler? If not, is this documented?

   What is the acceptable range of temperature?
   What is corrective action if outside of this range?

   Type of thermometer - digital - mercury - IR - ?
   Calibration documentation?

2. Are sample container labels checked for proper preservatives, and the pH measured (except volatiles) upon receipt?
   Is pH paper dipped in sample container, sample aliquot, or pipette used?
   Is pH check documented for each sample?

   Is COC checked for correct presevative documentation?
   What is done if preservation, HCl, is not noted on the COC for aqueous samples for volatiles analyses?

   Are preservatives added if the pH needs correction?
   Is client notified first?
   Is the purity of reagents verified?

   Where are checks and discrepancies documented?

3. Is the accompanying chain-of-custody checked against the sample labels?

   Are discrepancies documented?

   Are samples kept separate so that individual coolers are checked against the COC pertaining to that cooler?

   Are COCs properly signed off, relinquished and received?

4. Are samples assigned a unique laboratory number, and is this number traceable (correlated) to the field sample number?
   How is this documented?
   Are field shipments or coolers batched together in a sample group (job number/work order number)?

   Are any procedures in place to double check the labeling of sample containers in the lab to preclude mislabeling, i.e., mixing up containers? Are all container aliquots for a sample kept together?

5. Are nonconformance reports written concerning problems with samples at receipt?

   How and when is Earth Tech informed?

6. Are checklists used to make sure that the sample receiving protocol is followed?

   Does the protocol include checking analyses requests for short hold time samples and making specific notice of this?

   How are short hold time samples logged, tagged or otherwise made obvious to the analysts?

7. Is a bound logbook maintained documenting date of sample receipt, source of sample, sample number, parameters to be analyzed, and final disposition?

   Are samples routinely logged into the LIMS system upon receipt?

8. Are internal chain-of-custodies (sample tracking sheets) in use and properly signed when samples are removed for analysis and returned?

   Are samples logged in on the internal COC at receipt?
   If not, where is documentation for storage location?

9. Are samples placed in the designated refrigerator after log-in?
   Are samples for volatiles analysis kept separate from other samples?

10. Are the refrigerators monitored daily and records kept documenting this?

    Does the daily monitoring include Saturday? Sunday? Holidays?

    Who is responsible for the monitoring?

    What are acceptance criteria?

    Are refrigerator thermometers calibrated annually?
    How?

    Are thermometers tagged with calibration due date?

Are correction factors noted and applied?
Is it clear whether the recorded temperature is corrected or not?

What is the corrective action if the refrigerators are outside of acceptance criteria?
How is corrective action documented?

11. Are instructions (SOP) for sample receipt available?
Is SOP approved and current?
Does the SOP include corrective action procedures?

EXTRACTION/SAMPLE PREPARATION - organics

Laboratory personnel: _____

1. Are standard operating procedures (SOPs) available? 3510 (liquid-liquid), 3520 (continuous liquid-liquid), 3550 (sonication), 3540 (Soxhlet), 3580 (Waste dilution)? [indicate SW846 version #]
List other.

   Are EPA method numbers included in the SOP name or is the name only descriptive?

   Are EPA method numbers referenced in the procedure?

   Date(s)?

   Approval Signature?
   Current?

   Are SOPs controlled?

2. Are standards traceable to nationally recognized standards?
Which?

   If not, what standards are they traceable to?

3. Is a standard logbook kept indicating source of standard, lot number, concentration, final volume, name of solution, preparer?
Are procedures for standard preparation available?

4. Are standards kept in segregated refrigerators and are those monitored daily?

5. Are standard bottles labeled with reference to standard log, identification of material or method, concentration, preparation and expiration date, preparers name or initials?

6. How are expiration dates determined?
Is there a general policy for setting expiration dates?

   Do working standards exceed the source standard expiration date?
   Are standards discarded or revalidated after the expiration date?
   If revalidated, how long is the validation good for?

7. Are analytical balances calibrated using "Class S" or "Class 1" weights before and after use (calibration check bracketing sample weight)?
Documented?
What size weights are used?
What is the weighing range?
What tolerance? Is this listed in logbook?
What are the corrective action procedures?

8. Are balances calibrated at least yearly and does documentation support this?

Are weights calibrated at least every three years?
Documented?

9. Are samples batched at sample preparation time and do sample extraction/prep logs list method blanks and blank spikes (LCS, DCS) for each batch ($\leq$20 samples)?

11. How are matrix spikes assigned to the batch?

Do LCS, MS/MSD contain all target analytes? "analytes of interest"?
Short list?

12. Do logbooks indicate addition of surrogates to each blank, sample and QC sample?
Is ID of surrogate standard recorded?

13. Do extraction logs list the extraction **method numbers** and not just names?

14. When is spiking done, *before* or *after* addition of solvent?

15. Is there a spike witness program in place?

16. For method 504.1 (EDB), is the sample centrifuged after extraction to ensure that part of the solvent is not held in an emulsion?

17. When is GPC cleanup used? for each sample?

Is the temperature of the room monitored and recorded?
What are the criteria for the temperature range?

18. Are sonicators tuned, and is there a log to document this?

19. Are drying oven temperatures monitored, and documentation available?

    Thermometers calibrated? Documentation?

WET CHEMISTRY LAB

Laboratory
personnel:_____

1. Are SOPs available in the lab? Do they include cyanide (9012 and CLP), TDS (160.1), anions (300.0 and 340.2 [4500-F/C]), alkalinity (310.1 [2320]), ammonia-N (350.1), TKN (351.2), phosphorous (365.3), sulfide (376.2), oil and grease (1664; 413.1), TOC (415.1, 9060), MBAS (425.1),?
   List other.

   Date?
   Signature?
   Current?
   Are SOPs controlled?

2. Are maintenance logbooks kept for each instrument and are they up-to-date?

   Are entries initialed by analyst?
   Are logbooks reviewed and signed/initialed by supervisor?

   Methods (instruments) reviewed:

3. Is the calibration of balances checked before use?
   Documented?
   Range?
   Tolerance?

   Are balances calibrated at least once a year?
   Are weights calibrated?
   Calibration current?

4. Are individual instrument sample logbook records complete and up-to-date? For instance, does the run log for cyanide indicate CCV every 10th sample including QC samples?

   Other methods:

5. Are calibrations done per method requirements? for instance, six standards for initial calibration for cyanide?

   Other methods:

   Are acceptance criteria for calibration available and records showing acceptable calibration?

6. Is the conductivity meter calibrated daily and calibration documented?

7. Is the pH-meter standardized daily or before use and documented?

8. Is a thermometer used for any analyses?
   Is it calibrated?

9. Are standards preparation SOPs available, and are standards labeled properly including expiration dates?

   What expiration period (length of time) is given for reagents?

10. Are standards and check standards NIST traceable and certificate of analysis available?

11. Are reagents logged with receipt and expiration dates?

12. Are written corrective action procedures available?

13. Is a midi-distillation used in cyanide preparation for AFCEE projects? If so, does the lab have a variance from AFCEE?

TYPE II WATER / REAGENT WATER

1. What kind of system is used for preparing reagent water?

2. What checking is done on this water before use?

3. Is the water used for volatiles calibration and method blanks prepared in a different manner from other water?

   Different source?

4. Does the water meet ASTM Type II reagent grade water specs?

   Spec's per ASTM D1193 (Water 11.01)
   - should be distilled
   - must have a conductivity <1.0 umho/cm at 25 degrees Celsius
   - total matter <0.1 mg/L
   - low organics (<RL) - must pass permanganate test with minimum color retention time 60 min
   - silica = ND
   - Type II water must be protected from atmospheric contamination and contamination from container or tubing

FIELD SAMPLE CONTAINER PREP / FIELD BLANK PREP

Laboratory personnel: _____

1. Is an SOP available for bottle preparation including preservatives required for each method?

2. Are acids used for preservation checked for purity before use?

3. Where in the laboratory are trip blanks prepared?
   Is the environment contaminant free?
   How is this determined?

4. How are batches for trip blanks prepared?
   How are batches for field use (EBs, ABs..) prepared?

   What analyses are performed?
   Documentation?

5. How are lots tracked for trip blanks and water shipments to the field?

METALS LABORATORY (ICP Screen, CVAA, FAA, GFAA, Opb, CLP)

Laboratory personnel: _____

1. Are SOPs available in the lab? Do they include ICP (23 elements), ICP trace, ICP/MS, mercury (CVAA), lead (GFAA and FAA - for organic lead), Pb, Se and As (GFAA)?

   Date?
   Signature?
   Current?

   Are SOPs controlled?

   Are SW846 updated methods used? Which?
   If CLP is used, which version of the CLP SOW?

2. Are QC limits available in the lab or on the LIMS system, and are these used to validate the data?
   Are project specific QC requirements available to the analysts?
   How do they find this information?

3. Are maintenance logbooks kept for each instrument and are they up-to-date?

   Are entries initialed/dated by analyst?
   Does documentation indicate supervisor review?

   Do Logbooks include a routine maintenance schedule or procedure?

   Is return to control documented?

Atomic Absorption

1. Are standards traceable to NIST or other nationally accepted standard?

   Are certificates of analysis on file?

2. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

3. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

Metals, page 2

4. Are standards checked routinely for expiration?
Does expiration date on secondary standards exceed that for the primary standard?
Are expired standards discarded?

5. Are pipettes calibrated?
What frequency?
Is this documented in a logbook?
What are acceptance criteria, and are these listed in the logbook?

Is the logbook organized such that documentation can be clearly understood?

Are pipettes tagged with calibration due date?

Are out-of-criteria pipettes taken out of service and is this recorded?

6. Do records indicate a daily initial calibration of minimum 3 standards (low standard at PQL)and a blank for GFAA and FAA instruments?
Minimum 5 standards (low standard at PQL) and a blank for CVAA?
Acceptance of $r \geq 0.995$? Is the lowest standard at the PQL?

7. Are continuing calibration checks indicated every 10th sample and at the end of the run? (AA)

8. Is method of standards addition (MSA) performed?

ICP

1. Are standards traceable to NIST or other nationally accepted standard?

Are certificates of analysis on file?

2. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

3. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

Metals, page 3

4. Are standards checked routinely for expiration?
   Does expiration date on secondary standards exceed that for the primary standard?
   Are expired standards discarded?

5. Are pipettes calibrated?
   What frequency?
   Is this documented in a logbook?
   What are acceptance criteria, and are these listed in the logbook?

   Is the logbook organized such that documentation can be clearly understood?

   Are pipettes tagged with calibration due date?

   Are out-of-criteria pipettes taken out of service and is this recorded?

6. Calibration criteria for ICP? Blank + 1 standard? 3 standards?

7. For ICP is a calibration blank(ICB) and calibration check (ICV) run after the initial calibration?

8. For ICP, is a continuing calibration check (CCV and CCB) run at 10% frequency?

   What is corrective action for failure?

9. Is an ICP interference check run at the beginning of each daily run?
Is acceptance indicated within 80-120%?

What is corrective action?

10. Are ICP interelement correction factors updated annually, and verified quarterly?

What is corrective action if the verification fails?

METALS PREPARATION; personnel:

1. Are SOPs available for 3005?, 3015?, 3050? [list SW846 version #]
   List other.

   Do SOPs specify temperature for digestion?
   Is thermometer calibrated? How?

2. Is a method blank and LCS included in each digestion batch?

3. Are standards traceable to NIST or other nationally accepted standard?

   Are certificates of analysis on file?

4. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are standards checked routinely for expiration?
   Does expiration date on secondary standards exceed that for the primary standard?
   Are expired standards discarded?

7. Do prep logs indicate a method blank, MS/MSD and LCS run for every batch of maximum 20 samples?

8. Are pipettes calibrated?
   What frequency?
   Is this documented in a logbook?
   What are acceptance criteria, and are these listed in the logbook?

   Is the logbook organized such that documentation can be clearly understood?

   Are pipettes tagged with calibration due date?

Are out-of-criteria pipettes taken out of service and is this recorded?

Metals prep, page 2

9. Are samples homogenized before weighing?

10. Is balance calibration current? Date due?

Are criteria for the daily check listed in the logbook?

GAS CHROMATOGRAPHY, volatiles (8021, 504 (EDB))

Laboratory personnel:

_____

1. Are SOPs available in the lab? List.

   Updated methods used? [indicate version #]
   Date?
   Signature?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to analyst?

3. Are bound maintenance logbooks kept for each instrument? Paginated?

   Are they up-to-date?

   Are entries dated and initialed by analyst?

   Is supervisory review documented?

   Is return to control documented?

4. Are standards logbooks complete and up-to-date?

   Do they contain instructions for preparation of standards?
   Do the standards include the complete analyte list?
   Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. What is the policy for determining expiration dates?
   Do secondary standards expiration date exceed the source standard's?

   Are expired standards discarded?
   Is there a policy for routinely checking standards to remove expired standards?

7. Do analysis logbooks indicate initial calibration using at least 5 concentration levels of

standards for 8000 series methods?
Are standards levels identified? Is the 1

Are acceptance criteria *mean* % RSD ≤20% or r ≥.995? Is it specified in SOP?
How and when are retention time windows determined?

8. Are continuing calibration check standards run daily before sample analysis?

- Do records indicate that the CCS is from a different source than the initial calibration standards?

Are acceptance criteria *mean* %D ±15%?

Are CCVs run at 10% and end of sequence?
What is corrective action for failure?

Are calibrations accepted without meeting criteria?
If so, when?

9. Are analysis logbooks up-to-date?

Is the pH of samples verified and documented before analysis?

10. Do run logs document a method blank, MS/MSD and LCS for every 20 samples? (method blanks daily if <20 samples per day)

Do LCS and MS/MSD contain all analytes of interest?

11. Are surrogates added to each sample, blank and standard (8021)?
Are surrogate IDs entered in the logbook?

Are internal standards used? IDs entered in logbook?

12. Are second column confirmations being done on all analytes present in concentrations greater than the detection limit?

Are second column confirmations done using a different column or GC/MS?

Done condurrently with first column analyses?

Are second column confirmations done within holding times?

Are second column confirmations reported quantitatively?

Is the same level of QC applied to second column confirmations? Same acceptance criteria?

GC, volatiles, page 3

13. Are MDL studies performed on both columns?

Are the columns identified on the MDL studies tables?

14. Are trip blanks run as part of the same batch as the samples for water?
Where in the sample run are they scheduled?
Do analysts know that these are trip blanks?

Are hits in trip blanks confirmed on second column?
What if carryover is suspected?

15. Are method blanks being monitored with respect to contamination?
Is corrective action being performed if the level of contamination is > detection limit?
New method blank?
Rerun of samples?

16. Are volatiles samples stored in the volatiles section refrigerators?

Are there holding blanks in the volatiles refrigerators?

How often are these changed and analyzed?

17. Is there a tracking mechanism in place so that confirmations and diluted runs of a sample can be easily tracked in the lab?

18. Are dilutions decided based on pre-screening of samples?

Are dilutions expressed as a dilution factor, e.g., 5x, or as a ratio, e.g., 1:5?

19. Is the amount of headspace monitored in vials containing surrogates and internal standards?

Is there a policy regarding using standards with excessive headspace? What is regarded as excessive headspace?

20. Are written corrective action procedures available?

GAS CHROMATOGRAPHY, extractable (8081, 8082, 8151, 8141, CLP)

Laboratory personnel:

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1. Are SOPs available in the lab?
   Updated methods used? [indicate version #]
   Is CLP method used? Which version?
   SOP Date(s)?
   Approval Signature?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analysts?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?

   Are entries dated and initialed by analyst?

   Is supervisory review documented?

   Do logbooks document return to control?

4. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

   Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are standards checked routinely for expiration?

   Does the expiration date on secondary standards exceed that for the primary standard?

   Are expired standards discarded?

7. Do calibration logbooks indicate initial calibration using at least 5 concentration levels of standards for 8000 series methods?
Are standards levels identified?
Is the lowest standard at the PQL?

Do the standards contain the complete analyte list?
Are acceptance criteria *mean* % RSD ≤20% or r ≥.995? Is this specified in the SOP?

How are retention time windows determined? How often?

Is a second source verification standard run?
Acceptance criteria ±15%?

8. Are continuing calibration check standards run daily before sample analysis?
Do records indicate that the CCS is from a different source than the initial calibration standards? [not required if a second source verification standard is run]

Does this standard contain the complete analyte list?
Are acceptance criteria *mean* %D ±15%?

9. Are analysis logbooks up-to-date?

Do they indicate a CCV at 10% including samples and QC samples, and end of run?

10. Do run logs document a method blank, MS/MSD and LCS for every 20 samples? (method blanks daily if <20 samples per day)

Do LCS and MS/MSDs contain all analytes of interest?

11. Are surrogates added to each sample, blank and standard (8000 series)? Are surrogate IDs entered in the logbook?

12. Are second column confirmations being done on all analytes present in concentrations greater than the detection limit?

What type of columns are used?

13. Are second column confirmations done using a different column or GC/MS? Run simultaneously?

GC, extractable, page 3

14. If not run simultaneously, are second column confirmations done within holding times?

15. Are second column confirmations reported quantitatively?

    Are the same QC criteria applied to both first and second column?

16. Are MDL studies performed on both columns?

    Are the columns identified on the MDL studies tables?

17. Is dilution expressed as a dilution factor, e.g., 5x or as a ratio, e.g., 1:5

18. Are written corrective action procedures available?

LUFT, volatiles

Laboratory personnel: _____

1. Are SOPs available in the lab?

   Dated?
   Signed?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analyst?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?
   Are entries dated and initialed by the analyst?
   Is supervisory review documented?
   Is return to control documented?

4. Are standards logbooks complete and up-to-date?
   Do they contain instructions for preparation of standards?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are expired standards discarded?
   Is there a policy for checking for expired standards?

7. Do analysis logbooks indicate initial calibration using at least 3 concentration levels? 5?
   Are standards levels identified? Lowest standard at PQL?
   Are acceptance criteria RSD $\leq$20% or r $\geq$.995?
   What is specified in the SOP?

8. Are continuing calibration check standards run daily before sample analysis?
   Are acceptance criteria D $\pm$15%?
   Are CCVs run after every 10 samples including QC samples, and end of run?
   What is corrective action for failure?

9. Are analysis logbooks up-to-date?

   Is the pH of samples verified and documented before analysis?

10. Do run logs document a method blank, MS/MSD and LCS for every 20 samples? (method blanks daily if <20 samples per day)

11. If client specific calibration standards are used, how are these handled as far as initial calibration, daily checks, comparison to sample chromatograms, etc.?

12. Are surrogates used for this method?
    Which?
    Are surrogate IDs listed in the run log?

13. Are internal standards used for this method?
    Which?
    Are I.S. IDs listed in the run log?

14. Is dilution expressed as a dilution factor, e.g., 5x, or as a ratio, e.g., 1:5?

LUFT, extractables

Laboratory personnel: _____

1. Are SOPs available in the lab?

   Dated?
   Signed?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analyst?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?
   Are entries dated and initialed by the analyst?
   Is supervisory review documented?
   Is return to control documented?

4. Are standards logbooks complete and up-to-date?
   Do they contain instructions for preparation of standards?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are expired standards discarded?
   Is there a policy for checking for expired standards?

7. Do analysis logbooks indicate initial calibration using at least 3 concentration levels of both diesel and jet fuel? Other standards?

   Are standards levels identified? Lowest standard at PQL?

   Are acceptance criteria RSD $\leq$20% or r $\geq$.995?
   What is specified in the SOP?

8. Are continuing calibration check standards run daily before sample analysis?
   Do these include all components: jet fuel and diesel plus any other requested special fuel?
   Are acceptance criteria D ±15%?

   Are CCVs run at the frequency of 10% counting samples and QC samples?
   Are CCVs run for both diesel and jet fuel?

   What is corrective action for failure?

9. Are analysis logbooks up-to-date?

10. Do extraction and run logs document a method blank, MS/MSD and LCS for every 20 samples? (method blanks daily if <20 samples per day)

11. If client specific calibration standards are used, how are these handled as far as initial calibration, daily checks, comparison to sample chromatograms, etc.?

12. Are surrogates used for this method?
    Which?

13. Are I.S.s used for this method?

14. Are dilutions expressed as dilution factors or a ratio?

GAS CHROMATOGRAPHY/MASS SPECTROMETRY, volatiles (8260, 524.2, CLP)

Laboratory personnel: _____

1. Are SOPs available in the lab?

   Date?
   Signature?
   Current?
   Controlled?
   Are SW846 Updated methods used? [indicate version]
   Is CLP method used? What version?
   Is method 5035 (soils volatiles) available?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analyst?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?
   Are entries dated and initialed by the analyst?
   Is supervisory review documented?
   Is return to control documented?

4. Are standards logbooks complete and up-to-date?
   Do they contain instructions for preparation of standards?
   Do standards contain the complete analyte list?
   Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. What criteria are used to set standards expiration dates?
   Do expiration dates for working standards exceed those of the source standard?
   Are expired standards discarded?
   Is there a policy for checking for expired standards?

   Is the amount of headspace monitored in surrogate and I.S. vials?

   Is there a policy regarding using standards with extreme headspace? What is regarded as extreme headspace?

7. Do logbooks indicate initial calibration using at least 5

concentration levels of standards? Are standards concentrations identified?
Is the lowest standard at or below the reporting limit (PQL)?
Are acceptance criteria consistent with the version of the method?

GC/MS volatiles, page 2

8. Are instruments tuned every 12 hours (8 or 12 hours for 524.2) using BFB?
Are method specific criteria used for acceptance?

9. Are continuing calibration check standards run every 12 hours before sample analysis (8 or 12 hours for 524.2)?

10. Are the data evaluated against acceptance criteria before continuing sample analysis?
If updated methods are used are correct criteria used?
Are calibrations accepted even if not all criteria are met?
In which situation would this be done?

11. Are internal standards monitored and evaluated against acceptance criteria? Are internal standard IDs identified?
What are the acceptance criteria?

12. Are analysis logbooks up-to-date?
Do run logs identify analysis methods (e.g., 8260B, 524.2)?
Is pH of the samples verified and documented before analysis?

13. Do logbooks document a method blank and MS/MSD for every 20 samples?
(No MS/MSD required for 524.2)

Are all target analytes spiked into the MS/MSD and LCS?
"Analytes of interest"? Short list?

Is an LCS run with every batch?
How is an LCS defined? Blank spike? Laboratory Fortified Blank?

How is the batch defined?
Does a batch cover more than one tuning period?

14. Do logbooks document addition of surrogates to each sample,

blank and standard?

Are surrogate IDs identified?

15. Are samples rerun if a surrogate is out?
How is matrix interference determined?

GC/MS volatiles, page 3

16. Are volatiles samples stored in the volatiles sections?
Are holding blanks in the volatiles refrigerator?
How often are these changed and analyzed?

17. Have problems with common laboratory contaminants been found?

18. Are written corrective action procedures available?

19. Is dilution expressed as a dilution factor or as a ratio?

GAS CHROMATOGRAPHY/MASS SPECTROMETRY, semivolatiles (8270, CLP)

Laboratory personnel:

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1. Are SOPs available in the lab?

   Date?
   Signature?
   Current?
   Controlled?
   Are updated SW846 methods used? [indicate version]
   Is CLP method used? Which version?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analyst?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?
   Are entries dated and initialed by the analyst?
   Is supervisory review documented?
   Is return to control documented?

4. Do logbooks indicate initial calibration using at least 5 concentration levels of standards? Are standards levels identified in the run log?
   Is the lowest standard at the PQL?
   Are all analytes included in the calibration?

   Is analysis method, i.e., 8270, 8270B, 8270C, identified in the logbooks?

   Are acceptance criteria consistent with the version of the method?

5. Are instruments tuned every 12 hours using DFTPP?
   Are method specific criteria used?

6. Are continuing calibration check standards run every 12 hours before sample analysis?
   Complete analyte list used?
   Updated criteria used if updated method indicated?

7. Are the data evaluated against acceptance criteria before continuing sample analysis?

8. Do logbooks indicate addition of internal standards to each blank, sample and QC sample?

Is ID of internal standard documented?
Are internal standards monitored and evaluated against acceptance criteria?

9. What criteria are used to set standard expiration daates?

   Do expiration dates for working standards exceed those of the source standard?

   Are expired standards discarded?

   Is there a policy for checking expired standards?

10. Are analysis logbooks up-to-date?

11. Are samples rerun if
    a. 1 surrogate is out
    b. 2 surrogates are out
    c. 2 surrogates from the same group (acid/BNA) are out?

    Are samples re-extracted/rerun for these reasons?

12. Are dilutions expressed as dilution factors, e.g., 5x, or as a ratio, e.g., 1:5?

13. Are written corrective action procedures available?

SUBSAMPLING OF SOILS

1. Are soil samples for volatiles analyses taken in such a manner that they have been least exposed to air, i.e, a portion of the sample discarded before subsampling?

2. Is a separate liner or jar used for volatiles?
When only one soil sample liner is supplied is a subsample taken for VOA before any other samples are taken out of the liner?

3. Are soil samples for volatiles weighed in such a manner that they are least exposed to air?

4. Are soils for volatiles added to the sparge tube/vial before or after the water is added?

5. Are soil samples for semivolatiles and inorganics homogenized so that a representative sample is obtained?

DATA REVIEW/DATA MANAGEMENT

1. Is a level 1 review (analyst performing analysis) performed on 100% of the data?

   Is this documented?

   Is a level 2 review (supervisor or equivalent) performed on every data package?

2. Does this include checking calibration and QC data against criteria?

   Is this review documented?

3. Does the project manager review the data package before it is sent to the client?

   Is this review documented?

4. Does QA review a percentage of the data packages?

   Is this documented?

5. How is the electronic deliverable reviewed/checked?

6. Does the hard copy correlate with the electronic deliverable?
   How is this checked?

7. How are changes to the report handled/ documented?

CONTROL CHARTS/LIMITS

1. Are control charts showing warning and control limits available in the lab?

2. Are these being used by the analysts to monitor trends as well as check acceptance criteria?

3. How often are QC limits updated?

4. What criteria are used for including results in the calculations?

AUDITS

1. Are internal audits performed periodically?
   How often?
   Who performs them?

2. Do these audits include blind samples as well as a laboratory walk-through audit?

3. Are data audits performed?
   How often?
   Who performs them?

4 Are these audits documented?

5. Is there a system for verifying corrective action of nonconformances or quality deficiencies noted during these audits?

NONCONFORMANCE/CORRECTIVE ACTION

1. Are nonconformance (anomaly) reports generated in the various laboratories kept on record by the QA Officer?

   Is there a tracking log?

2. Are these nonconformances signed off properly and do they indicate return of control.

3. Are data generated during an out-of-control situation evaluated before being used?

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STANDARD OPERATING PROCEDURES

1 Is there a SOP log? Are approval dates listed?

Is the log up-to-date?

2. Does information on the log correspond to actual approved SOPs?

3. Does the lab have a current, approved SOP for all methods performed?

4. Are correct method references used in the SOPs?

5. Are requirements listed in the SOPs consistent with EPA requirements?

6. Are SOPs reviewed on a specific schedule, and is this being done?

7. How is it documented?

DOCUMENT CONTROL

1. Are SOPs issued as controlled documents?

3. Are individual SOPs issued or is the entire set issued as a whole?

3. How is distribution controlled?

4. Are revisions issued to all holders of the original document?

PERFORMANCE EVALUATION SAMPLES

1. What PE programs does the lab subscribe to?

2. Are the results communicated to the staff responsible for the analyses?

3. Are responses to deficiencies prepared?

4. Are any corrective actions performed as a consequence of unacceptable PE sample results?

   How are these documented?

METHOD DETECTION LIMIT STUDIES

1. Are MDL studies performed on every instrument? Each column?

   If not, how can you be sure that your MDL is applicable to all instruments?

2. Are metals and inorganics MDLs done annually? IDLs quarterly? Verify.

3. Are organics MDLs done annually? List per method.

4. What protocols are used for the MDL studies?

5. How are reporting limits(RL)/practical quantitation limits (PQL) related to MDLs?

6. Are RL/PQL checked by analyzing standards at or below the set level?

   How often is this done?

CALIBRATION RECORDS FOR BALANCES, WEIGHTS AND THERMOMETERS

1. Thermometers:

   Do records indicate annual calibration?

   Do records indicate that thermometers are calibrated at the appropriate range?

   Do records indicate use range for all thermometers?

   Are there calibration records for the NIST traceable thermometers?

   Do these thermometers cover the various ranges used in the laboratory?

2. Balances

   Are certificates for balance calibration available?

   Do these indicate annual outside calibration?

   Is there a log to track the location and ID of each balance?

3. Weights:

   Is there a log to track the ID and location of each weight set?

   How often are weight sets calibrated?

   Is done by an outside agency?

   To what tolerances are the weights calibrated? Class S? Class 1? Class 2?

PERSONNEL TRAINING

1. Are personnel training records available for all personnel? Who is responsible for these records?

2. Are resumes available for all personnel?

3. Is there a SOP for personnel training?

4. Are criteria specified in the SOP?

5. What training are sample receiving personnel given?

6. Who is responsible for training of new hires and continuing evaluation of analysts?

7. Who determines capability to perform an analysis?
   How is this determined?
   Documented for each method?

8. Does training include training on QA/QC procedures?

9. Are analysts informed of project-specific QA/QC procedures before the start of a project?

   How is this communicated to the analyst?

10. How are temporary personnel trained? capabilities evaluated? record? are they informed of project specific requirements?

HIGH PERFORMANCE LIQUID CHROMATOGRAPHY, extractable (8310, 8321, 8330, 632)

Laboratory personnel: _____

1. Are SOPs available in the lab?

   Updated methods used?
   Date?
   Signature?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analysts?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?

   Are entries dated and initialed by analyst?

   Is supervisory review documented?

   Do logbooks document return to control?

4. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

   Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are standards checked routinely for expiration?

   Does the expiration date on secondary standards exceed that for the primary standard?

   Are expired standards discarded?

7. Do calibration logbooks indicate initial calibration using at least 5 concentration levels of standards for 8000 series methods?
Are standards levels identified?
Is the lowest standard at the PQL?

Do the standards contain the complete analyte list?
Are acceptance criteria RSD ≤20% or r ≥.995? Is this specified in the SOP?

How are retention time windows determined? How often?

8. Are continuing calibration check standards run daily before sample analysis?
Do records indicate that the CCS is from a different source than the initial calibration standards?

Does this standard contain the complete analyte list?
Are acceptance criteria D ±15%?

9. Do runlogs indicate a CCV at 10% including samples and QC samples, and end of run?

10. Do run logs document a method blank, MS/MSD and LCS for every 20 samples? (method blanks daily if <20 samples per day)

Do LCS and MS/MSDs contain all analytes of interest?

11. Are surrogates added to each sample, blank and standard (8000 series)? Are surrogate IDs entered in the logbook?

12. Are second column confirmations being done on all analytes present in concentrations greater than the detection limit?

What type of columns are used?

13. Are confirmations done using a different column, second detector or GC/MS? Run simultaneously?

14. If not run simultaneously, are confirmations done within holding times?

15. Are confirmations reported quantitatively?

    Are the same QC criteria applied to both first and second column?

    Are MDL studies performed on both columns? Detectors?

16. Is dilution expressed as a dilution factor, e.g., 5x or as a ratio, e.g., 1:5

17. Are written corrective action procedures available?

DIOXINS/FURANS - LOW RESOLUTION (8280, 8280A)

Laboratory personnel:

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1. Are SOPs available in the lab?

   Updated methods used?
   Date?
   Signature?
   Current?
   Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

   How are project requirements communicated to the analysts?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?

   Are entries dated and initialed by analyst?

   Is supervisory review documented?

   Do logbooks document return to control?

4. Are standards logbooks complete and up-to-date?

   Do they contain procedures for preparing standards?

   Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are standards checked routinely for expiration?

   Does the expiration date on secondary standards exceed that for the primary standard?

   Are expired standards discarded?

7. Is instrument tuning checked?
How often?

8. Do logbooks indicate initial calibration using at least 5 concentration levels of standards?
Are standards levels identified?
Is the lowest standard at the PQL?

Do the standards contain the complete analyte list?
Are acceptance criteria specified in the SOP? Are these followed?

How are retention time windows determined? How often?
Acceptance criteria specified in SOP?

9. Are continuing calibration check standards run every 12 hours before sample analysis?

Does this standard contain the complete analyte list?

10. Do run logs document a method blank, and LCS for every 20 samples?

Are MS/MSDs performed?

Do LCS and MS/MSDs contain all analytes of interest?

11. Are internal standards added to each sample, blank and standard? Are standard IDs entered in the logbook?

Are recovery standards added to each sample, blank and standard?

12. Are second column confirmations being done when 2378-TCDD or TCDF is detected?

13. Are confirmations done using a different column?

14. If not run simultaneously, are confirmations done within holding times?

15. Are confirmations reported quantitatively?

Are the same QC criteria applied to both first and second column?

DIOXINS/FURANS - HIGH RESOLUTION (8290)

Laboratory personnel:

---

1. Are SOPs available in the lab?

Updated methods used?
Date?
Signature?
Current?
Controlled?

2. Are current (project) QC limits available in the lab or on the LIMS system, and are these used to validate the data?

How are project requirements communicated to the analysts?

3. Are maintenance logbooks kept for each instrument and are they up-to-date? Paginated?

Are entries dated and initialed by analyst?

Is supervisory review documented?

Do logbooks document return to control?

4. Are standards logbooks complete and up-to-date?

Do they contain procedures for preparing standards?

Are standards traceable to nationally recognized sources?

5. Are standards labeled with reference to standard log, identification of material, concentration, preparation and expiration date, and preparer ID?

6. Are standards checked routinely for expiration?

Does the expiration date on secondary standards exceed that for the primary standard?

Are expired standards discarded?

7. Is instrument tuning checked?
   How often?

8. Do logbooks indicate initial calibration using at least 5 concentration levels of standards?
   Are standards levels identified?
   Is the lowest standard at the PQL?

   Do the standards contain the complete analyte list?
   Are acceptance criteria specified in the SOP? Are these followed?

   How are retention time windows determined? How often?

9. Are continuing calibration check standards run every 12 hours before sample analysis?

   Does this standard contain the complete analyte list?

10. Do run logs document a method blank, and LCS for every 20 samples?

    Are MS/MSDs performed?

    Do LCS and MS/MSDs contain all analytes of interest?

11. Are internal standards added to each sample, blank and standard? Are standard IDs entered in the logbook?

    Are recovery standards added to each sample, blank and standard?

12. Are second column confirmations to determine isomer specificity being done when 2378-TCDD or TCDF is detected?

13. What columns are used?

14. If not run simultaneously, are confirmations done within holding times?

15. Are the same QC criteria applied to both columns?

FRONTIER GEOSCIENCES, INC.

ENVIRONMENTAL STANDARDS

Setting the Standards for Innovative Environmental Solutions

LABORATORY EVALUATION OF

FRONTIER GEOSCIENCE INC.

OF SEATTLE, WASHINGTON

September 6, 2000

Prepared for:

BLUE292
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Prepared by:

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INTRODUCTION

On August 21, 2000, Environmental Standards, Inc. (Environmental Standards) personnel Donald J. Lancaster, MS, and Lester J. Dupes, CEAC, CPC, performed an on-site audit/evaluation of the analytical procedures of the Frontier Geosciences Inc. (FRONTIER) facility located in Seattle, Washington. This audit was performed to provide Blue292 with feedback on laboratory operating issues relative to three general areas: method compliance, laboratory systems, and good laboratory practices. The audit was also specifically conducted to evaluate this laboratory's ability to perform analytical work for air, industrial hygiene, and environmental samples for Blue292 projects. The findings of the audit are presented herein.

FRONTIER is an environmental laboratory specializing in the analysis of environmental and industrial hygiene samples. The laboratory began in 1991 in the basement of a residence. In July 1992, the laboratory moved into its present location, and has steadily grown to occupy 8,600 square feet of space in its present location. The laboratory is currently looking for a larger facility in the same (or a nearby) neighborhood. The move to a larger facility would occur in 2002. The laboratory holds certifications from the states of California, Florida, Louisiana, New Jersey, New York, Washington, and Wisconsin. The laboratory employs 46 personnel, of which 30 hold technical positions. The Quality Assurance (QA) Officer is a member of the American Society for Quality and is an IRCA-certified Quality Systems Provisional Auditor.

Approximately 43% of FRONTIER's analytical work is from consulting firms, 15% is directly from industry, 14% is from governmental agencies (federal, state, and municipal), 12% is from universities and research institutions, and the remaining is from public utilities and other laboratories. The most frequently performed methods for environmental testing are total mercury in waters, tissues, soils, and sediments, total atmospheric mercury, methyl mercury in waters, soils, sediments, and tissues, and inductively-coupled plasma mass spectroscopy (ICP-MS) analyses for metals in waters, sediments, and tissues.

The normal operating hours of the laboratory is 0800 to 1600, Monday through Saturday. The laboratory often operating much longer hours based on workload demands. Routine sample receiving and login are conducted during the Saturday hours. The laboratory is currently 8,600 square feet in size; of this, 2,000 square feet is dedicated to analytical space. Attachment 1 is the laboratory floor plan.

The laboratory has a central alarm system that is monitored by an outside agency. The fire alarms and refrigerator temperature monitoring system are not tied into the security alarm system. Sample receiving does not have its own entrance to the building – sample shipments are brought through the front door. The front entrance has an alarm activated when the door is opened. Visitors to the laboratory are required to sign in and are escorted though the laboratory. Uninterrupted power supplies (UPSs) and surge protectors are used to protect the desktop computers and the aqueous mercury analyzer. All other instruments are not connected to the UPS. The laboratory's server is protected with a monitored UPS and surge protector; all power events for the server are logged via

Department of Energy

Oak Ridge Operations Office
P.O. Box 2001
Oak Ridge, Tennessee 37831—

October 6, 1999

Mr. Nicholas Bloom
Frontier Geosciences
414 Pontius Avenue, North
Seattle, Washington 98109

Dear Mr. Bloom:

THE DEPARTMENT OF ENERGY OAK RIDGE OPERATIONS OFFICE ENVIRONMENTAL MANAGEMENT - ANNUAL LABORATORY QUALIFICATION AUDIT OF FRONTIER GEOSCIENCES, INC, SEATTLE, WASHINGTON

An annual laboratory qualification audit of Frontier Geosciences, Inc. (FGI) was conducted on August 26, 1999, by the Department of Energy (DOE), Oak Ridge Operations Office (ORO), Environmental Management, Waste Management & Technical Integration Team. A detailed audit report is enclosed for your review and action. The report reflects the information presented in the audit teams closeout briefing on August 26, 1999. Comments made during that meeting have been reconciled.

We are transmitting by certified mail, the August 1999 audit report for FGI. This will establish the official date where by the Corrective Action Plan (CAP) due date can be tracked in our Integrated Performance Indicator Program (IPIP). Your receipt of this letter, and enclosed report indicates the start date of the 30 calendar day period for corrective action plan submittal. Late submittals may result in penalties in our IPIP. Following is the specific format that the CAP must be in:

The CAP must address each Finding listed in Section 3.2 of the report. Responses to Priority II Findings will be formally tracked in our Corrective Action Tracking System until resolution as a part of our IPIP. Responses to Priority III Findings will not be formally tracked; however, we may check their status during the next audit or surveillance to ensure that noted problems have been resolved. Observations included in Section 3.3 of the report are provided for laboratory information only; any corrective action response is optional but encouraged.

(1) Finding: Restate the finding and its associated number. It is not necessary to repeat the discussion portion of the finding.

CERTIFIED MAIL - RETURN RECEIPT REQUESTED - (P 151 529 849)

(2) Response: Provide a summary response to the finding that states the cause of the finding and what will be done to prevent recurrence. This is basically an explanation, in sentence format, of what you have determined to be the underlying reason that the finding occurred and what actions you will take to correct the finding and prevent recurrence.

(3) Itemized Corrective Actions: Present a tabular listing of the individual corrective actions that will be performed to address a finding and the estimated completion date for each corrective action. NOTE: In the past, several labs have not given us the date that actions are planned to be complete. Failure to supply the date will result in rejection of your CAP.

The CAP must be submitted to:

David Carden
Waste Management & Technical Integration Team
Environmental Management (EM-921)
U. S. Department of Energy
Post Office Box 2001
Oak Ridge, Tennessee 37830

Please provide your CAP to me in hard copy and electronic format (WordPerfect or Microsoft Word).

If you have any questions on this report or on the required format for corrective action response, please contact the Audit Team Leader, Peggy Wilson at (423) 576-5766.

Sincerely,

Clayton L. G.

Clayton Gist, Team Leader
Waste Management & Technical
Integration Team

Enclosure

cc w/enclosure:
David Carden, EM-923
Peggy Wilson, EM-923
Kelli Henry, BJC, K-1007, MS 7056
Chip Davis, Parallax, Inc.
Tom Jeskie, Parallax, Inc.
David Springmeier, PAI
Ahmed Halouma, NFT Inc.

ATTACHMENT 6

AQUEOUS SW8310 QES/STL LABORATORY CONTROL SAMPLE RESULTS FOR 26 APRIL 2000

LABORATORY CONTROL SAMPLE EVALUATION REPORT

HPLC

Client Lot #...: G0D140300 **Work Order #...:** DC4GR102-LCS **Matrix.....:** WATER
LCS Lot-Sample#: G0D190000-156 DC4GR103-LCSD
Prep Date.....: 04/19/00 **Analysis Date..:** 04/25/00
Prep Batch #...: 0110156
Dilution Factor: 1

PARAMETER	PERCENT RECOVERY	RECOVERY LIMITS	RPD	RPD LIMITS	METHOD
Acenaphthene	66	(50 - 150)			SW846 8310
	61	(50 - 150)	6.8	(0-35)	SW846 8310
Acenaphthylene	69	(50 - 150)			SW846 8310
	63	(50 - 150)	7.9	(0-35)	SW846 8310
Anthracene	68	(50 - 150)			SW846 8310
	68	(50 - 150)	1.2	(0-35)	SW846 8310
Benzo(a)anthracene	74	(50 - 150)			SW846 8310
	74	(50 - 150)	0.48	(0-35)	SW846 8310
Benzo(a)pyrene	**74**	**(48 - 112)**			**SW846 8310**
	72	**(48 - 112)**	**2.6**	**(0-17)**	**SW846 8310**
Benzo(b)fluoranthene	76	(50 - 150)			SW846 8310
	74	(50 - 150)	2.3	(0-35)	SW846 8310
Benzo(ghi)perylene	73	(50 - 150)			SW846 8310
	71	(50 - 150)	2.7	(0-35)	SW846 8310
Benzo(k)fluoranthene	76	(50 - 150)			SW846 8310
	74	(50 - 150)	2.5	(0-35)	SW846 8310
Chrysene	74	(50 - 150)			SW846 8310
	73	(50 - 150)	1.0	(0-35)	SW846 8310
Dibenz(a,h)anthracene	63	(50 - 150)			SW846 8310
	65	(50 - 150)	3.4	(0-50)	SW846 8310
Fluoranthene	70	(50 - 150)			SW846 8310
	73	(50 - 150)	4.2	(0-35)	SW846 8310
Fluorene	**66**	**(45 - 109)**			**SW846 8310**
	64	**(45 - 109)**	**3.6**	**(0-20)**	**SW846 8310**
Indeno(1,2,3-cd)pyrene	**76**	**(54 - 112)**			**SW846 8310**
	74	**(54 - 112)**	**2.2**	**(0-18)**	**SW846 8310**
Naphthalene	**69**	**(30 - 113)**			**SW846 8310**
	61	**(30 - 113)**	**11**	**(0-28)**	**SW846 8310**
Phenanthrene	65	(50 - 150)			SW846 8310
	69	(50 - 150)	4.9	(0-35)	SW846 8310
Pyrene	**68**	**(45 - 118)**			**SW846 8310**
	69	**(45 - 118)**	**1.7**	**(0-18)**	**SW846 8310**

SURROGATE	PERCENT RECOVERY	RECOVERY LIMITS
1-Methylnaphthalene	135 *	(33 - 119)
	114	(33 - 119)

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results

Bold print denotes control parameters

* Surrogate recovery is outside stated control limits.

LABORATORY CONTROL SAMPLE EVALUATION REPORT

HPLC

Client Lot #...: G0D120282 **Work Order #...:** DC1WF102-LCS **Matrix.....:** WATER
LCS Lot-Sample#: G0D170000-255 DC1WF103-LCSD
Prep Date.....: 04/17/00 **Analysis Date..:** 04/26/00
Prep Batch #...: 0108255
Dilution Factor: 1

PARAMETER	PERCENT RECOVERY	RECOVERY LIMITS	RPD	RPD LIMITS	METHOD
Benzo(a)pyrene	**87**	**(48 - 112)**			**SW846 8310**
	90	**(48 - 112)**	**3.5**	**(0-17)**	**SW846 8310**
Fluorene	**59**	**(45 - 109)**			**SW846 8310**
	58	**(45 - 109)**	**2.7**	**(0-20)**	**SW846 8310**
Indeno(1,2,3-cd)pyrene	**96**	**(54 - 112)**			**SW846 8310**
	101	**(54 - 112)**	**5.7**	**(0-18)**	**SW846 8310**
Naphthalene	**57**	**(30 - 113)**			**SW846 8310**
	54	**(30 - 113)**	**5.8**	**(0-28)**	**SW846 8310**
Pyrene	**83**	**(45 - 118)**			**SW846 8310**
	86	**(45 - 118)**	**3.4**	**(0-18)**	**SW846 8310**

SURROGATE	PERCENT RECOVERY	RECOVERY LIMITS
1-Methylnaphthalene	66	(33 - 119)
	56	(33 - 119)

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results.

Bold print denotes control parameters

LABORATORY CONTROL SAMPLE DATA REPORT

HPLC

Client Lot #...: G0D060231 **Work Order #...:** DC1VR102 **Matrix.....:** SOLID
LCS Lot-Sample#: G0D170000-244
Prep Date.....: 04/17/00 **Analysis Date..:** 04/26/00
Prep Batch #...: 0108244
Dilution Factor: 1

PARAMETER	SPIKE AMOUNT	MEASURED AMOUNT	UNITS	PERCENT RECOVERY	METHOD
Benzo(a)pyrene	**66.7**	**73.4 a**	**ug/kg**	**110**	**SW846 8310**
Fluorene	**133**	**125**	**ug/kg**	**94**	**SW846 8310**
Indeno(1,2,3-cd)pyrene	**66.7**	**80.9 a**	**ug/kg**	**121**	**SW846 8310**
Naphthalene	**667**	**653**	**ug/kg**	**98**	**SW846 8310**
Pyrene	**133**	**142**	**ug/kg**	**107**	**SW846 8310**

SURROGATE	PERCENT RECOVERY	RECOVERY LIMITS
1-Methylnaphthalene	108	(41 - 115)

NOTE(S):

Calculations are performed before rounding to avoid round-off errors in calculated results

Bold print denotes control parameters

a Spiked analyte recovery is outside stated control limits.

STL Reference Data Summary

Target Analyte List: Q. SW846 Method 8310 Standard List

Matrix: WATER
Extraction: LIQ/LIQ, SEP FUNNEL (PAH, P/P, TPH, DIOXIN)-> Large V
Method: Hydrocarbons, Polynuclear Aromatic (HPLC - 8310)
QC Program: STANDARD TEST SET
Location: STL Sacramento

Target List 2500	Detection Limits					Check List 20798					Spike List 20799				
Compound	RL	Units	MDL	Units	Run Date	Amt	Units	LCL	UCL	RPD	Amt	Units	LCL	UCL	RPD
Acenaphthene	0.10	ug/L	0.05	ug/L	19991130	1.0	ug/L	50	150	35	1.0	ug/L	50	150	35
Acenaphthylene	0.05	ug/L	0.025	ug/L	19991130	0.5	ug/L	50	150	35	0.5	ug/L	50	150	35
Anthracene	0.002	ug/L	0.001	ug/L	19991130	0.02	ug/L	50	150	35	0.02	ug/L	50	150	35
Benzo(a)anthracene	0.005	ug/L	0.0025	ug/L	19991130	0.05	ug/L	50	150	35	0.05	ug/L	50	150	35
Benzo(b)fluoranthene	0.0025	ug/L	0.0012	ug/L	19991130	0.02	ug/L	50	150	35	0.02	ug/L	50	150	35
Benzo(k)fluoranthene	0.0025	ug/L	0.0012	ug/L	19991130	0.02	ug/L	50	150	35	0.02	ug/L	50	150	35
Benzo(ghi)perylene	0.01	ug/L	0.005	ug/L	19991130	0.08	ug/L	50	150	35	0.08	ug/L	50	150	35
Benzo(a)pyrene	0.005	ug/L	0.0025	ug/L	19991130	0.05	ug/L	48	112	17	0.05	ug/L	48	112	17
Chrysene	0.005	ug/L	0.0025	ug/L	19991130	0.05	ug/L	50	150	35	0.05	ug/L	50	150	35
Dibenz(a,h)anthracene	0.02	ug/L	0.01	ug/L	19991130	0.2	ug/L	50	150	50	0.2	ug/L	50	150	50
Fluoranthene	0.005	ug/L	0.0025	ug/L	19991130	0.05	ug/L	50	150	35	0.05	ug/L	50	150	35
Fluorene	0.01	ug/L	0.005	ug/L	19991130	0.1	ug/L	45	109	20	0.1	ug/L	45	109	20
Indeno(1,2,3-cd)pyrene	0.005	ug/L	0.0025	ug/L	19991130	0.05	ug/L	54	112	18	0.05	ug/L	54	112	18
Naphthalene	0.05	ug/L	0.025	ug/L	19991130	0.5	ug/L	30	113	28	0.5	ug/L	30	113	28
Phenanthrene	0.004	ug/L	0.002	ug/L	19991130	0.04	ug/L	50	150	35	0.04	ug/L	50	150	35
Pyrene	0.01	ug/L	0.005	ug/L	19991130	0.1	ug/L	45	118	18	0.1	ug/L	45	118	18
1-Methylnaphthalene						5.0	ug/L	33	119	0	5.0	ug/L	33	119	0

STL Reference Data Summary

Structured Analysis Code: A-13-SG-01-07

Target Analyte List: Q. SW846 Method 8310 Standard List

Matrix: SOLID
Extraction: SONICATION - Low Level
Method: Hydrocarbons, Polynuclear Aromatic (HPLC - 8310)
QC Program: STANDARD TEST SET
Location: STL Sacramento

Target List 2500		Detection Limits					Check List 20610							Spike List 20611						
Syn	Compound	RL	Units	MDL	Units	Run Date	T	A	Amt	Units	LCL	UCL	RPD	T	A	Amt	Units	LCL	UCL	RPD
1	Acenaphthene	400	ug/kg	63	ug/kg	19980717		Y	1330	ug/kg	50	150	50		Y	1330	ug/kg	50	150	50
5	Acenaphthylene	200	ug/kg	46	ug/kg	19980717		Y	667	ug/kg	50	150	50		Y	667	ug/kg	50	150	50
122	Anthracene	8	ug/kg	1.1	ug/kg	19980717		Y	26.6	ug/kg	50	150	50		Y	26.6	ug/kg	50	150	50
202	Benzo(a)anthracene	16	ug/kg	1.7	ug/kg	19980717		Y	66.7	ug/kg	50	150	50		Y	66.7	ug/kg	50	150	50
205	Benzo(b)fluoranthene	4.0	ug/kg	2.4	ug/kg	19980402		Y	26.6	ug/kg	50	150	50		Y	26.6	ug/kg	50	150	50
208	Benzo(k)fluoranthene	4.0	ug/kg	1.1	ug/kg	19980717		Y	26.6	ug/kg	50	150	50		Y	26.6	ug/kg	50	150	50
210	Benzo(ghi)perylene	16	ug/kg	3.1	ug/kg	19980717		Y	106.4	ug/kg	50	150	50		Y	106.4	ug/kg	50	150	50
211	Benzo(a)pyrene	10	ug/kg	3.1	ug/kg	19980717	C	Y	66.7	ug/kg	49	107	53	C	Y	66.7	ug/kg	49	107	53
633	Chrysene	20	ug/kg	14	ug/kg	19980717		Y	66.7	ug/kg	50	150	50		Y	66.7	ug/kg	50	150	50
860	Dibenz(a,h)anthracene	40	ug/kg	9.2	ug/kg	19980717		Y	266	ug/L	50	150	50		Y	266	ug/kg	50	150	50
1414	Fluoranthene	20	ug/kg	4.8	ug/kg	19980717		Y	66.7	ug/kg	50	150	50		Y	66.7	ug/kg	50	150	50
1417	Fluorene	40	ug/kg	6.7	ug/kg	19980717	C	Y	133	ug/kg	43	112	56	C	Y	133	ug/kg	43	112	56
1535	Indeno(1,2,3-cd)pyrene	20	ug/kg	3.1	ug/kg	19980717	C	Y	66.7	ug/kg	54	114	51	C	Y	66.7	ug/kg	54	114	51
1932	Naphthalene	200	ug/kg	23	ug/kg	19980717	C	Y	667	ug/kg	44	110	50	C	Y	667	ug/kg	44	110	50
2154	Phenanthrene	16	ug/kg	2.6	ug/kg	19980717		Y	53.2	ug/kg	50	150	50		Y	53.2	ug/kg	50	150	50
2252	Pyrene	40	ug/kg	11	ug/kg	19980717	C	Y	133	ug/kg	49	115	54	C	Y	133	ug/kg	49	115	54
2770	1-Methylnaphthalene						X	Y	3330	ug/kg	41	115	0	X	Y	3330	ug/kg	41	115	0

ATTACHMENT 7

QES/STL REVIEW OF TEPH LABORATORY CONTROL SAMPLE NON-CONFORMANCES, AUGUST 2000

Quanterra
Environmental Services

Pamela A. Schemmer
Quality Assurance Manager

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

(916) 374-4441 Direct
(916) 372-1059 Fax

RECEIVED
NOV 1 - 2000

September 25, 2000

Mr. Chris Davis
Earth Tech, San Jose
695 River Oaks Parkway
San Jose, CA 95134

Subject: QA Review of TEPH Diesel problems

Dear Mr. Davis:

I have reviewed the issues associated with the TEPH analysis for the Earth Tech Benicia projects. In August 2000, STL Sacramento experienced problems with Diesel recovery in water LCS samples. I want to assure you this situation was completely investigated, and has been resolved.

Due to time delays between sample preparation and analysis during this period, the problem affected several weeks worth of prep batches. This included 6 Benicia lots, over 4 preparation batches. Once the problem was identified by the instrumentation group, all further preparations and re-extractions were ceased. All prep analysts performed a series of 4 LCSs for method verification at this point. All reagents, standards, and the preparation glassware were tested for possible sources of loss, to no avail. As further data was acquired on the instrumentation, it was evident the problem was isolated to low level water LCS samples, and did not affect soil extractions or higher level water samples that had a larger spike volume and resulted in a higher final volume. Also, the chromatograms showed that Diesel was unevenly affected, and the early portion was more degraded. Since Motor Oil and surrogate recoveries were unaffected for the same batches, we believed there was a loss of the more volatile portion of the Diesel spike. Since extract concentration procedures had already been verified as acceptable, it was determined there may have been a slight change in spike techniques which led to the loss.

LCS samples are spiked differently from other samples and spikes. Samples and MS/MSD samples are spiked in their original containers just above the water level, and are then transferred to separatory funnels. Since there are no original sample containers for blanks and LCSs, 1000 mL of reagent water is measured and transferred to a 2-L separatory funnel, and then spiked. Since the water level is well below top of the separatory funnel, an analyst is only able to apply the spike a several inches above the water level. When this is done, there is some dispersion of the spike in the separatory funnel. The acetone-diesel spike that does reach the surface of the

water can be noted to sit on the surface like a diesel spill, and "creep" up the sides of the separatory funnel. During this particularly busy time in the preparation laboratory, the separatory funnels were allowed to sit open after spiking while the rest of the batch was transferred to separatory funnels. When we isolated this problem, we determined that simply tilting the separatory funnel to spike closer to the water surface, and giving the funnel a quick mix after spiking, was sufficient to greatly impact Diesel recoveries. All separatory funnels are now capped after spiking or transfer to prevent loss.

Since determining the source of the problem and educating the preparation chemists on this technique, we have seen no further failures of LCSs. A copy of our most recent control chart is included. I have also included a copy of our most recent PE sample, which was prepared after this problem was resolved. Since the problem was due to the physical spiking of Diesel in the LCSs, and extraction efficiency was supported by Motor Oil and surrogate recoveries, any Diesel inherent in the samples should not have been affected. This problem was completely unrelated to the soil MS/MSD failures you noted. MS/MSD failures are generally a function of matrix interference, especially with soil TPH samples, and need to be addressed on a matrix specific basis.

I apologize for the inconvenience this may have caused you. If you have any further questions with this issue, please feel free to contact me at (916)-374-4441.

Sincerely,

Pamela Schemmer

Pamela A. Schemmer
Quality Assurance Manager

CC: Diana Brooks, Project Manager, STL Sacramento

Diesel Range Organics

QuantIMS Compound #: 797

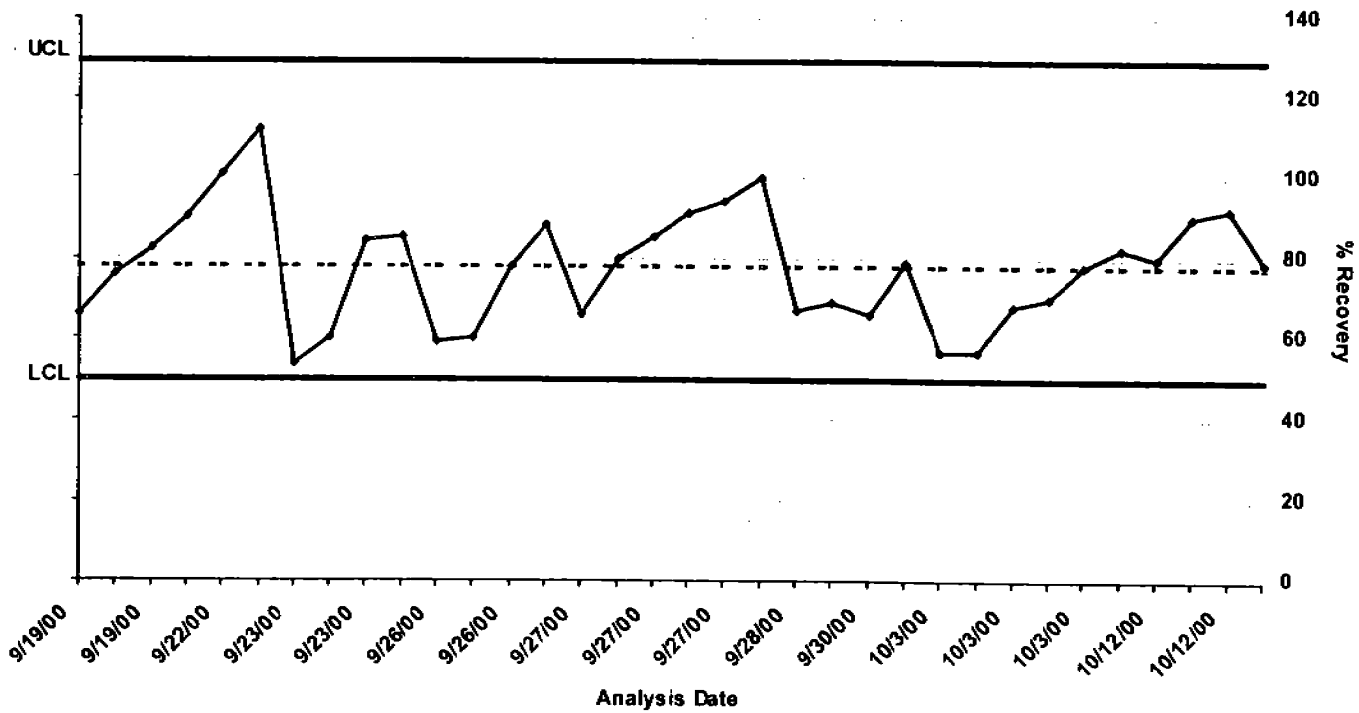
QuantIMS Spike List: 20600 (SAC: GCSEMI 8015MOD-TPH LCS)

Laboratory Control Sample
Aqueous

Analysis Dates: 09/19/2000 - 10/13/2000

Method	Method Description	Extraction
8015 MOD	Hydrocarbons, Extractable Petroleum (8015 MOD)	Not specified

Quality Control Chart



Statistical Calculations

N	36
Mean %R:	77.889
Std. Dev.:	13.761
Spike Level:	300
Spike Units:	ug/L
QIMS LCL	50
QIMS UCL	129
QIMS RPD	23

Calculated Limits

CALC LWL	50
CALC UWL	105
CALC LCL	37
CALC UCL	119
Calc RPD	42.79

Rejected data points are not charted

ENVIRONMENTAL RESOURCE ASSOCIATES®

UST-31 Final Customer Report

ERA Laboratory Code: R1456-02 **EPA ID: CA00044** **State ID:** **Report Issued: 10/17/00**

ERA Standard	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Gasoline in Soil	Unleaded Gasoline	mg/Kg	447.0000	644	D.L. - 1290	Acceptable	AK101
	Benzene	mg/Kg	1.8800	7.08	D.L. - 7.08	Acceptable	AK101
	Ethylbenzene	mg/Kg	11.4000	18.0	4.03 - 20.2	Acceptable	AK101
	Toluene	mg/Kg	29.4000	62.3	D.L. - 69.2	Acceptable	AK101
	Xylenes, total	mg/Kg	62.2000	61.1	20.2 - 113	Acceptable	AK101
Diesel in Soil	No. 2 Diesel	mg/Kg	536	602	D.L. - 951	Acceptable	AK102
TPH in Soil	TPH (gravimetric)	mg/Kg		2250	D.L. - 6990		
	TPH (IR)	mg/Kg		2700	D.L. - 5310		
BTEX in Soil	Benzene	µg/Kg	385.0000	345	204 - 480	Acceptable	8021B
	Ethylbenzene	µg/Kg	60.5000	54.5	22.6 - 84.4	Acceptable	8021B
	Toluene	µg/Kg	173.0000	154	85.5 - 227	Acceptable	8021B
	Xylenes, total	µg/Kg	321.0000	283	113 - 467	Acceptable	8021B
MTBE in Soil	t-Butylmethylether (MTBE)	µg/Kg		32.1	19.5 - 52.1		
Gasoline in Water	Unleaded Gasoline	µg/l	4380.0000	3540	1400 - 7160	Acceptable	AK101
	Benzene	µg/l	34.5000	39.3	13.0 - 61.2	Acceptable	AK101
	Ethylbenzene	µg/l	83.2000	91.3	65.8 - 102	Acceptable	AK101
	Toluene	µg/l	275.0000	274	99.9 - 494	Acceptable	AK101
	Xylenes, total	µg/l	433.0000	354	199 - 727	Acceptable	AK101
Diesel in Water	No. 2 Diesel	µg/l	3320	3440	D.L. - 5800	Acceptable	AK102
TPH in Water	TPH (gravimetric)	mg/bttl		54.3	D.L. - 84.7		
	TPH (IR)	mg/bttl		65.2	9.30 - 125		
BTEX in Water	Benzene	µg/l	51.8000	58.6	33.4 - 81.8	Acceptable	8021B
	Ethylbenzene	µg/l	119.0000	142	83.3 - 197	Acceptable	8021B
	Toluene	µg/l	64.3000	73.9	43.2 - 103	Acceptable	8021B
	Xylenes, total	µg/l	97.9000	112	70.8 - 151	Acceptable	8021B
MTBE in Water	t-Butylmethylether (MTBE)	µg/l		62.1	41.6 - 93.0		

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ENVIRONMENTAL RESOURCE ASSOCIATES®

UST-31 Final Customer Report

ERA Laboratory Code: R1456-02 **EPA ID: CA00044** **State ID:** **Report Issued: 10/17/00**

ERA Standard	Analyte	Units	Reported Value	Assigned Value	Acceptance Limits	Performance Evaluation	Method Description
Gasoline in Soil	Unleaded Gasoline	mg/Kg	642.0000	644	D.L. - 1290	Acceptable	CA LUFT
	Benzene	mg/Kg	1.8800	7.08	D.L. - 7.08	Acceptable	CA LUFT
	Ethylbenzene	mg/Kg	11.4000	18.0	4.03 - 20.2	Acceptable	CA LUFT
	Toluene	mg/Kg	29.4000	62.3	D.L. - 69.2	Acceptable	CA LUFT
	Xylenes, total	mg/Kg	62.2000	61.1	20.2 - 113	Acceptable	CA LUFT
Diesel in Soil	No. 2 Diesel	mg/Kg	562	602	D.L. - 951	Acceptable	CA LUFT
TPH in Soil	TPH (gravimetric)	mg/Kg		2250	D.L. - 6990		
	TPH (IR)	mg/Kg		2700	D.L. - 5310		
BTEX in Soil	Benzene	µg/Kg		345	204 - 480		
	Ethylbenzene	µg/Kg		54.5	22.6 - 84.4		
	Toluene	µg/Kg		154	85.5 - 227		
	Xylenes, total	µg/Kg		283	113 - 467		
MTBE in Soil	t-Butylmethylether (MTBE)	µg/Kg		32.1	19.5 - 52.1		
Gasoline in Water	Unleaded Gasoline	µg/l	5170.0000	3540	1400 - 7160	Acceptable	CA LUFT
	Benzene	µg/l	34.5000	39.3	13.0 - 61.2	Acceptable	CA LUFT
	Ethylbenzene	µg/l	83.2000	91.3	65.8 - 102	Acceptable	CA LUFT
	Toluene	µg/l	275.0000	274	99.9 - 494	Acceptable	CA LUFT
	Xylenes, total	µg/l	433.0000	354	199 - 727	Acceptable	CA LUFT
Diesel in Water	No. 2 Diesel	µg/l	3320	3440	D.L. - 5800	Acceptable	CA LUFT
TPH in Water	TPH (gravimetric)	mg/bttl		54.3	D.L. - 84.7		
	TPH (IR)	mg/bttl		65.2	9.30 - 125		
BTEX in Water	Benzene	µg/l		58.6	33.4 - 81.8		
	Ethylbenzene	µg/l		142	83.3 - 197		
	Toluene	µg/l		73.9	43.2 - 103		
	Xylenes, total	µg/l		112	70.8 - 151		
MTBE in Water	t-Butylmethylether (MTBE)	µg/l		62.1	41.6 - 93.0		

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ERA Laboratory Code: R1456-02 **EPA ID: CA00044** **State ID:** **Report Issued: 10/17/00**

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Gasoline in Soil	Unleaded Gasoline	mg/Kg	589.0000	644	D.L. - 1290	Acceptable	NWTPH GX
	Benzene	mg/Kg	1.8800	7.08	D.L. - 7.08	Acceptable	NWTPH GX
	Ethylbenzene	mg/Kg	11.4000	18.0	4.03 - 20.2	Acceptable	NWTPH GX
	Toluene	mg/Kg	29.4000	62.3	D.L. - 69.2	Acceptable	NWTPH GX
	Xylenes, total	mg/Kg	62.2000	61.1	20.2 - 113	Acceptable	NWTPH GX
Diesel in Soil	No. 2 Diesel	mg/Kg	353	602	D.L. - 951	Acceptable	NWTPH DX
TPH in Soil	TPH (gravimetric)	mg/Kg		2250	D.L. - 6990		
	TPH (IR)	mg/Kg		2700	D.L. - 5310		
BTEX in Soil	Benzene	µg/Kg		345	204 - 480		
	Ethylbenzene	µg/Kg		54.5	22.6 - 84.4		
	Toluene	µg/Kg		154	85.5 - 227		
	Xylenes, total	µg/Kg		283	113 - 467		
MTBE in Soil	t-Butylmethylether (MTBE)	µg/Kg		32.1	19.5 - 52.1		
Gasoline in Water	Unleaded Gasoline	µg/l	4820.0000	3540	1400 - 7160	Acceptable	NWTPH GX
	Benzene	µg/l	34.5000	39.3	13.0 - 61.2	Acceptable	NWTPH GX
	Ethylbenzene	µg/l	83.2000	91.3	65.8 - 102	Acceptable	NWTPH GX
	Toluene	µg/l	275.0000	274	99.9 - 494	Acceptable	NWTPH GX
	Xylenes, total	µg/l	433.0000	354	199 - 727	Acceptable	NWTPH GX
Diesel in Water	No. 2 Diesel	µg/l	3460	3440	D.L. - 5800	Acceptable	NWTPH DX
TPH in Water	TPH (gravimetric)	mg/bttl		54.3	D.L. - 84.7		
	TPH (IR)	mg/bttl		65.2	9.30 - 125		
BTEX in Water	Benzene	µg/l		58.6	33.4 - 81.8		
	Ethylbenzene	µg/l		142	83.3 - 197		
	Toluene	µg/l		73.9	43.2 - 103		
	Xylenes, total	µg/l		112	70.8 - 151		
MTBE in Water	t-Butylmethylether (MTBE)	µg/l		62.1	41.6 - 93.0		

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Quanterra
Environmental Services

Pamela A. Schemmer
Quality Assurance Manager

STL Sacramento
880 Riverside Parkway
West Sacramento, CA 95605

(916) 374-4441 Direct
(916) 372-1059 Fax

September 25, 2000

Mr. Chris Davis
Earth Tech, San Jose
695 River Oaks Parkway
San Jose, CA 95134

Subject: QA Review of TEPH Diesel problems

Dear Mr. Davis:

I have reviewed the issues associated with the TEPH analysis for the Earth Tech Benicia projects. In August 2000, STL Sacramento experienced problems with Diesel recovery in water LCS samples. I want to assure you this situation was completely investigated, and has been resolved.

Due to time delays between sample preparation and analysis during this period, the problem affected several weeks worth of prep batches. This included 6 Benicia lots, over 4 preparation batches. Once the problem was identified by the instrumentation group, all further preparations and re-extractions were ceased. All prep analysts performed a series of 4 LCSs for method verification at this point. All reagents, standards, and the preparation glassware were tested for possible sources of loss, to no avail. As further data was acquired on the instrumentation, it was evident the problem was isolated to low level water LCS samples, and did not affect soil extractions or higher level water samples that had a larger spike volume and resulted in a higher final volume. Also, the chromatograms showed that Diesel was unevenly affected, and the early portion was more degraded. Since Motor Oil and surrogate recoveries were unaffected for the same batches, we believed there was a loss of the more volatile portion of the Diesel spike. Since extract concentration procedures had already been verified as acceptable, it was determined there may have been a slight change in spike techniques which led to the loss.

LCS samples are spiked differently from other samples and spikes. Samples and MS/MSD samples are spiked in their original containers just above the water level, and are then transferred to separatory funnels. Since there are no original sample containers for blanks and LCSs, 1000 mL of reagent water is measured and transferred to a 2-L separatory funnel, and then spiked. Since the water level is well below top of the separatory funnel, an analyst is only able to apply the spike a several inches above the water level. When this is done, there is some dispersion of the spike in the separatory funnel. The acetone-diesel spike that does reach the surface of the

water can be noted to sit on the surface like a diesel spill, and "creep" up the sides of the separatory funnel. During this particularly busy time in the preparation laboratory, the separatory funnels were allowed to sit open after spiking while the rest of the batch was transferred to separatory funnels. When we isolated this problem, we determined that simply tilting the separatory funnel to spike closer to the water surface, and giving the funnel a quick mix after spiking, was sufficient to greatly impact Diesel recoveries. All separatory funnels are now capped after spiking or transfer to prevent loss.

Since determining the source of the problem and educating the preparation chemists on this technique, we have seen no further failures of LCSs. A copy of our most recent control chart is included. I have also included a copy of our most recent PE sample, which was prepared after this problem was resolved. Since the problem was due to the physical spiking of Diesel in the LCSs, and extraction efficiency was supported by Motor Oil and surrogate recoveries, any Diesel inherent in the samples should not have been affected. This problem was completely unrelated to the soil MS/MSD failures you noted. MS/MSD failures are generally a function of matrix interference, especially with soil TPH samples, and need to be addressed on a matrix specific basis.

I apologize for the inconvenience this may have caused you. If you have any further questions with this issue, please feel free to contact me at (916)-374-4441.

Sincerely,

Pamela Schemmer

Pamela A. Schemmer
Quality Assurance Manager

CC: Diana Brooks, Project Manager, STL Sacramento

Diesel Range Organics

QuantIMS Compound #: 797

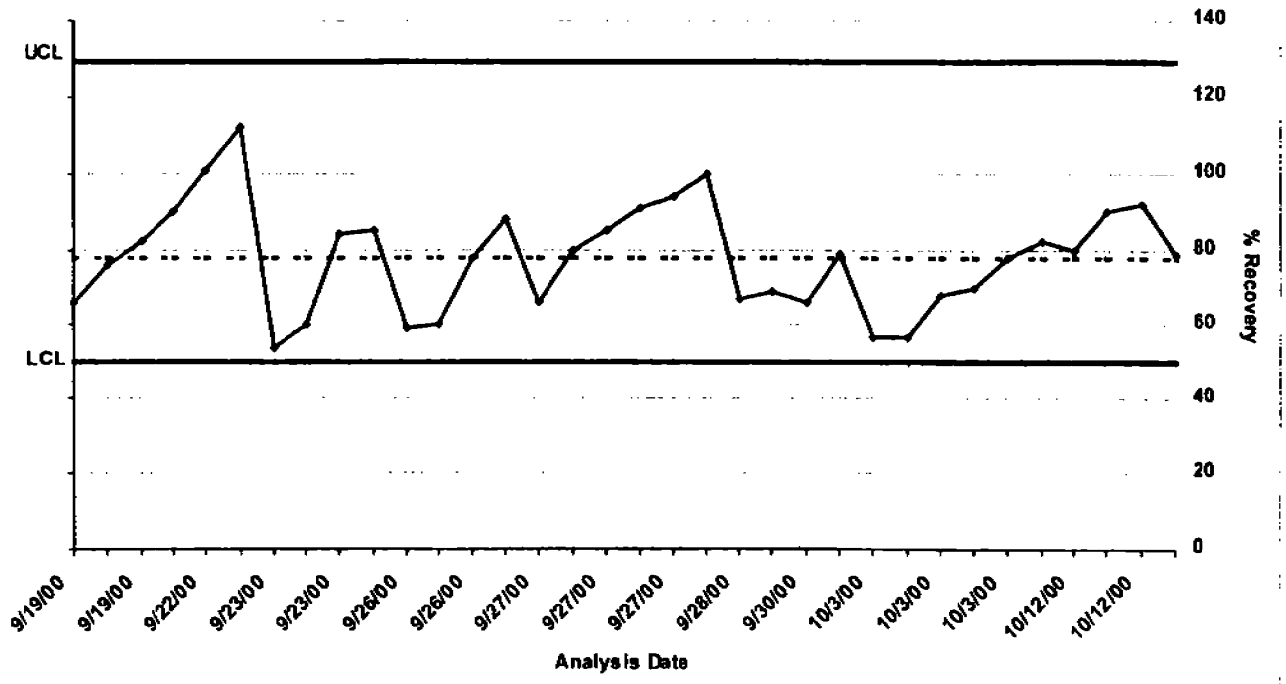
QuantIMS Spike List: 20600 (SAC: GCSEMI 8015MOD-TPH LCS)

Laboratory Control Sample
Aqueous

Analysis Dates: 09/19/2000 - 10/13/2000

Method	Method Description	Extraction
8015 MOD	Hydrocarbons, Extractable Petroleum (8015 MOD)	Not specified

Quality Control Chart



Statistical Calculations

N	36
Mean %R:	77.889
Std. Dev.:	13.761
Spike Level:	300
Spike Units:	ug/L
QIMS LCL	50
QIMS UCL	129
QIMS RPD	23

Calculated Limits

CALC LWL	50
CALC UWL	105
CALC LCL	37
CALC UCL	119
Calc RPD	42.79

Rejected data points are not charted

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Gasoline in Soil	Unleaded Gasoline	mg/Kg	447.0000	644	D.L. - 1290	Acceptable	AK101
	Benzene	mg/Kg	1.8800	7.08	D.L. - 7.08	Acceptable	AK101
	Ethylbenzene	mg/Kg	11.4000	18.0	4.03 - 20.2	Acceptable	AK101
	Toluene	mg/Kg	29.4000	62.3	D.L. - 69.2	Acceptable	AK101
	Xylenes, total	mg/Kg	62.2000	61.1	20.2 - 113	Acceptable	AK101
Diesel in Soil	No. 2 Diesel	mg/Kg	536	602	D.L. - 951	Acceptable	AK102
TPH in Soil	TPH (gravimetric)	mg/Kg		2250	D.L. - 6990		
	TPH (IR)	mg/Kg		2700	D.L. - 5310		
BTEX in Soil	Benzene	µg/Kg	385.0000	345	204 - 480	Acceptable	8021B
	Ethylbenzene	µg/Kg	60.5000	54.5	22.6 - 84.4	Acceptable	8021B
	Toluene	µg/Kg	173.0000	154	85.5 - 227	Acceptable	8021B
	Xylenes, total	µg/Kg	321.0000	283	113 - 467	Acceptable	8021B
MTBE in Soil	t-Butylmethylether (MTBE)	µg/Kg		32.1	19.5 - 52.1		
Gasoline in Water	Unleaded Gasoline	µg/l	4380.0000	3540	1400 - 7160	Acceptable	AK101
	Benzene	µg/l	34.5000	39.3	13.0 - 61.2	Acceptable	AK101
	Ethylbenzene	µg/l	83.2000	91.3	65.8 - 102	Acceptable	AK101
	Toluene	µg/l	275.0000	274	99.9 - 494	Acceptable	AK101
	Xylenes, total	µg/l	433.0000	354	199 - 727	Acceptable	AK101
Diesel in Water	No. 2 Diesel	µg/l	3320	3440	D.L. - 5800	Acceptable	AK102
TPH in Water	TPH (gravimetric)	mg/bttl		54.3	D.L. - 84.7		
	TPH (IR)	mg/bttl		65.2	9.30 - 125		
BTEX in Water	Benzene	µg/l	51.8000	58.6	33.4 - 81.8	Acceptable	8021B
	Ethylbenzene	µg/l	119.0000	142	83.3 - 197	Acceptable	8021B
	Toluene	µg/l	64.3000	73.9	43.2 - 103	Acceptable	8021B
	Xylenes, total	µg/l	97.9000	112	70.8 - 151	Acceptable	8021B
MTBE in Water	t-Butylmethylether (MTBE)	µg/l		62.1	41.6 - 93.0		

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	Benzene	mg/Kg	1.8800	7.08	D.L. - 7.08	Acceptable	CA LUFT
	Ethylbenzene	mg/Kg	11.4000	18.0	4.03 - 20.2	Acceptable	CA LUFT
	Toluene	mg/Kg	29.4000	62.3	D.L. - 69.2	Acceptable	CA LUFT
	Xylenes, total	mg/Kg	62.2000	61.1	20.2 - 113	Acceptable	CA LUFT
Diesel in Soil	No. 2 Diesel	mg/Kg	562	602	D.L. - 951	Acceptable	CA LUFT
TPH in Soil	TPH (gravimetric)	mg/Kg		2250	D.L. - 6990		
	TPH (IR)	mg/Kg		2700	D.L. - 5310		
BTEX in Soil	Benzene	µg/Kg		345	204 - 480		
	Ethylbenzene	µg/Kg		54.5	22.6 - 84.4		
	Toluene	µg/Kg		154	85.5 - 227		
	Xylenes, total	µg/Kg		283	113 - 467		
MTBE in Soil	t-Butylmethylether (MTBE)	µg/Kg		32.1	19.5 - 52.1		
Gasoline in Water	Unleaded Gasoline	µg/l	5170.0000	3540	1400 - 7160	Acceptable	CA LUFT
	Benzene	µg/l	34.5000	39.3	13.0 - 61.2	Acceptable	CA LUFT
	Ethylbenzene	µg/l	83.2000	91.3	65.8 - 102	Acceptable	CA LUFT
	Toluene	µg/l	275.0000	274	99.9 - 494	Acceptable	CA LUFT
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Diesel in Water	No. 2 Diesel	µg/l	3320	3440	D.L. - 5800	Acceptable	CA LUFT
TPH in Water	TPH (gravimetric)	mg/bttl		54.3	D.L. - 84.7		
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BTEX in Water	Benzene	µg/l		58.6	33.4 - 81.8		
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