DOE/PC/9 4251--TI-App.

RECEIVED

JUN 11 1996

OSTI

Advanced Emissions Control Development Project

Phase I Final Report Appendices

for the period: November 1, 1993 to February 29, 1996

Submitted: February 29, 1996

RDD:96:43195-005-000:01

RECEIVED
USDOE/PETC
36 APR 22 AM 8:44
ACQUISITION & ASSISTANCE DIV.

Babcock & Wilcox Alliance Research Center
1562 Beeson Street
Alliance, Ohio 44601-219

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

OCDO Grant Agreement: CDO/D-922-13
U.S. DOE - PETC Contract: DE-FC22-94PC94251
Babcock & Wilcox Contract: CRD-1310

George A. Farthing
Project Manager
Phone: 216/829-7494

CLEARED BY
PATENT COUNSEL

MASTER

DISTRIBUTION OF THIS DOCUMENT IS UNLIMITED

Babcock & Wilcox assumes no liability with respect to the use of, or for damages resulting from the use of, or makes any warranty or representation regarding any information, apparatus, method, or process disclosed in this report.

Babcock & Wilcox expressly excludes any and all warranties either expressed or implied, which might arise under law or custom or trade, including without limitation, warranties of merchantability and of fitness for specified or intended purpose.

TABLE OF CONTENTS

APPENDIX A

Wet Scrubber Sampling and Analysis Schedules
DBA/Lime Chemical Analysis
Limestone Forced Oxidation Chemical Analysis

APPENDIX B

Air Toxic Benchmarking Baghouse Conditions
Air Toxic Benchmarking ESP Conditions
Air Toxic Benchmarking Wet Scrubber Conditions

APPENDIX C

Quality Assurance Results

APPENDIX A

Wet Scrubber Sampling and Analysis Schedules
DBA/Lime Chemical Analysis
Limestone Forced Oxidation Chemical Analysis

	DBA	Makeup Water	Lime Slurry	Recirculation Slurry	Hydroclone Overflow	Hydroclone Underflow	Settling Bin	MEW Tank	CRW Tank
Total Sulfate				1/day					
Total Sulfite				1/day					
Total Carbonate				1/day					
Total Calcium			1/batch	1/day					
Total Magnesium				1/day					
Total Solids	1/batch		1/batch	1/day			1/day	1/day	1/day
Suspended Solids				1/day	1/day	1/day	1/day	1/day	1/day
Specific Gravity				1/day					
pH				1/day					
Solids Size Distribution				1/ week					
Dissolved Species									
DBA				1/ 8 hours				1/day	1/day
Ca, Mg		1/series		1/ 8 hours					
Na, K, Fe, Mn, Al		1/series		1/series					
Cl		1/series		1/ 8 hours				1/day	1/day
SO3				1/ 8 hours					

Sampling Matrix and Analysis Schedule - DBA/Lime Tests

	Makeup Water	Lime Slurry	Recirculation Slurry	Hydroclone Overflow	Hydroclone Underflow	Settling Bin	MEW Tank	CRW Tank
Total Sulfate			SS/daily					
Total Sulfite			SS/daily					
Total Carbonate			SS/daily					
Total Calcium		1/batch	SS/daily					
Total Magnesium		1/batch	SS/daily					
Total Solids		1/batch	SS/daily			1/day	1/day	1/day
Suspended Solids			SS/daily	1/day	1/day	1/day	1/day	1/day
Specific Gravity			SS/daily					
pH			SS/daily					
Reactivity		1/batch						
Solids Size Distribution			1/ week					
Dissolved Species								
Ca, Mg	1/series		1/ 8 hours					
Na, K, Fe, Mn, Al	1/series		1/series					
Cl	1/series		1/ 8 hours				1/day	1/day
SO3			1/ 8 hours					

Sampling Matrix and Analysis Schedule - Limestone Forced Oxidation

AECDP March DBA-Lime Tests

Date		Time	Total					Total		Susp	Spec	Solids		Dissolved											
Ca	Ca(OH)	CO3	SO3	SO4	MgO	Ca/S	Oxid	Solids	Solids	Grav	Dist	DBA	Ca	Mg	Na	K	Fe	Mn	Al	Cl	S	SO3			
mmole/l	wt%,d	mmole/l	mmole/l	mmole/l	%, dry	molar	%	%	%	g/g	MMu	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm		
DBA																									
30195	930							36.95																	
Makeup H2O																									
31795	320												0.67	17	33.6	4.31	0.27	0.1	0.23	45	0.43				
Lime Slurry																									
31795	320	98.81						17.53																	
Recirc Slurry																									
31695	137								0.94	1.0069															
31695	620									1.0034													65		
31695	700									1.0054													46		
31695	1000							3.09	2.35	1.0122		1594											168		
31695	1400							5.41	4.39	1.0250		1319													
31695	1630									1.0331		1826													
31695	1800								9.21	1.0410		2230													
31695	2000																						505		
31695	2300								9.38																
31695	2330							9.38	8.62	1.0509		2035								992					
31795	130									1.0324															
31795	320	337	4.2	8.1	372	1.042	99.8	6.60	5.19	1.0313		2362								1595			567		
31795	600									1.0400															
31795	900							9.18	7.31	1.0436		2233								1914			269		
31795	1300							10.73																	
31795	1400							11.71	10.53	1.0495		1951								2516			611		
31795	1600											2145													
31795	2030									1.0361															
31895	215											2243											56		
31895	415	540	2.1	19.5	587	2.26	1.036	99.6	10.18	8.47	1.0531	26.9	2140	1550	1450	78.4	99	0.19	1.07	2.91	1737	1520	493		
31895	900							12.74	10.58	1.0686		2520								2304			315		
31895	1300	509	2.1	20.8	573	1.040	99.6		8.65	1.0554		2913								2900			568		
Hydroclone Overflow																									
31795	1400							4.94	4.06																
Hydroclone Underflow																									
31795	1400							56.04	37.80																
Settling Bin																									
(1)	31795	900						60.49	59.14																
	31795	--						67.69	66.17																
	31895	--						69.28	66.26																
Mist Eliminator Water																									
31895	415											0								106					
Clarified Recycle Water																									
31795	900																								
31895	415											268								461					

(1) Both total & suspended solids are biased low due to sample taken from top of 55 gal drum, not 6" down.

AECDP March, 1995 LSFO Tests

c:\43195\lab1\labsum1 a.wk1

Date	Time	Total					Total			Susp	Spec	Solids Dissolved												Samples	
		Ca	CO3	SO3	SO4	MgO	Ca/S	Oxid	Solids	Solids	Grav	Dist	DBA	Ca	Mg	Na	K	Fe	Mn	Al	Cl	S	SO3		
		mmole/l	mmole/l	mmole/l	mmole/l	%, Dry	molar	%	%	%	g/g	MMu	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	ppm	tot	liq
Limestone Slurry	22495	---										20.5												"Solids"	
	32295	1245							15.30															X	
	32595	1100								16.52		22.8												X	
	33095	230							21.39	21.30	1.1497														

Recirc Slurry

32295	1115							2.49	2.46	1.0124																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																																								
-------	------	--	--	--	--	--	--	------	------	--------	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--	--

33095	2300	794	76.0	1.65	537		1.141	99.7	13.35											12796		0.54	X	X		
33195	215	973	19.0	2.90	611	2.13	1.310	99.5	14.67	10.88	1.0818			4930	1460	202	142	0.35	5.32	8.37	12139	753	2.10	X	X	
33195	815																							X		
33195	850												338												X	
33195	1000												344													
33195	1020												456												X	
33195	1100												670												X	
33195	1150												901												X	X
33195	1230												1398												X	X
33195	1250												1836	5340	1710	208	135	1.62	6.5	8.77		641			X	X
33195	1320												2565	5670	1660	205	133	1.59	8.67	8.77		660			X	X

Hydroclone Overflow

32395	600									7.99															
32395	2245																							X	
32395	1230								9.87	8.87															
32395	2250									9.88															
32595	1130									9.26															
33095	430									5.84														X	
33195	215									5.41															

Hydroclone Underflow

32395	2245																							X	
32395	1230								62.00	49.60															
32395	2250									66.77															
32595	1130									56.10															
33095	430									63.68														X	
33195	215									61.09															

Settling Bin

32395	----								71.79	69.07														X	
32495	----								74.19	70.10														X	
32595	----								76.71	71.64														X	
32995	----								82.78	78.54														X	
33095	----								72.55	68.95														X	
33195	----								73.57	66.08														X	

Mist Eliminator Water

32495	2300								0.01																	6877			X		
32595	930																									7125					
32595	2130																									7055			X		
32595	2240								0.01																						
32995	1400																									5388					
32995	2030								0.01																				X		
32995	2130																												X		
33095	1400																									5494					
33095	2300								0.01																						
33195	215								0.01																		5424			X	

Clarified Recycle Water

32395	2245							0.51	0.01									602				
32495	2300								0.01									11900			X	
32595	930																	12088				
32595	2130																	11982			X	
32595	2240								0.01													
32995	1400																	12266				
32995	2030								0.01												X	
32995	2130																				X	
33095	1400																	4714				
33095	2300								0.19													
33195	215								0.15									4502			X	

Other

Limestone (2-95)

Control Reactivity	0.229
Reactivity	0.293
CaO	50.4
MgO	1.4
CO2	44.2
SiO2	2.1
Al2O3	0.8
Fe2O3	0.2
TiO2	6.1

APPENDIX B

Air Toxic Benchmarking Baghouse Conditions

Air Toxic Benchmarking ESP Conditions

Air Toxic Benchmarking Wet Scrubber Conditions

Baghouse Operating Conditions During Air Toxic Benchmark Testing

Date	Test #	Gas Sample	Sample Time	Coal Flow lb/hr	Convection Pass Temp Deg F	CEDF Total Load MBTU/hr	BH Inlet Temp Deg F	BH Module Temp Deg F	BH Outlet Temp Deg F	BH Average Pressure Drop in H2O	Air-to- Cloth Ratio ft/min	Convection Pass O2 % (Dry)	BH Inlet O2 % (Dry)	BH Outlet O2 % (Dry)
6/26/1995	BENCH4	M26-1a*	1520-1644	7,849	816.3	100.0	345.3	321.9	286.7	5.16	3.94	3.36	3.44	4.45
6/26/1995	BENCH5	M29-1*	2005-0004	7,814	837.9	99.6	339.5	315.0	280.0	4.87	3.64	3.73	4.23	5.79
6/27/95	BENCH 6	M29-2*	203-603	7,827	844.3	99.7	344.0	317.7	283.0	4.79	3.71	3.71	4.40	5.63
6/27/1995	BENCH 7	M26-2a*	1500-1620	7,839	841.5	99.9	348.7	331.0	286.4	4.39	3.67	3.50	4.09	5.61
6/27/1995	BENCH 8	M29-3*	1942-2359	7,855	855.9	100.1	355.6	336.3	289.8	4.56	3.70	3.61	4.40	5.84
6/28/1995	BENCH 9	M29-4	144-544	7,859	814.1	100.1	347.5	336.8	276.4	3.96	3.67	3.68	4.44	5.59
6/29/1995	BENCH 16	M26-1b	1815-1940	7,825	846.5	99.7	316.6	305.4	237.1	4.79	3.61	3.82	4.67	5.80
6/29/1995	BENCH 16B	M26-2b	2048-2208	7,704	810.8	98.2	320.6	311.7	248.7	4.35	3.63	3.76	4.28	5.43
6/30/1995	BENCH 16C	M29-5	2352-352	7,955	828.0	101.4	321.0	312.3	247.9	4.39	3.67	3.52	4.36	5.37
Average				7,836	832.8	99.9	337.6	320.9	270.7	4.58	3.69	3.63	4.26	5.50
Standard Deviation				65	16.2	0.8	14.4	11.4	20.2	0.36	0.10	0.15	0.35	0.43
% Standard Deviation				0.8%	1.9%	0.8%	4.3%	3.5%	7.5%	7.8%	2.7%	4.1%	8.1%	7.8%

* - indicates baghouse particulate bypass

ESP Operating Conditions During Air Toxic Benchmark Testing

Date	Test #	Gas Sample	Sample Time	Coal Flow lb/hr	Convection Pass Temp Deg F	CEDF Total Load MBTU/hr	ESP Inlet Temp Deg F	ESP Outlet Temp Deg F	ESP Outlet Opacity %	Specific Collection Area (SCA)	Convection Pass O2 % (Dry)	ESP Inlet O2 % (Dry)	ESP Outlet O2 % (Dry)
6/28/1995	BENCH 12	M26-1	1555-1735	7,765	830.5	99.0	357.3	341.3	1.72	277.1	3.52	3.64	4.15
6/28/1995	BENCH 13	M26-2	2023-2200	7,726	840.1	98.4	358.3	342.5	4.62	279.7	3.69	3.55	4.27
6/29/1995	BENCH14	M29-1	0034-435	7,941	823.3	101.2	355.2	340.6	1.77	270.4	3.40	3.75	4.44
6/29/1995	BENCH15	M29-2	1127-1527	7,787	833.8	99.2	362.3	346.3	1.73	273.5	3.42	3.72	4.37
Average				7,805	831.9	99.5	358.3	342.7	2.46	275.2	3.51	3.66	4.31
Standard Deviation				94	7.0	1.2	3.0	2.5	1.44	4.1	0.13	0.09	0.13
% Standard Deviation				1.2%	0.8%	1.2%	0.8%	0.7%	58.5%	1.5%	3.8%	2.5%	2.9%

b-2

Wet Scrubber Operating Conditions During Air Toxic Benchmark Testing

Date	Test #	Gas Sample	Sample Time	FGD Outlet O2 % (Dry)	FGD Inlet SO2 ppm	FGD Outlet SO2 ppm	SO2 Removal %	L/G Ratio gpm/kacfm	Tower Velocity fps	Slurry pH	Tray pH	Tray Pressure Drop in H2O	Total Tower Pressure Drop in H2O
6/28/1995	BENCH 12	M26-1	1555-1735	4.17	2,056	371.5	82.0	91.1	8.10	5.74	4.12	1.73	3.51
6/28/1995	BENCH 13	M26-2*	2023-2200	4.30	2,036	163.8	91.9	91.1	8.11	5.67	4.03	1.73	3.56
6/29/1995	BENCH14	M29-1	0034-435	4.55	1,992	268.0	86.4	91.1	8.10	5.76	4.09	1.74	3.63
6/29/1995	BENCH15	M29-2	1127-1527	4.47	1,981	318.7	83.8	90.7	8.11	5.73	3.70	1.85	3.74
Average				4.37	2,016	280	86.0	91.0	8.10	5.72	3.98	1.76	3.61
Standard Deviation				0.17	35	89	4.3	0.2	0.00	0.04	0.19	0.06	0.10
% Standard Deviation				3.9%	1.8%	31.6%	5.0%	0.2%	0.0%	0.7%	4.9%	3.4%	2.7%

* - water in the FGD outlet impingers contributed to high SO2 Removal

APPENDIX C

Quality Assurance Results

Appendix C: Quality Assurance

A detailed summary of the analytical effort required of the configuration 1 and configuration 2 benchmarking tests are presented in Tables 1 and 2.

Table 1 Air Toxics Analysis Summary - Baghouse Configuration

Sample Matrix	# Samples	Particulate Weight	Trace Metals Analysis	Acid Gases	Hg Species	#HGAAS Analyses	# GFAAS Analyses	# CVAAS Analyses	# IC Analyses	# ISE Analyse
M26A Solids	5	✓		✓					8	8
M26A Impingers	10			✓					13	13
M29 Solids	6	✓	✓			12	12	12		
M29 Impingers	42		✓		✓	14	14	53		
BH Ash	2		✓	✓		3	3	3		6
Coal	2		✓	✓		3	3	3		4

Number of analyses include blanks, spikes, and duplicates

Table 2 Air Toxics Analysis Summary - ESP/WET Scrubber Configuration

Sample Matrix	# Samples	Particulate Weight	Trace Metals Analysis	Acid Gases	Hg Species	#HGAAS Analyses	#GFAAS Analyses	#CVAAS Analyses	#IC Analyses	#ISE Analyse
M26A	6	✓		✓					9	9
M26A	6			✓					7	9
M29 Solids	6	✓	✓			9	9	9		
M29 Impingers	42		✓		✓	14	14	53		
ESP Ash	2		✓	✓		3	3	3		6
Coal	2		✓	✓		3	3	3		4
Limestone Slurry	2		✓	✓		4	4	4		2
WFGD Gypsum	2		✓	✓		4	4	4		4
WFGD	2		✓	✓		4	4	4		4

Number of analyses include blanks, spikes, and duplicates

Recalibration Rates - GFAAS, CVAAS, HGAAS, ICP

Check standards prepared from a source independent of the calibration standards must fall between 90 - 110% of the true value. When outside of this range, the check standard was repeated. If still outside of the acceptable range, the standard was prepared again and reanalyzed. When values are still unacceptable, the instrument was recalibrated until acceptable check standard values were obtained. If necessary, new calibration standards were prepared.

Spike Recoveries

Summaries of the analytical precision and accuracy results are provided in Tables 3 through Table 12. Analytical accuracies reflected by the percent recovery for spiked samples are well within the targeted range of 50 - 150%.

Table 3 Spike Recoveries for M26A Fluoride

Method	Sample ppm	Spike ppm	Detection ppm	% Recovery
H ₂ SO ₄ Impinger				
Fluoride, ISE	6.37	10	16.4	100
Fluoride, IC	6.37	10	16.02	98
NaOH Impinger				
Fluoride, ISE	1.29	0.5	1.80	101
Fluoride, IC	1.29	0.5	1.86	104

Table 4 Spike Recoveries for M26A Chloride

Method	Sample ppm	Spike ppm	Detection ppm	% Recovery
H ₂ SO ₄ Impinger				
Chloride, ISE	29.7	50	72	90
NaOH Impinger				
Chloride, ISE	28.3	20	50	97

The chloride spiked sample samples prepared for IC analysis were found to be contaminated.

Table 5 Spike Recoveries for M26A Filter Solids

Configuration	Sample ppm	Spike ppm	Detection ppm	% Recovery
Chloride				
Baghouse	34.6	20	58	106
ESP	64.7	20	100	96
Fluoride				
Baghouse	273	200	490	104
ESP	240	200	440	98

Table 6 Spike Recoveries for M29 H₂O₂-HNO₃ Impingers

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	< 1	---	---	---
Arsenic	17.3	20	35.5	98
Barium	35	50	75	88
Beryllium	2.0	2	3.3	83
Cadmium	12	10	20	91
Chromium	86	100	190	102
Cobalt	5.3	5	10	97
Lead	14	20	30	89
Nickel	35	50	80	94
Manganese	27	25	51	98
Mercury	95	100	190	97
Selenium	107	100	202	98

Table 7 Mercury Spike Recoveries M29 Impinger Solution

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Impingers 5 & 6: H ₂ SO ₄ -KMnO ₄				
Mercury	30	25	52	95
Impinger 4				
Mercury	1.0	1.0	1.8	90
8 N HCl Impinger Rinse				
Mercury	1.11	1	2.0	95

Table 8 Spike Recoveries for Coal Composite

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	3.05	2	5.1	101
Arsenic	4.52	5	10	105
Barium	111	100	200	95
Beryllium	14.5	20	30	87
Cadmium	1.01	1	1.8	90
Chromium	71.8	50	100	82
Cobalt	4.80	5	9.3	95
Lead	21.5	20	41	99
Nickel	40	505	890	99
Manganese	104	100	200	98
Mercury	4.805	5.2	9.399	95
Selenium	9.32	10	20	104

Table 9 Spike Recoveries for Limestone Slurry

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	1.19	1	1.9	86
Arsenic	32.8	25	56	97
Barium	9.62	10	19.0	96
Beryllium	< 0.25	---	---	---
Cadmium	0.56	0.5	1.01	95
Chromium	1.64	2	3.50	96
Cobalt	< 1	---	---	---
Lead	6.09	10	16.4	102
Nickel	5.55	5	10.4	98
Manganese	42.1	50	95	103
Mercury	< 0.1	---	---	---
Selenium	9.77	10	19.0	96

Table 10 Spike Recoveries for Wet Scrubber Filtrate

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	5.73	5	9.4	88
Arsenic	< 1.63	---	---	---
Barium	20	20	41	103
Beryllium	< 0.41	---	---	---
Cadmium	1.44	1	2.20	92
Chromium	5.91	500	10.8	99
Cobalt	3.62	5	7.9	92
Lead	2.89	50	7.0	89
Nickel	31.2	25	55	98
Manganese	6.38	55	12	105
Mercury	<0.01	---	---	---
Selenium	28.3	250	50.3	94

Table 11 Spike Recoveries for Wet Scrubber Gypsum Solids

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	2.29	2	4.1	96
Arsenic	3.19	5	7.9	96
Barium	47.3	50	97	100
Beryllium	< 0.25	---	---	---
Cadmium	0.76	1	1.50	85
Chromium	10.4	100	21.8	103
Cobalt	2.21	2	4.1	97
Lead	20.4	20	370	92
Nickel	7.89	10	15	84
Manganese	35.8	50	83	97
Mercury	0.351	0.5	0.79	93
Selenium	0.44	1	1.5	104

Table 12 Spike Recoveries for Wet Scrubber Filtrate

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	5.73	5	9.4	88
Arsenic	< 1.63	---	---	---
Barium	20.0	20	41	103
Beryllium	< 0.41	---	---	---
Cadmium	1.44	1	2.20	92
Chromium	5.91	5	10.8	99
Cobalt	3.62	5	7.9	92
Lead	2.89	5	7.0	89
Nickel	31.2	25	55	98
Manganese	6.38	5	12	105
Mercury	< 0.01	---	---	---
Selenium	28.3	25	50.3	94

Table 13 Spike Recoveries for Ash

Analyte	Sample ppb	Spike ppb	Detection ppb	% Recovery
Antimony	0.052	0.05	0.104	101
Arsenic	1.56	2	3.2	89
Barium	0.23	1	1.23	100
Beryllium	0.17	0.2	0.35	95
Cadmium	0.12	0.1	0.20	91
Chromium	0.83	1	1.7	93
Cobalt	0.27	0.25	0.50	96
Lead	0.25	0.25	0.48	96
Nickel	0.68	0.5	1.0	85
Manganese	1.18	1	1.9	86
Mercury	0.195	0.2	0.399	101
Selenium	0.022	0.02	0.41	98

Replicate Analysis

Analytical precision reflected by the relative percent deviation (RPD) for replicate samples was generally less than 25%. Replicate analysis of the limestone slurry proved to be the most difficult. The replicates are separate preparations of the same sample by acid digestion. Replicates do not apply to liquid samples requiring no digestion or requiring filtration only.

Table 14 Precision Results - Relative Percent Deviation of Replicate Samples

Analyte	Composite Coal	M29 Solids	Limestone Slurry	Gypsum Solids
Antimony	53.2	0.2	9.5	5.1
Arsenic	31.6	0	18.6	0.4
Barium	2.22	0.7	5.0	0.1
Beryllium	2.79	4.9	4.3	---
Cadmium	13.3	21.8	43.7	1.5
Chromium	16.9	2.3	22.2	1.1
Cobalt	10.1	5.7	10.5	5.4
Lead	16.8	4.8	65.9	1.1
Nickel	6.1	4.0	55.4	1.4
Manganese	5.7	0.8	4.3	0.3
Mercury	7.2	5.7	7.3	5.6
Selenium	25.1	20.7	68.2	2.4

Blank Results

Trace metal grade acids employed for the Reagent (Method) blanks were used to zero the instruments.

Table 15 Method 26 A Reagent (Method) Blanks

0.1 N H₂SO₄ Reagent, ppm					
Fluoride, ISE	0.06	Chloride, ISE	0.51	Bromide, ISE	0.27
Fluoride, IC	< 1	Chloride, IC	< 1	Bromide, IC	---
0.1 N NaOH Reagent, ppm					
Fluoride, ISE	0.04	Chloride, ISE	11.84	Bromide, ISE	26.8
Fluoride, IC	< 1	Chloride, IC	< 1	Bromide, IC	---
Deionized Water Blank, ppm					
Fluoride, ISE	0.05	Chloride, ISE	0.12	Bromide, ISE	0.21
Fluoride, IC	< 1	Chloride, IC	< 1	Bromide, IC	---

Chloride determinations in the NaOH impingers by ISE were not corrected for the blank value of 11.84 ppm since many of the impinger solutions had Cl concentrations less than 10 ppm. Get the IC blank value to make sure it was contaminated.

Table 15 Method 29 Blank Solution Analysis, ppb

Analyte	0.1N HNO3	H2O2-HNO3	Acetone Rinse	DI Water
Antimony	< 1	< 1	< 1	< 1
Arsenic	< 1	< 1	< 1	< 1
Barium	< 1	< 1	< 1	< 1
Beryllium	< 0.5	< 0.5	< 0.5	< 0.5
Cadmium	< 0.1	< 0.1	< 0.1	< 0.1
Chromium	< 1	< 1	< 1	< 1
Cobalt	< 1	< 1	< 1	< 1
Lead	2.5	4.5	< 1	3.5
Nickel	< 1	22	< 1	5.4
Manganese	< 1	< 1	< 1	< 1
Mercury	< 1	< 1	< 1	< 1
Selenium	< 1	< 1	< 1	< 1

Table 16 Blank Whatman Glass Filter, $\mu\text{g}/\text{filter}$

Analyte	Lot 002945	Lot 647371A	Whatman Analysis ppm
Antimony	0.52	0.60	---
Arsenic	0.024	0.73	---
Barium	82	82	50
Beryllium	< 0.06	< 0.06	0.1
Cadmium	0.086	0.074	---
Chromium	36	33	0.1
Cobalt	3.2	2.6	1
Lead	4.85	4.9	< 0.1
Nickel	52	39	5
Manganese	21	22	1
Mercury	---	---	< 0.1
Selenium	0.044	0.028	< 0.1