

SOURCE SAMPLING FIELD DATA SHEET

Plant Name Plant Yates Station Boiler No. 1
 Sampling Location Inlet Train Particulate / Metals Run No. 3/Phase 2
 Date 6/27/93 Time Start _____ Time Finish _____ Test Duration _____ min.
 Duct Dimensions _____ X _____ Diameter _____ ft Initial Leak Rate _____ cfm
 PTCF _____ DGMCF _____ NOZZLE DIA. _____ inches Final Leak Rate _____ cfm
 Bar Press _____ " Hg
 Static Press _____ " H2O Operator SWN

Travers Point	Clock Time	Dry gas meter reading ft3	^P in H2O	^H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K Factor
						Inlet	Outlet					
W2-1	1049	752.642	0.02	0.22	285	87	84	-	204	60	4	
2	1054	754.1	0.03	0.33	298	87	84	-	221	55	5	
3	1059	753.7	0.04	0.44	307	88	85	-	243	55	6	
4	1104	757.7	0.09	0.99	299	89	86	-	257	49	11	11.06
5	1109	760.2	0.14	1.55	302	91	87	-	241	47	19	
6	121114	763.9	0.15	1.7	301	94	88	-		58	22	
stop	1119	767.251	Good at 22" Hg leak check		Good initial leak check @ 25" Hg							
E7-1	1148	768.450	0.03	0.33	317	89	87	-	216	65	6	11.12 10.88
2	1153	770.2	0.04	0.41	322	89	87	-	240	55	7	
3	1158	772.0	0.06	0.66	328	90	87	-	242	52	8	
4	1203	774.3	0.09	1.0	330	91	88	-	244	57	12	
5	1208	777.0	0.18	1.6	333	93	89	-	242	54	22	
6	1213	780.6	0.19	1.6	336	95	90	-	249	57	22	
stop	1218	783.812	Good leak check @ 22" Hg (0.018 A3/min)									
S-1	1225	784.027	0.02	0.22	316	92	90	-	222	67	5	
2	1230	785.6	0.04	0.45	322	92	89	-	247	60	7	
3	1235	787.4	0.07	0.77	321	92	90	-	248	57	11	
4	1240	790.0	0.11	1.25	321	92	91	-	250	58	18	
5	1245	792.5	0.14	1.55	322	96	92	-	251	59	21	
6	1250	796.6	0.16	1.4	322	97	92	-	245	63	22	
top	1255	799.342										
SOP												
		111.690	0.2525									
ok'd												

SOLE # _____
 ER # _____
 IENT TEMP. _____
 3E LENGTH _____
 R MATERIAL _____

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

ARKS

SOURCE SAMPLING FIELD DATA SHEET

Plant Name Plant Yates Station Boiler No. 1
 Sampling Location Inlet Train Particulate / Metals Run No. 3 / Phase 2
 Date 6/27/93 Time Start _____ Time Finish _____ Test Duration _____ min.
 Duct Dimensions _____ X _____ Diameter _____ ft Initial Leak Rate _____ cfm
 PTCF _____ DGMCF _____ NOZZLE DIA. _____ inches Final Leak Rate _____ cfm
 Bar Press _____ " Hg
 Static Press _____ " H2O Operator JWM

Travers Point	Clock Time	Dry gas meter reading ft3	ΔP in H2O	ΔH in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg
						Inlet	Outlet				
E3-1	1259	799.545	0.02	0.22	307	94	92	-	206	66	6
2	1304	801.1	0.03	0.33	315	94	92	-	226	65	7
3	1309	802.7	0.05	0.35	318	95	92	-	249	65	10
4	1314	804.8	0.09	1.2	316	96	93	-	247	55	15
5	1319	807.3	0.11	1.5	316	99	94	-	245	54	20
6	1324	810.5	0.14	1.3	314	99	94	-	242	56	22
Stop	1329	813.735	Good leak check @ 22" Hg								
E1-1	1335	814.000	0.07	0.77	309	97	94	-	205	58	13
2	1338	816.5	0.09	1.0	314	97	94	-	239	54	17
3	1345	819.4	0.04	0.44	312	97	93	-	241	54	11
4	1358	821.5	0.03	0.33	310	98	94	-	260	56	9
5	1355	823.3	0.04	0.44	309	97	94	-	252	56	10
6	1400	825.2	0.05	0.55	303	97	94	-	257	57	11
Stop	1405	827.278									
Avg.	-										
Check'd											

14.190

1.278

CONSOLE # _____
 FILTER # _____
 AMBIENT TEMP. _____
 PROBE LENGTH _____
 LINER MATERIAL _____

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

Flue-Gas Sampling Log

Sponsor: Plant Yates Station Belle Meade	Sample Run #: 1
Plant Location: ESP INLET	Soda-Lime Trap#: SL 404
Date: 06-25-93	Iodated Carbon #: 16 404
Fuel Type: COAL	Pump#: Box # 1
Pollution Control: ESP	Probe#: Z
Sampling Point: ESP RW 1	Filter ID:

time (hh:mm)	start		stop		elapsed time (min)	mean zero (l/min)	mean flow (l/min)
	zero (l/min)	flow (l/min)	time (hh:mm)	zero (l/min)			
07:05	-0.012	0.500	1:00	-0.005	246 235	-0.0085	0.3865
TOTALS:					235	-0.0085	0.3865

Integrator Volume (l):	100.0
Offset Correction (l):	
Total Integrator Volume:	
CO ₂ Mass Flow Correction:	
Actual (dry STP) volume (l):	
% O ₂ :	8.0
% CO ₂ :	10.0
% H ₂ O:	7.0
ppm SO ₂ :	1500 To 2000

COMMENTS:
START: LEAK V @ METER → -0.012
STOP: LEAK V @ METER → -0.005
HEAT SHEATHED PROBE TEMP 105°C TO 115°C

Flue-Gas Sampling Log

Sponsor:	YATES STATION Coal #1	Sample Run #:	Z
Plant Location:	ESP INLET	Soda-Lime Trap#:	SL-421
Date:	06-26-73	Iodated Carbon #:	IC-421
Fuel Type:	COAL	Pump#:	Box #1
Pollution Control:	ESP	Probe#:	#2
Sampling Point:	W-1	Filter ID:	

time (hh:mm)	start		stop		elapsed time (min)	mean zero (l/min)	mean flow (l/min)
	zero (l/min)	flow (l/min)	time (hh:mm)	zero (l/min)			
1045	-0.003	0.500	22:51Z	-0.006	267		
TOTALS:							

Integrator Volume (l):	100.0
Offset Correction (l):	
Total Integrator Volume:	
CO ₂ Mass Flow Correction:	
Actual (dry STP) volume (l):	
% O ₂ :	8.0
% CO ₂ :	10.0
% H ₂ O:	7.0
ppm SO ₂ :	1500 To 2000

COMMENTS:
START LEAK ✓ @ METER -0.006
STOP LEAK ✓ @ METER -0.008
HEAT SHEATHED PROBE TEMP
105°C To 115°C

Flue-Gas Sampling Log

Sponsor:	Yareb Station Boiler #1	Sample Run #:	3
Plant Location:	ESP INLET	Soda-Lime Trap#:	SL 410
Date:	06-21-95	Iodated Carbon #:	IC-410
Fuel Type:	COAL	Pump#:	Box #1
Pollution Control:	ESP	Probe#:	#2
Sampling Point:	W-1	Filter ID:	

time (hh:mm)	start		time (hh:mm)	stop		elapsed time (min)	mean zero (l/min)	mean flow (l/min)
	zero (l/min)	flow (l/min)		zero (l/min)	flow (l/min)			
07:15	-0.006	0.500	11:45	0.265 RUV	0.265	2:70	0.38 RUV	0.3825
TOTALS:								

Integrator Volume (l):	100.0
Offset Correction (l):	
Total Integrator Volume:	
CO ₂ Mass Flow Correction:	
Actual (dry STP) volume (l):	
% O ₂ :	8.0
% CO ₂ :	10.0
% H ₂ O:	7.0
ppm SO ₂ :	1500 to 2000

COMMENTS:
START LEAK ✓ METER - 0.006
STOP LEAK ✓ METER -- 0.008
HEAT SHEATH PROBE TEMP
105 °C TO 115 °C

Flue-Gas Sampling Log

Sponsor: YATES	Sample Run #: FIELD BLANK
Plant Location: ESP INLET	Soda-Lime Trap#: 406
Date: 06.24.93	Iodated Carbon #: 406
Fuel Type: COAL	Pump#: Box # 1
Pollution Control: ESP	Probe#: 2
Sampling Point:	Filter ID:

time (hh:mm)	start		time (hh:mm)	stop		elapsed time (min)	mean zero (l/min)	mean flow (l/min)
	zero (l/min)	flow (l/min)		zero (l/min)	flow (l/min)			
1700	-0.011	0.50	1210	-0.011	0.500			
TOTALS:								

Integrator Volume (l):	0
Offset Correction (l):	
Total Integrator Volume:	
CO ₂ Mass Flow Correction:	
Actual (dry STP) volume (l):	
% O ₂ :	8.0
% CO ₂ :	10.0
% H ₂ O:	7.0
ppm SO ₂ :	1500 ~ 2000

COMMENTS:
LEAK V @ METER → -0.011
HEAT SHEATHED PROBE TEMP 105 °C

SOURCE SAMPLING FIELD DATA SHEET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet

Train Anions

Run No. 1

Date 6-25-93 Time Start 1225

Time Finish 1405

Test Duration 100 min

Duct Dimensions 8 1/4" X 45"

Diameter _____ ft

Initial Leak Rate 0.004 cm

0.01 @ 10"

PTCF 84 DGMCF 1.003

Nozzle Dia. .375 inches

Final Leak Rate 0.004 cm

8"

Bar Press 29.55 " Hg

Static Press -5.8 " H2O

Operator MRK

PO4W-3

Travers Point	Clock Time	Dry gas meter reading ft3	Δ P in H2O	Δ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K=15.6
						Inlet	Outlet					
UA	1225	383.897										
	1245	397.46	.11	1.49	280	79	78		258	64	3.0	
	1305	410.73	.10	1.36	290	87	81		263	57	3.0	
	1325	423.28	.10	1.36	291	87	81		260	55	3.5	
	1345	435.99	.09	1.22	280	93	87		258	53	3.5	
	1405	448.71	.10	1.36	290	93	87		260	52	5.0	
Avg.	-	418.16	.1316	1.3580	290		85					
Check'd												

CONSOLE # A 161404
 FILTER # 1229
 AMBIENT TEMP. 78
 PROBE LENGTH 10'
 LINER MATERIAL glass

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1 ANIONS
 Sampling Location INLET Train Bulk Particulate Radionuclides Run No. 7
 Date 6-26-93 Time Start 1108 Time Finish 1213 Test Duration 65 min.
 Duct Dimensions 8'6" X 45 57 Diameter _____ ft Initial Leak Rate 0.004 cfm @ 10"
 PTCF 284 DGMCF 1.003 Nozzle Dia. 1.375 inches Final Leak Rate 0.009 cfm
 Bar Press 29.50 " Hg
 Static Press 5.8 " H2O Operator MKO at 84

Travers Point	Clock Time	Dry gas meter reading ft3	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K-13.9
						Inlet	Outlet					
<u>M/A</u>	<u>1208</u>	<u>550.080</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>	<u>—</u>
	<u>1128</u>	<u>562.70</u>	<u>1.10</u>	<u>1.38</u>	<u>27</u>	<u>84</u>	<u>83</u>		<u>245</u>	<u>62</u>	<u>3.0</u>	
	<u>1148</u>	<u>575.72</u>	<u>1.10</u>	<u>1.38</u>	<u>27</u>	<u>84</u>	<u>84</u>		<u>254</u>	<u>54</u>	<u>3.0</u>	
	<u>1208</u>	<u>589.54</u>	<u>1.11</u>	<u>1.51</u>	<u>28</u>	<u>84</u>	<u>87</u>		<u>257</u>	<u>54</u>	<u>3.5</u>	
	<u>1213</u>	<u>594.327</u>	<u>1.10</u>	<u>1.37</u>	<u>28</u>	<u>85</u>	<u>87</u>		<u>258</u>	<u>55</u>	<u>3.5</u>	<u>13.5</u> k
Avg.	—	<u>44.745</u>	<u>0.320</u>	<u>1.405</u>	<u>28.5</u>	<u>88.0</u>						
Check'd												

CONSOLE # A161.401
 THIMBLE # #1232 ✓
 AMBIENT TEMP. 74
 PROBE LENGTH 10
 LINER MATERIAL 9/16"

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

Page 1 of

Plant Name Plant Yates Station Boiler No. 1

Sampling Location INLET

Train Anions

Run No. 3

Date 6-27-93 Time Start 0715

Time Finish 0837

Test Duration 92 min.

Duct Dimensions 8'6" x 45'

Diameter ft

Initial Leak Rate 0.009 cfm at 12"

PTCF 844 DGMCF 1.003

Nozzle Dia. .375 inches

Final Leak Rate 0.006 cfm

Bar Press 29.50 " Hg

Static Press -5.9 " H₂O

Operator MKO

Port E-5 at 9"

Travers Point	Clock Time	Dry gas meter reading ft ³	Δ P in H ₂ O	Δ H in H ₂ O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K=12.8
						Inlet	Outlet					
<u>N/A</u>	<u>0715</u>	<u>655.883</u>										
	<u>0735</u>	<u>666.72</u>	<u>.08</u>	<u>1.02</u>	<u>310</u>	<u>72</u>	<u>71</u>		<u>256</u>	<u>58</u>	<u>2.0</u>	
	<u>0755</u>	<u>678.17</u>	<u>.08</u>	<u>1.02</u>	<u>308</u>	<u>73</u>	<u>71</u>		<u>268</u>	<u>53</u>	<u>2.5</u>	
	<u>0815</u>	<u>689.30</u>	<u>.08</u>	<u>1.02</u>	<u>310</u>	<u>83</u>	<u>76</u>		<u>261</u>	<u>56</u>	<u>2.5</u>	
	<u>0837</u>	<u>701.023</u>	<u>.07</u>	<u>1.89</u>	<u>311</u>	<u>84</u>	<u>76</u>		<u>258</u>	<u>56</u>	<u>2.5</u>	
Avg.	<u>-</u>	<u>45.140</u>	<u>.2783</u>	<u>.9875</u>	<u>310</u>		<u>76</u>					
Check'd												

CONSOLE # A161401

FILTER #

AMBIENT TEMP. 74

PROBE LENGTH 10'

LINER MATERIAL G/ASJ

Velocity
 % Moisture
 Flowrate (DSCFM)
 Isokinetic (%)

REMARKS

SOURCE SAMPLING FIELD DATA SHEET

Plant Name Plant Yates Station Boiler No. 1

Sampling Location SFT ESP Inlet Train Ammonia/Hydrogen Cyanide Run No. 1

Date 6-25-93 Time Start 1450 Time Finish 1650 Test Duration 70 min

Duct Dimensions 86" X 45 Diameter _____ ft Initial Leak Rate 0.010 at 10" cfm

PTCF .84 DG MCF 1.003 Nozzle Dia. .375 inches Final Leak Rate 0.009 at 12" cfm

Bar Press 29.55 " Hg

Static Press -5.8 " H2O

Operator MKO

Part 4-3

Travers Point	Clock Time	Dry gas meter reading ft ³	^ P. in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg
						Inlet	Outlet				
1450	1450	451.30									
1450	1450	450.134									
	1500	464.63	1.0	1.36	285	88	86		250	61	3
	1530	477.49	1.0	1.36	291	88	86		257	63	3
	STOP	leak check 0.006 at 12" put back particulate filter on because thick in H2O was losing particulate									
		CAME OFF TO TRAP LINE. Tip of probe broke fine polished the end									
		leak check 0.009 at 12"									
	1620	477.89									
	1640	490.34	1.07	1.72	287	89	86		253	62	3.5
	1650	497.14	1.0	1.36	291	89	86		252	58	4.5
		46.663									
Avg.	-	46.683	1.222	1.320	289		88				
Check'd											

CONSOLE # A161401
 FILTER # _____
 AMBIENT TEMP. 80
 PROBE LENGTH 10' 9/16"
 LINER MATERIAL 9/16"

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

Plant Name Plant Yates Station Boiler No. 1

Sampling Location INLET Train Ammonia/Hydrogen Cyanide Run No. 2

Date 6-26-93 Time Start 0930 Time Finish 1035 Test Duration 65 min.

Duct Dimensions 86" X 45' Diameter _____ ft Initial Leak Rate 0.009 cfm at 10"

PTCF .84 DGMCF 1.003 Nozzle Dia. .375 inches Final Leak Rate 0.006 cfm at 8"

Bar Press 29.56 " Hg

Static Press -5.8 " H2O

Operator MKO

Part W-4

Travers Point	Clock Time	Dry gas meter reading ft ³	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	13.8 -k
						Inlet	Outlet					
	0930	507.503										
	0950	522.62	.10	1.38	280	75	74		260	67	4.5	
	1010	533.67	.10	1.38	281	83	77		255	52	4.5	
	1030	546.65	.10	1.38	285	80	77		258	54	4.5	
	1035	549.18	.09	1.24	285	88	81		254	54	4.5	
Avg. -												
Check'd												

CONSOLE # A161401
 FILTER # -
 AMBIENT TEMP. 70
 PROBE LENGTH 10'
 LINER MATERIAL 9/1A22

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

*Sue Day
as Runz
Jun*

Plant Name Plant Yates Station Boiler No. 1

Sampling Location INLET Train Ammonia/Hydrogen Cyanide Run No. 3

Date 6-26-97 Time Start 1420 Time Finish 1520 Test Duration 60 min.

Duct Dimensions 8'6" X 45 Diameter ft Initial Leak Rate 0.009 cfm at 10"

PTCF 84 DGMCF 1.003 Nozzle Dia. 1.375 inches Final Leak Rate 0.006 cfm

Bar Press 29.85 " Hg

Static Press -5.8 " H2O

Operator MKO

Part w/ Lot 12"

Travers Point	Clock Time	Dry gas meter reading ft ³	Δ P in H ₂ O	Δ H in H ₂ O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg
						Inlet	Outlet				
<i>N/A</i>	<i>1420</i>	<i>5953.10</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>—</i>	<i>13.9</i>
	<i>1440</i>	<i>610.13</i>	<i>.11</i>	<i>1.48</i>	<i>281</i>	<i>96</i>	<i>93</i>		<i>266.68</i>		<i>5.0</i>
	<i>1500</i>	<i>622.85</i>	<i>.09</i>	<i>1.21</i>	<i>289</i>	<i>95</i>	<i>92</i>		<i>266.63</i>		<i>5.5</i>
	<i>1520</i>	<i>636.44</i>	<i>.10</i>	<i>1.35</i>	<i>286</i>	<i>95</i>	<i>92</i>		<i>266.62</i>		<i>5.5</i>
Avg.	—	<i>411.654</i>	<i>0.077</i>	<i>1.34</i>	<i>284</i>		<i>94</i>				
Check'd											

CONSOLE # A161401
 FILTER #
 AMBIENT TEMP. 70
 PROBE LENGTH 10'
 LINER MATERIAL 51855

Velocity
 % Moisture
 Flowrate (DSCFM)
 Isokinetic (%)

REMARKS

SOURCE SAMPLING FIELD DATA SHEET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1
 Sampling Location INLET Train Ammonia/Hydrogen Cyanide Run No. 4
 Date 6-21-93 Time Start 0920 Time Finish 1040 Test Duration 80 min.
 Duct Dimensions 8'6" X 45" Diameter _____ ft Initial Leak Rate 0.006 cfm
 PTCF 1.24 DGMCF 1.003 Nozzle Dia. 3.15 inches Final Leak Rate 0.004 cfm
 Bar Press 29.40 " Hg Operator MEI E-5 part
 Static Press -5.8 " H2O

Travers Point	Clock Time	Dry gas meter reading ft3	^ P in-H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K=1.8
						Inlet	Outlet					
	0940	701.417										
	0940	713.02	.08	1.02	313	82	81		260	66	5.0	
	1000	725.17	.09	1.15	315	83	81		259	59	5.0	
	1020	736.62	.08	1.02	315	85	82		257	60	5.0	
	1040	748.30	.08	1.02	316	86	81		257	61	5.0	
Avg.	-	46.825	0.0871	1.0850	315		85					
Check'd												

CONSOLE # A161401
 FILTER # _____
 AMBIENT TEMP. 76
 PROBE LENGTH 10'
 LINER MATERIAL GLASS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet Train Bulk Particulate-Radionuclides Run No. 1
 Date 6-25-93 Time Start 0745 Time Finish 0907 Test Duration 02 min.
 Duct Dimensions 8'6" X 45 Diameter _____ ft Initial Leak Rate 0.009 cfm
 PTCF .84 DGMCF 1.009 Nozzle Dia. .375 inches Final Leak Rate 0.006 cfm
 Bar Press 29.55 " Hg
 Static Press -6.4 " H2O Operator MKL Part E-4

Travers Point	Clock Time	Dry gas meter reading ft3	ΔP in H2O	ΔH in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K=13.5
						Inlet	Outlet					
<u>N/A</u>	<u>0745</u>	<u>656.235</u>	<u>0.08</u>	<u>1.08</u>								
	<u>0805</u>	<u>667.72</u>	<u>0.08</u>	<u>1.08</u>	<u>300</u>	<u>78</u>	<u>77</u>		<u>250</u>	<u>61</u>	<u>3.0</u>	
	<u>0825</u>	<u>679.80</u>	<u>.10</u>	<u>1.35</u>	<u>300</u>	<u>84</u>	<u>79</u>		<u>247</u>	<u>54</u>	<u>3.0</u>	
	<u>0845</u>	<u>694.05</u>	<u>.13</u>	<u>1.75</u>	<u>300</u>	<u>85</u>	<u>80</u>		<u>243</u>	<u>56</u>	<u>4.0</u>	
	<u>0907</u>	<u>708.84</u>	<u>.13</u>	<u>1.75</u>	<u>301</u>	<u>80</u>	<u>83</u>		<u>244</u>	<u>69</u>	<u>4.0</u>	
<p>Same impinger train was used for Radionuclides and Bulk Part/Extractions</p> <p>JW</p>												
Avg.	<u>-2"</u>	<u>53.605</u>	<u>1.3300</u>	<u>1.4825</u>	<u>301</u>		<u>82</u>					
Check'd												

CONSOLE # A161407
 THIMBLE # -
 AMBIENT TEMP. 74
 PROBE LENGTH 10'
 LINER MATERIAL 9/ass

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

53.6

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page ____ of ____

Plant Name Plant Yates Station Boiler No. 1

Sampling Location 6-2-83 Train Bulk Particulate-Radionuclides Run No. 3

Date 6-27-93 Time Start 1120 Time Finish 1240 Test Duration 80 MIN min.

Duct Dimensions 8'6" X 45 Diameter _____ ft Initial Leak Rate 0.017 cfm

PTCF .84 DGMCF 110.63 Nozzle Dia. 1.24 inches Final Leak Rate 0.009 cfm @ 10"

Bar Press 29.40 " Hg Operator MKC

Static Press -5.3 " H2O

part. E-5

Travers Point	Clock Time	Dry gas meter reading ft3	ΔP in H2O	ΔH in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	
						Inlet	Outlet					
<i>1120</i>	<i>1120</i>	<i>748.695</i>										
	<i>1140</i>	<i>760.33</i>	<i>.08</i>	<i>1.02</i>	<i>317</i>	<i>91</i>	<i>89</i>		<i>256</i>	<i>65</i>	<i>3.5</i>	
	<i>1200</i>	<i>771.20</i>	<i>.67</i>	<i>.89</i>	<i>316</i>	<i>91</i>	<i>89</i>		<i>261</i>	<i>57</i>	<i>3.5</i>	
	<i>1220</i>	<i>783.40</i>	<i>.08</i>	<i>1.32</i>	<i>315</i>	<i>93</i>	<i>90</i>		<i>255</i>	<i>56</i>	<i>3.5</i>	
	<i>1240</i>	<i>793.79</i>	<i>.07</i>	<i>.87</i>	<i>316</i>	<i>98</i>	<i>93</i>		<i>272</i>	<i>59</i>	<i>4.0</i>	
Avg.	-	<i>75.096</i>	<i>0.237</i>	<i>.955</i>	<i>316</i>		<i>93</i>					
Check'd												

12.8

CONSOLE # A16401
 THIMBLE # -
 AMBIENT TEMP. 78
 PROBE LENGTH 10'
 LINER MATERIAL GLASS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet Train Bulk Particulate-Ex. Metals Run No. 1

Date 6-25-93 Time Start 0945 Time Finish 1045 Test Duration 60 min.

Duct Dimensions 8' 6" X 45" Diameter _____ ft Initial Leak Rate 0.001 cfm @ 12"

PTCF .84 DGMCF 1.009 Nozzle Dia. .375 inches Final Leak Rate 0.004 cfm @ 10"

Bar Press 29.55 " Hg

Static Press -5.8 " H2O

Operator MKD

E-4

Travers Point	Clock Time	Dry gas meter reading ft3	ΔP in H2O	ΔH in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	13.5
						Inlet	Outlet					
N/A	0945	11.425										
	1005	725.95	.13	1.75	297	86	84		252	59	3.0	
	1025	741.00	.13	1.75	295	86	84		251	49	3.5	
	1045	754.849	.13	1.75	298	86	85		252	50	4.0	
Avg.	-	43.420	3606	1.75	296		85					
Check'd												

CONSOLE # A161402
 FILTER # (Th. mble)
 AMBIENT TEMP. 78
 PROBE LENGTH 10' CLASS
 LINER MATERIAL G/AS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet Train Bulk Particulate-Ex. Metals Run No. 3

Date 6-27-97 Time Start 1300 Time Finish 1410 Test Duration 70 min.

Duct Dimensions 36" X 45" Diameter _____ ft Initial Leak Rate 6.009 cfm at 10"

PTCF .84 DGMCF 1.003 Nozzle Dia. 1.275 inches Final Leak Rate 0.006 cfm at 10"

Bar Press 29.40 " Hg Operator M/KO

Static Press -5.8 " H2O

E-5

Travers Point	Clock Time	Dry gas meter reading ft3	ΔP in H2O	ΔH in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	12.8
						Inlet	Outlet					
<u>N/A</u>	<u>1305</u>	<u>793.42</u>										
	<u>1320</u>	<u>806.03</u>	<u>.09</u>	<u>1.17</u>	<u>311</u>	<u>94</u>	<u>92</u>		<u>268</u>	<u>66</u>	<u>3.5</u>	
	<u>1340</u>	<u>818.55</u>	<u>.09</u>	<u>1.15</u>	<u>318</u>	<u>98</u>	<u>93</u>		<u>251</u>	<u>53</u>	<u>4.0</u>	
	<u>1400</u>	<u>831.57</u>	<u>.10</u>	<u>1.28</u>	<u>317</u>	<u>96</u>	<u>92</u>		<u>263</u>	<u>57</u>	<u>4.5</u>	
	<u>1410</u>	<u>837.63</u>	<u>.10</u>	<u>1.28</u>	<u>318</u>	<u>97</u>	<u>93</u>		<u>260</u>	<u>58</u>	<u>4.5</u>	
Avg.	-	<u>44.194</u>	<u>.3081</u>	<u>1.215</u>	<u>316</u>		<u>94</u>					
Check'd												

CONSOLE # A16146
 FILTER # 1001 (Thimble)
 AMBIENT TEMP. 76
 PROBE LENGTH 10
 LINER MATERIAL GLASS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS Spilled some bulk particulate at end of run
≈ 1 gram was lost.

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet Train Size Fract. Particulate Run No. 1

Date 6-25-93 Time Start 0800 Time Finish 1020 Test Duration 140 min.

Duct Dimensions 8' 6" X 45 Diameter _____ ft Initial Leak Rate 0.009 cfm at 14"

PTCF .87 DGMCF .9880 Nozzle Dia. .375 inches Final Leak Rate _____ cfm

Bar Press 29.55 " Hg

Static Press -6.4 " H2O Operator MKO

Travers Point	Clock Time	Dry gas meter reading ft3	^ P in H2O	^ H in H2O	Stack Temp. F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg	K=3.93
						Inlet	Outlet					
N/A	0800	341.86										
	0820	348.23	.08	.31	290	76	75		248	61	2.0	
	0840	354.58	.08	.31	286	87	77		245	61	2.0	
	0900	360.97	.07	.31	288	88	78		250	62	2.0	
	0920	367.08	.08	.31	288	87	78		261	66	2.0	
	0940	373.42	.08	.31	289	83	78		262	64	2.0	
	1000	379.80	.09	.31	290	86	82		257	65	3.0	
	1010	383.07	.08	.31	288	87	81		254	64	3.5	
Avg.	-	41.161	2826	31	288		81					
Check'd												

CONSOLE # A661401
 FILTER # F1308 (47mm) Thimble
 AMBIENT TEMP. 70°
 PROBE LENGTH 16'
 LINER MATERIAL SS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

SOURCE SAMPLING FIELD DATA SHEET

ESP INLET

Page 1 of 1

Plant Name Plant Yates Station Boiler No. 1

Sampling Location inlet Train Size Fract. Particulate Run No. 2

Date 6-26-93 Time Start 8:09 Time Finish 11:25 Test Duration 120 min.

Duct Dimensions 8' 6" X 45' Diameter 22.5 ft Initial Leak Rate 0.017 cfm at 10"

PTCF .84 DGMCF 1.009 Nozzle Dia. .275 inches Final Leak Rate NA cfm

Bar Press 28.56 " Hg

Static Press -5.8 " H2O

Operator MFO

Port E-8

Travers Point	Clock Time	Dry gas meter reading ft ³ in H2O	^ P in H2O	^ H in H2O	Stack Temp. °F	Dry gas meter temp.		Hot box Temp.	Probe Temp	Last Impinger	Vacuum in. Hg		
						Inlet	Outlet						
<u>4A</u>	<u>0915</u>	<u>769.05</u>											
	<u>0935</u>	<u>777.03</u>	<u>.11</u>	<u>.42</u>	<u>311</u>	<u>81</u>	<u>79</u>	<u>NA</u>	<u>248</u>	<u>62</u>	<u>3.0</u>		
	<u>0955</u>	<u>783.87</u>	<u>.10</u>	<u>.38</u>	<u>311</u>	<u>82</u>	<u>80</u>		<u>246</u>	<u>61</u>	<u>3.0</u>		
	<u>1015</u>	<u>790.70</u>	<u>.10</u>	<u>.30</u>	<u>311</u>	<u>80</u>	<u>84</u>		<u>244</u>	<u>60</u>	<u>3.0</u>		
	<u>1045</u>	<u>798.24</u>	<u>.12</u>	<u>.46</u>	<u>310</u>	<u>85</u>	<u>82</u>		<u>242</u>	<u>61</u>	<u>3.0</u>		
	<u>1105</u>	<u>805.76</u>	<u>.12</u>	<u>.46</u>	<u>311</u>	<u>88</u>	<u>84</u>		<u>241</u>	<u>60</u>	<u>3.0</u>		
	<u>1125</u>	<u>813.78</u>	<u>.10</u>	<u>.38</u>	<u>311</u>	<u>89</u>	<u>86</u>		<u>252</u>	<u>59</u>	<u>3.5</u>		
Avg.	—	<u>43,733.0</u>	<u>0.3289</u>	<u>0.413</u>	<u>310.8</u>	<u>83.8</u>							
Check'd													

KE 386

CONSOLE # A 161462
 FILTER # 1311 (47mm)
 AMBIENT TEMP. 76
 PROBE LENGTH 10'
 LINER MATERIAL GLASS

Velocity _____
 % Moisture _____
 Flowrate (DSCFM) _____
 Isokinetic (%) _____

REMARKS _____

ORSAT DATA SHEET

Plant Plant Yates Station Boiler No. 1 Comments _____
 Location ESP IN _____
 Run No. 1 _____
 Date _____ Operator JWM

Sorbing Reagents: _____ (CO₂) _____ (O₂) _____ (CO)

Replicate Number	Original Volume Reading	(CO ₂) Reading 2 (ml)	(CO ₂) Volume (2-1) (ml)	(O ₂) Reading 3 (ml)	(O ₂) Volume (3-2) (ml)	(CO) Reading 4 (ml)	(CO) Volume (4-3) (ml)
1	0.0	0.8	18.0	17.2			
<i>BAD Bag Sample</i>							

WAW *ASSUME CO₂ = 10.5 N₂ = 81*
O₂ = 8.5

Averaged Results: % CO₂ _____ % O₂ _____
 % CO _____ % N₂ _____

Dry Molecular Weight, MW (dry) =

$$= 0.44 \frac{\text{_____}}{(\% \text{CO}_2)} + 0.32 \frac{\text{_____}}{(\% \text{O}_2)} + 0.28 \frac{\text{_____}}{(\% \text{CO} + \% \text{N}_2)}$$

$$= \text{_____} + \text{_____} + \text{_____}$$

Y-097

Run # 1 Train orsat ESP Inlet
ESP Outlet Stack
 Component bag
 Date 6-21-93 Time 1930 Smplr JWM
 Lab on site Analysis CO₂ O₂
 Tare Wt. Na Final Wt. Na

ORSAT DATA SHEET

Plant Plant Yates Station Boiler No. 1 Comments _____
 Location ESP Inlet _____
 Run No. 2 _____
 Date 6/22/93 Operator JWM / TMP _____
 Sorbing Reagents: ✓ (CO₂) ✓ (O₂) (CO)

Replicate Number	Original Volume Reading	(CO ₂) Reading 2 (ml)	(CO ₂) Volume (2-1) (ml)	(O ₂) Reading 3 (ml)	(O ₂) Volume (3-2) (ml)	(CO) Reading 4 (ml)	(CO) Volume (4-3) (ml)
1	0.0	10.2	10.2	18.0	7.8		
2	0.0	10.0	10.0	18.6	8.6		
3	0.0	10.0	10.0	18.6	8.6		

Averaged Results: % CO₂ 10.2 % O₂ 8.6
 % CO _____ % N₂ 81.2

Dry Molecular Weight, MW (dry) =

$$= 0.44 \frac{\text{_____}}{(\% \text{CO}_2)} + 0.32 \frac{\text{_____}}{(\% \text{O}_2)} + 0.28 \frac{\text{_____}}{(\% \text{CO} + \% \text{N}_2)}$$

= _____ + _____ + _____

Y-251

Run # 2 Train orsat ESP Inlet
ESP Outlet
Stack
 Component bag
 Date 6-22-93 Time _____ Smplr. JWM
 Lab on site Analysis CO₂ O₂
 Tare Wt. _____ Final Wt. _____

ORSAT DATA SHEET

Plant Plant Yates Station Boiler No. 1 Comments _____
 Location ESP Inlet
 Run No. 3
 Date 6/23/93 Operator TMP

Sorbing Reagents: ✓ (CO₂) ✓ (O₂) (CO)

Replicate Number	Original Volume Reading	(CO ₂) Reading 2 (ml)	(CO ₂) Volume (2-1) (ml)	(O ₂) Reading 3 (ml)	(O ₂) Volume (3-2) (ml)	(CO) Reading 4 (ml)	(CO) Volume (4-3) (ml)
1	0.0	10.8	10.8	19.0	8.2		
2	0.0	10.9	10.9	19.4	8.5		
3	0.0	10.8	10.8	19.0	8.2		

Averaged Results: % CO₂ 10.8 % O₂ 8.3
 % CO _____ % N₂ 80.9

Dry Molecular Weight, MW (dry) =
 = 0.44 _____ + 0.32 _____ + 0.28 _____
 (%CO₂) (%O₂) (%CO + %N₂)
 = _____ + _____ + _____

Y-256

Run # 3 Train orsat ESP Inlet
ESP Outlet Stack
 Component bag
 Date 6-23-93 Time _____ Smplr JWM
 Lab on site Analysis CO₂ O₂
 Tare Wt. _____ Final Wt. _____ C-211

ORSAT DATA SHEET

Plant Plant Yates Station Boiler No. 1 Comments _____
 Location ESP Inlet _____
 Run No. Metals Run 2-1 _____
 Date 6/25/93 Operator TMP

Sorbing Reagents: _____ (CO₂) _____ (O₂) _____ (CO)

Replicate Number	Original Volume Reading	(CO ₂) Reading 2 (ml)	(CO ₂) Volume (2-1) (ml)	(O ₂) Reading 3 (ml)	(O ₂) Volume (3-2) (ml)	(CO) Reading 4 (ml)	(CO) Volume (4-3) (ml)
1	0.0	10.2	10.2	19.0	8.8		
2	0.0	10.0	10.0	19.0	9.0		

Averaged Results: % CO₂ 10.1 % O₂ 9.9
 % CO _____ % N₂ _____

Dry Molecular Weight, MW (dry) =

$$= 0.44 \frac{\quad}{(\% \text{CO}_2)} + 0.32 \frac{\quad}{(\% \text{O}_2)} + 0.28 \frac{\quad}{(\% \text{CO} + \% \text{N}_2)}$$

Y-337

Run # 1 Train Orscit ESP Inlet
ESP Outlet
Stack
 Component bag - Phase Two
 Date 6/25/93 Time 1430 Smplr JWM
 Lab on site Analysis CO₂ O₂
 Tare WT(g) _____ Final Wt(g) _____

ORSAT DATA SHEET

Plant Plant Yates Station Boiler No. 1 Comments _____
 Location ESP Inlet _____
 Run No. 2-3 _____
 Date 6/27/93 Operator TMP

Sorbing Reagents: _____ (CO₂) _____ (O₂) _____ (CO)

Replicate Number	Original Volume Reading	(CO ₂) Reading 2 (ml)	(CO ₂) Volume (2-1) (ml)	(O ₂) Reading 3 (ml)	(O ₂) Volume (3-2) (ml)	(CO) Reading 4 (ml)	(CO) Volume (4-3) (ml)
1	0.0	11.8	18.8	7.0			
2	0.0	11.8	18.8	7.0			

Averaged Results: % CO₂ 11.8 % O₂ 7.0
 % CO _____ % N₂ _____

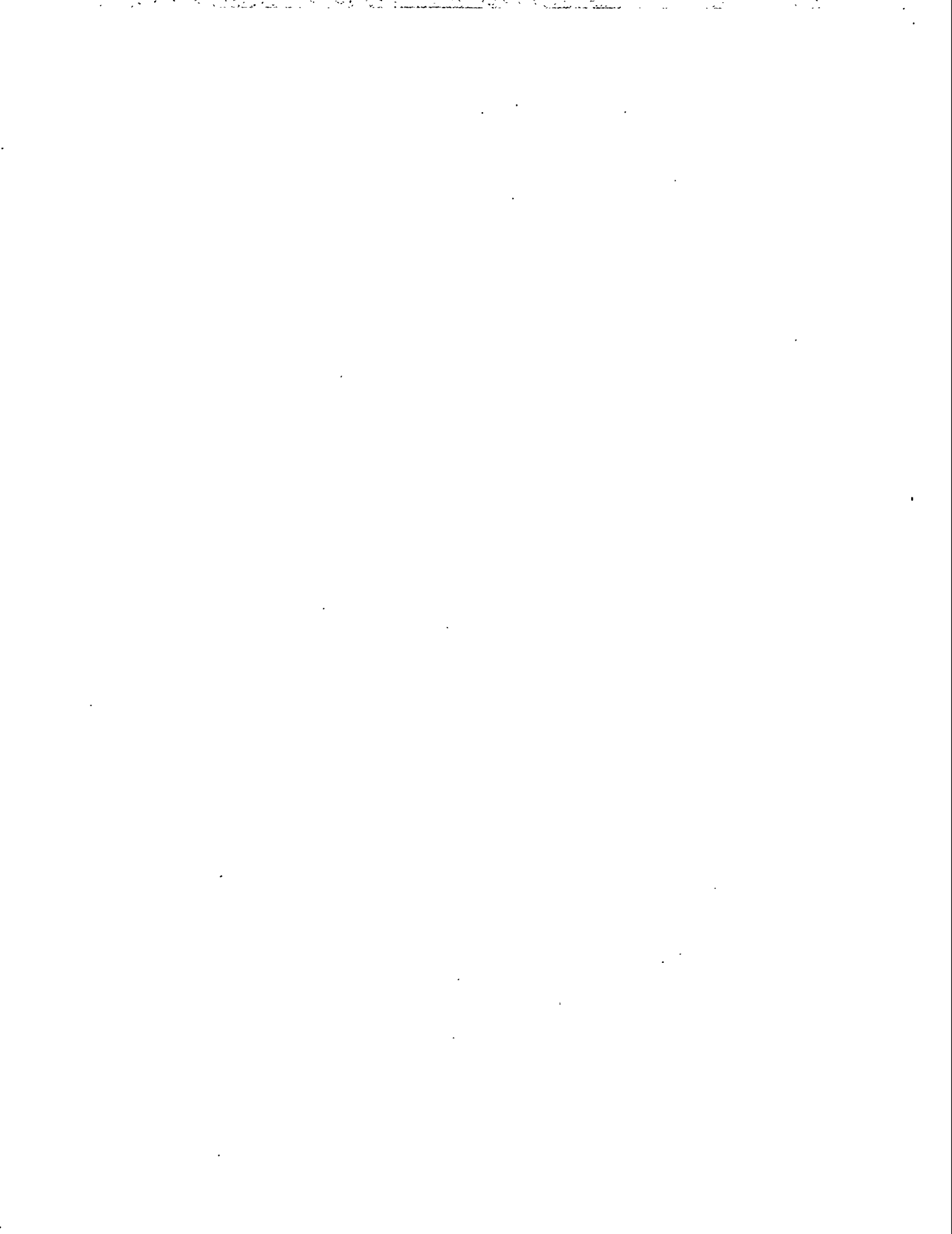
Dry Molecular Weight, MW (dry) =

$$= 0.44 \frac{\text{_____}}{(\% \text{CO}_2)} + 0.32 \frac{\text{_____}}{(\% \text{O}_2)} + 0.28 \frac{\text{_____}}{(\% \text{CO} + \% \text{N}_2)}$$

= _____ + _____ + _____

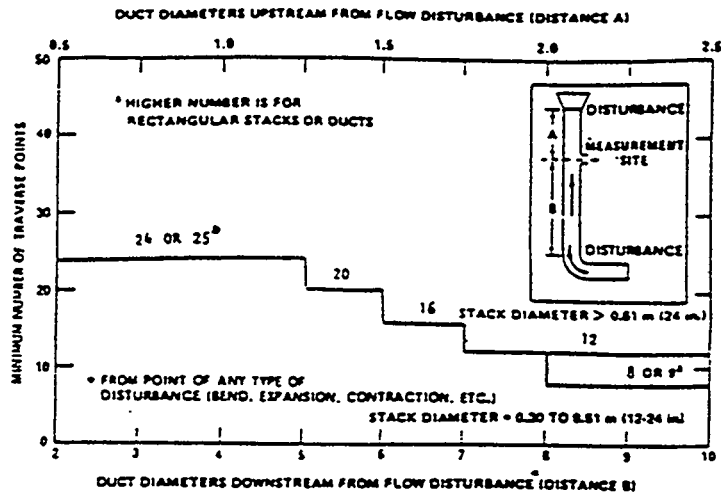
Y-454

Run # 2-3 Train ORSAT ESP Inlet
 _____ ESP Outlet
 _____ Stack
 Component ORSAT
 Date 6/27/93 Time 1400 Smplr JWM
 Lab On Site Analysis O₂/CO₂
 Tare Wt(g) — Final Wt(g) — C-213



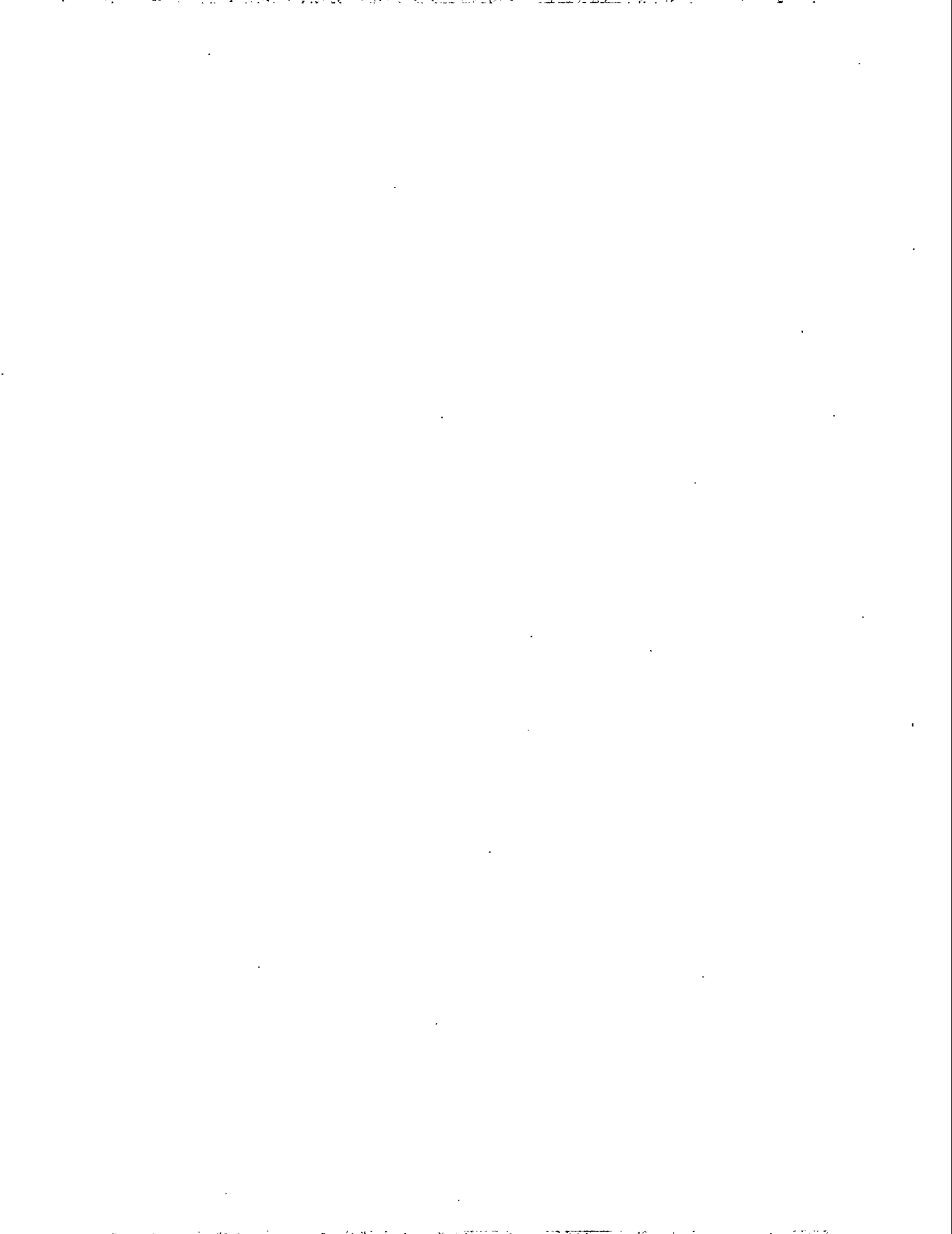
TRAVERSE FIELD DATA SHEET

Plant Name Plant Yates Station Boiler No1 Stack Diameter 8'6" x 45'
 Sampling Location ESP INLET Sample Port Diameter 4"
 Date 06-18-93 Sample Port Depth 14"
 Operator RWW/DJV/JWM Distance Upstream _____
 Distance downstream _____



Traverse Point Number	Number Traverse Points On A Diameter											
	2	4	6	8	10	12	14	16	18	20	22	24
1	14.6	6.7	4.4	3.2	2.6	2.1	1.8	1.6	1.4	1.3	1.1	1.1
2	85.4	25.0	14.6	10.5	8.2	6.7	5.7	4.9	4.4	3.9	3.5	3.2
3	75.0	29.6	19.4	14.6	11.8	9.9	8.5	7.5	6.7	6.0	5.5	
4	93.3	70.4	32.3	22.6	17.7	14.6	12.5	10.9	9.7	8.7	7.9	
5		85.4	67.7	34.2	25.0	20.1	16.9	14.6	12.9	11.6	10.6	
6		95.6	80.6	65.8	35.6	26.9	22.0	18.8	16.5	14.6	13.2	
7			89.5	77.4	64.4	36.6	28.3	23.6	20.4	18.0	16.1	
8			96.8	85.4	75.0	63.4	37.5	29.6	25.0	21.8	19.4	
9			91.8	82.3	73.1	62.5	38.2	30.6	26.2	23.0	21.0	
10			97.4	88.2	79.9	71.7	61.8	38.8	31.6	27.2	24.2	
11				93.3	85.4	78.0	70.4	61.2	39.3	32.3	28.3	
12				97.9	90.1	83.1	76.4	69.4	60.7	39.8	34.8	
13					94.3	87.5	81.2	75.0	68.5	60.2	39.2	
14						96.2	91.5	85.4	79.6	73.8	67.7	
15							95.1	89.1	83.6	78.2	72.8	
16							96.4	92.5	87.1	82.0	77.0	
17								95.6	90.3	85.4	80.6	
18								96.6	93.3	88.4	83.9	
19									96.1	91.3	86.8	
20									96.7	94.0	89.5	
21										96.5	92.1	
22											96.9	94.5
23												96.8
24												96.9

Traverse Points	
No.	Distance From Wall
	<i>PORT DEPTH INCLUDED</i>
1	22.5
2	39.5
3	56.5
4	73.5
5	90.5
6	107.5
7	
8	
9	
10	
11	
12	
13	
14	
15	
16	
17	
18	
19	
20	
21	
22	
23	
24	



VELOCITY PROFILE FIELD DATA

Plant Name Inlet Preliminary velocity traverse (75 MW Production
 Sampling Location Inlet Sample Ident. _____
 Date 6/19/93 (MMDDYY) Time Start 1100 (HHMM) Time Finish 1230 (HHMM)
 Duct Dimensions 8.5' x 45' ft. or Diameter _____ ft.
 PTCF 0.84 % H₂O ≈ 7.0
 Bar Press. 29.58 " Hg % CO _____ % N₂ _____
 Static Press. -6.5 " H₂O % CO₂ 9.0 % H₂ _____
 Operator Initials R DSV, JWM % O₂ 7.4 % CH₄ _____

I call the way in

97 MW's

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
E1-1	283	284		0.055	0.06				
2	285	285		0.06	0.06				
3	285	285		0.04	0.035				
4	284	285		0.025	0.02				
5	283	283		0.025	0.03				
6	269	269		0.02	0.02				
E2-1	281	282		0.02	0.02				
2	282	283		0.02	0.015				
3	284	284		0.01	0.02				
4	282	282		0.03	0.02				
5	274	275		0.04	0.03				
6	261	263		0.04	0.05				
E3-1	294			0.02					
2	295			0.02					
3	295			0.04					
4	296			0.08					
5	294			0.09					
6	270			0.13					

Weather Ave $\sqrt{dp} = 0.2537$
Stack Temp = 283 °F
 Remarks Vel = 17.12 fps
ACFM = 392,904
DSCFM

VELOCITY PROFILE FIELD DATA

Plant Name _____
 Sampling Location _____ Sample Ident. _____
 Date _____ (MMDDYY) Time Start _____ (HHMM) Time Finish _____ (HHMM)
 Duct Dimensions _____ x _____ ft. or Diameter _____ ft.
 PTCF _____ % H₂O _____
 Bar Press. _____ " Hg % CO _____ % N₂ _____
 Static Press. _____ " H₂O % CO₂ _____ % H₂ _____
 Operator Initials _____ % O₂ _____ % CH₄ _____

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
F-1	290		0.06	0.02					
2	293	0.05	0.05	0.05					
3	298			0.06					
4	299			0.14					
5	299			0.16					
6	290			0.19					
ES-1	290			0.03					
2	292			0.03					
3	295			0.06					
4	299			0.12					
5	300			0.15					
6	296			0.20					
E6-1	295			0.03					
2	296			0.04					
3	302			0.07					
4	305			0.14					
5	307			0.17					
6	308			0.18					

Weather _____

Remarks _____

VELOCITY PROFILE FIELD DATA

Plant Name _____
 Sampling Location _____ Sample Ident. _____
 Date _____ (MMDDYY) Time Start _____ (HHMM) Time Finish _____ (HHMM)
 Duct Dimensions _____ x _____ ft. or Diameter _____ ft.
 PTCF _____ % H₂O _____
 Bar Press. _____ " Hg % CO _____ % N₂ _____
 Static Press. 0.6 6.4 " H₂O % CO₂ _____ % H₂ _____
 Operator Initials _____ % O₂ _____ % CH₄ _____

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
E7-1	309			0.03					
2	309			0.04					
3	311			0.09					
4	311			0.12					
5	314			0.19					
6	314			0.20					
<i>access?</i> E8-1	304			0.07					
2	305			0.1					
3	307			0.08					
4	308			0.11					
5	314			0.14					
6	315			0.22					
<i>w1-1</i> E9-1	274			0.06					
2	275			0.07					
3	281			0.09					
4	282			0.06					
5	277			0.1					
6	262			0.13					

Weather _____

 Remarks _____

VELOCITY PROFILE FIELD DATA

Plant Name _____
 Sampling Location _____ Sample Ident. _____
 Date _____ (MMDDYY) Time Start _____ (HHMM) Time Finish _____ (HHMM)
 Duct Dimensions _____ x _____ ft. or Diameter _____ ft.
 PTCF _____ % H₂O _____
 Bar Press. _____ " Hg % CO _____ % N₂ _____
 Static Press. _____ " H₂O % CO₂ _____ % H₂ _____
 Operator Initials _____ % O₂ _____ % CH₄ _____

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
W2-1	273			0.04					
2	277			0.03					
3	277			0.05					
4	279			0.07					
5	286			0.16					
6	276			0.15					
W3-1	278			0.02					
2	282			0.04					
3	284			0.06					
4	282			0.13					
5	281			0.17					
6	279			0.19					
W4-1	284			0.03					
2	280			0.05					
3	282			0.08					
4	284			0.13					
5	280			0.17					
6	272			0.13					

Weather _____

 Remarks _____

VELOCITY PROFILE FIELD DATA

Plant Name _____
 Sampling Location _____ Sample Ident. _____
 Date _____ (MMDDYY) Time Start _____ (HHMM) Time Finish _____ (HHMM)
 Duct Dimensions _____ x _____ ft. or Diameter _____ ft.
 PTCF _____ % H₂O _____
 Bar Press. _____ " Hg % CO _____ % N₂ _____
 Static Press. _____ " H₂O % CO₂ _____ % H₂ _____
 Operator Initials _____ % O₂ _____ % CH₄ _____

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
W5-1	275			0.07					
2	275			0.06					
3	274			0.10					
4	277			0.11					
5	273			0.13					
6	262			0.15					
W6-1	266			0.03					
2	266			0.03					
3	272			0.03					
4	271			0.06					
5	265			0.06					
6	261			0.16					
W7-1	260			0.02					
2	263			0.02					
3	263			0.02					
4	258			0.02					
5	260			0.04					
6	243			0.04					

Weather _____

Remarks _____

VELOCITY PROFILE FIELD DATA

Plant Name _____

Sampling Location _____ Sample Ident. _____

Date _____ (MMDDYY) Time Start _____ (HHMM) Time Finish _____ (HHMM)

Duct Dimensions _____ x _____ ft. or Diameter _____ ft.

PTCF _____ 0.84 _____ % H₂O _____ ≈ 7.0 _____

Bar Press. _____ 29.58 _____ " Hg _____ % CO _____ % N₂ _____

Static Press. _____ - 6.4 _____ " H₂O _____ % CO₂ _____ % H₂ _____

Operator Initials _____ JWM _____ % O₂ _____ 7.4 _____ % CH₄ _____

Pt.	Stack Temp. °F			Velocity Pressure " H ₂ O			Other ()		
	#1	#2	Ave.	#1	#2	Ave.	#1	#2	Ave.
WS8-1	254			0.04					
2	262			0.04					
3	267			0.02					
4	264			0.01					
5	250			0.02					
6	255			0.06					
	282.9			0.2556					

MWkt
107.7
Finish

Weather _____ Vel = 17.113

_____ ACEM = 191,966

Remarks _____ DSCFM = 123,417

Facing parts
(East) E11 - E8 with WS8 (west)

APPENDIX D: QUALITY ASSURANCE/QUALITY CONTROL

Appendix D presents a summary of analytical results for QC samples, estimates of measurement precision and accuracy based on analysis of QC samples, and potential limitations in the use of the data.

Overall, QA/QC data associated with this program indicate that measurement data are acceptable and defensible. The QA/QC data indicate that the quality control mechanisms were effective in ensuring measurement data reliability within the expected limits of sampling and analytical error.

Quality control data provide information for identifying and defining qualitative limitations associated with measurement data. The following key types of QC procedures provide the primary basis for quantitatively evaluating data quality:

- Field and laboratory blank samples;
- Duplicate field samples;
- Matrix and surrogate spiked samples;
- Laboratory control samples; and
- Performance evaluation (audit) samples.

Additional details of the project QA/QC program are documented in the DOE Quality Assurance Project Plan.

Sample Collection

Several factors are evaluated to determine acceptable sample collection. Key components of the sampling equipment including the Pitot tubes, thermocouples, orifice meters, dry gas meters, and sampling nozzles were calibrated in the Radian Source Sampling Laboratory before use in the field. These calibrations were also checked after the equipment was returned to the laboratory after the field activities. The presampling calibrations were reviewed by the Radian QA Coordinator as part of the on-site Technical Systems audit.

These calibrations as well as the post sampling calibrations are on file at Radian Corporation. Standard EPA methods or other acceptable sampling methods were used to collect the organic, metal, and anion samples. The sampling runs were well documented, and all gas samples were collected at rates of between 90 and 110% of the isokinetic rates. Sufficient data were collected to ensure acceptable data completeness and comparability of the measurements.

Gas samples were collected from the ESP inlet, ESP outlet, and stack as integrated samples for most analyses over a specified time period. Solid samples of coal, limestone, bottom ash, ESP fly ash, and FGD slurry were collected at hourly intervals over each of the test runs. These individual grabs were combined to provide a single composite sample of each stream for each of the three test runs. Liquid streams were also collected as hourly grabs which were combined to provide a single composite for analysis for each test run. Liquid streams include the ash pond, gypsum recycle water, ash sluice filtrates, FGD slurry filtrate, limestone slurry filtrate, and the inlet and outlet to the condenser. All sampling was conducted while the plant was operating at 85 to 100% of full load and should be representative of typical operation for Plant Yates.

Analytical Quality Control Results

Generally, the type of quality control information obtained pertains to measurement precision, accuracy (which includes precision and bias), and blank effects that are determined using various types of replicate, spiked and blank samples. The specific characteristics evaluated depend on the type of quality control checks performed. For example, blanks may be prepared at different stages in the sampling and analysis process to isolate the source of the blank effect. Similarly, replicate samples may be generated at different stages to isolate and measure sources of variability. The QA/QC measures used as part of this program data evaluation protocol and the characteristic information obtained are summarized in Table D-1. The absence of any of these types of quality control checks from the data for a particular analytical technique does not necessarily reflect poorly on the quality of the data but does limit the ability to estimate the magnitude of the measurement error and hence, prevents placing an estimate of confidence in the results.

As shown in Table D-1, different QC checks provide different types of information, particularly pertaining to the sources of inaccuracy, imprecision, and blank effects. As part of this program, measurement precision and accuracy are typically being estimated from QC indicators that cover as much of the total sampling and analytical process as feasible. Precision and accuracy measurements are based primarily on the actual sample matrix. The precision and accuracy estimates obtained experimentally during the test program are compared to the data quality objectives (DQOs) established for the program as listed in the project QAPP.

These DQOs were not intended to be used as validation criteria but as empirical estimates of the precision and accuracy that would be expected from existing reference measurement methods and that would be considered acceptable. The precision and accuracy objectives are not necessarily derived from analyses of the same types of samples being investigated.

Table D-1
Types of Quality Control Samples

QC Activity	Characteristic Measured
Precision	
Replicate samples collected over time under the same conditions	Total variability, including process or temporal, sampling, and analytical, but not bias.
Duplicate field samples collected simultaneously	Sampling plus analytical variability at the actual sample concentrations.
Duplicate Analyses of a Single Sample	Analytical variability at the actual sample concentrations.
Matrix- or Media-Spiked Duplicates	Sampling plus analytical variability at an established concentration.
Laboratory Control Sample Duplicates	Analytical variability in the absence of sample matrix effects.
Surrogate-Spiked Sample Sets	Analytical variability in the sample matrix but at an established concentration.
Accuracy (Including Bias and Precision)	
Matrix-Spiked Samples	Analyte recovery in the sample matrix, indicating possible matrix interferences and other effects. In a single sample indicates both random error (imprecision) and systematic error (bias).
Media-Spiked Samples	Same as matrix-spiked samples. Used where a matrix-spiked sample is not feasible, such as the stack sampling methods.
Surrogate-Spiked Samples	Analyte recovery in the sample matrix, to the extent that the surrogate compounds are chemically similar to the compounds of interest. Primarily used as indicator of analytical efficacy.
Laboratory Control Samples (LCS)	Analyte recovery in the absence of actual sample matrix effects. Used as an indicator of analytical control.

Table D-1 (Continued)

QC Activity	Characteristic Measured
Standard Reference Material	Analyte recovery in a matrix similar to the actual samples.
Blank Effects	
Field Blank	Total sampling plus analytical blank effect, including sampling equipment and reagents, sample transport and storage, and analytical reagents and equipment.
Trip Blank	Blank effects arising from sample transport and storage. Typically only used for volatile organic compound analyses.
Method Blank	Blank effects inherent in analytical method, including reagents and equipment.
Reagent Blank	Blank effects from reagents used.

Although analytical precision and accuracy are relatively easy to quantify and control, sampling precision and accuracy are unique to each sample matrix. Data that do not meet these objectives are not necessarily unacceptable. Rather, the intent is to document the precision and accuracy obtained, and the objectives serve as benchmarks for comparison. The effects of not meeting the objectives should be considered in light of the intended use of the data.

Table D-2 presents the types of quality control data reported for the program and a summary of precision and accuracy estimates. Almost all of the quality control results met the project objectives.

The following potential problems were identified by the quality control data.

- Chloromethane, methylene chloride, and tetrachloroethene were found in one or more of the field blanks analyzed for VOST. In many cases, the same concentrations were also found in the field samples.
- A standard limestone sample (NIST 1C) was submitted blind as a performance audit sample. Aluminum, silicon, and sodium recoveries in this sample were below 50%, and the recovery of potassium was greater than 200 percent. This may indicate a similar low bias for these elements in the limestone process streams.
- Selenium showed no spike recovery in the impinger solutions analyzed by GFAAS. However, selenium recoveries in the audit samples submitted by RTI showed recoveries of 104 and 113 percent.

A discussion of the overall measurement precision, accuracy and blank effects is presented below for each measurement type.

Precision is a measure of the reproducibility of measurements under a given set of conditions. It is expressed in terms of the distribution, or scatter, of the data, calculated as the standard deviation or coefficient of variation (CV, standard deviation divided by the mean). For duplicates, precision is expressed as the relative percent difference (RPD).

Accuracy is a measure of the degree of conformity of a value generated by a specific procedure to be assumed or accepted true value, and includes both precision and bias. Bias is the persistent positive or negative deviation of the method average value from the assumed or accepted true value.

The efficiency of the analytical procedure for a given sample matrix is quantified by the analysis of spiked samples containing target or indicator analytes or other quality assurance measures, as necessary. However, all spikes, unless made to the flowing stream ahead of the sampling, produce only estimates of the recovery of the analyte through all of the measurement steps occurring after the addition of the spike. A good spike recovery tells little about the true value of the sample before spiking.

Table D-2
Summary of Precision and Accuracy Estimates

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Semivolatile Organics in Gas Solid Phase - SW8270	Precision- Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Acenaphthene		54	47-145	4.1	86
4-Chloro-3-methylphenol		69	22-147	5.0	84
2-Chlorophenol		62	23-134	3.0	82
1,4-Dichlorobenzene		58	20-124	3.2	80
2,4-Dinitrotoluene		55	39-139	3.2	78
n-Nitrosodipropylamine		130	0.1-230	6.3	60
4-Nitrophenol		78	0.1-132	7.0	89
Pentachlorophenol		84	14-176	9.0	45
Phenol		43	5-112	3.4	58
Pyrene		36	52-115	4.1	86
1,2,4-Trichlorobenzene		55	44-142	4.0	90
Semivolatile Organics in Fly Ash - SW8270	Precision- Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Acenaphthene		54	47-145	1.3	82
4-Chloro-3-methylphenol		69	22-147	5.6	84
2-Chlorophenol		62	23-134	1.8	84
1,4-Dichlorobenzene		58	20-124	2.5	81
2,4-Dinitrotoluene		55	39-139	2.7	76
n-Nitrosodipropylamine		130	0.1-230	7.8	60
4-Nitrophenol		78	0.1-132	37	49
Pentachlorophenol		84	14-176	5.3	64
Phenol		43	5-112	2.7	76
Pyrene		36	52-115	17.7	48Q
1,2,4-Trichlorobenzene		55	44-142	1.2	89
Semivolatile Organics in FGD Solids - SW8270	Precision- Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Acenaphthene		54	47-145	7.3	82
4-Chloro-3-methylphenol		69	22-147	9.3	76
2-Chlorophenol		62	23-134	7.1	84
1,4-Dichlorobenzene		58	20-124	8.7	80
2,4-Dinitrotoluene		55	39-139	4.0	74
n-Nitrosodipropylamine		130	0.1-230	14	52
4-Nitrophenol		78	0.1-132	14	92
Pentachlorophenol		84	14-176	4.1	74
Phenol		43	5-112	5.5	73
Pyrene		36	52-115	4.4	90
1,2,4-Trichlorobenzene		55	44-142	9.8	92
Semivolatile Organics in Aqueous Streams - SW8270	Precision- Matrix-Spiked Duplicates Accuracy - Matrix Spikes				
Acenaphthene		54	47-145	11	79
4-Chloro-3-methylphenol		69	22-147	10	83
2-Chlorophenol		62	23-134	10	80
1,4-Dichlorobenzene		58	20-124	6.8	72
2,4-Dinitrotoluene		55	39-139	7.4	82
n-Nitrosodipropylamine		130	0.1-230	12	75
4-Nitrophenol		78	0.1-132	8.6	47
Pentachlorophenol		84	14-176	11	72
Phenol		43	5-112	12	40
Pyrene		36	52-115	7.6	78
1,2,4-Trichlorobenzene		55	44-142	9.7	82

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Dioxins and Furans in Stack Gas Solid Phase	Precision: NA Accuracy: Internal Standard Recovery				
¹³ C ₁₂ -2,3,7,8-TCDF		50	40-120		60
¹³ C ₁₂ -2,3,7,8-TCDD		50	40-120		61
¹³ C ₁₂ -1,2,3,7,8-PeCDF		50	40-120		56
¹³ C ₁₂ -1,2,3,7,8-PeCDD		50	40-120		63
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF		50	40-120		69
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD		50	40-120		69
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF		50	40-120		57
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD		50	40-120		64
¹³ C ₁₂ -1,2,3,4,6,7,8,9-OCDD		50	40-120		50
PCDD/PCDF	Precision - NA Accuracy - Internal Standard Recovery, average for all samples analyzed.				
¹³ C ₁₂ 2,3,7,8-TCDF			40-120		57.2
¹³ C ₁₂ -2,3,7,8-TCDD			40-120		54.7
¹³ C ₁₂ -1,2,3,7,8-PeCDF			40-120		55.7
¹³ C ₁₂ -1,2,3,7,8-PeCDD			40-120		63.3
¹³ C ₁₂ -1,2,3,6,7,8-HxCDF			40-120		69.2
¹³ C ₁₂ -1,2,3,6,7,8-HxCDD			40-120		69.0
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDF			40-120		57.1
¹³ C ₁₂ -1,2,3,4,6,7,8-HpCDD			40-120		63.6
¹³ C ₁₂ -1,2,3,4,6,7,8,9-OCDD			40-120		50.0
PCDD/PCDF in Stack Gas	Precision - NA Accuracy - Surrogate Spike Recovery, average for all samples analyzed.				
³⁷ Cl ₄ -2,3,7,8-TCDD			70-130		118.4
¹³ C ₁₂ -2,3,4,7,8-PeCDF			70-130		113.2
¹³ C ₁₂ -1,2,3,4,7,8-HxCDF			70-130		120.8
¹³ C ₁₂ -1,2,3,4,7,8-HxCDD			70-130		141.6
¹³ C ₁₂ -1,2,3,4,7,8,9-HpCDF			70-130		104.7
¹³ C ₁₂ -1,2,3,7,8,9-HxCDF			70-130		75.4
¹³ C ₁₂ -2,3,4,6,7,8-HxCDF			70-130		84.3
Volatile Organics in Vapor Phase - SW8240	Precision - NA Accuracy - Surrogate Spike Recovery				
1,2-Dichloroethane-d4		50	70-130		114
Toluene-d8		50	70-130		101
4-Bromofluorobenzene		50	70-130		108
Aldehydes in Vapor Phase	Precision - Duplicate Analyses Accuracy - Matrix Spiked Samples				
Acetaldehyde		50	50-150	10	94
Formaldehyde		50	50-150	36	90
Aldehydes in Aqueous Streams	Precision - Duplicate Analyses Accuracy - Matrix Spiked Samples				
Acetaldehyde		50	50-150	14	101
Formaldehyde		50	50-150	18	94

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Gas Solid Phase - ICP-AES					
Precision - Matrix-spiked pairs					
Accuracy - Matrix-spiked Sample					
Aluminum		20	75-125	62Q	62Q
Antimony		20	75-125	20	84
Barium		20	75-125	30Q	75
Beryllium		20	75-125	<1	89
Chromium		20	75-125	2.9	88
Cobalt		20	75-125	1	91
Copper		20	75-125	<1	93
Manganese		20	75-125	2.2	91
Molybdenum		20	75-125	3.7	94
Nickel		20	75-125	5	89
Vanadium		20	75-125	2.2	94
Metals in Gas Solid Phase - ICP-AES					
Precision - NA					
Accuracy - Standard reference material (NIST 1633a Fly Ash)					
Aluminum		20	75-125		94
Antimony		20	75-125		NC
Barium		20	75-125		82
Beryllium		20	75-125		147Q
Calcium		20	75-125		99
Chromium		20	75-125		96
Cobalt		20	75-125		88
Copper		20	75-125		95
Iron		20	75-125		93
Magnesium		20	75-125		95
Manganese		20	75-125		94
Potassium		20	75-125		109
Nickel		20	75-125		94
Silicon		20	75-125		98
Sodium		20	75-125		96
Strontium		20	75-125		92
Titanium		20	75-125		97
Vanadium		20	75-125		95
Zinc		20	75-125		97
Metals in Gas Vapor Phase - ICP-AES					
Precision - Matrix-spiked Duplicates					
Accuracy - Matrix-spiked Sample					
Aluminum		20	75-125	<1	104
Antimony		20	75-125	4	101
Barium		20	75-125	0	106
Beryllium		20	75-125	0	108
Boron		20	75-125	2.9	104
Chromium		20	75-125	0	105
Cobalt		20	75-125	0	102
Copper		20	75-125	0	105
Manganese		20	75-125	<1	104
Molybdenum		20	75-125	2.0	100
Nickel		20	75-125	0	102
Vanadium		20	75-125	0	107

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Gas Vapor Phase - ICP-AES (HNO₃/H₂O₂ Impinger Solution)	Precision - NA Accuracy - Standard reference material (EPA ICP-19)				
Antimony		20	75-125		93
Beryllium		20	75-125		101
Calcium		20	75-125		109
Chromium		20	75-125		99
Cobalt		20	75-125		100
Copper		20	75-125		119
Iron		20	75-125		93
Manganese		20	75-125		97
Molybdenum		20	75-125		108
Nickel		20	75-125		102
Vanadium		20	75-125		103
Metals in Coal - INAAS	Precision - NA Accuracy - Standard Reference Material (NIST 1632b coal)				
Antimony		20	80-120		94
Barium		20	80-120		99
Beryllium		20	80-120		109
Boron		20	80-120		99
Chromium		20	80-120		99
Cobalt		20	80-120		NC
Copper		20	80-120		99
Manganese		20	80-120		103
Molybdenum		20	80-120		102
Nickel		20	80-120		99
Vanadium		20	80-120		97
Metals in Limestone - ICP-AES	Precision - NA Accuracy- Standard reference material (NIST Limestone 1c)				
Aluminum		20	75-125		14Q
Calcium		20	75-125		101
Iron		20	75-125		70Q
Magnesium		20	75-125		69Q
Manganese		20	75-125		74Q
Potassium		20	75-125		224Q
Silicon		20	75-125		1.5Q
Sodium		20	75-125		47Q
Strontium		20	75-125		97
Metals in FGD Solids - ICP-AES	Precision - Matrix-spiked Duplicates Accuracy - Matrix-spiked Samples				
Aluminum		20	75-125	8.7	94
Antimony		20	75-125	4.7	83
Barium		20	75-125	6.0	84
Beryllium		20	75-125	4.6	81
Boron		20	75-125	28Q	91
Chromium		20	75-125	5.7	82
Cobalt		20	75-125	5.6	78
Copper		20	75-125	5.1	87
Manganese		20	75-125	15	79
Molybdenum		20	75-125	5.1	79
Nickel		20	75-125	5.0	79
Vanadium		20	75-125	5.6	84

Appendix D: Quality Assurance/Quality Control

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in ESP Fly Ash - ICP-AES	Precision - Matrix-spiked Duplicates Accuracy - Matrix-spiked Samples				
Aluminum		20	75-125	16	78
Antimony		20	75-125	8.4	91
Barium		20	75-125	10.2	85
Beryllium		20	75-125	1.8	92
Chromium		20	75-125	1.7	94
Cobalt		20	75-125	1.8	93
Copper		20	75-125	2.4	95
Manganese		20	75-125	2.5	92
Molybdenum		20	75-125	4.5	84
Nickel		20	75-125	5.2	96
Vanadium		20	75-125	2.8	94
Metals in Aqueous Process Streams - ICP-AES	Precision - Matrix-spiked Duplicates Accuracy - Matrix-spiked Samples				
Aluminum		20	75-125	4.4	96
Antimony		20	75-125	16	87
Barium		20	75-125	7.6	99
Beryllium		20	75-125	4.4	92
Boron		20	75-125	1.0	96
Chromium		20	75-125	4.9	92
Cobalt		20	75-125	4.6	89
Copper		20	75-125	4.0	96
Manganese		20	75-125	4.5	92
Molybdenum		20	75-125	4.8	89
Nickel		20	75-125	7.3	90
Vanadium		20	75-125	3.6	95
Metals in Aqueous Process Streams - ICP-AES	Precision - NA Accuracy - Performance Audit Samples (2 concentrations)				
Antimony		20	75-125		127Q/82
Beryllium		20	75-125		99/93
Calcium		20	75-125		169Q
Chromium		20	75-125		94/97
Cobalt		20	75-125		100/87
Copper		20	75-125		96/110
Iron		20	75-125		103/139Q
Magnesium		20	75-125		131Q
Manganese		20	75-125		96/95
Molybdenum		20	75-125		98/114
Nickel		20	75-125		104/111
Titanium		20	75-125		98
Vanadium		20	75-125		96/104
Zinc		20	75-125		99
Metals in Gas Vapor Phase - GFAAS and CVAAS	Precision - Matrix spiked Duplicates Accuracy - Matrix Spiked Samples				
Arsenic		20	75-125	4.0	100
Cadmium		20	75-125	<1	114
Lead		20	75-125	45Q	84
Mercury		20	75-125	1.3	98
Selenium		20	75-125	94Q	0
Metals in Gas Solid Phase - CVAAS	Precision - Matrix spiked Duplicates Accuracy - Matrix Spiked Samples				
Mercury		20	75-125	1.0	128Q
Metals in Gas Vapor Phase - CVAAS	Precision - NA Accuracy - Performance Audit Samples				
Mercury (KMnO ₄ Impinger Solution)		20	75-125		33Q

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Metals in Process Solid Streams - GFAAS and CVAAS	Precision - Matrix spiked Duplicates Accuracy - Matrix Spiked Samples				
Arsenic		20	75-125	<1	104
Cadmium		20	75-125	8.8	110
Lead		20	75-125	1.2	86
Mercury		20	75-125	2.6	107
Selenium		20	75-125	25.3Q	103
Metals in Solid Phase - GFAAS and CVAAS	Precision - NA Accuracy - Standard reference material (NIST 1633a Fly Ash)				
Arsenic		20	75-125		NA
Cadmium		20	75-125		NA
Lead		20	75-125		NA
Mercury		20	75-125		119
Selenium		20	75-125		NA
Metals in Aqueous Process Streams - GFAAS and CVAAS	Precision - Matrix Spiked Duplicates Accuracy - Matrix Spiked Samples				
Arsenic		20	75-125	4.2	99
Cadmium		20	75-125	2.2	108
Lead		20	75-125	12	76
Mercury		20	75-125	24.6Q	35Q
Selenium		20	75-125	41.2Q	76.4
Metals in Aqueous Process Streams - GFAAS and CVAAS	Precision - NA Accuracy - Performance Audit Samples (2 concentrations)				
Arsenic		20	75-125		94/100
Cadmium		20	75-125		93/100
Lead		20	75-125		99/96
Selenium		20	75-125		96/50
Metals in Gas Vapor - ICP/MS (HNO₃/H₂O₂ Impinger Solution)	Precision - NA Accuracy - Performance Audit Samples				
Antimony		NA	NA		89
Arsenic		NA	NA		109
Beryllium		NA	NA		98
Cadmium		NA	NA		97
Chromium		NA	NA		97
Cobalt		NA	NA		88
Copper		NA	NA		83
Lead		NA	NA		87
Manganese		NA	NA		97
Molybdenum		NA	NA		94
Nickel		NA	NA		90
Selenium		NA	NA		106
Vanadium		NA	NA		93

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Extractable Metals - ICP/MS Nitric acid digestate)	Precision - Duplicate Analysis Accuracy - Matrix-Spiked Samples				
Antimony		20	NA	40Q	NA
Arsenic		20	75-125	434Q	118
Barium		20	75-125	5.8	94
Beryllium		20	75-125	11	108
Cadmium		20	75-125	0	94
Chromium		20	75-125	9.4	98
Cobalt		20	75-125	7.7	100
Copper		20	75-125	19	100
Lead		20	75-125	1.6	83
Manganese		20	75-125	9.6	108
Mercury		20	75-125	NC	852Q
Molybdenum		20	NA	12	NA
Nickel		20	75-125	13	103
Selenium		20	75-125	43Q	138Q
Vanadium		20	75-125	3.6	109
Extractable Metals - ICP/MS (Gastric fluid leachate)	Precision - Duplicate analysis Accuracy - Matrix-spiked samples				
Antimony		20	NA	6.5	NA
Arsenic		20	75-125	NC	0Q
Barium		20	75-125	1.5	85
Beryllium		20	75-125	12	79
Cadmium		20	75-125	27Q	107
Chromium		20	75-125	4.2	88
Cobalt		20	75-125	3.4	92
Copper		20	75-125	14	92
Lead		20	75-125	3.2	97
Manganese		20	75-125	3.2	71Q
Mercury		20	75-125	61Q	124
Molybdenum		20	NA	10	NA
Nickel		20	75-125	3.7	81
Selenium		20	75-125	NC	84
Vanadium		20	75-125	NC	0Q
Metals in Gas Solid Phase - GDMS	Precision - NA Accuracy - Standard Reference Material (NIST 1633a Fly Ash)				
Aluminum					
Antimony		NA	NA		180Q
Barium		NA	NA		NC
Beryllium		NA	NA		357Q
Calcium		NA	NA		NC
Chromium		NA	NA		70Q
Cobalt		NA	NA		140Q
Copper		NA	NA		NC
Iron		NA	NA		203Q
Magnesium		NA	NA		79
Manganese		NA	NA		120
Potassium		NA	NA		58Q
Nickel		NA	NA		119
Silicon		NA	NA		115
Sodium		NA	NA		111
Strontium		NA	NA		39Q
Titanium		NA	NA		320Q
Vanadium		NA	NA		131Q
Zinc		NA	NA		141Q
		NA	NA		129Q

Table D-2 (Continued)

Measurement Parameter	How Measured	Objectives		Measured	
		Precision (% RPD)	Accuracy (% Recovery)	Precision (% RPD)	Accuracy (% Recovery)
Anions in Aqueous Process Streams -	Precision - NA				
	Accuracy - Performance Audit Samples				
Chloride		20	80-120		0Q
Fluoride		20	80-120		39Q
Sulfate		20	75-125		350Q
Anions in Gas Vapor Phase -	Precision - Matrix spiked Duplicates				
	Accuracy - Matrix Spiked Samples				
Chloride		20	80-120	9.7	100
Fluoride		20	80-120	1.9	107
Anions in Process Solid Streams	Precision - Matrix spiked Duplicates				
	Accuracy - Matrix Spiked Samples				
Chloride		20	80-120	<1	95
Fluoride		20	80-120	3.5	70
Anions in Aqueous Process Streams	Precision - Matrix spiked Duplicates				
	Accuracy - Matrix Spiked Samples				
Chloride		20	80-120	3.6	111
Fluoride		20	80-120	1.6	101
Sulfate		20	75-125	1.5	97
Ammonia in Gas Vapor Phase by 350.2	Precision - Matrix spiked Duplicates				
	Accuracy - Performance Audit Standard				
Ammonia		20	80-120	39Q	63Q
Ammonia in Aqueous Streams by 350.1	Precision - Matrix spiked Duplicates				
	Accuracy - Performance Audit Standard				
Ammonia		20	80-120	60Q	88
Cyanide in Gas Vapor Phase by 335.2	Precision - Matrix spiked Duplicates				
	Accuracy - Performance Audit Standard				
Cyanide		20	75-125	16	50
Cyanide in Aqueous Streams by 335.2	Precision - Matrix spiked Duplicates				
	Accuracy - Performance Audit Standard				
Cyanide		20	75-125	13	80
Phosphate in Aqueous Streams by 365.2	Precision - Matrix spiked Duplicates				
	Accuracy - Performance Audit Standard				
Phosphate		20	75-125	6.1	97

NA = Not applicable.

NC = Not calculated.

Q = Outside project QC objectives.

Representativeness expresses the degree to which sample data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. The representativeness criterion is based on making certain that the sampling locations are properly selected and that a sufficient number of samples are collected.

Comparability is a qualitative parameter expressing the confidence with which one data set can be compared to another. Sampling data should be comparable with other measurement data for similar samples under similar conditions. This goal is achieved using standard techniques to collect and analyze representative samples and by reporting results in appropriate units. Data sets can be compared with confidence when the precision and accuracy is known.

Completeness is an expression of the number of valid measurements obtained compared with the number planned for a given study. The goal is to generate a sufficient amount of valid data.

Semivolatile Organics

Precision. The precision of the semivolatile organic analyses was estimated using matrix spiked duplicate pairs. The precision was met for all of the gas-phase solid samples, the gas vapor-phase samples, the solid stream samples, and aqueous-phase sample streams. The precision estimates are summarized for each stream in Table D-2.

Accuracy. The accuracy of the semivolatile analyses was estimated using matrix spiked duplicate samples. All of the spiked compounds analyzed in the gas solid-phase samples and the aqueous process streams were within the accuracy objectives. Matrix spikes into the solid process streams were all within the recovery objects for all analytes in the FGD solid stream and all the except pyrene in the ESP ash solids. Recovery for pyrene was 51% and 56% (project objective--52-115%) for the ESP ash sample and 48% and 37% for the ESP ash field duplicate.

Blank Effects. Acetophenone and benzoic acid were found in one or more of the field blanks associated with the gas-phase solids analyses. The concentrations of these compounds in the blanks, however, were not significant in comparison to the concentrations found in the samples. Several phthalates were also found in the field blanks. The concentrations found in the samples were about the same level as found in the blanks and are therefore considered an artifact of the sampling and handling process.

Volatile Organics

Precision. Precision for volatile organic analysis of the aqueous process streams was estimated using matrix spiked duplicate samples. The 50% precision objectives were met for each of the volatile analytes used for the matrix spikes.

Accuracy. Accuracy for the volatile organic analyses in the aqueous process streams was estimated using matrix spiked samples and accuracy for the gas vapor-phase streams was estimated using surrogates spiked into each sample prior to analysis. The accuracy objectives for recoveries ranging from 0.1% to 234% were met for all analytes of interest (actual recoveries ranged from 70-136%) for the aqueous streams. Accuracy objectives for surrogate recoveries of 70 to 130% for the gas-phase streams were met for all samples except for toluene-d8 in one stack sample. Accuracy based on the analysis of two laboratory method spikes met the recovery objectives for all analytes of interest except for one acetone, chloromethane, chloroethane, and methylene chloride spike.

Blank Effects. Chloromethane, methylene chloride, and tetrachloroethene were found in one or more of the field gas vapor-phase blank samples. In most cases these compounds were found in the investigative field samples at about the same level as in the field blank or at lower concentrations. The sampling, handling, and transport from the field may have contributed this observed contamination. Chloromethane and methylene chloride were also found in one laboratory blank.

Aldehydes

Precision. Precision for the aldehyde analyses was estimated using duplicate sample analyses. The precision objectives of 50% were met for both formaldehyde and acetaldehyde in the gas vapor-phase samples and the aqueous process stream sample analyses.

Accuracy. Accuracy for the aldehydes was estimated using matrix spiked samples. The project accuracy objectives of recoveries of 50-150% were met for the gas vapor-phase and aqueous stream sample spikes for both formaldehyde and acetaldehyde.

Blank Effects. Formaldehyde and acetaldehyde were found in concentrations (3.8-8.2 μg , formaldehyde; 2.7-8.6 μg , acetaldehyde) above the reporting limits in the field blanks to the gas vapor-phase sampling train. Low levels (within 3 times the detection limit) of these analytes were also found in two of the four laboratory (method) blanks but were not found in the trip blanks.

Metals

Precision. The precision of metals analyses by ICP-AES, GFAAS, and CVAAS was estimated for samples using matrix-spiked duplicate samples. The precision objectives (RPD <20%) were met for all target analytes analyzed by ICP-AES except aluminum and barium in the gas solid-phase spiked samples and boron in the process solid-spiked samples. The precision objectives for the GFAAS analyses were met except for lead in the gas vapor-phase matrix-spiked samples, selenium in the process solid matrix-spiked samples, and mercury and selenium in the aqueous process stream matrix spikes. In most of these cases, the concentrations of the analytes of interest were within 10 times the detection limit where the precision would not be expected as good or the spiked amount was low (<4 times) the amount found in the original sample.

Accuracy. The accuracy of metals analyses was estimated for the gas solid-phase samples using standard reference material (NIST 1633a fly ash) submitted blind to the laboratory as a performance audit sample. All of the metals analyzed by ICP-AES were within the 75-125% accuracy objectives except for beryllium (147%) which was recovered above the objectives. The fly ash (NIST 1633a) reference standard was also submitted for GDMS analysis. The results for this analysis are shown in Table D-2. Accuracy objectives were not assigned to the GDMS analyses since this technique has not been validated or widely used for these types of samples at the present time. However, the recoveries have been compared to the accuracy objectives for ICP-AES and flagged with a Q when outside the QC objectives.

The accuracy of the metals analyses was estimated for coal samples using a standard reference coal sample (NIST 1632b) submitted blind to the laboratory. All of the metals analyzed by INAA in the reference sample were within the 75-125% accuracy objective.

The accuracy of the metals analyses was estimated for the limestone samples using a standard reference limestone (NIST Limestone 1C) submitted blind to the laboratory. The results show that the recoveries for most of the metals were outside the 75-125% accuracy objectives. Aluminum, silicon, and sodium recoveries were 50%, and the recovery for potassium was greater than 200 percent. The recoveries of these analytes may show a similar bias in the limestone process streams.

The accuracy of the metals analyses for the gas vapor-phase samples and the aqueous process streams were estimated using performance audit samples prepared from EPA reference standards. The gas-phase audit sample was prepared in the solutions used for the impingers (multi-metals train) and the two aqueous-phase samples were prepared in HPLC grade water. The results show that the recoveries of all the metals analyzed by ICP-AES and GFAAS were within the 75-125% accuracy objectives except Sb (127%), Ca (169%), Fe (139%), and Mg (131%) by ICP-AES and Se (50%) and Hg (33%) by GFAAS. The concentrations of these elements in the samples were at or near the detection limit and are not expected to be as accurate as concentrations at higher levels (at least 10 times the detection limit). The gas-phase audit sample prepared in the $\text{HNO}_3/\text{H}_2\text{O}_2$ impinger solution was also analyzed by ICP/MS. The results for this analysis showed recoveries ranging from 83 to 109%, all within the accuracy objectives for ICP-AES (accuracy objectives were not assigned for ICP/MS).

Matrix-spiked samples were also used to determine the accuracy of the metals analyses in the gas, process solids, and aqueous process matrices. Recoveries for the target analytes were within the 75-125% accuracy objectives except for selenium (0% recovery) in the gas vapor-phase matrix mercury (35% recovery) in the aqueous process stream matrix.

Blank Effects. Aluminum, iron, manganese, and nickel were found at concentrations above the reporting limits in the field blanks to the gas vapor-phase sampling train. These elements were also found to a lesser extent in the impinger reagent blank solutions. Field blank filters combined with probe/nozzle rinses were also analyzed to determine the contribution of the filter media to the gas solid-phase components. Background or blank correction was

performed for the gas-phase samples using the results of the analysis of the impinger reagent blanks and the blank filter media.

Anions

Precision. Precision for the anions analyses was estimated for the gas vapor-phase samples, process solid streams, and aqueous process streams by the analysis of matrix spiked samples. The precision objectives of 20% were met for chloride, fluoride, and sulfate except for chloride and sulfate in one matrix spike pair from the stack with RPDs of 22% and 24%, respectively.

Accuracy. Accuracy for the anions analyses was estimated using matrix spiked duplicate samples. The accuracy objectives of 80-120% recovery were met for all analytes and all sample matrices except for the fluoride spikes into the ESP ash solid samples with recoveries of 56% and 60 percent. A performance audit sample was submitted for analysis of the target anions in an aqueous matrix. The recoveries for this sample were outside the accuracy objectives for all three analytes. This sample was prepared with each analyte concentration at the MDL; therefore, no corrective action was initiated.

Cyanide, Ammonia, and Phosphate

Precision. Precision for the cyanide, ammonia, and phosphate analyses was estimated using matrix spiked duplicate sample analyses. The precision objectives of 20% were met for each of the analytes for both the gas vapor-phase and aqueous process streams except for ammonia spikes into the JBR process liquids. The spike concentration was too low in comparison to the level found in the native process sample.

Accuracy. Accuracy for ammonia, cyanide and phosphate was estimated using both matrix spiked duplicate samples and "double blind" performance audit samples. The accuracy objectives (cyanide, 75-125%; ammonia, 80-120%; phosphate, 75-125%) were met for all matrix spiked samples except for the ammonia spikes into the JBR process liquids with recoveries at 60 and 273 percent. Recoveries for the performance audit samples met the accuracy objectives for all analytes with recoveries of 88% for ammonia, 80% for cyanide, and 97% for phosphate. Recoveries for performance audit samples spiked into the gas vapor-phase impinger solutions were not as good as the aqueous spiked audit samples. The recovery for ammonia in the impinger solutions was 63% and the recovery for cyanide was 50 percent. The aqueous spikes and impinger spikes were performed using the same spiking solutions and were spiked at the same concentration levels.

Performance Evaluation Audit Samples

Performance audit samples are samples of known composition which provide a point-in-time assessment of analytical performance. Audit samples were prepared for this study by spiking known concentrations of target analytes from EPA Quality Control Check material, vendor-certified standard material, or standards obtained from NIST (formerly NBS). Audit samples are similar to QCCS except that they are submitted "double blind" to the analytical laboratory. That is, the laboratory does not know the identity or composition of the audit samples.

Audit samples were prepared at concentration levels simulating the expected range of the analytes in the field samples when possible. Organic audit samples were not prepared because the laboratories performing organic analyses have consistently shown acceptable performance on surrogate recoveries and internal quality control samples. Results for these samples are shown in Table D-2.

Quality Assurance Audits

The purpose of a quality assurance audit is to provide an objective, independent assessment of a sampling or measurement effort. It ensures that the sampling procedures, data generating, data gathering, and measurement activities produce reliable and useful results. Sometimes inadequacies are identified in the sampling/measurement system and/or the quality control program. In such cases, audits provide the mechanism for implementing corrective action.

A technical systems audit (TSA) is an on-site, qualitative review of the various aspects of a total sampling and/or analytical system. It is an assessment of overall effectiveness and represents a subjective evaluation of a set of interactive systems with respect to strengths, deficiencies, and potential areas of concern. The audit consists of observations and documentation of all aspects of the measurement effort. Checklists that delineate the critical aspects of each methodology are used by the Radian auditor during the audit to document all observations. In addition to evaluating sampling and analytical procedures and techniques, the systems audit emphasizes review of all recordkeeping and data handling systems including:

- Calibration documentation for analytical instrumentation and sampling apparatus;
- Documentation of quality control data (control charts, etc.);
- Completeness of data forms and notebooks;
- Data review and validation procedures;
- Sample logging procedures;
- Chain-of-custody procedures;

- Documentation of maintenance; and
- Review of malfunction reporting procedures.

A technical systems audit of the Radian sampling and on-site analytical efforts was conducted on June 23 - 25, 1993 at Plant Yates by Barbara Hayes, a member of Radian's Quality Assurance Section. No critical or major concerns were observed during the audit; therefore, no Recommendations for Corrective Action (RCAs) were made. The sampling team was led by Dave Virbick and the analytical team was led by David Maxwell. The sampling team appeared well versed in the sampling methodology and requirements of the program. The equipment and instrumentation were generally in good working condition. All sampling and measurement procedures conformed to those described in the site Management Plan. Sampling information and any problems encountered were recorded onto preformatted data sheets or into bound laboratory notebooks. Duplicate samples were collected for the solid and aqueous streams at a rate of ten percent or one duplicate set per sample type (bottom ash, fly ash, etc.).

Sample collection procedures used by the sampling team followed those outlined in the site test plan. A detailed sampling schedule was used by the team to guide the collection of the samples for each analytical species at each sampling point.

No problems were identified with the sample custody procedures or documentation. A detailed master logbook was prepared prior to the field effort for all samples to be collected during each sampling period. This log was updated as the various samples were collected with the actual dates and times of sample collection. Samples were labelled with preformatted sample labels and stored at ambient temperature or cooled as required by the analytical species. Chain-of-custody forms were filled out and the samples were prepared for shipment to the laboratories for analysis.

Calibration of all on-site equipment was checked and found to be up-to-date. The analytical balance and top loading balance in the on-site laboratory trailer had been calibrated and certified within the past year. In addition, certified weights were available for daily balance checkout. All dry gas meters, consoles, Pitot tubes, and nozzles had been calibrated in the Radian Source Sampling Laboratory prior to being transported to the field location. Documentation for each of the observed instruments and equipment in use could be found in the records maintained by the sampling crew chief in the on-site laboratory. Sufficient replacement units were on hand to allow for breakage or equipment malfunction.

Recordkeeping practices by the project team were observed to be sound. Entries were made onto preformatted data sheets in ink, without erasures, signed and the time noted as each sample was collected.

Coal Round Robin

An interlaboratory study consisting of a coal round robin analysis was conducted by CONSOL, Inc. The objective of this round robin study was to estimate the analytical

variability one can expect on trace element analyses when comparing results from the same laboratory or results from two or more laboratories. The results of CONSOL's study is contained in the document entitled "Interlaboratory Variability and Accuracy of Coal Analyses in the U.S. Department of Energy Utility Air Toxics Assessment Program," which follows this section. The results from Radian's laboratory are designated as "Lab III" in the above referenced document. Radian's objectives in assessing this data are (1) to compare Radian's round robin results with the overall results of the study, and (2) based on this assessment, determine if a change in any of the analytical methods for Phase II should be made.

The analytical accuracy for each laboratory involved in the round robin study was measured by a comparative analysis of a standard reference material (SRM) coal sample (NIST 1632b). Each laboratory's analytical results for the standard reference material were compared to the certified or informational (non-certified) values. The round robin criteria for accurate results was 90-110% recovery of the SRM's certified value. (This is more stringent than the 80-120% recovery objective established for the program at Plant Yates). The following discussion addresses the performance of Radian's subcontracted coal laboratories with respect to the accuracy and precision assessments conducted by CONSOL on the NIST SRM.

Discussion of Results

The results of Radian's analysis of the SRM and the SRM-certified values are shown in Table D-3. Accuracy and precision objectives for the SRM coal in the round robin study were met by Radian for all ultimate and proximate parameters (% ash, C, H, N, S, and HHV) with the exception of one sulfur analysis which was reported outside the objective range for accuracy and precision. The methods used for ultimate, proximate, and HHV analyses are current ASTM protocols and are consistent with the methods used by most of the other laboratories. No change in the analytical approach for Phase II of this project is warranted.

Major ash minerals were primarily determined by instrumental neutron activation analysis (INAA). Silicon dioxide (SiO_2) and sulfur trioxide (SO_3) were not reported for the Plant Yates or the round robin study. The accuracy and precision objectives were met for all major ash minerals reported except calcium, magnesium and potassium. For future work, other ASTM methods (ASTM D-4326 or alternate) should be used to improve analytical bias and precision for these elements. This is especially important where these major elements are considered key factors in assessing mass flow rates in material balance closures.

Radian analyzed most of the trace elements in coal by INAA. Other methods of analysis using different preparation techniques were performed for As, B, Be, Cd, F, Hg, Pb, and Se. Of the target trace elements, 82% were detected. Cadmium, copper, and nickel were not detected. The results for copper and nickel are surprising, since this same SRM (1632b) was used as an internal audit sample during the Plant Yates study, and recovery by the same method (INAA) was 99% for both elements. Cadmium was determined by ICP-AES and this technique does not have the sensitivity to detect cadmium at the levels present in the SRM. Analysis of cadmium by graphite furnace-AA will be specified in Phase II of this project.

The accuracy objectives of the round robin study were met for 50% of the detected trace elements. Elements meeting accuracy objectives were barium, chromium, cobalt, and vanadium. Certified values for boron, beryllium, fluorine, and mercury are not available for this SRM, so no accuracy measurements were performed for these elements in the round robin report. However, the results for these noncertified elements appear consistent with those from the other laboratories. Elements that did not meet the 90-110% recovery range were arsenic, cobalt (1 result), manganese, molybdenum, lead, antimony, and selenium. (Antimony, manganese and molybdenum SRM recovery values obtained during the Plant Yates study were well within the 90-110% objective of the round robin study. See Table D-2.)

One of the requirements of the round robin study was to report analytical results for the target analytes that were determined by the same methods used to report plant coal sample results. For the Yates project (and the coal round robin study), Radian performed multiple techniques for some elements (i.e., INAA vs. GFAA or ICP-AES) to provide comparative results, especially where questionable results by any one technique had been previously encountered. Performance evaluation (PE) audit samples (SRMs) were submitted for analysis by each method and the accuracy and precision were assessed before selecting the best qualified data for reporting and for use in material balance calculations.

Comments

One of the conclusions evident from the round robin study is that there is a high degree of variability and repeatability between methods, laboratories, and duplicate results for trace elements. Evidence of the variability in trace element analyses can be shown, for example, with neutron activation analysis where unacceptable results were reported for the analysis of the NIST SRM in the round robin study, but the same technique produced 90-110% recovery for the same elements in the NIST 1632b standard reference coal submitted as an audit sample during this project. This suggests that the performance of some techniques, like INAA, may vary substantially between repeated analysis and analytical batches. Neutron activation appears to be a cost effective analytical technique; however, as with all analytical techniques, the results must be evaluated on a case-by-case basis.

Although the round robin analysis is useful for indicating problematic methods and poor quality control, the project-specific quality control activities should be used for assessing the accuracy and precision of the coal analyses performed at each site.

Table D-3
Radian Lab analysis of Standard Reference Coal, 1632b

Parameter	Certified Value	Analytical Method	Average % Recovery	Run 1	Run 2
Ultimate/Proximate (% Dry Basis)					
Ash	6.80	D 3174	99.6	6.78	6.77
Carbon	78.11	D 5373	99.4	77.74	77.52
Hydrogen	5.07	D 5373	101.2	5.14	5.12
Nitrogen	1.56	D 5373	97.1	1.54	1.49
Sulfur	1.89	D 4239	140.7	1.93	3.39 ^a
Chlorine	0.126	D 4208	84.5	0.107	0.106
BTU/lb	13,890	D 2015	99.2	13,767	13,797
Major Ash Minerals					
SiO ₂	44.03	--	--	--	--
Al ₂ O ₃	23.75	INAA	98.5	24.37	22.43
TiO ₂	1.11	INAA	92.8	0.97	1.09
Fe ₂ O ₃	15.96	INAA	91.7	14.24	15.04
CaO	4.2	INAA	53.5	2.3 ^a	2.19 ^a
MgO	0.93	INAA	80.1	0.77	0.72 ^a
Na ₂ O	1.02	INAA	85.3	0.87	0.87
K ₂ O	1.33	INAA	74.1	1.07 ^a	0.9 ^a
P ₂ O ₅	--	ICP-AES	--	0.36	0.39
SO ₃	--	--	--	--	--

Table D-3 (Continued)

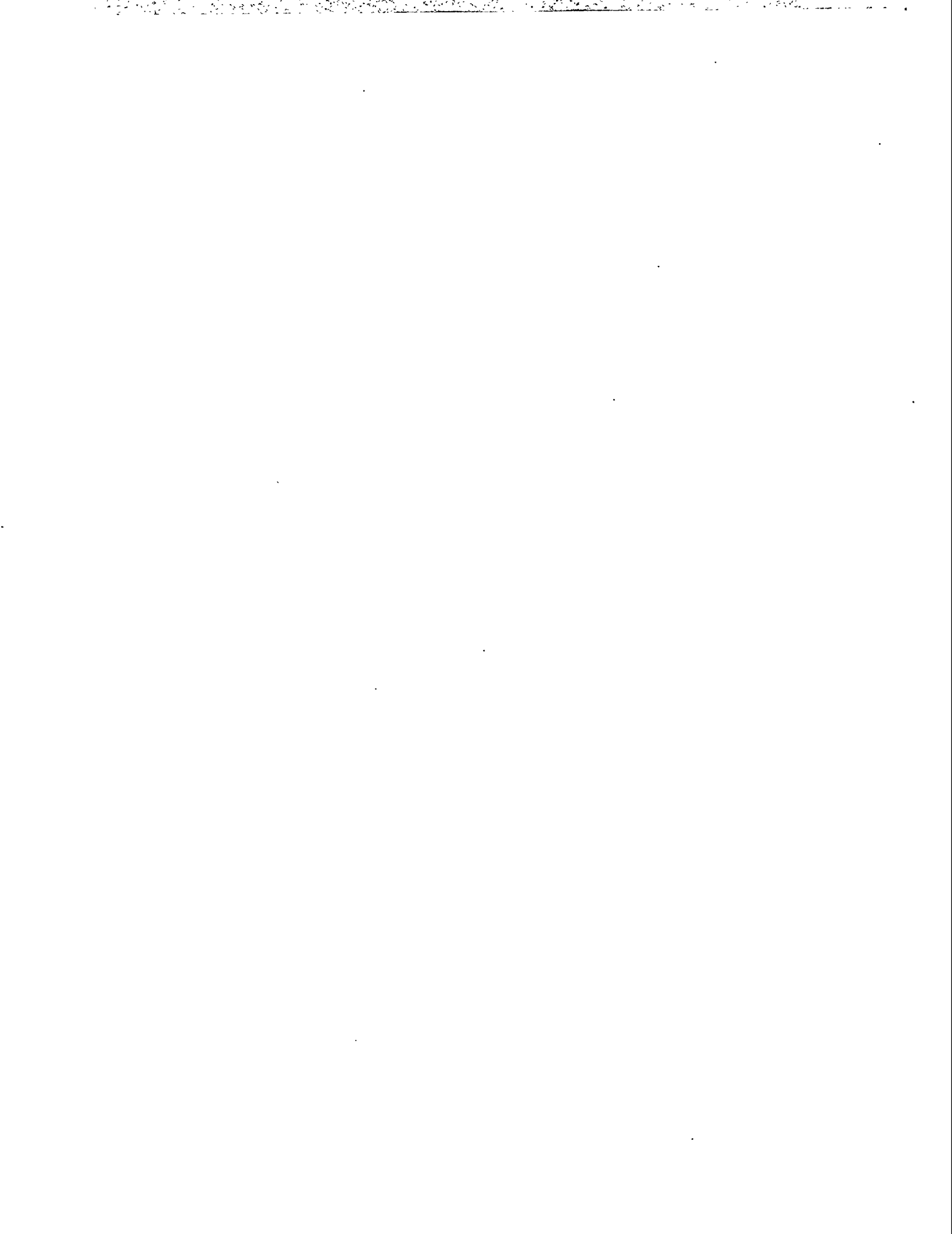
Parameter	Certified Value	Analytical Method	Average % Recovery	Run 1	Run 2
Trace Elements					
As	3.72	GF/AA	53.8	2 ^b	2 ^b
B	--	ICP-AES	--	61	60
Ba	67.5	INAA	106.6	71.2	72.7
Be	--	ICP-AES	--	0.6	0.6
Cd	0.0573	ICP-AES	--	<0.2	<0.2
Cr	11 ^d	INAA	96.4	11	10.2
Co	2.29	INAA	89.5	2.09	2.01 ^c
Cu	6.28	INAA	--	<35.3	<35.7
F	--	D 3761	--	40	40
Hg	--	DGA/CVAA	--	0.05	0.05
Mn	12.4	INAA	86.3	10.8 ^c	10.6 ^c
Mo	0.9 ^d	INAA	191.7	1.55 ^b	1.9 ^b
Ni	6.1	INAA	145.1	<8.8	<8.9
Pb	3.67	ICP-AES	81.7	3 ^c	3 ^c
Sb	0.24 ^d	INAA	81.3	0.196 ^c	0.194 ^c
Se	1.29	GF/AA	77.5	1 ^c	1 ^c
V	14 ^d	INAA	101.1	14.2	14.1

^a Results exceed ASTM reproducibility limits.

^b Results exceed certified values by more than 25 percent.

^c Results exceed certified values by more than 10 percent.

^d Informational value (not certified).



Interlaboratory Variability and Accuracy of Coal Analyses in the
U.S. Department of Energy Utility Air Toxics Assessment Program

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INTRODUCTION

The 1990 Clean Air Act Amendments (CAAA) empower the Environmental Protection Agency to set emission standards for a variety of potentially hazardous air pollutants from combustion sources. In order to define emissions from coal combustion sources, the U.S. Department of Energy (DOE) is coordinating an air toxics assessment program to characterize stack emissions from coal-fired utility boilers of volatile and semi-volatile organics, metals and anions specified in Title III of the Clean Air Act Amendments of 1990. The information from the DOE study will enable the Environmental Protection Agency to properly classify coal-fired utility boilers with regard to the CAAA and evaluate the potential risk to human health posed by these types of emission sources.

The first phase of DOE study consisted of sampling eight power plants. These plants represented a diverse range of boiler configurations, emission controls, and coal feeds. Part of the sampling protocol at each of the sites was to collect representative samples of the feed coal to the boiler. By analyzing the feed coal as well as all gas, solid, and water effluent streams, a material balance around each site could be established. A material balance closure near 100% would indicate that sampling and analyses of all streams was handled properly, and reliable emission estimates could be calculated.

Five laboratories participated in analyzing samples that were collected at the eight test sites. As part of the DOE program, CONSOL R&D conducted a coal analysis round robin among these laboratories. The primary purpose of this study was to estimate the analytical variability one can expect on trace element analyses when comparing results from the same laboratory or results from two or more laboratories.

Trace elements in coal generally are defined as those elements that occur at concentrations of 100 parts per million (ppm) or less. Seventeen trace elements were included in this study. Thirteen of these elements are listed in the 1990 CAAA as hazardous air pollutants. Earlier studies¹ have shown the interlaboratory variability of trace element analyses can be quite large. This analytical variability should be considered when determining the potential emissions from coal combustion sources.

The variability of other commonly measured coal quality parameters also was evaluated.

COAL SAMPLES

The coal samples used in the round robin study were supplied to CONSOL R&D by the prime contractor at each of the eight test sites. These were the same coals that were being fed to the boilers during the testing period at each site. The coals were geologically diverse and ranged from lignite to bituminous in rank. Once received, all sample reduction and preparation was according to ASTM D 2013 "Standard Method of Preparing Coal Samples for Analyses".² A spinning riffle was used to divide the gross sample prepared from each coal into homogenous splits. This is the preferred method in the coal industry to divide a sample of coal into several samples having the same composition and is widely used in commercially sponsored coal analyses round robin programs.

ROUND ROBIN DESIGN

Each participating laboratory was provided duplicate samples of each of the eight coals, along with a sample of a National Institute for Standard and Technology (NIST) certified reference coal. The samples were randomized and were identified only by code letters. Each laboratory was requested to analyze the samples in duplicate using the same procedures used to analyze the samples from the DOE Air Toxics Assessment programs. By using this round robin design, intra-laboratory repeatability and interlaboratory reproducibility, as well as individual laboratory precision, could be established. The suite of analyses included in this study is shown below:

<u>Proximate-Ultimate</u>	<u>Major Ash Elements</u>	<u>Trace Elements</u>	
Moisture	SiO ₂	As	Hg
Ash	Al ₂ O ₃	B	Mn
Carbon	TiO ₂	Ba	Mo
Hydrogen	Fe ₂ O ₃	Be	Ni
Nitrogen	CaO	Cd	Pb
Sulfur	MgO	Cr	Sb
Chlorine	Na ₂ O	Co	Se
Heating Value (Btu/lb)	K ₂ O	Cu	V
	P ₂ O ₅	F	
	SO ₃		

The average interlaboratory results for this suite of analyses for all eight samples are shown in Table 1. Individual laboratory results for all samples are presented in Appendix A. Samples identified as A&J and B&K are Illinois basin bituminous coals. Samples C&L, F&O, and H&Q are mid-sulfur bituminous coals. Sample D&M is a subbituminous coal from the Powder River basin. Sample G&P is also a subbituminous coal. Sample E&N is ranked as a lignite.

ANALYTICAL TECHNIQUES

The analytical techniques used by the participating laboratories to complete the suite of analysis in this study are shown in Table 2. No one parameter was measured by all laboratories by the same analytical technique. All of the labs used ASTM standard methods for the Proximate and Ultimate analyses. However, numerous techniques were used for the major ash and trace element analyses. The techniques included graphite furnace atomic absorption (GF/AA), inductively coupled plasma emission spectroscopy (ICP/ES), inductively coupled plasma mass spectroscopy (ICP/MS), instrumental neutron activation analyses (INAA), ion chromatography (IC), cold vapor atomic fluorescence (CV/AF), and X-ray fluorescence (XRF). Mercury was measured by gold amalgam cold vapor atomic absorption (GA/CVAA), double gold amalgam cold vapor atomic absorption (DGA/CVAA), and cold vapor atomic fluorescence (CV/AF). The techniques of AA, GF/AA, ICP/ES, ICP/MS, IC, and CVAA require that the analysis sample first be put into solution before being introduced into the instrument. INAA, XRF, GA/CVAA, and DGA/CVAA analyses can be performed on the whole coal or an ash sample of the coal.

ACCURACY

The accuracy of analyses performed by each laboratory was evaluated using the NIST Standard Reference Coal 1632b. This Pittsburgh seam coal is the most characterized standard reference material available from NIST. Certified or informational values are listed for all of the parameters included in this study except for boron, barium, fluorine, phosphorus, and mercury. For trace elements, all definitive results (" $<$ " values ignored) that fell within 10% of the certified or informational value arbitrarily were considered accurate values. Values outside this range were considered to be inaccurate. ASTM interlaboratory reproducibility limits were the criteria for accuracy on all other analyses. Table 3 shows the results reported by the each laboratory for NIST SRM 1632b. Using the previously described criteria for accuracy, the percentage of accurate results (accurate results/total definitive results) was calculated. Parameters without a certified or informational value were not included.

The table below shows the percentage of accurate results reported by each lab for the suite of trace elements, the percentage of accurate results for all analyses, and the percentage of trace element results that were reported as definitive. Although lab IV showed the highest percentage of accurate results (75%), that figure is based on only the 80% of definitive results reported by that laboratory.

As shown in the table below, the percentage of accurate trace element analyses ranged from 38% to 75%. Non-definitive results reported for antimony, cadmium, copper, fluorine, molybdenum, nickel, and selenium. Only one laboratory reported definitive results for the entire suite of trace elements. The most troublesome elements, with respect to accuracy, were arsenic, cadmium, molybdenum, antimony, and selenium. Only one lab reported accurate results for cadmium, molybdenum or antimony.

The Proximate and Ultimate analyses reported by labs II, III, IV, and V were all within ASTM reproducibility limits except for a single sulfur analysis. Lab I reported results that exceeded ASTM reproducibility limits for hydrogen, nitrogen, sulfur chlorine and heating value. Two labs reported all major ash elements within ASTM limits. Lab I exceeded limits for silicon, iron, calcium, magnesium, and potassium. Lab III exceeded limits for calcium, magnesium, and potassium. Lab IV performed only a limited number of major ash element analyses, but reported results for aluminum and potassium that were outside established ASTM reproducibility limits.

% ACCURATE RESULTS ON NIST 1632b

<u>Lab</u>	<u>Definitive Trace Element Results</u>	<u>Trace Elements</u>	<u>All Analyses</u>
I	88	38	43
II	100	73	88
III	82	50	63
IV	80	75	80
V	100	48	78

REPRODUCIBILITY

The percent relative standard deviation (PRSD) of the analytical results was chosen to represent interlaboratory reproducibility in this study. Table 4 shows the average PRSD for all labs, on all samples, for the entire suite of analyses. Reproducibility for trace elements ranged from 11.0 PRSD for vanadium to 60.7 PRSD for molybdenum. The average PRSD for all of the trace elements (all coals, all labs) was 27.9%. In most cases the PRSDs for cadmium, copper and antimony are based on results from only three laboratories. These elements were either below detection limits at laboratories II and III or were not determined.

Excluding Lab I's results, Proximate and Ultimate analyses were generally within ASTM limits. Aside from the determination of percent ash this particular laboratory reported only a single sulfur analyses on the standard reference material that was within established ASTM limits. Chlorine, although not generally considered a trace element in coal, is listed in the 1990 CAAA as a hazardous air pollutant. It showed an average PRSD for all labs of 37.2 %. Three of the coals sampled in the study are ranked subbituminous or lignites. Chlorine on these samples (D&M, E&N, G&P) was reported as below detection limits (0.01 and 0.02%) by two laboratories and not determined by another laboratory. Therefore, the PRSD for these three samples was calculated with data from only two labs. The reproducibility estimates for chlorine may have been larger if more labs had reported data.

Major ash elements were determined with an average PRSD of 21.7%. This is only slightly better than the average PRSD of 27.9% for trace elements. Phosphorous, calcium, and magnesium had PRSDs greater than 35%. Including only labs II and V, the overall average PRSD for the major ash elements drops to 7%. These were the only labs that did not exceed ASTM limits on the certified reference material. Labs I and III showed a consistent low bias for calcium and magnesium on most samples as well as on the certified reference material. Lab I showed poor intralaboratory repeatability for most major ash elements.

Figure 1 shows the interlaboratory reproducibility as PRSD for the suite of trace elements on all samples. The overall average PRSDs for V, F, Be, Mn, B, Hg, Cu, Sb, and Cr, and Ba are between 9.6 and 22.9%. PRSDs for Ba, Co, Ni, and Se were somewhat poorer, averaging nearly 30%. Ni, As, Cd, Pb and Mo showed the most variability with PRSDs from 36.2 to 60.7%.

Figure 2 shows the average interlaboratory reproducibility for the suite of trace elements, as well as the range of PRSDs, for each element on each sample. Although the average PRSD for many elements is reasonably good (~20%), on any given sample the range of reported values can be quite large. The average minimum PRSD for interlaboratory trace element analyses was 13.6%. The average maximum was 48.1%. Ba, Cd, Cu, Hg, Mo, Ni, Pb and Sb all had a PRSD range over 30%. The range of reported values for Mo, Ni, and Cd on some samples was 52%, 76%, and 110% respectively. This shows that outliers are to be expected when comparing trace element analyses between laboratories.

REPEATABILITY

Figure 3 shows the average intralaboratory repeatability for each trace element for all coals. Intralaboratory repeatability was calculated as the average percent difference in a given

laboratory's results on the eight paired samples. The data show that the overall laboratory repeatability on trace elements ranged from a low of 7.8% for chromium to a high of 32.5% for cadmium. The average repeatability for all trace elements was 14.6%. Overall intralaboratory repeatability for all elements by all labs was less than 10% on half of the analyses, less than 20% on 68%, and less than 30% on 75% of all trace element results. In general, elements with lower between-lab reproducibility also had lower same-lab repeatability. Similarly, elements like cadmium, that showed reproducibilities with a high PRSD, had higher average repeatabilities, with the exception of molybdenum. This element had a relatively low repeatability (16.8%), but showed the highest reproducibility (60.7%). This may suggest bias in the various methods used for its determination. Data showing the complete list of individual laboratory repeatability for all samples is presented in Appendix B.

VARIABILITY vs COAL RANK

Figure 4 shows the variability in interlaboratory trace element analyses as PRSD plotted as a function of the as-determined heating value for the eight coals. The as-determined heating value of a coal is one way to roughly establish coal rank. The data clearly show that trace element analytical variability is a function of coal rank, increasing as the coal rank decreases. This is not unusual; many ASTM coal standards have precision statements that are rank-dependant. In the case of the eight coals studied here, as the heating value of the coal (Btu/lb) decreases, the analytical variability of trace elements increases. Sample pairs A&J, C&L, H&Q, F&O, and B&K are bituminous coals. Samples G&P and D&M are subbituminous and samples E&N are classified as lignites. A regression analyses of the data is shown in Figure 5 and has an r^2 value of 0.95. Average trace element intralaboratory repeatability showed a similar trend. The overall trace element repeatability for the bituminous coals was slightly better (14.8%) than that for the subbituminous and lignite samples (20.2%).

MERCURY

Of the potential hazardous air pollutants mentioned in the CAAA, mercury is receiving the most attention regarding possible emissions from coal combustion sources. As mentioned earlier, four of the five laboratories in this study used some form of gold amalgamation followed by cold vapor atomic absorption for mercury analyses, the other used cold vapor atomic fluorescence. The table below summarizes intralaboratory repeatability and interlaboratory reproducibility for mercury analyses. Repeatability is shown as the percent difference in a laboratory's results on the eight paired samples, and reproducibility is shown as PRSDs.

REPEATABILITY AND REPRODUCIBILITY OF MERCURY RESULTS

	<u>A&J</u>	<u>B&K</u>	<u>C&L</u>	<u>D&M</u>	<u>E&N</u>	<u>F&O</u>	<u>G&P</u>	<u>H&Q</u>	<u>Avg.</u>
Repeatability, as % difference	11.3	46.3	19.1	19.1	25.8	11.7	8.6	21.2	17.6
Reproducibility, as PRSD	10.4	40.6	24.8	16.7	16.9	20.4	9.1	26.1	20.6

A recent, more extensive round robin on mercury analyses³ estimated interlaboratory reproducibility and intralaboratory repeatability at 25 and 50%, respectively. That particular round robin

involved three coal samples and 12 laboratories. Although the majority of laboratories in that study also used cold vapor atomic absorption for mercury analyses, some data were provided by labs using neutron activation and cold vapor atomic fluorescence.

SUMMARY AND CONCLUSIONS

Based on the analyses of the certified reference coal, even the best laboratory in this study reported trace element levels to within 10% of their certified value only about 80% of the time. On average, only 57% of the reported data from all labs met this 10% level of accuracy.

The techniques used in many laboratories for trace element analyses produced a significant number of non-definitive (" $<$ ") results. If certain detection limits are required, analytical techniques must be specified.

Although the overall interlaboratory trace element reproducibility is 28%, it may be very poor, approaching 60% for some elements.

Interlaboratory reproducibility for trace element analyses is dependent on coal rank. As coal rank decreases, analytical variability increases.

The variability of coal trace element analyses makes accurate estimates of emissions from combustion sources difficult, especially if the estimates are based solely on feed coal analyses.

RECOMMENDATIONS FOR CONDUCTING FUTURE COAL ANALYSES ROUND ROBIN PROGRAM

1. Follow ASTM standard method E 691. This standard lists specific guidelines for conducting an interlaboratory coal analysis round robin program. The standard also specifies software for the statistical interpretation of results. Both the method and the software are available from ASTM for a nominal fee. One of the guidelines violated in this round robin was the number of participating laboratories. E 691 states that a minimum of six laboratories is necessary to generate ASTM precision statements. For that reason we were unable to use the software from this standard that would have generated ASTM limits for repeatability and reproducibility.
2. Laboratories that are candidates for the round robin should be evaluated. Based on the data reported on the standard reference coal in this study, it is obvious that Lab I was not proficient with coal analyses. Laboratories that are candidates for round robins should be audited by someone familiar with the guidelines set forth in ASTM D 4182, "Evaluation of Laboratories Using ASTM Procedures in the Sampling and Analysis of Coal and Coke". These labs also should be able to demonstrate their ability to conform with ASTM D 4621, "Accountability and Quality Control in the Coal Analysis Laboratory". A lab not in compliance with either of the standards should not be included in the study. As a minimum, candidate labs should be able to demonstrate proficiency by analyzing a certified reference material within specified precision limits prior to conducting the actual round robin.
3. Specify the minimum detection limits that are required for each element. Based on the large number of non-definitive results reported for several of the trace elements it is apparent that

most laboratories are not using techniques that can accurately assess the levels of some of the trace elements found in coal. Using half the detection limit, which is the common practice for treating this type of result, would lead to a considerable overestimation of some trace element levels. Examples of this overestimation based on half the detection limit are found in Table 3. For instance, Lab III reported an average detection limit for Cu as 35.5 ppm. Using one half of this value, or 17.8 ppm, would overstate the certified value for Cu on this sample by nearly three fold.

REFERENCES

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2. "ASTM Volume 05.05 Gaseous Fuels; Coal and Coke", American Society for Testing and Materials, Philadelphia, PA, 1993.
3. Lengyel, John Jr., Devito, M. S., and Bilonick, R. A. "Interlaboratory and Intralaboratory Variability in The Analyses of Mercury In Coal". Paper to be presented at the Air Waste Management Association Annual Meeting, Cincinnati, OH, 6/19-24/94.

Table 1. Average of Interlaboratory Results for All Samples.

	<u>A&J</u> <u>IL BASIN</u>	<u>B&K</u> <u>IL BASIN</u>	<u>C&L</u> <u>BIT.</u>	<u>D&M</u> <u>PRB</u>	<u>E&N</u> <u>ND LIG.</u>	<u>F&O</u> <u>BIT.</u>	<u>G&P</u> <u>SUB. BIT.</u>	<u>H&Q</u> <u>BIT.</u>
<u>Trace Elements</u> ppm Dry Coal								
As	2.39	2.74	9.43	1.24	7.64	26.0	1.70	3.45
B	227	212	72.3	83.4	126	70.7	76.5	169
Ba	47.3	48.9	31.1	370	568	76.1	312	48.6
Be	1.33	1.61	1.33	0.42	0.72	2.37	1.29	1.41
Cd	0.580	1.013	0.112	0.058	0.079	0.085	0.560	0.508
Cr	28.3	34.7	16.3	4.40	8.05	20.0	9.61	21.4
Co	3.87	3.57	5.50	0.86	2.10	6.95	4.14	4.42
Cu	10.7	11.3	8.47	9.52	9.28	21.2	14.5	13.1
F	97.1	112	58.0	44.3	56.9	81.3	80.3	79.5
Hg	0.101	0.109	0.126	0.084	0.145	0.260	0.080	0.085
Mn	41.3	34.3	18.4	145	123	26.5	76.6	29.0
Mo	8.34	7.91	1.87	7.93	3.98	4.54	2.11	5.80
Ni	17.6	18.5	14.1	5.09	7.26	28.2	6.84	18.3
Pb	9.12	13.1	6.00	5.22	3.31	13.6	8.86	8.47
Sb	0.49	0.79	0.64	0.47	0.75	2.10	1.74	0.62
Se	2.94	3.16	1.92	0.84	0.80	2.56	1.18	2.21
V	36.6	46.3	31.0	9.36	16.8	34.0	26.1	38.5
<u>Proximate & Ultimate</u> % Dry Basis								
Ash	11.99	12.54	11.56	11.7	16.71	13.35	20.57	10.59
Carbon	69.58	69.80	72.08	67.6	58.80	70.26	61.27	71.03
Hydrogen	4.87	4.78	4.96	4.80	4.53	4.86	4.78	5.14
Nitrogen	1.33	1.33	1.39	1.01	0.89	1.37	1.05	1.42
Sulfur	3.42	3.53	3.26	1.15	1.12	3.01	0.65	2.89
Chlorine	0.064	0.074	0.085	0.03	0.040	0.140	0.039	0.115
Heating Valt	12214	12189	12888	11350	9601	12452	10636	12587
<u>Major Ash Elements</u> % Dry Ash								
SiO ₂	44.58	49.7	44.98	42.12	39.48	45.67	59.26	51.55
Al ₂ O ₃	16.78	18.6	21.41	16.48	10.58	22.54	20.62	21.74
TiO ₂	0.89	0.99	0.99	0.88	0.47	1.22	1.00	1.03
Fe ₂ O ₃	15.95	15.1	24.75	6.07	6.14	21.33	4.45	16.39
CaO	4.11	2.69	1.04	7.79	10.54	1.50	3.29	2.46
MgO	0.77	0.82	0.60	2.55	2.97	0.73	0.93	0.79
Na ₂ O ₃	0.91	0.75	0.43	0.29	0.84	0.30	0.23	0.84
K ₂ O	1.95	2.20	1.84	0.51	1.35	2.17	1.26	2.50
P ₂ O ₅	0.29	0.36	0.16	0.37	0.17	0.58	0.04	0.26
SO ₃	4.57	2.61	1.39	11.41	15.08	1.71	3.68	2.56

Table 2. Analytical Methods Used on DOE Air Toxics Assessment Coal Samples.

Parameter	Lab I	Lab II	Lab III	Lab IV	Lab V
Moisture	D3173	D 5142	D 3173	D 3173	D 3173
Ash	D3174	D 5142	D 3174	D 3174	D 3174
Carbon	D3178	D 5373	D 5373	D 3178	D 5373
Hydrogen	D3178	D 5373	D 5373	D 3178	D 5373
Nitrogen	D3179	D 5373	D 5373	D 3179	D 5373
Sulfur	D3177	D 4239	D 4239	D 4239	D 4239
Chlorine	D4208	LECO	D 4208	***IC	D 4208
Btu/lb	D2015	D 1989	D 2015	D 2015	D 2015
Major Ash Elements					
SiO ₂	ICP/ES	ICP/ES	ND	D 4326 XRF	ICP/ES
Al ₂ O ₃	"	"	INAA	"	"
TiO ₂	"	"	"	"	"
Fe ₂ O ₃	"	"	"	ND	"
CaO	"	"	"	ND	"
MgO	"	"	"	ND	"
NaO	"	"	"	D 4326 XRF	"
K ₂ O ₃	"	"	"	"	"
P ₂ O ₅	"	"	ICP/ES	ND	"
SO ₃	"	"	ND	ND	"
Trace Elements					
As	GF/AA	ICP/MS	GF/AA	GF/AA	CV/AF
B	ICP/ES	"	ICP/ES	ICP/ES	ICP/ES
Ba	"	ICP/ES	INAA	"	"
Be	"	ICP/MS	ICP/ES	"	"
Cd	AA	"	"	"	GF/AA
Cr	ICP/ES	"	INAA	"	ICP/ES
Co	"	"	"	"	"
Cu	"	"	"	"	"
Cu	"	"	"	"	"
F	D3761	*IC	D 3761	***IC	"
Hg	CVAA	DGA/CVAA	DGA/CVAA	GA/CVAA	CV/AF
Mn	ICP/ES	ICP/MS	INAA	ICP/ES	ICP/ES
Mo	"	"	"	"	"
Ni	"	"	"	"	"
Pb	AA	"	ICP/ES	GF/AA	GF/AA
Sb	GF/AA	"	INAA	"	CV/AF
Se	GF/AA	**#ICP/MS	GF/AA	"	"
V	ICP/ES	ICP/MS	INAA	ICP/ES	ICP/ES

*IC Hydropyrolysis with IC Finish

**#ICP/MS Hydropyrolysis with ICP/MS Finish

***IC-Soluble Species Only

AA	Atomic Absorption	ICP/ES	Inductively Coupled Plasma Emission Spectroscopy
CVAA	Cold Vapor Atomic Absorption	ICP/MS	Inductively Coupled Plasma Mass Spectroscopy
CV/AF	Cold Vapor Atomic Fluorescence	INAA	Instrumental Neutron Activation Analyses
DGA/CVAA	Double Gold Amalgam Cold Vapor Atomic Absorption	ND	Not Determined
GF/AA	Graphite Furnace Atomic Absorption	XRF	X-ray Fluorescence
IC	Ion Chromatography		

Table 3. Individual Laboratory Analyses of National Institute of Standard and Technology, Standard Reference Coal 1632b.

PARAMETER	CERTIFIED VALUE		LAB I		LAB II		LAB III		LAB IV		LAB V	
	Run_1	Run_2	Run_1	Run_2	Run_1	Run_2	Run_1	Run_2	Run_1	Run_2	Run_1	Run_2
Parts Per Million, Dry Coal												
As	3.04	3.54	3.71	3.87	2	2	2	2	2	2	4.1	3.1
B	87.1	81.0	52.0	51.0	61	60	60	60	55	55	31.1	18.6
Ba	67.5	48.5	66.0	66.4	71.2	72.7	72.7	72.7	63	70	67.2	67.6
Be	0.0573	0.82	0.58	0.59	0.8	0.6	0.6	0.6	0.5	0.6	0.693	0.668
Cd	0.27	0.41	0.040	0.060	<0.2	<0.2	<0.2	<0.2	<0.2	<0.2	0.028	ND
Cr	11.1	8.7	9.84	9.84	11	10.2	10.2	10.2	9	11	7.9	8.1
Co	4.15	2.83	1.89	2.06	2.09	2.01	2.01	2.01	2	2	2.18	1.42
Cu	6.07	5.87	5.4	5.86	<35.3	<35.7	<35.7	<35.7	6	6	8.4	8.6
F	<100	<100	43.0	35.9	40	40	40	40	ND	ND	41	41
Hg	0.17	0.15	0.099	0.095	0.05	0.05	0.05	0.05	0.05	0.07	0.057	0.062
Mn	12.4	11.1	11.1	12	12	10.6	10.6	10.6	12	13	10.9	11.1
Mo	*0.9	6.38	2.63	0.9	1.55	1.8	1.8	1.8	<3	<3	ND	ND
Ni	8.91	7.39	5.65	6.23	<8.8	<8.9	<8.9	<8.9	6	7	5.2	6
Pb	3.44	3.44	3.81	4.03	3	3	3	3	4	4	3.2	1.8
Sb	<0.8	<0.8	0.22	0.23	0.196	0.194	0.194	0.194	<1	<1	ND	ND
Se	1.42	0.75	1.46	1.52	1	1	1	1	1	1	1.23	0.751
V	*14	19.2	11.0	12.7	14.2	14.1	14.1	14.1	14	15	15.3	14.8
wt %, Dry Coal												
Ash	6.80	6.92	6.91	6.91	6.78	6.77	6.77	6.77	6.78	6.79	6.78	6.83
Carbon	78.11	76.43	77.93	78.39	77.74	77.52	77.52	77.52	76.89	76.72	77.62	77.2
Hydrogen	5.07	6.78	5.03	4.99	5.14	5.12	5.12	5.12	4.94	4.94	5.06	5.01
Nitrogen	1.56	0.47	1.54	1.6	1.54	1.49	1.49	1.49	1.5	1.56	1.46	1.41
Sulfur	1.89	2.27	1.89	1.92	1.93	3.39	3.39	3.39	1.96	1.95	1.92	1.91
Chlorine	0.126	0.030	0.112	0.109	0.107	0.106	0.106	0.106	ND	ND	0.12	0.12
Btu/lb	13890	12788	13809	13809	13767	13767	13767	13767	13760	13763	13774	13778
Underlined results exceed ASTM reproducibility limits												
SiO ₂	44.03	50.15	47.08	45.41	ND	ND	ND	ND	44.62	ND	44.4	43.8
Al ₂ O ₃	23.75	25.7	23.79	25.06	24.37	22.43	22.43	22.43	18.37	ND	24.3	24.2
TiO ₂	1.11	1.37	1.25	1.1	0.97	1.09	1.09	1.09	0.94	ND	1	0.8
Fe ₂ O ₃	15.96	17.99	16.75	17.03	14.24	15.04	15.04	15.04	ND	ND	16.4	16
CaO	4.2	1.72	4.64	4.63	2.3	2.19	2.19	2.19	ND	ND	4.2	4.2
MgO	0.93	0.24	1.03	1.02	0.77	0.72	0.72	0.72	ND	ND	0.95	0.97
Na ₂ O	1.02	1.12	1.05	1.04	0.87	0.87	0.87	0.87	1.01	ND	1	1
K ₂ O	1.33	1.8	1.24	1.39	1.07	0.9	0.9	0.9	2.05	ND	1.3	1.3
P ₂ O ₅		0.29	0.24	0.18	0.36	0.39	0.39	0.39	ND	ND	0.23	0.23
SO ₂		ND	ND	4.15	ND	ND	ND	ND	ND	ND	4.68	4.54
Underlined results exceed ASTM reproducibility limits												

Single--underlined results exceed certified values by more than 10%
 Double--underlined results exceed certified values by more than 25%
 * Informational Value
 ND=Not Determined

Table 4. Percent Relative Standard Deviation for All Samples.

Trace Elements	A&J	B&K	C&L	D&M	E&N	F&O	G&P	H&Q	Average PRSD	Maximum PRSD	Minimum PRSD
As	24.3	37.7	36.2	40.4	43.7	39.5	39.3	29.3	36.2	49.7	24.3
B	14.6	14.7	16.0	33.8	35.0	18.3	16.7	21.6	21.3	35.0	14.6
Ba	37.7	21.5	20.6	53.4	34.7	26.6	45.0	14.4	31.7	53.4	14.4
Be	11.4	15.9	15.7	17.9	17.0	11.8	8.40	21.7	15.0	17.9	8.4
Cd	35.1	58.4	32.0	62.9	38.6	39.0	142	57.9	58.3	142	32.0
Cr	9.91	4.34	19.0	19.2	13.0	20.9	14.1	14.3	14.3	20.9	4.34
Co	29.0	21.4	30.6	43.1	45.5	27.8	25.4	40.3	32.9	45.5	21.4
Cu	17.7	17.7	19.8	22.1	29.7	12.5	49.2	14.8	22.9	49.2	12.5
F	16.0	14.9	7.75	14.4	7.7	15.4	16.8	9.83	12.9	16.8	7.7
Hg	10.4	40.6	24.8	16.7	16.9	20.4	9.05	26.1	20.6	40.6	9.05
Mn	24.4	14.0	10.1	19.1	17.1	11.3	20.1	13.0	16.1	24.4	10.1
Mo	51.6	53.6	46.9	55.1	88.0	46.3	97	47.5	60.7	97	46.3
Ni	15.5	13.9	15.4	89.8	49.9	38.2	17.5	25.0	33.1	89.8	13.9
Pb	34.8	29.9	43.8	27.2	63.6	33.7	22.6	38.5	36.8	63.6	22.6
Sb	5.86	35.2	7.59	4.98	36.9	11.6	44.0	25.0	21.4	44.0	5.86
Se	20.5	37.6	25.6	28.2	33.9	24.0	33.8	26.1	28.7	37.6	20.5
V	11.5	9.4	13.7	9.31	8.73	13.9	6.07	15.3	11.0	13.9	6.07
AVG.	21.8	25.9	22.7	32.8	34.1	24.1	35.7	25.9	27.9	49.1	16.1
<u>Proximate & Ultimate</u>											
Ash	1.48	0.66	0.56	1.68	2.66	0.68	0.66	0.72	1.14	10.4	0.66
Carbon	0.85	2.71	0.82	1.97	2.15	2.07	1.87	2.78	1.90	10.4	0.85
Hydrogen	5.79	4.01	4.66	14.0	23.9	4.50	7.65	11.4	9.49	10.4	4.01
Nitrogen	5.42	3.70	3.21	8.60	19.5	3.24	4.93	4.11	6.58	10.4	3.21
Sulfur	6.78	4.45	4.01	53.3	3.50	2.42	16.0	4.75	11.9	10.4	4.01
Chlorine	23.1	31.0	25.4	46.0	25.0	60.5	58.1	28.5	37.2	10.4	25.4
Heating Value, Btu/lb	4.89	3.44	1.58	6.79	6.35	3.55	4.39	3.53	4.32	10.4	1.58
AVG.	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4	10.4
<u>Major Ash Elements</u>											
SiO ₂	17.71	3.38	1.93	3.27	3.18	3.09	3.25	10.7	5.81	10.7	1.93
Al ₂ O ₃	17.08	4.60	4.2	26.0	34.9	4.72	11.4	7.23	13.8	10.7	4.2
TiO ₂	8.77	25.7	5.58	15.6	5.67	32.0	16.3	30.0	17.5	10.7	5.58
Fe ₂ O ₃	7.74	36.8	5.71	15.2	16.1	3.43	7.40	10.8	12.9	10.7	5.71
CaO	50.7	38.5	27.2	62.2	63.9	21.5	37.9	30.6	41.6	10.7	21.5
MgO	43.7	42.4	30.7	60.2	66.2	28.8	47.5	38.1	44.7	10.7	30.7
Na ₂ O	9.35	14.1	13.7	12.2	7.84	26.0	26.3	9.64	14.9	10.7	13.7
K ₂ O	13.1	7.38	7.80	18.3	23.2	8.94	8.38	12.2	12.4	10.7	7.80
P ₂ O ₅	34.9	30.2	37.2	38.7	39.3	33.5	37.8	31.0	35.3	10.7	37.2
SO ₃	36.6	32.4	17.8	29.1	3.37	8.01	4.18	12.8	18.0	10.7	17.8
AVG.	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7	21.7

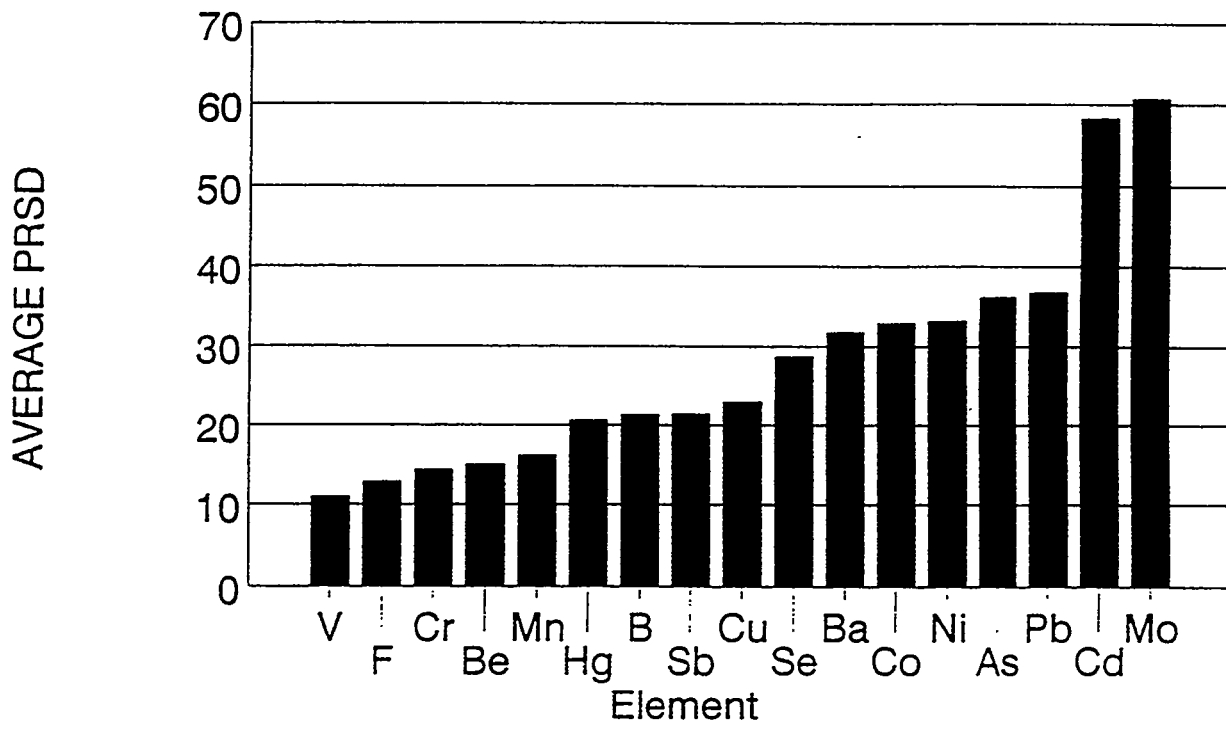


Figure 1. Average Variability for All Coals.

Bars Show Range, Dash Shows Average For All Coals

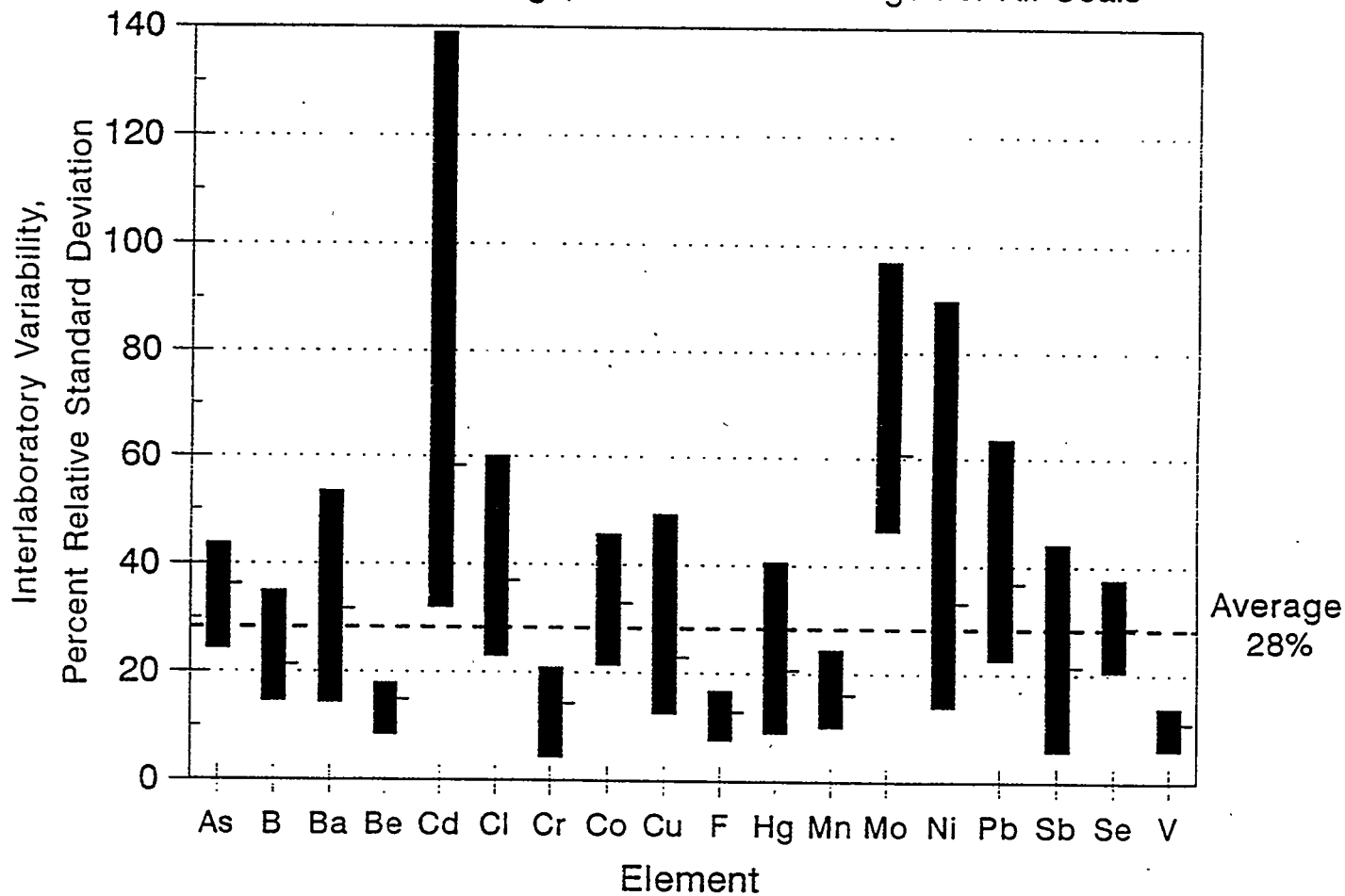


Figure 2. Interlaboratory Variability by Element

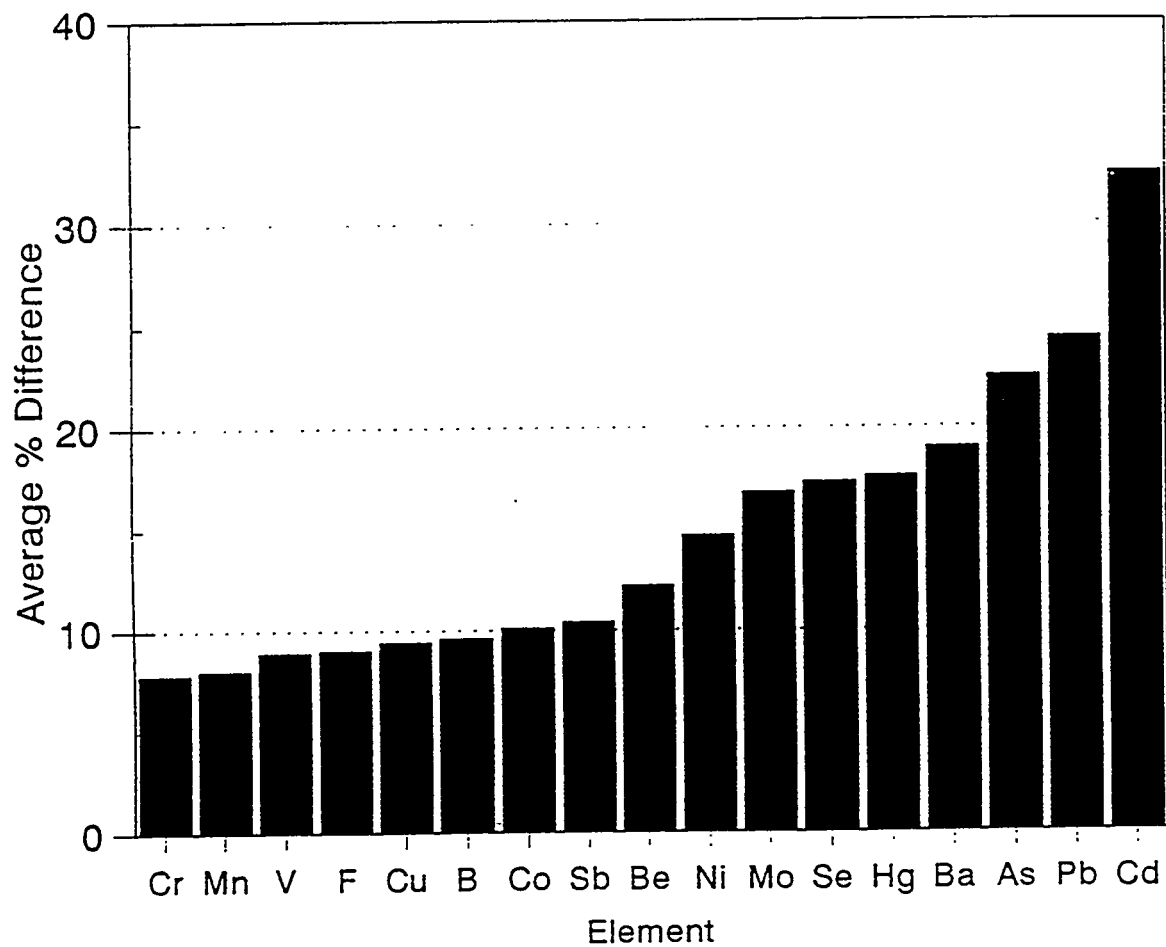


Figure 3. Average Interlaboratory Repeatability.

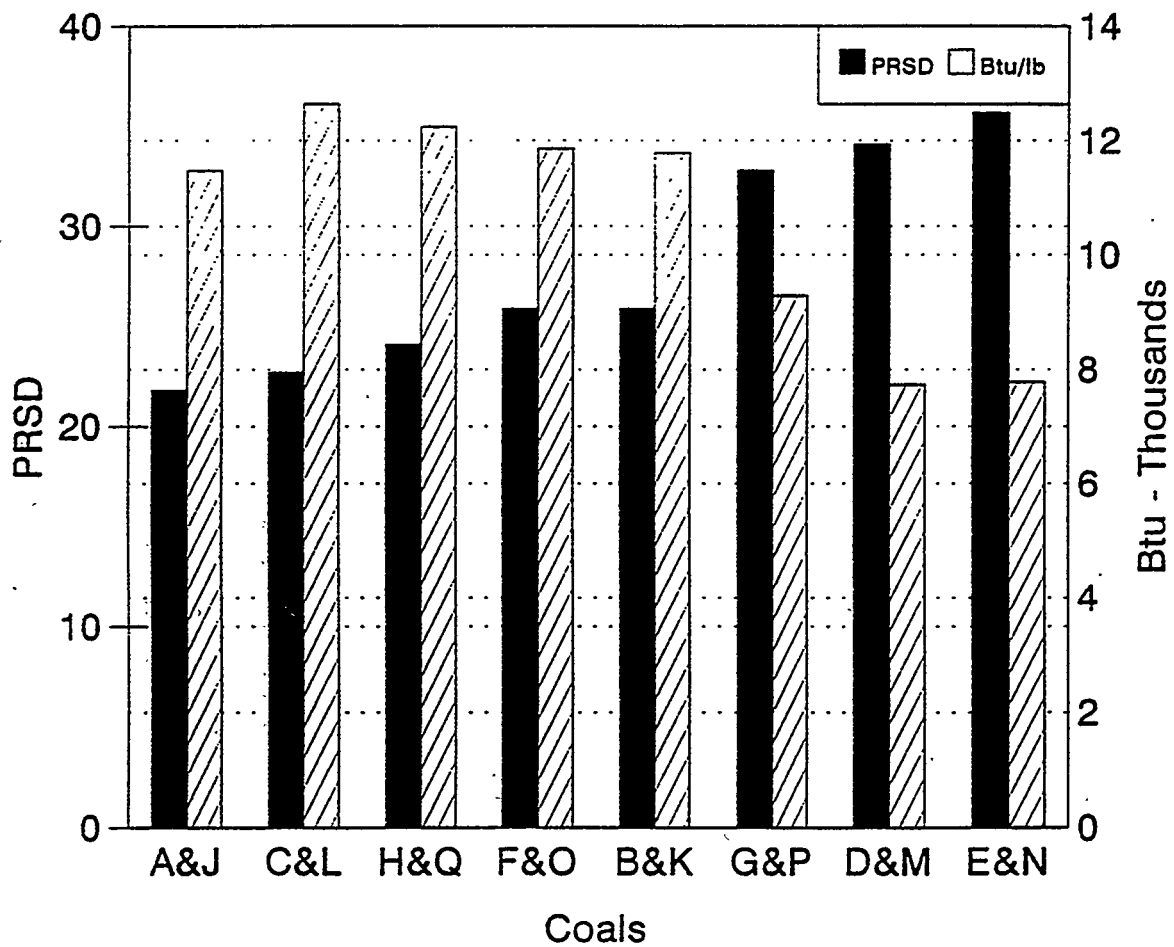


Figure 4. Comparison of Interlaboratory Variability vs. Heating Value.

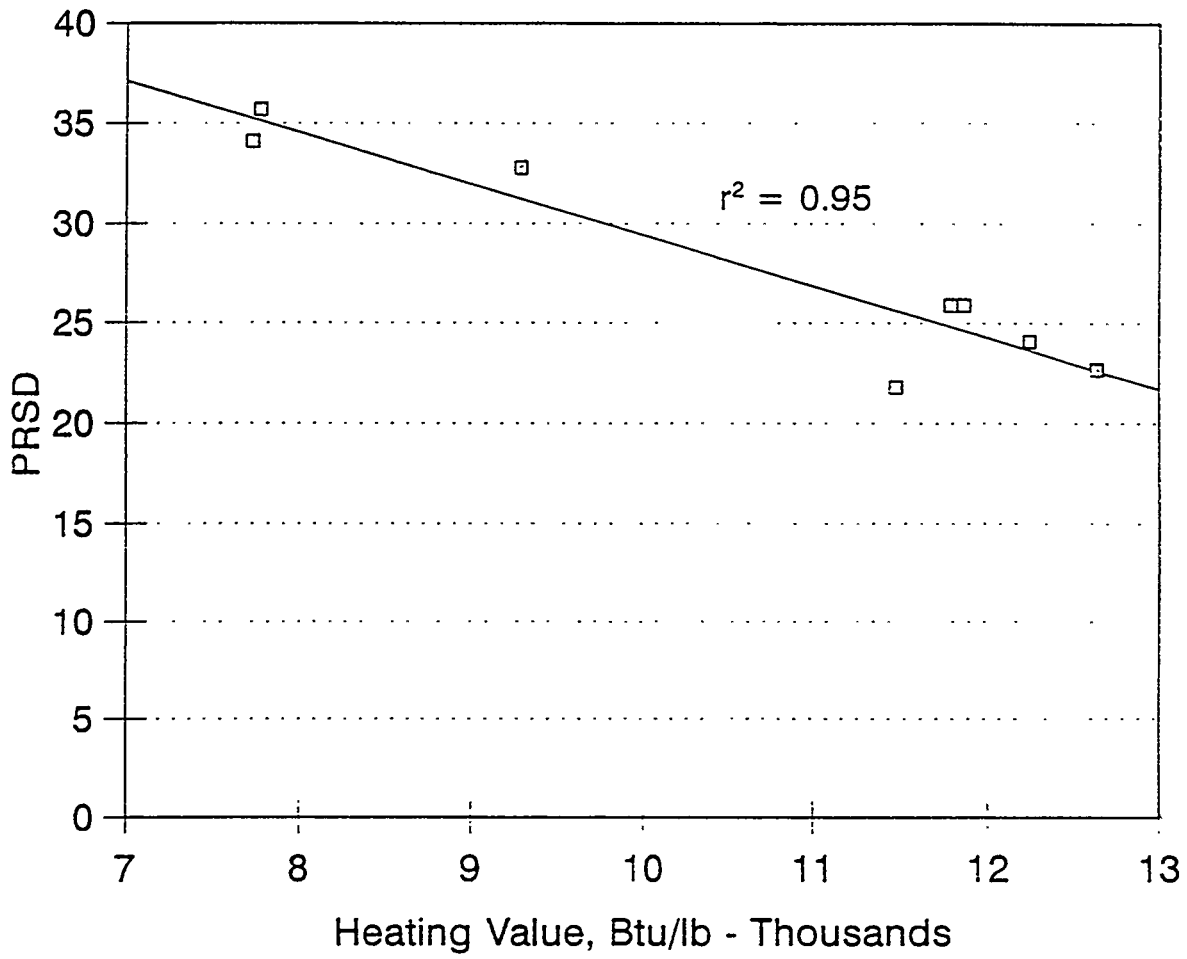


Figure 5. Correlation of Variability vs. Heating Value.

APPENDIX A

**INDIVIDUAL LABORATORY ANALYSIS
OF ROUND ROBIN SAMPLES**

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE A

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	2.36	3.01	3.43	3.46	2	2	2	2	2.6	2
B	236.33	290.64	179.81	182.77	250	230	230	230	276	217
Ba	29	32.29	55.8	63.3	76.7	72	58	55	49.4	49.8
Be	1.29	1.61	1.1	1.09	1.3	1.3	1.4	1.3	1.49	1.35
Cd	0.5	0.55	0.57	0.59	<0.3	<0.3	<0.4	<0.4	0.373	0.545
Cr	25.78	30.14	26.56	24.44	25.2	28	35	34	24.6	25.5
Co	4.73	5.81	3.41	3.35	3.28	3.44	5	5	2.71	3.79
Cu	8.7	10.76	8.76	8.54	<37.3	<38.3	12	12	13.2	13.1
F	<100	<100	108.92	107.67	100	100	ND	ND	72	67
Hg	<0.1	<0.1	0.094	0.095	0.09	0.09	0.1	0.1	0.123	0.109
Mn	36.52	39.89	51.8	51.8	29.3	31.2	53	55	42.8	41.7
Mo	11.82	8.61	7.01	6.78	15.1	14.6	6	<6	6.5	5.1
Ni	17.19	19.38	16.32	15.28	12.8	14	23	21	18	17.6
Pb	4.08	6.89	10.89	10.71	8	8	13	12	6.7	6.8
Sb	<0.8	<0.8	0.52	0.53	0.466	0.512	<1	<1	ND	ND
Se	3.01	3.55	3.58	3.61	3	2	3	ND	1.3	2.9
V	35.45	40.9	29.03	28.9	34.3	35.8	45	41	40.1	38.1

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	12.16	12.18	12.08	12.13	12.18	12.26	11.83	11.75	11.86	11.95
CARBON	66.39	68.88	70.09	70.22	70.32	70.15	69.2	69.25	69.2	69.14
HYDROGEN	4.97	5.88	4.83	4.79	4.89	4.92	4.67	4.6	4.76	4.71
NITROGEN	1.25	1.26	1.37	1.41	1.48	1.4	1.35	1.31	1.25	1.28
SULFUR	3.44	3.6	3.44	3.48	3.46	3.42	3.41	3.4	3.43	3.4
CHLORINE	0.06	0.05	0.077	0.064	0.075	0.072	ND	ND	0.1	0.09
Btu/lb	12460	11297	12478	12462	12425	12477	12453	12455	12427	12402

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	39.75	49.24	48.71	48.87	ND	ND	47.03	ND	45.9	46.8
Al ₂ O ₃	13.18	15.36	18.5	18.84	16.37	17.74	18.21	ND	17.9	17.8
TiO ₂	0.8	0.97	0.95	0.95	0.72	0.7	0.89	ND	0.9	0.8
Fe ₂ O ₃	13.89	16.43	17.27	17.35	15.21	15.17	ND	ND	17	17.3
CaO	2.47	2.23	6.48	6.43	2.36	2.21	ND	ND	5.5	5.9
MgO	0.4	0.35	1.06	1.06	0.59	0.72	ND	ND	1.1	1
Na ₂ O ₃	0.77	0.96	0.95	0.95	0.78	0.78	0.97	ND	0.99	0.97
K ₂ O	1.92	2.24	2.18	2.18	1.68	1.85	2.01	ND	2.1	2
P ₂ O ₅	0.26	0.3	0.25	0.22	0.2	0.24	ND	ND	0.24	0.26
SO ₃	ND	ND	3.33	3.29	ND	ND	ND	ND	6.18	5.97

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE B

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN_1	RUN_2	RUN_1	RUN_2	RUN_1	RUN_2	RUN_1	RUN_2	RUN_1	RUN_2
As	2.7	2.79	3.26	3.18	1	2	5	5.3	1.2	
B	260.01	258.48	193.41	189.71	260	270	190	227	192	
Ba	37.44	34.12	47.8	49.3	64.3	73.7	47	46.5	46.5	
Be	1.87	1.86	1.37	1.44	1.5	1.5	1.4	1.8	1.89	
Cd	0.92	0.77	0.97	1	<0.3	<0.3	<0.4	0.573	0.941	
Cr	36.4	35.15	32.57	35.14	35.2	34.5	38	31.1	32.8	
Co	5.62	4.55	3.43	3.58	3.57	3.53	3	2.77	3.07	
Cu	11.44	11.37	9.61	9.79	<41.8	<42.4	10	13.2	13.8	
F	<100	<100	124.37	125.42	110	120	ND	88	95	
Hg	<0.1	<0.1	0.131	0.115	0.11	0.11	0.12	0.105	0.106	
Mn	32.24	31.02	40.6	39.9	29.9	28.1	38	37	34.8	
Mo	7.28	7.03	7.2	7.36	<13.6	<13.8	5	4.9	6.6	
Ni	19.76	18.61	16.94	17.49	22.9	21.8	21	17.3	19.4	
Pb	7.49	2.38	14.95	14.77	15	16	15	10.2	9.1	
Sb	<0.8	<0.8	0.69	0.69	0.707	0.566	<1	2.56	ND	
Se	3.33	1.45	4.71	4.78	3	4	1	3.2	2.5	
V	49.92	50.66	38.79	39.29	44.7	49.1	47	49.5	49.8	

% DRY BASIS

ASH	12.68	12.54	12.69	12.72	12.56	12.53	12.45	12.43	12.48
CARBON	68.33	67.79	70.23	70.07	70.12	69.95	68.86	68.84	68.78
HYDROGEN	5.1	5.29	4.82	4.84	4.83	4.81	4.51	4.68	4.68
NITROGEN	1.26	1.23	1.33	1.44	1.42	1.4	1.35	1.33	1.27
SULFUR	3.63	3.63	3.43	3.49	3.46	3.47	3.48	3.51	3.48
CHLORINE	0.05	0.05	0.084	0.077	0.079	0.078	ND	0.1	0.12
Btu/lb	11900	11480	12398	12402	12376	12367	12390	12350	12321

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	49.21	49.47	51.04	51.01	ND	ND	50.45	49.2	49.3
Al ₂ O ₃	18.59	18.69	19.41	19.46	18.28	19.4	19.05	19	18.9
TiO ₂	1.01	1	1	0.99	0.86	0.78	0.96	0.8	0.8
Fe ₂ O ₃	16.42	16.5	17.97	17.83	16.7	16.42	ND	17.1	16.7
CaO	1.84	1.62	3.94	3.85	1.84	1.97	ND	3.6	3.3
MgO	0.3	0.36	1.09	1.1	0.77	0.77	ND	1.1	1.1
Na ₂ O ₃	0.7	0.71	0.79	0.79	0.78	0.7	0.73	0.86	0.84
K ₂ O	2.37	2.19	2.39	2.38	2.25	2.22	2.26	2.2	2.2
P ₂ O ₅	0.3	0.3	0.26	0.27	0.51	0.51	ND	0.43	0.3
SO ₃	ND	ND	1.94	1.96	ND	ND	ND	3	3.2

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE C

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	3.06	5.82	13.61	13.04	5	7	6	ND	12.1	13.4
B	91.69	86.72	66.46	63.46	80	80	73	76	79.1	54
Ba	28.52	27.55	32.2	34.9	34.6	45.2	35	35	35.2	34.3
Be	1.63	1.63	1.13	1.17	1.3	1.2	1.2	1.2	1.53	1.51
Cd	0.08	0.08	0.1	0.11	<0.3	<0.3	<0.4	<0.4	0.079	0.237
Cr	21.39	20.4	16.77	15.9	17.2	18.2	19	20	9.9	12.4
Co	9.37	8.67	4.51	4.46	4.92	5.13	5	7	4.03	4.6
Cu	9.88	8.06	6.98	6.95	<38	<39.3	8	8	10.9	11.2
F	<100	<100	63.96	63.22	60	60	ND	ND	58	53
Hg	<0.1	0.16	0.147	0.143	0.1	0.11	0.14	0.14	0.135	0.145
Mn	18.34	18.36	15.7	16.4	19	18.5	22	23	19	19.6
Mo	3.77	3.06	1.62	1.65	1.71	2.87	<6	<6	ND	0.977
Ni	16.3	16.32	12.44	12.06	10.4	10.7	15	15	14.2	13.8
Pb	3.77	0.65	6.44	6.52	5	7	7	8	4.5	5.7
Sb	<0.8	<0.8	0.66	0.7	0.603	0.654	<1	<1	ND	ND
Se	1.94	1.53	2.47	2.57	1	2	2	1	0.837	1.1
V	37.69	37.75	24.63	25.27	30.6	29.9	33	33	33	33.2

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	11.59	11.62	11.52	11.53	11.64	11.6	11.57	11.51	11.61	11.59
CARBON	71.69	71.12	72.79	72.63	72.98	72.72	71.64	71.72	71.99	71.86
HYDROGEN	5.76	5.01	4.85	4.86	4.99	4.98	4.6	4.6	4.85	4.86
NITROGEN	1.4	1.38	1.44	1.39	1.42	1.42	1.45	1.45	1.43	1.36
SULFUR	3.44	3.55	3.15	3.16	3.28	3.27	3.13	3.18	3.37	3.16
CHLORINE	0.05	0.05	0.092	0.089	0.098	0.104	ND	ND	0.11	0.11
Btu/lb	11987	12644	12957	12971	12972	12932	12989	12953	12906	12906

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	47.09	45.15	45.02	45.57	ND	ND	44.73	ND	44.5	44.3
Al ₂ O ₃	23.24	21.56	21.31	21.63	19.36	18.78	21.74	ND	21.4	21.5
TiO ₂	1.08	1.04	0.96	0.98	1.1	1.09	0.99	ND	0.9	0.9
Fe ₂ O ₃	25.13	25.11	25.63	26.06	23.41	24.61	ND	ND	25.2	25.4
CaO	0.64	0.64	1.22	1.29	1.17	1.04	ND	ND	1.1	1.1
MgO	0.35	0.33	0.71	0.69	0.72	0.63	ND	ND	0.73	0.71
Na ₂ O ₃	0.54	0.37	0.4	0.42	0.38	0.41	0.42	ND	0.44	0.46
K ₂ O	2.12	2.01	1.93	1.89	1.77	1.9	1.85	ND	1.7	1.8
P ₂ O ₅	0.13	0.12	0.07	0.08	0.21	0.23	ND	ND	0.15	0.23
SO ₃	ND	ND	1.44	1.51	ND	ND	ND	ND	1.04	1.05

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE D

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	1.25	1.87	1.93	1.89	<1	<1	1	ND	0.45	0.63
B	117.71	104.86	40.44	39.86	100	110	90	90	102	76
Ba	187.83	137.31	432.1	424.4	697	640	170	150	450	536
Be	0.53	0.47	0.45	0.41	0.4	0.4	0.3	0.3	0.421	0.408
Cd	<0.06	<0.06	0.07	0.07	<0.3	<0.3	<0.4	<0.4	0.049	ND
Cr	6.01	4.62	6.17	4.64	4.79	3.97	4	4	3.03	3.54
Co	<2.0	<2.0	1.19	1.17	0.757	0.663	<1	<1	ND	0.77
Cu	11.65	8.74	7.5	7.07	<45	<43.9	8	8	14	12.1
F	<100	<100	48.59	48.12	50	50	ND	ND	39	35
Hg	<0.1	<0.1	0.086	0.096	0.08	0.08	0.1	0.1	0.102	0.091
Mn	137.74	121.08	188.4	186.3	98	99.8	160	150	148	151
Mo	8.01	6.99	6.55	6.39	11.1	10.8	7	4	4.84	5.37
Ni	5.01	3.74	4.15	3.34	16	15.2	2	1	2.08	2.08
Pb	5.13	5.12	5.44	5.45	8	7	5	5	3.8	2.9
Sb	<0.8	<0.8	0.48	0.48	0.451	0.429	<1	<1	ND	ND
Se	<0.6	0.97	0.9	0.92	<1	<1	1	ND	ND	ND
V	11.65	10.86	8.54	7.99	9.73	8.3	9	9	9.5	10

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	12	11.67	11.86	11.93	11.51	11.51	11.7	11.79	11.48	11.55
CARBON	62.27	65.9	68.55	68.65	68.41	68.21	67.27	67.36	67.38	67.43
HYDROGEN	7.42	1.95	4.68	4.71	4.63	4.61	4.51	4.52	4.33	4.38
NITROGEN	0.98	0.94	0.93	0.85	1.08	1.1	1.02	1.01	1.08	1.12
SULFUR	0.93	0.91	0.96	0.96	0.95	4.82	0.91	0.92	0.97	1
CHLORINE	0.04	0.04	<0.02	<0.02	<0.01	<0.1	ND	ND	0.03	0.04
Btu/lb	11342	9083	11735	11717	11693	11601	11751	11759	11634	11663

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	42.49	38.97	44.35	44.06	ND	ND	41.94	ND	41.7	41.8
Al ₂ O ₃	9.07	8.89	19.24	19.09	18.48	18.26	18.88	ND	18.2	18.1
TiO ₂	0.92	0.86	0.86	0.85	0.8	0.87	0.85	ND	0.8	0.8
Fe ₂ O ₃	5.22	4.74	6.8	7.12	6.97	6.1	ND	ND	6.3	6.8
CaO	2.92	2.55	13.24	13.12	4.2	4.01	ND	ND	11.7	11.5
MgO	1	1.01	4.13	4.09	1.22	1.21	ND	ND	4	3.8
Na ₂ O ₃	0.31	0.25	0.32	0.32	0.27	0.26	0.3	ND	0.25	0.24
K ₂ O	0.72	0.67	0.52	0.51	0.4	0.4	0.5	ND	0.47	0.48
P ₂ O ₅	0.33	0.29	0.3	0.3	0.56	0.56	ND	ND	0.26	0.24
SO ₃	ND	ND	11.31	11.21	ND	ND	ND	ND	13.02	13.54

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE E

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	<0.6	8.63	11.53	11.69	4	5	7	8	8.4	9.2
B	153.79	139.65	45.15	45.15	150	150	130	140	151	138
Ba	192.23	266.6	658.9	669.8	795	687	400	380	714	701
Be	0.82	0.71	0.72	0.73	0.7	0.7	0.6	0.7	0.639	0.732
Cd	0.12	<0.06	0.1	0.1	<0.3	<0.3	<0.4	<0.4	0.052	0.092
Cr	8.59	7.87	7.88	9.85	9.8	8.14	8	8	6.4	6
Co	3.33	2.79	2.66	2.54	1.91	1.91	1	2	2.02	ND
Cu	9.36	8.89	6.97	7.1	<48.2	<46.3	7	7	11.9	11
F	<100	<100	55.8	60.87	60	60	ND	ND	54	57
Hg	<0.1	<0.1	0.159	0.144	0.17	0.17	0.18	0.18	0.113	0.196
Mn	108.93	99.02	149.2	151.2	93.9	88.8	140	140	124	122
Mo	3.59	3.3	2.77	2.75	7.51	7.13	<6	<6	ND	0.977
Ni	8.07	5.71	6.97	7.62	18.1	<17.4	6	3	3.7	4.92
Pb	1.92	<0.6	3.04	2.63	4	4	4	4	4	1.8
Sb	<0.8	<0.8	0.77	0.72	0.679	0.073	<1	<1	ND	2.3
Se	<0.6	<0.6	0.88	0.98	<1	<1	1	1	ND	0.746
V	17.94	16.5	14.34	14.32	16.4	15.5	17	18	16.2	16.2

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	17.69	16.32	16.15	16.19	16.23	16.29	17.45	17.5	16.83	16.67
CARBON	55.85	54.93	59.58	59.55	59.05	59.17	58.91	58.61	59.38	59.33
HYDROGEN	7.41	7.38	4.21	4.15	4.24	4.31	3.92	3.9	3.91	3.87
NITROGEN	0.09	0.88	0.99	0.91	1.02	1.02	0.79	0.82	1.05	1.02
SULFUR	1.15	1.09	1.13	1.13	1.15	1.14	1.13	1.12	1.11	1.12
CHLORINE	0.03	0.03	<0.02	<0.02	<0.01	<0.01	ND	ND	0.05	0.05
Btu/lb	9252	8208	9842	9841	9920	9914	9777	9823	9939	9917

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	37.26	36.67	39.92	40.18	ND	ND	39.61	ND	38.5	38.7
Al ₂ O ₃	3.01	3.53	12.68	12.69	12.35	11.98	12.38	ND	11.6	11.7
TiO ₂	0.45	0.44	0.45	0.46	0.55	0.43	0.49	ND	0.5	0.4
Fe ₂ O ₃	4.95	4.67	6.93	7	6.43	6.17	ND	ND	6.7	6.6
CaO	2.23	3.16	17.34	17.53	5.32	5.04	ND	ND	16.2	16.3
MgO	0.94	1.15	4.96	4.97	1.19	1.05	ND	ND	4.8	4.8
Na ₂ O ₃	0.67	0.72	0.89	0.89	0.8	0.78	0.87	ND	0.84	0.84
K ₂ O	1.4	0.12	1.39	1.39	1.99	2.1	1.25	ND	1.3	1.3
P ₂ O ₅	0.13	0.13	0.13	0.14	0.22	0.21	ND	ND	0.13	0.11
SO ₃	ND	ND	14.99	15.21	ND	ND	ND	ND	15.42	16.02

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE F

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	4.82	50.43	35.51	35.07	17	17	24	ND	28.7	28.1
B	89.23	96.74	64.7	63.46	73	76	68	65	64.9	53.4
Ba	55.38	53.51	86.4	98.1	93	85.6	83	83	59	87.3
Be	2.67	2.78	2.14	2.18	1.9	2	2.3	2.4	2.75	2.41
Cd	0.07	0.09	0.1	0.12	<0.3	<0.3	<0.4	<0.4	0.093	ND
Cr	22.56	22.64	25.35	28.97	19.7	21.7	20	21	11	14.4
Co	9.74	11.32	5.9	5.88	6.23	6	8	7	4.56	3.59
Cu	21.54	21.61	17.35	17.07	<37.8	<37.4	20	21	22.2	29
F	<100	<100	90.46	92.55	90	100	ND	ND	63	65
Hg	0.21	0.27	0.238	0.251	0.24	0.26	0.25	0.25	0.398	0.323
Mn	25.64	15.73	26.6	26.6	27	23.2	30	31	25.5	26.6
Mo	7.38	6.48	4.25	4.46	3.65	3.8	<6	<6	1.92	1.51
Ni	26.67	29.84	21.98	22.39	26.8	28.2	25	28	23.5	23.8
Pb	7.28	<0.6	15.67	15.66	15	15	16	17	12.3	11.5
Sb	1.95	2.88	2.2	2.25	1.97	2.1	2	2	ND	ND
Se	1.13	2.26	3.27	3.29	2	2	3	ND	1.5	2.2
V	40	42.19	28.06	27.98	33.6	36.3	35	36	35.5	36.2

% DRY BASIS

ASH	13.42	13.42	13.42	13.45	13.44	13.4	13.21	13.27	13.39	13.37
CARBON	66.75	66.24	71.23	71.11	71.34	71.38	70.41	70.23	69.98	69.94
HYDROGEN	5.3	5.28	4.76	4.77	4.94	4.88	4.63	4.58	4.77	4.75
NITROGEN	1.39	1.35	1.43	1.43	1.45	1.39	1.39	1.35	1.28	1.32
SULFUR	3.1	2.96	2.9	2.95	3.1	3	2.96	2.99	3.16	3.13
CHLORINE	0.6	0.05	0.155	0.14	0.119	0.122	ND	ND	0.12	0.13
Btu/lb	12207	10953	12674	12648	12645	12609	12665	12688	12623	12583

% DRY ASH

MAJOR ASH ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
SiO ₂	45.86	49.3	47.18	46.04	ND	ND	44.91	ND	46.1	45.3
Al ₂ O ₃	23.1	24.62	23.19	23.37	22.98	23.97	22.55	ND	22.1	22.2
TiO ₂	1.12	1.23	1.06	1.07	1.3	1.12	1.12	ND	1	1
Fe ₂ O ₃	20.76	23.02	21.68	22.01	20.9	19.67	ND	ND	21.4	21
CaO	1.18	1.07	1.85	1.91	1.44	1.36	ND	ND	1.7	1.8
MgO	0.43	0.33	0.87	0.87	0.73	0.86	ND	ND	0.86	0.86
Na ₂ O	0.22	0.21	0.3	0.3	0.25	0.25	0.38	ND	0.24	0.26
K ₂ O	2.39	2.22	2.3	2.32	1.92	2.11	2.05	ND	2.2	2.2
P ₂ O ₅	0.47	0.53	0.48	0.46	0.89	0.89	ND	ND	0.45	0.46
SO ₃	ND	ND	1.78	1.83	ND	ND	ND	ND	1.53	1.49

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE G

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	1.53	1.65	2.53	2.57	1	1	2	ND	0.75	1.21
B	95.36	74.61	87.94	90.9	86	84	82	65	60.7	47.4
Ba	95.36	88.87	404.6	417.4	402	461	250	250	365	389
Be	1.53	1.21	1.33	1.4	1.2	1.2	1	1.3	1.47	1.39
Cd	<0.06	<0.06	0.01	<0.01	<0.4	<0.4	<0.6	<0.6	0.036	0.34
Cr	10.96	8.56	12.89	10.44	10.2	10	9	10	7	7.5
Co	5.59	4.83	3.96	4.07	4.24	4.38	4	4	2.34	2.38
Cu	52.61	10.53	10.44	10.47	<40.5	<42.9	10	10	14.1	14.7
F	<100	<100	92.65	91.53	90	90	ND	ND	73	78
Hg	<0.1	<0.1	0.097	0.093	0.08	0.08	0.07	0.07	0.078	0.071
Mn	54.81	50.47	99.5	100.7	79.9	82.7	77	82	76.3	75.5
Mo	2.52	<2	1.75	1.73	6.65	5.92	<6	<6	0.429	0.795
Ni	7.45	6.69	8.4	7.74	<15.2	<16.1	6	4	6.4	6.4
Pb	6.25	5.05	10.02	10.06	12	12	10	11	6.1	7.5
Sb	1.97	0.99	1.72	1.72	1.38	1.63	1	1	4.43	3.22
Se	<0.6	<0.6	1.62	1.84	1	1	2	ND	1.07	1.77
V	29.6	24.14	24.82	25.52	25	26.8	27	29	27	27.9

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	20.52	20.6	20.54	20.66	20.6	20.63	20.64	20.61	20.71	20.86
CARBON	58.09	58.61	62.29	62.02	61.81	61.95	61.25	61.29	61.35	61.13
HYDROGEN	5.82	5.38	4.6	4.62	4.71	4.76	4.51	4.5	4.35	4.56
NITROGEN	1.01	1.02	0.89	1.03	1.1	1.06	1.04	1.04	1.09	1.09
SULFUR	0.6	0.53	0.71	0.73	0.71	0.69	0.69	0.67	0.72	0.69
CHLORINE	0.08	0.05	<0.02	<0.02	<0.01	<0.01	ND	nd	0.04	0.04
Btu/lb	9769	9471	10855	10859	10848	10848	10848	10857	10804	10797

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	62.96	50.22	61.22	61.62	ND	ND	59.37	60.34	59.8	59.6
Al ₂ O ₃	18.16	16.1	22.76	22.61	20.51	22.92	21.59	21.81	21.1	20.7
TiO ₂	0.98	0.81	0.96	0.93	1.35	1.48	1.19	0.94	0.8	0.8
Fe ₂ O ₃	4.35	3.5	4.63	4.68	4.48	4.87	ND	ND	4.6	4.6
CaO	2.24	2.01	4.68	4.68	1.52	2.13	ND	ND	4.2	4.2
MgO	0.23	0.33	1.33	1.32	0.73	0.84	ND	ND	1.3	1.3
Na ₂ O ₃	0.12	0.11	0.25	0.27	0.2	0.23	0.31	0.19	0.25	0.29
K ₂ O	1.16	1.09	1.4	1.39	1.15	1.27	1.23	1.28	1.3	1.3
P ₂ O ₅	0.04	0.03	0.03	0.03	0.05	0.03	ND	ND	0.05	0.1
SO ₃	ND	ND	3.53	3.54	ND	ND	ND	ND	3.79	3.7

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE H

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	<0.6	3.63	4.63	4.51	3	3	3	3	ND	ND
B	203.01	181.49	142.76	134.99	160	170	160	160	150	142
Ba	37.4	33.09	51.1	50	54.5	49.9	50	50	48	46
Be	1.71	1.49	1.23	1.26	1.1	1.1	1.3	1.3	1.2	1.46
Cd	0.46	0.4	0.56	0.63	<0.2	<0.2	<0.4	<0.4	<0.4	0.107
Cr	22.44	21.35	19.58	19.89	23.1	20.1	22	22	22	17.8
Co	6.41	7.78	3.59	3.62	4.08	3.71	5	4	2.48	2.47
Cu	13.89	11.74	10.99	11.19	<34.0	<32.2	12	12	15.1	14.8
F	<100	<100	92.92	93.09	80	80	2.84	ND	71	71
Hg	<0.1	0.15	0.098	0.098	0.05	0.05	0.05	0.06	0.118	0.098
Mn	26.71	23.49	31.7	31.7	21.2	22.3	31	31	27.3	26.7
Mo	5.98	6.73	5.05	5.1	11.5	11.4	4	2	3.4	3.54
Ni	20.3	18.15	15.19	15.32	13.6	13.4	18	18	16.2	16.3
Pb	5.45	<0.6	10.6	10.73	9	9	11	11	7.6	6.8
Sb	<0.8	<0.8	0.62	0.61	0.585	0.417	<1	<1	ND	ND
Se	1.39	1.71	3.09	3.01	2	2	2	2	1.5	1.9
V	40.6	37.37	31.43	31.58	36.1	36.5	39	38	40.4	39.6

% DRY BASIS

ASH	10.47	10.68	10.67	10.71	10.57	10.5	10.51	10.48	10.59	10.69
CARBON	67.37	67.64	72.52	72.14	72.59	72.71	71.27	71.23	71.23	71.19
HYDROGEN	6	6.63	4.91	4.8	5.1	5.01	4.7	4.73	4.81	4.79
NITROGEN	1.29	1.43	1.4	1.54	1.46	1.5	1.43	1.43	1.43	1.35
SULFUR	3.08	2.87	2.8	2.84	2.87	2.86	2.86	2.89	2.86	2.82
CHLORINE	0.06	0.06	0.122	0.124	0.135	0.148	ND	ND	0.14	0.14
Bitu/lb	11827	11670	12858	12828	12815	12809	12776	12767	12734	12706

% DRY ASH

MAJOR ASH ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
SiO ₂	50.3	44.98	50.71	50.64	ND	ND	48.97	49.7	49.7	49.6
Al ₂ O ₃	23.13	18.88	22.1	22.14	21.61	20.21	21.32	21.4	21.4	21
TiO ₂	1.16	0.98	1.09	1.09	1.1	1.02	1.08	0.9	0.9	0.9
Fe ₂ O ₃	16.05	14.01	16.52	16.59	15.37	13.41	ND	16.1	16.1	16.1
CaO	1.71	1.54	3.3	3.39	1.89	1.68	ND	2.9	2.9	2.9
MgO	0.39	0.38	1.06	1.07	0.85	0.85	ND	1	1	1
Na ₂ O ₃	0.85	0.69	0.91	0.93	0.79	0.72	0.76	0.96	0.96	0.88
K ₂ O	2.83	2.41	2.6	2.61	2.78	2.29	2.28	2.4	2.4	2.4
P ₂ O ₅	0.21	0.18	0.18	0.2	0.35	0.34	ND	0.27	0.27	0.22
SO ₃	ND	ND	2.24	2.3	ND	ND	ND	2.93	2.93	2.7

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE J

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	1.03	2.9	3.42	3.34	2	2	2	2	2.1	2.2
B	311.53	257.9	186.96	179.95	230	220	200	210	222	205
Ba	3.22	27.94	54.1	57.7	54.7	55.6	51	49	ND	51.6
Be	1.61	1.5	1.15	1.19	1.4	1.4	1.1	1.2	1.47	1.43
Cd	0.88	1.01	0.6	0.65	<0.3	<0.3	<0.4	<0.4	0.417	0.276
Cr	34.38	26.86	26.3	26.97	28.6	26.5	30	28	26	33.3
Co	6.45	5.48	3.65	3.59	3.49	3.13	3	3	2.52	2.63
Cu	8.81	10.75	9.35	9.18	<99.2	<95.2	10	9	13.1	13.9
F	0.01	0.01	111.15	110.27	100	110	4.35	ND	91	87
Hg	0.18	<0.1	0.117	0.111	0.1	0.1	0.09	0.09	0.113	0.107
Mn	17.19	35.46	53.4	51.8	30.6	28.2	45	45	42.6	43.8
Mo	10.74	7.52	7.07	7.04	16.7	15.6	5	5	5.2	5.4
Ni	21.48	19.34	16.12	16.55	15.3	13.2	19	17	18.3	21.5
Pb	<0.6	8.81	11.16	10.85	16	12	11	11	7.6	7
Sb	<0.8	<0.8	0.51	0.49	0.49	0.421	<1	<1	ND	ND
Se	1.93	2.79	4.16	3.99	3	3	3	ND	2.5	2.5
V	45.12	36.54	32.32	33.8	33.5	32.1	36	36	38.5	38.6

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	12.2	12.17	12.13	12.16	11.75	11.77	11.89	11.87	11.84	11.85
CARBON	69.43	69.07	70.45	70.4	70.01	69.92	69.56	69.4	68.98	69.34
HYDROGEN	4.78	5.7	4.86	4.84	4.88	4.95	4.43	4.47	4.77	4.73
NITROGEN	1.29	1.3	1.26	1.26	1.37	1.42	1.27	1.31	1.32	1.25
SULFUR	3.52	4.05	3.46	3.42	3.53	2.15	3.42	3.44	3.44	3.39
CHLORINE	0.04	0.04	0.08	0.075	0.079	0.076	ND	ND	0.11	0.01
Btu/lb	10161	11035	12502	12493	12493	12515	12446	12451	12410	12422

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	7.17	43.52	46.35	46.95	ND	ND	48.17	ND	47.5	48.2
Al ₂ O ₃	5.65	13.51	18.95	18.67	16.56	17.23	18.57	ND	17.6	17.8
TiO ₂	1.09	0.9	0.94	0.93	0.9	0.74	0.94	ND	0.9	0.9
Fe ₂ O ₃	15.11	13.89	17.2	17.23	14.94	14.01	ND	ND	16.4	16.8
CaO	1.85	1.98	6.55	6.45	2.21	2.1	ND	ND	5.3	5.5
MgO	0.15	0.28	1.06	1.04	0.92	0.74	ND	ND	1	1
Na ₂ O ₃	0.69	0.9	0.93	0.91	0.85	0.82	1.01	ND	0.99	1
K ₂ O	1.7	2.02	2.17	2.15	1.47	1.25	2.05	ND	2.1	2.1
P ₂ O ₃	0.28	0.24	0.27	0.29	0.51	0.55	ND	ND	0.24	0.28
SO ₃	ND	ND	2.98	2.88	ND	ND	ND	ND	5.86	6.05

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE K

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	2.49	2.8	3.18	3.22	1	2	2	2	1.9	2.8
B	228.69	238.84	173	167.32	200	180	200	200	201	205
Ba	32.22	32.19	48.5	46.7	61.1	57.4	52	49	50.1	56.5
Be	1.66	1.77	1.34	1.42	1.7	1.7	1.2	1.4	1.69	2.37
Cd	2.18	2.08	1.01	0.96	<0.3	<0.3	<0.4	<0.4	0.564	0.184
Cr	33.26	33.23	35.54	33.49	36.9	34.2	32	38	30.5	38.1
Co	4.57	4.88	3.44	3.68	3.67	3.42	3	3	1.82	3.84
Cu	11.43	11.42	9.61	9.7	<35.4	<33.9	9	10	13.8	15.9
F	0.01	0.01	128.14	134.87	110	120	ND	ND	96	88
Hg	0.18	0.19	0.107	0.105	0.011	0.011	0.012	0.12	0.121	0.095
Mn	28.07	28.04	39.9	39.9	27.3	28.5	34	39	33.9	40.8
Mo	7.07	7.58	7.39	7.38	17.6	18.4	<6	<6	5.8	6.9
Ni	17.67	17.65	17.46	17.52	13.5	12.2	18	19	17.4	23
Pb	9.77	14.54	15.25	15.61	18	18	15	16	10.2	9.5
Sb	<0.8	<0.8	0.68	0.73	0.657	0.597	<1	<1	ND	ND
Se	2.81	3.53	5.56	4.94	3	3	3	3	3.1	2.2
V	46.78	45.69	38.76	41	44.8	46.8	42	48	48.6	57.9

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	12.59	12.38	12.63	12.6	12.44	12.49	12.47	12.46	12.44	12.62
CARBON	68.06	81.55	70.23	70.02	69.61	69.21	68.99	68.92	68.7	68.93
HYDROGEN	4.98	4.6	4.82	4.87	4.91	4.9	4.55	4.53	4.69	4.7
NITROGEN	1.33	1.35	1.34	1.32	1.41	1.36	1.29	1.35	1.33	1.26
SULFUR	4	3.88	3.4	3.43	3.51	3.54	3.48	3.45	3.44	3.39
CHLORINE	0.04	0.03	0.073	0.09	0.086	0.088	ND	ND	0.07	0.07
Btu/lb	11326	11013	12359	12363	12391	12411	12392	12389	12384	12388

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	46	46.73	51.78	51.75	ND	ND	48.95	ND	50.6	51
Al ₂ O ₃	15.6	17.43	19.36	19.39	18.74	17	18.5	ND	18.6	18.4
TiO ₂	0.94	0.98	0.99	0.98	0.85	0.71	1.68	ND	0.9	1
Fe ₂ O ₃	14.17	15.59	19.07	18.9	1.74	1.59	ND	ND	17.5	17.4
CaO	1.5	1.41	3.81	3.68	1.92	1.81	ND	ND	3.5	3.5
MgO	0.3	0.26	1.09	1.09	0.74	0.81	ND	ND	1.1	1.1
Na ₂ O ₃	0.7	0.27	0.77	0.76	0.8	0.7	0.77	ND	0.92	0.82
K ₂ O	2.19	2.32	2.36	2.35	1.71	2.05	1.95	ND	2.2	2.2
P ₂ O ₅	0.26	0.27	0.31	0.28	0.59	0.51	ND	ND	0.39	0.32
SO ₃	ND	ND	1.87	1.82	ND	ND	ND	ND	3.56	3.54

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE L

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	14.26	10.18	13.18	13.29	7	8	8	ND	10.9	10.7
B	61.46	98.77	62.87	64.7	79	72	59	63	63.9	50.1
Ba	25.46	27.49	32.7	32.9	34.4	34.1	30	30	ND	33
Be	1.32	1.63	0.99	1.05	1.5	1.5	1.1	1.1	1.43	1.44
Cd	0.09	0.12	0.15	0.15	<0.3	<0.3	<0.2	<0.2	0.088	0.054
Cr	16.29	20.36	15.08	14.92	17	16.5	15	16	11.7	11.9
Co	6.11	9.67	4.54	4.43	5.18	5.56	4	4	4.49	4.33
Cu	7.33	8.96	6.91	6.76	<34.9	<32.6	7	7	11	10.6
F	<100	<100	61.87	61.71	60	50	ND	ND	52	52
Hg	<0.1	0.19	0.155	0.154	0.12	0.12	0.1	0.1	0.185	0.176
Mn	15.27	18.33	16.3	16.3	19.3	17.5	18	18	18.9	19.2
Mo	<2	3.56	1.58	1.54	1.87	2.02	<3	<3	ND	ND
Ni	13.24	16.29	12.39	12.18	19.3	16.7	14	12	14.3	14.7
Pb	3.46	3.67	6.67	6.55	15	9	6	6	4.7	4.3
Sb	<0.8	<0.8	0.67	0.66	0.546	0.597	<1	<1	ND	ND
Se	1.93	2.14	2.65	2.74	2	2	2	ND	1.1	2.3
V	30.55	37.67	24.64	24.07	32.7	32.5	28	27	32.8	32.3

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	11.58	11.56	11.45	11.53	11.61	11.61	11.45	11.38	11.49	11.67
CARBON	71.12	71.51	72.74	72.82	72.69	72.38	71.92	71.97	71.59	71.67
HYDROGEN	5.17	5.41	4.9	4.88	5.03	5.05	4.88	4.87	4.82	4.81
NITROGEN	1.36	1.37	1.39	1.42	1.43	1.42	1.41	1.38	1.34	1.23
SULFUR	3.41	3.52	3.12	3.17	3.26	3.29	3.08	3.15	3.29	3.25
CHLORINE	0.05	0.06	0.091	0.078	0.099	0.096	ND	ND	0.1	0.09
Btu/lb	12539	13308	13004	13009	12925	12950	12977	12973	12925	12936

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	39.57	47.19	45.66	45.89	ND	ND	45.42	ND	45.1	44.3
Al ₂ O ₃	19.93	23.29	21.77	21.86	21.55	21.56	22.07	ND	21.2	20.7
TiO ₂	0.94	1.07	0.97	0.98	0.96	1.08	0.97	ND	0.9	1
Fe ₂ O ₃	22.63	26.45	26.21	26.12	21.83	21.53	ND	ND	25.4	25.2
CaO	0.5	0.6	1.28	1.29	1.21	1.17	ND	ND	1.2	1.2
MgO	0.26	0.28	0.7	0.71	0.68	0.69	ND	ND	0.73	0.71
Na ₂ O ₃	0.37	0.38	0.41	0.41	0.36	0.33	0.5	ND	0.57	0.52
K ₂ O	1.7	2.02	1.9	1.92	1.58	1.42	1.83	ND	1.9	1.8
P ₂ O ₅	0.11	0.13	0.1	0.1	0.24	0.21	ND	ND	0.19	0.21
SO ₃	ND	ND	1.64	1.62	ND	ND	ND	ND	1.42	1.4

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE M

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	1.36	1.36	1.74	1.75	2	1	1	ND	0.54	0.52
B	101.72	105.45	26.13	31.55	99	100	97	96	79.6	61.2
Ba	161.27	173.68	438.5	451.3	559	589	120	140	445	503
Be	0.52	0.5	0.52	0.53	0.4	0.4	0.3	0.4	0.383	0.319
Cd	<0.06	<0.06	0.1	0.11	<0.3	<0.3	<0.4	<0.4	0.068	ND
Cr	5.21	5.09	4.8	4.76	4.94	5.01	3	4	3.3	3.2
Co	<2.0	2.73	1.19	1.16	0.619	0.641	1	1	ND	0.86
Cu	10.42	10.3	8.21	8.21	<36.7	<41.6	7	8	11.5	11.6
F	<100	<100	50.01	53.25	40	40	ND	ND	39	39
Hg	<0.1	<0.1	0.082	0.08	0.07	0.07	0.06	0.06	0.102	0.088
Mn	119.09	119.09	184.4	186.5	126.3	132.7	140	160	141	141
Mo	7.57	7.2	6.93	7.33	18.7	19.8	5	4	5.7	5.3
Ni	4.09	4.47	3.6	3.56	7.91	10.4	1	2	ND	ND
Pb	4.09	4.84	5.49	5.44	7	8	5	5	3.8	2.9
Sb	<0.8	<0.8	0.49	0.5	0.479	0.481	<1	<1	ND	ND
Se	<0.6	<0.6	0.98	0.95	1	1	1	ND	1.01	ND
V	9.68	10.67	9.3	9.35	8.68	9.07	8	9	9.3	9.6

% DRY BASIS

PROXIMATE & ULTIMATE	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
ASH	11.94	12.07	11.74	11.86	11.54	11.49	11.43	11.46	11.53	11.62
CARBON	67.62	67.2	68.81	68.56	68.36	68.49	67.86	67.72	67.51	67.67
HYDROGEN	7.29	6.03	4.72	4.75	4.72	4.79	4.59	4.58	4.38	4.33
NITROGEN	0.98	1	0.84	0.97	1.15	1.11	0.96	0.96	1.11	1.1
SULFUR	0.89	0.83	0.98	0.99	1.05	1.03	0.94	0.96	0.98	0.98
CHLORINE	0.01	0.01	<0.02	<0.02	<0.1	<0.01	ND	ND	0.04	0.04
Btu/lb	10898	8333	11747	11743	11738	11707	11775	11745	11670	11662

% DRY ASH

MAJOR ASH ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
SiO ₂	40.07	41.85	43.44	43.5	ND	ND	43.29	ND	40.3	40.9
Al ₂ O ₃	8.64	7.77	18.96	18.97	15.34	16.2	18.98	ND	19.5	19.1
TiO ₂	0.81	0.86	0.81	0.81	1.29	1.23	0.84	ND	0.8	0.8
Fe ₂ O ₃	4.75	4.85	7.05	7.64	5.21	6.02	ND	ND	5.9	5.6
CaO	2.76	2.16	12.82	12.83	3.75	4.21	ND	ND	11.4	11.4
MgO	1.45	1.11	4.04	4.02	0.96	0.99	ND	ND	3.9	3.9
Na ₂ O ₃	0.24	0.24	0.29	0.32	0.26	0.29	0.32	ND	0.32	0.36
K ₂ O	0.84	0.46	0.5	0.51	0.37	0.44	0.48	ND	0.49	0.51
P ₂ O ₅	0.31	0.31	0.29	0.29	0.64	0.6	ND	ND	0.29	0.27
SO ₃	ND	ND	1.77	11.84	ND	ND	ND	ND	14.48	14.08

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE N

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	11.83	10.07	11.69	11.71	4	5	3	ND	8.9	10.1
B	199.3	149.18	41.06	49.2	145	145	150	140	136	124
Ba	361.24	335.65	695.5	655	73	77.9	390	460	754	729
Be	0.97	0.77	0.51	0.51	0.8	0.8	0.6	0.6	0.711	1.13
Cd	0.11	<0.06	0.13	0.13	<0.4	<0.4	<0.4	<0.4	0.11	ND
Cr	10.09	7.71	8.01	8.45	9.06	8.67	8	20	6.4	6
Co	4.73	3.11	2.78	2.67	1.85	1.95	1	1	1.27	1.51
Cu	11.58	8.95	7.72	7.57	<39.7	<40.7	7	7	16.2	13.2
F	<100	<100	62.27	62.91	50	50	2.05	ND	54	56
Hg	<0.1	<0.1	0.129	0.167	0.11	0.1	0.13	0.13	0.153	0.155
Mn	137.02	105.67	150.8	147.9	93.1	96.5	130	130	120	124
Mo	3.99	3.85	2.82	2.88	10.5	11	<6	<6	0.142	0.44
Ni	7.22	5.59	7.3	7.49	15.5	16.4	4	4	5.32	11.8
Pb	3.61	2.98	2.6	2.5	9	8	3	3	1.97	0.05
Sb	<0.8	<0.8	0.77	0.79	0.672	0.704	<1	<1	ND	ND
Se	0.95	<0.6	1.03	1.03	<1	<1	1	1	1.56	ND
V	21.18	16.16	15.72	15.78	19.4	19.2	16	17	16	16.2

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	16.73	16.96	16.11	16.15	16.64	16.46	16.74	16.69	17.06	17.32
CARBON	58.35	58.36	59.77	59.64	59.28	59.22	58.96	59.22	59.26	59.64
HYDROGEN	6.03	4.36	4.23	4.25	4.28	4.31	4.06	4.02	3.8	3.87
NITROGEN	0.82	0.82	0.91	0.91	1.06	1.05	0.81	0.77	1.02	0.99
SULFUR	1.02	1.02	1.15	1.13	1.18	1.16	1.13	1.14	1.13	1.12
CHLORINE	ND	ND	<0.02	<0.02	<0.01	<0.01	ND	ND	0.04	0.04
Btu/lb	9107	7314	9887	9881	9924	9906	9906	9894	9892	9885

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	46.27	34.56	41.36	40.88	ND	ND	39.59	ND	39.6	39.4
Al ₂ O ₃	4.36	3.6	12.34	12.09	12.98	13.13	12.28	ND	12.1	12.2
TiO ₂	0.57	0.44	0.43	0.42	0.44	0.53	0.48	ND	0.5	0.5
Fe ₂ O ₃	5.22	4.19	7.09	7.06	6.25	6.38	ND	ND	6.6	6.6
CaO	4.9	3.9	17.58	17.35	4.77	5.06	ND	ND	15.9	16
MgO	1.23	1.34	4.8	4.79	1.11	1.05	ND	ND	4.7	4.7
Na ₂ O ₃	0.94	0.82	0.87	0.84	0.8	0.81	0.85	ND	0.95	0.92
K ₂ O	1.79	1.24	1.37	1.33	1.19	1.36	1.26	ND	1.4	1.4
P ₂ O ₅	0.19	0.14	0.13	0.12	0.33	0.31	ND	ND	0.18	0.15
SO ₃	ND	ND	14.56	14.4	ND	ND	ND	ND	15.11	14.94

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE O

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	46.12	35.85	34.96	36.04	21	18	27	ND	29.4	31
B	99.41	74.78	58.5	62.38	73	77	72	80	54.5	47.8
Ba	53.29	48.15	84.8	86.2	107	100	85	89	ND	85
Be	2.97	2.25	1.9	2.03	2.7	2.4	2.1	2.4	2.58	2.61
Cd	0.11	0.06	0.14	0.13	<0.3	<0.3	<0.4	<0.4	0.11	ND
Cr	22.55	18.44	20.68	19.39	21	22.6	20	20	13.3	13.9
Co	9.74	9.12	5.9	6.16	6.67	7.72	8	7	4.6	5.8
Cu	23.57	18.44	17.81	18.62	<31	<32.6	22	22	22.7	23.5
F	0.01	0.1	80.03	66.12	90	90	ND	ND	73	73
Hg	0.14	0.3	0.248	0.273	0.23	0.23	0.2	0.2	0.399	0.353
Mn	26.64	21.51	27.2	24.8	27.4	30.4	30	31	26	26.4
Mo	10.25	5.33	4.05	4.09	5.09	5.68	<6	<6	1.85	2.79
Ni	27.67	21.51	21.12	22.1	54.5	61.9	26	25	23.7	24.1
Pb	9.94	11.27	15.29	15.14	17	20	19	18	10.5	9.7
Sb	2.15	1.43	2.09	2.17	1.85	1.78	2	2	2.6	2.4
Se	2.05	2.15	3.39	3.37	2	3	3	ND	3.3	2.2
V	44.07	33.8	28.47	30.13	32.3	32.1	28	26	35.6	36.6

% DRY BASIS

ASH	13.29	13.46	13.32	13.3	13.26	13.4	13.12	13.16	13.31	13.46
CARBON	69.61	69.56	71.35	71.16	71.19	71.46	70.98	70.79	70.5	70.66
HYDROGEN	5.06	5.24	4.82	4.78	4.91	5	4.6	4.65	4.76	4.79
NITROGEN	1.3	1.31	1.34	1.47	1.41	1.37	1.36	1.38	1.33	1.35
SULFUR	3.08	3.1	2.92	2.97	3.02	2.99	3.01	3.04	2.93	2.91
CHLORINE	0.02	0.02	0.13	0.13	0.127	0.13	ND	ND	0.12	0.12
Btu/lb	11774	11530	12737	12720	12654	12655	12690	12708	12644	12637

PROXIMATE & ULTIMATE

% DRY ASH

MAJOR ASH ELEMENTS	% DRY ASH	
SiO ₂	52.88	ND
Al ₂ O ₃	24.76	20.52
TiO ₂	1.29	1.03
Fe ₂ O ₃	23.15	20.6
CaO	1.19	1.31
MgO	0.37	0.83
Na ₂ O ₃	0.24	0.25
K ₂ O	2.51	2.27
P ₂ O ₅	0.53	0.97
SO ₃	ND	ND

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE P

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	1.75	2.19	2.48	2.6	1	1	1	ND	2.3	2.4
B	89.74	90.81	75.17	75.9	60	77	82	74	45.2	65.9
Ba	82.08	79.87	397.2	377.8	495	462	230	240	385	374
Be	1.42	1.42	1.14	1.16	1.2	1.4	1.3	1.1	1.32	1.35
Cd	<0.06	2.95	<0.1	<0.1	<0.4	<0.4	<0.6	<0.6	0.018	ND
Cr	10.84	10.72	9.42	9.9	10.8	9.9	10	9	7.6	7.4
Co	6.24	6.24	4.15	4	4.34	3.79	5	3	2.74	3.41
Cu	13.13	14.22	11.56	11.49	<39.2	<35.7	11	10	13.7	13.4
F	0.01	<100	87.98	88.66	80	80	ND	ND	56	56
Hg	<0.1	0.16	0.082	0.089	0.07	0.08	0.08	0.08	0.078	0.077
Mn	51.44	47.05	90.6	90.6	82.5	79.1	87	79	77.1	75.4
Mo	2.96	2.95	1.68	1.66	<19.6	<17.8	<8	<8	0.488	ND
Ni	8.54	8.1	7.28	8.23	<14.7	<13.4	5	6	5.9	7.3
Pb	7.33	8.1	9.45	9.63	11	9	10	9	6.72	7
Sb	1.42	1.97	1.7	1.73	1.77	1.55	2	1	2.6	ND
Se	1.2	1.03	1.59	1.72	1	1	<1	ND	0.79	0.26
V	26.27	27.35	22.37	23.81	26.1	21.6	28	26	27	26.1

% DRY BASIS

PROXIMATE & ULTIMATE

ASH	20.56	20.54	20.58	20.6	20.44	20.43	20.24	20.29	20.51	20.75
CARBON	60.52	61.06	62.04	62.16	62.33	62.4	61.56	61.24	61.09	61.27
HYDROGEN	5.12	5.39	4.65	4.66	4.8	4.84	4.61	4.65	4.51	4.58
NITROGEN	1.02	1.02	0.98	1	1.11	1.15	1.08	1	1.09	1.08
SULFUR	0.61	0.64	0.7	0.072	0.71	0.7	0.69	0.7	0.7	0.71
CHLORINE	0.01	0.01	<0.02	<0.02	<0.01	<0.01	ND	ND	0.04	0.04
Btu/lb	10329	9469	10913	10898	10872	10870	10852	10865	10857	10830

% DRY ASH

MAJOR ASH ELEMENTS

SiO ₂	57.03	55.93	61.79	61.01	ND	ND	60.75	59.54	58.2	58.8
Al ₂ O ₃	16.09	15.09	22.5	22.16	22.53	19.93	22.08	21.79	21.1	20.9
TiO ₂	0.98	0.98	0.95	0.93	1	1.04	0.94	0.93	1	1
Fe ₂ O ₃	4.11	3.88	4.84	4.86	4.5	4.35	ND	ND	4.4	4.5
CaO	2.31	2.01	4.57	4.51	2.38	2.52	ND	ND	4.3	4.3
MgO	0.32	0.28	1.34	1.31	0.93	0.91	ND	ND	1.2	1.2
Na ₂ O ₃	0.13	0.11	0.27	0.26	0.26	0.24	0.26	0.22	0.27	0.27
K ₂ O	1.09	1.03	1.39	1.39	1.32	1.26	1.3	1.27	1.3	1.3
P ₂ O ₅	0.03	0.04	0.04	0.04	0.02	0.03	ND	ND	0.04	0.04
SO ₃	ND	ND	3.61	3.52	ND	ND	ND	ND	3.92	3.8

INDIVIDUAL LABORATORY ANALYSES OF ROUND ROBIN SAMPLE Q

PPM DRY WHOLE COAL BASIS

TRACE ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
As	4.72	5.67	4.39	4.43	3	3	3	3	2.3	3.3
B	235.98	288.74	138.35	135.55	160	180	160	170	140	160
Ba	39.69	41.71	50.9	50.8	56.8	67.1	50	47	48.6	50.2
Be	1.93	2.35	1.11	1.18	1.5	1.5	1.2	1.2	1.44	1.48
Cd	0.55	1.39	0.66	0.63	<0.2	<0.2	<0.3	<0.3	0.286	0.089
Cr	25.74	32.08	19.39	20.28	21.9	21.5	22	22	19.2	17.4
Co	7.08	8.77	3.61	3.59	3.88	4.23	4	5	2.79	2.29
Cu	12.87	18.18	10.72	10.79	<34.3	<34.8	12	12	15.3	15.3
F	<100	<100	80.61	80.89	80	80	ND	ND	70	75
Hg	<0.1	0.18	0.098	0.109	0.07	0.07	0.09	0.08	0.117	0.113
Mn	33.25	34.22	32.4	33.2	29.7	29.3	31	30	26.6	26.9
Mo	6.54	9.09	5.06	5.1	<8.6	<8.7	<6	<6	4.34	4
Ni	21.45	24.6	15.19	15.24	28.4	29.7	18	16	17.3	16.5
Pb	6.22	1.39	10.87	10.68	11	12	11	12	6.8	6.2
Sb	<0.8	<0.8	0.61	0.58	0.445	0.596	<1	<1	1.78	ND
Se	1.39	2.03	3.04	2.91	3	3	2	ND	2.4	1.87
V	48.27	57.75	31.51	32.81	36.6	35.7	39	39	40	39.4

% DRY BASIS

ASH	10.61	10.58	10.65	10.76	10.56	10.55	10.46	10.51	10.62	10.64
CARBON	66.15	68.69	72.54	72.7	72.68	72.58	71.32	71.6	71.25	71.23
HYDROGEN	6.38	5.87	4.89	4.89	5.11	5.09	4.71	4.73	4.77	4.79
NITROGEN	1.41	1.42	1.48	1.48	1.52	1.45	1.37	1.36	1.34	1.31
SULFUR	3.25	3.25	2.77	2.76	2.78	2.86	2.9	2.92	2.87	2.78
CHLORINE	0.07	0.08	0.139	0.137	0.148	0.136	ND	ND	0.1	0.1
Bit/lb	12206	11290	12901	12875	12785	12769	12815	12809	12768	12733

PROXIMATE & ULTIMATE

% DRY ASH

MAJOR ASH ELEMENTS	LAB I		LAB II		LAB III		LAB IV		LAB V	
	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2	RUN 1	RUN 2
SiO ₂	60.66	69.31	50.76	50.64	ND	ND	49.64	ND	50.1	50.2
Al ₂ O ₃	24.82	26.73	22.2	22.07	19.79	19.71	21.62	ND	21.7	21.5
TiO ₂	1.37	1.64	1.11	1.01	0.27	0.27	1.07	ND	1.2	1.2
Fe ₂ O ₃	18.79	21.68	16.92	16.92	16.53	16.39	ND	ND	15.4	15.5
CaO	1.7	1.56	3.31	3.4	2.22	2.01	ND	ND	2.9	3
MgO	0.34	0.28	1.03	1.04	0.69	0.66	ND	ND	1	1
Na ₂ O ₃	0.91	1.01	0.87	0.89	0.7	0.81	0.81	ND	0.9	0.91
K ₂ O	2.92	3.41	2.58	2.56	1.9	2.04	2.88	ND	2.5	2.4
P ₂ O ₅	0.22	0.3	0.2	0.21	0.39	0.43	ND	ND	0.21	0.21
SO ₃	ND	ND	2.23	2.32	ND	ND	ND	ND	2.82	2.9

APPENDIX B

INTRALABORATORY TRACE ELEMENT REPEATABILITY
AS PERCENT DIFFERENCE

**Laboratory Repeatability
Samples A & J**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	31.0%	1.9%	0.0%	0.0%	6.7%	7.9%	13.2%	166.3%
B	7.7%	1.0%	6.5%	11.5%	14.3%	8.2%	5.1%	62.0%
Ba	65.2%	6.3%	29.7%	12.2%	63.1%	35.3%	27.7%	78.5%
Be	57.1%	6.6%	7.4%	16.0%	2.1%	17.8%	22.5%	126.2%
Cd	9.1%	7.5%			27.9%	14.8%	11.4%	76.7%
Cr	12.4%	4.4%	3.5%	17.3%	16.8%	10.9%	6.6%	61.0%
Co	0.5%	6.9%	1.5%	50.0%	23.2%	16.4%	20.9%	127.2%
Cu		6.9%		23.3%	2.6%	10.9%	10.9%	99.8%
F		2.2%	4.9%		24.6%	10.6%	12.2%	115.8%
Hg		18.7%	10.5%	10.5%	5.3%	11.3%	5.5%	49.1%
Mn	36.7%	1.5%	2.8%	18.2%	2.2%	12.3%	15.3%	124.4%
Mo	11.2%	2.3%	8.4%	50.0%	9.0%	16.2%	19.2%	118.6%
Ni	11.0%	3.3%	6.1%	20.0%	11.1%	10.3%	6.3%	61.5%
Pb	21.8%	1.9%	54.5%	12.8%	7.8%	19.8%	20.8%	105.1%
Sb		4.9%	7.1%			6.0%	1.6%	26.2%
Se	32.6%	12.5%	18.2%	0.0%	17.4%	16.1%	11.7%	72.7%
V	6.7%	13.2%	6.6%	17.7%	1.4%	9.1%	6.4%	69.7%
Average	23.3%	6.0%	11.2%	18.5%	14.7%	13.8%	7.9%	57.8%

**Laboratory Repeatability
Samples B & K**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	3.7%	0.6%	0.0%	85.7%	32.1%	24.4%	36.8%	150.5%
B	10.3%	11.8%	33.0%	2.5%	3.2%	12.2%	12.4%	101.6%
Ba	10.5%	2.0%	15.2%	8.2%	13.6%	9.9%	5.2%	52.4%
Be	8.4%	1.8%	12.5%	3.8%	9.5%	7.2%	4.4%	60.5%
Cd	86.4%	0.0%			67.7%	51.4%	45.5%	88.5%
Cr	7.3%	1.9%	2.0%	6.9%	7.1%	5.0%	2.8%	55.9%
Co	7.3%	1.6%	0.1%	0.0%	3.1%	2.4%	3.0%	123.9%
Cu	0.2%	0.5%		5.1%	9.5%	3.8%	4.4%	115.4%
F		5.2%	0.0%		0.5%	1.9%	2.8%	149.1%
Hg		14.8%	163.6%	4.3%	2.3%	46.3%	78.4%	169.5%
Mn	12.0%	0.9%	3.9%	2.7%	6.8%	5.2%	4.3%	82.8%
Mo	2.3%	1.4%			9.9%	4.5%	4.7%	102.6%
Ni	8.3%	1.6%	54.0%	12.7%	9.6%	17.2%	20.9%	121.6%
Pb	84.5%	3.8%	14.9%	3.3%	2.1%	21.7%	35.5%	163.5%
Sb		2.2%	1.5%			1.8%	0.5%	25.0%
Se	28.1%	10.1%	15.4%	100.0%	7.3%	32.2%	38.7%	120.4%
V		2.1%	2.4%	4.3%	7.0%	4.0%	2.3%	56.9%
Average	20.7%	3.7%	22.7%	17.2%	12.0%	14.8%	23.8%	160.7%

**Laboratory Repeatability
Samples C & L**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	93.4%	0.7%	22.2%	28.6%	16.6%	32.3%	35.7%	110.6%
B	1.0%	1.8%	5.8%	19.9%	15.5%	8.8%	8.5%	96.2%
Ba	5.7%	2.3%	15.2%	15.4%	71.2%	22.0%	28.1%	128.1%
Be	10.0%	12.0%	18.2%	8.7%	5.8%	10.9%	4.6%	42.5%
Cd	27.0%	35.3%			76.0%	46.1%	26.2%	56.9%
Cr	13.1%	8.5%	5.5%	22.9%	5.7%	11.1%	7.2%	65.0%
Co	13.4%	0.0%	6.6%	40.0%	2.2%	12.4%	16.2%	130.5%
Cu	9.6%	1.9%		13.3%	2.3%	6.8%	5.6%	83.1%
F		2.9%	8.7%		6.5%	6.0%	2.9%	48.8%
Hg	17.1%	6.3%	13.3%	33.3%	25.3%	19.1%	10.5%	55.0%
Mn	8.8%	1.5%	1.9%	22.2%	1.3%	7.2%	9.0%	125.7%
Mo	62.9%	4.7%	16.3%			28.0%	30.8%	110.2%
Ni	9.9%	0.3%	52.2%	14.3%	3.5%	16.0%	20.9%	130.6%
Pb	46.9%	2.0%	66.7%	22.2%	12.5%	30.1%	26.4%	87.7%
Sb		2.2%	9.5%			5.9%	5.1%	87.6%
Se	15.9%	6.7%	28.6%	0.0%	54.8%	21.2%	21.6%	102.0%
V	10.1%	2.4%	7.5%	18.2%	1.7%	8.0%	6.7%	84.1%
Average	23.0%	5.4%	18.5%	19.9%	20.0%	17.2%	11.6%	67.9%

**Laboratory Repeatability
Samples D & M**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	13.7%	9.0%		0.0%	1.9%	6.1%	6.4%	103.5%
B	7.2%	32.8%	5.4%	7.0%	23.3%	15.1%	12.3%	81.2%
Ba	0.3%	3.8%	15.2%	20.7%	3.9%	8.8%	8.7%	99.1%
Be	0.2%	19.9%	0.0%	15.4%	16.6%	10.4%	9.6%	91.8%
Cd		40.0%			32.5%	36.2%	5.3%	14.7%
Cr	3.2%	12.3%	12.7%	13.3%	1.1%	8.5%	5.9%	69.1%
Co		0.4%	11.9%		11.0%	7.8%	6.4%	82.1%
Cu	1.6%	11.9%		6.5%	12.2%	8.0%	5.0%	62.7%
F		6.6%	22.2%		5.3%	11.3%	9.4%	83.2%
Hg		11.6%	13.3%	50.0%	1.6%	19.1%	21.2%	110.9%
Mn	8.3%	1.0%	26.8%	3.3%	5.9%	9.0%	10.3%	113.7%
Mo	1.5%	9.7%	55.0%	20.0%	7.4%	18.7%	21.3%	113.9%
Ni	2.2%	4.5%	52.1%	0.0%		14.7%	25.0%	170.0%
Pb	13.8%	0.4%	0.0%	0.0%	0.0%	2.8%	6.1%	216.4%
Sb		3.1%	8.7%			5.9%	4.0%	67.5%
Se		5.9%		0.0%		2.9%	4.1%	141.4%
V	10.1%	12.1%	1.6%	5.7%	3.1%	6.5%	4.5%	68.7%
	5.6%	10.9%	17.3%	10.9%	9.0%	11.3%	7.1%	63.0%

**Laboratory Repeatability
Samples E & N**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	86.9%	0.8%	0.0%	85.7%	7.7%	36.2%	45.8%	126.6%
B	17.1%	0.0%	3.4%	7.1%	10.6%	7.6%	6.6%	86.2%
Ba	41.2%	1.6%	163.0%	8.6%	4.7%	43.8%	68.5%	156.3%
Be	12.8%	34.8%	13.3%	8.0%	29.3%	19.6%	11.7%	59.4%
Cd	8.7%	26.1%			26.8%	20.5%	10.2%	49.9%
Cr	7.8%	7.4%	1.2%	0.0%	0.0%	3.3%	4.0%	121.5%
Co	24.6%	4.7%	0.5%	40.0%	31.7%	20.3%	17.1%	84.3%
Cu	11.8%	8.3%		0.0%	24.9%	11.2%	10.3%	92.0%
F		7.0%	18.2%		0.9%	8.7%	8.8%	100.6%
Hg		2.3%	47.3%	32.3%	21.2%	25.8%	18.9%	73.5%
Mn	15.4%	0.6%	3.7%	7.4%	0.8%	5.6%	6.1%	110.2%
Mo	12.9%	3.2%	38.0%		50.7%	26.2%	21.9%	83.7%
Ni	7.3%	1.4%		11.8%	66.0%	21.6%	29.9%	138.4%
Pb	109.8%	10.6%	72.0%	28.6%	96.7%	63.5%	42.8%	67.4%
Sb		4.6%	58.6%			31.6%	38.2%	120.9%
Se		10.2%		0.0%	70.6%	26.9%	38.2%	141.7%
V	81.0%	9.4%	19.0%	5.9%	0.6%	23.2%	33.0%	142.3%
Average	33.6%	7.8%	33.7%	18.1%	27.7%	23.3%	19.5%	83.9%

**Laboratory Repeatability
Samples F & O**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	38.9%	0.6%	13.7%	11.8%	6.1%	14.2%	14.7%	103.5%
B	6.5%	5.8%	0.7%	13.3%	14.5%	8.2%	5.7%	70.1%
Ba	7.1%	7.6%	14.7%	4.7%	53.0%	17.4%	20.2%	116.1%
Be	4.3%	9.5%	26.7%	4.3%	0.6%	9.1%	10.3%	113.9%
Cd	6.1%	20.4%			16.7%	14.4%	7.4%	51.6%
Cr	9.8%	30.2%	5.2%	2.5%	6.8%	10.9%	11.1%	102.0%
Co	11.0%	2.3%	16.2%	0.0%	24.3%	10.8%	10.0%	92.7%
Cu	2.7%	5.7%		7.1%	10.3%	6.4%	3.1%	48.9%
F		21.1%	5.4%		13.1%	13.2%	7.8%	59.3%
Hg	8.7%	6.3%	8.3%	22.2%	12.9%	11.7%	6.3%	54.3%
Mn	15.1%	2.3%	14.1%	0.0%	0.6%	6.4%	7.5%	117.5%
Mo	11.7%	6.8%	36.4%		30.0%	21.2%	14.2%	67.1%
Ni	13.9%	2.6%	71.6%	3.8%	1.1%	18.6%	30.1%	161.5%
Pb	97.8%	2.9%	20.9%	11.4%	16.4%	29.9%	38.5%	129.0%
Sb	29.7%	4.4%	11.4%	0.0%		11.4%	13.1%	115.1%
Se	21.3%	3.0%	22.2%	0.0%	39.1%	17.1%	16.0%	93.2%
V	5.4%	4.5%	8.2%	27.2%	2.1%	9.5%	10.2%	107.3%
Average	18.1%	8.0%	18.4%	7.7%	15.5%	13.6%	9.8%	72.6%

**Laboratory Repeatability
Samples G & P**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	21.3%	0.4%	0.0%	66.7%	82.3%	34.1%	38.2%	112.0%
B	6.0%	16.8%	8.0%	5.9%	2.7%	7.9%	5.3%	67.6%
Ba	12.9%	5.9%	10.3%	6.2%	0.7%	7.2%	4.7%	65.1%
Be	3.6%	17.1%	8.0%	4.3%	6.9%	8.0%	5.4%	68.0%
Cd					165.0%	165.0%		
Cr	9.9%	18.8%	2.4%	0.0%	3.4%	6.9%	7.6%	109.9%
Co	18.0%	1.5%	5.9%	0.0%	26.3%	10.3%	11.4%	110.3%
Cu	79.1%	9.7%		4.9%	6.1%	24.9%	36.2%	144.9%
F		4.2%	11.8%		29.7%	15.2%	13.1%	86.1%
Hg		10.5%	6.5%	13.3%	3.9%	8.6%	4.2%	48.8%
Mn	6.7%	10.0%	3.1%	4.3%	0.5%	4.9%	3.6%	73.4%
Mo	80.4%	4.1%			86.0%	56.8%	45.7%	80.5%
Ni	16.2%	4.0%		9.5%	3.1%	8.2%	6.0%	73.8%
Pb	30.9%	5.1%	18.2%	10.0%	0.9%	13.0%	11.9%	91.4%
Sb	13.5%	0.3%	9.8%	40.0%	98.5%	32.4%	39.8%	122.7%
Se		4.4%	0.0%		92.0%	32.2%	51.9%	161.4%
V	0.2%	8.6%	8.2%	3.6%	3.3%	4.8%	3.6%	74.4%
Average	23.0%	7.6%	7.1%	13.0%	36.0%	25.9%	18.9%	73.1%

**Laboratory Repeatability
Samples H & Q**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	96.4%	3.6%	0.0%	0.0%	27.7%	25.5%	41.3%	161.7%
B	30.8%	1.4%	3.0%	6.3%	3.0%	8.9%	12.4%	139.5%
Ba	14.4%	0.6%	17.1%	1.0%	4.1%	7.4%	7.8%	104.1%
Be	28.9%	8.4%	30.8%	4.1%	1.4%	14.7%	14.1%	95.7%
Cd	77.1%	8.1%			15.7%	33.6%	37.8%	112.5%
Cr	27.6%	0.5%	0.5%	0.0%	1.7%	6.0%	12.1%	199.6%
Co	11.0%	0.1%	4.0%	0.0%	2.6%	3.6%	4.5%	126.6%
Cu	19.1%	3.1%		0.0%	2.3%	6.1%	8.8%	143.0%
F		14.1%	0.0%		2.1%	5.4%	7.6%	141.0%
Hg	18.2%	5.5%	33.3%	42.9%	6.3%	21.2%	16.6%	78.0%
Mn	29.4%	3.4%	30.2%	1.6%	0.9%	13.1%	15.3%	116.4%
Mo	20.6%	0.1%			18.3%	13.0%	11.2%	86.4%
Ni	18.0%	0.3%	73.1%	5.7%	3.9%	20.2%	30.3%	150.1%
Pb	33.1%	1.0%	24.4%	4.4%	10.2%	14.6%	13.6%	93.2%
Sb		3.3%	3.8%		100.0%	35.7%	55.7%	155.9%
Se	9.8%	2.5%	40.0%	0.0%	22.7%	15.0%	16.5%	110.2%
V	30.5%	2.1%	0.4%	1.3%	0.8%	7.0%	13.1%	187.8%
Average	31.0%	3.4%	18.6%	5.2%	13.2%	14.8%	15.2%	103.2%

**Laboratory Repeatability
All Coals**

TRACE ELEMENTS	LAB I	LAB II	LAB III	LAB IV	LAB V	MEAN	SDEV	PRSD
As	48.2%	2.2%	4.5%	34.8%	22.6%	22.5%	19.7%	87.6%
B	10.8%	8.9%	8.2%	9.2%	10.9%	9.6%	1.2%	12.4%
Ba	19.7%	3.8%	35.1%	9.6%	26.8%	19.0%	12.6%	66.6%
Be	15.7%	13.8%	14.6%	8.1%	9.0%	12.2%	3.4%	28.2%
Cd	26.8%	17.2%			53.6%	32.5%	18.9%	58.0%
Cr	11.4%	10.5%	4.1%	7.9%	5.3%	7.8%	3.2%	40.3%
Co	10.7%	2.2%	5.9%	16.3%	15.5%	10.1%	6.1%	60.2%
Cu	15.5%	6.0%		7.5%	8.8%	9.4%	4.2%	44.5%
F		7.9%	8.9%		10.3%	9.0%	1.2%	13.6%
Hg	5.5%	9.5%	37.0%	26.1%	9.8%	17.6%	13.4%	76.3%
Mn	16.6%	2.6%	10.8%	7.5%	2.4%	8.0%	6.0%	74.7%
Mo	25.4%	4.0%	19.3%	8.8%	26.4%	16.8%	10.0%	59.7%
Ni	10.9%	2.2%	38.6%	9.7%	12.3%	14.7%	13.9%	94.3%
Pb	54.8%	3.5%	34.0%	11.6%	18.3%	24.4%	20.4%	83.3%
Sb	5.4%	3.1%	13.8%	5.0%	24.8%	10.4%	9.0%	86.7%
Se	13.5%	6.9%	15.5%	12.5%	38.0%	17.3%	12.0%	69.5%
V	18.0%	6.8%	6.7%	10.5%	2.5%	8.9%	5.8%	65.3%
Average	19.3%	6.5%	17.1%	12.3%	17.5%	14.7%	6.9%	46.6%

Average Repeatability by Coals	
Coal	Avg
A & J	13.8%
B & K	14.8%
C & L	17.2%
D & M	11.3%
E & N	23.3%
F & O	13.6%
G & P	25.9%
H & Q	14.7%

DOE COAL ROUND ROBIN TRACE ELEMENT REPEATABILITY RESULTS
% of Individual Lab Analysis Within Repeatability Ranges

Repeatability Range	Lab I	Lab II	Lab III	Lab IV	Lab V	All Labs
Less than 10%	31.0	79.0	42.0	46.0	52.0	50.0
10 to 20%	24.0	14.0	19.0	17.0	15.0	18.0
20 to 30%	8.0	2.0	7.0	7.0	11.0	7.0
30 to 50%	7.0	4.0	6.0	6.0	3.0	5.0
Greater than 50%	10.0	0.0	9.0	4.0	12.0	7.0
Non Determined	20.0	1.0	18.0	21.0	7.0	13.0