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ON-LINE LOW COST SULFUR AND ASH ANALYZER

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1.0 SUMMARY

A program of design, fabrication, and field testing of an on-line sulfur and ash analyzer was undertaken by The Babcock & Wilcox Company. The analyzer is intended for use on coal slurry streams such as those found at coal cleaning facilities. The analyzer design consists of a sample preparation and delivery system (SPAD) and an inductively coupled plasma atomic emission spectrometer (ICP-AES).

The program consisted of the following major tasks:

- Selection and screening of delivery systems
- Design of the analyzer system
- Fabrication of SPAD system
- Field testing of the SPAD system
- Laboratory ICP testing of field collected samples

The field testing was conducted at CQ Inc. (Homer City, Pa. pilot plant). Testing was completed without taking the ICP to the field, since the analysis of coal slurry by ICP had been demonstrated during the delivery system screening tests and the field tests were aimed primarily at demonstrating the performance of the SPAD system. Although the ICP was not deployed to the field, the subsequent laboratory testing of field collected samples simulated the performance of the entire system. The use of an ICP in a laboratory environment circumvented the need to "field harden" an ICP and, although ICPs are now being used in industrial environments, custom preparation of a commercial ICP would be required for a coal preparation plant environment.

Major successes of the project include:

- Demonstration of a sampling system which provides a continuous representative sample of slurry at a "cut down rate" of between 60,000:1 to 940,000:1.
- Development of a mass opacity meter.
- Demonstration of the complete sample preparation system in the field.
- Simulation of the entire system.

Major shortcomings identified include:

- Low (and variable from element to element) recovery of element concentration in field collected slurries of ICP analysis using the ARL MDSN nebulizer.
- Undesirable sensitivity to background levels of sulfur and calcium in the plant water.

2.0 INTRODUCTION

The purpose of the current project was to develop, fabricate, and field test an on-line sulfur and ash analyzer for use on coal slurry streams typical of those found in coal cleaning plants. The analyzer design consists of a sample preparation and delivery (SPAD) system and an Inductively Coupled Plasma Argon Atomic Emission Spectrometer (ICP) as the centerpiece of the analyzer. The SPAD system consists of sampling, dilution, grinding, and solids measurement equipment as well as a device for delivering coal slurry to the ICP "torch" (a flame shaped argon plasma within an RF induction coil). The two delivery devices tested (or screened) during the project were both Babington type pneumatic nebulizers (see figures 3.1 & 3.2).

The ICP is a well developed laboratory instrument which is used routinely to determine the elemental composition of substances in solution form. ICP analysis is carried out by pneumatically nebulizing the subject solution (i.e. forming an aerosol of solution droplets within an argon gas stream) and delivering the aerosol to an RF induction coil where it passes through the center of an argon plasma and is heated so intensely by RF Induction that it dissociates into elemental constituents which are excited and give off characteristic emission. The emission lines are monitored and their intensities are directly proportional to the concentration of each element in solution. The Babington nebulizers extend the capabilities of the ICP to finely ground slurries as well as solutions.

In order for the ICP to be used in the field, a SPAD system had to be designed and fabricated. Before the SPAD system could be designed, however, a nebulizer design had to be selected and extensive tests on a variety of slurry types and slurry preparations were conducted. These tests are referred to as "screening tests" and have been previously reported on in reference 1. An overview of the testing and a summary of the test results are discussed in this report.

Because the coal is in slurry form, it is critically important to know the moisture content (or %-solids) of the slurry being delivered to the ICP in order to convert ICP measurements to % of elements in dry coal. As a result, an opacity meter which measures the %-solids in the coal slurry was developed as part of this project. The opacity meter works extremely well at low %- solids (below .1 %) of consistent particle size distributions.

Once the slurry preparation parameters and the delivery system parameters were defined (i.e. results of screening tests), a SPAD system fulfilling these requirements had to be designed and fabricated. The SPAD system had to perform one primary and many secondary samplings, grind the slurry to a mean particle size of approximately 5 microns, dilute the coal slurry to 3% or less solids concentration, measure the %-solids, and deliver a continuous sample to the ICP plasma. The sampling portion was designed to use commercial equipment, where available. However, only the primary sampler could be purchased commercially and special sampling equipment in accordance with theory of reference 2 was designed and fabricated to accomplish continuous secondary sampling and dilution. A detailed discussion of the design of the analyzer is presented in reference 3.

Recent studies [4, 5] involving ICP's and coal slurries have indicated that, under proper conditions, finely ground coal in slurry form could be nebulized and delivered to an ICP plasma and that the emissions would be characteristic of the elemental concentration in the slurry. These studies, as well as others [6 - 8], used Babington type nebulizers to deliver the coal slurry to the ICP plasma (sometimes referred to as the ICP "torch" since it is bright and flame shaped). Babington type nebulizers cause the flowing slurry to form a liquid film which is made to pass over a pressurized argon orifice. The resulting fine aerosol, containing fluid droplets which carry the tiny coal particles, is directed to the ICP plasma by a shaped piece of glassware called a "spray chamber". Two of these Babington nebulizers were chosen for the screening tests and their performance is reported on in reference 1. Although these nebulizers place fairly stringent requirements on the coal slurry preparation, they offered the most reliable and most successful of the slurryto-ICP delivery methods documented in the open literature to date.

During the nebulizer/ICP testing, the coal slurries used were prepared in the laboratory and were well characterized - i.e.: independent chemical analysis, well described particle size distribution, and known solids concentrations. The last of these slurry parameters, solids concentration in the slurry, must be measured by the field analyzer since the purpose of the analyzer is to measure the concentration of elements in the dry coal and not the concentration in the slurry. This necessitated the development of a device which can measure the solids concentration (or mass concentration) of the coal slurry on-line. To this end, a concept of measuring the opacity of coal slurry and relating the opacity to the mass concentration of coal in the slurry was also pursued and the resulting design is described in reference 3.

3.0 OVERVIEW OF SCREENING TESTS

3.1 Screening Test Objectives

The primary objective of the screening tests was to characterize the ranges of particle size distribution and %-solids of the coal slurry for which repeatable and representative ICP emission signal intensity could be obtained using two candidate Babington nebulizers. Parameters which could affect the characterization, in addition to particle size and %-solids, include RF-power, argon gas flow, analyte uptake rate, nebulizer design, spray chamber geometry, and effectiveness of sump agitation.

The secondary objective was to develop sufficient first hand experience with the two delivery systems to determine which, if either, was most suitable for a field analyzer.

An additional cojective which was introduced to the screening tests was to evaluate an opacity meter design for use in measuring the mass of coal being delivered to the ICP "torch".

3.2 Screening Test Results

Detailed results of each of the many tests performed on the two delivery systems are presented in reference 1 and only generalized results are repeated here. An overview of the conditions tested and the coal slurries prepared are presented in tables 3-1 to 3-4. Note that the majority of the testing took place with slurries composed of Pittsburgh # 8 coal since it was believed that the effects of variation in parameters such as &-solids, particle size, argon flow, etc. would be essentially independent of coal type.

The slurries tested were laboratory prepared grinds made from coal samples obtained from the Penn State Coal Bank. High purity water was added to the coal during the grinding process (to facilitate grinding) and subsequently (to obtain the desired solids concentrations). After grinding and diluting, the slurries were analyzed by both conventional assay methods and by ICP analysis. The conventional assay methods were performed on residues of the slurry after drying (i.e. on both suspended and dissolved solids) in order to have a basis of comparison for the ICP results (which by its very nature is an analysis of both suspended and dissolved solids). No attempt was made to determine what portion of the slurry assay was in solution form and what portion was in suspended solids form.

The initial tests were all conducted on the Precision Glass nebulizer (figure 3.1) because of delivery delays associated with the ARL MDSN nebulizer (figure 3.2). However, once the ARL nebulizer was ready for testing, it was used exclusively from that point on.

The generalized results are:

- 1. Both the Precision Glass nebulizer and the ARL MDSN nebulizer are capable of delivering coal slurry to an ICP plasma and providing recovery of elements in the slurry approaching 100 % (recovery > 80 %), provided that certain conditions are fulfilled. Those conditions are listed below in their approximate order of importance.
 - a) The coal slurry should be ground to a particle size of approximately 5 microns in mean size.
 - b) The slurry should have a solids concentration of less than 3.0%. Slurries with as low as .01 %-solids concentration were analyzed with good recovery for the elements considered in the screening test

program (Al, Fe, S, & Si), which were all greater than 1% of the dry coal by weight.

- c) A multilinear calibration curve should be used. This was not done during the screening tests but would be easily adaptable to a custom field analyzer.
- d) The carrier argon flow rate should be set as low as practical (i.e. as low as can be while still maintaining a stable plasma). For the laboratory ICP used during the screening tests, this is approximately 0.30 l/min. The lower argon flow rates provided good recovery despite having used a linear calibration curve. This indicates that lower argon flow rates are more forgiving.
- e) The analyte uptake rate should be set to an optimum value. The effect of analyte uptake rate is dependent upon argon flow rate when linear calibration is used and the precise value for all argon flow rates was not determined. For an argon flow rate of .37 1/min an analyte uptake rate of approximately 3 ml/min appeared to yield very good results for both nebulizers tested. At lower argon flow rates there is an indication that even lower uptake rates may be optimum. Higher power levels to the ICP plasma (1.25 kW vs. 1.1 kW) make
- maintenance of stable plasma easier.

2.

- 3. The ARL MDSN nebulizer required less maintenance (cleaning) than the Precision Glass nebulizer. The Precision Glass nebulizer, possibly due to the 90 degree bend in the spray chamber, appeared to be much more sensitive to alignment to the ICP torch than the ARL MDSN.
- 4. Both of the tested nebulizer systems experienced torch fouling and required periodic cleaning. At %-solids concentrations of 3 % and less, with the Pittsburgh # 8 slurries, and with water flush between trials, the system could be used for many hours (i.e. 25 - 30 one minute long aspirations of slurry with 5 minute long distilled water aspirations between slurry aspirations) without cleaning.
- 5. Only the ARL nebulizer was tested over a variety of coal types and it was demonstrated that coal type did not affect the recovery. The coal type did however play a significant role in the rate at which the glassware became fouled.

3.3 Screening Test Conclusions & Recommendations

The screening testing performed was sufficient both in breadth and depth to conclude that an ICP, using a Babington type nebulizer as the delivery system, is a good candidate for a field analyzer of coal slurry. It is both rugged enough and would require only moderate amounts of attention (i.e. change out of glassware once or twice per shift).

Although not all open questions were answered (eg. optimum analyte uptake rate at all argon flow rates, benefit of using cleaning solutions between trials vs. water only), the following recommendations could be made:

• Preferred Nebulizer

ARL MDSN

• Preferred ICP Settings

Multilinear Calibration Minimum RF Power of 1.25 kW Preferred Nebulizer Parameters Argon Flow Rate = 0.30 1/min Analyte Uptake Rate = 2 - 4 ml/min Preferred Coal Preparation Mean Particle Size < 5 microns Solids Concentration < 3.0 %

Following these recommendations should result in greater than 80 % recovery of elements in the slurry (i.e. concentration of dissolved and suspended solids).

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DOE S & ASH ANALYZER - COAL SLURRY DATABASE ("SLURDATA.WK1") REV. 7/14/89

URRY # 7	9 # 11I	6071389-1	7/13/89	33.25	4.00	00.00	471.82	55.77			4.45	9.3	0.51		14.8	3.79		ISH	2.73	1.09	2.37	0.06	0.76	0.06	0.05	0.20		
			. <u></u>					·						F-4176			MOLEC.	X OF	39.52	13.90	22.92	0.67	7.20	0.69	10.47	1.61		10.36
RY # 6	0- FR 1 POF	50389-	5/03/89	34.10	2.00	0.00	166.45	55.84			2.91	6.5	0.00		30.3	1.88		Ŧ	7.45	3.35	3.07	0.19	0.36	0.23	0.05	0.78	0.03	
SLUI	5	SUF	•											F-4143			MOLEC.	X OF ASI	52.57	20.91	14.48	1.05	1.65	1.24	0.23	3.11	0.21	2.27
RY # 5	11 # 8	52289-5	/22/89	34.50	0.50	0.00	151.08	8.65			6.97	14	3.06		10.32	1.7			2.27	1.26	1.41	0.07	0.10	0.04	0.05	0.14	0.00	
SLUR	Id	SP80	2											:-4152			IOLEC.	K OF ASH	:7.15	23.04	19.60	1.11	1.32	0.66	0.61	1.66	0.09	1.65
7 # X	8#1	1989-4	19/89	30.50	2.00	0.00	64.62	33.53			4.16	8.9	0.44		10.42	1.75	. <u>-</u>	<u>~</u>	1.86 4	1.09 5	2.20 1	0.06	0.17	0.05	0.04	0.12	0.01	
SLURF	LId	SP805	5/				•							-4154			OLEC.	OF ASH	8.23	9.70	0.21	0.93	2.33	0.74	0.58	1.40	0.26	3.46
(#3 	1 # 8	1989-3	68/61	34.50	1.50	0.00	56.45	55.84			4.68	10.2	0.95	<u> </u>	10.05	1.69	<u>.</u>	<u>×</u>	2.12 3	1.23 1	1.42 3	0.07	0.09	0.04	0.04	0.13	0.00	
SLURR	11d	SP805	5/	,.			1	•						4153			LEC.	OF ASH	.15	.06	.20	.08	.29	.65	.55	.57	.09	.85
# S	#	89-2	/80	.70	.50	00.	.05	.61	1.2	6.3	.89	8.4	.61		0.5	.78	MO	*	.28 45	.24 23	.65 20	1 20.1	10 1	0 70'	0 20-	1.13 1	.02 0	-
SLURRY	111d	SP80502	5/5	33		U	232	1		J			U	142	-	•	EC.	F ASH	46 2	31 1	40	14 C	27 0	67 C	31 C	51 0	0 97	46
-	80	9-1	39	30	00	00	0	00			55	- 2	8	F-4	55	35	MOL	2 0	1 46.	28 22.	8 22.	07 1.	9 1.	0 20	0 70	11.1	1 0.	-
URRY #	# 111d	804128	4/12/8	24.8	7.(14.0	0.0	0.0			5.5	13.	4.4		11.5	1.8		HS	2.4	-	1.5	0.0	0.0	0.0	0.0	0.1	0.0	
אר 		SP				• ••••••				·				F-4094			MOLEC.	X OF A	44.58	20.91	24.53	10.97	1.10	0.67	0.51	1.57	0.18	1.63
									TIPOISE				SNO				GRAVIM	FACTOR	0.4674	0.5293	0.6994	0.5995	0.7147	0.6031	0.7419	0.8306	0.4364	
		~					~	(B)	SEC, CEN	ES/CM			75 MICR				MOLEC	FORM	S i 02	A1203	Fe203	1102	CaO	Ng0	Na20	K20	P205	
		(B&U 1D	0		(HRS)	(g) (g)	6) 360s	190280	, a2 700/	C, DYN	(RONS)	(SNC	5 > 18.	MP. #	IY COAL	COAL			COAL	COAL	COAL	COAL	COAL	COAL	COAL	COAL	COAL	
	ΤΥΡΕ	RY ID (GROUNE	LIDS	D TIME	ERS-A-2	ERS-SF5	- FOAM D	.a 25C,	TNS 822	AN CMIC	(MI CRC	RTICLES	LAB SA	H IN DR	IN DRY			IN DRY	IN DRY	IN DRY	IN DRY	IN DRY	IN DRY	IN DRY	IN DRY	IN DRY	IN ASH
	COAL	SLUR	DATE	% SO	GRIN	DISP	DISP	ANTI	 v1 sc 	* SUR	d-ME	06 - P	X PA	CHEM	X ASI	X S			X Si	X AI	X Fe	X 1i	X Ca	у мд	z Na	XX	ч Х	% S

* TEST PERFORMED ON 2.9 % SOLIDS SOLN.

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TABLE 3.2

TEST MATRIX FOR PRECISION GLASS NEBULIZER (SINGLE COAL TYPE)

IRGH # 8

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	3.9																						<u>.</u>		 .	-
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0.3	4.2		×	×				×																		_
	3.9		×	×	×	×	×	×	×	×				×				×	×		×		×			_
	~															_						_				
\$	4.7									_						_			_		_					
0.2	4.2			-														_				_				
	3.9		×				×				_								_							
Q-ARGON=	d-MEAN=	Q-UPTAKE	3.0	3.4	4.8	6.5	3.0	3.4	4.8	6.5	3.0	3.4	4.8	6.5	3.0	3.4	4.8	6.5	3.0	3.4	4.8	6.5	3.0	3.4	4.8	1
<u> </u>	_ <u>~</u> -			ň				5				6.5				8.5				10				15		

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TABLE 3.3

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TEST MATRIX FOR ARL MDSN NEBULIZER (SINGLE COAL TYPE)

COAL TYPE = PITISBURGH # 8

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TABLE 3.4

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TEST MATRIX FOR ARL MDSN NEBULIZER (THREE COAL TYPES)

COAL TYPE = PITTSBURGH # 8, UPPER FREEPORT, ILLINOIS # 6

	4.5	111	×
0.35	2.9	U.F.	×
	3.9	P111	×
ARGON=	4EAN=	אר דידן	1.5 2.0 3.5 3.5 7.0 7.5 7.0
<u>-</u>	_ _p	LIDS CO	
		% sol	



PRECISION GLASS BABINGTON NEBULIZER

Figure 3.1



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ARL MDSN (MAXIMUM DISOLVED SOLIDS NEBULIZER)

Figure 3.2

4.0 OVERVIEW OF ANALYZER DESIGN

A detailed discussion of the design of the analyzer and, in particular, the development of the multistage sampling system is presented in reference 3. The current discussion is limited to an overview of the entire system.

The sulfur and ash analyzer is shown schematically in figure 4.1. Although the ICP portion of the design was not fabricated under this contract, the entire system is described in the following paragraphs.

An intermittent flow of coal slurry at approximately 20 liters/min from either the feed stream or the clean coal product (i.e. froth concentrate) stream of the froth flotation cell of the coal cleaning plant is obtained using the permanently installed "wig-wag"samplers (SO) at the host site. The slurry has a solid: content of between 5 and 30 %-solids, depending on the coal type being processed and whether the "feed" or the "clean coal concentrate" is being sampled. The slurry is directed into a funnel which converts the intermittent flow to a nearly continuous flow. A 5%, 6" vezin sampler (S1) is installed in this 20 1/min flow stream. The 1 1/min output of S1 is directed to a Netzsch LME4 continuous grinding mill and the 19 1/min "throughput" of S1 is directed to a second 5% sampler, S2, whose 0.95 1/min output is diverted to a reference 55 gallon drum. The second vezin sampler is required only for the prototype system to enable the collection of a reference sample to which the results of the multistage sampling system can be compared.

The size of the coal particles in the slurry enter the Netzsch grinder at -100 mesh and are reduced to a mean size of approximately 5 microns. Results of laboratory testing of the LME4 at the Netzsch laboratory are presented in appendix A. The 1 l/min output of the grinder is directed to the multistage sampling and dilution system where the slurry is prepared to the requirements for ICP nebulization (e.g. continuous 2 -4 ml/min slurry with less than 3 %-solids content.

Because a sampling/dilution system meeting the needs of the analyzer was not available commercially, a prototype system was developed and fabricated specifically for this project (see figures 4.2 - 4.3). A detailed discussion of the design of that system is presented in reference 3. The output of the multistage sampling system will be nearly steady at 3 - 4 ml/min, thanks to constant addition of filtered well water at approximately 9 times the rate of inlet slurry. Variations in the rate at which the slurry stream enters the system are attenuated by an order of magnitude by virtue of dilution with well water. Such small changes are easily accommodated by the control of the peristaltic pump, P4, delivering slurry to the ICP.

When elemental analysis is requested (via the computer terminal), the output of pump P4 is directed to the ICP nebulizer. When the nebulizer is being flushed or calibrated, the output of pump P4 is diverted to the opacity meter station. In this mode of operation, the metering pump P3 will inject a precise amount of water into the static mixer, causing the slurry to become much more dilute (< 0.1 %-solids). Under these dilute conditions, the opacity of the slurry is functionally related to the solids content of the slurry. This functional relationship makes possible the conversion of elemental concentrations in the slurry to elemental concentrations in the dry coal.

The ICP torch box, RF generator, and Ar distribution system are purchased items and, depending on the model selected, minor modifications will be made to accommodate the present application. The opto-electronics/computer system may be purchased as part of the ICP and modified (both hardware and software) or may be fabricated from parts and software written from "scratch". Computer controlled ICP units are now commercially available with many, but not all of the features needed for the present application. Since this part of the system was not fabricated during the current project, a number of options are discussed in reference 3.

Software to control all functions of the system (with the exception of the ICP) was written in "C" for the field test. The computer hardware included an IBM PC (286) compatible and the associated I/O hardware depicted in figure 4.4. A listing of the software is included in appendix I. The main functions of the software include:

- Calibrate FMI metering pump and opacity meter.
- Setup system
- Maintain a data log
- Regulate the fluid level in the bottom cup by feedback of pressure sensor signal to peristaltic pump, P4.
- On command from user, the software will obtain an opacity reading on the slurry by flushing the opacity meter with clean water, temporarily freezing the rate of the peristaltic pump P4, switching flow of Q4 from SC4 to opacity meter, making opacity reading (integrated over 30 seconds), storing results, returning Q4 to SC4, and again regulating the slurry level in the bottom cup.

A comprehensive list of the parts purchased for the analyzer is presented in table 4.1.





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Figure 4.4

Table 4.1

SULFUR & ASH ANALYZER PARTS LIST

TEM P	ART DESCRIPTION	SOURCE	MODEL #	QUANT	STORAGE LOCATION
TEM P/	ART DESCRIPTION	SOURCE	MODEL #	QUANT	STORAGE LOCATION
1 \$1,	/S2 ROTARY SAMPLER, SINGLE ARM 5 % 30 RPM	DENVER EQUIP. CO.	6" VEZIN	2	
2 P1	GRINDING SYSTEM .75 HP PERISTALTIC PUMP GRINDER GRINDER TRANSPORT. GRINDING MEDIA 1.1 mm SS BAL	NETZSCH NETZSCH NETZSCH LNETZSCH	LME4 630383	4 WK'S 1 42 LBS	
	DISPERSANTS	GAF CHEMICALS	CO-630	45 LBS	
3	WATER FILTRATION SYS. 20" FILTER HOUSING 10" 5 MICRON FILTERS 10" 0.45 MICRON FILTERS	AQUA-LINE TECH. CANTON, OH	 1L-0037-B MC 12 FPN941AAS	2 4 4	
4 P2	METER. PUMP,2 - 16 l/min GEAR PUMP & MOTOR and FOCUS 11 DC CONTROLLER	GELBER	400001GR-S6	1	
5	STATIC MIXER	K J LESKER ĈO.	1/2-10-321-5	1	
6 P4	PERISTALTIC PUMP "P4" TUBING AND FITTINGS	GILSON	MINIPULS3 F117939	1 3	
7	OPACITY METER PHOTODIODE LAMP LAMP ADAPTER FIBER OPTIC LIGHT GUIDE LAMP HOLDER OPTICAL TOOL RED FILTER CIRCUIT BOARD LOG RATIO AMP POWER SUPPLY +-15 VOLT	B&W AMTEC SILICON DETECTOR CORP. DOLAN-JENNER DOLAN-JENNER DOLAN-JENNER DOLAN-JENNER DOLAN-JENNER DOLAN-JENNER B&W RDD BURR BROWN COMPUTER PRODUCTS INC	SD172-11-11-22 180(110 V) SX-5 EE848 EKE SYF-100 T-10 FR1-60 SPECIAL LOG 100JP PM-302	1 2 1 1 1 1 1 1 1 1 1 1 1	
8	STATIC MIXER (OPAC METER)	K J LESKER CO	070-434	1	
9 P3	30-576 ml/min METERING PUMP POWER SUPPLY 24 VOLT IN-LINE PULSE SUPRESSOR	FLUID METERING INC ACOPIAN CO. FLUID METERING INC	QV-1KCKV107 24EB10 PD-HF	1 1 2	

Table 4.1 (Continued)

SULFUR & ASH ANALYZER PARTS LIST

ITEM PA	RT DESCRIPTION	SOURCE	MODEL # QUANT	STORAGE LOCATION
10 V1	SOLENOID PINCH VALVE 12 V, 30 PSI, NORM. OPEN POWER SUPPLY 12 VOLT DC OUTPUT MODULE	COLE PARMER (NResearch Inc, N.J.) ACOPIAN CO. OPTO	NR P/N 225P091-2 1 12EB20 1 OPTO 22 #ODC5 2	
11	POWER SUPPLY 24 VOLT	ACOPIAN CO.	24EB10 1	
12	FUNNELS	US PLASTICS	5 SIZES 5	
13	TABLE (SUPPLIED BY B&W)	HALLOWELL	WHEEL SET 1 GFI BREAKERS 3	
	COMPUTER CONTROL SYSTEM A/D CARD SCREW TERMINAL BOARD CABLE INTERFACE DIGITAL 1/O BOX SCREW TERMINAL BOARD D/A CARD 286 PC SUPPLIED BY B&W	METRABYTE METRABYTE METRABYTE METRABYTE METRABYTE METRABYTE	DAS-8PGA 1 STA-08PGA 1 C-1800 3 PIO-24 1 STA-U 2 DDA-06 1	
12	TRANSPORTATION COST TO/FROM S	ITE		
	MULTI-STAGE SAMPLING SYSTEM TEFLON O-RING PACKING SET APOLLO ROLLER VARIABLE SPEED DRIVE BEVEL GEAR MOTOR FIBERGLASS TUBING NYLON BEARING STOCK THREADED ROD JARS 17 LITERS	MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR MCMASTER-CARR FISHER SCIENTIFIC	#9609K45 3 #6407K33 3 #5985K42 1 #6456K17 1 #5990K34 1 #8535K21 4 #8664K22 1 #90038A036 4 11-823-13 4	
	DUAL SHAFT STIRRER & SPEED CON TYGON TUBE TUBING CONNECTOR KIT DIFF. PRESS. TRANS. (0-5"H2O) PLASTIC STATIC MIXER	N COLE-PALMER COLE-PALMER COLE-PALMER COLE-PALMER OMEGA	N-04651-00 4 N-060408-60 1 N-06286-00 1 N-07352-12 1 FMX 8213 1	

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Table 4.1 (Continued)

SULFUR & ASH ANALYZER PARTS LIST

I TEM	PART	DESCRIPTION	SOURCE	MODEL #	QUANT	STORAGE LOCATION
		POLY ROD (14"0D x 18")	ALMAC PLASTICS	••••	1	
1		POLY SHEET (1"x 18"x 36")	UNIVERSAL PLASTICS	•••	8	
		POLY SHEET (1.5"x 3.5"x 12")	UNIVERSAL PLASTICS		5	
		POLY ROD (1.5"00 x 12")	UNIVERSAL PLASTICS		1	
1		POLY ROD (6.5"0D x 18")	UNIVERSAL PLASTICS		1	
1		POLY ROD (2.5"00 x 12")	UNIVERSAL PLASTICS	····	1	
		POLY SHEET (1/4"x 18"x 96")	UNIVERSAL PLASTICS		2	
1		POLY SHEET (1/4"x 36"x 96")	UNIVERSAL PLASTICS		2	
1				ł		
		COUPLINGS & SCREWS	MCMASTER-CARR		1	
		POLY ROD 3"O.D.	MCMASTER-CARR		3	
		FLEXIBLE COUPLING	MCMASTER-CARR	•••	1	
		HOFFMAN ENCLOSURE & PANEL	SACKS ELECTRIC		1	
1		CPVC SCH 80 SOCKET FITTING	J.F.GOOD		4	
1		PVC FITTINGS (ELS TEES PIPE)	MCMASTER-CARR		1	
		SCREWS & TUBING	MCMASTER-CARR		1	
1		FITTINGS	COLE-PALMER	•••	1	
		PROPELLERS, RODS, UNIONS	COLE-PALMER		1	
		POWDER FUNNELS, PVC TUBING	COLE-PALMER	····	1	
14		MISCELLANEOUS EQUIPMENT				
		BARBED TUBE FITTINGS	COLE - PALMER		1 BOX	
		POLY BOTTLES	FISHER SCIENTIFIC		3 CASES	
		PUMP HEAD AND TUBING (TO FIT B&W SUPPLIED PERIST. PUMP FOR DISPERSANT DRIP)	COLE-PALMER		1	
		QUART CONTAINERS	PRO-GRAM PLASTICS		1 CASE	
	• • • • • •	NFRUI 17FR	AR!	MDSN173470-2		
1 16		тоясн	PREC. GLASS	1100-05	1	1
1 17		SPRAY CHAMBER	PREC. GLASS	100-14	1	
1 1/		SPRAT CHAMBER	PREC. GLASS	1100-14	1	

5.0 LABORATORY TESTING AND DEBUGGING OF SYSTEM PRIOR TO FIELD DEPLOYMENT

5.1 Vezin Samplers S1 and S2

The two 6" vezin samplers (purchased from Denver Equipment) were arranged in tandem on a tripod in the identical configuration in which they would be used in the field (see fig. 5.1). Each sampler was designed to cut 5 % of a continuous flow. These rotary samplers contain a single 18° "pie" shaped arm and operate at a speed of 30 RPM (i.e. one cut every 2 seconds). A funnel was placed in front of the first sampler to "smooth out" the intermittent flows which might occur in the field. In order to simulate extreme intermittent flow conditions, a test was conducted in which four 8-liter containers of water were poured, as rapidly as possible (1 -2 seconds), into the funnel attached to the top sampler. The four containers were poured at 30 second intervals. A second test was conducted in which the containers were poured more slowly (8 - 10 seconds).

The lab set-up and data for the two tests are presented in figure 5.1. The results indicate that, even at extreme intermittent flow conditions, the samplers operate near the intended 5 % rate. This is due to the fact that the funnel and hose, mounted to the entrance to the top sampler, acts as a flow "smoother". Note that, because the top sampler acts somewhat like an additional flow smoother, the bottom sampler performance is better than that of the top sampler.

This testing was conducted only to empirically demonstrate the % cutting of the vezin samplers and, although there is no reason to doubt the samplers' representativeness, these lab tests are not a proof of representativeness.

5.2 Multistage Sampling System

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The multistage sampling system was designed to allow many secondary sampling stages to be used in series by creating a situation in which the output of each secondary sampling stage could be transformed from intermittent flow to continuous flow prior to the next sampling stage. The laboratory testing of this system, like that of the vezin samplers, focused on obtaining the proper % cut from each sampling stage rather than demonstrating "representative" sampling.

The testing procedure consisted of introducing a steady flow of tap water (using the Gelber metering pump) into the top mixing tank and diverting the output of the sampler being tested into a graduated cylinder (or a tared container). The "waste" water (that portion of the water not sampled) was also collected and measured. The ratio of the sampled volume to the total volume (sample + waste) represents the fraction of the flow sampled. The metering pump was run at approximately 10 1/min and introduced into the top tank, regardless of which sampling stage was being tested. This means the first stage was sampling during the test on the middle stage and the top two samplers were operating when the bottom stage was being tested.

As the tests were conducted, three problems were encountered and solved:

5.2.1 Intermittent Flow Through Overflow Tubes (see Fig. 5.2)

The first major problem encountered was the occurrence of intermittent flow through the overflow tubes in each of the three mixing tanks. Despite having a weir shaped opening at the entrance, the weir tube in the top mixing tank was always under water at a feed rate of 10 1/min. The equilibrium level of water in the top tank was approximately 1" above the overflow tube under stable conditions. Shortly after having reached a steady state condition, however, a whirlpool would form at the entrance to the overflow tube. This whirlpool whirlpool introduced an air "funnel" into the tube, slowed the flow through the tube, and caused the water level in the tank to rise. The rising level would sometimes eliminate the whirlpool and bring the level back to its original position. Other times the tank would overflow. Neither of these conditions were tolerable. The problem was solved by placing a thin brass plate (whose width was approximately twice the tube diameter) directly above the overflow tube opening (approx. 0.25" above). Later, in field testing, it was discovered that a small funnel attached to the top of the overflow tube resulted in an even more stable flow condition and also eliminated the foam layer which would occasionally form on the surface of the water.

The intermittent flow conditions in the overflow tubes at the second and third stages were not "whirlpool" induced and did not result in an overflowing condition. The intermittent phenomenon observed was one in which the water in the tank would rise above the tube entrance before any flow would occur. Once a sufficient head was developed, the water would flow through the tube continuously, but at a rate which exceeded the inlet flow to the tank. The water level in the tank would quickly decrease to a level near the tube opening, at which time the flow would stop until sufficient head was again developed. The problem was solved in the middle stage by inserting a thin wire through the overflow tube. An attempt to solve the bottom stage problem in the same manner failed because the wire insert stopped the flow entirely. For that stage, a plastic orifice was constructed to restrict flow rate at the tube bottom (the orifice, however, proved to be a clogging nuisance in the field when unground coal was being sampled).

The inside diameters of the weir tubes in the top, middle, and bottom mixing tanks are 0.495", 0.1825", and 0.055" respectively.

5.2.2 High Flow Velocity Through Top Overflow Tube Resulting in Splashing In and Out of the Sampling Cups (see Fig. 5.3)

The cutter cups for all three stages were identical in shape and size. Although they worked extremely well at the second and third stages, they performed poorly at the top stage. The velocity of water exiting the top overflow tube was great enough to impinge upon the bottom of the cutter cup and splash higher than the cup height. Since the cutter cup was continually in motion, some of the water which splashed upward escaped the cutter. Similarly, as the cutter approached the exit flow of the tube, the water would impinge upon the flat surface surrounding the cutter and splash upward. As the cutter progressed through the flow stream it would inevitably catch some of this splashed water. The first splashing problem (out of the cup) was solved by replacing the original cup with one with three times its depth. The second problem was solved by installing plastic prism on the flats of the cutter base in such a manner that the impinging flow stream would be deflected laterally and thus avoiding upward splashing.

5.2.3 Scattering of Waste Water at Bottom Sampler Stage (see Fig. 5.3)

For the field test, the water which is passed through the bottom sampler would also be collected (for chemical assay by conventional methods). In order to minimize the amount of water which is thrown onto the walls of the sampler (thus causing a delay in the response of the sample collected at SC4'), a triangular trough was installed on the fore and back sides of the cutter. These troughs caused the "waste" to fall more directly into the collection pan on the bottom of the sampler.

5.2.4 Results of Testing

As the problems discussed above were discovered and solved, additional testing was conducted and the data recorded. Table 5.1 presents the data collected during each of 10 tests conducted. Most of the notes need no explanation in

light of the above discussion. Note 3, however, deserves some explanation. As the waste water was collected from the upper two samplers (RUN's 1,2,6,7,8,& 9), the waste water remaining in the drain hose was insignificant when compared to the total volume. At the bottom sampler, however, the amount of waste water was more significant, owing to the small volume of water processed through this stage. For RUN's 5 & 10, the waste hose was removed and the waste was collected directly from the outlet nipple in the bottom sampler tank.

5.3 Opacity Meter Testing

The opacity meter was tested with the entire control system (all equipment shown in fig. 4.4). The software includes an option for calibrating the opacity meter and for obtaining opacity meter readings upon demand. The software setup routine permits the user to specify the FMI-pump background flow rate (for continuous cleaning of the opacity probe), the FMI-pump flow rate for diluting slurry during opacity readings and for flushing after opacity readings, and the length of time over which the opacity reading is to be integrated.

Coal samples prepared during the Screening Test Phase of this project were used for the testing and calibration. The following problems were encountered:

5.3.1 Nonlinearity of the FMI Metering Pump

Except at very low flow rates the flow rate of the FMI pump versus input signal (4 - 20 ma) was non linear and, at high input signals, the flow actually decreased with increasing signal. FMI service personnel advised that we install an "in-line pulse suppressor" when we were dealing with nondegassed water (distilled, but non-degassed water was being used). An additional measure that was taken involved maintaining the distilled water tank at an elevation above the metering pump, causing a pressure head to be present in the inlet line to the pump. These measures helped but did not cure the problem completely. As a result, the metering pump was not operated at values above 150 ml/min.

5.3.2 Gas Bubbles Forming in the Distilled Water

The FMI metering pump caused tiny bubbles to form in the distilled water (which led to the nonlinearity problem discussed above). In addition to the nonlinearity of flow, however, the bubbles acted as light scatterers and rendered the opacity measurements useless. The same fixes that were implemented to improve the pump nonlinearity (namely pulse suppressor, raising the elevation of the distilled water tank, and operating below 150 ml/min) improved the performance significantly.





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BOTTOM MIXING TANK REVISIONS

Figure 5.2





ORIGINAL CUTTER DESIGN

REVISED TOP CUTTER (WEDGE ON BOTH SIDES)



REVISED BOTTOM CUTTER (WEDGE ON BOTH SIDES)

Figure 5.3

Table 5.1

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DOE SULFUR AND ASH ANALYZER LABORATORY TESTING OF MULTISTAGE SAMPLING SYSTEM

NOTE	Ч	8			m	8	4	н	ي. ا	3,6
LOW TOTAL (ml/min)	10189.01	745.53	53.60	43.31	51.68	773.62	760.38	10140.26	9616.01	56.06
SAMPLE (ml/min)	760.37	54.67	4.15	3.29	4.01	50.03	58.55	778.00	716.77	4.15
% CUT	7.46	7.33	7.75	7.60	7.75	6.47	7.70	7.67	7.45	7.41
DELTA TIME (min)	4.603	5.03	15.112	18.99	15.73	4.837	4.987	4.563	4.883	15.22
TOTAL (ml)	46900	3750	810.05	822.5	813	3742	3792	46270	46955	853.22
.VOLUME. WASTE (ml)	43400 *	3475	747.28 *	760	750	3500	3500	42720 *	43455 *	062
SAMPLE (ml)	3500	275	62.77 *	.62.5	63	242	292	3550	3500	63.22 *
CUTTER ELEVAT.	TOP	MIDDLE	BOTTOM	BOTTOM	BOTTOM	MIDDLE	MIDDLE	TOP	TOP	BOTTOM
ITTER VEED XPM)	17	17	17	17	17	17	17	17	17	17
DATE CU SF (F	6/26/90	6/26/90	6/26/90	6/26/90	6/26/90	6/26/90	6/26/90	6/26/90	6/21/90	6/21/90
RUN #		2	ć	4	л С	9	7	8	6	10

STEEL CUTTERS (1" HIGH) INSTALLED ON UPPER SAMPLER PERIODIC FLOW IN OVERFLOW TUBE OF MIDDLE TANK WASTE COLLECTION BEAKER PLACED DIRECTLY UNDER DRAIN (i.e. NO HOSE) FLOW RESTRICTOR PLACED IN OVERFLOW TUBE OF MIDDLE TANK PLEXIGALSS CUTTERS (2" HIGH) INSTALLED ON UPPER SAMPLER FLOW ORIFACE INSTALLED AT EXIT OF BOTTOM OVERFLOW TUBE WEIGHED SAMPLE NOTES1.

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*0.44.00

6.0 DESCRIPTION OF FIELD TESTS

Four tests were conducted at the EPRI coal cleaning pilot plant in Homer City PA (CQ Inc.) between July 30 and August 7, 1990. Additionally, three days of system setup and system debugging took place from July 27 through July 29, 1990. The preparation and planning for the field testing is described in reference 9.

Tests 1 & 2 were executed on an Upper Freeport coal (see data sheet in appendix D) being run for a Battelle project which was evaluating a new dewatering device. Tests 3 & 4 were executed on an Oklahoma bituminous coal (see data sheet in appendix D). Prior to the first test, the system was operated for two days with coal slurry from the froth flotation feed line. During that time, it was discovered that the drain tubes on the lowest cutter (i.e. sampler) in the multistage sampler were prone to clogging on unground coal slurry. The lowest stage sampler had been designed with smaller tube diameters to minimize inventory when sampling ground coal slurry for feed to the ICP. The tests on unground slurry therefore had to be executed using the first and second samplers rather than the second and third sampler, as described in the Field Test Plan (reference 9).

Also prior to the first test, the Netzsch grinder was loaded with grinding media and operated with coal slurry from the froth flotation tank. The output of the grinder was pumped to the multistage sampler and diluted with filtered well water. During this pretest operation, it was discovered that anti-foam agent would be required to minimize foaming in the upper mixing tank. A problem with the solenoid actuated pinch valve was also identified. As the pinch valve was actuated, closing one leg of a Y-connection, the other leg of the Y did not open immediately (it took some seconds for the pinched tubing to recover its shape). The result was that the peristaltic pump caused excess pressure in the tubing and the tubing separated from the fittings. In addition, even the ground coal slurry could occasionally clog the tiny tube fittings connecting the peristaltic pump to the opacity meter (see photographs of pinch valve in section 10).

Because of the problems with the peristaltic pump tubing/fittings/valves, the tubing was replaced with larger diameter tubing and the solenoid valve was replaced by hand operated valves. A future system would have to be equipped with separate valves for each leg of the Y-connection (or better yet, the slurry feed to the ICP should be sufficiently dilute to allow continuous online opacity/solids measurement, thus eliminating the need for a separate opacity branch). It was B&W's intention to continue with pretest operation when C.Q. Inc. was unexpectedly informed by Battelle that they would be halting their testing of Upper Freeport coal until later in the year. Since the next day's coal had already been loaded into the system, C.Q. offered to run the plant one more day with the Upper Freeport coal for B&W's purposes. Since this would be the last chance to obtain data on the Upper Freeport coal, the first two tests were executed, even though the system was not 100 % debugged.

6.1 Test 1: Feed - No Grinder - Upper Freeport (see Fig. 6.1)

Initially, it was intended to execute the test with the grinder in-line. However the Netzsch grinding mill could not be brought to the proper speed and eventually shut itself down on an overload signal. After wasting approximately two hours trying to solve the grinder problem, it was decided to switch over to a "No-Grinder" test. The arrangement of the equipment for that test is depicted in Figure 6.1. Note that the elevation of the bottom of the first vezin sampler is only about 12" above the top of the first mixing tank on the multistage sampler. The hose connecting the sampler to the top tank
multistage sampler. The hose connecting the sampler to the top tank was supported at multiple locations to overhead beams but still had some minor sags note that it was originally intended to use the second and third sampling stages rather than the first two).

The test was executed using the feed flow of the froth flotation tank (primary sampler set to divert approximately 15 liters/min to the first vezin sampler). The test was run for approximately an hour when it was observed that the first vezin sampler was overflowing. The test was halted and it was discovered that the hose connecting the vezin sampler, S1, to the top tank had become clogged with coal particles (caused by too slight a downward slope of the connecting hose and intermittent sagging).

During the hour-long test however, the problem with the Netzsch grinder had been traced to a defective RPM meter and it was possible to proceed with the next test (with the grinder in line). The RPM meter indicated a lower RPM than that at which the mill was actually operating. The meter was drifting lower as time went on. As the throttle on the mill was increased to compensate for the apparent low speed, the grinder drew too much power and shut down. On advice from Netzsch, the grinder was set to run at a power consumption of between 6 and 7 amps. At this setting, the grinder performed well as evidenced by the PSDs presented in appendix F.

Test 1 Loq

Title : Feed - NO Grinder - Upper Freeport (FNGU) Coal Type : Upper Freeport (see appendix D) Date : 7-30-90 Multistage Sampler Speed : 32 RPM Q_0 : approximately 15 1/min (* calculated) Q_c : approximately 0.75 1/min (* calculated) * Solids entering Vezin Sampler: approximately 6.6 * Q_{p2} : 6.3 1/min Dilution Ratio = 6.3/.75 +1 = 9.4:1 Grinder : Not Used 15:53 BEGIN REFERENCE SAMPLE 16:05 START SQ3-1 17:05 STOP SQ3-1 STOP REFERENCE SAMPLE

CLOG IN LINE BETWEEN VEZIN SAMPLER AND MULTISTAGE SAMPLER

*NOTE: Calculated Flow rates

 $Q_0 = [(VOL_{REF}) / (DELTA TIME)] / [(0.05) x (0.95)]$

 $Q_{c} = 0.05 \times Q_{0}$

COLLECTED APPROX. WT. SAMPLE

SQ3-1	(3,600 q)			
REFERENCE	(82,600 q)	OVER	117	MINS
WELL H.O	$(1,000 \alpha)$			

Fraction of Process Stream Sampled:

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F \approx (F_{s0}) (F_{s1}) (F_{MSS})

\approx (0.05) (0.05) (0.075^2) = 1.41 \times 10^{-5}

or 70,900:1
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6.2 Test 2: Feed - Grinder - Upper Freeport (see Fig. 6.2)

With the Netzsch grinder problem overcome, the 5 % cut from the first vezin sampler was directed to the Netzsch grinder feed pump. A drip solution of anti-foam agent (approximately 0.2 ml/min of Colloid 642, Colloids Inc., Newark, N.J.) and 1 ml/min of dispersant (IGEPAL CO-630 surfactant, GAF Chemicals Corp., Wayne, N.J.) was started at the same time. The reference sample was started approximately 10 minutes prior to the first sample extraction from the multistage sampler.

As a result of problems encountered during the pretesting with the small peristaltic pump tubing and fittings, the output of the last stage of sampling was equipped with larger diameter tubing. This tubing worked extremely well in that it did not clog, however the velocity of slurry through the tubing was so slow that it made opacity readings impractical (by the time slurry reached the opacity meter, the measurement time was over and the slurry was diverted back to the sample container SC4). The tubing connection between the sampler output and the sample container SC4 was made as short as practical by placing the peristaltic pump on the floor of the multistage sampler (see photographs in section 10).

The pressure sensor and software which controlled the fluid level in the vessel collecting the slurry worked very effectively to minimize and stabilize the inventory of slurry in the path between the sampler and the sample container SC4.

During the test, a layer of foam would slowly build on the surface of the slurry in the top mixing tank (the water level in this tank was always above the top of the overflow tube). Periodically the top tank was agitated by hand to mix the foam with the bulk of the slurry and allow it to enter the overflow tube.

Also during the course of this test, it was decided to draw off a sample (SQ3') of the waste slurry exiting the second stage of the multistage sampler for purposes of having a relatively large slurry specimen on which to calibrate the opacity meter and the ICP during the post-site laboratory testing at the B&W research center. This was an unplanned sample and no provision had been made to assure that a representative sample could be taken at the waste drain. This sample would be necessary since all samples which were sent to the Homer City Coal Lab (samples SC4'-1 to 8) would be totally consumed and the samples obtained for ICP testing at B&W (samples SC4-1 to 8) were quite small (approx. 50 ml each). The configuration of the waste exit piping is shown in figures 6.5 - 6.6. Although the configuration might that an unrepresentative sample of the field slurry would be better for calibration purposes than a laboratory prepared sample.

The test was concluded when C.Q. had exhausted the coal that they had loaded on the previous work day.

Test 2 Log

Title : Feed - Grinder - Upper Freeport (FGU) Coal Type : Upper Freeport (see appendix D) Date : 7-30-90 Multistage Sampler Speed : 28 RPM Q₀: approximately 17 1/min (* calculated) Q_c : approximately 0.85 1/min (* calculated) % Solids entering Grinder : approximately 6.4 % Q_{p_2} : 8.0 - 10.3 l/min (flow meter reading) Dilution Ratio = 9.15/.85 +1 = 11.8:1 Grinder : Netzsch LME4 In Line (1 mm dia. balls) Dispersant : approx. 1 ml/min IGEPAL 630 Surfactant (GAF) Anti-Foam : approx. 0.2 ml/min of Colloid 642 Grinder : Run at 7 - 9 amps. Grinder Output Temperature : 50 - 57 C 18:05 BEGIN REFERENCE SAMPLE 18:18 START SC4-1 & SC4'-1 18:30 REMOVE SC4-1 & SC4'-1 START OPACITY READING 18:33 START SC4-2 & SC4'-2 18:50 STOP SC4-2 & SC4'-2 START OPACITY READING 18:53 START SC4-3 & SC4'-3 19:05 STOP SC4-3 & SC4'-3 TAKE OPACITY READING RECOGNIZE FUTILITY OF TAKING OPACITY READING 19:08 START SC4-4 & SC4'-4 19:20 STOP SC4-4 & SC4'-4 START SC4-5 & SC4'-5 19:35 STOP SC4-5 & SC4'-5 START SC4-6 & SC4'-6 19:50 STOP SC4-6 & SC4'-6 START SC4-7 & SC4'-7 20:05 STOP SC4-7 & SC4'-7 START SC4-8 & SC4'-8 20:15 LINE FROM VEZIN SAMPLER TO MULTISTAGE SAMPLER CLOGGED STOP REFERENCE SAMPLE 20:18 STOP SC4-8 & SC4'-8 *NOTE: Calculated Flow rates $Q_0 = [(VOL_{REF}) / (DELTA TIME)] / [(0.05) x (0.95)]$ $Q_c = 0.05 \times Q_o$ COLLECTED APPROX. WT. SAMPLE SC4 -1 THRU 8 (65 g EACH) SC4'-1 THRU 8 (750 g EACH) (107,100 g) OVER 130 MINS REFERENCE so3'-1 (2,000 q)Fraction of Process Stream Sapled: $F \simeq (F_{s0}) (F_{s1}) (F_{MSS})$ = (0.05) (0.05) (0.075³) ≈ 1.06 x 10⁻⁶ or 940,000:1

6.3 Test 3: Concentrate - Grinder - Oklahoma (Figure 6.3)

The next opportunity to test coal slurry at the C.Q. facility was presented on a project aimed at determining the parameters associated with cleaning of an Oklahoma bituminous coal. Prior to this test, the tubing connecting the output of the sampler, the peristaltic pump, and the opacity meter was redesigned to provide larger tubing in the regions where clogs had been troublesome and smaller diameter tubing in the longer lengths (e.g. around the peristaltic pump head). With the exception of a sliver of plastic getting caught in a fitting (4 of 12 opacity measurements were missed as a result) the system performed exceptionally well and was trouble free. Once again, the Yconnection was switched by hand operated pinch valves to avoid having both legs closed simultaneously.

Since C.Q. Inc. required their own samples for these tests, they attached a third vezin sampler to the two vezin samplers which B&W had installed. This required that the tripod on which the vezin samplers were mounted be raised approximately 8". This additional height was also desirable for the test which would be run on unground coal slurry to provide a greater slope for slurry to travel between the outlet of the first vezin sampler and the top mixing tank of the multistage sampler. The C.Q. supplied vezin sampler had a slight overflow problem throughout the test. It was later discovered (during test # 4) that the sampler inlet had been assembled 180 degrees out of phase with the sample outlet, resulting in the overflow condition. In general, the vezin samplers supplied by Denver Equipment were not very precisely manufactured and should not be considered extremely accurate.

As the C.Q. test began, the flow to the first vezin sampler was much below the expected 20 liter/minute. B&W waited until the flow could be increased. After much discussion, it was determined that the Oklahoma coal had very few fines (the coal was not ground in the ball grinder and thus the only fines were the natural fines). At that point in time it was decided to use the throughput of the third vezin sampler as the input to the grinder (since this approximated the 1 liter/min flow upon which the equipment was designed) and divert the 5 % cut from the first vezin sampler to the reference sample. The arrangement of equipment for this test is depicted in figure 6.3.

As with test # 2, a steady drip of anti-foam and dispersant was added to the inlet of the grinder feed pump. Also, as in test # 2, an unrepresentative slurry sample from the waste drain of the second sampler stage was collected for calibration purposes (SQ3'). Prior to conducting this test, a small plastic funnel was inserted onto the overflow tube of the top mixing tank of the multistage sampler (see figure 5.2). The purpose of the funnel was to attempt to allow foam buildup in this tank to overflow into the tube more regularly (in the previous test the water level in the tank would sometimes be approximately 1 " above the top of the overflow tube and thus the foam would sit on the water surface and build up). The funnel worked as planned and no appreciable foam buildup was observed.

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Test 3 Log

Title : Concentrate - Grinder - Oklahoma (CGO) Coal Type : Oklahoma Bituminous (see appendix D) Date : 8-01-90 Multistage Sampler Speed : 42 RPM Q_0 : approximately 2 1/min (* calculated) Q_c : approximately 1.6 1/min (* calculated) % Solids entering Grinder : approximately 24 % $Q_{P2}: 9.0 \ 1/min$ Dilution Ratio = 9.00/1.6 + 1 = 6.6:1Grinder : Netzsch LME4 In Line (1 mm dia. balls) Dispersant : approx. 1 ml/min IGEPAL 630 Surfactant (GAF) Anti-Foam : approx. 0.2 ml/min of Colloid 642 Grinder : Run at 6 - 7 amps. Grinder Output Temperature : 42 - 48 C 10:20 TAKE PUSH WATER (CLAR. H₂O) SAMPLE 13:08 START REFERENCE SAMPLE 13:15 START SC4-1 & SC4'-1 13:30 STOP SC4-1 & SC4'-1 TAKE OPACITY READING 13:32 START SC4-2 & SC4'-2 13:45 STOP SC4-2 & SC4'-2 TAKE OPACITY READING 13:47 START SC4-3 & SC4'-3 14:00 STOP SC4-3 & SC4'-3 TAKE OPACITY READING 14:02 START SC4-4 & SC4'-4 14:04-2:11 COLLECT SAMPLE FROM BACK OF SECOND MIX TANK 14:15 STOP SC4-4 & SC4'-4 TAKE OPACITY READING 14:17 START SC4-5 & SC4'-5 14:30 STOP SC4-5 & SC4'-5 TAKE OPACITY READING (CLOG IN LINE Y-CONNECTOR) 14:32 START SC4-6 & SC4'-6 14:45 STOP SC4-6 & SC4'-6 14:46 START SC4-7 & SC4'-7 15:00 STOF SC4-7 & SC4'-7 15:01 START SC4-8 & SC4'-8 15:14 STOP SC4-8 & SC4'-8 15:16 START SC4-9 & SC4'-9 15:17 CLOG CLEARED AND OPACITY AGAIN WORKING 15:32 STOP SC4-9 & SC4'-9 TAKE OPACITY READING 15:34 START SC4-10 & SC4'-10 15:49 STOP SC4-10 & SC4'-10 TAKE OPACITY READING 15:50 START SC4-11 & SC4'-11 16:05 STOP SC4-11 & SC4'-11 TAKE OPACITY READING 16:06 START SC4-126 SC4'-12 16:11 STOP REFERENCE SAMPLE 16:22 STOP SC4-12 & SC4'-12 TAKE OPACITY READING

*NOTE: Calculated Flow rates

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 $Q_0 = [(VOL_{REF}) / (DELTA TIME)] / [(0.05)]$

 $Q_{c} = 0.95 \times 0.95 \times 0.90 \times Q_{o}$

COLLECTED SAMPLE	APPROX.	WT.
SC4 -1 THRU 12	(60	g EACH)
SC4'-1 THRU 12	(700	g EACH)
REFERENCE	(18,200	g) OVER 183 MINS
SQ3'-1	(4,000	g)
CLAR. H ₂ O	(2,000	g)

.

Fraction of Process Stream Sampled:

- $F = (F_{*0}) (F_{*1}) (F_{*2}) (F_{*3}) (F_{HSS})$ $= (0.05) (0.95) (0.95) (0.90) (0.075^3)$ = 1.71 x 10-5or 58,500:1

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6.4 Test 4: Feed - No Grinder - Oklahoma (Figure 6.4)

As a result of the low volume problem with the concentrate from the froth flotation tank and the inability to arrange the vezin samplers in such a way that the output of the third sampler was at an elevation above the top mixing tank of the multistage sampler, it was decided to execute the last test using froth feed.

To avoid the hose clogging problem that was encountered during test # 1, the entire vezin sampler tripod was raised approximately 8" and a piece of angle iron was placed between the output of the first vezin sampler and the top of the top mixing tank. The 5 % cutter hose was placed inside the angle iron and thus guided it at a constant slope without any sags.

The arrangement of equipment for this test is depicted in figure 6.4. An additional unplanned sample of slurry was obtained by extracting slurry from the top mixing tank through a peristaltic pump tube suspended at approximately mid depth in the tank. This sample, labeled ST1-1, was sent to the Homer City Coal Lab for analysis.

A sample of the waste at the drain of the second stage sampler (SQ3') was also collected for PSD analysis at B&W.

Test 4 Loq

Title : Feed - NO Grinder - Oklahoma (FNGO) Coal Type : Oklahoma Bituminous (see appendix D) Date : 8-07-90 Multistage Sampler Speed : 40 RPM Mixer Speed (all) : 333 RPM Q_0 : approximately 8 1/min (* calculated) Q_c : approximately 0.4 l/min (* calculated) % Solids entering Vezin Sampler: approximately 8 % Q_{P2}: 7.3 l/min Dilution Ratio = 7.30/.40 + 1 = 19.2:1Grinder : Not Used 9:30 BEGIN REFERENCE SAMPLE 9:40 START SQ3-1 START ST1-1 12:55 STOP REFERENCE SAMPLE 13:05 STOP SQ3-1 STOP ST1-1 *NOTE: Calculated Flow rates $Q_0 = [(VOL_{REF})/(DELTA TIME)]/[(0.05) \times (0.95)]$ $Q_c = 0.05 \times Q_0$ COLLECTED APPROX. WT. SAMPLE SQ3-1 (8,100 g)ST1-1 (3,400 g)(80,000 g)REFERENCE OVER 205 MINS CLAR. H,O (2,000 g)WELL H,O (2,000 q)Fraction of Process Stream Sampled: $F = (F_{s0}) (F_{s1}) (F_{MSS})$ = (0.05) (0.05) (0.075) = 1.41 x 10-5 or 70,900:1





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EQUIPMENT ARRANGEMENT FOR TEST #2 "FEED-GRINDER-UPPER FREEPORT" (Ie: "FGU") TEST DATE 7/30/91 (18:05 TO 20:18) FIGURE 6.2

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Figure 6.5

Multi-stage Sampling System (showing mixing tank drain configuration)





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7.0 DESCRIPTION OF LABORATORY TESTS ON FIELD COLLECTED SAMPLES

Laboratory testing of field collected samples was performed at the Homer City Coal Lab (conventional assay) and at B&W PDD Lab (PSD, ICP, & conventional assay). The types of assay performed on the individual samples and the lab performing the assays are outlined in figures 7.1 - 7.4. Reference samples were collected during all four tests for the purpose of defining the coal composition in the slurry prior to entering the analyzer's sample preparation system.

Tests 1 and 4 bypassed the grinder and provided a basis for determining how well the sampling system performed on unground (i.e. -100 mesh) coal. These tests were of interest because a sampling system which can representatively cut a slurry stream to a very small fraction of the "mother" stream, may have important commercial application, even without an ICP (e.g. the ability to obtain a small sample for off-line assay).

Tests 2 and 3 utilized the entire sample preparation system including the grinder, the opacity meter, and the control system. The small slurry samples obtained from these tests were analyzed by ICP at B&W.

7.1 Disposition of Samples Obtained During Test #1 (See Fig. 7.1)

Both samples collected during test # 1 (reference and SQ3-1) were sent to the Homer City (H.C.) Coal Lab for assay. The samples contained unground coal (i.e. -100 mesh coal slurry). The large volume reference sample was filtered through five 25 micron filters and the solids were analyzed by conventional methods described in appendix H. The smaller volume sample, SQ3-1, was filtered through a single 2.5 micron filter and the solids analyzed in a manner identical to that used for the reference sample. The mean particle size of the coal in both slurry samples is approximately 85 microns with 25 % of particles less than 25 microns and 3 % of particles less than 2.5 microns. Although the difference in filter sizes has the potential to make the comparison of results of the two samples difficult, the H.C. Coal Lab supervisor stated that the 25 micron filters become clogged rapidly and actually act as much finer filters.

7.2 Disposition of Samples Obtained During Test #2 (See Fig. 7.2)

The reference sample and the eight "flow-through" samples, SC4'-1 to 8, collected at the last stage of sampling, were sent to the Homer City Coal Lab for conventional assay. The "final" samples, SC4-1 to 8, from the last stage of the multistage sampler were returned to B&W for opacity testing and ICP analysis. An additional sample, SQ3'-1, was obtained from the "flow-through" of the second sampling stage and was returned to B&W. This sample was collected unscientifically from the end of a 30' collapsible hose running from the drain pipe on the second sampling stage to the waste sump at the Homer City site (see fig. 6.5). The purpose of this sample was to provide a relatively large volume of the ground slurry for experimentation and calibration at B&W (since the SC4 samples were of such small volume, they could not be "wasted" by using them to perform shakedown, calibration, and PSD testing).

At the Homer City Coal Lab, the reference sample was filtered through five 25 micron filters and the solids assayed. The eight SC4' samples were filtered through single 2.5 micron filters and assayed in the same manner as the reference sample (with the exception that the analysis of the major elements in the ash was not performed).

Particle size distribution in the reference sample is similar to that of the reference sample in test 1. Mean particle size of the coal in the ground

slurry samples (SC4, SC4', & SQ3') is approximately 6 microns with 30 % of the particles less than 2.5 microns, 1 % less than 0.5 microns, and 1.6 % of the particles greater than 19 microns.

At B&W, the eight slurry samples, SC4-1 to 8, were diluted 5:1 with distilled water. The dilution was considered necessary since the sample volumes were small and, prior to performing ICP analysis, the samples were "run" through the opacity meter (note that the opacity meter did not operate properly in the field test, as described in section 6). The dilution made possible both the opacity testing and ICP testing without seriously depleting the sample volume.

The eight SC4 samples were "run" through the opacity meter to determine - solids. During this operation, less than 10 ml of slurry was removed from the slurry samples through a peristaltic pump. The slurry container was agitated by an electric mixer during the opacity testing. The eight samples were then analyzed by ICP using the ARL MDSN (suspended solids) nebulizer. Again the slurry was under constant agitation. Upon completion of the ICP analysis, the slurries were allowed to settle overnight and approximately 50 ml of clear fluid from the top of the slurry was poured off, through a 2.5 micron filter (for safety) and the filtrates were analyzed by ICP using the Meinhardt (dissolved solids) nebulizer. Placing the filtrate in a clear glass beaker and holding it to a light revealed perfectly clear and transparent fluid with no trace of particles visible to the naked eye.

The SQ3'-1 "calibration" sample was riffled into eight approximately equal volumes by inserting an electric mixer and one end of a peristaltic pump tube into the original container and filling eight new containers in series, as described below (prior to riffling, the original container was weighed):

The eight new containers were weighed. Approximately 5 ml was pumped into container 1, then 5 ml into container 2....etc (the 5 ml volumes are approximate - the actual process was timed with a stopwatch). This process was repeated until all but the last 10 % of slurry in the original container had been riffled into the eight new containers. The eight containers were again weighed. A precise weight of distilled water was then added to the original container and the riffling process repeated until the original container was empty. The eight new containers were weighed a final time. Based upon the weights recorded, a precise weight of distilled water was then added to each of the eight new containers to bring the laboratory dilution to 2:1 in each container.

The eight new samples were labeled SQ3'-1A through 1H. Various tests on these samples were conducted at the B&W RDD chemistry lab:

- a Sample FGU-SQ3'-1D was shaken vigorously and approximately 100 g was poured off and analyzed for PSD. The remainder of the sample was dried in an oven, the residue weighed, and the % total (suspended and dissolved) solids computed. The dry residue was then analyzed for total sulfur.
- b Sample FGU-SQ3'-1G was shaken vigorously and approximately 100 g of slurry was poured off. This 100 g was poured through a 0.45 micron filter. The filter residue was dried, weighed and the dry residue was analyzed for total sulfur and % ash. The filtrate was analyzed by ICP using the Meinhardt (dissolved solids) nebulizer.
- c Sample FGU-SQ3'-lE was shaken vigorously and approximately 100 g of slurry was poured off. This 100 g was poured through a 0.45 micron filter. The filter residue was dried, weighed and the % suspended solids was computed. The residue was then analyzed for total sulfur.

- d Sample FGU-SQ3'-1H was poured, in its entirety, through a 0.45 micron filter. A precise weight of distilled water was then added to the empty container and its contents poured through the 0.45 micron filter. The filter residue was dried, weighed and the % suspended solids was computed. The dry residue was divided into two specimens, on which total sulfur and % ash content were determined.
- e Sample FGU-SQ3'-1C was further riffled into six containers in a manner similar to that used to riffle the original SQ3'-1 sample and each diluted to a different %-solids:

FGU-SQ3'-1C-1, 0.05 % tot solids, 0.038% susp solids, 585 g FGU-SQ3'-1C-2, 0.08 % tot solids, 0.060% susp solids, 357 g FGU-SQ3'-1C-3, 0.11 % tot solids, 0.083% susp solids, 264 g FGU-SQ3'-1C-4, 0.14 % tot solids, 0.105% susp solids, 202 g FGU-SQ3'-1C-5, 0.17 % tot solids, 0.128% susp solids, 165 g FGU-SQ3'-1C-6, 0.20 % tot solids, 0.150% susp solids, 146 g

These six samples were used to calibrate the opacity meter (as described in section 8) and sample FGU-SQ3'-1C-1 was used to make preliminary checks on the ICP during set-up of the MDSN nebulizer. The suspended solids are computed by multiplying the total solids by the ratio of (% susp. solids/ %tot. solids), or 0.54/0.72 (see table 8.2.2, results of test # 2, samples $SQ3'-1D \notin SQ3'-1H$).

7.3 Disposition of Samples Obtained during Test #3 (See Fig. 7.3)

With minor differences, the disposition of the slurry samples obtained during test # 3 is identical to that of test # 2. The differences include:

- a) At site, the reference sample was collected in two separate containers. The flow from the concentrate tanks was very low and it was initially believed that the reference sample could be collected in a single small container. When this proved inadequate, a second container was utilized. At the test's conclusion, the first container was emptied into the second (larger) container and the first container was rinsed twice with deionized water and poured into the second. A total of 3.5 liters of deionized water was used for the rinse. At the Homer City Coal Lab, the reference sample was filtered through five 25 micron filters prior to assay of the solids (as was the case for reference samples from tests 1 & 2).
 - b) Twelve, rather than eight, SC4 and SC4' samples were obtained. Other than this difference, the samples were handled in a manner identical to that of test # 2.
 - c) The "calibration" sample was riffled into 10 containers (versus 8 for test # 2) and diluted 1:1 (versus 2:1 for test # 2). In addition, significantly more caution was exercised in obtaining this sample. In test # 2, the sample was extracted at the end of a 30' collapsible hose and during test # 3, it was extracted at the drain pipe exit of the second sampler stage (see figure 6.5).
- d) Particle size distribution in the reference sample is similar to that of the reference sample in tests 1 & 2. Mean particle size of the coal in the ground slurry samples (SC4, SC4', & SQ3') is approximately 8 microns with 18 % of the particles less than 2.5 microns, 1 % of the particles less than 0.5 microns, and 7 % of the particles greater than 19 microns.

e) The CGO-SQ3'-1G sample was riffled into six containers and diluted as follows:

CGO-SQ3'-1G-1, 0.289% tot solids, 0.276% susp solids,952 g CGO-SQ3'-1G-2, 0.595% tot solids, 0.567% susp solids,482 g CGO-SQ3'-1G-3, 0.733% tot solids, 0.699% susp solids,343 g CGO-SQ3'-1G-4, 0.935% tot solids, 0.891% susp solids,291 g CGO-SQ3'-1G-5, 1.101% tot solids, 1.050% susp solids,266 g CGO-SQ3'-1G-6, 1.70 % tot solids, 1.621% susp solids,166 g

These six samples were used to calibrate the opacity meter (as described in section 8) and sample FGU-SQ3'-1G-1 was used to make preliminary checks on the ICP during set-up of the MDSN nebulizer. The suspended solids are computed by multiplying the total solids by the ratio of (% susp. solids/ %tot. solids), or 4.08/4.28 (see table 8.3.2, results of test # 3, samples $SQ3'-1D \notin SQ3'-1H$).

7.4 Disposition of Samples Obtained during Test #4 (See Fig. 7.4)

The handling of the reference sample and the sample SQ3-1 were identical to that of test # 1 (.i.e. reference sample filtered through five 25 micron filters and sample SQ3-1 was filtered through a single 2.5 micron filter. Both samples were assayed at the Homer City Coal Lab.)

In addition, sample STI-1 was obtained by drawing off a the top mixing tank at a low, but constant rate through the Gilson peristaltic pump. This sample was also sent to the Homer City Coal Lab and filtered through a single 2.5 micron filter prior to analysis of solids. Although this sampling method does not guarantee a representative sample, it was relatively easy to obtain and was of interest.

All coal slurry sampled during this test was unground (mean particle size of the coal is approximately 85 microns with 25 % of particles and 3 % of particles less than 25 microns and 2.5 microns, respectively).

7.5 Opacity/Solids Testing on Field Collected Slurries

The opacity meter was used on-line in the field for tests 2 & 3. As mentioned earlier, however, the results were usable only for test # 3 (8 of 12 measurements). It was therefore desirable to reanalyze the field collected slurries, SC4, for %-solids by opacity at the B&W research facility subsequent to the field tests.

The equipment setup used for the laboratory testing was identical to the field setup with the exception that level regulation equipment (i.e. bottom sampler cup and pressure sensor) was not used. The lab setup is depicted in figure 7.5







7-6

DISPOSITION OF FIELD COLLECTED SLURRY SAMPLES FROM TEST #2 "FEED-GRINDER-UPPER FREEPORT" FIGURE 7.2



DISPOSITION OF FIELD COLLECTED SLURRY SAMPLES FROM TEST #3 "CONCENTRATE-GRINDER-OKLAHOMA" FIGURE 7.3

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FIGURE 7.4 DISPOSITION OF FIELD COLLECTED SLURRY SAMPLES FROM TEST #4 "FEED-NO GRINDER-OKLAHOMA"



EQUIPMENT ARRANGEMENT FOR "POST-SITE" OPACITY MEASUREMENTS Figure 7.5

8.0 PRESENTATION AND DISCUSSION OF RESULTS

The main purpose of the current program was to measure sulfur and ash content of the slurry streams at the Homer City test site. As described earlier, a reference sample was collected in an undiluted (i.e. same consistency as the process stream) and unground condition and, depending on the test, various samples were collected at exit locations of the multistage sampling system. Certain of the field collected samples were analyzed at the Homer City Coal Lab and the remainder at B&W by a variety of methods, including ICP analysis of slurry.

Tests 1 & 4 were conducted solely to demonstrate the representativeness of sampling on unground (i.e. -100 mesh) coal slurry while tests 2 & 3 were conducted to demonstrate the representativeness of sampling on ground samples as well as evaluate the performance of the of ICP measurement of sulfur and ash. The results of each test are discussed individually since the data reduction process varies from test to test.

8.1 Sulfur and Ash Results, Test # 1

Test # 1 was conducted on the froth flotation feed stream of an Upper Freeport coal with the grinder bypassed. Only two samples were collected, the reference sample and the "well water" diluted sample at the exit of the second stage of the multistage sampling system, SQ3-1 (see figure 6.1). The dilution ratio of well water to slurry entering the multistage sampler was approximately 9:1 (see section 6.1). The well water contained a large amount of dissolved sulfur but was relatively low in all other elements (see table 8.2.5).

The most important parameters for test # 1 slurries are described in table 8.1.1 and the laboratory results are presented in table 8.1.2. Both the reference sample and the exit sample, SQ3-1, have the same particle size distribution but, as noted in table 8.1.1, the Homer City Coal lab filtered the two samples differently - the reference sample was filtered using five 25 micron filters in series and the exit sample was filtered through a single 2.5 micron filter. The slurries had a particle size distribution in which 25 % of the particles were smaller than 25 microns and 3 % of the particles smaller than 2.5 microns. Considering the fact that the % suspended solids measured in the two slurries are in good agreement (7 % according to table 8.1.2), after accounting for the dilution ratio of approximately 9:1, the five 25 micron filters in series must not have passed a significant amount of particles smaller than 25 microns.

Comparing the concentrations of elements and of ash in the two slurries (see table 8.1.2 and figure 8.1.1) reveals that, of the major elements in coal (i.e. S, Si, Al, & Fe), S and Fe in the exit sample are approximately 20 % lower than the reference sample while the Si and Al are in good agreement. Of the remaining elements that are present in more than negligible quantities (CA, Mg, & K), the Ca is approximately 50 % lower in the exit sample while the Mg and K are in good agreement. Both the measured and calculated (using CONAC equations) ash in sample SQ3-1 are approximately 10 % lower than in the reference sample (see appendix C for a discussion of the algorithm for calculating % ash). The discrepancies may be due to one or more of the following:

- a Use of different filters sizes during laboratory sample preparation.
- b The clog which occurred in the slurry hose connecting the vezin sampler to the multistage sampler toward the end of the test (see section 6.1).

- c Leeching of the elements Ca and Mg (and to a lesser extent S) out of the coal into the well water diluted sample SQ3-1. The well water is nearly saturated with S but almost free of Ca and Mg. The Plant water is nearly saturated with all three elements. The forms of the remaining elements in the Upper Freeport coal are not soluble in water. (This phenomena is discussed in detail in the next section).
- d Insufficient agitation of the mixing tanks for the unground coal (this could result in a longer delay time for the tanks to reach steady state).
- e Error introduced by the commercially obtained vezin samplers.

Considering the above listed potential sources of error, the agreement between the reference sample and sample SQ3-1 is excellent and demonstrates the representativeness of the multistage sampling system.

Table 8.1.1

03/04/91 FILE "TESTLOG.UK1"

TEST # 1 DATA

					-	APPROX.	••••••	1SO4S10	TION & PREPI	ARATION OI	E SAMPLE	
			1	LIME	.DEL.	UE1GHT	SENT TO	ASSAY OF	ANAL OF	FILTER	FILTER DILUT	. TESTS TO BE PERFORMED
TEST ID	SAMPLE	DATE	BEGIN	END	TIME			TOTAL SLURRY	FILTRATE	TYPE	RETENTION RATI	0
			HR : MIN	I HR : MI	N(MIN)			ð	FOR	TO GET	(MICRONS) DEIO	-
						(6)		SUSP SOLIDS ONL	Y BACKGROUNI	D SOLIDS	н20	
					1 1							
FEED	REF	7-30-90	15 :53	17:50	117	82,600	H.C. LAB	SUSP SOLIDS ONL	Y NO	5×541	25 NONE	SOLIDS, S, ASH, ELEM'S
NO GRINDER	So3 - 1	7-30-90	16:5	17 :50	105	3,600	H.C. LAB	SUSP SOLIDS ONL	Y NO	# 42	2.5 NONE	SOLIDS, S, ASH, ELEM'S
UP. FREEP.												

Table 8.1.2

TEST # 1 RESULTS

			OVE	RVIEN	OF LAE	RESU	LTS, X	IN DR	Y PROD	UCT	•		:		
		X SOLI	X SO	ASH	K ASH	x s	X Si	X Al	% Fe	X Ca	X Mg	X Na	х Х	X Ti	×
TEST ID	SAMPLE	IN FIE	כ	ALC	HEAS.										
		COLLEC	TED												
		SAMPLE													
FEED	e REF	1 1 1 1 1 1	6.57 1	7.23	18.04	2.49	4.18	2.04	2.19	0.5	0.13	0.03	0.38	0.05	0.03
NO-GR I ND	e SO3 - 1	<u>+</u>	0.65 1	5.71	16.14	2.00	3.99	2.03	1.7	0.23	0.12	0.01	0.36	0.03	0.03
UP-FREEP	e SO3 - 1	6	6.11 1	5.71	16.14	2.00	3.99	2.03	1.75	0.23	0.12	0.01	0.36	0.03	0.03
	DELTA	· · ·	- 97.0	1.52	-1.90	0.49	-0.19	-0.01	-0.44	-0.27	-0.01	-0.02	-0.02	-0.02	0.00
	Z DIFF		2/-	24-	211-	202-	2C-	20	202-	242-	28- -	2/9-	×C-	205-	20
	OG = SLURF	RY FLOW 1	NTO MU	LTIST/	VGE SAM	IPLER :		0.75	l/min						

6.30 l/min QP2 = MEAS. WELL WATER FLOW INTO SAMPLER =

X SUSPEMDED SOLIDS BY FILTERING SLURRY X SOLIDS AS COLLECTED IN FIELD X SOLIDS REFERRED BACK TO PRE-WELL WATER DILUTION [i.e. X SOLIDS*(QG/QP2 +1)] a -- o

α I * X ASH CACLULATED=[2.139*(X Si) + 1.889*(X AL) + 1.43*(X Fe) + 1.399*(X Ca) + 1.348*(X Na) + 1.205*(X K) + 1.668*(X Ti)]+ 2.32*(X Mg)/[FIRST 7 TERMS]

																												_						
																		-	+	R-OK		34.53	ano	IN COAL	9.83	3.52	2.02	0.87	0.25	0.30	0.83	0.32	0.01	
																			TEST	FEED-NO G	s <u>0</u> 3-1		MOLEC &	OF ASH	60.89	19.27	8.36	3.51	1.22	1.16	2.88	1.53	0.05	0.09
		12	(z.		18.82	æ	IN COAL	3.66	1.94	4.03	0.04	0.10	гđ	ŋ	0.10	0.04		-	# 4	R-OK		31.2	đþ	IN COAL	7.76	2.68	3.50	1.91	0.20	0.15	0.59	1.68	0.01	
S IN WHICH		TEST 4	FEED-GR-UI	SQ3'-1H		MOLEC &	OF ASH	41.64	19.51	30.6	0.27	0.88	rđ	đ	0.93	0.49			TEST	FEED-NO G	ST1-1		MOLEC &	OF ASH	53.21	16.21	16.06	8.56	1.07	0.64	2.28	8.98	0.05	0.14
LL TESTS		# 2	ы		18.92	о р	IN COAL	4.34	2.17	2.41	0.55	0.13	0.02	0.38	0.03	0.02		•	# 4	R-OK		38.44	æ	IN COAL	9.04	2.83	4.71	3.12	0.21	0.15	0.67	0.17	0.03	
COAL FOR A		TEST	FEED-GR-U	REFERENCE		MOLEC &	OF ASH	49.04	21.7	18.18	4.08	1.15	0.16	2.43	0.29	0.21	0.06	_	TEST	FEED-NO G	REFERENCE		MOLEC 8	OF ASH	50.32	13.92	17.52	11.34	0.91	0.52	2.09	0.74	0.15	0.14
LEM IN C	CTED	 #	R-UF		16.14	сно С	IN COAL	3.99	2.03	1.75	0.23	0.12	0.01	0.36	0.03	0.03			# ~	Å		8.64	а ю	IN COAL	1.97	0.96	0.94	0.17	0.09	0.05	0.27	0.07	0.02	
SH TO \$ E	WAS CONDU	TEST	FEED-NO G	s <u>0</u> 3-1		MOLEC %	OF ASH	52.89	23.77	15.47	1.98	1.2	0.07	2.71	0.31	0.38	0.04	_	TEST	CONC-GR-O	H1-, 6Q3		MOLEC &	OF ASH	48.81	20.89	15.53	2.7	1.66	0.73	3.73	1.27	0.41	
1.3 AR WT OF 7	OF ASH	# 1	R-UF		18.04	عيو	IN COAL	4.18	2.04	2.19	0.50	0.13	0.03	0.38	0.05	0.03			~	К		9.16	an S	IN COAL	2.20	0.93	0.91	0.51	0.09	0.04	0.24	0.04	0.00	
TABLE 8.1. MOLECHLAR	ANALYSIS	TEST 4	FEED-NO G	REFERENCE		MOLEC %	OF ASH	49.56	21.41	17.37	3.84	1.16	0.26	2.52	0.45	0.38	0.06		TEST	CONC-GR-O	REFERENCE		MOLEC &	OF ASH	51.5	19.1	14.2	7.78	1.7	0.61	3.15	0.77	0.05	0.15
CTON OF 8	ELEMENTAL				RY COAL =	GRAVIM.	FACTOR	0.4674	0.5293	0.6994	0.7147	0.6031	0.7419	0.8306	0.5995	0.4364						RY COAL	CRAVIM	FACTOR	0.4674	0.5293	0.6994	0.7147	0.6031	0.7419	0.8306	0.5995	0.4364	
CONTED					SH IN D	MMOLEC	FORM	Si02	A1203	Fe203	CaO	MqO	Na2O	K20	Ti02	P205	MnO2					ASH IN I	NOTED	FORM	Si02	A1203	Fe203	CaO	MgO	Na2O	K20	Ti02	P205	MnO2
					8 A	ELE		l Si	2 A1	3 Fe	4 Ca	5 Mg	6 Na	7 K	8 Ti	д 6						8			1 Si	2 Al	3 Fe	4 Ca	5 Mg	6 Na	7 K	8 Tİ	9 Р	

| 0.15 - ***SUI+/CDAVIM FACTODI*/MOIFC & OF ASH)/(10

% IN COAL = (%ASH)*(GRAVIM. FACTOR)*(MOLEC % OF ASH)/(100)
a INSUFFICIENT SAMPLE MASS TO ALLOW ELEM ANAL OF NA AND K

-

8-4.1

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8-5

8.2 Sulfur and Ash Results, Test # 2

Test # 2 was conducted on the froth feed stream of an Upper Freeport coal with the grinder in-line. In addition to the unground reference sample, eight pairs of samples were collected sequentially in time at the exit of the third (bottom) sampling stage (SC4-1 thru 8) of the multistage sampler and at the drain of the third sampling stage (SC4'-1 thru 8) as shown in figure 6.2. The dilution ratio of well water to slurry entering the multistage sampler was approximately 12:1 (see section 6.2). The well water contained a large amount of dissolved sulfur but was relatively low in all other elements (see table 8.2.5). A "calibration" sample was also collected at the drain of the second stage sampler (SQ3'-1). This calibration sample was subsequently divided and diluted and used to calibrate the opacity meter, test for PSDs, and check out the ICP - making it possible to conduct these checks without depleting the small volume samples collected at SC4.

8.2.1 Results Summary

The most important parameters for test # 2 are presented in table 8.2.1 for the slurry samples and in table 8.2.3 for the filtrate of these slurry samples. Filtrates of certain samples were prepared for ICP testing of background levels subsequent to ICP testing of the slurries themselves.

The reduced results of conventional testing and ICP testing are summarized in table 8.2.2. The results of individual elemental concentrations are separated into two categories - the first assuming 85 % ICP recovery on slurry elements and the second assuming 100 % ICP recovery. The details of these computations are presented and discussed in later tables. The title "85 % ICP RECOVERY" pertains only to those quantities determined by ICP analysis (i.e. samples SC4-1 to 8). The assumed value of 85 % ICP recovery was selected after both ICP and conventional analysis was performed on the "riffles" of the calibration samples, SQ3'-1, collected during test # 2 and test # 3. Comparison of the ICP calculated sulfur concentration (sample SQ3'-1C-1) to the sulfur concentration based on conventional assay (sample SQ3'-1H) indicates that the ICP was operating at approximately 90 % recovery on test # 2's calibration sample and 85 % on test # 3's calibration sample. Studying the results of all ICP analysis of field samples (i.e. FGU-SC4-1 to 8 for test # 2 and CGO-SC4-1 to 12 for test # 3), it was surmised that the ICP was operating over a range of 80 % - 90 % recovery for sulfur. Therefore the value of 85 % was selected and used for all data reduction. The results for the theoretically achievable 100 % ICP recovery are also presented for information. The % recovery which would have to be assumed to make ICP slurry analysis agree with analysis of ash of the same sample (SQ3'-1C-1) is presented in Table 8.2.7b.

The \$-solids listed in table 8.2.2 are referenced back to the "as collected in the field" condition (i.e. including site dilution with well water but not the later laboratory dilution with deionized water). The only exception to this is for the second lines labeled "AVG SC4-1 to 8" and "AVG SC4'-1 to 8" (at the bottom of the table). For these data rows, the average \$-solids in the samples are multiplied by the ratio of $(Q_c/Q_{P2} + 1)$ in order to make a comparison to the \$-solids in the reference sample possible (i.e. 6.44 \$ \pounds 6.38 \$ versus 6.40 \$, which is excellent agreement considering the fact that the flow meter reading during the field testing varied by as much as +/- 0.5 $1/\min$).

The calculated ash concentrations are computed using the CONAC equation (ref. 16). The explicit formulation is listed at the bottom of table 8.2.2 and is discussed in appendix C. Since the CONAC equation requires the knowledge of the ash concentration prior to computing the contribution of Mg, an estimate of the ash concentration, using the first seven terms of the equation (which are known in advance), is used in the computation of Mg contribution.

The lines labeled "AVG....." at the bottom of table 8.2.2 were computed based on straight averages (rather than a mass weighted average) of the 8 quantities listed in the columns directly above them. The purpose of computing average values of the eight individual samples is to make possible a single comparison to the reference sample. This comparison is presented graphically in figure 8.2.1. From this figure it is seen that the 8-solids in the reference sample and the average of the eight SC4' samples are in excellent agreement. The average of the eight "opacity determined" %-solids is also in excellent agreement with the former. The average calculated ash in the eight SC4 samples is considerably lower than the calculated ash in the reference sample and the average measured ash in the SC4' samples is higher than the measured ash in the reference sample. The average sulfur content in the SC4 and SC4' samples is in very good agreement with the reference sample. No measurements were made on the SC4'-samples at the Homer City Coal Lab for elements other than S, therefore the remaining elemental comparisons are made between the ICP results and the reference sample results only. This comparison indicates that Si and Al concentrations determined by ICP are substantially lower than those in the reference sample, while Fe concentration is higher. The concentrations of Ca and Mg are in good agreement.

The results indicate that the sampling system performed well but that the ICPanalysis was marginal at best.

8.2.2 ICP Results Prior to Correction for Low ICP Recovery

The parameters for test 2 are repeated in table 8.2.3 for the slurry samples, the filtrates of slurry samples, site well water samples, and site clarified water samples. All slurry samples were analyzed by ICP prior to filtering to collect filtrate. In the case of clarified water, only the filtrates were analyzed and in the case of well water, no solids were present in the field collected samples.

The raw results, in PPM, of the ICP tests are presented in table 8.2.4. The % suspended solids are "as analyzed" by ICP (rather than being referenced back to the field collected condition, as is the case in other tables). The individual concentrations of each element are listed for the slurry and for the filtrate (i.e. background). The slurries were nebulized to the ICP using the ARL MDSN nebulizer while the filtrates were nebulized with a Meinhardt nebulizer, which is the standard nebulizer for solutions.

In table 8.2.5, the results of table 8.2.4 have been increased by the dilution ratio + 1 (e.g. in the case of sample SC4-1, the S content in the slurry = (5+1)*146 = 876 and the S content of the filtrate = (5+1)*131 = 786). Note that since the well water and clarified water were never diluted, the raw ICP results in table 8.2.4 are identical to the results presented in table 8.2.5. Also note that, although all slurry samples for this test (with the exception of the reference sample, which was not analyzed by ICP) were diluted with well water in the multistage sampling system by an approximate ratio of 12:1, the dilution ratio shown in the rightmost column of table 8.2.5 represents only the additional dilution performed at the B&W lab using high purity water. There are two very notable aspects of the results presented in table 8.2.5:

a The concentration of elements in the filtrate (background) of the slurries is higher than that in the plant well water and, with the exception of Ca, higher than that in the plant clarified water as well. This is a result of the significant dilution of the field slurries with high purity water at the B&W lab (recall that the values in this table were corrected upward to be consistent with the site collected %-solids). The implication is that the plant clarified water is saturated with the elements S, Ca, and Mg and the well water is saturated with the element S. The dilution with site well water permitted leeching of the elements Ca & Mg into solution and the laboratory dilution with high purity water, therefore, permitted additional leeching of these elements as well as leeching of the element S. These results also imply that the forms of S, Ca, and Mg found in the Upper Freeport coal are, to a large degree, soluble in water, while the Si, Al, and Fe are only minimally soluble in water.

b In many of the samples, the background levels of S, Ca, and Mg (as measured by ICP using Meinhardt nebulizer) are larger than those in the slurry (as measured by ICP using MDSN nebulizer). Therefore the ICP must be operating at less than 100 % recovery for one or both of the nebulizers. Since the filtrates are solutions and were nebulized with a proven nebulizer (Meinhardt), there is little doubt that almost all of the error is associated with slurry nebulization (using ARL MDSN nebulizer, which was shown to give less than 100 % recovery during the study phase of the current project - see ref. 1). This "less than 100 ICP % recovery" in the slurries combined with high background levels of S, Ca, and Mg create a serious potential for error in the measurement of element concentrations in the dry product. One is strongly tempted to use one of the elements which is not found in the plant water and which is nearly insoluble in water (such as Si, Al, or Fe) to determine the actual recovery of the ICP. However, the testing conducted during the study phase of the project indicated that the recovery of individual elements is not identical for the same slurry. This effect is most likely due to the fact that the proportions of each slurry element which are present in solid and solution form vary significantly from element to element and due to elements not being evenly distributed among all particle sizes.

After completing ICP analysis on the calibration slurry sample (SQ3'-1C-1) and its filtrate and comparing the sulfur result to the B&W lab result on a different "riffle" of that same sample (SQ3'-1H), a value of 90 % ICP recovery for sulfur can be deduced (the same analysis for the calibration sample for test 3, discussed in the next section, leads to an 85 % ICP recovery conclusion). After viewing the results of the data reduction at a number of different assumed % ICP recoveries, the value of 85 % was decided upon for data presentation. This may not be appropriate for all slurry samples or for all elements, but it permits a consistent presentation format.

The concentrations of elements (determined by ICP) in the slurries are plotted in figure 8.2.2 as a function of sample number (i.e. essentially a function of time). The values are those listed in table 8.2.5 - referenced to the field collected conditions (i.e. pre-laboratory dilution).

Figures 8.2.3 - 8.2.5 present a comparison of the element concentrations in the slurry and the filtrate for those elements which are soluble in water (S, Ca, and Mg). These plots demonstrate the problem, discussed above, of back-ground levels being higher than slurry levels.

8.2.3 ICP Results After Correction for Low ICP Recovery

As mentioned earlier, a decision to assume 85 % recovery for presentation of results was made after inspecting the calibration results and after viewing the reduced results at a number of assumed % recoveries. Table 8.2.6 contains the raw ICP data from the ICP slurry testing (three trials per slurry), ICP data from the filtrate (i.e. background) testing, and computation of the concentrations of elements in the dry product (dry suspended coal).

Two sets of computations are made - the first assuming ICP slurry recovery of 85 % and the second assuming 100 % ICP slurry recovery. The equation used for

computing the "% in dry product" is shown at the bottom of the second page of table 8.2.6.

Table 8.2.7 presents the "% in dry product" results from the previous table in summary form along with the conventional assay results on the "matching" slurries (e.g. reference slurry, SC4'-slurries, etc.). The very last line in this table presents the results for the calibration slurry, where it can be seen that the % S from B&W lab conventional assay (0.69 %) is not in agreement with the % S from the ICP analysis (1.60 %), assuming 85 % ICP recovery. Table 8.2.7a presents the same summary assuming 90 % recovery and it can be seen that the agreement between conventional assay and ICP analysis is good (0.69 % vs. 0.68 %).

Figure 8.2.6 compares the weighted average ICP results (assuming 85 % ICP recovery) to the reference sample results. This figure is nearly identical to that of figure 8.2.1 except that figure 8.2.1 uses a straight average of 8 samples, while figure 8.2.6 uses a mass weighted average of the same 8 samples. Note that with the 85 % recovery assumption, the S, Ca, and Mg are in excellent agreement while the Si and Al are low and the Fe high. The discrepancies may be due to one or more of the following:

- a Use of coarse filter on the reference sample at the Homer City Coal Lab (see discussion in section 7.2).
- b Leeching of the elements Ca and Mg (and to a lesser extent S) out of the coal into the well water diluted samples. The well water is nearly saturated with S but almost free of Ca and Mg. The Plant water is nearly saturated with all three elements. The forms of the remaining elements in the Upper Freeport coal are not soluble in water. Significant additional leeching of S into solution occurred after the samples were diluted in the B&W lab with high purity water.
- c Error introduced by the commercially obtained vezin samplers.
- d Inaccuracy of the ICP analysis of slurries using the MDSN nebulizer. This includes sample to sample variation and element to element variation.
- e Inability of the NETZSCH grinder to attain a 5 micron mean particle size (actual was 6 microns).

Figures 8.2.7 - 8.2.13 present a comparison of the individual sample results to the reference sample results. The ICP results presented assume 85 % ICP recovery. Figures 8.2.7 & 8.2.8 (% ash & % S) include results from the ICP analysis of samples SC4-1 to 8, HC Coal Lab analysis of samples SC4'-1 to 8, and the HC Coal Lab analysis of the reference sample. The remaining figures do not include HC Coal Lab results on samples SC4'-1 to 8 since the HC Coal lab did not conduct elemental analysis on the SC4' samples.

Finally, table 8.2.8 and figure 8.2.14 present the data and plots of the ICP analyses of secondary solution standards which were conducted after each individual slurry sample analysis by ICP. Both the slurry and standard solution were conducted using the MDSN nebulizer. The secondary standards used during the ICP testing were:

s	100	ppm	Fe	25	ppm
Si	25	ppm	Ca	50	ppm
A 1	25	ppm	Mg	10	ppm

The plotted results show the variability of ICP analysis using the MDSN nebulizer on solutions only and must be assumed to be worse for slurries.

		_	—					—								—				—						
	TESTS TO BE PERFORMED					ICP, OPACITY	SOLIDS, S, ASH	SOLIDS, S, ASH, ELEM'S	SOLIDS, S, PSD	ASH, S, ICP FILTRATE	SUSP SOLIDS, S	SUSP SOLIDS, S, ASH														
F SAMPLE	FILTER DILUT.	RETENTION RATIO	(MICRONS) DEION	H20		5:1	5:1	5:1	5:1	5:1	5:1	5:1	5:1	2.5 NONE	25 NONE	2:1	0.45 2:1	0.45 2:1	0.45 2:1							
ARATION 0	FILTER	TYPE	TO GET	0 SOLIDS		NONE	# 42	# 42	# 42	75 #	# 42	# 42	# 42	# 42	5×541	NONE										
ON & PREP	ANAL OF	ILTRATE	FOR	BACKGROUN	4 8 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1	YES	ON	QN	Q	QN	ON	NO	0N	NO	NO	0N	YES	NO	ON N							
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	SENT TO					8&W	B&V	B&V	B&U	884	B&U	B&W	B&u	H.C. LAB	B&U	884	8&W	884								
WE I GHT	BEFORE	IE DEION.	IN)DILUT.	(6)	1 	15 45	17 80	15 54	12 60	15 62	15 70	15 73	15 68	15 520	17 880	15 690	12 690	15 800	15 810	15 810	15 820	30 107100	b 260	b 260	b 260	b 260
	MEDEI	END TH	HR :MIN(M			18:33	18:50	19:8	19:20	19:35	19:50	20:5	20:20	18:33	18:50	19:8	19 :20	19:35	19:50	20:5	20:20	20 :15 1			••	
	TT	BEGIN	HR :MIN		, , , , , ,	90 18 :18	90 18 :33	90 18 :53	90 19 : 8	90 19 :20	90 19 :35	90 19 :50	90 20 : 5	90 18 :18	90 18 :33	90 18 :53	90 19 : 8	90 19 :20	90 19 :35	90 19 :50	90 20 : 5	90 18 : 5	: 06	: 06	: 06	: 06
		DATE				7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-	7-30-
		SAMPLE				sc4 - 1	SC4 - 2	SC4 - 3	SC4 - 4	SC4 - 5	SC4 - 6	SC4 - 7	SC4 - 8	SC41- 1	SC41- 2	SC41- 3	5C41- 4	SC41- 5	SC41- 6	SC41- 7	SC41- 8	REF B&U	a so3'-10	a, cSo3' - 1G	a, cSo3' - 1E	a, dSo3'-1H
		TEST ID				FEED	GR I NDER	UP. FREEP.	=	=	=	=	Ŧ	=	=	=	=	=	=	=	=	2	-	-		=

Table 8.2.1

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TEST # 2 DATA

COLLECTED UNSCIENTIFICALLY FOR CALIBRATION PURPOSES ONLY ORIGIN. SAMPLE WAS DILLUTED 2:1 AND RIFFLED INTO 8 VOLS, SQ3'-1A THROUGH H. APPROX 100 mL OF SAMPLE POURED THROUGH FILTER FOR ANALYSIS OF SUSP. SOLIDS ENTIRE SAMPLE POURED THROUGH FILTER FOR ANALYSIS OF SUSP. SOLIDS

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PRODUCT	USING PER SAMPLE BACKGROUND SUBTRACTION	XASH X S X SI X AL X FE X CA X M9 CALC	0.0 % - 3 % 5 1 % - 0.07	13.00 1.12 2.16 1.22 2.83 -0.55 -0.05 10.66 0.67 2.39 1.22 2.83 -0.55 -0.06 9.64 0.67 2.39 1.22 2.38 -0.87 -0.04	10.92 0.75 2.63 1.26 3.18 -1.14 -0.19	11.49 -0.94 2.60 1.39 2.81 -0.50 -0.08	9.92 -0.98 1.99 0.99 2.97 -0.50 -0.20 17 10.82 -0.50 2.38 1.30 2.30 0.02 -0.17							ZELEM (EXCEPT S) DETERMINED FROM ANAL UF ASH AT H.C. LAB		SUSP SOLIDS ONLY, XELEM (EXCEPT S) DETERM		TO PRE-WELL WATER DILUTION	TO PRE-WELL WATER DILUTION		68*(% Ti)]+ 2.32*(% Mg)/[FIRST 7 TERMS]
F DRY		2												0.02		0.04	DILUT	BACK	BACK		+ 1.6
N X O		Ţi												0.03		0.10	FIELD	ERRED	ERRED		נג ג)
JLTS.		х Х Х												.38		÷	NI SO	SC REF	DS REF		1.205*
IB RESU		6 X												.02		*	t sol II	Sol II	SOLII SOLII	т т	Ha) +
u of La		X 6W		222	.10	5.5	0.02							0.13 0		0.10	0.15	0.15		THROUG	348*(X
rs Vedvtf	CTION.	E E E		2,8,5	58	<u>ເ</u>	38.2	9						0.55		0.04	0.46	0.46		3'-1A	+
RESULT	UBTRA	Fe X		50 0 0 0 0	ğκ. b d	22.52		- 2						. 41		NLY)	17.5	.41		/min /min NLY LS, SQ	(X Ca)
LE 8.2 T # 2	OUND S	A IC		84 24 24 24 24 24 24 24 24 24 24 24 24 24	2 5 2 6 2 6	59 54 74	6 ₽ :	4 7						17 2	LIDS O	102 0 94 4	545	- 54			.399*
TAB	BACKGR			282	28 	82	 8 % 8	85 						34 2	ISP SOLV	8 8 0 1 0 1	88	88		PURPO	e) + 1
	IPLE"			283	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	2.2		-2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -2 -	រជ	5	53	25	<u>8</u> 5	3	282	1 2 2 2 3 2 3 2 3 3 3 3 3 3 3 3 3 3 3 3	518	8 8 10	2K	ER = ER = ATION	RY 3+(% F
	ER SA	ж. 		4 Mi	mm	~		~ ~ ¤	5 28 28	، نہ بو و	~ ~ 5 5	32.5	2. 	5. 75	0 0 2	ం రాల్లు జిల్లు		i ni	2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2	SAMPI SAMPI CALIBI	IETER SLUR 8 K + 1.4
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		LIDS LELD IELD ECTED	E	0.50	0.0	50	0.0		0.5	0.5	5.0		0.5	0.0	0.7	0.5 2.0		4.9 4.9	0.54	L UNTO L VATE L VATE IENTIF	UDISOLV LIDS E LIDS E MPLE 1
	::	X SO N F COLL	SAMP			<u></u>													~	I FLO S. VEL UNSC SAMPLE	DED SO DED SO DED SO DED SO DED SO A Si * (% Si
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		TEST ID		FEED GRINDE	UP-FREE		: :	=	= =	: =	=	: :	: =	2 =	=	:	:				X ASH

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Z OF QUANTITY IN DRY SUSP SOLIDS

Figure 8.2.1

4PLE	ICP ELEMS	2.5 ICP ELEMS	2.5 ICP ELEMS	ICP ELEMS	2 5 5:1 ICP ELEMS			2.5 5:1 JUP ELEMS	2.5 5:1 ICP ELEMS	2.5 5:1 ICP ELEMS	2.5 5:1 ICP ELEMS	2.5 5:1 ICP ELEMS	2.5 5:1 ICP ELEMS	1 25 2+1 ICP FIEMS		2.3 13.4.1 ICF ELENS
ATION OF SAN FILTER FILI TYPE RETE TO GET (MIC FILTRATE	NONE	# 42	4 27	NONE	C7 #	1 C	77 H	# 42	# 42	# 42	# 42	# 42	27 #			25 #
N & PREPAR NAL OF LTRATE	NA	YES	YFS	4	217	103	YES	YES	YES	YES	YES	YES	YFS		TES	
DISPOSITIO ICP ASSAY OF A SLURRY, FILTRATEFI OR BOTH	SOLUTION	FILTRATE ONLY	ETTENTE ONLY		SULUTION	SLUKKT & FILIKAIC	SLURRY & FILTRATE	SLURRY & FILTRATE	SLURRY & FILTRATE	SLURRY & FILTRATE	SLURRY & FILTRATE	SUIRBY & FILTRATE	CLURDY & ELLTDATE		FILTRATE ONLY	SLURRY & FILTRATE
SENT TO	RRU			100	00.00	B&U	B&U	B&W	884	B&U	RLU			BOW	B&U	B&U
APPROX.					2000											
MEDEL. END TIME HR :MIN(MIN)					••	<1 E: 31	18 :50 17	19:8 15	19:20 12	10 . 35 15	31 03. 01			<1 02: 0Z	••	
E BEGIN HR :MIN				: 06-20	: 06-20	50-90 18 :18	50-90 18 :33	30-90 18 :53	30-90 19 : 8	00.10.20	00 10 12 12 04 00 02 04 04 04 04 04 04 04 04 04 04 04 04 04			30-90 20 : 5	30-90 :	30-90 :
SAMPLE DAT			CLAR H20 8-1	CLAR H20 8-1	WELL H20 8-1	SC4 - 1 7-	SC4 - 2 7-	SC4 - 3 7-	Sr4 - 4 7-				SC4 - / / -	SC4 - 8 7-	SQ31-1G 7-	SQ31-1C-1 7-
TEST ID						FEED	GR I NDFR	LID FREED		: :	=	2	=	Ŧ	=	2

Table 8.2.3

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TEST # 2 DATA (AND WELL WATER AND CLARIFIED WATER DATA) ICP RAW DATA ON LABORATORY DILUTED SLURRY, SLURRY FILTRATE, WELL WATER, AND CLARIFIED WATER FILTRATE

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Table 8.2.4

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TEST # 2 (INCL. WELL WATER AND CLARIF. WATER) ICP RAW RESULTS ON LABORATORY DILUTED SLURRY, SLURRY FILTRATE, WELL WATER, AND CLARIFIED WATER FILTRATE

TEST ID	SAMPLE	z susp solids in LAB diluted slurry	S Si Al Fe (PPM) (PPM) (PPM) (P	Ca Mg Na K PPN)(PPN)(PPN)()	Ті Р (МРМ) (РРМ)	(X) Sq1 Ios	S (Mdd)	si (PPM) (Ał (PPM) (f	Fe PPM) (P	PM) (PF	9 01LL 1 RA 1 DE 1 DE 1
						0.00	665	3.3	0.0	0.0	18	
						0.00	690	0.0	0.0	0.0 4	1 02	- 5
	CLAK HZU	· · · ·				0.00	680	0.0	0.0	0.0 4	02	4
	LLAK HZU					0.00	689	0.0	0.0	0.0	22	2
	WELL HZU		0 22 0 21 0 76 771	71 07		00.00	131	0.9	0.0	0.0	. 12	5 5:
FEED	SC4 - 1	00.0 l			_	0.00	128	1.1	0.5 (0.0	51	5
GRINDER	SC4 - 2	140.0		71 74		00.00	128	1.1	0.6	0.0	92	- 1 7
UP. FREEP.	SC4 - 5	0.097	0.03 0.01 0.43 401	51 13		0.00	132	0.8	0.5 (0.5	61	5 -
5	SC4 - 4	C8U.U		5 5 5 1 7		00.00	128	1.0	0.6 (0.0	. 19	
2	sc4 - 5	0.000	0.22 0.21 0.12 021	5 C C C C C C C C C C C C C C C C C C C		0.00	133	1.0	0.6	0.0	3	
=	SU4 - 0	0.000		5 CT 05		0.00	139	0.8	0.7	0.6	62	. 7
=	SC4 - 7	140.0	120 22 0 12 0 21 0	64 12		0.00	135	1.2	0.7	0.0	3	. 7
=	SC4 - 8	0.100		i		0.00	280	2.3	1.8	0.9	124	50 2:
	sa3'-16 sa3'-1c-1	0.038	53 10.2 5.4 11.0 24	4.3 6.1		0.00	57	0.0	0.0	0.0 25	.4 5	9 13.
SOLIDS IN SI	LURRY AFTER	DILUTION WITH DEIO	NIIZED H20			ICP FIL MEINHA	TRATE RDT NE	ANALYS BULIZEI	IS PERFI	ORMED 1	41 T H	

a X SOLIDS IN SLURRY AFTER DILUTION WITH DEIONIZED H2O ICP SLURRY ANALYSIS PERFORMED WITH ARL MDSN NEBULIZER ICP RESULTS ARE THE AVERAGE OF 3 TRIALS

Table 8.2.5

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TEST # 2 (INCL. WELL WATER AND CLARIFIED WATER, UNDILUTED FIELD COLLECTED BACK TO UNDILUTED FIELD COLLECTED CONDITIONS

DILUT. RATIO DEION H20					5:1	=		=				=	2:1	13.4:1
l (Mdd)	9	45	44	2	06	8	84	06	84	20	20	78	8	2
TRATE. Ca (PPM)	18	470	470	22	282	306	336	366	366	384	372	384	372	274
IN FIL Fe (PPM)	0.0	0.0	0.0	0.0	0	0	0	m	0	0	4	0	m	0
MENTS Al (PPM)	0.0	0.0	0.0	0.0	0	m	4	m	4	4	4	4	ŝ	0
φM ELE Si (PPM)	3.3	0.0	0.0	0.0	s	~	~	ŝ	9	9	ŝ	2	2	0
JLTS, P S (PPM)	665	690	680	689	786	768	768	262	768	798	834	810	840	616
susp susp sol IDS (X)	0.00	0.00	0.00	0.00	0.00	0.00	00.0	0.00	0.00	0.00	0.00	0.00	0.00	0.00
d Wdd)														
(Mdd) įl														
(Mdd) X														
Na (MPM)														
SLURRY. Mg (PPM)					84	84	84	78	78	78	. 22	22		8
.UTED S Ca (PPM)					240	276	282	306	348	354	354	384		262
I UNDIL Fe (PPM)					198	162	138	162	144	150	174	126		119
ENTS IN Al (PPM)					78	72	78	9 9	72	78	60	78		58
A ELEME Si (PPM)	1				144	144	144	138	126	144	120	138		110
(PPM)					876	816	804	828	768	750	780	780		572
LAB RESULT	1 1 1 1 1 1 1 1 1													
X SUSP SOLIDS X SUSP SOLIDS IN FIELD COLLECTED SAMPLE	0.00	¢.	r.	0.000	0.51	0.58	0.58	0.50	0.50	0.54	0.58	0.55	0.54	0.54
SAMPLE	VELL H20	CLAR H20	CLAR H20	MELL H20	SC4 - 1	SC4 - 2	SC4 - 3	SC4 - 4	SC4 - 5	SC4 - 6	SC4 - 7	SC4 - 8	So31-16	so31C-1
TEST ID					FEED	GR I NDER	UP. FREEP.	=	=		=	=	=	Ŧ

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PPM IN PREDILUTED SAMPLE

Figure 8.2.2



SEM S IN EREDILUTED SAMPLE

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BEAN OF IN EREDIFICED STATES



BRAKES CHINIGANA NI PW WAA

RECOVERY= MULTIPLIER= FREEP.") FIELD SAMPLES .RESULTS USING PER SAMPLE BACKGROUND.ASSUMING 85% ICP RECOVERY..... S Si Al Fe Ca Mg RECOVERY= 0.85 MULTIPLIER= 1.176 TRIAL 06-27-91 FILE MFGUICP.WK1M RESULTS OF ICP TESTING ON TEST # 2 (MFEED-GRINDER-UPPER -NM K SOLIDS AFTER DILUTION 0.0850 0.0850 0.0850 × X SUSP DILUTION SOLIDS NIGH FROM FIELD PURITY H20 ----ເຈັບ

 136.8
 26.3
 12.0
 25.5
 43.6
 14.2

 140.9
 23.8
 12.8
 30.3
 44.8
 15.3

 130.7
 22.2
 12.1
 26.5
 49.8
 13.6

 130.7
 22.2
 12.1
 26.5
 49.8
 13.6

 136.1
 24.1
 12.3
 27.4
 46.1
 14.4

 4.190
 1.689
 0.375
 2.070
 2.703
 0.688

 128
 1.1
 0.52
 0
 51.4
 4.8

 8.133
 22.99
 11.77
 27.4
 5.31
 -0.43

 8.133
 22.99
 11.272
 27.4
 5.31
 -0.43

127.1 24.7 13.2 22.8 49.0 13.8 138.8 23.1 12.4 23.4 45.0 13.8 137.4 24.9 12.3 22.7 48.0 14.3 134.4 24.2 12.6 23.0 47.3 14.0 5.216 0.801 0.396 0.300 1.705 0.247 128 1.1 0.56 0 55.8 14.3 6.433 23.11 12.06 22.97 -8.45 -0.35 0.67x 2.39x 1.25X 2.36x-0.87x-0.04x 153.5 26.3 12.8 34.6 41.3 14.2 144.6 23.8 14.3 33.6 38.8 13.8 138.7 21.9 12.0 29.9 39.4 14.1 145.6 24.0 13.0 32.7 39.9 14.0 6.083 1.809 0.973 2.013 1.049 0.175 131 0.9 0 0 47.2 14.6 14.6 23.11 13.03 32.72 -7.34 -0.59 1.72x 2.72x 1.53x 3.85x-0.86x-0.07x 8 11.5 27.4 49.3 13.1 0 10.6 27.0 49.3 14.1 1 10.9 26.8 54.6 12.9 7 11.0 27.0 51.1 13.3 2 0.374 0.251 2.493 0.544 1 0.5 0.52 60.6 14.9 2 10.53 26.51 -9.53 -1.55 11.262 3.182-1.142-0.192 .RESULTS USING PER SAMPLE BACKGROUNDSSSUMING 100 % ICP RECOVERY.... S Si Al Fe Ca Mg 129.6 24.8 11.5 27.4 49.3 1 146.2 22.0 10.6 27.0 49.3 1 139.0 21.4 10.9 26.8 54.6 1 138.3 22.7 11.0 27.0 51.1 1 138.3 22.7 11.0 27.0 51.1 1 132 0.8 0.5 0.52 60.6 1 132 0.8 0.5 0.52 60.6 1 0.75x 2.63x 1.26x 3.18x-1.14x-0 55.8 14.3 -0.10 2.111 2-0.01X 0.22X 0.5 0.52 60.6 14.9 12.47 31.28 -0.52 0.801 1 1.50x 3.75x-0.06x 0.10x 0 0 47.2 14.6 15.33 38.49 -0.31 1.874 : 1.802 4.532-0.042 0.222 128 1.1 0.52 0 51.4 14.8 32.15 27.24 13.94 32.23 2.815 2.098 3.33X 2.82X 1.44X 3.33X 0.29X 0.22X 13.1 14.1 12.9 15.7 14.2 15.3 13.6 16.9 13.8 13.8 14.3 16.4 14.2 13.8 14.1 16.5 49.0 45.0 48.0 55.7 49.3 49.3 54.6 41.3 38.8 39.4 46.9 43.6 44.8 49.8 54.2 128 1.1 0.56 0 30.15 27.38 14.28 27.02 -1 3.12x 2.83x 1.48x 2.80x-34.6 33.6 33.6 22.8 23.4 22.7 27.0 25.5 30.3 32.2 27.4 27.0 26.8 31.8 11.5 10.6 13.0 13.2 12.4 12.3 14.8 12.8 14.3 12.0 15.3 12.0 12.8 12.1 14.5 131 0.9 40.29 27.34 15 4.74X 3.22X 1 0.8 25.93 1 3.112 24.7 23.1 24.9 28.5 24.8 22.0 21.4 26.7 26.3 23.8 21.9 28.2 26.3 23.8 22.2 28.3 132 882 129.6 146.2 139.0 162.7 127.1 138.8 137.4 158.2 136.8 140.9 130.7 160.2 153.5 144.6 138.7 171.3 Вч. - ~ M - NM - NM 0.0967 0.0967 0.0967 0.0967 0.0967 0.0967 0833 000 ----500 **~~**~~ 555 (PPN) 0.5 (PPN) 0.5 (PPN) 0.5 (PPN)*NULTIPLIER 3 (PPN) 0.58 3 (PPN) 0.58 3 (PPN) 0.58 6 (PPN)*MULTIPLIER (РРМ) 0.58 2 (РРМ) 0.58 2 (РРМ) 0.58 0.58 0.58 (РРМ)*МULTIPLIER FGU-SC4- 4 (PPM) 0.5 FGU-SC4- 4 (PPM) 0.5 FGU-SC4- 4 (PPM) 0.5 FGU-SC4- 4 (PPM) 0.5 AVG 05 3 (PPM) MULTIPLIER STD 05 3 (PPM) MULTIPLIER BACKGROUND (PPM) AVG MINUS BACKGROUND (PPM) IN DRY PRODUCT (X) 0.51 0.51 0.51 °LIER JUND (PPM) NUS BACKGROUND (PPM) PRODUCT (%) (Wdd) (Mdd) (PPM)*MULTIPL (PPM) 0 (PPM) 0 (PPM) 0 (PPM) 0 FGU-SC4- 2 (PPM) FGU-SC4- 2 (PPM) FGU-SC4- 2 (PPM) AVG 0F 3 (PPM) AVG 0F 3 (PPM) STD DEV BACKGROUND (PPM) AVG MINUS BACKGROUND (F IN DRY PRODUCT (2) FGU-SC4- 3 (PPH) FGU-SC4- 3 (PPH) FGU-SC4- 3 (PPH) FGU-SC4- 3 (PPH) AVG 6 3 (PPH) STD DEV BACCGROUND (PPH) AVG MIMUS BACCGROUND (I IN DRY PRODUCT (X) FGU-SC4-1 (FGU-SC4-1 (FGU-SC4-1 (AVG OF 3 STD DEV BATCGROUND (AVG MINUS BAG IN DRY PRODUG SAMPLE

TABLE 8.2.6

TEST # 2 ("FEED-GRINDER-UPPER FREEP.") FIELD SAMPLES RESULTS OF ICP TESTING ON

					RESUL	rs using	IG PER 85%	SAMPLI ICP RI	E BACK	sround.	.RESULTS USING PER SAMPLE	E BACKGROUND RECOVERY
SAMPLE	% SUSP SOLIDS FROM FIELD	DILUTION HIGH PURITY H20	X SOLIDS AFTER DILUTION	TRIAL	S	Si	AL	e L	Ca	6 W	s si Al Fe	e)
EGU-SC4- 5 (PPM) EGU-SC4- 5 (PPM) EGU-SC4- 5 (PPM) EGU-SC4- 5 (PPM) VUG OF 3 (PPM) IN DEV ACKGROUND (PPM) VUG MINUS BACKGROU N DRY PRODUCT (%)	0.5 0.5 0.5 0.5 0.5 NU (PPM)	~~~ 	0.0833 0.0833 0.0833		129.5 123.0 132.1 50.8 128 22.82 22.74X	21.2 18.9 22.4 24.5 24.5 23.45 23.45 23.45	12.3 11.6 13.5 14.6 1.69%	23.1 25.5 222.0 27.7 3.32%	59.7 60.7 52.0 67.6 6.903 0.83%	13.8 12.9 12.5 15.4 14.1 1.268 0.152	129.5 21.2 12.3 23.1 123.0 18.9 11.6 25.5 132.1 22.4 13.5 22.0 128.2 20.8 12.4 23.5 3.827 1.443 0.791 1.475 128 1 0.55 0 128 1 0.55 0 0.2 19.79 11.89 23.54 0.022 2.372 1.432 2.824	59.7 13.8 60.7 12.9 52.0 12.5 57.5 13.1 3.854 0.565 60.7 14.1 -3.23 -1.03 -0.39%-0.12%
FGU-SC4- 6 (PPM) FGU-SC4- 6 (PPM) FGU-SC4- 6 (PPM) FGU-SC4- 6 (PPM) FGU-SC4- 6 (PPM) TGU-SC4- 6 (PPM) NG DEV ACKGROUND (PPM) NG MINUS BACKGROU N DRY PRODUCT (%)	0.54 0.54 0.54 MULTIPLIER ND (PPM)	555 555	0.0900 0.0900 0.0900	- NM	128.9 122.3 122.3 122.3 146.5 133 133 133 133	23.7 21.0 24.4 27.1 27.1 26.11	14.3 12.5 15.6 15.4 1.65%	23.9 26.6 25.4 29.8 29.78 3.31%	56.7 60.3 61.2 69.9 63.9 5.974 0.66 X	12.3 13.6 15.6 15.4 13.8 13.8	128.9 23.7 14.3 23.9 122.3 21.0 12.5 26.6 122.3 24.4 12.6 25.4 124.5 23.1 13.1 25.3 3.111 1.458 0.805 1.088 133 1 0.6 0 -8.5 22.05 12.52 25.32 -0.94x 2.45x 1.39x 2.81x	56.7 12.3 60.3 13.2 61.2 13.6 59.4 13.1 1.968 0.534 63.9 13.8 63.9 13.8 63.9 13.8 51.0.75
FGU-SC4- 7 (PPM) FGU-SC4- 7 (PPM) FGU-SC4- 7 (PPM) VG OF 3 (PPM)* STD DEV STD DEV VG MINUS BACKGROUN IN DRY PRODUCT (2)	0.58 0.58 0.58 0.58 MULTIPLIER ND (PPM)	255 222	0.0967 0.0967 0.0967	- NM	133.1 129.1 126.3 152.4 139 13.35 1.38%	25.5 25.0 9.7 23.6 0.82 23.6 2.36%	5.2 12.8 12.1 12.1 0.7 1.18%	27.5 29.3 31.2 34.5 0.6 33.85 33.85	57.8 57.4 62.5 69.7 62.1 7.562 0.78 x	11.5 12.9 12.7 14.5 14.3 0.229 0.229	133.1 25.5 5.2 27.5 129.1 25.0 12.8 29.3 126.3 9.7 12.9 31.2 129.5 20.1 10.3 29.3 2.790 7.329 3.595 1.514 139 0.82 0.7 0.6 -9.5 19.25 9.613 28.69	57.8 11.5 57.4 12.9 62.5 12.7 59.2 12.4 2.357 0.612 62.1 14.3 62.1 14.3 5.0.30%-0.20%
FGU-SC4- 8 (PPM) FGU-SC4- 8 (PPM) FGU-SC4- 8 (PPM) AVG OF 3 (PPM) VG OF 3 (PPM)* STD DEV VG MINUS BACKGROUN IN DRY PRODUCT (X)	0.55 0.55 0.55 0.55 0.55 0.55 MULTIPLIER	~~~ 	7190.0 0.0917 0.0917	- NM	136.3 132.3 132.7 153.5 135 135 135 135 2.012	21.6 25.5 27.1 27.1 27.1 25.92 2.83%	12.6 12.5 12.5 14.8 14.09 1.54X	24.2 15.8 23.2 24.8 24.77 2.70%	66.7 62.5 63.4 75.5 75.5 11.50	12.2 12.8 12.0 14.5 13.9 0.609 0.07 X	136.3 21.6 12.6 24.2 132.3 25.5 12.7 15.8 122.7 22.1 12.5 23.2 130.4 23.1 12.6 21.1 5.706 1.747 0.071 3.740 135 1.2 0.7 0 135 1.2 0.7 0 -4.56 21.85 11.87 21.05	66.7 12.2 62.5 12.8 63.4 12.0 64.2 12.3 1.833 0.357 1.833 0.357 1.833 0.357 0.18 -1.56 0.18 -1.56

X IN DRY PRODUCT= [(AVG PPM - BACKGR PPM)*100]/[(X SOLIDS AFTER DILUTION)*1000000]

TABLE 8.2.6 (CONTINUED)

RESULTS OF ICP TESTING ON TEST # 2 ("FEED-GRINDER-UPPER FREEP.") FIELD SAMPLES

D. RESULTS USING PER SAMPLE BACKGROUND	S Si Al Fe Ca Mg	52.1 10.5 5.8 10.5 25.5 6.1 50.9 9.7 4.9 11.4 24.9 6.2 56.8 10.5 5.4 11.1 22.5 6.0 53.3 10.2 5.4 11.1 22.5 6.0 53.3 10.2 5.4 11.1 22.5 6.0 55.6 0.377 0.368 0.377 0.26 0.081 56.6 0 0 0 25.4 5.9 56.6 0 0 25.4 5.9 -3.33 10.23 5.366 11 -1.1 0.2 -0.88% 2.69% 1.41% 2.89%-0.29% 0.05%	133.4 22.8 12.2 26.2 53.1 13.3 4.7 2.2 0.9 1.5 2.2 0.5 3.5% 9.8% 7.6% 6.0% 4.2% 3.5%
KGROUN		6.1 6.2 7.2 7.2 7.2 7.2 7.2 7.0 3.4	
LE BAC	Ca	25.55 24.99 28.6 28.6 25.4 25.4 3.188 8.6 25.4	
Idws 3	e e	10.5 11.4 11.1 12.9 12.94 12.94	
NG PER	AL	5.8 5.4 6.3 6.3 6.3 1.663	
1-1C-1 TS USI	IS IS	10.5 9.7 10.5 12.0 12.03 3.17x	
RESUL	S	52.1 50.9 56.8 62.7 56.6 5.066 1.60%	
PLE FC	TRIAL	-NM	
IBRATION SAM	X SOLIDS AFTER DILUTION	0.0380 0.0380 0.0380	
CAL	DILUTION HIGH PURITY H20	13.21 :1 13.21 :1 13.21 :1	SC4-8) SC4-8)
	X SUSP Solids From Field	0.54 0.54 0.54 0.54 MULTIPLIER	SC4-1 THRU (SC4-1 THRU F AVG)*1002
	SAMPLE	FGU-SQ3"1C-1(PPM) FGU-SQ3"1C-1(PPM) FGU-SQ3"1C-1(PPM) AVG OF 3 (PPM) STD DEV BACKGRUU AVG MNUS BACKGRU IN DRY PRODUCT (2)	AVG OF AVERAGES (AVG OF STD. DEV. ((AVG OF STD/AVG OF

TABLE 8.2.7

RECOVERY= 0.85 RECOVERY= 1 MULTIPLIER= 1.176 MULTIPLIER= 1

RESULTS OF ICP TESTING ON TEST # 2 ("FEED-GRINDER-UPPER FREEP.") FIELD SAMPLES

		ъ.	0.07 0.05 0.19 0.12 0.12 0.12 0.12	061 -	0.05
	CIND.	ů.		u ucc.	0.29
	CT BACKGR			19X	-
	PRODU MPLE 1	Al Al	28. 28. 29. 29. 29. 29. 29. 29. 29. 29. 29. 29	-412	. 41 2
	PER SI	si x	28 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	45X	\$. \$.
	USING USING MG 100	<u>ر</u> ،	23622385 B	10 4.	88 88
	ULTS 1	HS.	- 0000000	15 2.8 1814 -	17 -0.
	RES	CALCA	100 00 00 00 00 00 00 00 00 00 00 00 00	17.6	12.
		ъ В н	0.142 0.22 0.22 0.15 0.15 0.17 0.142	0.130 91	0.34
	ROUND	ъ.	0.29 0.29 0.28 0.28 0.28 0.28 0.28 0.28 0.28 0.28	- 9 %	0.84
	BACKG BACKG COVERY	res	2.23	40%	3.41
	r PROD SAMPLE ICP RE	k SAMP Al	1.56 1.56 1.56 1.56 1.58 1.58	.170 2 -30%	3.1
	IN DR	× 50		.340 2 -35 X	3.17
	USING NG	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	72 33 33 50 72 50 72 50 72 50 72 50 72 50 72 50 72 50 72 50 72 50 72 50 50 50 50 50 50 50 50 50 50 50 50 50	.810 4 -4X	1.60
	SULTS SSUMI	ASH X	75 22 22 22 23 24 24 24 24 24 24 24 24 24 24 24 24 24	615 2. -18X	10.
		CALC	425455554 <u>3</u>	17.0	16
		ВW	0.13 0.13 0.13 0.13 0.13 0.13 0.13 0.13		0.1
		ů	0.55 0.55 0.55 0.55 0.55 0.55 0.55 0.55		N ASH.
	SAMPLE	F.	2.22.22.24		LENS 1 4.03
	DRY PR RENCE	VI	2.17		LAB E 1.94
,	X IN	Si	****		88N 3.66
	HC LAE	s	555555555555		0.69
;;;		ASH	888888888		7.33
		ASH X	71 292 112 292 117 292 117 293 117 203 117 200	- 26	8. 1
5	::	.s	80 K0	18	98 : 98 :
5	:-LAB.	SAMPL	2 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5		LAB
		SC4'	20.32 20.32 20.31 20.32 20.33 20.33 20.33 20.33 20.33 20.33 20.53		-B&V 18.8 18.8
	:		520 880 690 880 880 880 880 880 880 880 880 880 8		
		SC4 MGHT.	- 00 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	PLE RENCE	-1 58:
		SAMPLE	SC4 - SC4 - SC4 - SC4 - SC4 - SC4 - SC4 - SC4 - SC4 -	REF SAM X DIFFE	se3/-1c so3/1H

* X ASH COMPUTED USING ONLY THOSE ELEMENTS LISTED e.g. X ASH = [2.139*(XSi) + 1.889*(X Al) + 1.43*(X Fe) + 1.339*(XCa)] + 2.32*(X Mg)/(FIRST 4 TERMS)

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Table 8.2.7a

RECOVERY= 0.9 MULTIPLIER= 1.111

RECOVERY= 1 MULTIPLIER= 1

RESULTS OF ICP TESTING ON TEST # 2 ("FEED-GRINDER-UPPER FREEP.") FIELD SW/PLES

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RESULTS USING PER SAMPLE BACKGROUND ASSUMING 100 % ICP RECOVERY	CALC ASH S Si Al Fe Ce	13.00 1.72 2.72 1.53 3.65 -0.86 -0. 10.66 0.67 2.39 1.25 2.83 -0.55 -0. 10.92 0.75 2.63 1.26 3.18 -1.14 -0. 11.25 0.02 2.37 1.43 2.62 -0.39 -0. 11.17 -0.94 2.45 1.39 2.81 -0.50 -0. 9.92 -0.98 1.99 0.99 2.97 -0.30 -0. 10.846 0.108 2.394 1.282 2.860 -0.54 -0. 17.615 2.810 4.340 2.170 2.410 0.550 0.1 -382 -962 -452 -412 192 -1992 -1	12.17 -0.88 2.69 1.41 2.89 -0.29 0.
DRY PRODUCT R SAMPLE BACKGROUND X ICP RECOVERY	SC4 SAMPLES.	1.70 4.28 -0.34 0.11 1.36 3.15 -0.02 0.12 1.39 3.16 -0.46 -0.12 1.41 3.54 -0.46 -0.12 1.55 3.18 0.38 0.05 1.19 3.53 0.38 0.06 1.55 3.13 0.23 0.08 1.11 3.30 0.38 -0.06 1.11 3.30 0.38 0.05 1.11 3.30 0.38 0.06 1.11 3.30 0.38 0.06 1.11 3.30 0.38 0.06 1.11 3.30 0.38 0.06 1.11 3.30 0.38 0.06 1.45 2.55 0.80 -0.02 1.45 2.410 0.550 0.46 1.2170 2.410 0.550 0.130 1.3 -32X -79X -65X	0 1.57 3.22 0.42 0.23
	GALC ASH S Si * * * *	3 15.36 3.62 3.03 3 12.74 2.41 2.66 3 13.56 2.61 2.65 3 13.56 2.61 2.65 3 13.57 1.75 2.65 3 13.59 0.59 2.65 3 12.10 0.51 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 3 13.154 1.758 2.65 7 -25x -367 -36	- 14.59 0.68 2.99
X IN DRY PRODUCT	Si Al Fe Ca Mg		B&W LAB ELEMS IN ASH 5.66 1.94 4.03 0.04 0.
	.ES XASH XASH S NEAS CALC	88 18.92 17.62 2.81 13 18.92 17.62 2.81 11 18.92 17.62 2.81 11 18.92 17.62 2.81 13 18.92 17.62 2.81 14 18.92 17.62 2.81 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4 18.92 17.62 2.81 4	 59 18.84 17.33 0.69 ⁻
HC-LAB.	AMPLE SC4SC4' SAMPL UGHT. UGHT XASH S (9) (9) WEAS X	C4- 1 45 520 21.08 2.1 C4- 2 80 880 20.71 2.4 C4- 3 54 690 20.71 2.4 C4- 3 54 690 20.71 2.4 C4- 5 6.00 20.46 2.1 2.4 C4- 5 6.00 20.47 2.4 2.4 C4- 5 6.00 20.77 2.4 2.4 C4- 7 70 810 20.31 2.5 2.5 C4- 7 73 810 20.31 2.2 2.5 C4- 8 68 820 20.22 2.2 2.5 C4- 8 68 820 20.62 2.4 2.5 2.5 C4- 8 68 820 20.62 2.4 2.5 2.5 C4- 8 68 820 20.62 2.4 2.5	03'-1C-1 585 780 18.84 LAB 03'-1C-1 585 780 18.84 0.4 18.84 0.4

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* X ASH COMPUTED USING ONLY THOSE ELEMENTS LISTED e.g. X ASH = [2.139*(XSi) + 1.889*(X Al) + 1.43*(X Fe) + 1.339*(XCa)] + 2.32*(X Mg)/(FIRST 4 TERMS)

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TABLE8.2.7bTEST2

WHAT & RECOVERY WOULD HAVE TO HAVE BEEN USED IN TABLE 8.2.7 IN ORDER TO GET ICP ELEM IN COAL SLURRY SQ3'-1C-1 TO AGREE WITH ELEM CONCENTR. FROM ANAL OF ASH IN SAMPLE SQ3'-1H ?

FOR	ASSUME ICP RECOVERY OF				
S	90%				
Si	74%				
Al	73%				
Fe	72%				
Ca	95%				
MG	978				
*CALC ASH	81%				

* USING ONLY THE FIVE ELEMENTS LISTED



FIGURE 8.2.6

Using only those elements shown

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TEST #2

8-25.1

FIGURE 8.2.6a

TEST #2: ASH RESULTS: ICP VS REF & SC4 SAMPLES

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1000000 X80 NI HSV %





1000099 YAO NI IS %

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СP

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SAMPLE NUMBER REF +

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Figure 8.2.9

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TOUGORA YAO NI IA %

 ∞ + TEST #2: IRON RESULTS: ICP VS REF SAMPLES ICP RECOV. ASSUMED TO BE 85% ("FGUFE") +9 IСР + S SAMPLE NUMBER REF + + Figure 8.2.11 + 4 + \sim \sim + \sim 9 ഹ 4 ς 3 0 -

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CALCIUM RESULTS: ICP VS REF SAMPLES ICP RECOV. ASSUMED TO BE 85% ("FGUCA") TEST #2:



2000084 ABO NI PO %





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_ 30 TEST #2 SECONDARY STANDARD RESULTS DURING ICP TESTING OF FGU-SC4-1 to 8

	TIME SLUE	кку	TIME	S	Si	Al	Fe	Ca	Mg
	SAMI	PLE	AFTER	(100)	(22)	(22)	(25)	(20)	(10)
	PREC	CEDING	START						//
	STAN	NDARD	(min)	(mdd)	(mdd)	(mdd)	(mdd)	(mdd)	(mqq)
06/22/11	CAL	IBRATE							
	۹.35 د		0	106.8	22.57	24.27	25.05	46.98	9.73
2	0 • 40		S	114.7	23.41	25.6	24.08	49.63	9.15
=			101	100	24.05	24.47	22.71	44.94	10.07
: 3			- - -	95.09	22.53	29.49	22.66	47.01	10.09
: 1		F	40	100.6	21.07	26.17	22.67	47.56	9.3
: =		4 C	5 Y	95.98	20.99	25.02	21.72	43.08	9.09
: 2		, , , , , , , , , , , , , , , , , , ,	06	98.55	23.67	23.9	23.93	29.38	8.72
: =		v	115	109.2	24.13	24.13	21.75	48.88	8.87
: 2		ALTRDATE	205						
: 3	10 . U NEVU	TUNNTIN	020	91,56	20.49	24.66	25.35	49.73	8.84
:	CZ: CT	ı			0 1 0	75 94	23.38	47.6	8.93
8	13 : 20	D	667	74.07	2.12			20.75	α
=	14:20	9	285	92.97	22.96	Z5.41	23.12		
=	14:45	7	310	97.85	21.55	24.09	24.74	53.02	9. 16
=	15 :20	~ ~ ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	345	91.74	22.18	23.09	24.74	50.56	8.63
=	15:25	1	350	91.39		23.16	23.56	53.28	8.23
	MEA	N		98.63	22.42	24.96	23.58	47.29	9.12
	TRU	Ε	• • • •	100.00	25.00	25.00	25.00	50.00	10.00
	BIA	S	•	1.37	2.58	0.04	1.42	2.71	0.88
	STD) DEV	• • • •	6.88	1.15	1.55	1.12	5.65	0.52
	VAR	VIANCE	• • • •	47.32	1.32	2.41	1.26	31.93	0.27

Figure 8.2.14



PPM PER ICP

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8.3 Sulfur and Ash Results, Test # 3

Test # 3 was conducted on the froth concentrate stream of an Oklahoma coal with the grinder in-line. In addition to the unground reference sample, twelve pairs of samples were collected sequentially in time at the exit of the third (bottom) sampling stage (SC4-1 thru 12) of the multistage sampler and at the drain of the third sampling stage (SC4'-1 thru 12) as shown in figure 6.3. The dilution ratio of well water to slurry entering the multistage sampler was approximately 7:1 (see section 6.3). The well water contained a large amount of dissolved sulfur but was relatively low in all other elements (see table 8.3.5). A "calibration" sample was also collected at the drain of the second stage sampler (SQ3'-1). This calibration sample was subsequently divided and diluted and used to calibrate the opacity meter, test for PSDs, and check out the ICP - making it possible to conduct these checks without depleting the small volume samples collected at SC4.

8.3.1 Results Summary

The most important parameters for test # 3 are presented in table 8.3.1 for the slurry samples and in table 8.3.3 for the filtrate of these slurry samples. Filtrates of certain samples were prepared for ICP testing of background levels subsequent to ICP testing of the slurries themselves.

The reduced results of conventional testing and ICP testing are summarized in table 8.3.2. The results of individual elemental concentrations are separated into two categories - the first assuming 85 % ICP recovery on slurry elements and the second assuming 100 % ICP recovery. The details of these computations are presented and discussed in later tables. The title "85 % ICP RECOVERY" pertains only to those quantities determined by ICP analysis (i.e. samples SC4-1 to 12). The assumed value of 85 % ICP recovery was selected after both ICP and conventional analysis was performed on the "riffles" of the calibration samples, SQ3'-1, collected during test # 2 and test # 3. Comparison of the ICP calculated sulfur concentration (sample SQ3'-1G-1) to the sulfur concentration based on conventional assay (sample SQ3'-1H) indicates that the ICP was operating at approximately 85 % recovery on test # 3's calibration sample. Studying the results of all ICP analysis of field samples (i.e. FGU-SC4-1 to 8 for test # 2 and CGO-SC4-1 to 12 for test # 3), it was surmised that the ICP was operating over a range of 80 % - 90 % recovery for sulfur. Therefore the value of 85 % was selected and used for all data reduction. The results for the theoretically achievable 100 % ICP recovery are also presented for information. The % recovery which would have to be assumed to make the ICP slurry analysis agree with the ash analysis of the same sample (SQ3'-1G-1) is presented in Table 8.3.7a.

The \$-solids listed in table 8.3.2 are referenced back to the "as collected in the field" condition (i.e. including site dilution with well water but not the later laboratory dilution with deionized water). The only exception to this is for the second lines labeled "AVG SC4-1 to 12" and "AVG SC4'-1 to 12" (at the bottom of the table). For these data rows, the average \$-solids in the samples are multiplied by the ratio of (Q_c/Q_{P2} +1) in order to make a comparison to the \$-solids in the reference sample possible (i.e. 22.62 \$ \pounds 21.75 \$ versus 23.86 \$, which is excellent agreement considering the fact that the flow meter reading during the field testing varied by as much as +/- 0.5 l/min).

The calculated ash concentrations are computed using the CONAC equation (ref. 16). The explicit formulation is listed at the bottom of table 8.3.2 and is discussed in appendix C. Since the CONAC equation requires the knowledge of the ash concentration prior to computing the contribution of Mg, an estimate of the ash concentration, using the first seven terms of the equation (which are known in advance), is used in the computation of Mg contribution.

The lines labeled "AVG....." at the bottom of table 8.3.2 were computed based

on straight averages (rather than a mass weighted average) of the 12 quantities listed in the columns directly above them. The purpose of computing average values of the twelve individual samples is to make possible a single comparison to the reference sample. This comparison is presented graphically in figure 8.3.1. From this figure it is seen that the %-solids in the reference sample and the average of the twelve SC4' samples are in good agreement. The average of the twelve "opacity determined" &-solids is also in good agreement with the former. The average calculated ash in the twelve SC4 samples is considerably lower than the calculated ash in the reference sample and the average measured ash in the SC4' samples is higher than the measured ash in the reference sample. The average sulfur content in the SC4 and SC4' samples is in very good agreement with the reference sample. No measurements were made on the SC4'samples at the Homer City Coal Lab on elements other than S, therefore the remaining elemental comparisons are made between the ICP results and the reference sample results only. This comparison indicates that Si and Al concentration determined by ICP are slightly lower than those in the reference sample, while Fe concentration is slightly higher. The concentration of Ca is lower, while the concentration of Mg is in good agreement. These results are consistent with the results of test # 2.

The results indicate that the sampling system performed well but that the ICPanalysis was marginal at best.

8.3.2 ICP Results Prior to Correction for Low ICP Recovery

The parameters for test 3 are repeated in table 8.3.3 for the slurry samples, the filtrates of slurry samples, site well water samples, and site clarified water samples. All slurry samples were analyzed by ICP prior to filtering to collect filtrate. In the case of clarified water, only the filtrates were analyzed and in the case of well water, no solids were present in the field collected samples.

The raw results, in PPM, of the ICP tests are presented in table 8.3.4. The % suspended solids are "as analyzed" by ICP (rather than being referenced back to the field collected condition, as is the case in other tables). The individual concentrations of each element are listed for the slurry and for the filtrate (i.e. background). The slurries were nebulized to the ICP using the ARL MDSN nebulizer while the filtrates were nebulized with a Meinhardt nebulizer, which is the standard nebulizer for solutions.

In table 8.3.5, the results of table 8.3.4 have been increased by the dilution ratio + 1 (e.g. in the case of sample SC4-1, the S content in the slurry = (5+1)*121 = 726 and the S content of the filtrate = (5+1)*110 = 660). Note that since the well water and clarified water were never diluted, the raw ICP results in table 8.3.4 are identical to the results presented in table 8.3.5. Also note that, although all slurry samples for this test (with the exception of the reference sample, which was not analyzed by ICP) were diluted with well water in the multistage sampling system by an approximate ratio of 7:1, the dilution ratio shown in the rightmost column of table 8.3.5 represents only the additional dilution performed at the B&W lab using high purity water.

There are two very notable aspects of the results presented in table 8.3.5:

a The concentration of the elements Ca and Mg in the filtrate (background) of the slurries is higher than that in the plant well water and, in the case of Mg, higher than that in the plant clarified water as well. This is a result of the significant dilution of the field slurries with high purity water at the B&W lab (recall that the values in this table were corrected upward to be consistent with the site collected %-solids). The implication is that the plant clarified water is saturated with the elements S, Ca, and Mg and the well water is saturated with the element S. The dilution with site well water permitted leeching of the elements Ca & Mg into solution and the laboratory dilution with high purity water, therefore, permitted additional leeching of these elements. These results also imply that the forms of Ca and Mg found in the Oklahoma coal are, to a large degree, soluble in water, while the S, Si, Al, and Fe are only minimally soluble in water.

b In many of the samples, the background levels of S, Ca, and Mg (as measured by ICP using Meinhardt nebulizer) are nearly as large as those in the slurry (as measured by ICP using MDSN nebulizer) and in a couple of instances are actually larger. Therefore the ICP must be operating at less than 100 % recovery for one or both of the nebulizers. Since the filtrates are solutions and were nebulized with a proven nebulizer (Meinhardt), there is little doubt that almost all of the error is associated with slurry nebulization (using ARL MDSN nebulizer, which was shown to give less than 100 % recovery during the study phase of the current project - see ref. 1). This "less than 100 ICP % recovery" in the slurries combined with high background levels of S, Ca, and Mg create a serious potential for error in the measurement of element concentrations in the dry product. One is strongly tempted to use one of the elements which is not found in the plant water and which is nearly insoluble in water (such as Si, Al, or Fe) to determine the actual recovery of the ICP. However, the testing conducted during the study phase of the project indicated that the recovery of individual elements is not identical for the same slurry. This effect is most likely due to the fact that the proportions of each slurry element which are present in solid and solution form vary significantly from element to element and due to elements not being evenly distributed among all particle sizes.

After completing ICP analysis on the calibration slurry sample (SQ3'-1G-1) and its filtrate and comparing the sulfur result to the B&W lab result on a different "riffle" of that same sample (SQ3'-1H), a value of 85 % ICP recovery for sulfur can be deduced (the same analysis for the calibration sample for test 2, discussed in the previous section, leads to a 90 % ICP recovery conclusion). After viewing the results of the data reduction at a number of different assumed % ICP recoveries, the value of 85 % was decided upon for data presentation. This may not be appropriate for all slurry samples or for all elements, but it permits a consistent presentation format.

The concentrations of elements (determined by ICP) in the slurries are plotted in figure 8.3.2 as a function of sample number (i.e. essentially a function of time). The values are those listed in table 8.3.5 - referenced to the field collected conditions (i.e. pre-laboratory dilution).

Figures 8.3.3 - 8.3.5 present a comparison of the element concentrations in the slurry and the filtrate for S and those elements which are soluble in water (Ca and Mg). These plots demonstrate the problem, discussed above, of background levels being nearly as high or higher than slurry levels.

8.3.3 ICP Results After Correction for Low ICP Recovery

As mentioned earlier, a decision to assume 85 % recovery for presentation of results was made after inspecting the calibration results and after viewing the reduced results at a number of assumed % recoveries. Table 8.3.6 contains the raw ICP data from the ICP slurry testing (three trials per slurry), ICP data from the filtrate (i.e. background) testing, and computation of the concentrations of elements in the dry product (i.e. dry suspended coal).

Two sets of computations are made - the first assuming ICP slurry recovery of 85 % and the second assuming 100 % ICP slurry recovery. The equation used for computing the "% in dry product" is shown at the bottom of the second page of table 8.3.6.

Table 8.3.7 presents the "% in dry product" results from the previous table in summary form along with the conventional assay results on the "matching" slurries (e.g. reference slurry, SC4'-slurries, etc.). The very last line in this table presents the results for the calibration slurry, where it can be seen that the % S from B&W lab conventional assay (0.48 %) is in excellent agreement with the % S from the ICP analysis (0.468 %), assuming 85 % ICP recovery.

Figure 8.3.6 compares the weighted average ICP results (assuming 85 % ICP recovery) to the reference sample results. This figure is nearly identical to that of figure 8.3.1 except that figure 8.3.1 uses a straight average of 12 samples, while figure 8.3.6 uses a mass weighted average of the same 12 samples. Note that with the 85 % recovery assumption, the S and Mg are in excellent agreement while the Si, Al, and Ca are low and the Fe high. The discrepancies may be due to one or more of the following:

- a Use of coarse filter on the reference sample at the Homer City Coal Lab (see discussion in section 7.3).
- b Leeching of the elements Ca and Mg out of the coal into the well water diluted samples. The well water is nearly saturated with S but almost free of Ca and Mg. The Plant water is nearly saturated with all three elements. The forms of S, Si, Al, & Fe in the Oklahoma coal are not soluble in water.
- c Error introduced by the commercially obtained vezin samplers.
- d Inaccuracy of the ICP analysis of slurries using the MDSN nebulizer. This includes sample to sample variation and element to element variation.
- e Inability of the NETZSCH grinder to attain a 5 micron mean size (actual was 8 microns).

Figures 8.3.7 - 8.3.13 present a comparison of the individual sample results to the reference sample results. The ICP results presented assume 85 % ICP recovery. Figures 8.3.7 \pounds 8.3.8 (% ash \pounds % S) include results from the ICP analysis of samples SC4-1 to 12, HC Coal Lab analysis of samples SC4'-1 to 12, and the HC Coal Lab analysis of the reference sample. The remaining figures do not include HC Coal Lab results on samples SC4'-1 to 12 since the HC Coal lab did not conduct elemental analysis on the SC4' samples.

Finally, table 8.3.8 and figure 8.3.14 present the data and plots of the ICP analyses of secondary solution standards which were conducted after each individual slurry sample analysis by ICP. Both the slurry and standard solution were conducted using the MDSN nebulizer. The secondary standards used during the ICP testing were:

m
m
m
m
m
m

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The plotted results show the variability of ICP analysis using the MDSN nebulizer on solutions only and must be assumed to be worse for slurries.

Table 8.3.1

TEST # 3 DATA

					2	FIGHT			ISOdS I 0	TION & PRE	PARATION 0	F SAMPLE		
			1	1ME	DEL. B	EFORE	SENT TO		ASSAY OF	ANAL OF	FILTER	FILTER DILUT.	TESTS TO BE PERFORMED	
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		-	NIW- dh	HR MIN	DUNIS	ILUT.			×	FOR	TO GET	(MICRONS) DEION		•
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											1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1 1			
			17 .15	02. 21	: ¥	55	RRU	101	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
CONCENTRATE	- +1S	04-10-0		2/. C	2 f	5 2	вки	1	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
GRINDER	SC4 - 2	8-11-90	20: 01		2 5		R	2 1	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
OKLAHOMA	SC4 - 5	04-10-2	14: CI	14 - 15	2 2	2.5	RÅL	2 01	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	<u> </u>
E :	5C4 - 4	06-10-0	2 : 41	51. 41 12. 11	: 2	3 5	BRU	10	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
: :	5 - 713 5 - 7	04-10-0 8-01-00	CE- 71	57- 21	i ti	797	B&U	2	ITAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
: :	504 - 0 504 - 0	04-10-0 0-10-8	14 - 14	1 · 1	71	2.5	B&U	21	TAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
: :		04-10-0 B-01-00	5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	15 - 14	÷ F	22	B&U	2	TAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
: =	504 - 50 517 - 0	8-01-00	15 -16	15 : 32	16	61	B&U	5	DTAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	·
: :	sr4 - 40	8-01-00	15 - 74 24 - 74	15 : 40	; £	12	B&U	2	DTAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
: :	504 - 10 504 - 11	04-10-0	2		ς Έ	60	B&U	2	TAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	
; 1	sr4 - 12	8-01-00		16 :22	16	3 5	B&W	Ĕ	DTAL SLURRY	YES	NONE	5:1	ICP, OPACITY, ICP BACKGR.	<u> </u>
	SC4 - 12	8-01-00	13 - 15	13 :30	: £	760	H.C. LA	เ ย รูเ	INO SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
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: :	304 5	8-01-00	27- 21	0 - 71	5	740	H.C. LA	NB SL	INO SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
: =	SC41- 4	8-01-00	2 : 71	14 :16	14	710	H.C. LA	NB SL	USP SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
: 1	5 + 7 - 5 5 - 7 - 5	8-01-90	21: 71	14 :31	14	660	H.C. LA	AB SL	INO SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
: 1	507 P	8-01-00	C2 - 71	14 -45	1	620	IH.C. LV	NB SI	IND SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
: =	SC41- 7	8-01-90	14 :46	15:0	14	002	H.C. LA	NB SL	IND SOLIDS ONI	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	-
. 2	SC41- 8	8-01-90	15 : 1	15 :14	13	640	H.C. U	AB SI	USP SOLIDS ON	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
=	5	8-01-90	15 : 16	15 :32	16	680	[н.с. <i>LI</i>	AB SL	USP SOLIDS ON	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
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=	SC41-11	8-01-90	15 :50	16:5	15	640	H.C. LJ	AB SI	USP SOLIDS ON	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
Ξ	5641-12	8-01-90	16:6	16 :22	16	750	H.C. L	AB SI	USP SOLIDS ON	LY NO	# 42	2.5 NONE	SOLIDS, S, ASH	
=	H RRU RFF	8-01-90	13:8	16 :11	183	b18200	H.C. L	AB SI	USP SOLIDS ON	LY NO	5x541	25 +3.5 (SOLIDS, S, ASH, ELEM'S	
:	CO-RFF	8-01-90	13:8	16:11			H.C. L	AB SI	NO SOLIDS ON	LY NO	5x541	25 NONE	SOLIDS, ASH	
=	a So31-10	8-01-90	14 : 4	••		c 400	B&V	Ĩ	OTAL SLUKRY	NO	NONE	1:1	SOLIDS, S, PSD	
:	a.dso3'-1A	8-01-90	14 : 4			c 400	B&U	Ñ	USP SOLIDS ON	LY YES		0.45 1:1	ASH, S, ICP BACKGR.	
=	a dso3'-1E	8-01-90	14 : 4	••		c 400	884	Ŝ	USP SOLIDS ON	ILY NO		0.45 1:1	SUSP SOLISD, S	
=	a, eSQ3'-1H	8-01-90	14 : 4	••		c 400	884	ŝ	USP SOLIDS ON	ILY NO		0.45 1:1	SUSP SOLISD, S, ASH	

COLLECTED UNSCIENTIFICALLY FOR CALIBRATION PURPOSES ONLY

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FIELD SAMPLE WAS COLLECTED IN TWO CONTAINERS. AT TEST CONCLUSION, THE FIRST WAS EMPTIED INTO THE SECOND. THE FIRST WAS THEN RINSED WITH 3.5 (DISTILLED WATER AND EMPTIED INTO THE SECOND. 18.2 (REPRESENTS THE UNDILUTED VOLUME ORIGIN. SAMPLE WAS DILLUTED 1:1 AND RIFFLED INTO 10 VOLS, SO3'-1A THROUGH J.

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APPRCX 100 mt OF SAMPLE POURED THROUGH FILTER FOR ANALYSIS OF SUSP. SOLIDS

ENTIRE SAMPLE POURED THROUGH FILTER FOR ANALYSIS OF SUSP. SOLIDS

	V PRODUCT	ZASH X S X SI X AL X RECOVERY	3.72 0.13 1.04 0.50 0.41 -0.04 0.03 4.59 0.14 1.22 1.13 0.50 0.51 0.02 4.59 0.14 1.22 0.61 0.59 0.01 0.04 4.50 0.22 1.32 0.61 0.59 0.01 0.02 4.80 0.22 1.32 0.61 0.59 0.01 0.02 5.01 0.22 1.32 0.66 0.68 0.03 0.05 5.01 0.22 1.32 0.66 0.75 0.01 0.05 5.67 0.27 1.54 0.75 0.01 0.05 5.67 0.25 1.76 0.89 0.11 0.05 5.54 0.53 1.76 0.86 0.14 0.05 6.54 0.48 1.56 0.73 1.20 0.06 0.07	ZELEM (EXCEPT S) DETERMINED FROM ANAL OF ASH AT H.C. LAB	SUSP SOLIDS ONLY, XELEM (EXCEPT S) DETERM FROM AMALYSIS OF ASH IN B&U LAB	TO PRE-WELL WATER DILUTION TO PPE-VELL WATER DILUTION	THE SECOND. ESENTS % SOLIDS IN THE UNDILUTED SLURRY. 68*(% Ti)1+ 2.32*(% MG)/[FIRST 7 TERMS]
		× •		0.00	0.02	DILUT	6 REPR + 1.6
		X Ti		0.04	0.07	FIELD ERRED FIELD FRED	PTIED 23.84
	SULTS	×		0.24	0.27	DS IN DS REF DS REF DS REF	WAS EW COND.
		% N3		0.04	0.05		FIRST THE SE Na) +
		SM X	0.066	0.09	0.09	0.10	, THE INTO 48*(X
)LTS	UVERV	X Ca	089 (0000) 145 (0000)	0.51 DS)	0.17	0.29	LUSION MPTIED + 1.3
3.2 3 RESU	SUBTR	- *	2000 100 100 100 100 100 100 100 100 100	P SOLI	0.94	.88	/min /min vLY T CONC AND EI (X Ca)
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% OF QUANTITY IN DRY SUSP SOLIDS

ID SAM	alq	DATE	BEGIN HR :MI	TIME	.DEL. 1 TIME V(MIN)	FIGHT	SENT TO	ICP ASSAY OF A SLURRY, FILTRATEFI OR	NAL OF LTRATE	FILTER TYPE TO GET	FILTER RETENTION (MICRONS)	DILUT TESTS TO BE PERFOR Ratio Deion
					1	(6)		BOŤH		FILTRAT	E	H20
	L H20	7-30-90	16 :35			1000	B&U	SOLUTION	NA	NONE		ICP ELEMS
C	NR H20	8-01-90	10 20	~		2000	884	FILTRATE ONLY	YES	# 42	2.5	ICP ELEMS
CL	NR H20	8-07-90	••			2000	B&U	FILTRATE ONLY	YES	# 42	2.5	ICP ELEMS
	L H20	8-07-90	••			2000	B&U	SOLUTION	NA	NONE		ICP ELEMS
TRATE SC4		8-01-90	13 :15	13:30	15		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
ER SC4	N 1	8-01-90	13 :32	3 :3 :45	13		B&V	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
HOMA SC4	- m	8-01-90	13 : 47	7 14 : 0	13		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
SCI		8-01-90	14 : 2	2 14 :16	14		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
SC2		8-01-90	14 :17	7 14 :31	14		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
SC.	• •	8-01-90	14 :32	214 :45	13		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
205	· · ·	8-01-90	14 :46	5 15 : 0	14		884	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
25		8-01-90	15 : 1	15 :14	13		B&N	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
205) O ,	8-01-90	15 :16	15 :32	16		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELENS
	10	8-01-90	15 : 34	15 :49	15		B&W	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
		8-01-00	15 :50	16:5	15		B&U	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
	-1-	8-01-90	16:6	5 16 :22	16		884	SLURRY & FILTRATE	YES	# 42	2.5	5:1 ICP ELEMS
	21-1A	8-01-90	,				1884	FILTRATE ONLY	YES		0.45	1:1 ICP ELEMS
			,					CLUDRY & FILTRATE		C7 8	2	12 0.1 1 P E E LC

TEST # 3 DATA ICP RAW DATA ON LABORATORY DILUTED SLURRY, SLURRY FILTRATE, WELL WATER, AND CLARIFIED WATER FILTRATE

Table 8.3.3 st # 3 DATA

Table 8.3.4

TEST # 3

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ICP RAW RESULTS ON LABORATORY DILUTED SLURRY, SLURRY FILTRATE, WELL WATER, AND CLARIFIED WATER FILTRATE

				3 RESULTS, PI	M ELEME	INTS IN	LAB DI	LUTED	SLURRY				RESUL	TS, PP	M ELEM	ENTS IN	A FILTR	ATE	.	
	TEST ID	SAMPLE	X SUSP	(Hdd) S	Si (PPM) (F	Al PM) (P	Fe C PM) (PP	H) (PF	ig Na M) (PPM)	K (Ndd)	Ti (PPM) (G (Wdd	USP OL IDS () (Hdd S	Si PPM) (Al PPM) (F	Fe PPM) (P	Ca PM) (P	Mg 0 PM) R 0	LUT. ATIO EION
			LAB DILUIED SLURRY								1			6 6 6 7 7						H20
		UFLL K20	0.00										0.00	665	3.3	0.0	0.0	18		
		CLAP H20											0.00	690	0.0	0.0	0.0 4	20	45	—
		CLAR H20											0.00	680	0.0	0.0	0.0 4	22	- 77	
		UELL H20	0.000										0.00	689	0.0	0.0	0.0	22	2	
C	CONCENTRATE	sr4 - 1	0.812	121	35.0 40	0.0 34	.0	80	6				0.00	110	0.7	0.0	0.0	61	13 5	
	CDIMPED		0.780	120	39.0 39	0.0 39	.0 6	5	9				0.00	103	0.7	0.0	0.0	62	13	
		2 - 42S	0.702	124	\$6.0 4	5.0 42	.0	7	2				0.00	114	0.0	0.0	0.0	68	14	
		504 - 7 507 - 7	0.667	126	36.0 4;	0 41	.0 6	5	2				0.00	112	0.0	0.0	0.0	67	13	
0 4	=	5 + 50 Srb - 5	0.570	611	5.0 3	.0 35	.0 6	5	6				0.00	106	0.0	0.0	0.0	61		
	=	504 - A	0.570	112	74.0 3	2.0 39	.0 6	2	ñ			-	0.00	63	0.0	0.0	0.0	56		
0 4		50 - 20 SF6 - 7	0.558	124	36.0 3	7.0 42	9	2	5				0.00	109	0.0	0.0	0.0	65	12	
	=	576 - R	0.570	121	0.10	2.0 42	.0	0	5				0.00	106	0.0	0.0	0.0	6 8	12	
8-	=	50 - 50 SF4 - 0	0.467	119	0.0	7.0 42	.0		5				0.00	112	0.0	0.0	0.0	8	13	
43	=	SC4 - 10	0.382	110	57.0 3	1.0 45	.0	2	7				0.00	100	0.0	0.0	0.0	62		
• •	=	sc4 -11	0.163	107	1 0.01	\$.0 3C	.0 5	.0	2				0.00	8	0.0	0.0	0.0	22	=	
	3	sc4 -12	0.327	115	51.0 2	4.0 39	.0 5	4					0.00	100	0.0	0.0	0.0	52	*	 z
	3	A1 - 1702	2,040										0.00	361	1.4	1.6	0.0 2	90	42 1	.
	Ξ	so3 - 16-1	0.276	51	37 18	3.6 16	.4 27.	4 7	m			-	0.00	25	0.0	0.0	0.0 27	•••		.8:1
æ	X SOLIDS IN S	SLURRY AFTER	DILUTION WITH DEIONI	ZED HZU										PATE /	ISA INN	S DERF		HTH.		
	ICP SLURRY AN	VALYSIS PERFC	REMED WITH ARL MOSN N	EBULIZER									MEINHA	NET NET	SUL IZER					
	ICP RESULTS A	ARE THE AVERA	IGE OF 3 IKIALS																	

ICP RESULTS ON SLURRY, SLURRY FILTRATE, WELL WATER, AND CLARIFIED WATER, CORRECTED BACK TO UNDILUTED FIELD COLLECTED CONDITIONS TEST # 3

Table 8.3.5

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	<u>- 11</u>	110	NOI	120									-					-				8:1
<u>-</u>	11a 6	<u>s</u>	8 						<u> </u>											-		5 13.
Ш. Ш.	Ĭ	ldd) (Ū	4	41		2	22	20	22	8	3	22	22	22	3	3	3	20	ĸ
LTRAT	са Са	Mdd) (18	470	470	22	365	372	408	402	366	336	390	408	396	372	312	312	412	399
IN FI	Fe	(Mdd)			0.0	0.0	0.0	0.0	0	0	0	0	0	0	0	0	0	0	0	0	0	0
EMENTS	٩I	(Mdd)			0.0	0.0	0.0	0.0	0	0	0	0	0	0	0	0	0	0	0	•	m	0
PM ELE	Si	(Hdd)			3.3	0.0	0.0	0.0	4	4	0	0	0	0	0	0	0	0	0	0	m	0
LTS, P	s	(Mdd)			6 65	690	680	689	0 99	618	78 9	672	636	558	654	636	672	600	594	600	222	695
RESU	Sp	SOLIDS	3		0.00	0.00	0.0	0.00	0.0	0.00	0.00	8.0	.00	00.0	0.0	8.0	00.0	00.0	8.0	00.0	0.00	8.0
	P S	H) S(_			_	_	_	_	_	_	_	_	_	_	_	_	_	_	_	_	_
		H) (P																				
• • • • • • • • • •		IG9) (1																				
	-	idd) (
	Na	Mdd) (
SLURR	БW	(Mdd)							8	8	<u>&</u>	8	8	82	8	8	8	2	22	82		108
LUTED	Ca	(Mdd)							348	378	402	390	378	372	396	414	426	402	336	324		405
I UND I	Fe	(Mdd)							204	234	252	246	234	234	252	252	252	270	180	234		242
NTS I	١v	(Hdd)							240	234	258	222	204	192	222	252	222	186	28	144		273
B RESULTS, PPM ELEME	Si	(Hdd)							510	534	516	516	450	777	516	546	474	402	174	306		547
	s) (Mdd			1 1 1				726	720	ንተረ	756	714	672	772	726	714	660	642	690		754
		J																				
n	X SUSP SOLIDS	IN FIELD	COLLECTED	SAMPLE	0.00	~	د.	0.000	4.87	4.68	4.21	4.00	3.42	3.42	3.35	3.42	2.80	2.29	0.98	1.96	4.08	4.08
		Ē			H20	HZO	H20	HZO	-	~	m	4	5	\$	~	80	0	10		12	X	16-1
		SAMPL			NELL	CLAR	CLAR	NELL	- * 2C	- +22	- 7 2	- 40S	- † 25	SC4 -	- * 2C	- * 25	SC4 -	sc4 -	- * 2C	- * 22	503'-	5031-
		TEST ID							CONCENTRATE	GRINDER	OKLAHOMA	=	=	z	=	2	Ŧ	=	T	z	Ŧ	I
																		8-	-4	4		



PPM IN PREDILUTED SAMPLE





PPM Co IN PREDILUTED SAMPLE

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8-47


PPM Mg IN PREDILUTED SAMPLE

8-48

Figure 8.3.5

TABLE 8.3.6

RECOVERY= 0.85 MULTIPLIER= 1.176

RECOVERY= MULTIPLIER=

06-27-91 FILE "CGOICP.WK1"

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RESULTS USING PER SAMPLE BACKGROUND	s si Al Fe Ca Mg	116.5 80.4 40.5 31.1 55.5 17.5 126.4 88.0 40.2 36.8 58.0 14.5 119.0 86.3 40.6 32.9 59.7 15.1 120.6 84.9 40.4 33.6 57.7 15.7 4.20 3.22 0.17 2.40 1.74 1.30 110 0.7 0 0 60.6 13.1 10.63 84.18 40.41 33.59 -2.9 2.636 0.131 1.042 0.502 0.412-0.042 0.033	113.5 84.4 36.0 38.3 59.9 15.8 128.3 92.9 40.7 38.9 62.4 15.6 118.7 88.4 39.4 39.2 66.1 16.8 120.2 88.6 38.7 38.8 62.8 16.0 6.13 3.48 1.97 0.36 2.53 0.50 103 0.7 0 0.61.8 12.8 17.16 87.86 38.69 38.78 0.966 3.246 0.222 1.132 0.502 0.502 0.012 0.042	119.8 89.2 43.3 46.7 61.6 15.0 127.0 81.4 39.7 41.4 66.5 15.6 124.0 87.0 44.5 42.9 72.6 14.7 123.6 85.9 42.5 41.7 66.9 15.1 2.95 3.28 2.04 0.89 4.49 0.37 114 0 0 68.2 13.8 9.6 85.85 42.49 41.65 -1.34 1.28 9.6 85.85 42.49 41.65 -1.34 1.28	124.4 95.8 41.1 39.3 65.6 14.9 131.0 86.8 37.3 39.6 62.5 13.9 123.7 76.6 46.9 43.1 67.2 15.2 126.4 86.4 41.7 40.7 65.1 14.6 3.29 7.87 3.95 1.77 1.95 0.52 112 0 0 66.9 12.8 14.36 86.40 41.74 40.65 -1.80 1.843 0.222 1.302 0.633 0.613-0.033 0.033
INDER-OKLAHOMA") FIELD SAMPLES .RESULTS USING PER SAMPLE BACKGROUND. assuming 85 % ICP RECOVERY	DSTRIAL S Si Al Fe Ca Mg ER TON	1117 1 116.5 80.4 40.5 31.1 55.5 17.5 1117 2 126.4 88.0 40.2 36.8 58.0 14.5 1117 3 119.0 86.3 40.6 32.9 59.7 15.1 141.9 99.9 47.5 39.5 67.9 18.5 110 0.7 0 0 60.6 13.1 31.92 90.16 47.54 39.51 7.282 5.413 0.392 1.222 0.592 0.492 0.092 0.072	7800 1 113.5 84.4 36.0 38.3 59.9 15.8 7800 2 128.3 92.9 40.7 38.9 62.4 15.6 7800 3 118.7 88.4 39.4 39.2 66.1 16.8 7800 3 118.7 88.4 39.4 39.2 66.1 16.8 7800 3 118.7 88.4 39.4 59.2 66.1 16.8 7800 3 118.7 88.4 55.5 45.6 73.8 18.9 703 0.7 0 0 61.8 12.8 38.37 103.4 45.52 45.62 12.04 6.078 0.492 1.333 0.582 0.582 0.1552 0.087	7017 1 19.8 89.2 43.3 40.7 61.6 15.0 7017 2 127.0 81.4 39.7 41.4 66.5 15.6 7017 3 124.0 87.0 44.5 42.9 72.6 14.7 7017 3 124.0 87.0 44.5 42.9 72.6 14.7 7017 3 124.0 87.0 49.0 49.0 7 77.7 7 145.4 101.0 50.0 49.0 7 77.7 77.7 31.41 101 49.99 49.00 10.45 3.941 31.41 101 49.99 49.00 10.45 3.941 0.455 1.442 0.7712 0.702 0.152 6.063	6667 1 124.4 95.8 41.1 39.3 65.6 14.9 6667 2 131.0 85.8 37.3 39.6 62.5 13.9 6667 3 123.7 76.6 46.9 43.1 67.2 15.2 148.7 101.7 49.1 47.8 76.6 17.2 36.66 101.6 49.10 47.82 9.680 4.427 0.55x 1.52x 0.74x 0.72x 0.15x 0.07x
RESULTS OF ICP TESTING ON TEST # 3 ("CONCENTR-GR	SAMPLE X SUSP DILUTION X SOLI Solids High Aft From Field Purity Dilut H20	CGO-SC4- 1 (PPM) 4.87 5:1 0.8 CGO-SC4- 1 (PPM) 4.87 5:1 0.8 CGO-SC4- 1 (PPM) 4.87 5:1 0.8 CGO-SC4- 1 (PPM) 4.87 5:1 0.8 AVG OF 3 (PPM)*MULTIPLIER STD DEV STD DEV BACKGROUND (PPM) AVG MINUS BACKGROUND (PPM) IN DRY PRODUCT (%)	CGO-SC4- 2 (PPN) 4.68 5 :1 0.7 CGO-SC4- 2 (PPN) 4.68 5 :1 0.7 CGO-SC4- 2 (PPN) 4.68 5 :1 0.7 AVG OF 3 (PPN)*MULTIPLIER STD DEV BACKGROUND (PPN) AVG MINUS BACKGROUND (PPM) IN DRY PRODUCT (%)	CGO-SC4- 3 (PPM) 4.21 5 :1 0.7 CGO-SC4- 3 (PPM) 4.21 5 :1 0.7 CGO-SC4- 3 (PPM) 4.21 5 :1 0.7 CGO-SC4- 3 (PPM) 4.21 5 :1 0.7 AVG OF 3 (PPM)*MULTIPLIER STD DEV BACKGROUND (PPM) AVG NINUS BACKGROUND (PPM) IN DRY PRODUCT (%)	CGO-SC4- 4 (PPM) 4.00 5 :1 0.6 CGO-SC4- 4 (PPM) 4.00 5 :1 0.6 CGO-SC4- 4 (PPM) 4.00 5 :1 0.6 AVG 0 3 (PPM)*MULTIPLIER STD DEV BACKGROUND (PPM) AVG MINUS BACKGROUND (PPM) IN DRY PRODUCT (%)

TABLE 8.3.6 (CONTINUED)

RESULTS OF ICP TESTING ON TEST # 3 ("CONCENTR-GRINDER-OKLANOMA") FIELD SAMPLES

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						SULTS	USING 8	PER SA	P RECO	ACKGR	. ONUC	RESUL	TS US	ING PE	K SAM	LE BA	CKGROU VERY	2
SAMPLE		X SUSP SOLIDS FROM FIELD	DILUTION High Purity H20	X SOLIDS AFTER DILUTION	TRIAL	S	Si	Y	e	Ca	Б М	S	Si	Ĭ	Z	υ A	æ.	D.
CGO-SC4- CGO-SC4- CGO-SC4- CGO-SC4- AVG OF 3 STD DEV BACKGROUND AVG MINUS B IN DRY PROD	5 (PPH) 5 (PPH) 5 (PPH) (PPH)+ (PPH)+ ACKGROUN	3.42 3.42 3.42 multiplier D (PPM)	555 555	0.5700 0.5700 0.5700	1116 3116 33.1 0.0	8 76 9 74 7 75 7 7	1 34 7 34 7 40 7 40. 562 0.	.2 40 .5 40 .0 35 .3 45 .5 45. 71% 0.	.2 64 .6 58 .8 63 .7 73 .7 73 .0 60 .0 60 .0 50 .0 70 .0 br>70 .0 70	6.4 6.4 6.6 6.6 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7 7	5.2 5.8 8.3 8.3 7 2 2 1 1 2 2 1 1 2	116.8 121.9 116.7 118.5 2.43 106 12.46 0.223	7.72 7.75 7.62 7.62 7.62 7.62	34.5 34.5 34.22 37.22 0.20 0.20 0.00	40.9 33.35.1 38.1 30.0 30.0 30.0 30.0 30.0 30.0 30.0 30	58. 58. 58. 58. 58. 58. 58. 58. 58. 58.	8 15. 8	0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0
CGO-SC4- CGO-SC4- CGO-SC4- CGO-SC4- AVG OF 3 STD DEV BACKGROUND AVG MINUS B AVG MINUS B IN DRY PROD	6 (PPH) 6 (PPH) 6 (PPH) (PPH) 1 (PPH) ACKGROUNI	3.42 3.42 3.42 3.42 MULTIPLIER D (PPM)	555	0.5700	30110 3110 3110 30.0 30.0		9 33 2 32 3 30 3 37 .2 37 .23 37.	.8 38 .2 39 .7 39 .9 46 .9 46 .6% 0.	.7 63 .5 63 .4 59 .1 72 .1 72 13 16. 81% 0.	29% 0.11 29% 0.11 29% 0.11	5.3 5.8 5.8 5.3 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0 5.0	106.4 119.7 5.60 5.60 92.4 19.66	75.9	33.8 32.22 30.7 32.21 32.21 1.28 32.21 0 32.21 5 0.57	866 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	63. 63. 63. 59. 55. 73. 7.73 56. 7.73 56. 7.73 7.73 7.73 7.73 7.73 7.73	0 13. 0	-0.1.4.15.4.15.15
CGO-SC4- CGO-SC4- CGO-SC4- CGO-SC4- AVG OF 3 STD DEV BACKGROUND AVG MINUS B IN DRY PROD	7 (PPH) 7 (PPH) 7 (PPH) (PPH) 6 (PPH) ACKGROUM	3.35 3.35 3.35 3.35 MULTIPLIER D (PPN)	555	0.5583 0.5583 0.5583	121 221 231 231 231 231 231 232 232 232	25 1.1 80 101 0.2 20 101 0.2	7 38 2 36 5 36 3 43 3 43. 3 43.	-0 41 -2 39 -2 39 -5 49 -0 -0 -0 -0 -0 -0 -0 -0 -0 -0 -0 -0 -0	.9 63 .4 67 .6 77 .6 67 .6 7 .6	244-1 50 WW	100% 201 201 201 201 201 201 201 201 201 201	126.3 121.1 125.0 125.0 124.1 2.21 109 15.13 0.273	95.7 80.2 86.1 6.82 6.82 6.82 1.54 1.54	38.0 36.2 36.8 37.0 0.73 0.73 0.73	41. 45. 7. 7. 7. 7. 7. 7.	6.7. 6.7. 6.7. 6.7. 6.7. 6.7.	24 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5 5	2.5.8.9.5 - 8.3 X
CGO-SC4- CGO-SC4- CGO-SC4- CGO-SC4- AVG OF 3 STD DEV BACKGROUND AVG MINUS B IN DRY PROD	8 (PPN) 8 (PPN) 8 (PPN) (PPN) ACKGROUN	3.42 3.42 3.42 3.42 MULTIPLIER D (PPM)	555	0.5700 0.5700 0.5700	- 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	8 4	9 44 3 40 5 49 5 49	.1 46 .0 40 .2 39 .1 49 .13 49. .852 0.	.1 67 .6 69 .5 819 .5 813 .5 813 .5 813 .5 813 .5 813 .0 68	23X 0.11	5.6 5.0 7.8 0.3 11%	120.4 120.2 123.2 123.2 123.2 15.26 15.26 0.277	84.9 87.3 99.5 99.5 90.6 90.6 1.59	40-04 40-04 41-24 41-28 41-26 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-76 41-77 410 41-77 410-77 410-77 410-77 410 410-77 410 410-77 410 410-77 410 410 410-77 410 410 410 410 410 410 410 410 410 410	40.0 2.2 2.0 40.0 70.0	22 28 29 29 29 29 29 29 29 29 29 29 29 29 29	15.0 15.0 15.0 15.0 15.0 15.0 15.0 15.0	% % % % % % % %

TABLE 8.3.6 (CONTINUED)

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RESULTS OF ICP TESTING ON TEST # 3 ("CONCENTR-GRINDER-OKLAHOMA") FIELD SAMPLES

				.RESI	JLTS USI	ING PER	SAMPLE	BACKGR COVERY	OUND.	RESULT	NISU S	100 X	SAMPLE ICP R	BACKGR ECOVERY	
SAMPLE	X SUSP SOLIDS FROM FIELD	DILUTION HIGH PURITY H20	X SOLIDS AFTER DILUTION	TRIAL	S.	Ĩ	Fe	Ca	Б Ж	S	Si	AL	Fe	Ca	б м
CGO-SC4-9 (PPM) CGO-SC4-9 (PPM) CGO-SC4-9 (PPM) CGO-SC4-9 (PPM) STD DEV STD DEV STCGROUND (PPM) IN DRY PRODUCT (%)	2.80 2.80 2.80 2.80 *MULTIPLIER ND (PPM)	888 555	0.4667 0.4667 0.4667	1112. 2121. 3123.1 140. 11 28.0 0.6	2 74.8 1 76.3 8 85.8 0 92.9 3 92.89 0% 1.999	40.0 35.7 35.1 43.5 43.46 40.93%	38.4 44.4 42.4 49.1 1.05 1.05 1.05 1	66.7 1 74.2 1 70.9 1 83.1 1 83.1 1 65.5 1 7.56 5. 7.56 5.	15.1 14.6 17.6 17.6 039	112.2 121.1 123.8 119.0 4.96 4.96 7.033 7.033 0.15 X	74.8 76.3 85.8 79.0 79.0 4.84 1.69%	40.0 35.7 35.1 35.1 26.9 0.79	38.4 44.4 42.4 41.7 2.48 0.89%	66.7 76.2 70.9 70.6 3.08 65.5 .106 2.11% (0.11\% (0.11\% (0.11\%)))))))))))))))))))))))))))))))))))	15.1 14.6 15.0 1.30 1.30 12.6 12.6
CGO-SC4- 10 (PPM) CGO-SC4- 10 (PPM) CGO-SC4- 10 (PPM) CGO-SC4- 10 (PPM) SCD DEV STD DEV BACKGROUND (PPM) AVG MINUS BACKGROU IN DRY PRODUCT (%)	2.29 2.29 2.29 2.29 2.29 4MULTIPLIER	555	0.3817 0.3817 0.3817	1114- 2 103. 3 110- 128- 99- 29-0	6 70.0 9 65.4 8 79.2 8 79.2 6 2.07 6 2.07	29.1 33.7 28.6 35.9 35.85 20.94%	46.3 44.5 53.0 53.01 1.39%	64.1 72.2 78.9 61.9 0.45% (14.7 12.4 15.9 10.5 1.14 X	114.6 103.9 110.0 4.38 99.8 9.7 0.25%	70.0 65.4 66.5 67.3 1.96 7.28 7.28	29.1 33.7 28.6 30.5 2.31 2.31 2.31 0.80%	46.3 44.5 44.5 45.1 0.90 5.06 5.18%	64.1 66.8 67.1 3.68 61.9 9.14%	14.7 12.4 13.5 13.5 10.5 0.08%
CGO-SC4- 11 (PPM) CGO-SC4- 11 (PPM) CGO-SC4- 11 (PPM) AVG 0F 3 (PPM) STD DEV BACKGROUND (PPM) AVG MINUS BACKGROU IN DRY PRODUCT (X)	0.98 0.98 0.98 0.98 0.98 1.00 1.00	5555 5555	0.1633 0.1633 0.1633	1 109. 2 111. 3 100. 126. 98.	1 28.3 8 28.9 4 28.9 0 33.7 5 33.74 8% 2.07	14.5 14.9 12.7 16.5 16.5 1.01x	29.6 30.6 35.3 35.33 1 2.16%	56.5 57.8 54.2 66.1 52.2 3.86 3 0.85%	11.8 11.0 13.7 13.7 10.5 .237 0.20 X	109.1 111.8 100.4 107.1 4.85 98.5 8.65 8.65 0.532	28.3 28.9 28.7 28.7 0.31 0.31 1.76 X	14.5 14.9 12.7 14.00 14.02 0.86%	29.6 30.6 30.0 0.41 1.84 x	56.5 557.8 557.8 554.2 554.2 554.2 1.51 1.51 1.51 1.51 1.51 1.51 0.24 x	11.8 11.7 11.7 11.7 10.53 10.53 0.07 X
CGO-SC4- 12 (PPM) CGO-SC4- 12 (PPM) CGO-SC4- 12 (PPM) CGO-SC4- 12 (PPM) AVG OF 3 (PPM) STD DEV AVG MHUS BACKGROU AVG MHUS BACKGROU IN DRY PRODUCT (X)	1.96 1.96 1.96 1.96 1.96	5.5.5	0.3267 0.3267 0.3267	1119. 2109. 3116. 3116. 35.9	7 50.4 7 55.2 7 47.6 7 60.1 8 0 8 0 0% 1.84	24.0 23.3 24.3 28.1 28.1 28.05 28.05	39.7 37.8 39.6 45.9 45.94	54.2 53.2 53.7 63.2 51.8 11.36 4 0.35%	12.3 13.1 12.5 14.9 .350 0.13 x	119.7 109.7 116.7 115.4 9.19 99.19 99.19 15.56	50.4 55.2 47.6 51.1 3.12 3.12 1.56 x	24.0 24.3 24.3 0.42 0.42 0.73%	39.7 37.8 39.5 39.1 0.87 1.20%	54.2 53.7 53.7 53.7 53.7 53.8 0.6%	12.3 13.1 12.6 12.6 10.5 0.34 0.07

X IN DRY PRODUCT = [(AVG PPM - BACKGR PPM)*100]/[(X SOLIDS AFTER DILUTION)*100000]

TABLE 8.3.6 (CONTINUED)

RESULTS OF ICP TESTING ON TEST # 3 ("CONCENTR-GRINDER-OKLAHOMA") FIELD SAMPLES

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ASSUMING PER SAMPLE BACKGROUND	S Si Al Fe Ca Mg	49.6 40.9 19.4 16.0 28.3 7.3 53.3 33.7 18.1 17.8 26.6 7.0 49.4 36.5 18.2 15.4 27.2 7.5 50.8 37.0 18.6 16.4 27.4 7.3 1.79 2.96 0.59 1.02 0.70 0.21 46.8 0.0 0.0 0.0 27.0 5.1 3.966 37.03 18.56 16.4 0.366 2.366 0.14x 1.34x 0.67x 0.59x 0.01x 0.08x	118.1 74.8 34.5 39.4 63.3 14.4 3.9 3.8 1.5 1.5 2.3 0.5 3.3X 5.0X 4.3X 3.8X 3.6X 3.4X
. UND.	Б Ж	7.3 7.5 3.5 3.5 12%	
BACKGR(OVERY .	Ca	8.3 6.6 7.2 7.0 7.0 19% 3.0	
SAMPLE Icp rec	e e	16.0 2 17.8 2 19.3 3 19.3 3 0.0 2 0.29 5. 0.70% 0	
G PER 9 85 %	Al	19.4 18.1 18.2 21.8 21.8 1.84 19 0.79%	
-1G-1 IS USIN SSUMING	Si	40.9 33.7 36.5 43.6 43.6 0.0 1.58%	
- So3' - RESUL1	S	49.6 53.3 49.4 59.7 12.92 12.92 10.47%	
י כפנ	TRIAL	- NM	
BRATION SAM	X SOLIDS AFTER DILUTION	0.2760 0.2760 0.2760	
CALI	DILUTION HIGH PURITY H20	13.78 :1 13.78 :1 13.78 :1	54-12) 564-12)
	X SUSP SOLIDS FROM FIELD	4.08 4.08 4.08 4.08 Multiplier	-1 THRU SC 24-1 THRU S 1VG)*100%
	-	1G-1(PPM) 1G-1(PPM) 1G-1(PPM) (PPM)*)*)*)*)*) (PPM)*)*)*)*)*)*)*)*)*)*)*)*)*)*)*)*)*)**)*	ERAGES (SC ¹). DEV. (SC TD/AVG OF A
	SAMPLE	CGO-SQ3'- CGO-SQ3'- CGO-SQ3'- CGO-SQ3'- AVG OF 3 STD DEV BACKGROUNI AVG MINUS IN DRY PR(AVG OF AVI AVG OF ST(AVG OF S1

												RECON	VERY= IPLIER	0.8 = 1.17	v, x0				RECOVER	:Y= .IER=				
RESULTS OF ICP	TESTING	on tes	T#3("CONCE	NTR-G	R NDER	-OKLAH	("AMO	FIELD	SAMPLE	ES													
		HC-LAB.			ĤĊ	LAB R	EFEREN	roduct Ce sam	PLE		RE	sults u	.% IN SING P ING 85	DRY PH Er Sah	ODUCT.	CKGROU ERY	- ON	RESU	LTS USI ASSUMING	IN DRY IG PER	PRODUC SAMPLE	CT BACKG	ROUND.	: : :
SC4 SAMPLE WGHT. (9)	sc4's .NGHT.XA (g) ME	AMPLES. ISH S AS X	XASI- MEAS	H XASH		s.	¥	U L	Ğ	Ĩ	g CALC	ASH X X	S S	SC4	AMPLES	0 K	е	CALC AS	× ^v	si x		2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	× G	. Z ×
sc4- 1 65 sc4- 2 59	760 7. 650 8	72 0.6	6 9.16 7 9.16	5 8.50 5 8.50	0.7	1 2.5	56°0 3	10.91 10.91	0.51	0.0	9 9 - 7 5	58 0.39 03 0.49	3 1.22	21 0.55 26 0.55	35 0.44 33 0.56	X 0.06	9 0.066	3.72	0.131 0.220	1.126 0	.497 0 .496 0	- 413 -	0.03 0	.032 .041
SC4- 3 62 SC4- 4 63	740 §. 710 9.	3.9 0.6 45 0.6	57 9.14 56 9.14	5 8.50 5 8.50	0.7	~ ~ ~	0.92	0.9	0.5	0.0	~~~ ~~~	66 0.44 91 0.5	7 1.4 5 1.5 5	39 0.7 24 0.7 34 0.7	12 0.6 36 0.7 8 0.8	7.0 20 1.0 71 2.0 71	9 0.05 5 0.064 7 0.104	v. 4 . 80	0.215 0.218 0.218	277 1.323 0 825 1.323	0 929.0 0 929.0	- 609.0	0.02 0	.027
SC4- 5 59 SC4- 6 46	660 9. 620 9. 700 0	-59 0.6 91 0.7 66 0.6	8 0 0 1 0 0 0 1 0 0 0 1 0 0 0 0 0 0 0 0	6 8.50 5 8.50 5 8.50	0.7 0.7 7.0		6 0 6 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0	9.0 5 9.0 2 9.0 2	0.5.0	0.0	 	13 0.69 97 0.66	1.5 1.8	30 0.6 14 0.7	8 8 0 8 8 0 8 8 0 8	99 0.25 37 0.25	2 0.09	5.67	0.345	1.542 0	0.565 0 0.662 0	. 6 87 0 . 754 0	.100 0	.051
sc4- 8 52 sc4- 9 61	640 9 680 10	91 0.6	55 9.1t	6 8.5C	0.7	~ ~ ~	2 0.9	6 0 0 6 0 0	0.0	1 0.0	~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~	22 0.64 08 0.60 86 0.75	8-1-00 	69 0.8 90 0.9 74 0.9	62 0.8 31 1.0 39 1.3	67 0.2 51 0.3 38 0.4	28 0.10 76 0.10 5 0.14	8.2 2.6 1.1 1.1	0.267 0.150 0.254	1.589 (1.692 (1.762 (0.732 0.732 0.791 0.791 0.798 1). 737 0 . 893 0 . 180 0	. 109 (vcu. 120.1
SC4- 10 77 SC4- 11 60 SC4- 12 69	770 13 640 15 750 12	9.0 10.0 10.0	- 8 9 - 1 8 9 - 1 8 9 - 1	6 8 5 8 5 8 5 7 1 7	0.7		2 0 0 0 2 0 0	2 0 0 0	0.5	1 0.0		65 1.66 09 1.05	83 2.0 99 1.8	66 1.0 39 0.8	10 2.1	63 0.8 06 0.3	48 0. 19 48 0. 13	8 8.3 5 6.5	0.526	1.756 (1.563 (0.858 1 0.730 1	1.838 0	0.057 (.072
WEIGHTED AVG	10	.45 0.1	69 9.1	6 8.5(0.7	1 2.	2 0.9	3 0.9	1 0.5	1 0.0	9 6.9	7.0.76	22 1.6	96 0.7	86 1.0	04 0.2	97 0.10	5 5.66	0.267	1.441 (0.668 (0.853 (0.053 (0.51
REF SAMPLE			9.1	9							60	500 0.7	10 2.2	00 0.9	30 0.9	10 0.5	10 0.09	0 9.16	0.710	2.200 (0.930 (0.910 (0.510 (060.0
X DIFFERENCE												-18%	- x2	- 752	15%	10X -	1 727	Ř	29- X8	X7E- 3	-28%	%9 -	X 06-	277-
	8&	LAB				:	B&N LA	B ELEM	S IN A	нс														
sa3' - 16- 1 sa3' - 1H	8 8 008 8	.65 0.	48 43 8.6	5 7.6	4. 0.4	68 1.9	7 0.9	6 0.9	4 0.1	7 0.(و کو	.18 0.4	68 1.5	78 0.7	91 0.6	98 0.1	88 0.12	4 5.0	4 0.143	1.341	0.672	0.594 (0.013	0.078
* X ASH COMP e.g. X ASH :	JTED USIN : [2.139*	ig only (XSi) +	THOSE E	CIA X)	s L15'	TED 43*(X	fe) +	1.339*	(%Ca))	+ 2.	32*(X M	g)/(F1R:	ST 4 1	ERMS)										

TABLE 8.3.7

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. . TABLE 8.3.7a TEST #3 WHAT & RECOVERY WOULD HAVE TO HAVE BEEN USED IN TABLE 8.3.7 IN ORDER TO GET ICP ELEM IN COAL SLURRY SQ3'-1G-1 TO AGREE WITH ELEM CONCENTR. FROM ANAL OF ASH IN SAMPLE SQ3'-1H ?

FOR	ASSUME ICP
	RECOVERY OF
-	
S	85∛
Si	68%
Al	70%
Fe	638
Ca	87%
MG	968
*CALC ASH	71%

-

* USING ONLY THE FIVE ELEMENTS LISTED



TEST #3: ASH RESULTS: ICP VS REF & SC4 SAMPLES



TOUDORY YRO NI HRA %

TEST #3: SULFUR RESULTS: ICP VS REF & SC4 SAMPLES

 $|\cdot| + |\cdot|$



TOUCORS YRO NI S %

TEST #3: SILICON RESULTS:ICP VS REF SAMPLE ICP RECOV ASSUMED TO BE 85% ("CGOSI")



2 ZIN DEA BEODACT





TOUDORY PRODUCT

TEST #3: IRON RESULTS:ICP VS REF SAMPLE ICP RECOV ASSUMED TO BE 85% ("CGOFE")



TOUGOAA YAG NI SA %



% Ca IN DRY PRODUCT



TOUDOAR YAD NI EM 28

TEST #3

_

TABLE 8.3.8

٢ SECONDARY STANDARD RESULTS

	(20)	(mdd)	21.8 20.5	19.3 20.4 1	19.0 21.7	- 484 - 484	1180	690 880 611	19.1 19.4	16.6 17.9	19.6 19.8 9	19.48	20.00	0.52	1.52	2.31
-1 to 12	Ca (50)	(mdd)	51.4	0,446 0,000 0,000 0,000	50.1 51.4	400 8014 1010	001 001	140 140	44 49.44 49.84	49.6 50.1	43 50.3 49.3	49.91	50.00	0.09	3.32	11.05
CGO-SC4	Fe (50)	(mdd)	455.4 455.4	4440 6470 80720	53.4 53.4	444 689 500 500	446 46 46 40 40	40.04	444 888 68 68 78	50.2 47.1	46.34 46.3 3.38	48.22	50.00	1.78	2.93	8.60
ESTING OF	(50)	(mdd)	400 00 00 00 00	649 670.00	440 647 0.00	444 0400 041	404 929 99	804 000	443.04 47.60	46.4 51.4	53.7 47.6 55.9	49.53	50.00	0.47	4.69	21.97
NG TCP TI	(100)	(wdd)	100.5 100.5	105.1 104.7	92.92 100.89	105.0 945.0	103-30 103-30 186-10 186-10 195-10 10 195-10 10 195-10 10 195-10 10 10 10 10 10 10 10 10 10 10 10 10 1	102.2	102.6	104.4	8992 2.50 8.4	99.24	100.00	0.76	6.46	41.70
FIS DUKI	(100)	(udd)	103.5	101.8 104.7	106.2 959.8	101.3	105.1 105.9	107.6 98.3	107.79	93.8 99.2	96.6 93.1 93.1	102.02	100.00	-2.02	6.75	45.56
IKD KESU	TIME AFTER START	(uim)	000	1-14 1-14 1-14 1-14 1-14 1-17	1700 1700 1700	2020 2020 2020	245 265 285 285 285 285 285 285 285 285 285 28	200 200 200		444 0000	2000 2200 2000		• • • •	•	• • • • • • •	• • • •
UNDARI STANDA	SLURRY SAMPLE PRECEDTNG	STANDARD CALTBRATE		amkdai tech	TRADIDARIE	112	00œ	ר ס נ	4 3 DEAL TDDAME	льсантрима в 2 1 Recal.t Reate	FGU-S03 ' -1C-1 CGO-S03 ' -1G-1	MEAN	TRUE	BIAS	STD DEV	VARIANCE
つむく	ЧE	:30		0500	0000		000	-104 005		5000	2000 2000					
	TII	9		~~~œo	10/0/0	000,		777		~~~~~	600					
	DATE	12/02/90			* * * *			: : : :								



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8.4 Test # 4 (Unground Oklahoma Froth Feed)

Test # 4 was conducted on the froth flotation feed stream of an Oklahoma coal. In addition to the two samples which were needed for comparison (the reference sample and the "well water" diluted sample at the exit of the second stage, SQ3-1), CQ Inc. collected their own reference sample (only %-solids and ash analysis were performed) and B&W collected a sample by drawing off slurry through a peristaltic pump from the top mixing tank (see figure 6.4). The dilution ratio of well water to slurry entering the multistage sampler was approximately 19:1. The well water contained a large amount of dissolved sulfur but was relatively low in all other elements (see table 8.3.5).

The most important parameters for test # 4 slurries are described in table 8.4.1 and the laboratory results are presented in table 8.4.2 and figures 8.4.1 - 8.4.3. All samples have the same particle size distribution but, as noted in table 8.4.1, samples SQ3-1 and ST1-1 were filtered through 2.5 micron filters while the reference samples were filtered through five 25 micron filters. The slurries had a particle size distribution in which 25 % of the particles were smaller than 25 microns and 3 % of the particles smaller than 2.5 microns.

The \$-solids in sample SQ3-1 are in very good agreement with both reference samples, while the \$-solids in sample ST1-1 are approximately 20 \$ lower than the \$-solids in the reference sample. Both the \$-solids and the \$ ash in the B&W ref. sample and the CQ ref. sample are in excellent agreement with one another.

Comparing the concentrations of elements and of ash in sample SQ3-1 with the concentrations in the reference sample (see table 8.4.2 and figure 8.4.1) reveals that the sulfur concentration in SQ3-1 is approximately 20 % lower than the reference sample. Of the remaining major elements in this coal (i.e. Si, Al, Fe, & Ca), Si and Al are high by 9 and 24 %, respectively and Fe and Ca are low by 57 & 72 %, respectively. One possible explanation of this is that the Si and Al are not soluble in the well water while the Ca is (recall that the dilution ratio of well water to slurry in the field was 19:1 and very little Ca is present in the well water).

Comparing concentrations of elements and ash in sample ST1-1 with the concentrations in the reference sample (table 8.4.2 and figure 8.4.2) indicates a sulfur concentration approximately 30 % below that of the reference sample. Of the remaining major elements (Si, Al, Fe, & Ca), all are in concentrations less than that of the reference sample of between 5 and 40 %. When compared to sample SQ3-1, the concentration of major elements in sample ST1-1 are lower in Si and Al but higher in Fe and Ca (see figure 8.4.3). The method which was used to obtain sample ST1-1 (suction through a submersed tube in a mixing tank) is not recommended by reference 2 or by reference 15. Although the method used for collecting sample ST1-1 may not be theoretically representative, the results appear to be in better agreement with the reference sample, for certain elements, than sample SQ3-1.

As in the case of test # 1, the discrepancies between sample SQ3-1 and the reference sample may be due to one or more of the following:

- a Use of different filters sizes during sample preparation (see section 7.4).
- b Leeching of the elements Ca and Mg out of the coal into the highly diluted sample SQ3-1.

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- c Insufficient agitation of the mixing tanks for the unground coal (this could result in a longer delay time for the tanks to reach steady state).
- d Error introduced by the commercially obtained vezin samplers.
- e Nonscientific sampling from the top mixing tank (for sample ST1-1 only).

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Table 8.4.1

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TEST # 4 DATA

	TESTS TO BE PERFORMED		_		 SOLIDS, ASH	SOLIDS, S, ASH, ELEM'S	SOLIDS, S, ASH, ELEN'S	SOLIDS, S, ASH, ELEM'S	PSD
JF SAMPLE	FILTER DILUT.	RETENTION RATIC	(MICROWS) DEION	H20	 25 NONE	25 NONE	2.5 NONE	2.5 NONE	NONE
ARATION C	FILTER	TYPE	TO GET	ID SOLIDS	5x541	5×541	# 42	# 42	NONE
ION & PREF	ANAL OF	FILTRATE	FOR	BACKGROUN	ON	ON	NO	NO	NA
11SO4S10	ASSAY OF	TOTAL SLURRY	ð	SUSP SOLIDS ONLY	 SUSP SOLIDS ONLY	SUSP SOLIDS ONLY	SUSP SOLIDS ONLY	SUSP SOLIDS ONLY	
	SENT TO				 H.C. LAB	H.C. LAB	H.C. LAB	H.C. LAB	B&U
APPROX.	.DEL. WEIGHT	TIME	N(MIN)	(B)		205 80,000	205 8,100	205 3,400	2,000
	TIME	END	HR :MI		 12 :55	12 :55	13 : 5	13 : 5	••
		BEGIN	HR :MI		 9:30	9:30	07:6 (07:6 (
		DATE			8-07-90	8-07-90	8-07-90	8-07-90	8-07-90
		SAMPLE			 CO-REF	B&W REF	So3 - 1	ST1 - 1	So31- 1
		TEST ID			FEED	NO GRINDER	OKLAHOMA		Ð

B COLLECTED UNSCIENTIFICALLY FOR PSD ANALYSIS PURPOSES ONLY

===== ST1-1 VS. REF. ===== \$03-1 VS. REF. COMPARE COMPARE 0.15 0.67 0.17 0.03 0.03 0.04 -3.35 -3.91 -0.15 0.79 0.69 -2.69 -2.25 0.04 0.15 0.16 0.15 -0.02 1 1x -9x -10x -21x 9x 24x -57x -72x 19x 100x 24x 88x -67x 8.04 37.09 38.44 0.70 9.04 2.83 4.71 3.12 0.21 0.15 0.67 0.17 0.03 0.33 33.07 31.20 0.51 7.76 2.68 3.50 1.91 0.20 0.15 0.59 1.68 0.01 0.35 33.07 31.20 0.51 7.76 2.68 3.50 1.91 0.20 0.15 0.59 1.68 0.01 0% -12% 888% -67% -1.69 -4.02 -7.24 -0.19 -1.28 -0.15 -1.21 -1.21 -0.01 0.00 -0.08 1.51 -0.02 0.83 0.32 0.01 0.32 0.01 **X** P 0.67 0.17 XS XSi XAI XFe XCa XMg XNa XK XTi 0.30 0.83 SOLIDS REFERRED BACK TO PRE-WELL WATER DILUTION [i.e. % SOLIDS*(QG/QP2 +1)] 0.15 0.30 -21% -11% -19% -27% -14% -5% -26% -39% -5%
 B.04
 37.09
 38.44
 0.70
 9.04
 2.83
 4.71
 3.12
 0.21

 0.42
 33.74
 34.53
 0.55
 9.83
 3.52
 2.02
 0.87
 0.25

 8.09
 33.74
 34.53
 0.55
 9.83
 3.52
 2.02
 0.87
 0.25

 8.09
 33.74
 34.53
 0.55
 9.83
 3.52
 2.02
 0.87
 0.25
 OVERVIEW OF LAB RESULTS, % IN DRY PRODUCT..... 0.40 l/min 7.30 l/min op2 = meas. Well water flow into sampler = QG = SLURRY FLOW INTO MULTISTAGE SAMPLER = X SUSPENDED SOLIDS BY FILTERING SLURRY X SUSPENDED SOLIDS BY FILTERING SLURRY X SOLIDS X ASH X ASH 38.65 CALC MEAS. X SOLIDS AS COLLECTED IN FIELD 8.00 COLLECTED IN FIELD SAMPLE <u>+ 0</u> 6 ¥e ST1 - 1 e ST1 - 1 e So3 - 1 e B&U REF e B&N REF e So3 - 1 X DIFF DELTA X DIFF DELTA e CO-REF SAMPLE х đ, NO GRIND OKLAHOMA TEST ID FEED

Table 8.4.2 rest # 4 results X ASH CACLULATED=[2.139*(X Si) + 1.889*(X Al) + 1.43*(X Fe) + 1.399*(X Ca) + 1.348*(X Na) + 1.205*(X K) + 1.668*(X Ti)]+ 2.32*(X Mg)/[FIRST 7 TERMS]





SOF QUANTITY IN DRY SUSP SOLIDS



2 OF QUANTITY IN DRY SUSP SOLIDS



SOLIOS ASUS YAO NI YTITNAUQ AO %

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8.5 Opacity/Solids Testing on Field Collected Slurries

The opacity meter was used on-line in the field for tests 2 & 3. As mentioned earlier, however, the results were usable only for test # 3 (8 of 12 measurements). It was therefore desirable to reanalyze the field collected slurries, SC4, for %-solids by opacity at the B&W research facility subsequent to the field tests.

The equipment setup used for the laboratory testing was identical to the field setup with the exception that level regulation equipment (i.e. bottom sampler cup and pressure sensor) was not used. The lab setup is depicted in figure 7.5.

8.5.1 Calibration of Opacity Meter

As a first step, the opacity meter was recalibrated using riffles of the "calibration" slurries, SQ3', collected during field tests 2 & 3. Although these slurries were not perfectly representative, they were considered preferable to laboratory prepared slurries. The PSD of the two slurries are presented in figures 8.5.2 & 8.5.4. Note that the Upper Freeport Coal was ground to a mean particle size of approximately 6 microns while the Oklahoma coal (much more difficult to grind) was ground to a mean particle size of approximately 8 microns.

To avoid problems with metering pump nonlinearities, six dilutions of the calibration slurry from each of the two field tests (test # 2 & test # 3) were prepared as described in section 7:

Test # 2:

FGU-SQ3'-1C-1, 0.05 % tot solids, 0.038% susp solids FGU-SQ3'-1C-2, 0.08 % tot solids, 0.060% susp solids FGU-SQ3'-1C-3, 0.11 % tot solids, 0.083% susp solids FGU-SQ3'-1C-4, 0.14 % tot solids, 0.105% susp solids FGU-SQ3'-1C-5, 0.17 % tot solids, 0.128% susp solids FGU-SQ3'-1C-6, 0.20 % tot solids, 0.150% susp solids

Test # 3:

CGO-SQ3'-1G-1, 0.289% tot solids, 0.276% susp solids CGO-SQ3'-1G-2, 0.595% tot solids, 0.567% susp solids CGO-SQ3'-1G-3, 0.733% tot solids, 0.699% susp solids CGO-SQ3'-1G-4, 0.935% tot solids, 0.891% susp solids CGO-SQ3'-1G-5, 1.101% tot solids, 1.050% susp solids CGO-SQ3'-1G-6, 1.70 % tot solids, 1.621% susp solids

The metering pump, P3, was operated at 45 ml/min and the peristaltic pump, P4, was operated at 5 ml/min for all calibration "runs". Table 8.5.1 presents the data from the calibration "runs" on test # 2. The top set of data uses the total solids in the slurry (dissolved and suspended) as a basis of the calibration, while the bottom set of data uses the suspended solids only as a basis. The two sets of data are plotted in figure 8.5.1 along with their respective linear regressions. Table 8.5.2 and figure 8.5.3 present the same information for test number 3. Note that the difference between the calibration curves for total and suspended solids for test # 2 is much more dramatic than for test # 3. Table 8.5.3 combines the data from tests 2 and 3 and figure 8.5.5 presents the plots and linear regression for both.

The total solids in the calibration slurry was determined in the B&W lab by drying sample SQ3'-1D, in its entirety, and the suspended solids was determined by filtering sample SQ3'-1H, in its entirety, through a 0.45 micron

filter before drying. The filtrate from sample SQ3'-1H was clear and perfectly transparent to the naked eye.

8.5.2 Opacity Measurements on Test # 2 Slurries, SC4-1 thru 8

After calibration was complete, each of the individual field collected slurries, SC4-1 thru SC4-8, were "run" through the opacity meter under the same conditions used in the calibration runs (i.e. QP3 = 45 ml/min and QP4 = 5 ml/min). The opacity reading was converted to a \$-solids value by using one of the linear regression curves of figure 8.5.1 or 8.5.3. Computations were conducted for the total solids calibration and for the suspended solids calibration and the data is presented in tables 8.5.4 & 8.5.5 and the results are plotted in figures 8.5.6 - 8.5.9.

Figure 8.5.6 compares the results of opacity measurements using total solids calibration to the % suspended solids measured in samples SC4'-1 through SC4'-8 at the Homer City lab for field test # 2, while figure 8.5.7 presents the same comparison using the suspended solids calibration. These plotted results demonstrate excellent agreement between opacity measurements, using the suspended solids calibration curve, and % suspended solids measured by the Homer City Lab. This excellent agreement exists despite the fact that the Homer City lab measured the % suspended solids by filtering the slurry sample through 2.5 micron filter paper (from the PSD run at B&W, slurry SQ3'-1 has 30 % of its particles less than 2.5 microns). Discussions with Homer City Coal lab indicates that the filter paper becomes clogged quickly with particles and does not pass all particle smaller than 2.5 microns.

Figure 8.5.8 and 8.5.9 present the comparison of opacity measurements to Homer City Lab results for test # 3. Since there is little difference between total and suspended solids for this slurry, figures 8.5.8 and 8.5.9 appear to be very similar. On these curves is also plotted the instantaneous opacity measurements made during the field test (only 8 of 12 measurements were successfully made). The comparison is good, but not as good as was observed for test # 2. This may be due to the fact that test #3 slurries were not ground as finely as those in test #2.

FILE "UFPCAL.WK1"

BASED ON % SUSP. & DISOLVED SOLIDS CALIBRATION OF UPPER FREEPORT COAL 9/9/90

FGU-SQ3'-1C (ORIGINAL FIELD SAMPLE AT .72 % TOTAL SOLIDS)

						LINEAR	Regression C	Output:
	SAMPLE			TESTED		REGR	Constant	-0.00066
SAMPLE	X SOLIDS	29Q	0P3	X SOLIDS	OPACI 1Y	X SOLIDS	Std Err of Y Est	0.000379
2252222							R Squared	0.996340
8	0.050	5.00	45.00	0.005	0.0374	0.005	No. of Observations	9
FGU-S031-1C-2	0.080	5.00	45.00	0.008	0.058	0.008	Degrees of Freedom	4
FGU-SQ31-1C-3	0.110	5.00	45.00	0.011	0.0774	0.011		
FGU-SQ31-1C-4	0710	5.00	45.00	0.014	0.0922	0.013	X Coefficient(s) 0.1	152857
FGU-SQ31-1C-5	0.170	5.00	45.00	0.017	0.1142	0.017	Std Err of Coef. 0.(004631
FGU-SQ31-1C-6	0.200	5.00	45.00	0.020	0.1376	0.020		

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BASED ON X SUSP. SOLIDS ONLY CALIBRATION OF UPPER FREEPORT COAL BASED ON X SUSP. SOU 9/9/90 FGU-SQ3'-1C (ORIGINAL FIELD SAMPLE AT .54 X SUSPENDED SOLIDS)

	ssion Output:	-0-00049	st 0.000284	0.996340	tions 6	edom 4		s) 0.114643	f. 0.003473
	Regree	Constant	Std Err of Y E	R Squared	No. of Observa	Degrees of Free		X Coefficient(:	Std Err of Coe
LINEAR	REGR	X SOLIDS		0.004	0.006	0.008	0.010	0.013	0.015
		OPACITY		0.0374	0.058	0.0774	0.0922	0.1142	0.1376
	TESTED	X SOLIDS		0.004	0.006	0.008	0.011	0.013	0.015
	-	CP3		45.00	45.00	45.00	45.00	45.00	45.00
		940		5.00	5.00	5.00	5.00	5.00	5.00
	SAMPLE	X SOLIDS		0.038	0.060	0.083	0.105	0.128	0.150
		SAMPLE		FGU-S031-1C-1	FGU-SQ31-1C-2	FGU-SQ3+-1C-3	FGU-S031-1C-4	FGU-SQ31-1C-5	FGI1-5031-1C-6

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SONDS %



Figure 8.5.2

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Table 8.5.2

FILE "OKCAL.WK1"

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BASED ON X SUSP. & DISOLVED SOLIDS CALIBRATION OF OKLAHOMA COAL 9/9/90

CGO-SQ31-1G (ORIGINAL FIELD SAMPLE AT 4.28 % TOTAL SOLIDS)

						LINEAR	Regression Outpu	
	SAMPLE			TESTED		REGR	Constant	-0.00368
SAMPLE	X SOLIDS	0P4	0P3	X SOLIDS (OPACITY	X SOLIDS	Std Err of Y Est	0.000992
			******				R Squared	0.999047
CG0-S03'-16-1	0.289	5.00	80.00	0.017	0.109	0.016	No. of Observations	9
CGO-S031-1G-2	0.595	5.00	79.67	0.035	0.213	0.035	Degrees of Freedom	4
CG0-S031-16-3	0.733	5.00	79.33	0.043	0.264	0.044		
CGO-S031-16-4	0.935	5.00	79.67	0.055	0.331	0.056	X Coefficient(s) 0.18083	7
CG0-S031-16-5	1.101	5.00	79.67	0.065	0.384	0.066	Std Err of Coef. 0.00279	-
CG0-S031-1G-6	1.700	5.00	79.33	0.101	0.572	0.100		

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9/9/90 CGO-SQ3+-1G (ORIGINAL FIELD SAMPLE AT 4.08 % SUSPENDED SOLIDS)

						LINEAR		
	SAMPLE		-	TESTED		REGR	Regression Outp	ut:
SAMPLE	X SOLIDS	740	OP3	X SOLIDS	OPACITY	X SOLIDS	Constant	-0.00350
			63 61 61 61 61 61				STU ELL OL 1 EST	
CGO-SQ3 - 1G-1	0.275	5.00	80.00	0.016	0.109	0.015	R Squared	0.999047
CGO-SQ31-16-2	0.567	5.00	79.67	0.033	0.213	0.033	No. of Observations	ę
CG0-S03!-1G-3	0.699	5.00	79.33	0.041	0.264	0.042	Degrees of Freedom	4
CGO-SQ3+-16-4	0.891	5.00	79.67	0.053	0.331	0.054		
CG0-SQ3'-1G-5	1.050	5.00	79.67	0.062	0.384	0.063	X Coefficient(s) 0.1723	87
CGO-SQ3+-16-6	1.621	5.00	79.33	0.096	0.572	0.095	Std Err of Coef. 0.0026	61



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Table 8.5.3

ILE "BOTHCAL.WK1" CALIBRATION OF UPPER FREEPORT COAL 9/9/90 FGU-SQ3'-1C (ORIGINAL FIELD SAMPLE AT .72 % SOLIDS) BASED ON % SUSP. & DISOLVED SOLIDS

						LINEAR	Regression Outpu	t:
	SAMPLE			TESTED		REGR	Constant	-0.00296
SAMPLE	X SOLIDS	QP4	QP3	% SOLIDS	OPACITY	% SOLIDS	Std Err of Y Est	0.000981
22222322	=========		*******	*******		*****	R Squared	0.998949
FGU-SQ3'-1C-1	0.050	5.00	45.00	0.005	0.0374	0.004	No. of Observations	12
FGU-SQ3'-1C-2	0.080	5.00	45.00	0.008	0.058	0.007	Degrees of Freedom	10
FGU-SQ3'-1C-3	0.110	5.00	45.00	0.011	0.0774	0.011		
FGU-SQ3'-1C-4	0.140	5.00	45.00	0.014	0.0922	0.014	X Coefficient(s) 0.17874	?
FGU-SQ3'-1C-5	0.170	5.00	45.00	0.017	0.1142	0.017	Std Err of Coef. 0.001833	3
FGU-SQ31-1C-6	0.200	5.00	45.00	0.020	0.1376	0.022		
CGO-SQ3'-1G-1	0.289	5.00	80.00	0.017	0.109	0.017		
CGO-SQ31-1G-2	0.595	5.00	79.67	0.035	0.213	0.035		
CGO-SQ3'-1G-3	0.733	5.00	79.33	0.043	0.264	0.044		
CGO-SQ31-1G-4	0.935	5.00	79.67	0.055	0.331	0.056		
CG0-SQ31-1G-5	1.101	5.00	79.67	0.065	0.384	0.066		
CGD-SQ31-1G-6	1.700	5.00	79.33	0.101	0.572	0.099		

CALIBRATION OF UPPER FREEPORT COAL 9/9/90 FGU-SG3'-1C (ORIGINAL FIELD SAMPLE AT .54 % SOLIDS) BASED ON % SUSP. SOLIDS ONLY

	SAMPLE			TESTED		LINEAR REGR	Regression Out	out:
SAMPLE	% SOLIDS	QP4	QP3	% SOLIDS	OPACITY	% SOLIDS	Constant	
*******	===========		=======	22282222	*******	=======	Std Err of Y Est	(
FGU- SQ3 - 1C - 1	0.038	5.00	45.00	0.004	0.0374	0.001	R Squared	(
FGU-5931-10-2	0.060	5.00	45.00	0.006	0.058	0.005	No. of Observations	
FGU-SQ3'-1C-3	0.083	5.00	45.00	0.008	0.0774	0.008	Degrees of Freedom	
FGU- 5931-10-4	0.105	5.00	45.00	0.011	0.0922	0.011	-	
FGU-SQ31-1C-5	0.128	5.00	45.00	0.013	0.1142	0.015	X Coefficient(s) 0.175	683
FGU- 593 - 10-6	0.150	5.00	45.00	0.015	0.1376	0.019	Std Err of Coef. 0.003	483
CGO-SQ3'-1G-1	0.275	5.00	80.00	0.016	0.109	0.014		
CGO-SQ31-1G-2	0.567	5.00	79.67	0.033	0.213	0.032		
CGO-SQ31-1G-3	0.699	5.00	79.33	0.041	0.264	0.041		
CGO-SQ31-1G-4	0.891	5.00	79.67	0.053	0.331	0.053		
CGO-SQ31-1G-5	1.050	5.00	79.67	0.062	0.384	0.062		
CGO-SQ3+-1G-6	1.621	5.00	79.33	0.096	0.572	0.095		

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-0.00514 0.001864 0.996083 12

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Table P - 1

FILE "UFSOL"

RESULTS FOR OPACITY % SOLIDS TESTS FOR UPPER FREEPORT COAL

06-71-6

HC LAB 0.51 0.58 0.58 0.58 0.58 0.58 0.58 0.58 0.54 0.54 0.58 0.58 BY DRYING ¥ ARC SOLIDS....BY OPAC SUSPEN AT SITE 0.478 0.506 0.561 0.561 0.519 0.544 0.544 0.530 0.530 0.502 0.478 0.523 0.523 0.705 0.696 0.696 0.668 0.668 0.636 0.636 0.673 0.673 0.746 0.746 0.723 0.691 TOTAL 0.723 62.1 77 77.1 92.1 107 32 32.1 50.1 22 107.1 15.1 50 **%** 0 15 1130 1130.1 1148 1148.1 1160 1160.1 1175 1175.1 1190 1190.1 1205 1205.1 1098 1113 1113.1 TIME (MIN) 11113 1130 1133 1148 1148 1148 1148 1160 1175 1175 1175 1190 1205 1205 1220 1098 Opacity 0.070 0.077 0.0 70.0 70.0 70.0 0.082 0.076 Bkgrnd -0.04 -0.04 -0.043 -0.042 -0.042 -0.042 -0.043 -0.041 ARC SOLIDS.... 5.00 0.723231 0.543601 5.00 0.704767 0.529753 5.00 0.635946 0.478130 5.00 0.746003 0.560673 5.00 0.690974 0.519401 5.00 0.695535 0.522829 5.00 0.667839 0.502057 5.00 0.672631 0.505644 Y-INTERC=-0.00066 -0.00049 1/XCOEFF=6.542062 8.722730 SUSPEN TOTAL **740** 45.33 45.00 45.00 45.00 45.33 45.33 45.33 0P3 FGU-SC4- 1 FGU-SC4- 2 FGU-SC4- 3 10-SC4- 4 FGU-SC4- 5 FGU-SC4- 6 FGU-SC4- 7 FGU-SC4- 8

0.519

0.691

122

1220



S01705 %

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SONOS %

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Table 8.5.5

FILE "OKSOL"

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RESULTS FOR OPACITY % SOLIDS TESTS FOR OKLAHOMA COAL 9-14-90 1/xcoeff=5.529841 5.800901 Y-INTERC=-0.00368 -0.0035

			ARC	SOLIDS			-	11ME		ARC S	OL IDS	BY OPAC	BY DRYI	UN C
	0P3	0D4	TOTAL	SUSPEN	Bkgrnd	Opac i ty	•	(MEN)		TOTAL	SUSPEN	AT SITE	AT HC (LAB
1 - 72C-052	79.67	5.00	4.636067	4.422590	-0.084	0.256	£	Sé.	0	4.636	4.423		4.8	87
CC0-SC4- 2	79.67	5.00	4.544198	4.335014	-0.075	0.251	810	810	15	4.636	4.423	4-144	4.8	87
CG0-SC4- 3	80.67	5.00	4.728003	4.510267	-0.078	0.258	812	810.01	15.01	4.544	4.335		4.6	8
CGO-SC4- 4	80.67	5.00	4.523505	4.315324	-0.07	0.247	825	825	30	4.544	4.335	4.837	4.4	83
CGO-SC4- 5	80.67	5.00	3.928600	3.748218	-0.067	0.215	827	825.01	30.01	4.728	4.510		4.2	5
CCO-SC4- 6	79.33	5.00	3.775652	3.602366	-0.07	0.21	840	840	45	4.728	4.510	4.305	4.2	5
CG0-SC4- 7	79.33	5.00	4.288051	4.090823	-0.068	0.238	842	840.01	45.01	4.524	4.315			4
CGO-SC4- 8	79.33	5.00	4.196551	4.003598	-0.064	0.233	856	856	61	4.524	4.315	3.641		4
CGO-SC4- 9	80.33	5.00	3.894492	3.715691	-0.076	0.214	857	856.01	61.01	3.929	3.748		З.6	5
CG0-SC4-10	80.33	5.00	2.487201	2.374158	-0.071	0.138	871	871	26	3.929	3.748	000-0	м. Ч	42
CGO-SC4-11	80.33	5.00	1.172495	1.120885	-0.074	0.067	872	871.01	76.01	3.776	3.602		м. К	42
cco-sc4-12	80.33	5.00	2.468684	2.356507	-0.075	0.137	885	885	8	3.776	3.602	000.0	м. К	42
							886	885.01	90.01	4.288	4.091		'n	35
							006	006	105	4.288	4.091	0.000	m	33
							901	900.01	105.01	4.197	4.004		л. М	42
							914	914	119	4.197	4.004	0.000	З.4	42
							916	914.01	119.01	3.894	3.716		N	80.
							932	932	137	3.894	3.716	3.279	N	ø.
							934	932.01	137.01	2.487	2.374		2.:	8
							676	576	154	2.487	2.374	1.469	~	8

0.98 0.98 1.96 1.96

1.551

1.121 1.121 2.357 2.357

1.172 1.172 2.469 2.469

154.01 170 170.01 187

949.01 965 965.01 982

950 965 982

2.690



S01105 %



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8.6 Evaluation of the CONAC Algorithm

The "calculated % ash" values presented in the preceding sections have been determined by application of the CONAC algorithm (see appendix C). There are a total of eight elements which enter into the CONAC algorithm (Si, Al, Fe, Ca, Mg, Na, K, and Ti). For the large samples, for which elemental ash analysis was conducted, all eight of these elements were used in the CONAC algorithm. For the coal slurry samples, for which only five elements (in addition to sulfur, which does not enter into the algorithm, Si, Al, Fe, Ca, & Mg were determined), only five elements are used in the algorithm.

With the exception of ignoring contribution of sulfur to the ash content (which is generally small) and modifying the contribution that magnesium makes to the ash content, the CONAC algorithm uses the gravimetric relationships between the element oxides assumed to be in the ash and the elements themselves. During an assay of the ash, the concentration of elements are measured and converted to oxide content by application of the gravimetric relationships for reporting purposes. The sum of the individual oxide contents (in %) are summed and compared to 100 %. The closeness to 100 % is a measure of the validity of the assumptions on which oxides the elements form in the coal. Analysis of gases from the ashing process have been performed on many occasions and, with the exception of sulfur, all elements remain with the ash. Sulfur is converted primarily into gas during the ashing process. Thus, if one can measure the concentration of elements in the coal, the ash can be computed through application of the gravimetric relationships, if the error in ignoring sulfur is acceptable. This is essentially the basis of the CONAC algorithm. If there should be a discrepancy in the application of the CONAC algorithm, it must be due to:

- a ignoring the contribution of sulfur
- b inappropriate assumptions about the oxide forms in the ash (which, for the coals tested in this project, are reasonable as evidenced by the closeness of the ash contributions of each element oxide to 100 %)
- c error in measuring the element concentrations in the coal prior to applying the CONAC algorithm

In order to evaluate the appropriateness of the CONAC algorithm, it is necessary to compare calculated ash values to measured ash values determined from consistent data (i.e. test results from specimens which were taken from the same samples and prepared identically prior to ash measurement and individual element measurements in the ash). The only consistent samples are the large references samples taken during tests 1 - 4, the SQ3-1 samples from tests 1 & 4, the ST1-1 sample from test 4, and the SQ3'-1 sample taken from tests 2 and 3. All of these samples were dewatered and dried prior to separating the residue into ash samples and element analysis samples (which were ashed prior to element analysis). No other samples were of sufficient weight to permit both ash and element analysis of ash to be performed. It would be inappropriate, for the purpose of justifying the use of the CONAC algorithm, to compare ash values computed from ICP analysis of the coal slurry to measured ash values in reference samples because of the variability of the ICP recovery from element to element and the differing sample preparation methods (e.g. filter types, dilution with well or deionized water, etc.). As mentioned in sections 8.2.1 and 8.3.1, the ICP results have been reduced assuming 85 % recovery based on the element sulfur. The recovery for each element in the coal is different, however, and some of the major contributors to the ash content, such as Si, exhibited lower recoveries which results in an underestimation of the ash content. This underestimation is not however due to the inappropriateness of the CONAC algorithm.

The results of the measured ash and the calculated ash from the consistent samples are presented in Table 8.6 and the comparison of calculated ash values to measured ash values are plotted in Figure 8.6.1. The average absolute difference between the measured ash and the "CONAC" calculated ash is 4.3%. Eight of the nine calculated values are less than the measured value. This is as expected since the CONAC Algorithm ignores the contribution of Sulfur and other minor elements. The ninth calculated sample is 6 % larger than the measured value. This is an unexpected result but the difference is not extreme. The maximum absolute difference between measured ash and calculated ash is for test # 3, sample SQ3'-1H. This is also expected since the SQ3'-1H sample (obtained by filtering the slurry) did not provide enough solids to allow analysis of the Na and K, Therefore the calculated ash is in more error than the other samples. The B&W lab method for determining elements in ash involve two methods. The first method involves dissolving a portion of the ash and using the ICP to determine concentrations of elements other than Na and K (the B&W ICP optics is not sensitive at Na and K wavelengths, a limitation not shared by most ICPs in production today). The remaining ash is used to determine the concentrations of Na and K by flame spectroscopy.

From the foregoing discussion it is concluded that the CONAC algorithm is an appropriate method for computing the ash value from individual element concentrations found in the ash. If one inspects the ash values calculated from the elements measured in the coal slurry (by ICP) a much poorer comparison is observed. This is due, to a certain extent, to the fact that only 5 of the 8 elements were determined by ICP analysis of coal slurry. Ignoring of 3 of the 3 elements ignored are contribute only minor amounts to the ash content. Two othe factors played a role in the poor comparison. First, the ash calculation based on ICP analysis of coal slurry ignores any difference in the sample preparation at the Homer City lab and the sample preparation on the coal slurries at B&W. Second, and most important, the assumed recovery of 85% used to reduce the ICP data is appropriate to Sulfur only and, as can be seen from Figures 8.6.2 & 8.6.3, the 85% recovery assumption understates the concentration of Si, Al, and Fe in the ash. Since these three elements (especially Si) contribute heavily to the ash content by CONAC, the poor comparison of %ash by calculation from ICP elements from the coal slurry is due, to a great extent, on poor recovery of elements from the coal slurry by the ICP.

Babcock & VVIICOX Research and Development Division

a McDermott Company

Alliance, Ohio 44601

To M. L. MALITO - STRUCTURAL MECHANICS SECTION, ARC

From

G. R. TAYLOR - CHEMISTRY SECTION (35), ARC

Customer D.O.E. File No. ACG-92-4554-07

Subject COAL WATER SLURRY ASH ANALYSES

July 18, 1991

Dete

This letter to cover one customer and one subject only

Attached are results obtained on two samples of coal water slurry that you submitted with your work order dated July 10, 1991. The attached is the elemental constituents in the ash of the samples. Suspended solids, total sulfur and ash concentration were previously reported.

This confirms a verbal report.

R. Trylor

Reviewed and Approved by

8-91

leb

Attachments

cc: M. L. Diehl P. R. Schliffka ARC Library

D.O.E. ACG-92-4554-07 July 17, 1991

SAMPLE NO.

F-4666

DESCRIPTION

Coal Water Slurry #CGO-SQ3'-1H

ASH ANALYSIS (ICP) & *

Silicon	(SiO_2)	48.81
Aluminum	$(Al_2\tilde{O}_2)$	20.89
Iron	(Fe_2O_3)	15.53
Titanium	(TiÕ ₂)	1.27
Calcium	(CaO)	2.70
Magnesium	(MgO)	1.66
Sodium**	(Na20)	0.73
Potassium**	(K ₂ Õ)	3.73
Sulfur	(SÕ ₃)	2.47
Phosphorus	(P ₂ Ŏ ₅)	0.41

- * The results of spectrographic analysis are reported by the Research Center as the oxides. This does not necessarily mean that the elements are present as such in the sample.
- ** Flame Emission

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D.O.E. ACG-92-4554-07 July 17, 1991

SAMPLE NO.

F-4667

DESCRIPTION

Coal Water Slurry #FGU-SQ3'-1H

ASH ANALYSIS (ICP) & *

Silicon	(SiO ₂)	41.64
Aluminum	$(Al_2\tilde{O}_2)$	19.51
Iron	(Fe_2O_3)	30.60
Titanium	(TiÕ ₂)	0.93
Calcium	(CaO)	0.27
Magnesium	(MgO)	0.88
Sodium**	(Na_2O)	Insufficient Sample
Potassium**	(K ₂ Õ)	Insufficient Sample
Sulfur	(SÕ ₃)	0.09
Phosphorus	(P ₂ Ŏ ₅)	0.49

* The results of spectrographic analysis are reported by the Research Center as the oxides. This does not necessarily mean that the elements are present as such in the sample.

****** Flame Emission

TABLE 8.6

-÷ COMPARISON OF CALCULATED % ASH (USING CONAC FORMULATION) TO MEASURED % ASH FOR FIELD SAMPLES ON WHICH BOTH ASH AND ELEMENTAL ANALYSIS OF THE ASH WERE CONDUCTED

TEST #	PLOT ID	SAMPLE DESCRIPTION	SAMPLE ID	% ASH CALC.	% ASH MEAS.*	ABSLO DI	FFER \$	REFER TO TABLE #	LAB DOING ASH ANAL
1	la	FEED/NO GRIND/UP-FREEPORT	REF	17.23	18.04	0.81	4.5%	8.1.2	H.C. LAB
Ч	1b	FEED/NO GRIND/UP-FREEPORT	SQ3-1	15.71	16.14	0.43	2.7\$	8.1.2	H.C. LAB
5	2a	FEED/GRINDER/ UP-FREEPORT	REF	18.15	18.92	0.77	4.1%	8.2.2	H.C. LAB
2	2b	FEED/GRINDER/ UP-FREEPORT	H1-, £Q3	e 17.49	18.84	1.35	0 7.2%	8.2.2	B&W LAB
ĉ	3a	CONC/GRINDER/ OKLAHOMA	REF	8.91	9.16	0.25	2.7\$	8.3.2	H.C. LAB
m	3b	CONC/GRINDER/ OKLAHOMA	H1-, £Q3	8.15	8.65	0.50	5.8%	8.3.2	B&W LAB
4	4a	FEED/NO GRIND/OKLAHOMA	REF	37.09	38.44	1.35	3.5%	8.4.2	H.C. LAB
4	4b	FEED/NO GRIND/OKLAHOMA	SQ3-1	33.74	34.53	0.79	2.3%	8.4.2	H.C. LAB
4	4 C	FEED/NO GRIND/OKLAHOMA	ST1-1	33.07	31.20	1.87	6.0\$	8.4.2	H.C. LAB
		AVERAGE OF ABSOLUTE DIFFER	ENCES			06.0	4.3\$	8 9 8 8 8 8)) 0 0 0 0 0 0

* USING CONAC EQUATION @ Na AND K NOT INCLUDED IN CALC ASH (DUE TO INSUFFICIENT SAMPLE MASS Na & K WERE NOT DETERMINED DURING ELEM ANAL OF ASH)

COMPARISON OF CALC. VS. MEAS. % ASH





NI N DRY PRODUCT



Х ЕГЕМ И ДКА БКОДЛСТ

8.7 Indication of Potential Precision

Although only limited data is available for determining the potential precision of a sulfur and ash analyzer using an ICP with the ARL MDSN nebulizer, an attempt is made in this section of the report to display a comparison of the ash and sulfur measurements made by ICP analysis of coal slurry (SC4 samples) to those made on the small "flow through" samples (SC4' samples) collected simultaneously and sent to the Homer City Coal Lab for assay (% ash and sulfur by conventional techniques).

In order to make these comparisons, the calculated ash values from ICP analysis were corrected to account for the low recovery of elements as indicated by the only samples which were analyzed both by ICP assay of coal slurry and by analysis of elements in ash and for which Leco sulfur analysis was conducted:

ANAL BY	TEST 2 SAMPLE	TEST 3 SAMPLE
ICP OF SLURRY	FGU-SQ3'-1C-1	CGO-SQ3'-1G-1
ELEM ANAL OF ASH	FGU-SQ3'-1H	сбо-503,-1н
LECO SULFUR ANAL	FGU-SQ3'-1H	CG0-SQ3'-1H

Due to the variable recovery of each element, the assumed recovery to make ash values agree are different than the assumed recovery to make sulfur content agree. From tables 8.2.7b and 8.3.7a the following recoveries are required:

_	FOR	TEST	2	SAMPLE	TEST 3	SAMPLE
융	ASH	8:	1	£	71	8
	S	90	0	£	85	S

Additionally, for the calculation of ash only, the ash calculated from the five ICP elements must be increased to account for the difference between % ash measured by combustion of filtered slurry solids and that calculated by the CONAC algorithm (using only 5 elements). This correction is smaller than the correction needed to offset the low ICP recovery and is 1.087 (=18.84/17.33) for test 2 and 1.132 (=8.65/7.64) for test 3.

The results of the corrected ICP ash and sulfur results are presented in tables 8.7.1 - 8.7.2 and figures 8.7.1 - 8.7.4 and compared to the corresponding values reported by the Homer City Lab. These results eliminate essentially all sampling effects. They do not, however, eliminate the variable sample preparations used (SQ3'-IH samples were filtered through 0.4 micron filter prior to ashing and Leco sulfur determination vs. 2.5 micron filter used at Homer City Coal Lab) and the B&W samples were diluted to various degrees with deionized water (resulting in leaching of certain of the elements, especially sulfur, into solution).

It should also be noted that, although a factor for correcting the CONAC algorithm to account for less than eight elements could be predicted for a particular coal in advance, the correction for low ICP recovery is difficult to predict beforehand. Thus the correction made to the ICP data for low ICP recovery was only possible through hindsight.

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Table 8.7.1

RECOVERY= NULTIPLIER= SPECIAL MANIPULATION FOR PURPOSE OF SHOUING "PRESICION" OF ASH FROM ICP VS MEAS ASH recovery= [0.81] multiplier= 1.234

RESULTS OF ICP TESTING ON TEST # 2 ("FEED-GRINDER-UPPER FREEP.") FIELD SAMPLES

	HC-LAB				LAB F	IN DRI Referen	r produ	CT		RESULTS U	X IN I SING PEI	RY PRO R SAMPL 81%	E BACK	ROUND.			RESULT ASSU	NING 1	IN DRY G PER S 00 X IC	AMPLE	SACKGRO	9	÷ :
SAMPILE SC4 UGHT . (9)	UCHT XASH SAMP UGHT XASH S (g) MEAS X	PLES 5 6	XASH XA HEAS CA		s	Si J	4	.e	5 M 8	CALC ASH CO X CALC	88 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8 8	sck sam x	PLES.	щж.	א ^ט :	Ξ×	CALC ASH	×	si x	AI AI	S L X	۲. By	ت
SC4- 1 45 SC4- 2 80 SC4- 3 54	520 21.08 2. 880 20.71 2. 690 20.46 2.	895	8.92 17. 8.92 17. 8.92 17.	82 55 58 57 58 57 58 57	244 244	222	17 2.4	0.00	5 0.13 5 0.13	17.98 19.5 15.05 16.3 13.93 15.1	56.5.74	3.38 2.88 86 2.88	1.52	2.52	0.24	0.30	13.00 26.65	22.0	2883	N 2 2 2	ဆိုဆိုဆိုန် ဗို.ဗို.ဗို.မိ	83.56 9.9.9 9.9.9	666
SC4- 4 6(SC4- 5 62 SC4- 5 62	810 20.7 2. 810 20.7 2.	2.23	18.92 17. 18.92 17. 8 02 17	2022	222	~~~ ***	1.2 2.1		2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2	16.04 17.4 16.43 17.8 16.27 17.6	6 3.63 2.303 2.303	2.8 2.8 2.8	582	4.67 7.49 7.49	8. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2. 2.	0.19 0.24 0.26		28.2	32.2	9.5. ñ.	2007 2007 2007	- 6 0 - 6 0	-Ha
sc4- 8 68	810 20.31 2. 820 20.22 2.		18.92 17. 18.92 17.	5.5 5.5 805	565	5 7 7 7 7 7 7 7	12 2.4	5.00	50.13	14.52 15.7	2.84	2.48	1.24	3.68	1.12	0.10 0.14	9.92 10.82	0.50	2.38	22 28 28 28	- 0- 0- - 0- 0-	9 9 8 8	N.F.
WEIGHTED AVG	20.62 2.	48 1	8.92 17.	62 2.5	31 4.2	34 2.	17 2.4	1 0.5	5 0.13	15.666 17.0	14 3.550	2.981	1.596	3.534 (.848 (0.227	10.846 (.108 2	.394 1	.282 2.	9- 0- 098	.54 -0.	н.
REF SAMPLE		-	18.92				-0			17.615	2.810	4.340	2.170	2.410 (.550 0	0.130	17.615	.810 4	.340 2	170 2.	610 O.S	50 0.1	ň
X DIFFERENCE							۱ ۲		/	-112	26	x -313	-26%	K ,	54%	75%	-38%	X96 -	-45%	×17-	<u>5</u>	- X66	ĕ
sq3/ -1C-1 585 sq3/ 1H	B&H LAB 780 18.84 0. 18.84 0.	: 69	8.84 17.		65 3.6	B&U LAI	B ELEM:	IN AS	H	12.71	2.41	3.32	1.74	3.57	1.21	0.43	12.17	0.88	2.69	1.41 2	0, 08,	e R	ð
* X ASH COMPL e.g. X ASH = ** CORR CALC	TED USING ONLY : [2.139*(XSi) + : ASH = (CALC AS	THOSE 1.88 54)*(12	E ELEMENT	S LISTI 1.47 33)	5*(% Fi	e) + 1.	.339*()	(ca)] +	2.32*(X Mg)/(FIRST 4	TERMS)												

\$ Note that assuming recovery = 81% results in agreement of ICP calc. ash in sample SQ3'-IC-1 with calc. ash in sample SQ3'-IH (elements by ash anal.), both calculations made using only 5 elements.





Figure 8.7.1

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Test #2 SULFUR RESULTS:ICP VS MEAS SC4 SAMPLES



2 IN DEA FRODUCT 86-8

SPECIAL MANIPULATION FOR PURPOSE OF	SHOWING "PRECISION" OF ASH FROM ICP VS MEAS ASH Recovery= 0.71 Multip! IER= 1.408	RECOVERY= 1 MULTIPLIER= 1
RESULTS OF ICP TESTING ON TEST # 3 ("CONCENTR-GRIMDER-OKLANONA") FIELD SAM	bles	
		X IN DRY PRODUCTX IN DRY PRODUCTX RESULTS USING PER SAMPLE BACKGROUND
SC4SC4' SAMPLES	SC4 SAMPLES	SC4 SAMPLES
SAMPLE UGHT.WGHT.XASH S XASH XASH S Si Al Fe Ca	Mg CALC ASH CORR. S Si Al Fe Ca Mg (
(g) (g) MEAS X MEAS CALC +	х сицся х а а а а а а а а а а а а а а а а а а	: : : : : : : : : : : : : : : : : : :
[
SC4- 1 65 760 7.72 0.66 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 5.69 6.447 0.738 1.464 0.701 0.582 0.254 0.111 0.00 4.35 7.035 0.840 1.500 0.408 0.700 0.341 0.125	2/2 0.02 50.07 50.00 700.00 10
SC4- 2 59 650 8.2 0.67 9.16 8.50 0.71 2.2 0.93 0.91 0.51 5.51 5.51 5.51 5.51 5.51 5.51 5.5	0.09 7.05 7.980 0.856 1.723 0.853 0.836 0.370 0.106	4.59 0.136 1.223 0.605 0.593 -0.01 0.018
Std- 2 05 140 9.45 0.66 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 7.36 8.331 0.989 1.825 0.881 0.858 0.371 0.117	4.80 0.215 1.296 0.626 0.609 -0.02 0.027
α Sc4- 5 59 660 9.59 0.66 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 7.67 8.692 1.067 1.863 0.845 0.959 0.476 0.172	5.01 0.218 1.323 0.600 0.681 0.029 0.061
6 SC4- 6 46 620 9.91 0.71 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 7.62 8.625 1.148 1.832 0.795 0.968 0.544 0.147	1 CU.U UUI.U 1800 U 265 U 269 U 260 U 200
SC4- 7 57 700 9.66 0.67 9.16 8.50 0.71 2.2 0.93 0.91 0.51	151.0 645.0 230.1 250.2 2.172 0.933 1.062 0.495 0.20	5.0/ U.2/1 1.342 U.002 U.104 U.004 5.0 U.04 0.059 5.0 M 50 0.757 0.059
SC4- 8 52 640 9.91 0.65 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 8.96/10.1/11.156 2.236 1.052 1.050 0.090 0.097 0.197 0.187 0.082	6.56 0.150 1.692 0.791 0.893 0.109 0.051
504- 9 61 680 10.96 0.67 9.16 5.50 0.71 2.2 0.99 0.91 0.51 2.2 0.91 0.51	0.09 11.05 12.52 1.425 2.483 1.124 1.662 0.852 0.223	7.18 0.254 1.762 0.798 1.180 0.135 0.079
SC4- 11 60 640 15.01 0.81 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 13.64 15.45 3.204 2.473 1.209 2.590 1.646 0.364	8.37 0.526 1.756 0.858 1.858 0.242 0.072 4 54 0 474 1 544 0 740 1.105 0.057 0.065
SC4- 12 69 750 12.08 0.68 9.16 8.50 0.71 2.2 0.93 0.91 0.51	222.0 621.0 600.1 820.1 102.2 616.1 177.11 81.01 60.0	
WEIGHTED AVG 10.45 0.69 9.16 8.50 0.71 2.2 0.93 0.91 0.51	0.09 8.747 9.908 1.319 2.030 0.941 1.202 0.617 0.176	5.660 0.267 1.441 0.668 0.853 0.053 0.051
REF SAMPLE 9.16	. 8.500 0.710 2.200 0.930 0.910 0.510 0.090	9.160 0.710 2.200 0.930 0.910 0.510 0.090
× Difference	3x 86x -8x 1x 32x 21x 96x	77- X06- X9- X82- X7E- X29- X8E-
sa3'-16-1 800 8.65 0.480.48	7.67 0.894 1.889 0.947 0.836 0.418 0.186	5.04 0.143 1.341 0.672 0.594 0.013 0.078
Sq3'-1H 8.65 0.48 8.65 [7.64] 0.48 1.97 0.96 0.94 0.17 * * Act cramphifen incluid chairy threefs filenewits listed	60°0	
e.g. X ASH = [2.139*(XSi) + 1.889*(X Al) + 1.43*(X Fe) + 1.339*(XCa)] +	2.32*(% Mg)/(FIRST & TERMS)	
** CORR CALC ASH = CALC ASH)*(8.65 / 7.64) \$ Note that assuming recovery = 71% results in agree	ement of ICP calc. ash in sample SQ3'-1G-1) hoth calculations made using only	with
calc. asn in sample og - in verements of von succe 5 elements,		

Table 8.7.2

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CORRECTED ICP ASH VS. MEAS ASH Test #3



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Test #3 SULFUR RESULTS:ICP VS MEAS SC4' SAMPLES



9.0 CONCLUSIONS AND RECOMMENDATIONS

9.1 Conclusions

During this project an on-line sulfur and ash analyzer was designed and the sample preparation system was fabricated and field tested. Each individual component of the analyzer, with the exception of the ICP, performed well. The performance of the entire system, because of the ICP performance, was marginal, at best. The results of conventional assay on reference samples and small "end stage" samples compare favorably. The results of ICP assay of "end stage" samples and conventional assay of reference samples compare favorably for sulfur and certain other elements, only after correcting the ICP results for a recovery of 85 % (a value determined after the tests were completed).

The multistage sampling system performed exceedingly well, especially considering the potential error introduced by the differing sample preparation methods used in the post site laboratory analysis. Although the multistage sampler could be improved further to make it manufacturable at a lower cost and more trouble free, especially in the plumbing (i.e. small tubing, valves, and fittings) after the last sampling stage, its current configuration provided an amazingly representative sample after the parent stream had been cut to as much as 1 part in 940,000. The sampling system can be produced at low cost (< \$ 10,000) and used without a grinder or ICP to collect very small, and yet, representative slurry samples for off-line assay. The sample size reduction afforded by this sampling system can provide a significant time and cost advantage over the current practice of collecting large volume samples for offsite assay.

The in-line Netzsch grinder also performed well. It produced a mean particle size of 6 microns on an Upper Freeport coal and 8 microns on the difficult to grind Oklahoma coal. Both of these mean particle sizes approach the 5 micron mean size which was set 75 a goal in the screening testing (Ref. 1).

The mass opacity meter, developed specifically for this project, performed well on the 6 micron Upper freeport coal, and adequately on the 8 micron Oklahoma coal. The large development effort required for this device was not anticipated at the start of the project and its development was halted before many design improvement ideas (such as measurement of scattered light and the addition of polarized filters at 90°) could be tested and implemented. B&W is confident that the opacity meter can be developed further to make possible accurate measurement of suspended solids in more dense slurries and over a broader range of particle size distributions.

During the study phase of the project, the ARL MDSN nebulizer (attached to an ARL ICP) was demonstrated as being capable of recovering at least 80 % of the element concentrations in a finely ground (approx. 5 microns mean size) and diluted slurry (less than 3 % solids concentration). During the study phase, no attempt was made to distinguish between the elements in solution and the elements in suspended solids. When the analysis of the field collected slurries was conducted, however, the difficulty in repeating this performance when high background levels (i.e. elements in solution) had to be subtracted from the element concentrations measured in the slurry (elements in solution and in suspended solids), became apparent. For example, a slurry which contains 1000 ppm S, 900 ppm of which is in solution form (i.e. background), would require an ICP/nebulizer system with a maximum error of 1 % on slurries and 0 % on solutions (for background measurement) in order to yield a maximum error of 10 % on the concentration in the suspended solids. e.g.:

PPM Elem. in Solids = (% ICP Recov.)*(PPM Elem. in Slurry) - (PPM Elem. in Solution)

 $= (0.99) \pm 1000 - 900$

= 90 ppm

% Error in Solids Measurement = (100 %)*(100 - 90)/(100)

= 10 %

This error band is outside the capabilities of the slurry nebulization hardware which is commercially available. Clearly, more development work is needed in the area of slurry nebulization to ICP before an ICP can be considered appropriate for on-line analysis of coal slurry.

The current project was successful in designing a system, testing each portion of the system, and identifying the features which need improvement to reach original project objective of on-line measurement of sulfur and ash on coal slurry streams. B&W continues to believe that this objective is achievable and has prepared recommendations for future work.

9.2 Recommendations

The performance of the sulfur and ash analyzer was determined to be inadequate for an on-line application in a coal cleaning plant due to the performance of the ICP. The ICP is still considered to have high potential for use in an online system but significant improvements must be made to the delivery of slurry to the plasma and possibly to the optics used to transmit emission spectra to the photodetection equipment. Specific recommendations are discussed below.

The sampling portion of the system performed very well and is suitable for use on coal slurry streams as an independent device where small representative samples, for off-line assay, are needed. The experience gained from the field testing at the CQ Inc. provided information on plumbing modifications which should be incorporated in the prototype. These modifications are also discussed below.

9.1.1 ICP Performance

The recovery of the ICP was not as good on field collected slurry samples as it was on laboratory prepared slurry samples (see ref. 1). The overall average recovery was poorer and the recovery of each element was different (see tables 8.2.7b and 8.3.7a). The recovery of S, Ca, and Mg was greater than 85 % while the recovery of Si, Al, and Fe was less than 75 %. This may be due to the fact that the forms of Si, Al, and Fe found in the coal are harder to grind than the other elements and, as a result, these elements made up the majority of the large particles (those > 19 microns) in the ground slurry. Since the calibration standards for the ICP were in solution form, two possible explanations for the poor recovery are hypothesized:

- 1. The larger particle sizes require more residence time in the plasma to fully dissociate and become excited.
- 2. The ARL MDSN nebulizer did not transport the larger particles to the ICP plasma representatively.

An additional problem encountered with the ICP, when the ARL MDSN nebulizer is used as the delivery device with coal slurries, is the necessity to have a very dilute slurry. Because certain of the elements in the coal are very soluble in water, the background levels (dissolved elements) which must be subtracted from the slurry levels are as much as 90 % of the levels in the slurry. Therefore small errors in the measurement of the slurry element concentrations and the background element concentrations are magnified.

For improving ICP performance on coal slurries, B&W makes the following recommendations:

- 1. B&W believes that an ICP delivery system which is more insensitive to particle size and is capable of delivering the slurry in a nearly dewatered condition to the plasma needs to be developed. This development will require a radically new approach to sample delivery in which the slurry sample passes through an energy absorption stage where it is dewatered and vaporized prior to delivery to the ICP plasma. Such a system is, in B&W's opinion, attainable. The system may, for example, be possible using microwave technology. This effort involves more basic research than was possible under the constraints of the current project, which had as its goal the testing of the entire system.
- 2. An optical collection system for viewing the plasma over a longer length and possibly around the plasma circumference should be developed and tested to determine if larger particles can be dissociated and excited with full recovery of the elements. This effort could be performed in conjunction with recommendation 1 and should involve a manufacturer of ICPs.
- 3. Successful development of the new delivery system should be incorporated into a production analyzer system designed in cooperation with an ICP manufacturer and a sampling system manufacturer (see below). Leeman Labs has expressed interest in the product, if a market exists, and was given two samples of the field collected slurries for experimentation.

As mentioned earlier, the sampling system developed under this project, has no significant deficiencies. The experience gained from the field testing provided sufficient data to make modifications to the prototype equipment for trouble free operation at a test site. The sampling system, used as a stand alone device, would not require any grinding equipment on the process stream, and would not require any computer control. Therefore B&W makes the following recommendations:

- 1. The potential market should be identified for a stand alone sampling system capable of obtaining extremely small, representative samples of coal slurry streams.
- Modifications to the prototype sampling system should be made, including:
 - The tubing on the bottom-most sampling stage should be increased in diameter to eliminate potential clogging on unground slurry.
 - The configuration of the overflow tubes in the mixing tanks should be modified to eliminate the potential for "slug" flow and to prohibit the buildup of foam. The field use of a funnel on the top of the overflow tube in the top mixing tank essentially solved that problem and the change from a constant diameter overflow tube to a tapered overflow tube has high potential to eliminate the unstable flow resulting from capillary action of the small diameter tubes.

- The mixers should be operated on the high range, rather than

the low range which was used during field testing.

- 3. The modified system should be installed and operated over many weeks at a commercial coal cleaning plant and sufficient data obtained to characterize the sampling system statistically.
- 4. A manufacturing design should be prepared with the help of an equipment manufacturer (such as Denver Equipment). The design should concentrate on durability, low maintenance, flexibility of application, and minimum manufacturing cost. The equipment manufacturer will decide to commercialize depending on the outcome of the market study.

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10.0 EQUIPMENT PHOTOGRAPHS

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