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**ENGINEERING DEVELOPMENT OF
SELECTIVE AGGLOMERATION**

TRACE ELEMENT REMOVAL STUDY

SEPTEMBER 1993

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ABBREVIATIONS

AAS	Atomic Absorption Spectrophotometry
AES	Atomic Emission Spectrophotometry
APC	Alabama Power Company
APCL	Alabama Power Company General Test Laboratory
ASTM	American Society for Testing and Materials
DOE	U.S. Department of Energy
EPA	U.S. Environmental Protection Agency
EPRI	Electric Power Research Institute
GFAAS	Graphite Furnace Atomic Absorption Spectrophotometry
HAPs	Hazardous Air Pollutants
HGAAS	Hydride Generation Atomic Absorption Spectrophotometry
ICP	Inductively Coupled Plasma
ISE	Ion Selective Electrode
NIST	National Institute of Standards
NSC	No Significant Change
POC	Proof-of-Concept
ppm	parts per million
QA	Quality Assurance
QC	Quality Control
RCV	Raw Coal Value
RSD	Relative Standard Deviation
SAFCV	Selective Agglomeration Feed Coal Value
SCS	Southern Company Services, Inc.
USGS	U.S. Geological Survey

EXECUTIVE SUMMARY

As coal producers and coal-burning utilities develop acid rain compliance strategies for the remainder of the decade and into the 21st century, they must be aware of the possible effects of air toxics emissions (hazardous air pollutants) on their industries. This is particularly important if power plants become classified as major sources of these pollutants under Title III of the Clean Air Act Amendments of 1990. All 12 of the elements and radionuclides listed in Title III are known to occur in coal, at least in trace concentrations.

Southern Company Services, Inc., (SCS) was contracted in 1989 by the U.S. Department of Energy (DOE) to develop a commercially acceptable selective agglomeration technology to enhance the use of high-sulfur coals (DOE Contract No. DE-AC22-89PC88879) by 1993. The project scope involved development of a bench-scale process and components, as well as the design, testing, and evaluation of a proof-of-concept (POC) facility. To that end, a two-ton-per-hour facility was constructed and tested near Wilsonville, Alabama. Although it was not the primary focus of the test program, SCS also measured the ability of selective agglomeration to remove trace elements from coal.

There are several companion reports to this Trace Element Removal Study Report. A Final Technical Report was issued in June 1993. It provides a performance evaluation of the advanced coal cleaning technology and presents a preliminary engineering design of a 200-ton-per-hour selective agglomeration process. A standalone Executive Summary (part of the Final Report) is also available. In addition, a Site Closeout Report is available which addresses all state and federal regulations in closing the selective agglomeration site.

Under the SCS plan, three different coals representing three different coal seams: Upper Freeport, Pittsburgh No. 8, and Illinois No. 5 were tested. Both the Upper Freeport and the Pittsburgh No. 8 coals were commercially precleaned prior to processing, while the Illinois No. 5 coal was processed raw at the POC

facility. After parametric testing, the POC facility was operated continuously for approximately 80 hours at steady state, optimized conditions, for each test coal.

The samples collected were shipped to the Alabama Power Company General Test Laboratory (APCL) in Vamons, Alabama, where they were analyzed for ash, carbon, hydrogen, nitrogen, sulfur forms, total sulfur, volatile matter, and gross calorific matter. For quality assurance/quality control (QA/QC) purposes, splits of seven samples, including raw coal, two feed coals, two product coals, and two tailing samples, were prepared by APCL and sent to four referee laboratories for analysis. While there were cases of poor data agreement between laboratories, the overall level of consistency indicates that analytical results are accurate within the limits of currently available technology.

The APCL QA/QC data were used to establish a criterion to determine if the difference between two measured trace-element concentrations was significant. This approach was used to determine if changes in trace-element concentrations observed with cleaning were within experimental error limits.

For the Upper Freeport coal, selective agglomeration combined with conventional cleaning significantly reduced all elements except beryllium, chlorine, fluorine, and mercury. For example, arsenic, cobalt, and lead were reduced by more than 75 percent. The concentration of barium, manganese, nickel, and zinc were more than halved. As a result of cleaning, the Pittsburgh No. 8 coal showed a decrease in all trace elements except chlorine and fluorine. Substantial reductions were achieved for arsenic (84 percent), barium (79 percent), cobalt (86 percent), lead (92 percent), and zinc (81 percent). For the Illinois No. 5 coal, selective agglomeration reduced all trace elements except cobalt and chlorine. More than 70 percent of the barium, manganese, and zinc were removed. Although not optimized for trace-element removal, selective agglomeration technology was shown to be effective in

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reducing the trace-element content of the test coals.

The study also looked at the potential leaching of trace elements into the process recycle water. The data indicates that leaching was not a significant concern for the majority of trace elements. However, these results are considered preliminary and the issue of trace-element leaching should be investigated further.

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INTRODUCTION

Introduction

This study is part of a project titled "Engineering Development of Selective Agglomeration" contracted to Southern Company Services, Inc., (SCS) in 1989 by the United States Department of Energy (DOE Contract No. DE-AC22-89PC88879). This study, an addendum to the Final Report prepared by Praxis Engineers, Inc., is an evaluation of the applicability of selective agglomeration as an advanced coal cleaning process to remove trace elements from coal. This study centers around the trace element reductions produced by operating a two-ton-per-hour proof-of-concept (POC) facility constructed in Wilsonville, Alabama. Figure 1-1 shows the POC pilot plant facility. Other organizations involved in this project were:

- Alberta Research Council, which provided process development and cofunding.
- Southern Electric International, which was responsible for POC construction and operations.
- Praxis Engineers, Inc., which provided process engineering.
- The Electric Power Research Institute, which provided cofunding and expertise in coal cleaning.

Project Objectives and Process Performance Targets

The main project objectives were to produce clean coal products at a 6 percent or lower ash while recovering 85 percent or more of the coal's heating value and rejecting 85 percent or more of the pyritic sulfur. While removal of trace elements was not a primary project objective with specific performance targets, it was an objective to measure the removal of selected trace elements during the demonstration tests for each project coal. No efforts were made to optimize trace element removal during parametric testing or demonstration runs.

Air Toxics Concerns

The geological processes that form coal can also concentrate trace elements in the coal. For example, the average concentration of arsenic in bituminous coal (20 ppm) is ten times the average concentration found in all other rocks that make up the earth's crust (2 ppm). Similarly, antimony, cadmium, mercury, and selenium are generally more concentrated in coal than in the earth's crust. Just as the sulfur and ash contents of coals vary from seam to seam and within seams, so do trace element concentrations.

Since the enactment of the 1990 Clean Air Act Amendments, many of the elements found in coals have been classified as hazardous air pollutants (HAPs). Although no new restrictions on trace element emissions were placed on the power generation industry under the new Clean Air Act Amendments, the act mandates studies of utility toxic air emissions. These studies are to be performed by the U.S. Environmental Protection Agency (EPA) which is required by law to recommend new air toxic regulations to protect human health and the environment. Furthermore, the legislation requires three separate studies of mercury emissions, deposition, and health effects. These studies are to be presented to Congress in 1995. New regulations aimed at coal-fired electric utilities may be forthcoming when these federally-mandated studies are complete. Coal producers and electric utilities that use coal are becoming more aware of potential compliance problems concerning not only sulfur and nitrogen oxides emissions, but also air toxics emissions.

Title III of the Clean Air Act Amendments of 1990 has identified 189 HAPs, including 12 elements and their compounds commonly found in coal: antimony, arsenic, beryllium, cadmium, chlorine, chromium, cobalt, lead, manganese, mercury, nickel, and selenium. In addition,

Figure 1-1. POC Facility in Wilsonville, Alabama

radionuclides, which are known to occur naturally in some coals, are also listed as HAPs.

Currently, arsenic and mercury are commonly viewed as posing the greatest risk to human health and the ecosystem when released in utility emissions. Also, the United States National Committee for Geochemistry lists arsenic, boron, cadmium, mercury, molybdenum, lead, and selenium as of primary concern for their potential damage to the environment. From these and other lists shown in Table 1-1, it was determined that the following trace elements would be monitored for removal: arsenic, barium, beryllium, cadmium, chlorine, cobalt, chromium, fluorine, lead, manganese, mercury, nickel, selenium, and zinc. Other elements such as boron, germanium, phosphorous, and silver were not selected (although they are of concern) because of inherent difficulties with analytical procedures and instruments used in determining concentrations.

Air Toxics Control Options Available to Utilities

While a significant research effort aimed at compliance with new regulations is likely to develop, three basic options are available to utilities:

1. Installation of improved particulate collection devices to increase capture of the finest-sized fly ash particles that usually contain relatively high concentrations of volatile trace elements.
2. Installation of wet scrubbers that serve to cool boiler flue gas, thereby promoting condensation of volatile trace elements. Wet scrubbers provide a high fine-particle capture efficiency and are, therefore, likely to be effective in capturing most HAPs (with the possible exception of highly volatile species such as mercury).
3. Use of coal cleaning or coal switching to reduce the concentration of trace elements entering the boiler.

As a HAP control technology, coal cleaning offers the advantages of relatively low cost, improved boiler thermal efficiency, and

reduction of other regulated emissions such as sulfur dioxide.

Testing of Selective Agglomeration for Trace Element Removal

Physical coal cleaning techniques are effective in removing mineral matter from coal and can, therefore, potentially remove some trace elements associated with specific minerals. Moreover, recent studies (Akers, 1992; Devito, 1993; and Fonseca, 1993) have shown that physical coal cleaning is effective in reducing the concentration of many trace elements, especially if they are present in the coal in relatively high concentrations. The degree of reductions achieved is coal-specific, relating in part to the degree of mineral association of the specific trace element and the degree to which the trace element-bearing mineral is removed by the cleaning processes employed.

This study investigates the use of a relatively new coal cleaning technology, selective agglomeration, as another possible coal cleaning process to aid in trace element reduction. Selective agglomeration is an advanced physical fine-coal cleaning process that uses diesel oil and mechanically-applied energy to form microagglomerates of clean coal, and flotation to separate the agglomerates from the mineral matter. It is based on surface chemistry differences between coal and its associated impurities.

During selective agglomeration, a liquid agglomerant (diesel oil) that is immiscible with water is introduced into a coal-water slurry and agitated allowing it to disperse and come in contact with all the particles in the slurry. The coal particles are attracted to the agglomerant while the mineral impurities remain in the water. Continued agitation of the agglomerant-coated coal particles causes them to coalesce and aggregate. Once the agglomerates are formed, they are separated from the mineral matter by subsequent processing steps.

Part of the test plan included measurement of the effectiveness of this process in removing trace element contaminants during the demonstration runs. The trace elements studied were analyzed in raw coals, conventionally cleaned coals, agglomerated coal products, and

Introduction

Table 1-1
Trace Element Matrix

Trace Elements Detected In U.S. Coals ¹	Hazardous Air Pollutants ²	Environmental Concern ³	Conventional Coal Cleaning Studies ⁴	Common Elements Selected For POC Operation Study ⁵
Antimony	Antimony			
Arsenic	Arsenic	Arsenic (Primary)	Arsenic	Arsenic
Barium		Barium (Minor)	Barium	Barium
Beryllium	Beryllium			Beryllium
Boron		Boron (Primary)		
Cadmium	Cadmium	Cadmium (Primary)	Cadmium	Cadmium
Chlorine	Chlorine			Chlorine
Chromium	Chromium	Chromium (Moderate)	Chromium	Chromium
Cobalt	Cobalt	Cobalt (Minor)		Cobalt
Copper		Copper (Moderate)		
Fluorine	Fluorine (HF)		Fluorine	Fluorine
Gallium				
Germanium		Germanium (Minor)		
Lanthanum				
Lead	Lead	Lead (Primary)	Lead	Lead
Lithium				
Manganese	Manganese	Manganese (Minor)		Manganese
Mercury	Mercury	Mercury (Primary)	Mercury	Mercury
Molybdenum		Molybdenum (Primary)		
Nickel	Nickel	Nickel (Moderate)	Nickel	Nickel
Phosphorus	Phosphorus			
Scandium				
Selenium	Selenium	Selenium (Primary)	Selenium	Selenium
Silver			Silver	
Strontium				
Thorium				
Uranium				
Vanadium		Vanadium (Moderate)		
Ytterbium				
Yttrium				
Zinc		Zinc (Moderate)	Zinc	Zinc
Zirconium				

1. Trace elements detected in coal, tabulated from the National Coal Resources Data System developed by the USGS.
2. Title III, Hazardous Air Pollutants List, Clean Air Act Amendments, 1990, pp. 139-143.
3. Trace elements considered potentially toxic to plant and animal life (U.S. National Committee of Geochemistry).
4. Trace element removal studies by conventional coal cleaning (CQ Inc.).
5. Trace elements selected for this study. Boron, germanium, phosphorus, and silver were excluded due to analytical difficulties.

coal refuse to determine if the elements were increased, reduced, or unchanged by cleaning. In addition, measurements were made of the trace element concentrations in the reagents used during testing and in the plant process water to determine if trace elements leached from the coal during cleaning.

During testing, the two-ton-per-hour test plant was operated continuously for approximately 80 hours. Test conditions were optimized for ash and pyritic sulfur rejection and heating value recovery. The change in trace element concentration during cleaning was measured, but no attempt was made to optimize trace element rejection.

Test Coals

Three coals from different seams were studied:

1. Upper Freeport coal, Sheesley Mine in Jefferson County, Pennsylvania (P&N Coal Company). This coal was conventionally precleaned at CQ Inc.'s commercial-scale demonstration cleaning plant in Homer City, Pennsylvania.
2. Pittsburgh No. 8 coal, Emerald Mine in Greene County, Pennsylvania (Cyprus Coal Company). This coal was precleaned in a commercial coal preparation plant.
3. Illinois No. 5 coal, Wabash Mine in Wabash County, Illinois (Amax Coal Company). This coal was processed raw.

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SAMPLING TEST PLAN

Introduction

The sampling test plan was designed to provide data to be used in the evaluation of the performance of the selective agglomeration process. All major process streams were sampled either manually or mechanically according to ASTM procedures.

Proof-of-Concept Facility Operations

The POC facility tested a diesel-based selective agglomeration process, which includes the unit operations as shown in Figure 2-1 and listed below:

- Coal receiving, handling, and crushing.
- Coal grinding.
- High-shear agglomeration.
- Microagglomerate recovery (flotation).
- Product vacuum filtration.
- Product handling, storage, and removal.
- Reagent storage and delivery.
- Water clarification.
- Tailings dewatering.
- Refuse handling, storage, and removal.

Because the POC facility contained only the equipment necessary to demonstrate the selective agglomeration process, conventional coal precleaning and product pelletization were performed by outside vendors although these processes are part of the overall commercial-scale design.

During plant operations, precleaned coal is received by trucks and delivered to the feed coal pad. A front-end loader transfers the coal to a bin and conveyor system. From a 24-ton hopper, the coal is fed to a hammermill crusher and reduced to 1/4-in. topsize before being transferred (by conveyor) to a smaller 3-ton hopper. A belt weigh-feeder controls the coal flow to the ball mills. Here water is added to the coal going to the mills, which produces a slurry of approximately 45 percent solids. A 200 mesh topsize product can be produced by operating the mills in series. In addition, the primary and

secondary mills can be calibrated to produce a 28 mesh or 100 mesh topsize product.

From the ball mills, the slurry is diluted to the desired percent solids for the high-shear agglomeration circuit by adding water. Two high-shear agglomeration vessels were tested. The vessels had volumes of 150 and 45 gallons.

Diesel oil in concentrations less than 1.5 percent (by weight on a dry coal feed basis) is added as an agglomerant before the high-shear vessels. The microagglomerates produced from the high-shear mixer are pumped to the flotation cells where the clean coal is recovered.

Separation of microagglomerated coal from the mineral-bearing water is accomplished in the flotation cells. Frother is added to the cells to generate fine bubbles and promote froth stability.

The product from the first-stage flotation bank of six cells is refloats in the second stage flotation cell bank (which also has six cells). The second-stage flotation product is pumped to a rotary vacuum drum filter for mechanical dewatering. Flotation tailings are sent to a thickener where flocculant is added to aid solids settling. Clarified water is recycled to the head tank and the solid refuse is pumped to a plate and frame press for dewatering and then dumped on the refuse pad for loadout.

The flotation product is dewatered by a rotary vacuum drum filter that produces a moisture content of approximately 25 percent. The filtrate from this process is recycled to the clarified water tank. The product is moved to the product pad on a belt conveyor for loadout.

Reagents used during the operation of the POC facility included:

- High-shear agglomerant (No. 2 diesel oil).
- Flotation frother (Betz M150).

Figure 2-1.
Proof-of-Concept Process and Instrument Drawing

- Flocculant for the thickener.
- Sodium hydroxide (caustic) for pH control.

Diesel oil was stored in a 5000-gallon storage tank and delivered to the process by a metering pump and associated piping. Frother and flocculant were also supplied by metering pumps. Sodium hydroxide was manually added to the system as needed.

Proof-of-Concept Facility Samplers

The POC facility was equipped with five mechanically-operated pneumatic diverter samplers designed for falling slurry streams with low flow rates. Overall, these samplers provided representative samples of main process streams to determine POC material balances. Samplers 2, 4, and 5 were used to collect samples for the trace element study.

These process streams were equipped with these slurry samplers:

Sampler 1.

Location: Above the first-stage flotation feed box.

Sample stream: First-stage flotation feed (high-shear product).

Sampler 2.

Location: First-stage flotation tailings output flange.

Sample stream: Tailings from the first-stage flotation cell bank.

Sampler 3.

Location: Above the first-stage froth tank.

Sample stream: Product slurry from the first-stage flotation cell bank.

Sampler 4.

Location: Second-stage flotation tailings output flange.

Sample stream: Tailings stream from the second-stage flotation cell bank.

Sampler 5.

Location: Above the second-stage froth tank.

Sample stream: Product slurry from the second-stage flotation cell bank.

Sample Collection

The sampling plan incorporated grab, manual, and mechanical sampling of the process streams and raw coal. ASTM and EPA standard methods for the collection and preservation of the samples were strictly employed. The location of sample collection points for the trace elements study is shown in Figure 2-2.

A comprehensive test sampling scheme was developed to obtain trace element material balances around the major plant operations. The process streams sampled were:

- Raw Coal (before conventional cleaning).
- Feed Coal 1/4-inch topsize (before advanced cleaning).
- Product Slurry and Filtrate.
- Tailings Slurry and Filtrate.
- Diesel Oil.
- Flocculant.
- Frother.
- Caustic.
- Water.
 - Laboratory deionized.
 - Plant recycle, make-up, and potable.

The raw coals were sampled prior to conventional precleaning at the individual sites, i.e., the Upper Freeport coal was sampled by CQ Inc., and the Pittsburgh No. 8 coal by Cyprus Coal Company. These samples were then sent to analytical laboratories for preparation and analysis. However, since the Illinois No. 5 coal was processed raw, without precleaning, there were no raw coal samples taken at the mine site. In this case, the feed coal sample served as the equivalent to the raw coal samples of the other two coals.

Samples of feed coal into the POC facility were collected by stop-belt sampling. The loaded conveyor belt was stopped a minimum of 15 times per 8-hour period. A full cross-section cut with parallel sides was removed from the coal stream each time the belt was stopped. These 15 subsamples, each weighing no less than two pounds each, were collected for each composite sample. One composite sample was collected every 8 hours during plant steady-state conditions.

Sampling Test Plan

Figure 2-2.
Proof-of-Concept Process Flowchart and Points of Sample Collection for Trace Element Removal Study

Mechanical sampling was used to collect samples of the first-stage flotation tailings slurry and the second-stage flotation product and tailings slurries. A minimum of 20 full-stream increments for each composite sample were collected with one composite sample collected every 4 hours during steady-state conditions.

Grab sampling was used to obtain secondary process stream samples and reagent samples. The secondary process streams and reagents were recycle, make-up, and potable waters, diesel oil, flocculant, frother, and caustic. Samples of each water stream were collected every 24 hours during steady-state conditions. Samples of diesel oil, flocculant, frother, and caustic were collected after every new shipment.

Table 2-1 identifies the sample, sampling method used, sample location, number of samples, and the analyses performed on these samples.

Process stream flowrate measurements and plant operating data were also gathered during demonstration runs to complete trace element material balances.

Sample Labeling and Chain-of-Custody Procedures

As with the sampling plan, a detailed analytical test plan was developed to track each sample from collection through analysis. Samples were

collected and prepared at the plant site and shipped via intracompany mail (at the conclusion of each test run) to the APCL. Individual self-adhesive labels, each with a unique sample code, were affixed to sample containers immediately following sample collection. The sample code along with run number, operating conditions, sample location and identification, sampler's comments, and other pertinent information was entered into a permanently-bound field notebook. Once the sample was received at APCL, their quality assurance organization became responsible for the samples. APCL also provided splits of some samples to the referee laboratories for duplicate analyses.

During sample collection, the sample coordinator was responsible for using proper containers and labeling, maintaining chain-of-custody forms, and shipment of samples to the APCL. The original chain-of-custody form prepared by the sample coordinator accompanied each shipment of samples while a copy was retained for the record. Field notebook entries provided a backup to data and information contained on the chain-of-custody form. At APCL, chain-of-custody became the responsibility of the quality assurance organization.

Table 2-1
Sampling Procedures for Trace Element Removal Study

Sample	Sampling Method	Sample Location	Number of Samples	Analyses
Raw Coal	Manual	Mine/CQ Inc.	Minimum 15 increments per composite 1 composite per coal type	Trace Elements Short Proximate Ultimate Sulfur Forms
Feed Coal	Manual	Belt Conveyor to 3-ton Bin	Minimum 15 increments per composite 1 composite per 8 hours 10 composites per demo run	Trace Elements Short Proximate Ultimate Sulfur Forms
Product Slurry	Mechanical	Above the second stage froth tank	Minimum 20 increments per composite 1 composite per 4 hours 20 composites per demo run	Trace Elements Short Proximate Ultimate Sulfur Forms
Tailings Slurry	Mechanical	Proportional sampling from tailing streams of No. 1 and No. 2 flotation cell banks	Minimum 20 increments per composite 1 composite per 4 hours 20 composites per demo run	Trace Elements Short Proximate Ultimate Sulfur Forms
Recycle Water	Grab	Thickener Overflow Pump Discharge	1 sample per 24 hours 4 samples per demo run	Trace Elements
Make-up Water	Grab	Make-up Water Header	1 sample per 24 hours 4 samples per demo run	Trace Elements
Potable Water	Grab	Safety Shower and Eye Wash Stations	1 sample per 24 hours 4 samples per demo run	Trace Elements
Diesel Oil	Grab	Storage Tank Discharge	1 sample per shipment	Trace Elements
Flocculant	Grab	55-gallon Drum	1 sample per shipment	Trace Elements
Frother	Grab	55-gallon Drum	1 sample per shipment	Trace Elements
Caustic	Grab	Storage Tank Discharge	1 sample per shipment	Trace Elements

3

ANALYTICAL TEST PLAN

Introduction

Because coal is highly variable and complex with elements bound or contained in different ways, analyzing for trace elements in coal presents special problems for laboratories. In addition, elements may be associated with either the organic or inorganic fractions of the coal or both, and a sample preparation (dissolution) technique that works for one element may not work for another element or even the same element in a different form. There are also special problems of accurately measuring concentrations at the parts per million level, whereby a very small analytical mistake can cause an error of several orders of magnitude. Therefore, accurate results not only depend on maintenance and calibration of laboratory equipment, but also on sample division, preparation, dissolution, and dilution. Moreover, detection of errors is difficult since there is little reliable information as to what is a normal concentration of a specific element in a particular coal.

In addition to trace elements, the coal samples were analyzed for moisture, ash, gross calorific value, total sulfur, carbon, hydrogen, nitrogen, volatile matter, and sulfur forms. The complete data set, including trace elements analyses, is contained in Appendix A.

Methods of Analysis

The Alabama Power Company General Test Laboratory (APCL) in Vamons, Alabama, had the primary responsibility for analyzing the samples. Quality assurance and quality control (QA/QC) sample testing was performed by APCL and the following designated referee laboratories:

- Southern Research Institute, Birmingham, Alabama.
- Iowa State University, Ames, Iowa.

- Huffman Laboratories, Golden, Colorado.
- Standard Laboratories, Cresson, Pennsylvania.

Quality assurance was performed at APCL by regularly analyzing certified reference samples to assure accurate operation of equipment. Also, duplicate analyses of both unknown and certified samples were performed routinely to confirm analytical precision.

Table 3-1 summarizes the samples, analyses performed on these samples, sample preservation techniques, analytical method number, and analytical method used.

Seven samples from each demonstration test were sent to referee laboratories. These samples included one raw coal, two feed coals, two product coals, and two tailings samples. Table 3-2 lists the laboratories and summarizes the methods used by each to determine trace elements. Detailed procedures provided by the individual laboratories are contained in Appendix B.

Additional Quality Assurance/Quality Control Checks

Given the difficulty in accurately measuring the concentration of trace elements in coal, a QA/QC effort in addition to that employed by APCL was implemented involving:

- *Precision Checks.* A review of APCL data for internal consistency.
- *Accuracy Checks.* A comparison of the results obtained on splits of the same sample by APCL and other laboratories.

This QA/QC effort was intended to support APCL's internal QA/QC program, not to supplant it. Primary responsibility for accurate results remained with the laboratory performing the analysis.

Table 3-1
APCL's Analytical Procedures for Trace Element Removal Study

Sample	Analyses	Preservation Method	Analytical Method Number	Analytical Method Description
COAL (Raw, Feed, Product, Tailings)	Gross Calorific Value	Air-tight Seal	ASTM D 3286	Oxygen Bomb Comb.
	Total Sulfur	Air-tight Seal	ASTM D 4239	High Temp Comb. with Infrared Absorption
	Moisture and Ash	Air-tight Seal	ASTM D 3173/3174	MAC-400 Analyzer
	Carbon, Hydrogen, Nitrogen	Air-tight Seal	ASTM D 3136	Combustion
	Volatile Matter	Air-tight Seal	ASTM D 3175	Gravimetric
	Sulfur Forms	Air-tight Seal	ASTM D 2492	Acid Extraction (Organic Sulfur Calculated)
	Mercury	Air-tight Seal	ASTM D 3684	Oxygen Bomb Comb. Hg-Cold Vapor AAS
	Arsenic and Selenium	Air-tight Seal	Developmental Method	Ultrasonic Slurry/GFAAS
	Fluorine	Air-tight Seal	ASTM D 3781 and EPA 300.0	Oxygen Bomb Comb. Ion Chromatography
	Chlorine	Air-tight Seal	ASTM D 4208 and EPA 300.0	Oxygen Bomb Comb. Ion Chromatography
WATER (Recycle, Make-up, Potable)	Other Trace Elements (Ba, Be, Cd, Co, Cr, Mn, Ni, Pb, Zn)	Air-tight Seal	ASTM D 3683	Low-Temperature Ashing and Acid Dissolution with ICP-AES and/or GFAAS
	Mercury	HNO ₃ to pH<2	EPA 600/4-79-020 Method 245.1	Cold Vapor AAS Method
	Fluorine and Chlorine	None	EPA 600/4-79-020 Method 300.0	Ion Chromatography
	Other Trace Elements (As, Ba, Be, Cd, Co, Cr, Mn, Ni, Pb, Se, Zn)	HNO ₃ to pH<2	EPA 600/4-79-020 Method 200.7	Acid Digestion with ICP-AES and/or AAS
PETROLEUM (Diesel Oil)	Other Trace Elements (As, Ba, Be, Cd, Co, Cr, Mn, Ni, Pb, Se, Zn)	None	EPA SW 846 Methods 3030 and 6010	Acid Digestion with ICP-AES and/or AAS
	Mercury	None	ASTM D 3684	Oxygen Bomb Comb.
	Fluorine and Chlorine	None	ASTM D 808 and EPA 300.0	Oxygen Bomb Comb. and Ion Chromatography

Table 3-2
Interlaboratory Agreement Analytical Procedures

		Standard Laboratories	Southern Research Institute	Iowa State University	Huffman Laboratories ^d	Alabama Power Company
As	Preparation Detection	ASTM D3684 GFAAS	Mod ASTM D3683 ^a GFAAS	ASTM D4606 HGAAS	ASTM D4606 HG-ICP	Ultrasonic Slurry ^c GFAAS
Ba	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES
Be	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES
Cd	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a GFAAS	Shapiro ^b GFAAS		ASTM D3683 ICP-AES
Cl	Preparation Detection	ASTM D4208 ISE	ASTM D2361 IC	ASTM D4208 EPA 300.0 (IC)		ASTM D4208 EPA 300.0 (IC)
Co	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 GFAAS
Cr	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES
F	Preparation Detection	ASTM D3761 ISE	ASTM D2361 ISE	ASTM D3761 EPA 300.0 (IC)		ASTM D3761 EPA 300.0 (IC)
Pb	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a GFAAS	Shapiro ^b GFAAS		ASTM D3683 GFAAS
Mn	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES
Hg	Preparation Detection	ASTM D3684 Cold Vapor AAS	SW-846 (7471) Cold Vapor AAS	ASTM D3684 Cold Vapor AAS	ASTM D3684 Cold Vapor AAS	ASTM D3684 Cold Vapor AAS
Ni	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES
Se	Preparation Detection	ASTM D3684 GFAAS	Mod ASTM D3683 ^a GFAAS	ASTM D4606 HGAAS	ASTM D4606 HGAAS	Ultrasonic Slurry ^c GFAAS
Zn	Preparation Detection	ASTM D3683 Flame AAS	Mod ASTM D3683 ^a ICP-AES	Shapiro ^b ICP-AES		ASTM D3683 ICP-AES

^a Samples were ashed according to ASTM D3683 and dissolved by a microwave dissolution method.

^b Samples were ashed according to ASTM D3683 and dissolved by a modified Shapiro Rapid Dissolution Method.

^c Spectrochemica Acta., Vol. 44B, No. 12, pp. 1245-1256, 1989. Bradshaw, Deborah and Slavin, Walter (Perkin-Elmer Corporation)

^d Huffman Laboratories only analyzed for arsenic, selenium, and mercury.

Precision Checks

Once the initial trace element laboratory data was received by SCS, it was evaluated for consistency to determine potential outliers in each data set. A potential outlier was defined as an analysis that differed from the average for the particular coal and sample type (such as feed, product, or tailings) by more than 15 percent. When such a data point was located, a recheck of that sample was performed.

Following reanalysis, the new value was compared with the original value and the average value for the sample type. In all cases where rechecks were involved, if the recheck value was closer to the average for the sample type, it replaced the original number. Appendix C contains the original data set and the results of each recheck.

After all rechecks were completed, an in-depth evaluation was performed to check the APCL values for statistical outliers. Statistically, an outlier is an observation that falls out in the tail of a distribution and can indicate a faulty or incorrectly recorded value (Kenkel, 1989). Each data set for each test series and sample point was analyzed using a box plot statistical technique. A box plot uses the 25th percentile and 75th percentile to identify potential outliers. From this analysis, it was determined that there were no extreme outliers in the APCL-produced numbers, therefore all values in the new data set could be used in interpreting the trace element data.

A second precision check involved the comparison of mass balances around the cleaning operation calculated using trace element concentrations with those calculated using the traditionally measured coal quality parameters of ash and heating value. This precision check is a measure of analytical closure around the cleaning operation. It also did not indicate any extreme outliers in the data set, confirming that all values could be used in interpreting the test results.

Accuracy Checks

Laboratories determine the accuracy of analytical procedures by analyzing standard samples certified by some group or agency to

contain specific concentrations of the parameters in question. The most widely recognized and respected source of analytical standards is the National Institute for Standards and Technology (NIST) in Gaithersburg, Maryland. Unfortunately, the available NIST-certified samples do not cover all the trace elements included in this study. Also, because of the variety of modes of occurrence of trace elements in coal, it is possible for a laboratory to have good results with an NIST standard sample, but experience difficulty with other samples.

In order to increase the confidence level in the results obtained during this test program, a sample exchange program, or "Round Robin," was instituted. In this program, APCL provided splits of some of the samples to referee laboratories for analysis. Iowa State University was provided splits of raw Upper Freeport and Pittsburgh coals as well as POC feed, product, and tailings samples for these same coals. Huffman Laboratory was subcontracted by Iowa State University to perform analyses on arsenic, selenium, and mercury. Standard Laboratories received raw Illinois No. 5 coal samples and POC samples of feed, product, and tailings for the same coal. Southern Research Institute performed trace element analyses on selected samples of all three coals.

The results of these accuracy checks are presented in Appendix A. While there were cases in which poor data agreement was obtained among laboratories, the overall level of consistency indicates that the results produced by APCL are accurate within the limits of currently available procedures and instrumentation.

Establishment of Level of Significance

Data supplied from APCL were used to establish the degree by which two measurements of trace element concentrations must differ to provide assurance that the difference is significant and not an artifact of analytical variability. As part of their internal QA/QC program, APCL personnel performed duplicate analyses of 15 different samples for each trace element included in this study. The Relative Standard deviation (RSD) of each sample pair was calculated according to Equation 1:

$$RSD = 200(A-B)/\sqrt{2(A+B)} \quad (\text{eq. 1})$$

Where A is the value obtained by the first analysis and B is the value obtained by the second analysis.

All 15 RSDs were averaged and this average (Table 3-3) was used to establish the range of concentration equivalent to one RSD for the trace element concentration data used in this report by modifying Equation 1 in the following manner:

$$A = B(\sqrt{2}ARSD + 200)/(200 - \sqrt{2}ARSD) \quad (\text{eq. 2})$$

where A is the concentration one RSD from the measured trace element concentration, B is the measured trace element concentration, and ARSD is the average RSD of APCL's QA/QC data (Table 3-3).

Table 3-3
Average RSDs Supplied by APCL

Element	Average RSD
Arsenic	7.07
Barium	0.75
Beryllium	1.67
Chlorine	3.29
Cobalt	2.91
Chromium	1.95
Fluorine	1.36
Lead	2.09
Mercury	10.64
Manganese	0.75
Nickel	1.74
Zinc	1.36

The value of A calculated in this manner is the range of values contained within one RSD of the measured trace element concentration under the assumption that these values are distributed in a manner defined by the average RSDs presented in Table 3-3. Because both measurements were made by the same laboratory, this is a reasonable assumption.

To provide a conservative analysis of the trace element data, numbers that do not differ by at least two RSDs are assumed to be the same number. For example, in the results of the Upper Freeport testing presented in the Test Results section, arsenic was reduced from 25.3

ppm to 15.8 ppm (Table 4-1) by conventional cleaning. Using both the average RSD from Table 3-2 for arsenic (7.07) and the raw coal concentration of 25.3 ppm in Equation 2, ± 2.7 is the value that defines the range of values within one RSD of 25.3 ppm arsenic. The range of two standard deviations is twice the range of one standard deviation or, in this case, ± 5.3 . Performing the same calculation for the 15.8 ppm arsenic concentration in the cleaned coal produces a range of ± 3.3 at two RSDs.

Two standard deviations are expected to contain 95 percent of a normally distributed population of data as displayed in Figure 3-1. Assuming that the measured concentration is the mean value within a normally distributed population (defined by the data in Table 3-2), then the establishment of a minimum value for arsenic concentration in the raw coal is at the 95-percent confidence level (two standard deviations below the mean) of 20.0 ppm and the establishment of a maximum concentration of 19.1 ppm for the clean coal is also at the 95-percent confidence level. Because the minimum value for the raw coal exceeds the maximum value for the clean coal, the values differ significantly under this criterion, indicating that arsenic was reduced by cleaning.

As a second example, the chromium concentration in the Upper Freeport raw coal was measured as 19 ppm and as 17 ppm in the conventionally cleaned coal (Table 4-1). However, performing the same calculations using 1.95 for chromium (Table 3-3) yields a minimum value of 17.9 for the raw coal and a maximum value of 18.0 for the clean coal. In this case at the 95 percent confidence level, the concentration of chromium in the cleaned coal can be either higher or lower than that of the raw coal and the data are interpreted as showing no significant change in chromium concentration with cleaning.

The range of concentrations calculated in this manner is presented along with the measured data in the Test Results section. In addition, the same approach is used to establish the significance of calculated reductions or increases in trace elements on a heat unit basis with cleaning. In this case, the values required to establish a significant reduction difference are calculated by using both the low and high

These values are also presented in the tables in the Test Results section.

Figure 3-1.
Normal Distribution Curve
(Approximate percentage distribution of data
about the mean)

4

TEST RESULTS

Introduction

Advanced coal cleaning processes such as selective agglomeration may offer advantages over conventional cleaning technologies in reducing trace elements because they are specifically designed to clean fine-sized coal, allowing the coal to be finely ground to potentially liberate additional trace element-bearing minerals. Also, many trace elements tend to be concentrated in fine-sized coal, making efficient methods of removing them especially important. In these tests, the Upper Freeport and Pittsburgh No. 8 coals were ground to minus 200 mesh topsize in the POC facility. The Illinois No. 5 coal was ground to two different topsizes, minus 100 mesh and minus 200 mesh, to evaluate the effects of grinding on liberation.

An added advantage of using the selective agglomeration process is that it can be used in conjunction with conventional equipment in an operating coal cleaning plant to produce additional trace element reductions. The trace element reduction potential of combining conventional cleaning and selective agglomeration can be evaluated for the Pittsburgh No. 8 and Upper Freeport coals because both were pre-cleaned by conventional processes before cleaning by selective agglomeration. However, no effort was made to optimize the combination of conventional and selective agglomeration cleaning for trace element reduction, so the results obtained should be considered conservative.

The interpretation of results presented in this section is based solely on APCL analyses. The data from the referee laboratories were used only to establish the accuracy of the APCL data.

Upper Freeport Coal

The results of the cleaning tests with Upper Freeport coal are presented in Tables 4-1 and 4-2. Table 4-1 presents the measured concentration (parts per million) of each trace element in the raw coal, the average measured concentration of each trace element for all samples of the feed to the agglomeration process (conventionally cleaned coal), and the average measured concentration of clean coal samples from the agglomeration process. Each measured concentration is followed by a number that represents the range of values that contain the true concentration at a 95 percent confidence level. The procedure for calculating this range is discussed in the Establishment of Level of Significance section. For purposes of interpreting the cleaning results, the difference between two concentrations is considered to be within experimental error if the ranges overlap.

As shown in Table 4-1, conventional coal cleaning (represented by the difference in the values of the raw coal and feed coal) appears to have increased the concentration of chlorine, mercury, manganese, and zinc. However, the change in the value of chlorine with cleaning is within experimental error because the high-range value of chlorine in the raw coal (1,916 ppm) exceeds the low-range value of chlorine in the feed coal (1,606 ppm). Since the ranges overlap, the change in chlorine concentration does not qualify as significant.

Conventional coal cleaning reduced the measured concentration of arsenic, barium, cobalt, chromium, fluorine, lead, and nickel. The only one of these elements that does not meet the significance criterion is chromium. The concentration of beryllium was unchanged by conventional cleaning.

Test Results

Table 4-1
Upper Freeport Trace Element Concentration (ppm, Dry Basis)

Element	Raw Coal	Feed Coal	Product Coal
Arsenic	25.3 ±5.3	15.8 ±3.3	6.8 ±1.4
Barium	88 ±1.9	66 ±1.4	48 ±1.0
Beryllium	2 ±0.1	2 ±0.1	2 ±0.1
Chlorine	1,749 ±167	1775 ±169	1900 ±181
Cobalt	13 ±1.1	8 ±0.7	4 ±0.3
Chromium	19 ±1.1	17 ±1.0	13 ±0.7
Fluorine	45 ±1.8	24 ±1.0	35 ±1.4
Lead	10 ±0.6	4.7 ±0.3	3 ±0.2
Mercury	0.03 ±0.01	0.09 ±0.03	0.08 ±0.03
Manganese	20 ±0.4	23 ±0.5	9 ±0.2
Nickel	19 ±1.0	14 ±0.7	9 ±0.5
Zinc	30 ±1.2	33 ±1.3	16 ±0.6
Ash (Wt %)	12.8	10.1	6.27

Table 4-2
Upper Freeport Trace Element Reductions (percent)

Element	Conventional Cleaning	Selective Agglomeration Cleaning	Both Processes Combined
Arsenic	40 ±27	59 ±18	75 ±11
Barium	27 ±3	31 ±3	50 ±2
Beryllium	3 ±9	5 ±9	8 ±9
Chlorine	2 ±19	-2 ±20	0 ±19
Cobalt	40 ±10	52 ±8	72 ±5
Chromium	13 ±10	27 ±8	37 ±7
Fluorine	48 ±4	-39 ±11	28 ±6
Lead	54 ±5	39 ±7	72 ±3
Mercury	-191 ±212	15 ±62	-147 ±179
Manganese	-11 ±5	63 ±2	58 ±2
Nickel	29 ±7	39 ±6	56 ±4
Zinc	-7 ±8	54 ±4	51 ±4
Ash	26	41	56

The performance of the selective agglomeration process, shown in the net change from the feed coal to the product coal, increased the concentration of chlorine and fluorine; however, the change in chlorine concentration fails to meet the significance criterion. No change was produced in the concentration of beryllium, while the concentration of the other elements dropped. The small drop in mercury concentration with cleaning does not meet the significance criterion.

The combination of conventional and selective agglomeration cleaning reduced the concentration of all elements except chlorine

and mercury, while the concentration of beryllium was unchanged. As before, the change in concentration of chlorine does not meet the significance criterion.

Table 4-2 shows the trace element reductions achieved by both conventional and selective agglomeration cleaning. The reductions achieved by conventional cleaning are calculated on a heat unit basis according to the following formula:

$$\text{Reduction} = \frac{100 \times (\text{RCV} - \text{SAFCV})}{\text{RCV}} \quad (\text{eq. 4-1})$$

Where all values are on a heat unit basis:

Raw Coal Value (RCV) = (ppm in Raw Coal)/(Heating Value of Raw Coal)

Selective Agglomeration Feed Coal Value (SAFCV) = (ppm in Selective Agglomeration Feed Coal)/(Heating Value of Selective Agglomeration Feed Coal).

The reductions achieved by the selective agglomeration process are calculated in the same manner except the selective agglomeration feed coal value is used in place of the raw coal value since the clean coal from the conventional cleaning process is also the feed coal to the selective agglomeration process.

Reductions in Table 4-2 are calculated on a heat unit basis because it provides the best measure of the total impact of coal cleaning on potential emissions of air toxics. Cleaning increases the heating value of coal, thereby reducing the tonnage that must be burned to achieve a given thermal output as well as (in most cases) lowering the concentration of impurities. For example, although the concentration of chromium was not significantly reduced by conventional cleaning, the increase in heating value was more than sufficient to offset this, producing a small but significant reduction in chromium. As with the measured concentration values, a significance criterion is presented along with each reduction in Table 4-2. The criterion is calculated by the equation using the range of values given for each element in Table 4-1.

Chromium, as well as arsenic, barium, beryllium, chlorine, cobalt, fluorine, lead, and nickel were reduced by conventional cleaning. The reductions in beryllium and chlorine do not meet the significance criterion. Conventional cleaning increased mercury, manganese, and zinc; however, the increase in zinc is not significant.

The selective agglomeration process reduced all elements except chlorine and fluorine. Of these elements, only the increase in fluorine was significant. For all of the elements reduced, only mercury met the significance criterion.

When the impact of conventional coal cleaning and selective agglomeration are considered together, all elements are reduced except mercury and chlorine. Neither beryllium nor chlorine pass the significance test.

Pittsburgh No. 8 Coal

The results of the cleaning tests with Pittsburgh No. 8 coal are presented in Tables 4-3 and 4-4. Table 4-3 shows the measured concentration (parts per million) of each trace element in the raw coal, the average measured concentration of each trace element for all samples of the feed to the agglomeration process (conventionally cleaned coal), and the average measured trace element concentration of all clean coal samples from the selective agglomeration process. As with the Upper Freeport coal, a significance criterion is given for each measured concentration.

Conventional cleaning appears to increase the concentration of beryllium, chlorine, and fluorine; however, only the change in fluorine is large enough to satisfy the significance criterion. The concentration of all other elements was decreased, though the change in mercury concentration does not satisfy the significance criterion.

The selective agglomeration process decreased the concentration of all elements except chlorine, chromium, and manganese. The following elements did not satisfy the significance criterion: arsenic, chlorine, chromium, fluorine, and mercury.

Together, both cleaning processes decreased the concentration of all elements except chlorine and fluorine, while the beryllium concentration was unchanged. All changes in concentration satisfied the significance criterion.

Table 4-4 presents the trace element reductions achieved by both conventional and selective agglomeration cleaning, as well as the significance criterion. The reductions shown in Table 4-4 are calculated on a heat unit basis in the same manner as for the Upper Freeport coal.

Test Results

Table 4-3

Pittsburgh No. 8 Trace Element Concentration (ppm, Dry Basis)

Element	Raw Coal	Feed Coal	Product Coal
Arsenic	6.9 ±1.5	1.8 ±0.4	1.6 ±0.3
Barium	140 ±3.0	46 ±1.0	42 ±0.9
Beryllium	2 ±0.1	3 ±0.1	2 ±0.1
Chlorine	688 ±66	909 ±87	921 ±88
Cobalt	10 ±0.8	3 ±0.3	2 ±0.2
Chromium	41 ±2.2	12 ±0.7	13 ±0.7
Fluorine	17 ±0.7	95 ±3.7	94 ±3.7
Lead	9 ±0.5	3 ±0.2	1 ±0.1
Mercury	0.10 ±0.03	0.08 ±0.03	0.04 ±0.01
Manganese	35 ±0.8	11 ±0.2	14 ±0.3
Nickel	15 ±0.8	10 ±0.5	8 ±0.4
Zinc	31 ±1.2	8 ±0.3	7 ±0.3
Ash (Wt %)	30.9	7.1	5.0

Table 4-4

Pittsburgh No. 8 Trace Element Reductions (percent)

Element	Conventional Cleaning	Selective Agglomeration Cleaning	Both Processes Combined
Arsenic	81 ±8	13 ±38	84 ±7
Barium	76 ±1	11 ±4	79 ±1
Beryllium	-7 ±10	35 ±6	30 ±7
Chlorine	5 ±18	1 ±19	6 ±18
Cobalt	79 ±4	35 ±11	86 ±2
Chromium	79 ±2	-6 ±12	78 ±2
Fluorine	-300 ±31	3 ±8	-288 ±30
Lead	76 ±3	67 ±4	92 ±1
Mercury	43 ±42	51 ±36	72 ±20
Manganese	78 ±1	-25 ±5	72 ±1
Nickel	52 ±5	22 ±8	63 ±4
Zinc	82 ±1	14 ±7	81 ±1
Ash	84	29	89

For the Pittsburgh No. 8 coal, conventional cleaning reduced all elements studied except beryllium and fluorine. Beryllium, chlorine, and mercury did not meet the significance criterion. The selective agglomeration process reduced all elements except chromium and manganese; however, arsenic, chlorine, chromium, fluorine, and mercury did not satisfy the significance criterion.

When conventional cleaning is considered in conjunction with the selective agglomeration process, all elements studied were reduced except fluorine. Only chlorine did not satisfy the significance criterion.

Illinois No. 5 Coal

The results of the cleaning tests with Illinois No. 5 coal are presented in Tables 4-5 and 4-6 along with the significance criteria. Table 4-5 presents the concentration (parts per million) of each trace element in the raw coal, which in this case is also the feed coal, sent to the POC facility. This coal was ground to two different topsizes (100 mesh and 200 mesh) during the demonstration run.

As shown in Table 4-5, selective agglomeration decreased the concentration of all elements except cobalt in the 100 mesh product and chlorine in the 200 mesh product. The change

in concentration of chlorine did not satisfy the significance criterion in either case, while the concentration of cobalt was unchanged in the 200 mesh product.

Comparing the 100 mesh and 200 mesh products, only the differences in the concentrations of barium, cobalt, chromium, fluorine, manganese, and zinc meet the significance criterion. Of these six cases, only the concentrations of cobalt and fluorine are lower in the 200 mesh product. Also, there is an indication of reduced mercury concentration in the 200 mesh product although this cannot be

quantified. Overall, the additional grinding does not appear to have aided the extraction of most trace elements in these tests. However, optimization of trace element removal was not part of this project. Additional research aimed specifically at trace element removal may provide improved results.

Table 4-6 presents the trace element reductions achieved by selective agglomeration for both sizes. The reductions are calculated on the same heat unit basis as the previous coals and include the significance criteria.

Table 4-5
Illinois No. 5 Trace Element Concentration (ppm, Dry Basis)

Element	Feed Coal	100 Mesh Product Coal	200 Mesh Product Coal
Arsenic	26.1 ±5.5	10.7 ±2.2	12 ±2.5
Barium	80 ±1.7	19 ±0.4	20 ±0.4
Beryllium	5 ±0.2	3 ±0.1	3 ±0.1
Chlorine	1,494 ±142	1,448 ±138	1,590 ±151
Cobalt	7 ±0.8	9 ±0.8	7 ±0.6
Chromium	18 ±1.0	10 ±0.6	12 ±0.7
Fluorine	88 ±3.4	51 ±2.0	43 ±1.7
Lead	31 ±1.9	19 ±1.1	19 ±1.1
Mercury	0.08 ±0.03	0.03 ±0.01	<0.02
Manganese	46 ±1.0	12 ±0.3	16 ±0.3
Nickel	25 ±1.3	18 ±0.9	17 ±0.9
Zinc	88 ±3.4	23 ±0.9	27 ±1.1
Ash (Wt %)	12.8	4.8	4.5

Table 4-6
Illinois No. 5 Trace Element Reductions (percent)

Element	Selective Agglomeration (-100 Mesh)	Selective Agglomeration (-200 Mesh)
Arsenic	64 ±16	61 ±17
Barium	79 ±1	79 ±1
Beryllium	48 ±5	49 ±5
Chlorine	16 ±16	9 ±17
Cobalt	-11 ±19	15 ±14
Chromium	52 ±5	43 ±6
Fluorine	50 ±4	58 ±3
Lead	47 ±6	48 ±6
Mercury	68 ±24	
Manganese	77 ±1	70 ±1
Nickel	38 ±6	42 ±6
Zinc	77 ±2	74 ±2
Ash	77	79

Test Results

For the Illinois No. 5 coal, selective agglomeration reduced all elements studied except cobalt, which increased in the 100 mesh product. Neither chlorine nor cobalt satisfied the significance criterion for either mesh size. As with the concentration data in Table 4-5, these data do not provide a clear indication that the additional grinding increases trace element reduction.

Comparisons Among Test Coals

Table 4-7 contains the measured trace element concentrations of the three raw coals tested. While the Pittsburgh No. 8 coal has the highest concentration of ash, the only trace element concentrations that are highest among the three coals studied are barium and chromium. The Upper Freeport coal, which has the lowest ash content, has the highest measured concentrations of chlorine and cobalt, while the Illinois No. 5 coal contains the highest measured concentrations of more trace elements than the other two coals. While some of the differences in measured concentration do not satisfy the significance criterion, Table 4-7 illustrates the high degree of variability of trace element concentrations in U.S. coal, and the fact that the concentration of ash in raw coal is not always a good predictor of trace element content.

Table 4-8 compares the overall trace element reductions produced by combining conventional coal cleaning and selective agglomeration for the Pittsburgh No. 8 and Upper Freeport coals as well as reductions for the Illinois No. 5 cleaned only by selective agglomeration.

As shown in Table 4-8, the highest reductions for the greatest number of elements occurred for Pittsburgh No. 8 coal cleaned by the combination of conventional and selective agglomeration processes. Similarly, the Upper Freeport coal, with the lowest ash reduction, tended to have trace element reductions lower than the other two coals.

Table 4-8 indicates that some trace element reductions track with ash reduction. To better examine this possible relationship, Figures 4-1 through 4-11 were created. Each figure is a plot of ash reduction versus trace element reduction for those reductions found to meet the significance criterion. Chlorine is not plotted

because no data points were found that met its significance criterion. For the Upper Freeport and Pittsburgh No. 8 coals, data for both conventional and selective agglomeration cleaning are plotted. Data from both the 100 mesh and 200 mesh selective agglomeration cleaning are used for Illinois No. 5 coal.

For most of the plots, a fair to strong linear relationship is evident. Trace elements in this category with at least four data points that meet the significance criteria include arsenic (Figure 4-1), barium (Figure 4-2), cobalt (Figure 4-4), chromium (Figure 4-5), manganese (Figure 4-9), nickel (Figure 4-10), and zinc (Figure 4-11). A linear relationship appears to exist between ash reduction and beryllium reduction (Figure 4-3); however, only three significant data points are available. The plot of mercury reduction (Figure 4-8) is included, but since only two significant data points are available, little can be said about the relationship with ash reduction.

The remaining two plots, fluorine (Figure 4-6 and lead (Figure 4-7), do not show any relationship with ash reduction. With the exception of these two elements and possibly beryllium and mercury, ash reduction seems to be a fairly good predictor of trace element reduction, and the difference in ash reduction among the various tests seems sufficient to explain much of the difference in trace element reductions. However, given the limited number of data points available, this observation should be substantiated by additional testing.

Comparisons with Other Test Results

A comparison of the results from one test program with results obtained by other organizations is useful as a means of confirming accuracy. Unfortunately, no cases that allow an exact comparison were found. Table 4-9 provides a comparison of trace element reductions for Upper Freeport coal cleaned using a combination heavy-media cyclone, water-only cyclone, and spiral concentrator circuit at CQ Inc.'s coal preparation facilities (Akers, et al., 1992). The Upper Freeport coal cleaned by CQ Inc., is not from the same mine as the Upper Freeport used in this test work; however, both coals are from the same region of Pennsylvania.

Table 4-7
Raw Coal Trace Element Concentrations (ppm, Dry Basis)

Element	Upper Freeport	Pittsburgh No. 8	Illinois No. 5
Arsenic	25.3	6.9	26.1
Barium	88	140	80
Beryllium	2	2	5
Chlorine	1,749	688	1,494
Cobalt	13	10	8
Chromium	19	41	18
Fluorine	45	17	88
Lead	10	9	31
Mercury	0.03	0.08	0.09
Manganese	20	35	46
Nickel	19	15	25
Zinc	30	31	88
Ash (Wt %)	12.84	30.88	26.1

Table 4-8
Trace Element Reductions (percent)

Element	Upper Freeport	Pittsburgh No. 8	Illinois No. 5	
			100 Mesh	200 Mesh
Arsenic	75	84	64	61
Barium	50	79	79	79
Beryllium	NSC	29	48	49
Chlorine	NSC	NSC	NSC	NSC
Cobalt	72	86	NSC	NSC
Chromium	37	78	52	43
Fluorine	28	-291	50	58
Lead	72	82	47	48
Mercury	-147	72	68	68
Manganese	58	72	77	70
Nickel	56	62	38	42
Zinc	51	84	77	74
Ash	56	89	77	79

NSC = No significant change

Table 4-9
Upper Freeport Trace Element Reduction Comparison (percent)

Element	CQ Inc., Study	Combined*
Ash	87-91	56
Arsenic	83-84	75
Barium	85-88	50
Chromium	67-69	61
Fluorine	85-87	61
Lead	44-76	72
Mercury	76-78	-147
Nickel	48-58	56
Zinc	71-77	51

* Combined Conventional and Selective Agglomeration

As Table 4-9 shows, the combined conventional and selective agglomeration processes fell within the ranges of the CQ Inc., tests for only two of the nine comparable trace elements. In most cases, the CQ Inc., tests provided higher trace element reductions; however, the ash content for the coal averaged over 23 percent while that used in the selective agglomeration test was less than 13 percent. The higher raw coal ash allowed higher ash reductions to be achieved with conventional cleaning technologies and, as trace element reduction is often proportional to ash reduction, higher trace element reductions were achieved in most cases. The combined conventional and selective agglomeration test produced

Test Results

Figure 4-1. Ash Versus Arsenic Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-2. Ash Versus Barium Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-3. Ash Versus Beryllium Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-4. Ash Versus Cobalt Reduction (Conventional and Selective Agglomeration Cleaning)

Test Results

Figure 4-5. Ash Versus Chromium Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-6. Ash Versus Fluorine Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-7. Ash Versus Lead Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-8. Ash Versus Mercury Reduction (Conventional and Selective Agglomeration Cleaning)

Test Results

Figure 4-9. Ash Versus Manganese Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-10. Ash Versus Nickel Reduction (Conventional and Selective Agglomeration Cleaning)

Figure 4-11. Ash Versus Zinc Reduction (Conventional and Selective Agglomeration Cleaning)

comparable reductions for lead and nickel at a much lower ash reduction, while increasing mercury content. (Mercury was reduced 76-78 percent in the CQ Inc., study.) With the exception of mercury, test results are generally comparable.

Table 4-10 compares the combined conventional and selective agglomeration trace element reductions and those reductions reported by CONSOL, Inc. (Fonseca, et al., 1993), for Pittsburgh seam coal. No specifics were given on the CONSOL test series other than they were done in a conventional coal preparation plant.

As Table 4-10 shows, the combined conventional and selective agglomeration processes provided a higher ash reduction than the CONSOL tests along with a higher reduction of all elements except beryllium, chromium, fluorine, and nickel. Fluorine was increased by the combined process, but reduced in the CONSOL tests. These coals are not from the same mine and these differences may be caused by differences in the mode of occurrence of beryllium, fluorine, and nickel in the two coals. With the exception of fluorine,

the results of these tests are generally comparable.

**Table 4-10
Pittsburgh No. 8 Trace Element Reduction
Comparison (percent)**

Element	CONSOL Study	Combined*
Ash	76-79	89
Arsenic	63-83	84
Beryllium	59-88	30
Cobalt	76-77	86
Chromium	74-78	78
Fluorine	70-83	-288
Lead	73-79	92
Mercury	12-50	72
Nickel	71-78	63

* Combined Conventional and Selective Agglomeration

Leaching of Trace Elements into Process Water

During the demonstration runs, samples were taken of process (recycle) water, make-up water, and the reagents used during testing including No. 2 diesel fuel, caustic, flocculant, and frother. Tables 4-11 and 4-12 provide data

Test Results

taken during Upper Freeport testing, Tables 4-13 and 4-14 for Pittsburgh No. 8 testing, and Tables 4-15 and 4-16 for Illinois No. 5 testing.

If a trace element is being leached from the coal into the recycle water, the concentration of the element in the recycle water should increase during the test run to a concentration significantly greater than that present in the make-up water. Even if this criterion is met, it is still possible that the trace element is being introduced into the recycle water by one or more of the reagents used during testing if the concentration of the element is higher in the reagent than in the recycle water. Given the complex chemistry involved, a conservative approach is used to interpret the data to avoid raising unfounded concerns about trace elements leaching into process water.

Arsenic

In the case of the Pittsburgh No. 8 coal, the arsenic concentration in the make-up and recycle water is reported as less than 0.005 ppm. In the case of the Upper Freeport coal and the Illinois No. 5 coal some of the arsenic concentrations in the recycle water may significantly exceed the concentration in the make-up water. Some of the reagents used during testing may have a higher concentration of arsenic than the make-up water; however, these same reagents were used during testing with the Pittsburgh No. 8 coal without increasing the concentration of arsenic in the recycle water. The data indicate the possibility that a small amount of arsenic leached from the Upper Freeport or Illinois No. 5 coal during the demonstration runs.

Barium

The concentration of barium in the recycle water does not significantly exceed the concentration in the make-up water for any of the three coals.

Beryllium

The concentration of beryllium in the recycle water and the make-up water is reported as less than 0.01 ppm for all three coals.

Cadmium

The concentration of cadmium in the recycle water and the make-up water is reported as less than 0.01 ppm for all three coals except for analysis of two samples of recycle water for Upper Freeport coal reported as 0.01 ppm. These two analyses are insufficient to support the conclusion that cadmium is being leached from the coal.

Chlorine

The concentration of chlorine in the recycle water significantly exceeds that of the make-up water for all tests and there is a general trend for chlorine concentration to increase during each test. While this indicates that chlorine is leaching from the coal into the recycle water, measurements of some of the reagents used during testing, especially the caustic used for pH control and frother, show high concentrations of chlorine. Also, the coal with the highest chlorine content, Upper Freeport, exhibits the lowest concentration of chlorine in the recycle water of the three coals studied. This combination of factors makes the results inconclusive relative to chlorine leaching.

Cobalt

The concentration of cobalt in the recycle water and the make-up water is reported as less than 0.01 ppm for all coals except Upper Freeport. In the case of Upper Freeport, some of the measured concentrations in the recycle water may significantly exceed that of the make-up water. Some of the reagents used during testing have a slightly higher concentration of cobalt than the make-up water; however, these same reagents were used during testing with the other two coals without increasing the concentration of cobalt in the process water. The data indicate the possibility that cobalt leached from the Upper Freeport coal during the demonstration run.

Chromium

The concentration of chromium in the recycle water does not significantly exceed the concentration in the make-up water for any of the three coals.

Table 4-11
Upper Freeport Demonstration Run System Water Analyses (ppm)

Recycle Water

Element	Sample Set				
	1	5	9	13	19
Arsenic	<0.005	0.010	0.008	0.005	<0.005
Barium	0.03	0.04	0.04	0.05	0.05
Beryllium	<0.01	<0.01	<0.01	<0.01	<0.01
Cadmium	<0.01	<0.01	<0.01	0.01	0.01
Chlorine	60.6	63.2	71.2	72.3	70.8
Cobalt	0.06	0.36	0.18	0.33	0.18
Chromium	0.01	0.02	0.02	0.02	0.02
Fluorine	2.76	3.01	3.53	3.58	3.66
Iron	5.93	110.00	70.00	56.64	38.20
Lead	<0.01	<0.01	<0.01	<0.01	<0.01
Mercury	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002
Manganese	0.27	3.80	3.34	4.59	2.33
Nickel	0.05	0.60	0.27	0.14	0.28
Selenium	<0.005	0.005	<0.005	<0.005	<0.005
Zinc	0.08	0.70	0.20	0.43	0.15

Make-up Water

Element	Sample Set 19
Arsenic	<0.005
Barium	0.05
Beryllium	<0.01
Cadmium	<0.01
Chlorine	2.7
Cobalt	0.06
Chromium	0.02
Fluorine	0.31
Iron	0.28
Lead	<0.01
Mercury	<0.0002
Manganese	0.06
Nickel	<0.01
Selenium	<0.005
Zinc	0.03

Table 4-12
Upper Freeport Demonstration Run System Reagent Analyses (ppm)

Reagents

Element	No. 2 Diesel	Caustic	Flocculant	Frother
Arsenic	<1	<0.005	<0.005	<0.005
Barium	<1	0.08	0.04	<0.01
Beryllium	<1	<0.01	<0.01	<0.01
Cadmium	<1	0.01	0.02	0.01
Chlorine	-	972.5	49.0	1800
Cobalt	<1	0.07	0.10	0.06
Chromium	<1	0.05	0.03	0.02
Fluorine	39	<0.01	38	27
Lead	3	0.65	0.66	0.16
Mercury	<0.12	<0.0002	<0.0002	0.0196
Manganese	<1	0.02	0.75	<0.01
Nickel	2	0.08	0.10	0.06
Selenium	-	<0.005	0.006	<0.005
Zinc	4	0.07	0.12	0.02

Test Results

Table 4-13
Pittsburgh No. 8 Demonstration Run System Water Analyses (ppm)

Recycle Water

Element	Sample Set						
	2	5	7	9	12	16	20
Arsenic	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005	<0.005
Barium	0.05	0.05	0.06	0.06	0.04	0.05	0.06
Beryllium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cadmium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Chlorine	92.0	80.6	80.6	84.7	100.0	76.4	87.9
Cobalt	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Chromium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Fluorine	1.48	1.69	1.53	1.74	2.52	2.73	2.52
Iron	2.03	2.52	1.67	3.32	<0.01	2.03	2.75
Lead	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mercury	<0.0002	<0.0002	<0.0002	0.0002	<0.0002	<0.0002	<0.0002
Mangan.	0.15	0.12	0.12	0.15	0.01	0.15	0.14
Nickel	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Selenium	0.005	0.005	0.005	0.005	0.005	0.005	0.005
Zinc	0.02	0.02	0.02	0.02	0.02	0.03	0.02

Make-up Water

Element	Sample Set 7
Arsenic	<0.005
Barium	0.04
Beryllium	<0.01
Cadmium	<0.01
Chlorine	41.7
Cobalt	<0.01
Chromium	<0.01
Fluorine	0.24
Iron	-
Lead	0.02
Mercury	<0.0002
Manganese	0.04
Nickel	<0.02
Selenium	<0.005
Zinc	0.01

Table 4-14
Pittsburgh No. 8 Demonstration Run System Reagent Analyses (ppm)

Reagents

Element	No. 2 Diesel	Caustic	Flocculant	Frother
Arsenic	<1	<0.005	0.10	0.32
Barium	<1	0.08	1.90	5.80
Beryllium	<1	<0.01	<0.04	<0.04
Cadmium	<1	0.01	<0.05	0.06
Chlorine	<150	972.5	18.1	13.6
Cobalt	<1	0.07	0.10	0.15
Chromium	<1	0.05	0.05	0.17
Fluorine	31	<0.01	0.44	3.15
Lead	<1	0.65	0.13	0.49
Mercury	0.0300	<0.0002	<0.0002	0.0756
Manganese	<1	0.02	0.45	<0.04
Nickel	<1	0.08	0.05	0.34
Selenium	<1	<0.005	0.11	0.24
Zinc	<1	0.07	0.38	0.46

Table 4-15
Illinois No. 5 Demonstration Run System Water Analyses (ppm)

Recycle Water

Element	Sample Set						
	4	6	11	13	15	17	20
Arsenic	0.005	0.008	0.007	0.003	0.002	0.003	0.005
Barium	0.03	0.03	0.03	0.03	0.03	0.03	0.03
Beryllium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Cadmium	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Chlorine	164.6	175.3	193.9	212.6	184.6	227.2	244.6
Cobalt	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Chrom.	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Fluorine	0.31	0.31	0.39	0.31	0.39	0.45	2.13
Iron	0.872	1.520	2.460	0.932	0.864	1.160	1.640
Lead	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01	<0.01
Mercury	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002	<0.0002
Mangan.	0.80	0.80	0.83	0.91	0.92	1.01	1.15
Nickel	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02	<0.02
Selenium	0.003	0.002	0.003	0.003	0.003	0.002	0.003
Zinc	0.04	0.02	0.02	0.02	0.01	0.01	0.01

Make-up Water

Element	Sample Set 17
Arsenic	<0.002
Barium	0.05
Beryllium	<0.01
Cadmium	<0.01
Chlorine	34.00
Cobalt	<0.01
Chromium	<0.01
Fluorine	0.15
Iron	-
Lead	<0.01
Mercury	<0.0002
Manganese	0.01
Nickel	<0.02
Selenium	<0.0002
Zinc	0.02

Table 4-16
Illinois No. 5 Demonstration Run System Reagent Analyses (ppm)

Reagents

Element	No. 2 Diesel Fuel
Arsenic	<1
Barium	<1
Beryllium	<1
Cadmium	<1
Chlorine	<150
Cobalt	<1
Chromium	<1
Fluorine	657
Iron	1.000
Lead	<1
Mercury	<0.02
Manganese	<1
Nickel	<1
Selenium	<1
Zinc	<1

Test Results

Fluorine

The concentration of fluorine in the recycle water significantly exceeds that of the make-up water for all tests and there is a strong trend for fluorine concentration to increase during each test. While this indicates that fluorine is leaching from the coal into the recycle water, measurements of some of the reagents used during testing, especially the No. 2 diesel, frother and flocculant, show high concentrations of fluorine. Also, the coal with the lowest fluorine content, Upper Freeport, exhibits the highest concentration of fluorine in the recycle water of the three coals studied. This combination of factors makes the results inconclusive relative to fluorine leaching.

Iron

Iron content in the make-up water was determined only for Upper Freeport coal and was measured only for one reagent (No. 2 diesel) during the Illinois No. 5 tests. Although limited data are available, the iron content of the recycle water during the Upper Freeport test is over 100 times greater than the make-up water, indicating that iron is leaching from the coal or as a result of contamination from the plant (e.g., ball mill, rust).

Lead

The concentration of lead in the recycle water and the make-up water is reported as less than 0.01 ppm for all three coals except for one analysis of make-up water for Pittsburgh coal reported as 0.02 ppm. There is no indication that lead is leaching from the coal during testing.

Mercury

The concentration of mercury in the recycle water and the make-up water is reported as less than 0.0002 ppm for all three coals except for one analysis of make-up water for Pittsburgh coal reported as 0.0002 ppm. There is no indication that mercury is leaching from the coal during testing.

Manganese

The concentration of manganese in the recycle water significantly exceeds that of the make-up water for almost all tests and manganese concentration tends to increase in the recycle water during testing with the Illinois No. 5 coal. The reagents used during testing contain measurable concentrations of manganese; however, in the case of the Upper Freeport coal, the concentration of manganese in the recycle water significantly exceeds that of any reagent. The data indicate the possibility that manganese leached from the Upper Freeport coal during the demonstration run.

Nickel

The concentration of nickel in the recycle water is reported as less than 0.02 ppm for all tests except Upper Freeport, which exhibited a significant increase in concentration over that of the make-up water. However, the concentration of nickel is also higher in the No. 2 diesel than in the recycle water. This combination of factors makes the results inconclusive relative to nickel leaching.

Selenium

The concentration of selenium in the recycle water of the Illinois No. 5 test appears significantly higher than the make-up water; however, some of the reagents used during testing have much higher concentrations than observed in the recycle water. Therefore, the data does not support the conclusion that selenium was leached from the coal during the demonstration run.

Zinc

The concentration of zinc in the Upper Freeport recycle water appears higher than in the make-up water. However, the concentration of zinc is also higher in the No. 2 diesel than in the recycle water, producing inconclusive results.

5

CONCLUSIONS AND RECOMMENDATIONS

Cleaning Test Conclusions

Table 5-1 summarizes the cleaning results for Upper Freeport coal. The reductions (or increases) in the table are calculated on a heat unit basis because this approach provides the best measure of the total impact of coal cleaning on potential emissions of air toxics. Conventional cleaning significantly increased mercury and manganese in the clean coal while arsenic, barium, chromium, cobalt, fluorine, lead, and nickel were reduced. There was no significant change in the other elements studied. The selective agglomeration process did not produce a significant change in beryllium, chlorine and mercury, while all other elements were significantly reduced except fluorine.

When the impact of conventional coal cleaning and selective agglomeration are considered together for Upper Freeport coal, all elements are reduced except beryllium, mercury, and chlorine. Neither beryllium nor chlorine changed significantly with cleaning while mercury increases.

For the Pittsburgh No. 8 coal (Table 5-2), conventional cleaning did not significantly change beryllium, chlorine, and mercury, and provided a reduction in all other elements studied except fluorine. The selective agglomeration process did not change arsenic, chlorine, chromium, and fluorine. All other elements except manganese were reduced.

When conventional cleaning is considered in conjunction with the selective agglomeration process for Pittsburgh No. 8 coal, all elements studied were reduced except fluorine, which increased, and chlorine, which was unchanged.

The results of the cleaning tests with Illinois No. 5 coal are presented in Table 5-3. There were no conventional cleaning tests performed for Illinois No. 5 coal; however, the raw coal was

ground to two different topsizes before cleaning, 100 mesh and 200 mesh.

For the Illinois No. 5 coal, selective agglomeration reduced all elements studied except cobalt and chlorine, which were not significantly changed. In these tests, grinding to 200 mesh before cleaning did not appear to make much overall difference in trace element removal.

For most of the trace elements studied, a fairly strong linear relationship appears to exist between ash reduction and trace element reduction. Trace elements in this category include arsenic, barium, cobalt, chromium, manganese, nickel, and zinc. A linear relationship may exist between ash reduction and both beryllium reduction and mercury reduction; however, limited data are available for these elements.

The remaining two elements, fluorine and lead, do not show any relationship with ash reduction. With the exception of these two elements and possibly beryllium and mercury, ash reduction seems to be a fairly good predictor of trace element reduction, and the difference in ash reduction between the various tests seems sufficient to explain much of the difference in trace element reductions. However, given the limited number of data points available, this observation should be substantiated by additional testing.

Even though neither process was optimized for trace element removal, both conventional coal cleaning and selective agglomeration were found to be effective in removing most trace elements from coal. The ability of these technologies to reduce trace elements is enhanced in situations in which high ash reductions can be achieved.

Conclusions and Recommendations

Leaching of Trace Elements into Process Water Conclusions

During the demonstration runs, samples were taken of process (recycle) water, make-up water, and the reagents used during testing, including No. 2 diesel fuel, caustic, flocculant,

and frother, to determine if any trace elements were leaching from the coal during cleaning. Based on analysis of these data, some indication was found that a small amount of arsenic, cobalt, iron, and manganese leached from one or more coals during testing.

Table 5-1
Trace Element Results: Upper Freeport Coal

Element	Conventional Cleaning	Selective Agglomeration	Combined
Arsenic	-	-	-
Barium	-	-	-
Beryllium	o	o	o
Chlorine	o	o	o
Cobalt	-	-	-
Chromium	-	-	-
Fluorine	-	+	-
Lead	-	-	-
Mercury	+	o	+
Manganese	+	-	-
Nickel	-	-	-
Zinc	o	-	-

+ Trace Element Increased
- Trace Element was Reduced
o No Significant Change

Table 5-2
Trace Element Results: Pittsburgh No. 8 Coal

Element	Conventional Cleaning	Selective Agglomeration	Combined
Arsenic	-	o	-
Barium	-	-	-
Beryllium	o	-	-
Chlorine	o	o	o
Cobalt	-	-	-
Chromium	-	o	-
Fluorine	+	o	+
Lead	-	-	-
Mercury	o	-	-
Manganese	-	+	-
Nickel	-	-	-
Zinc	-	-	-

+ Trace Element Increased
- Trace Element was Reduced
o No Significant Change

Table 5-3
Trace Element Results: Illinois No. 5 Coal

Element	(-100 Mesh) Selective Agglomeration	(-200 Mesh) Selective Agglomeration
Arsenic	-	-
Barium	-	-
Beryllium	-	-
Chlorine	o	o
Cobalt	o	o
Chromium	-	-
Fluorine	-	-
Lead	-	-
Mercury	-	-
Manganese	-	-
Nickel	-	-
Zinc	-	-

- Trace Element was Reduced
o No Significant Change

Recommendations

Many of the trace elements found in coal have been classified as hazardous air pollutants under the 1990 Clean Air Act Amendments. Removal of these elements before the coal is burned eliminates the possibility of atmospheric release. While it is unlikely that a physical coal cleaning process such as selective agglomeration will remove all trace elements, this project has demonstrated that large reductions are possible. In order to better define the role that coal cleaning can play in control of air toxics, additional work is required:

- The five laboratories involved in this study used a variety of analytical techniques to measure the concentration of trace elements in coal and, in some cases, agreement between laboratories was poor. Part of the difficulty is that there is no general agreement as to which analytical procedures are best for which trace elements. This problem should be addressed by additional research to evaluate various analytical procedures. Also, there is currently only one subbituminous coal NIST standard reference for trace elements and one bituminous standard along with a separate mercury standard. Increasing the number of NIST standard references would allow laboratories to evaluate analytical

procedures under a greater variety of conditions.

- Given that conventional and advanced coal cleaning technologies have a high potential as cost-effective air toxics control measures, additional work should be performed to evaluate the trace element removal abilities of these technologies at pilot and commercial scale.
- Most information about the removal of trace elements by coal cleaning is empirical. Fundamental studies of the mechanisms of removal, including the interaction between mineral form and behavior during coal cleaning, are likely to lead to improved methods of extracting trace elements from coal.
- The data concerning the potential leaching of trace elements into the process recycle water indicates that leaching was not a significant concern for the majority of trace elements. However, these results are considered preliminary and the issue of trace-element leaching should be investigated further.

Upper Freeport

Upper Freeport Demonstration Run
Raw and Feed Coal Analyses
All Analyses Reported On A Dry Basis
Results % by weight unless otherwise noted

Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Moist	Ash	Vol	FC	BTU/lb
AT-UF-000 *	Upper Freeport Raw Coal	1.24	12.84	30.77	56.39	13388
	CQ, Inc.	5.19	13.63	-	-	13300
	Geochemical Testing	1.16	13.37	-	-	13319
	SRI	-	13.2	-	-	-
	SRI (Dup AT-UF-000)	-	13.2	-	-	-
Average		2.53	13.26	-	-	13336
Standard Deviation		1.88	0.29	-	-	38
AT-UF-011	Feed, CDSB #1 & #2	5.82	10.87	31.83	57.30	13680
AT-UF-014	Feed, CDSB #3 & #4	5.79	10.55	32.41	57.04	13752
AT-UF-042 *	Feed, CDSB #5 & #6	5.81	9.73	32.71	57.56	13899
	Geochemical Testing	5.26	10.07	-	-	13799
	SRI	-	9.78	-	-	-
AT-UF-058	Feed, CDSB #7 & #8	5.95	10.12	31.38	58.50	13819
AT-UF-075	Feed, CDSB #9 & #10	5.75	10.22	32.61	57.17	13766
AT-UF-093	Feed, CDSB #11 & #12	5.96	9.96	32.77	57.27	13822
AT-UF-098	Feed, CDSB #13 & #14	5.43	10.02	32.39	57.59	13873
AT-UF-110	Feed, CDSB #15 & #16	5.66	9.56	32.03	58.41	13902
AT-UF-112	Feed, CDSB #17 & #18	5.88	9.82	31.93	58.25	13879
AT-UF-142 *	Feed, CDSB #19 & #20	5.46	10.22	31.94	57.84	13840
	Geochemical Testing	4.93	9.75	-	-	13851
Average		5.75	10.11	32.20	57.69	13823
Standard Deviation		2.61	5.39	15.91	28.68	6692

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-UF-000 *	Upper Freeport Raw Coal	75.87	4.20	1.32	3.13	2.64	1.08	0.11	1.45
	CQ, Inc.	-	-	-	-	2.83	0.84	0.03	1.96
	Geochemical Testing	-	-	-	-	2.75	0.71	0.05	1.99
	SRI	-	-	-	-	-	-	-	-
	SRI (Dup AT-UF-000)	-	-	-	-	-	-	-	-
Average		-	-	-	-	2.74	0.88	0.06	1.80
Standard Deviation		-	-	-	-	0.08	0.15	0.03	0.25
AT-UF-011	Feed, CDSB #1 & #2	77.32	4.69	1.39	4.18	1.55	0.55	0.18	0.82
AT-UF-014	Feed, CDSB #3 & #4	78.00	4.72	1.42	3.75	1.56	0.68	0.15	0.73
AT-UF-042 *	Feed, CDSB #5 & #6	78.54	4.71	1.43	3.99	1.60	0.63	0.16	0.81
	Geochemical Testing	-	-	-	-	1.50	0.54	0.22	0.74
	SRI	-	-	-	-	-	-	-	-
AT-UF-058	Feed, CDSB #7 & #8	78.38	4.64	1.42	3.86	1.58	0.70	0.17	0.71
AT-UF-075	Feed, CDSB #9 & #10	78.33	4.64	1.42	3.80	1.59	0.56	0.17	0.86
AT-UF-093	Feed, CDSB #11 & #12	78.22	4.54	1.43	4.27	1.58	0.66	0.16	0.76
AT-UF-098	Feed, CDSB #13 & #14	78.72	4.45	1.43	3.73	1.65	0.80	0.15	0.70
AT-UF-110	Feed, CDSB #15 & #16	78.95	4.43	1.44	3.98	1.64	0.92	0.18	0.54
AT-UF-112	Feed, CDSB #17 & #18	78.74	4.42	1.43	3.98	1.61	0.96	0.14	0.51
AT-UF-142 *	Feed, CDSB #19 & #20	78.87	4.74	1.37	3.14	1.66	0.95	0.14	0.57
	Geochemical Testing	-	-	-	-	1.51	0.56	0.22	0.73
Average		78.41	4.60	1.42	3.87	1.60	0.74	0.16	0.70
Standard Deviation		37.88	2.79	7.06	12.60	3075.33	0.40	0.07	0.66

Samples marked with (*) are samples that have QC analyses directly underneath.
CDSB = Crusher Discharge Stop-belt Feed Coal Samples.

Upper Freeport Demonstration Run
Raw and Feed Coal Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Method		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-UF-000 *	Upper Freeport Raw Coal	25.3	88	2	< 1.0	1749	13	12	19
	Iowa State University	77	117.2	1.6	0.07	1660	7.8	-	15.2
	Huffman Laboratories	87	-	-	-	-	-	-	-
	SRI	85.3	75.3	1.55	1.08	2209	6.64	-	19.4
	SRI (Dup AT-UF-000)	-	-	-	-	2142	-	-	-
AT-UF-011	Feed, CDSB #1 & #2	22.1	66	2	< 1.0	1868	8	8	19
AT-UF-014	Feed, CDSB #3 & #4	12.3	69	2	< 1.0	1343	10	6	18
AT-UF-042 *	Feed, CDSB #5 & #6	16.9	63	2	< 1.0	1778	7	5	16
	Iowa State University	22	78.5	1.6	0.05	1540	4.8	-	13.4
	Huffman Laboratories	28	-	-	-	-	-	-	-
	SRI	30.8	67.5	1.52	0.351	2293	4.32	-	16.9
AT-UF-058	Feed, CDSB #7 & #8	14.7	66	2	< 1.0	1857	9	5	17
AT-UF-075	Feed, CDSB #9 & #10	17.4	69	2	< 1.0	1869	7	6	16
AT-UF-093	Feed, CDSB #11 & #12	14.6	64	2	< 1.0	1507	9	6	16
AT-UF-096	Feed, CDSB #13 & #14	17.2	70	2	< 1.0	2216	7	7	16
AT-UF-110	Feed, CDSB #15 & #16	16.6	68	2	< 1.0	1619	7	5	15
AT-UF-112	Feed, CDSB #17 & #18	11.9	63	2	< 1.0	2055	6	5	16
AT-UF-142 *	Feed, CDSB #19 & #20	13.8	61	2	< 1.0	1641	9	6	16
	Iowa State University	24	89.5	1.6	0.06	1670	5.1	-	13.3
	Huffman Laboratories	28	-	-	-	-	-	-	-
Average		15.8	66	2	< 1.0	1775	8	6	17
Standard Deviation		2.8	3	0	0	245	1	1	1

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-UF-000 *	Upper Freeport Raw Coal	45	10	0.03	20	17	19	< 2	30
	Iowa State University	59	13.12	0.26	17.3	-	17.1	1.2	33.9
	Huffman Laboratories	-	-	0.06	-	-	-	0.24	-
	SRI	31.7	11.9	0.06	12.1	-	18.4	< 2	28.8
	SRI (Dup AT-UF-000)	38.3	-	-	-	-	-	-	-
AT-UF-011	Feed, CDSB #1 & #2	19	5	0.15	31	11	15	< 2	31
AT-UF-014	Feed, CDSB #3 & #4	17	5	0.05	24	5	15	< 2	38
AT-UF-042 *	Feed, CDSB #5 & #6	20	4	0.03	22	11	14	< 2	35
	Iowa State University	14	6.4	0.13	19.3	-	13.4	0.7	30.7
	Huffman Laboratories	-	-	0.09	-	-	-	0.10	-
	SRI	64.8	7.45	0.13	15.4	-	15.3	< 2	45.9
AT-UF-058	Feed, CDSB #7 & #8	17	5	< 0.02	24	5	15	< 2	39
AT-UF-075	Feed, CDSB #9 & #10	18	5	0.14	24	5	14	< 2	33
AT-UF-093	Feed, CDSB #11 & #12	17	5	0.13	21	5	14	< 2	35
AT-UF-096	Feed, CDSB #13 & #14	16	4	0.08	20	7	14	< 2	27
AT-UF-110	Feed, CDSB #15 & #16	55	4	0.12	20	5	14	< 2	32
AT-UF-112	Feed, CDSB #17 & #18	16	5	0.09	20	4	14	< 2	30
AT-UF-142 *	Feed, CDSB #19 & #20	49	5	0.14	20	5	14	< 2	31
	Iowa State University	111	6.53	0.14	17.9	-	13.5	0.8	30.9
	Huffman Laboratories	-	-	0.06	-	-	-	0.03	-
Average		24	4.7	0.09	23	6	14	< 2	33
Standard Deviation		14	0.5	0.05	3	2	0	0	4

Samples marked with (*) are samples that have QC analyses directly underneath.
(<) Indicates result was below instrument detection limits.
CDSB = Crusher Discharge Stop-belt Feed Coal Samples.

Upper Freeport Demonstration Run
Second Stage Flotation Product Analyses
All Analyses Reported On A Dry Basis
Results % By Weight Unless Otherwise Noted
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-UF-056	F2PR- 3, Coal Product	6.32	32.34	61.34	14496
AT-UF-065	F2PR- 4, Coal Product	6.09	32.12	61.79	14580
AT-UF-050	F2PR- 7, Coal Product	6.18	32.59	61.23	14522
AT-UF-088	F2PR-11, Coal Product	7.21	32.06	60.73	14021
AT-UF-103 *	F2PR-13, Coal Product	6.29	32.49	61.22	14550
	Geochemical Testing	6.35	-	-	14468
	SRI	6.33	-	-	-
AT-UF-117	F2PR-17, Coal Product	5.67	33.16	61.17	14534
AT-UF-125	F2PR-18, Coal Product	6.15	32.34	61.51	14571
AT-UF-127 *	F2PR-19, Coal Product	6.25	32.58	61.17	14565
	Geochemical Testing	6.20	-	-	14507
Average		6.27	32.46	61.27	14480
Standard Deviation		0.40	0.32	0.28	175
AT-UF-030	VFPR- 4, Filter Product	6.40	32.95	60.65	14526
AT-UF-124	VFPR-19, Filter Product	6.30	32.24	61.46	14527
Average		6.35	32.60	61.06	14527
Standard Deviation		0.05	0.35	0.40	1

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-UF-056	F2PR- 3, Coal Product	82.31	5.17	1.43	3.62	1.15	0.65	0.03	0.47
AT-UF-065	F2PR- 4, Coal Product	82.49	5.11	1.44	3.68	1.19	0.63	0.03	0.53
AT-UF-050	F2PR- 7, Coal Product	81.53	4.70	1.42	4.92	1.25	0.79	0.02	0.44
AT-UF-088	F2PR-11, Coal Product	79.05	4.14	1.41	6.96	1.23	0.76	0.13	0.34
AT-UF-103 *	F2PR-13, Coal Product	82.52	5.14	1.42	3.41	1.22	0.64	0.02	0.56
	Geochemical Testing	-	-	-	-	1.08	0.52	0.01	0.55
	SRI	-	-	-	-	-	-	-	-
AT-UF-117	F2PR-17, Coal Product	82.36	5.16	1.42	4.22	1.17	0.70	0.02	0.45
AT-UF-125	F2PR-18, Coal Product	82.58	5.19	1.42	3.49	1.17	0.67	0.04	0.46
AT-UF-127 *	F2PR-19, Coal Product	82.33	5.20	1.42	3.58	1.22	0.74	0.02	0.46
	Geochemical Testing	-	-	-	-	1.16	0.58	0.05	0.53
Average		81.90	4.98	1.42	4.24	1.20	0.70	0.04	0.46
Standard Deviation		1.12	0.35	0.01	1.13	0.03	0.06	0.04	0.06
AT-UF-030	VFPR- 4, Filter Product	82.87	4.90	1.43	3.18	1.22	0.37	0.34	0.51
AT-UF-124	VFPR-19, Filter Product	82.28	4.90	1.40	3.79	1.33	0.67	0.08	0.58
Average		82.58	4.90	1.42	3.49	1.28	0.52	0.21	0.55
Standard Deviation		0.30	0.00	0.01	0.30	0.06	0.15	0.13	0.03

Samples marked with (*) are samples that have QC analyses directly underneath.

F2PR = Second stage flotation product collected by mechanical sampler and dewatered in laboratory.

VFPR = Sample collected from rotary vacuum drum system in plant.

Upper Freeport Demonstration Run
Second Stage Flotation Product Samples
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Methods		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-UF-056	F2PR- 3, Coal Product	2.1	39	2	< 1.0	1876	5	7	16
AT-UF-065	F2PR- 4, Coal Product	4.1	39	2	< 1.0	2103	5	9	13
AT-UF-050	F2PR- 7, Coal Product	5.2	39	3	< 1.0	1736	3	9	6
AT-UF-088	F2PR-11, Coal Product	13.0	48	2	< 1.0	1712	4	11	19
AT-UF-103 *	F2PR-13, Coal Product	7.7	54	2	< 1.0	1972	4	9	8
	Iowa State University	15	34.7	1.5	0.04	1920	3.2	-	15.4
	Huffman Laboratories	18	-	-	-	-	-	-	-
	SRI	21.2	29.5	1.51	0.062	3818	2.84	-	20.7
AT-UF-117	F2PR-17, Coal Product	7.3	55	2	< 1.0	1888	4	9	17
AT-UF-125	F2PR-18, Coal Product	6.3	56	2	< 1.0	1968	5	10	8
AT-UF-127 *	F2PR-19, Coal Product	8.7	50	2	< 1.0	1947	5	8	18
	Iowa State University	16	34.5	1.5	0.03	1930	3.0	-	15.6
	Huffman Laboratories	17	-	-	-	-	-	-	-
Average		6.8	48	2	< 1.0	1900	4	9	13
Standard Deviation		3.1	7	0	-	121	1	1	5
AT-UF-030	VFPR- 4, Vacuum Filter Pr	13.7	61	2	< 1.0	2154	5	9	9
AT-UF-124	VFPR-19, Vacuum Filter P	10.7	66	2	< 1.0	2099	6	9	6
Average		12.2	64	2	< 1.0	2127	6	9	8
Standard Deviation		1.5	3	0	0	28	1	0	2

Test Methods		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-UF-056	F2PR- 3, Coal Product	26	3	0.10	9	11	9	< 2	17
AT-UF-065	F2PR- 4, Coal Product	27	3	0.06	9	14	10	< 2	16
AT-UF-050	F2PR- 7, Coal Product	65	3	0.10	10	7	9	< 2	14
AT-UF-088	F2PR-11, Coal Product	64	4	0.13	10	13	9	< 2	17
AT-UF-103 *	F2PR-13, Coal Product	42	4	< 0.02	8	8	10	< 2	17
	Iowa State University	94	3.62	0.081	6.8	-	10.5	0.6	14
	Huffman Laboratories	-	-	0.06	-	-	-	0.03	-
	SRI	< 25	4.61	< 0.03	5.62	-	11.7	< 2	12.8
AT-UF-117	F2PR-17, Coal Product	17	3	0.12	8	9	9	< 2	14
AT-UF-125	F2PR-18, Coal Product	18	3	0.08	8	9	9	< 2	16
AT-UF-127 *	F2PR-19, Coal Product	22	4	0.06	8	8	9	< 2	15
	Iowa State University	84	3.13	0.077	7.0	-	10.2	0.6	13.6
	Huffman Laboratories	-	-	0.07	-	-	-	0.05	-
Average		35	3	0.08	9	10	9	< 2	16
Standard Deviation		18	0	0.04	1	2	0	0	1
AT-UF-030	VFPR- 4, Filter Product	26	4	0.09	10	18	1	< 2	17
AT-UF-124	VFPR-19, Filter Product	11	2	0.11	10	6	9	< 2	17
Average		19	3	0.10	10	12	5	< 2	17
Standard Deviation		8	1	0.01	0	6	4	0	0

Samples marked with (*) are samples that have QC analyses directly underneath.

(<) Indicates result below instrument detection limits.

F2PR = Second stage flotation product collected by mechanical sampler and dewatered in laboratory.

VFPR = Sample collected from rotary vacuum drum system in plant.

Upper Freeport Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
Results % By Weight Unless Otherwise Noted
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-UF-067	FLT- 3, Flotation Tailings	46.08	25.21	28.71	7799
AT-UF-036	FLT- 4, Flotation Tailings	55.79	24.59	19.62	6160
AT-UF-051	FLT- 7, Flotation Tailings	40.43	26.63	32.94	8797
AT-UF-087	FLT-11, Flotation Tailings	33.23	25.43	41.34	10066
AT-UF-104 *	FLT-13, Flotation Tailings	32.27	25.47	42.26	10104
	Geochemical Testing	31.99	-	-	10067
	SRI	32.2	-	-	-
	SRI (Dup AT-UF104)	32.2	-	-	-
AT-UF-116	FLT-17, Flotation Tailings	38.90	24.93	36.17	9037
AT-UF-126	FLT-18, Flotation Tailings	42.64	23.55	33.81	8275
AT-UF-128 *	FLT-19, Flotation Tailings	49.14	23.17	27.69	7263
	Geochemical Testing	49.33	-	-	7206
Average		42.31	24.87	32.82	8438
Standard Deviation		7.43	1.04	6.99	1271

SCS SN	Sample Description	C	H	N	O	Tot	Sulfur Forms		
							Org	Sulf	Pyr
AT-UF-067	FLT- 3, Flotation Tailings	43.73	2.94	0.76	2.65	3.84	0.13	0.11	3.60
AT-UF-036	FLT- 4, Flotation Tailings	34.69	2.44	0.61	1.99	4.48	1.20	0.12	3.16
AT-UF-051	FLT- 7, Flotation Tailings	49.62	2.60	0.90	3.09	3.36	0.94	0.16	2.26
AT-UF-087	FLT-11, Flotation Tailings	54.24	2.69	0.95	6.00	2.89	0.48	0.10	2.31
AT-UF-104 *	FLT-13, Flotation Tailings	57.16	3.55	1.01	3.04	2.97	0.44	0.11	2.42
	Geochemical Testing	-	-	-	-	3.00	0.59	0.15	2.26
	SRI	-	-	-	-	-	-	-	-
	SRI (Dup AT-UF104)	-	-	-	-	-	-	-	-
AT-UF-116	FLT-17, Flotation Tailings	50.97	3.07	0.89	2.60	3.57	0.64	0.16	2.77
AT-UF-126	FLT-18, Flotation Tailings	46.39	2.71	0.82	3.82	3.62	0.35	0.22	3.05
AT-UF-128 *	FLT-19, Flotation Tailings	40.91	2.58	0.73	2.41	4.23	< 0.01	0.15	4.12
	Geochemical Testing	-	-	-	-	4.10	0.35	0.28	3.47
Average		47.21	2.82	0.83	3.20	3.62	0.52	0.14	2.96
Standard Deviation		6.87	0.33	0.12	1.17	0.52	0.37	0.04	0.62

Samples marked with (*) have QC results reported directly underneath.
(<) Indicates that the sample result was below instrument detection limits.
FLT = Flotation Tailings (slurry) collected with mechanical samplers and dewatered in laboratory.

Upper Freeport Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Methods		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-UF-067	FLT- 3, Flotation Tailings	111.7	523	4	1.6	881	19	5	124
AT-UF-036	FLT- 4, Flotation Tailings	19.3	689	4	1.5	469	22	6	127
AT-UF-051	FLT- 7, Flotation Tailings	77.4	294	4	1.8	921	19	5	127
AT-UF-087	FLT-11, Flotation Tailings	43.3	322	3	1.5	1074	17	7	97
AT-UF-104 *	FLT-13, Flotation Tailings	51.8	406	4	< 1.0	1326	14	7	114
	Iowa State University	72	373	1.6	0.08	830	8.3	-	98.7
	Huffman Laboratories	92	-	-	-	-	-	-	-
	SRI	109	275	1.56	0.11	1591	7.31	-	119
	SRI (Dup AT-UF104)	111	237	1.54	0.19	-	6.99	-	118
AT-UF-116	FLT-17, Flotation Tailings	63.1	589	3	2.0	1121	19	5	150
AT-UF-126	FLT-18, Flotation Tailings	84.1	664	3	2.2	924	20	10	148
AT-UF-128 *	FLT-19, Flotation Tailings	60.3	787	4	2.2	751	21	5	146
	Iowa State University	92	682.3	1.6	0.19	500	8.7	-	142.8
	Huffman Laboratories	132	-	-	-	-	-	-	-
Average		63.9	534	4	1.8	933	19	6	129
Standard Deviation		26.1	168	0	0.3	240	2	2	17

Test Methods		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-UF-067	FLT- 3, Flotation Tailings	17	20	0.19	135	7	34	3	70
AT-UF-036	FLT- 4, Flotation Tailings	66	39	0.04	186	8	30	3	63
AT-UF-051	FLT- 7, Flotation Tailings	75	25	0.25	151	3	33	2	58
AT-UF-087	FLT-11, Flotation Tailings	108	21	0.09	129	8	22	4	55
AT-UF-104 *	FLT-13, Flotation Tailings	19	29	0.05	108	6	31	< 2	54
	Iowa State University	52	30.34	0.29	91	-	33.1	1.5	52.7
	Huffman Laboratories	-	-	0.11	-	-	-	< 0.02	-
	SRI	29.0	25.2	0.07	63.5	-	29.4	< 2.0	41.4
	SRI (Dup AT-UF104)	-	24.7	-	64.0	-	29.4	< 2.0	41.9
AT-UF-116	FLT-17, Flotation Tailings	16	35	0.06	125	4	30	< 2	61
AT-UF-126	FLT-18, Flotation Tailings	17	37	0.07	130	11	32	2	65
AT-UF-128 *	FLT-19, Flotation Tailings	96	34	0.29	157	7	37	2	71
	Iowa State University	110	44.47	0.52	134	-	34.8	2.1	74.6
	Huffman Laboratories	-	-	0.09	-	-	-	< 0.02	-
Average		52	30	0.13	140	7	31	3	62
Standard Deviation		36	7	0.09	22	2	4	1	6

Samples marked with (*) have QC results reported directly underneath.

(<) Indicates that the sample result was below instrument detection limits.

FLT = Flotation Tailings (slurry) collected with mechanical samplers and dewatered in laboratory.

Upper Freeport Demonstration Run
System Water Analyses
All Analyses Reported On An As Received Basis
All Results In mg/L

Trace Elements

Recycle Water

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-UF-007,008	Set 1 Recycle Water	06/03/92	1700	<0.005	0.03	<0.01	<0.01	60.6	0.06	0.01
AT-UF-038,039	Set 5 Recycle Water	06/04/92	0900	0.010	0.04	<0.01	<0.01	63.2	0.36	0.02
AT-UF-071,072	Set 9 Recycle Water	06/05/92	0100	0.008	0.04	<0.01	<0.01	71.2	0.18	0.02
AT-UF-096,097	Set 13 Recycle Water	06/05/92	1700	0.005	0.05	<0.01	0.01	72.3	0.33	0.02
AT-UF-131,132	Set 19 Recycle Water	06/07/92	1000	<0.005	0.05	<0.01	0.01	70.8	0.18	0.02

Test Method				EPA 300.0	EPA 200.7	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn		
AT-UF-007,008	Set 1 Recycle Water	2.76	5.93	< 0.01	<0.0002	0.27	0.05	<0.005	0.08		
AT-UF-038,039	Set 5 Recycle Water	3.01	110.00	< 0.01	<0.0002	3.80	0.60	0.005	0.70		
AT-UF-071,072	Set 9 Recycle Water	3.53	70.00	< 0.01	<0.0002	3.34	0.27	<0.005	0.20		
AT-UF-096,097	Set 13 Recycle Water	3.58	56.64	< 0.01	<0.0002	4.59	0.14	<0.005	0.43		
AT-UF-131,132	Set 19 Recycle Water	3.66	38.20	< 0.01	<0.0002	2.33	0.28	<0.005	0.15		

Make-up and Potable Water

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-UF-129,130	Set 19 Make-up Water	06/07/92	1000	< 0.005	0.05	< 0.01	< 0.01	2.7	0.06	0.02
AT-UF-133,134	Set 19 Potable Water	06/07/92	1000	< 0.005	0.02	< 0.01	0.02	9.1	0.09	0.03

Test Method				EPA 300.0	EPA 200.7	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn		
AT-UF-129,130	Set 19 Make-up Water	0.31	0.28	< 0.01	< 0.0002	0.06	< 0.01	< 0.005	0.03		
AT-UF-133,134	Set 19 Potable Water	0.31	0.10	< 0.01	< 0.0002	< 0.01	0.08	< 0.005	0.05		

(<) Indicates that the result was below instrument detection limits.

Upper Freeport Demonstration Run
Product Filtrate Water Analyses
All Analyses Reported On An As Received Basis
All Results In mg/L

Trace Elements

Vacuum Filter Product Filtrate

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-UF-031,032 Set 4	Product Filtrate	06/03-04/92	2220-0220	<0.005	0.04	<0.01	0.01	61.2	0.21	0.02
AT-UF-135,136 Set 19	Product Filtrate	06/07/92	0800-1200	<0.005	0.05	<0.01	0.02	75.0	0.25	0.03

Test Method				EPA 300.0	EPA 200.7	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn		
AT-UF-031,032 Set 4	Product Filtrate	2.81	57.45	<0.01	<0.0002	2.68	0.48	<0.005	0.19		
AT-UF-135,136 Set 19	Product Filtrate	3.79	61.06	<0.01	<0.0002	3.25	0.44	<0.005	0.29		

Vacuum Filter Tailings Filtrate

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-UF-033,034 Set 4	Tailings Filtrate	06/03-04/92	2220-0220	<0.005	0.04	<0.01	0.02	65.5	0.32	0.02
AT-UF-137,138 Set 19	Tailings Filtrate	06/07/92	0800-1200	<0.005	0.04	<0.01	0.02	1100	0.28	0.03

Test Method				EPA 300.0	EPA 200.7	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn		
AT-UF-033,034 Set 4	Tailings Filtrate	3.53	55	<0.01	<0.0002	3.28	0.58	<0.005	0.22		
AT-UF-137,138 Set 19	Tailings Filtrate	334	100	<0.01	<0.0002	3.36	0.48	<0.005	0.47		

(<) Indicates that the result was below instrument detection limits

Upper Freeport Demonstration Run
 Diesel, Frother, Flocculant And Caustic Analyses
 All Analyses Reported On An As Received Basis
 All Results In mg/L, Unless Otherwise Noted

Trace Elements

SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co
AT-UF-148	No. 2 Diesel Fuel	01/29/92	-	1	< 1	< 1	< 1	-	< 1
AT-UF-001	Caustic, dilute (from drum)	06/03/92	1245	< 0.005	0.08	< 0.01	0.01	972.5	0.07
AT-UF-002	Flocculant, dilute (from drum)	06/03/92	1300	< 0.005	0.04	< 0.01	0.02	49.0	0.10
AT-UF-003	Frother, (from drum)	06/03/92	1300	< 0.005	< 0.01	< 0.01	0.01	1600	0.06

SCS SN	Sample Description	Cr	F	Pb	Hg	Mn	Ni	Se	Zn
AT-UF-148	No. 2 Diesel Fuel	< 1	39	3	< 0.12	< 1	2	-	4
AT-UF-001	Caustic, dilute (from drum)	0.05	< 0.01	0.65	< 0.0002	0.02	0.08	< 0.005	0.07
AT-UF-002	Flocculant, dilute (from drum)	0.03	38	0.66	< 0.0002	0.75	0.10	0.006	0.12
AT-UF-003	Frother, (from drum)	0.02	27	0.16	0.0196	< 0.01	0.06	< 0.005	0.02

Sulfur Forms					
SCS SN	Sample Description	Tot	Org	Sulf	Pyr
AT-UF-148	No. 2 Diesel Fuel	0.18	-	-	< 0.01
AT-UF-001	Caustic, dilute (from drum)	-	-	< 1	-
AT-UF-002	Flocculant, dilute (from drum)	-	-	< 1	-
AT-UF-003	Frother, (from drum)	0.09	-	< 1	-

(<) Indicates that the value is less than instrument detection limits.

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Upper Freeport Raw Coal Analyses

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	25.3	77	85.3	87
Barium	88	117.2	75.3	ND
Beryllium	2	1.6	1.55	ND
Cadmium	BD	.07	1.08	ND
Chlorine	1749	1660	2176*	ND
Cobalt	13	7.8	6.64	ND
Chromium (D3684)	12	ND	ND	ND
Chromium (D3683)	19	15.2	19.4	ND
Fluorine	45	59	35*	ND
Lead	10	13.12	11.9	ND
Mercury	.03	.26	.06	.06
Manganese	20	17.3	12.1	ND
Nickel (D3684)	17	ND	ND	ND
Nickel (D3683)	19	17.1	18.4	ND
Selenium	BD	1.2	BD	.24
Zinc	30	33.9	28.8	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Upper Freeport Feed Coal Analyses

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	15.8	23	30.8	28
Barium	66	84	67.5	ND
Beryllium	2	1.6	1.52	ND
Cadmium	BD	.06	.351	ND
Chlorine	1775	1605	2293	ND
Cobalt	8	5	4.32	ND
Chromium (D3684)	6	ND	ND	ND
Chromium (D3683)	17	13.4	16.9	ND
Fluorine	24	62.5	64.8	ND
Lead	4.7	6.5	7.45	ND
Mercury	0.09	.014	.13	.09
Manganese	23	18.6	15.4	ND
Nickel (D3684)	6	ND	ND	ND
Nickel (D3683)	14	13.5	15.3	ND
Selenium	BD	.8	BD	0.1
Zinc	33	30.8	45.9	ND

Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Upper Freeport Second Stage Flotation Product

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	6.8*	16*	21.2	18
Barium	48*	34.6*	29.5	ND
Beryllium	2*	1.5*	1.51	ND
Cadmium	BD	.04*	.062	ND
Chlorine	1900*	1925*	3818	ND
Cobalt	4*	3.1*	2.84	ND
Chromium (D3684)	9*	ND	ND	ND
Chromium (D3683)	13*	15.5*	20.7	ND
Fluorine	35*	89*	BD	ND
Lead	3*	3.4*	4.61	ND
Mercury	0.08*	.08*	BD	.06
Manganese	9*	6.9*	5.62	ND
Nickel (D3684)	10*	ND	ND	ND
Nickel (D3683)	9*	10.4*	11.7	ND
Selenium	BD	.6*	BD	.03
Zinc	16*	13.8*	12.8	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Upper Freeport Combined Flotation Tailings

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	63.9*	67*	110*	18
Barium	534*	528*	256*	ND
Beryllium	4*	1.6*	1.55*	ND
Cadmium	1.8*	.14*	0.15*	ND
Chlorine	933*	665*	1591	ND
Cobalt	19*	8.5*	7.2*	ND
Chromium (D3684)	6*	ND	ND	ND
Chromium (D3683)	129*	121*	119*	ND
Fluorine	52*	81*	29.0	ND
Lead	30*	37.4*	25*	ND
Mercury	0.13*	.41*	.07	.06
Manganese	140*	113*	64*	ND
Nickel (D3684)	7*	ND	ND	ND
Nickel (D3683)	31*	34*	29.4	ND
Selenium	3*	1.8*	BD	.03
Zinc	62*	63.7*	41.7*	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

Pittsburgh No. 8

Pittsburgh No. 8 Demonstration Run
Raw and Feed Coal Analyses
All Analyses Reported On A Dry Basis
Results % by weight unless otherwise noted

Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Moist	Ash	Vol	FC	BTU/lb
AT-P8-000 *	APCO, First Split	4.84	30.88	30.26	38.86	10167
	APCO, Second Split	4.31	31.61	-	-	10018
	Geochemical Testing, APCO's Second	1.48	30.73	-	-	-
	Geochemical Testing, Third Split	4.42	32.67	-	-	9783
	APCO, Geochemical's Third Split	-	32.58	-	-	9787
	Southern Research Institute	-	32.3	-	-	-
Average		-	31.80	-	-	9939
Standard Deviation		-	0.78	-	-	163
AT-P8-135	Commercial Testing & Engineering	4.12	27.59	-	-	10744
	Geochemical Testing	4.12	32.21	-	-	-
	APCO	-	31.65	-	-	9981
Average		-	30.48	-	-	10363
Standard Deviation		-	2.06	-	-	382

						Sulfur Forms			
SCS SN	Sample Description	C	H	N	O	Tot	Org	Sulf	Pyr
AT-P8-000 *	APCO, First Split	57.91	3.67	1.11	4.27	(2.16)	(1.36)	(0.02)	(0.78)
	APCO, Second Split	-	-	-	-	(1.99)	-	-	(0.93)
	Geochemical Testing, APCO's Second	-	-	-	-	2.15	0.84	0.08	1.23
	Geochemical Testing, Third Split	-	-	-	-	2.09	0.64	0.11	1.34
	APCO, Geochemical's Third Split	-	-	-	-	2.21	1.10	0.10	1.01
	Southern Research Institute	-	-	-	-	-	-	-	-
Average		-	-	-	-	2.15	0.86	0.10	1.19
Standard Deviation		-	-	-	-	0.05	0.19	0.01	0.14
AT-P8-135	Commercial Testing & Engineering	-	-	-	-	2.26	0.95	0.01	1.30
	Geochemical Testing	-	-	-	-	2.08	0.68	0.04	1.36
	APCO	-	-	-	-	2.24	1.03	0.10	1.11
Average		-	-	-	-	2.19	0.89	0.05	1.26
Standard Deviation		-	-	-	-	0.08	0.15	0.04	0.11

Pittsburgh No. 8 Demonstration Run
Feed Coal Analyses - APCO Laboratory
All Analyses Reported On A Dry Basis
Results % by weight unless otherwise noted
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Moist	Ash	Vol	FC	BTU/lb
AT-P8-001	Feed, CDSB # 1 & # 2	4.47	7.27	38.86	53.87	14117
AT-P8-020	Feed, CDSB # 3 & # 4	4.70	6.67	39.58	53.75	14128
AT-P8-030	Feed, CDSB # 5 & # 6	5.40	6.79	39.95	53.26	14082
AT-P8-042	Feed, CDSB # 7 & # 8	5.05	7.19	39.71	53.10	14041
AT-P8-052	Feed, CDSB # 9 & #10	5.36	6.68	39.24	54.08	14111
AT-P8-065	Feed, CDSB #11 & #12	5.57	6.49	40.74	52.77	14169
AT-P8-069	Feed, CDSB #13 & #14	5.02	7.08	39.74	53.18	14104
AT-P8-085	Feed, CDSB #15 & #16	5.35	6.95	38.62	54.43	14128
AT-P8-095	Feed, CDSB #17 & #18	5.88	6.99	38.16	54.85	14114
AT-P8-104	Feed, CDSB #19 & #20	5.54	7.64	38.52	53.84	13991
AT-P8-118	Feed, CDSB #21 & #22	4.41	6.93	38.33	54.74	14101
	SRI	-	7.19	-	-	-
AT-P8-126	Feed, CDSB #23 & #24	5.06	7.30	38.73	53.97	14096
Average		5.15	7.00	39.18	53.82	14099
Standard Deviation		0.43	0.31	0.74	0.63	43

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-P8-001	Feed, CDSB # 1 & # 2	78.55	5.13	1.47	5.59	1.99	1.32	0.02	0.65
AT-P8-020	Feed, CDSB # 3 & # 4	79.30	5.25	1.46	5.28	2.04	1.41	0.02	0.61
AT-P8-030	Feed, CDSB # 5 & # 6	79.11	4.98	1.47	5.60	2.05	1.40	0.02	0.63
AT-P8-042	Feed, CDSB # 7 & # 8	78.99	4.93	1.47	5.28	2.14	1.28	0.02	0.84
AT-P8-052	Feed, CDSB # 9 & #10	79.49	4.89	1.49	5.43	2.02	1.29	0.02	0.71
AT-P8-065	Feed, CDSB #11 & #12	79.73	4.94	1.48	5.34	2.02	1.24	0.02	0.76
AT-P8-069	Feed, CDSB #13 & #14	79.24	5.16	1.46	4.95	2.11	1.34	0.02	0.75
AT-P8-085	Feed, CDSB #15 & #16	79.05	5.24	1.47	5.30	1.99	1.22	0.02	0.75
AT-P8-095	Feed, CDSB #17 & #18	79.97	5.03	1.49	4.47	2.05	1.24	0.02	0.79
AT-P8-104	Feed, CDSB #19 & #20	78.49	5.08	1.46	5.09	2.24	1.38	0.03	0.83
AT-P8-118	Feed, CDSB #21 & #22	79.24	5.12	1.46	5.18	2.07	1.26	0.02	0.79
	SRI	-	-	-	-	-	-	-	-
AT-P8-126	Feed, CDSB #23 & #24	78.63	5.18	1.51	5.24	2.14	1.29	0.02	0.83
Average		79.15	5.08	1.47	5.23	2.07	1.31	0.02	0.75
Standard Deviation		0.43	0.12	0.01	0.29	0.07	0.06	0.00	0.08

CDSB = Crusher Discharge Stop-Belt Feed Coal Samples.

Pittsburgh No. 8 Demonstration Run

Feed Coal Analyses - Geochemical Testing (QA/QC)

All Analyses Reported On A Dry Basis

Results % by weight unless otherwise noted

Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Moist	Ash	Vol	FC	BTU/lb
AT-P8-001	Feed, CDSB # 1 & # 2	3.86	7.00	-	-	-
AT-P8-020	Feed, CDSB # 3 & # 4	6.92	6.90	-	-	-
AT-P8-030	Feed, CDSB # 5 & # 6	4.03	6.94	-	-	-
AT-P8-042	Feed, CDSB # 7 & # 8	4.47	7.49	-	-	13986
AT-P8-052	Feed, CDSB # 9 & # 10	4.82	7.12	-	-	-
AT-P8-065	Feed, CDSB # 11 & # 12	5.29	6.77	-	-	-
AT-P8-069	Feed, CDSB # 13 & # 14	4.43	7.18	-	-	-
AT-P8-085	Feed, CDSB # 15 & # 16	4.47	7.16	-	-	-
AT-P8-095	Feed, CDSB # 17 & # 18	4.47	6.95	-	-	-
AT-P8-104	Feed, CDSB # 19 & # 20	4.12	7.68	-	-	-
AT-P8-118	Feed, CDSB # 21 & # 22	3.58	7.19	-	-	14035
	Southern Research Institute	-	7.19	-	-	-
AT-P8-126	Feed, CDSB # 23 & # 24	4.02	7.38	-	-	-
Average		4.54	7.15	0.00	0.00	2335
Standard Deviation		0.84	0.25	0.00	0.00	5221

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-P8-001	Feed, CDSB # 1 & # 2	-	-	-	-	1.91	1.02	0.11	0.78
AT-P8-020	Feed, CDSB # 3 & # 4	-	-	-	-	1.95	1.05	0.14	0.76
AT-P8-030	Feed, CDSB # 5 & # 6	-	-	-	-	1.98	1.09	0.14	0.75
AT-P8-042	Feed, CDSB # 7 & # 8	-	-	-	-	2.00	0.93	0.09	0.98
AT-P8-052	Feed, CDSB # 9 & # 10	-	-	-	-	1.92	1.04	0.06	0.82
AT-P8-065	Feed, CDSB # 11 & # 12	-	-	-	-	2.01	1.15	0.07	0.79
AT-P8-069	Feed, CDSB # 13 & # 14	-	-	-	-	2.07	1.13	0.09	0.85
AT-P8-085	Feed, CDSB # 15 & # 16	-	-	-	-	2.01	1.02	0.06	0.93
AT-P8-095	Feed, CDSB # 17 & # 18	-	-	-	-	1.94	1.04	0.09	0.81
AT-P8-104	Feed, CDSB # 19 & # 20	-	-	-	-	2.22	1.10	0.18	0.94
AT-P8-118	Feed, CDSB # 21 & # 22	-	-	-	-	2.02	1.01	0.10	0.91
	Southern Research Institute	-	-	-	-	-	-	-	-
AT-P8-126	Feed, CDSB # 23 & # 24	-	-	-	-	2.09	1.04	0.18	0.87
Average		0.00	0.00	0.00	0.00	2.01	1.05	0.11	0.85
Standard Deviation		0.00	0.00	0.00	0.00	0.08	0.06	0.04	0.07

CDSB = Crusher Discharge Stop-Belt Feed Coal Samples.

Pittsburgh No. 8 Demonstration Run
Raw and Feed Coal Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Method		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-P8-000 *	Pittsburgh No. 8 Raw Co	6.9	140	2	< 1.0	688	10	9	41
	Iowa State University	8.9	121.6	1.6	0.12	953	5.7	-	38.6
	Huffman Laboratories	9	-	-	-	-	-	-	-
	SRI	35.2	109	1.20	0.251	1050	4.97	-	58.4
AT-P8-001 #	Feed, CDSB # 1 & # 2	-	-	-	-	-	-	-	-
AT-P8-020	Feed, CDSB # 3 & # 4	< 1.0	43	3	< 1.0	919	3	5	12
AT-P8-030	Feed, CDSB # 5 & # 6	1.9	49	3	< 1.0	834	3	11	12
AT-P8-042 *	Feed, CDSB # 7 & # 8	2.8	48	3	< 1.0	999	3	8	13
	Iowa State University	5.2	43.5	1.0	0.04	961	3.4	-	11.0
	Huffman Laboratories	4	-	-	-	-	-	-	-
AT-P8-052	Feed, CDSB # 9 & # 10	1.6	41	2	< 1.0	915	3	3	11
AT-P8-065	Feed, CDSB # 11 & # 12	1.3	46	3	< 1.0	893	3	3	10
AT-P8-069	Feed, CDSB # 13 & # 14	< 1.0	47	3	< 1.0	905	3	2	11
AT-P8-085	Feed, CDSB # 15 & # 16	< 1.0	48	3	< 1.0	910	3	7	12
AT-P8-095	Feed, CDSB # 17 & # 18	< 1.0	46	3	< 1.0	894	3	11	11
AT-P8-104	Feed, CDSB # 19 & # 20	< 1.0	50	3	< 1.0	866	3	11	13
AT-P8-118 *	Feed, CDSB # 21 & # 22	1.6	46	3	< 1.0	951	3	2	11
	Iowa State University	5.1	40.7	1.0	0.08	1110	2.6	-	10.0
	Huffman Laboratories	4	-	-	-	-	-	-	-
	SRI	12.5	36.3	0.83	0.048	1674	2.30	-	161
AT-P8-126 #	Feed, CDSB # 23 & # 24	-	-	-	-	-	-	-	-
	Average	1.8	46	3	< 1.0	909	3	6	12
	Standard Deviation	0.5	3	0	0	42	0	4	1

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-P8-000 *	Pittsburgh No. 8 Raw Co	17	9	0.10	35	8	15	< 2	31
	Iowa State University	101	17.23	0.09	34.4	-	16.2	2.7	28.2
	Huffman Laboratories	-	-	0.04	-	-	-	0.33	-
	SRI	48.8	6.29	0.04	26.2	-	19.9	< 2	24.7
AT-P8-001 #	Feed, CDSB # 1 & # 2	-	-	-	-	-	-	-	-
AT-P8-020	Feed, CDSB # 3 & # 4	36	3	0.04	11	4	10	< 2	7
AT-P8-030	Feed, CDSB # 5 & # 6	59	4	0.07	11	21	10	< 2	8
AT-P8-042 *	Feed, CDSB # 7 & # 8	65	3	0.10	12	13	11	< 2	9
	Iowa State University	101	2.32	0.02	10.2	-	7.3	1.6	9.8
	Huffman Laboratories	-	-	0.05	-	-	-	0.07	-
AT-P8-052	Feed, CDSB # 9 & # 10	55	3	0.10	11	11	9	< 2	7
AT-P8-065	Feed, CDSB # 11 & # 12	62	3	0.07	11	15	9	< 2	8
AT-P8-069	Feed, CDSB # 13 & # 14	224	3	0.08	12	15	10	< 2	8
AT-P8-085	Feed, CDSB # 15 & # 16	224	3	0.08	11	14	10	< 2	8
AT-P8-095	Feed, CDSB # 17 & # 18	89	3	0.07	11	5	9	< 2	10
AT-P8-104	Feed, CDSB # 19 & # 20	49	3	0.06	13	6	11	< 2	9
AT-P8-118 *	Feed, CDSB # 21 & # 22	88	3	0.10	11	5	9	< 2	8
	Iowa State University	40	2.97	0.08	10.3	-	6.9	1.6	9.0
	Huffman Laboratories	-	-	0.05	-	-	-	< 0.02	-
	SRI	< 25	1.94	< 0.03	7.00	-	8.42	< 2	7.78
AT-P8-126 #	Feed, CDSB # 23 & # 24	-	-	-	-	-	-	-	-
	Average	95	3	0.06	11	11	10	< 2	8
	Standard Deviation	66	0	0.02	1	5	1	0	1

Samples marked with (*) are samples that have QC analyses directly underneath.
Samples marked with (#) indicate samples not tested for trace elements.
(<) Indicates result was below instrument detection limits.
CDSB = Crusher Discharge Stop-Belt Feed Coal SampleS.

Pittsburgh No. 8 Demonstration Run
 Second Stage Flotation Product Analyses
 All Analyses Reported On A Dry Basis
 Results % By Weight Unless Otherwise Noted
 Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-P8-018	F2PR- 2, Coal Product	5.27	38.18	56.55	14283
AT-P8-022 *	F2PR- 3, Coal Product	4.94	38.21	56.85	14334
	Geochemical Testing	4.84	-	-	14296
AT-P8-035	F2PR- 5, Coal Product	4.57	38.61	56.82	14467
AT-P8-044	F2PR- 7, Coal Product	5.24	38.20	56.56	14344
AT-P8-076	F2PR-12, Coal Product	5.36	38.53	56.11	14383
AT-P8-079	F2PR-13, Coal Product	5.20	39.00	55.80	14424
AT-P8-097	F2PR-15, Coal Product	5.10	38.08	56.82	14415
AT-P8-102	F2PR-16, Coal Product	5.07	39.54	55.39	14428
AT-P8-124 *	F2PR-19, Coal Product	5.08	38.05	56.87	14328
	Geochemical Testing	5.04	-	-	14341
	SRI	5.12	-	-	-
	SRI (Dup AT-P8-124)	5.07	-	-	-
AT-P8-128	F2PR-20, Coal Product	4.60	38.88	56.52	14456
	Average	5.04	38.53	56.43	14386.20
	Standard Deviation	0.26	0.46	0.48	58.27

SCS SN	Sample Description	C	H	N	O	Tot	Sulfur Forms		
							Org	Sulf	Pyr
AT-P8-018	F2PR- 2, Coal Product	80.55	5.40	1.49	5.55	1.74	1.05	0.06	0.63
AT-P8-022 *	F2PR- 3, Coal Product	80.44	5.35	1.53	6.03	1.71	1.06	0.06	0.59
	Geochemical Testing	-	-	-	-	1.71	0.99	0.07	0.65
AT-P8-035	F2PR- 5, Coal Product	81.45	5.39	1.53	5.45	1.61	1.08	0.05	0.48
AT-P8-044	F2PR- 7, Coal Product	81.28	5.36	1.54	4.90	1.68	1.07	0.04	0.57
AT-P8-076	F2PR-12, Coal Product	81.15	5.38	1.49	4.80	1.82	1.13	0.05	0.64
AT-P8-079	F2PR-13, Coal Product	81.06	5.35	1.52	5.04	1.83	1.17	0.03	0.63
AT-P8-097	F2PR-15, Coal Product	81.28	5.41	1.54	4.84	1.83	1.15	0.05	0.63
AT-P8-102	F2PR-16, Coal Product	81.32	5.30	1.49	4.95	1.87	1.20	0.04	0.63
AT-P8-124 *	F2PR-19, Coal Product	80.75	5.35	1.58	5.43	1.81	1.13	0.05	0.63
	Geochemical Testing	-	-	-	-	1.74	1.01	0.06	0.67
	SRI	-	-	-	-	-	-	-	-
	SRI (Dup AT-P8-124)	-	-	-	-	-	-	-	-
AT-P8-128	F2PR-20, Coal Product	81.19	5.39	1.55	5.50	1.77	1.16	0.04	0.57
	Average	81.05	5.37	1.53	5.25	1.77	1.12	0.05	0.60
	Standard Deviation	0.33	0.03	0.03	0.38	0.08	0.05	0.01	0.05

Samples marked with (*) are samples that have QC analyses directly underneath.

F2PR = Second Stage Flotation Product collected by mechanical sampler and dewatered in laboratory.

Pittsburgh No. 8 Demonstration Run
 Second Stage Flotation Product Samples
 All Analyses Reported On A Dry Basis
 All Results Reported In mg/kg

Trace Elements

Test Method		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-P8-018	F2PR- 2, Coal Product	2.4	41	2	< 1.0	966	4	9	13
AT-P8-022 *	F2PR- 3, Coal Product	1.6	41	2	< 1.0	772	2	9	12
	Iowa State University	3.4	36.7	1.0	0.06	1061	2.6	-	11.1
	Huffman Laboratories	3	-	-	-	-	-	-	-
AT-P8-035	F2PR- 5, Coal Product	2.1	40	2	< 1.0	975	2	9	13
AT-P8-044	F2PR- 7, Coal Product	< 1.0	39	2	< 1.0	1053	2	16	13
AT-P8-076	F2PR-12, Coal Product	1.1	42	2	< 1.0	1063	2	9	13
AT-P8-079	F2PR-13, Coal Product	1.5	48	3	< 1.0	789	2	10	13
AT-P8-097	F2PR-15, Coal Product	< 1.0	46	3	< 1.0	851	2	23	13
AT-P8-102	F2PR-16, Coal Product	1.0	42	3	< 1.0	928	3	29	13
AT-P8-124 *	F2PR-19, Coal Product	1.7	41	3	< 1.0	994	2	7	13
	Iowa State University	3.8	40.3	1.1	0.04	870	2.5	-	12.0
	Huffman Laboratories	3	-	-	-	-	-	-	-
	SRI	9.49	34.7	0.84	0.043	1712	2.11	-	19.2
	SRI (Dup AT-P8-124)	8.43	34.2	0.83	0.063	1268	2.07	-	17.2
AT-P8-128	F2PR-20, Coal Product	1.5	41	2	< 1.0	820	2	24	12
	Average	1.6	42	2	< 1.0	921	2	15	13
	Standard Deviation	0.4	3	0	0	101	1	8	0

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-P8-018	F2PR- 2, Coal Product	55	< 1	< 0.02	14	3	9	3	6
AT-P8-022 *	F2PR- 3, Coal Product	54	< 1	< 0.02	12	3	8	3	6
	Iowa State University	84	1.45	0.05	10.0	-	6.6	1.3	6.6
	Huffman Laboratories	-	-	0.05	-	-	-	0.03	-
AT-P8-035	F2PR- 5, Coal Product	52	2	< 0.02	12	3	8	< 2	7
AT-P8-044	F2PR- 7, Coal Product	56	1	< 0.02	14	2	9	< 2	6
AT-P8-076	F2PR-12, Coal Product	92	1	0.04	14	3	8	< 2	7
AT-P8-079	F2PR-13, Coal Product	270	< 1	0.04	17	3	8	3	7
AT-P8-097	F2PR-15, Coal Product	133	1	0.03	16	2	9	< 2	7
AT-P8-102	F2PR-16, Coal Product	72	< 1	0.04	15	6	8	< 2	7
AT-P8-124 *	F2PR-19, Coal Product	78	1	0.04	15	2	8	< 2	7
	Iowa State University	80	3.14	< 0.01	0.5	-	7.3	1.3	7.4
	Huffman Laboratories	-	-	0.06	-	-	-	< 0.02	-
	SRI	< 25	6.56	< 0.03	7.54	-	6.82	< 2	6.26
	SRI (Dup AT-P8-124)	< 25	1.76	< 0.03	7.52	-	26.9	< 2	6.44
AT-P8-128	F2PR-20, Coal Product	73	< 1	0.07	13	3	8	3	7
	Average	94	1	0.04	14	3	8	3	7
	Standard Deviation	63	0	0.01	2	1	0	0	0

Samples marked with (*) are samples that have QC analyses directly underneath.

(<) Indicates result below instrument detection limits.

F2PR = Second Stage Flotation Product collected by mechanical sampler and dewatered in laboratory.

Pittsburgh No. 8 Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
Results % By Weight Unless Otherwise Noted
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-P8-017	FLT- 2, Tailings Product	27.18	30.41	42.41	10736
AT-P8-021 *	FLT- 3, Tailings Product	28.10	29.63	42.27	10566
	Geochemical Testing	27.69	-	-	10468
AT-P8-034	FLT- 5, Tailings Product	33.74	28.39	37.87	9817
AT-P8-043	FLT- 7, Tailings Product	41.85	25.99	32.16	8359
AT-P8-075	FLT-12, Tailings Product	31.74	29.66	38.60	10086
AT-P8-078	FLT-13, Tailings Product	32.80	28.94	38.26	10006
AT-P8-096	FLT-15, Tailings Product	30.41	29.67	39.92	10219
AT-P8-105	FLT-16, Tailings Product	38.46	26.90	34.64	8746
AT-P8-123 *	FLT-19, Tailings Product	33.71	28.59	37.70	9841
	Geochemical Testing	33.21	-	-	9697
	SRI	33.5	-	-	-
AT-P8-127	FLT-20, Tailings Product	28.65	30.45	40.90	10571
Average		32.66	28.86	38.47	9895
Standard Deviation		4.40	1.39	3.06	739

SCS SN	Sample Description	C	H	N	O	Tot	Sulfur Forms		
							Org	Sulf	Pyr
AT-P8-017	FLT- 2, Tailings Product	60.17	4.05	1.11	2.50	4.99	1.97	0.06	2.96
AT-P8-021 *	FLT- 3, Tailings Product	59.25	3.99	1.11	2.70	4.85	1.68	0.08	3.09
	Geochemical Testing	-	-	-	-	3.93	0.74	0.11	3.08
AT-P8-034	FLT- 5, Tailings Product	54.07	3.46	1.00	2.85	4.88	1.15	0.08	3.65
AT-P8-043	FLT- 7, Tailings Product	46.12	2.88	0.85	2.51	5.79	1.01	0.08	4.70
AT-P8-075	FLT-12, Tailings Product	56.53	3.53	1.04	2.51	4.65	1.23	0.05	3.37
AT-P8-078	FLT-13, Tailings Product	55.16	3.57	1.04	2.66	4.77	1.26	0.04	3.47
AT-P8-096	FLT-15, Tailings Product	57.17	3.66	1.07	3.02	4.67	1.31	0.05	3.31
AT-P8-105	FLT-16, Tailings Product	49.06	2.94	0.92	1.67	6.95	2.56	0.09	4.30
AT-P8-123 *	FLT-19, Tailings Product	54.01	3.02	1.00	3.21	5.05	1.43	0.07	3.55
	Geochemical Testing	-	-	-	-	4.95	0.65	0.13	4.17
	SRI	-	-	-	-	-	-	-	-
AT-P8-127	FLT-20, Tailings Product	58.94	3.48	1.10	3.40	4.43	1.35	0.07	3.01
Average		55.05	3.46	1.02	2.70	5.10	1.50	0.07	3.54
Standard Deviation		4.28	0.39	0.08	0.45	0.70	0.44	0.02	0.53

Samples marked with (*) have QC results reported directly underneath.
FLT = Flotation Tailings collected by mechanical sampler and dewatered in laboratory.

Pittsburgh No. 8 Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Method		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-P8-017	FLT- 2, Tailings Product	7.6	101	10	2.1	621	7	34	89
AT-P8-021 *	FLT- 3, Tailings Product	11.4	119	11	2.5	560	7	23	110
	Iowa State University	16	98.6	1.1	0.42	830	7.5	-	98.6
	Huffman Laboratories	17	-	-	-	-	-	-	-
AT-P8-034	FLT- 5, Tailings Product	16.8	133	13	2.7	565	8	18	116
AT-P8-043	FLT- 7, Tailings Product	31.1	160	16	2.9	559	9	21	127
AT-P8-075	FLT-12, Tailings Product	13.9	122	12	2.2	547	7	19	114
AT-P8-078	FLT-13, Tailings Product	9.2	131	13	2.6	550	8	19	116
AT-P8-096	FLT-15, Tailings Product	14.6	124	12	2.1	603	7	15	113
AT-P8-105	FLT-16, Tailings Product	7.8	152	16	2.7	442	9	18	136
AT-P8-123 *	FLT-19, Tailings Product	20.8	131	15	2.4	490	8	17	137
	Iowa State University	22	108.4	1.1	0.17	500	8.6	-	141.9
	Huffman Laboratories	22	-	-	-	-	-	-	-
	SRI	61.6	96.4	0.84	0.148	774	7.38	-	161
AT-P8-127	FLT-20, Tailings Product	12.1	113	12	2.0	562	7	16	125
Average		14.5	129	13	2.4	550	8	20	118
Standard Deviation		6.8	17	2	0.3	49	1	5	13

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-P8-017	FLT- 2, Tailings Product	88	9	0.13	146	33	40	5	30
AT-P8-021 *	FLT- 3, Tailings Product	166	10	0.12	185	15	44	6	33
	Iowa State University	110	6.65	0.20	159.1	-	34.8	3.8	30.6
	Huffman Laboratories	-	-	0.03	-	-	-	0.06	-
AT-P8-034	FLT- 5, Tailings Product	180	12	0.12	196	5	52	5	38
AT-P8-043	FLT- 7, Tailings Product	138	13	0.15	224	5	58	7	44
AT-P8-075	FLT-12, Tailings Product	970	11	0.18	192	3	48	7	34
AT-P8-078	FLT-13, Tailings Product	1396	10	0.16	208	4	51	5	35
AT-P8-096	FLT-15, Tailings Product	458	10	0.09	195	4	49	8	33
AT-P8-105	FLT-16, Tailings Product	243	14	0.18	243	3	60	6	39
AT-P8-123 *	FLT-19, Tailings Product	208	11	0.15	230	5	51	7	36
	Iowa State University	212	13.00	0.28	232.3	-	43.2	4.5	38.2
	Huffman Laboratories	-	-	0.07	-	-	-	0.04	-
	SRI	73.4	7.73	0.07	176	-	64.9	< 2	31.0
AT-P8-127	FLT-20, Tailings Product	261	9	0.17	203	4	50	6	31
Average		411	11	0.15	202	8	50	6	35
Standard Deviation		409	2	0.03	26	9	6	1	4

Samples marked with (*) have QC results reported directly underneath.
FLT = Flotation Tailings collected by mechanical sampler and dewatered in laboratory.

Pittsburgh No. 8 Demonstration Run
 Vacuum Filter Product Analyses
 All Analyses Reported On A Dry Basis
 Results % By Weight Unless Otherwise Noted
 Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-P8-023	VFPR- 3, Product Coal	5.06	38.78	56.16	14419
AT-P8-045	VFPR- 7, Product Coal	5.51	38.27	56.22	14396
AT-P8-080	VFPR-13, Product Coal	5.34	39.20	56.46	14310
AT-P8-129	VFPR-20, Product Coal	4.53	38.51	56.96	14482
Average		5.11	38.69	56.45	14402
Standard Deviation		0.37	0.35	0.32	62

SCS SN	Sample Description	C	H	N	O	Sulfur Forms			
						Tot	Org	Sulf	Pyr
AT-P8-023	VFPR- 3, Product Coal	81.59	4.26	1.52	5.82	1.75	1.07	0.03	0.65
AT-P8-045	VFPR- 7, Product Coal	81.16	4.54	1.51	5.61	1.67	1.04	0.04	0.59
AT-P8-080	VFPR-13, Product Coal	81.35	4.39	1.51	5.63	1.78	1.13	0.02	0.63
AT-P8-129	VFPR-20, Product Coal	81.47	5.48	1.52	5.27	1.73	1.14	0.03	0.56
Average		81.39	4.67	1.52	5.58	1.73	1.10	0.03	0.61
Standard Deviation		0.16	0.48	0.00	0.20	0.04	0.04	0.01	0.03

VFPR = Vacuum Filter Product collected from rotary vacuum drum system in plant.

Pittsburgh No. 8 Demonstration Run
 Vacuum Filter Product Samples
 All Analyses Reported On A Dry Basis
 All Results Reported In mg/kg

Trace Elements

Test Method		ASTM D4606	SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-P8-023	VFPR- 3, Product Coal	1.1	39	2	< 1.0	948	2	11	12
AT-P8-045	VFPR- 7, Product Coal	5.1	39	2	< 1.0	729	2	13	14
AT-P8-080	VFPR-13, Product Coal	< 1.0	42	2	< 1.0	940	2	9	13
AT-P8-129	VFPR-20, Product Coal	1.1	40	2	< 1.0	899	4	9	12
Average		2.4	40	2	< 1.0	879	3	11	13
Standard Deviation		1.9	1	0	0	89	1	2	1

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-P8-023	VFPR- 3, Product Coal	70	3	0.07	12	5	8	< 2	6
AT-P8-045	VFPR- 7, Product Coal	71	1	0.06	14	6	9	2	7
AT-P8-080	VFPR-13, Product Coal	155	< 1	0.03	14	8	8	< 2	6
AT-P8-129	VFPR-20, Product Coal	90	2	< 0.02	10	6	8	2	6
Average		97	2	0.05	13	6	8	2	6
Standard Deviation		35	1	0.02	2	1	0	0	0

(<) Indicates result below instrument detection limits.

VFPR = Vacuum Filter Product collected from rotary vacuum drum system in plant.

Pittsburgh No. 8 Demonstration Run
System Water Analyses
All Analyses Reported On An As Received Basis
All Results in mg/L

Trace Elements

Recycle Water

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-P8-013,014	CLWT- 2, Recycle Water	07/27/92	2100	< 0.005	0.05	< 0.01	< 0.01	92.0	< 0.01	< 0.01
AT-P8-037,038	CLWT- 5, Recycle Water	07/28/92	1300	< 0.005	0.05	< 0.01	< 0.01	80.6	< 0.01	< 0.01
AT-P8-050,051	CLWT- 7, Recycle Water	07/28/92	2100	< 0.005	0.06	< 0.01	< 0.01	80.6	< 0.01	< 0.01
AT-P8-063,064	CLWT- 9, Recycle Water	07/29/92	0500	< 0.005	0.06	< 0.01	< 0.01	84.7	< 0.01	< 0.01
AT-P8-073,074	CLWT-12, Recycle Water	07/29/92	2100	< 0.005	0.04	< 0.01	< 0.01	100.0	< 0.01	< 0.01
AT-P8-099,100	CLWT-16, Recycle Water	07/30/92	1300	< 0.005	0.05	< 0.01	< 0.01	76.4	< 0.01	< 0.01
AT-P8-130,131	CLWT-20, Recycle Water	07/31/92	0900	< 0.005	0.06	< 0.01	< 0.01	87.9	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-P8-013,014	CLWT- 2, Recycle Water	1.48	2.03	< 0.01	< 0.0002	0.15	< 0.02	0.005	0.02
AT-P8-037,038	CLWT- 5, Recycle Water	1.69	2.52	< 0.01	< 0.0002	0.12	< 0.02	0.005	0.02
AT-P8-050,051	CLWT- 7, Recycle Water	1.53	1.67	< 0.01	< 0.0002	0.12	< 0.02	0.005	0.02
AT-P8-063,064	CLWT- 9, Recycle Water	1.74	3.32	< 0.01	0.0002	0.15	< 0.02	0.005	0.02
AT-P8-073,074	CLWT-12, Recycle Water	2.52	< 0.01	< 0.01	< 0.0002	0.01	< 0.02	0.005	0.02
AT-P8-099,100	CLWT-16, Recycle Water	2.73	2.03	< 0.01	< 0.0002	0.15	< 0.02	0.005	0.03
AT-P8-130,131	CLWT-20, Recycle Water	2.52	2.75	< 0.01	< 0.0002	0.14	< 0.02	0.005	0.02

CLWT = Clarified water used for recycle water

Makeup/Potable Water

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-P8-048,049	Set-7, Makeup Water	07/28/92	2100	< 0.005	0.04	< 0.01	< 0.01	41.7	< 0.01	< 0.01
AT-P8-046,047	Set-7, Potable Water	07/28/92	2100	< 0.005	0.01	< 0.01	< 0.01	96.5	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-P8-048,049	Set-7, Makeup Water	0.24	-	0.02	< 0.0002	0.04	< 0.02	< 0.005	0.01
AT-P8-046,047	Set-7, Potable Water	0.17	-	0.02	< 0.0002	0.01	< 0.02	< 0.005	0.01

(<) Indicates that the result is below instrument detection limits

Pittsburgh No. 8 Demonstration Run
System Water Analyses
All Analyses Reported On An As Received Basis
All Results In mg/L

Trace Elements

Vacuum Filter Product Filtrate

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co
AT-P8-024,025	VFFLP- 3, Product Filtrate	07/28/92	0030-0430	< 0.005	0.05	< 0.01	< 0.01	79.2	< 0.01
AT-P8-081,082	VFFLP-13, Product Filtrate	07/29-30/92	2330-0330	< 0.005	0.04	< 0.01	< 0.01	91.7	< 0.01
AT-P8-119,120	VFFLP-18, Product Filtrate	07/30-31/92	2300-0300	< 0.005	0.04	< 0.01	< 0.01	86.1	< 0.01

Test Method				EPA 200.7	EPA 300.0	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	Cr	F	Pb	Hg	Mn	Ni	Se	Zn		
AT-P8-024,025	VFFLP- 3, Product Filtrate	< 0.01	1.69	< 0.01	< 0.0002	0.14	< 0.02	0.005	0.02		
AT-P8-081,082	VFFLP-13, Product Filtrate	< 0.01	3.25	< 0.01	< 0.0002	0.12	< 0.02	0.005	0.02		
AT-P8-119,120	VFFLP-18, Product Filtrate	< 0.01	2.37	< 0.01	< 0.0002	0.10	< 0.02	0.005	0.02		

Vacuum Filter Tailings Filtrate

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co
AT-P8-026,027	VFFLT- 3, Tailings Filtrate	07/28/92	0030-0430	< 0.005	0.05	< 0.01	< 0.01	73.4	< 0.01
AT-P8-083,084	VFFLT-13, Tailings Filtrate	07/29-30/92	2330-0330	< 0.005	0.04	< 0.01	< 0.01	90.3	< 0.01
AT-P8-121,122	VFFLT-18, Tailings Filtrate	07/30-31/92	2300-0300	< 0.005	0.04	< 0.01	< 0.01	91.0	< 0.01

Test Method				EPA 200.7	EPA 300.0	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	Cr	F	Pb	Hg	Mn	Ni	Se	Zn		
AT-P8-026,027	VFFLT- 3, Tailings Filtrate	< 0.01	1.32	< 0.01	< 0.0002	0.11	< 0.02	0.005	0.02		
AT-P8-083,084	VFFLT-13, Tailings Filtrate	< 0.01	3.10	0.02	< 0.0002	0.09	< 0.02	0.005	0.01		
AT-P8-121,122	VFFLT-18, Tailings Filtrate	< 0.01	2.63	< 0.01	< 0.0002	0.08	< 0.02	0.005	0.02		

(<) Indicates that the result is below instrument detection limits

Pittsburgh No. 8 Demonstration Run
 Diesel, Caustic, Flocculant and Frother Analyses
 All Analyses Reported On An As Received Basis
 All Results in mg/L, Unless Otherwise Noted

Trace Elements

SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co
AT-PB-005	No. 2 Diesel Oil, from tank	07/27/92	2000	< 1.00	< 1.00	< 1.00	< 1.00	< 150.0	< 1.00
AT-UF-001	Caustic, dilute (from drum)*	06/03/92	1245	< 0.005	0.08	< 0.01	0.01	972.5	0.07
AT-PB-007	Flocculant, from drum	07/27/92	2000	0.10	1.90	< 0.04	< 0.05	18.1	0.10
AT-PB-008	Frother, from drum	07/27/92	2000	0.32	5.80	< 0.04	0.06	13.6	0.15

SCS SN	Sample Description	Cr	F	Pb	Hg	Mn	Ni	Se	Zn
AT-PB-005	No. 2 Diesel Oil, from tank	< 1.00	31.00	< 1.00	0.0300	< 1.00	< 1.00	< 1.00	< 1.00
AT-UF-001	Caustic, dilute (from drum)*	0.05	< 0.01	0.65	< 0.0002	0.02	0.08	< 0.005	0.07
AT-PB-007	Flocculant, from drum	0.05	0.44	0.13	< 0.0002	0.45	0.05	0.11	0.38
AT-PB-008	Frother, from drum	0.17	3.15	0.49	0.0756	< 0.04	0.34	0.24	0.46

SCS SN	Sample Description	Sulfur Forms			
		Tot	Org	Sulf	Pyr
AT-PB-005	No. 2 Diesel Oil, from tank	-	-	-	-
AT-UF-001	Caustic, dilute (from drum)*	-	-	< 1	-
AT-PB-007	Flocculant, from drum	-	-	-	-
AT-PB-008	Frother, from drum	-	-	-	-

(<) Indicates that the value is less than instrument detection limits.

(*) Collected during Upper Freeport Demo Run.

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Pittsburgh No.8 Raw Coal

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	6.9	67*	8.9	9
Barium	140	528*	121.6	ND
Beryllium	2	1.6*	1.6	ND
Cadmium	BD	.14*	.12	ND
Chlorine	688	665*	953	ND
Cobalt	10	8.5*	5.7	ND
Chromium (D3684)	9	ND	ND	ND
Chromium (D3683)	41	121*	38.6	ND
Fluorine	17	81*	101	ND
Lead	9	37.4*	17.23	ND
Mercury	0.1	.41*	0.09	.04
Manganese	35	113*	34.4	ND
Nickel (D3684)	8	ND	ND	ND
Nickel (D3683)	15	34*	16.2	ND
Selenium	BD	1.8*	2.7	.33
Zinc	31	63.7*	28.2	ND

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Pittsburgh No. 8 Feed Coal Analyses

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	1.8*	5.2*	12.5	4*
Barium	46*	42.1*	36.3	ND
Beryllium	3*	1*	.83	ND
Cadmium	BD	.06*	.048	ND
Chlorine	909*	1036*	1674	ND
Cobalt	3*	3*	2.3	ND
Chromium (D3684)	6*	ND	ND	ND
Chromium (D3683)	12*	10.5*	161	ND
Fluorine	95*	71*	BD	ND
Lead	3*	2.7*	1.94	ND
Mercury	.08*	.05*	bd	.05*
Manganese	11*	10.3*	7	ND
Nickel (D3684)	11*	ND	ND	ND
Nickel (D3683)	10*	7.1*	8.42	ND
Selenium	BD	1.6*	BD	.07
Zinc	8*	9.4*	7.78	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Pittsburgh No. 8 Second Stage Flotation Product

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	1.6*	3.6*	9*	4*
Barium	42*	38.5*	34.5*	ND
Beryllium	2*	1.1*	.84*	ND
Cadmium	BD	.05*	.05*	ND
Chlorine	921*	966*	1490*	ND
Cobalt	2*	2.6*	2.1*	ND
Chromium (D3684)	15*	ND	ND	ND
Chromium (D3683)	13*	11.6*	18.2*	ND
Fluorine	94*	82*	BD	ND
Lead	1*	2.3*	4.16*	ND
Mercury	.04*	.05	BD	.05*
Manganese	14*	5*	7.53*	ND
Nickel (D3684)	3*	ND	ND	ND
Nickel (D3683)	8*	7*	16.9*	ND
Selenium	3*	1.3*	BD	.07
Zinc	7*	7*	6.4*	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Pittsburgh No. 8 Tailings

	Alabama Power Company Laboratory	Iowa State University	Southern Research Institute	Huffman Laboratories
Arsenic	14.5*	19*	61.6	20*
Barium	129*	104*	96.4	ND
Beryllium	13*	1.1*	.84	ND
Cadmium	2.4*	.3*	.148	ND
Chlorine	550*	665*	774	ND
Cobalt	8*	8.1*	7.38	ND
Chromium (D3684)	20*	ND	ND	ND
Chromium (D3683)	118*	120*	161	ND
Fluorine	411*	161*	73.4	ND
Lead	11*	9.8*	7.73	ND
Mercury	.15*	.24*	.07	.05*
Manganese	202*	196*	176	ND
Nickel (D3684)	8*	ND	ND	ND
Nickel (D3683)	50*	39*	64.9	ND
Selenium	6*	4.2*	BD	.05*
Zinc	35*	34*	31	ND

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

Illinois No. 5

Illinois No. 5 Demonstration Run

Raw and Feed Coal Analyses

All Analyses Reported On A Dry Basis

Results % by weight unless otherwise noted

Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Moist	Ash	Vol	FC	BTU/lb
AT-15-101	Feed, CDSB # 1 & # 2	13.92	18.9	32.71	48.39	11577
AT-15-118	Feed, CDSB # 3 & # 4	14.67	17.62	33.34	49.04	11760
AT-15-132	Feed, CDSB # 5 & # 6	14.87	18.01	32.92	49.07	11769
AT-15-143	Feed, CDSB # 7 & # 8	14.46	16.05	34.24	49.71	12033
AT-15-149	Feed, CDSB # 9 & #10	14.55	17.91	33.84	48.25	11731
AT-15-165	Feed, CDSB #11 & #12	14.43	17.73	33.62	48.65	11724
AT-15-186	Feed, CDSB #13 & #14	14.17	17.06	34.27	48.67	11815
AT-15-195	Feed, CDSB #15 & #16	14.71	17.70	33.95	48.35	11688
	Geochemical Testing	6.75	19.55	-	-	11432
	SRI	17.1	-	-	-	-
AT-15-203	Feed, CDSB #17 & #18	14.27	18.61	33.39	48.00	11708
AT-15-217	Feed, CDSB #19 & #20	14.65	20.95	32.42	46.63	11319
AT-15-233	Feed, CDSB #21 & #22	14.59	18.70	33.09	48.21	11588
	Geochemical Testing	7.92	18.85	-	-	11450
AT-P8-237	Feed, CDSB #23 & #24	14.48	16.32	34.38	49.30	12069
	Average	14.48	17.96	33.51	48.52	11732
	Standard Deviation	0.25	1.23	0.62	0.75	190

SCS SN	Sample Description	C	H	N	O	Tot	Sulfur Forms		
							Org	Sulf	Pyr
AT-15-101	Feed, CDSB # 1 & # 2	67.08	4.42	1.51	6.2	1.89	0.69	0.14	1.06
AT-15-118	Feed, CDSB # 3 & # 4	67.87	4.51	1.53	6.39	2.08	0.95	0.14	0.99
AT-15-132	Feed, CDSB # 5 & # 6	67.52	4.43	1.48	6.52	2.04	0.95	0.12	0.97
AT-15-143	Feed, CDSB # 7 & # 8	69.22	4.53	1.54	6.59	2.07	1.06	0.14	0.87
AT-15-149	Feed, CDSB # 9 & #10	67.88	4.37	1.47	6.32	2.05	0.94	0.15	0.96
AT-15-165	Feed, CDSB #11 & #12	67.78	4.34	1.53	6.63	1.99	0.92	0.15	0.92
AT-15-186	Feed, CDSB #13 & #14	68.05	4.35	1.54	7.00	2.00	0.79	0.15	1.06
AT-15-195	Feed, CDSB #15 & #16	67.80	4.35	1.53	6.43	2.19	1.04	0.17	0.98
	Geochemical Testing	-	-	-	-	1.97	0.61	0.16	1.20
	SRI	-	-	-	-	-	-	-	-
AT-15-203	Feed, CDSB #17 & #18	67.44	4.25	1.51	6.19	2.00	0.93	0.17	0.90
AT-15-217	Feed, CDSB #19 & #20	65.78	4.14	1.52	5.65	1.96	0.78	0.16	1.02
AT-15-233	Feed, CDSB #21 & #22	67.40	4.16	1.59	6.04	2.11	0.98	0.16	0.97
	Geochemical Testing	-	-	-	-	1.98	0.65	0.16	1.17
AT-P8-237	Feed, CDSB #23 & #24	69.25	4.2	1.7	6.45	2.08	0.89	0.15	1.04
	Average	67.76	4.34	1.54	6.37	2.04	0.91	0.15	0.98
	Standard Deviation	0.87	0.12	0.06	0.32	0.07	0.10	0.01	0.06

Samples marked with (*) are samples that have QC analyses directly underneath.

CDSB = Crusher Discharge Stop-Belt Feed Coal Samples.

Illinois No.5 Demonstration Run
Feed Coal Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Method		ASTM D4606	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-15-101	Feed, CDSB # 1 & # 2	10.5	91	4	< 1.0	1827	7	8	19
AT-15-118	Feed, CDSB # 3 & # 4	10.9	81	4	< 1.0	902	7	13	17
AT-15-131	Feed, CDSB # 5 & # 6	5.9	84	4	< 1.0	1196	7	7	18
AT-15-143	Feed, CDSB # 7 & # 8	17.6	73	4	< 1.0	931	6	10	16
AT-15-149	Feed, CDSB # 9 & #10	30.5	82	4	< 1.0	944	7	11	18
AT-15-165	Feed, CDSB #11 & #12	43.4	82	4	1.1	911	7	10	18
AT-15-186	Feed, CDSB #13 & #14	36.9	76	4	1.2	1470	7	13	16
AT-15-195*	Feed, CDSB #15 & #16	35.6	68	7	1.1	1350	7	7	17
	Standard Laboratories	6.35	63	2.00	< 0.05	2500	16.12	-	19.0
	SRI	52.4	69.9	1.67	0.074	2027	4.73	-	22.9
AT-15-203	Feed, CDSB #17 & #18	33.0	84	7	< 1.0	1962	7	4	19
AT-15-217	Feed, CDSB #19 & #20	32.6	94	4	< 1.0	623	7	15	20
AT-15-233*	Feed, CDSB #21 & #22	31.2	77	7	< 1.0	650	7	20	18
	Standard Laboratories	7.26	61.57	1.94	< 0.05	2300	16.75	-	20
AT-15-237	Feed, CDSB #23 & #24	24.8	67	4	< 1.0	714	6	19	16
	Average	26.1	80	5	1.1	1123	7	11	18
	Standard Deviation	11.5	8	1	0.0	426	0	5	1

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D4606	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-15-101	Feed, CDSB # 1 & # 2	121	30	< 0.02	45	8	26	2	84
AT-15-118	Feed, CDSB # 3 & # 4	149	34	< 0.02	43	4	26	< 2	95
AT-15-131	Feed, CDSB # 5 & # 6	57	28	0.01	43	7	24	< 2	94
AT-15-143	Feed, CDSB # 7 & # 8	70	30	< 0.02	38	9	25	< 2	67
AT-15-149	Feed, CDSB # 9 & #10	99	31	0.06	45	8	26	< 2	66
AT-15-165	Feed, CDSB #11 & #12	96	32	< 0.02	45	9	26	< 2	109
AT-15-186	Feed, CDSB #13 & #14	100	40	< 0.02	40	6	26	< 2	76
AT-15-195*	Feed, CDSB #15 & #16	89	29	< 0.02	54	9	25	< 2	97
	Standard Laboratories	120.8	28	0.22	43.5	-	28.0	0.98	54.5
	SRI	41.9	18.2	< 0.03	31.1	-	20.7	< 2	34.7
AT-15-203	Feed, CDSB #17 & #18	102	27	0.08	55	8	26	< 2	106
AT-15-217	Feed, CDSB #19 & #20	64	35	0.11	49	9	26	< 2	91
AT-15-233*	Feed, CDSB #21 & #22	58	30	0.10	53	9	24	< 2	70
	Standard Laboratories	133.45	26.7	0.16	40.8	-	28.15	1.18	43.72
AT-15-237	Feed, CDSB #23 & #24	52	27	0.10	47	8	24	< 2	106
	Average	88	31	0.08	46	8	25	< 2	88
	Standard Deviation	28	4	0.03	5	1	1	0	15

Samples marked with (*) are samples that have QC analyses directly underneath.
Samples marked with (#) indicate samples not tested for trace elements.
(<) Indicates result was below instrument detection limits.
CDSB = Crusher Discharge Stop-Belt Feed Coal SampleS.

Illinois No. 5 Demonstration Run
 Second Stage Flotation Product Analyses
 All Analyses Reported On A Dry Basis
 Results % By Weight Unless Otherwise Noted

100 Mesh Coal
 Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-15-116	F2PR- 3, Coal Product	4.50	37.38	58.12	13698
AT-15-125	F2PR- 4, Coal Product	4.70	36.76	58.54	13405
AT-15-184	F2PR-12, Coal Product	4.88	37.19	57.93	13530
AT-15-191 *	F2PR-14, Coal Product	4.61	36.68	58.71	13613
	Geochemical Testing	4.94	-	-	13697
	SRI	4.62	-	-	-
AT-15-212	F2PR-16, Coal Product	4.53	39.15	56.32	13462
AT-15-215	F2PR-17, Coal Product	4.61	36.9	58.49	13776
AT-15-219	F2PR-18, Coal Product	5.59	38.71	55.7	13336
Average		4.77	37.54	57.69	13546
Standard Deviation		0.35	0.92	1.10	147

SCS SN	Sample Description	C	H	N	O	Sulfur Forms			
						Tot	Org	Sulf	Pyr
AT-15-116	F2PR- 3, Coal Product	78.62	5.16	1.74	8.52	1.46	0.76	0.15	0.55
AT-15-125	F2PR- 4, Coal Product	79.34	5.14	1.77	7.60	1.45	0.83	0.08	0.54
AT-15-184	F2PR-12, Coal Product	78.50	5.12	1.75	8.28	1.47	0.74	0.14	0.59
AT-15-191 *	F2PR-14, Coal Product	79.10	5.09	1.76	7.98	1.46	0.73	0.15	0.58
	Geochemical Testing	-	-	-	-	1.44	0.74	0.15	0.55
	SRI	-	-	-	-	-	-	-	-
AT-15-212	F2PR-16, Coal Product	76.71	5.36	1.70	10.31	1.39	0.75	0.10	0.54
AT-15-215	F2PR-17, Coal Product	78.84	5.16	1.74	8.19	1.46	0.77	0.11	0.58
AT-15-219	F2PR-18, Coal Product	76.62	5.31	1.70	9.36	1.42	0.77	0.09	0.56
Average		78.25	5.19	1.74	8.61	1.44	0.76	0.12	0.56
Standard Deviation		1.03	0.09	0.03	0.86	0.03	0.03	0.03	0.02

Samples marked with (*) are samples that have QC analyses directly underneath.
 (<) Indicates result was below instrument detection limits.

Illinois No. 5 Demonstration Run
Second Stage Flotation Product Samples
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

100 Mesh Coal
Trace Elements

Test Methods		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-15-116	F2PR- 3, Coal Product	16.4	20	3	< 1.0	1330	9	12	10
AT-15-125	F2PR- 4, Coal Product	7.3	21	3	< 1.0	1364	8	15	11
AT-15-184	F2PR- 12, Coal Product	9.5	22	3	< 1.0	1520	9	14	10
AT-15-191 *	F2PR-14, Coal Product	11.6	16	3	< 1.0	1516	7	13	10
	Standard Laboratories	7.88	17	1.48	< 0.05	2100	7	-	11
	SRI	24.8	17.8	1.63	0.063	2356	3.46	-	12.9
AT-15-212	F2PR-16, Coal Product	15.7	18	3	< 1.0	1465	8	11	10
AT-15-215	F2PR-17, Coal Product	7.2	17	3	< 1.0	1471	9	12	10
AT-15-219	F2PR-18, Coal Product	7.1	22	3	< 1.0	1468	10	35	12
	Average	10.7	19	3	< 1.0	1448	9	16	10
	Standard Deviation	3.7	2	0	0	68	1	8	1

Test Methods		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-15-116	F2PR- 3, Coal Product	66	16	0.04	12	7	18	< 2	20
AT-15-125	F2PR- 4, Coal Product	41	20	0.02	12	9	19	< 2	22
AT-15-184	F2PR- 12, Coal Product	42	15	0.04	13	15	19	< 2	27
AT-15-191 *	F2PR-14, Coal Product	46	19	0.03	12	27	17	< 2	22
	Standard Laboratories	55.7	18.57	0.15	10	-	19	0.80	15.16
	SRI	< 25	9.77	< 0.03	8.88	-	16.1	< 2	24.4
AT-15-212	F2PR-16, Coal Product	58	23	0.02	11	10	17	< 2	20
AT-15-215	F2PR-17, Coal Product	60	16	0.04	11	16	17	< 2	23
AT-15-219	F2PR-18, Coal Product	43	21	< 0.02	12	7	19	< 2	27
	Average	51	19	0.03	12	13	18	< 2	23
	Standard Deviation	9	3	0.01	1	7	1	0	3

Samples marked with (*) are samples that have QC analyses directly underneath.
(<) Indicates result below instrument detection limits.

Illinois No. 5 Demonstration Run
 Second Stage Flotation Product Analyses
 All Analyses Reported On A Dry Basis
 Results % By Weight Unless Otherwise Noted
 200 Mesh Coal
 Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-15-228 *	F2PR-20, Coal Product	4.30	37.44	58.26	13601
	Geochemical Testing	4.63	-	-	13652
AT-15-235	F2PR-21, Coal Product	4.63	38.75	56.62	13715
AT-15-242	F2PR-23, Coal Product	4.51	37.63	57.86	13888
	Average	4.48	37.94	57.58	13735
	Standard Deviation	0.14	0.58	0.70	118

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-15-228 *	F2PR-20, Coal Product	78.73	5.18	1.73	8.67	1.39	0.72	0.12	0.55
	Geochemical Testing	-	-	-	-	1.40	0.76	0.14	0.50
AT-15-235	F2PR-21, Coal Product	78.69	5.16	1.72	8.39	1.41	0.77	0.12	0.52
AT-15-242	F2PR-23, Coal Product	78.18	5.12	1.70	9.07	1.42	0.83	0.12	0.47
	Average	78.53	5.15	1.72	8.71	1.41	0.77	0.12	0.51
	Standard Deviation	0.25	0.02	0.01	0.28	0.01	0.04	0.00	0.03

Samples marked with (*) are samples that have QC analyses directly underneath.
 (<) Indicates result was below instrument detection limits.

Illinois No. 5 Demonstration Run
 Second Stage Flotation Product Samples
 All Analyses Reported On A Dry Basis
 All Results Reported In mg/kg

200 Mesh Coal
 Trace Elements

Test Methods		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-15-228 *	F2PR-20, Coal Product	14.3	18	3	< 1.0	1658	7	16	11
	Standard Laboratories	5.94	18	1.33	< 0.05	2000	6	-	13
AT-15-235	F2PR-21, Coal Product	5.8	21	3	< 1.0	1684	8	31	12
AT-15-242	F2PR-23, Coal Product	15.9	20	3	< 1.0	1427	7	18	12
	Average	12.0	20	3	< 1.0	1590	7	22	12
	Standard Deviation	4.4	1	0	0	116	0	7	0

Test Methods		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-15-228 *	F2PR-20, Coal Product	46	18	0.02	15	7	16	< 2	27
	Standard Laboratories	50	15	0.17	12	-	19	0.72	21.8
AT-15-235	F2PR-21, Coal Product	41	19	< 0.02	16	6	17	< 2	28
AT-15-242	F2PR-23, Coal Product	42	20	< 0.02	16	7	18	< 2	25
	Average	43	19	< 0.02	16	7	17	< 2	27
	Standard Deviation	2	1	0	0	0	1	0	1

Samples marked with (*) are samples that have QC analyses directly underneath.
 (<) Indicates result below instrument detection limits.

Illinois No. 5 Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
Results % By Weight Unless Otherwise Noted

100 Mesh Coal
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-15-115	FLT- 3, Flotation Tailings	52.05	21.21	26.74	6194
AT-15-124	FLT- 4, Flotation Tailings	55.94	20.76	23.30	5277
AT-15-183	FLT-12, Flotation Tailings	58.18	19.80	22.02	5251
AT-15-190 *	FLT-14, Flotation Tailings	53.82	22.18	24.00	5951
	Geochemical Testing	54.40	-	-	5838
	SRI	53.1	-	-	-
	SRI (Dup AT-15-190)	53.1	-	-	-
AT-15-211	FLT-16, Flotation Tailings	54.28	22.32	23.40	5719
AT-15-214	FLT-17, Flotation Tailings	54.40	21.07	24.53	5629
AT-15-218	FLT-18, Flotation Tailings	59.45	19.51	21.04	5063
	Average	55.45	20.98	23.58	5583
	Standard Deviation	2.40	0.99	1.69	379

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-15-115	FLT- 3, Flotation Tailings	35.30	2.58	0.75	6.04	3.28	0.99	0.10	2.19
AT-15-124	FLT- 4, Flotation Tailings	31.78	2.39	0.68	5.81	3.40	1.13	0.09	2.18
AT-15-183	FLT-12, Flotation Tailings	30.51	2.34	0.64	5.29	3.04	0.76	0.13	2.15
AT-15-190 *	FLT-14, Flotation Tailings	34.09	2.54	0.72	5.86	2.97	0.74	0.21	2.06
	Geochemical Testing	-	-	-	-	2.97	0.24	0.26	2.47
	SRI	-	-	-	-	-	-	-	-
	SRI (Dup AT-15-190)	-	-	-	-	-	-	-	-
AT-15-211	FLT-16, Flotation Tailings	33.54	2.47	0.68	5.88	3.15	0.93	0.22	2.00
AT-15-214	FLT-17, Flotation Tailings	33.42	2.46	0.68	6.24	2.80	0.79	0.14	1.87
AT-15-218	FLT-18, Flotation Tailings	29.31	2.19	0.58	5.17	3.30	0.84	0.29	2.17
	Average	32.56	2.42	0.68	5.76	3.13	0.88	0.17	2.09
	Standard Deviation	1.96	0.12	0.05	0.36	0.20	0.13	0.07	0.11

Samples marked with (*) have QC results reported directly underneath.

Illinois No. 5 Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

100 Mesh Coal
Trace Elements

Test Methods		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-I5-115	FLT- 3, Flotation Tailings	46.2	178	12	2.7	701	29	34	73
AT-I5-124	FLT- 4, Flotation Tailings	50.6	191	12	2.9	856	30	20	74
AT-I5-183	FLT-12, Flotation Tailings	42.7	208	13	3.1	1624	40	14	78
AT-I5-190 *	FLT-14, Flotation Tailings	42.7	258	18	4.7	1211	34	13	88
	Standard Laboratories	10.8	126.8	2.7	< 0.05	1400	45	-	79
	SRI	124	214	1.96	0.46	1022	9.31	-	107
	SRI (Dup AT-I5-190)	-	222	1.94	0.44	-	9.20	-	94.8
AT-I5-211	FLT-16, Flotation Tailings	72.0	197	12	3.8	863	26	13	76
AT-I5-214	FLT-17, Flotation Tailings	71.0	174	12	4.3	799	31	13	80
AT-I5-218	FLT-18, Flotation Tailings	68.8	191	13	4.7	725	35	14	85
Average		56.3	200	13	3.74	968	32	17	79
Standard Deviation		12.7	26	2	0.79	310	4	7	5

Test Methods		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-I5-115	FLT- 3, Flotation Tailings	134	48	0.04	160	7	48	< 2	240
AT-I5-124	FLT- 4, Flotation Tailings	199	40	0.03	175	7	48	< 2	235
AT-I5-183	FLT-12, Flotation Tailings	141	42	0.08	180	30	53	< 2	222
AT-I5-190 *	FLT-14, Flotation Tailings	108	51	0.08	191	24	62	< 2	202
	Standard Laboratories	213.8	49	0.18	137.0	-	60	1.9	173
	SRI	< 25	41.5	0.05	116	-	34.5	< 2.5	190
	SRI (Dup AT-I5-190)	-	42.2	-	117	-	34.0	< 2.5	192
AT-I5-211	FLT-16, Flotation Tailings	183	35	0.05	164	17	51	< 2	189
AT-I5-214	FLT-17, Flotation Tailings	195	34	0.04	152	18	57	< 2	179
AT-I5-218	FLT-18, Flotation Tailings	153	46	0.06	173	21	58	< 2	215
Average		159	42	0.05	171	18	54	< 2	212
Standard Deviation		32	6	0.02	12	8	5	0	21

Samples marked with (*) have QC results reported directly underneath.
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Illinois No. 5 Demonstration Run
Combined Flotation Tailings Analyses
All Analyses Reported On A Dry Basis
Results % By Weight Unless Otherwise Noted

200 Mesh Coal
Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-I5-227 *	FLT-20, Flotation Tailings	63.38	17.59	19.03	4475
	Geochemical Testing	63.25	-	-	4762
AT-I5-234	FLT-21, Flotation Tailings	61.68	17.90	20.42	4619
AT-I5-243	FLT-23, Flotation Tailings	56.43	19.79	23.78	5526
	Average	60.50	18.43	21.08	4873
	Standard Deviation	2.96	0.97	1.99	465

SCS SN	Sample Description	Sulfur Forms							
		C	H	N	O	Tot	Org	Sulf	Pyr
AT-I5-227 *	FLT-20, Flotation Tailings	25.78	1.96	0.49	3.53	4.86	1.17	0.37	3.32
	Geochemical Testing	-	-	-	-	4.72	0.25	0.39	4.08
AT-I5-234	FLT-21, Flotation Tailings	26.83	1.96	0.54	4.78	4.21	0.95	0.35	2.91
AT-I5-243	FLT-23, Flotation Tailings	31.74	2.24	0.66	5.30	3.63	0.96	0.34	2.33
	Average	28.12	2.05	0.56	4.54	4.23	1.03	0.35	2.85
	Standard Deviation	2.60	0.13	0.07	0.74	0.50	0.10	0.01	0.41

Samples marked with (*) have QC results reported directly underneath.

Illinois No. 5 Demonstration Run
 Combined Flotation Tailings Analyses
 All Analyses Reported On A Dry Basis
 All Results Reported In mg/kg

200 Mesh Coal
 Trace Elements

SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-I5-227 *	FLT-20, Flotation Tailings	68.7	214	17	6.1	454	32	32	129
	Standard Laboratories	13.3	169.5	3.21	< 0.05	1100	53.03	-	125
AT-I5-234	FLT-21, Flotation Tailings	49.8	224	14	5.4	976	36	21	108
AT-I5-243	FLT-23, Flotation Tailings	53.3	246	13	5	580	30	14	101
	Average	57.3	228	15	5.5	670	33	22	113
	Standard Deviation	8.2	13	2	0.5	222	2	7	12

SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-I5-227 *	FLT-20, Flotation Tailings	170	54	< 0.02	237	18	75	< 2	305
	Standard Laboratories	211.2	58	0.33	201.5	-	77	2	242.66
AT-I5-234	FLT-21, Flotation Tailings	165	51	0.02	216	8	66	< 2	290
AT-I5-243	FLT-23, Flotation Tailings	142	47	0.06	197	10	62	< 2	220
	Average	159	51	0.03	217	12	68	< 2	272
	Standard Deviation	12	3	0.02	16	4	5	0.0	37

Samples marked with (*) have QC results reported directly underneath.
 (<) Indicates that the sample result was below instrument detection limits.

Illinois No.5 Demonstration Run
Vacuum Filter Product Samples
All Analyses Reported On A Dry Basis
All Results Reported In mg/kg

Trace Elements

Test Method		SLURRY /GFAAS	ASTM D3683	ASTM D3683	ASTM D3683	ASTM D4208	ASTM D3683	ASTM D3684	ASTM D3683
SCS SN	Sample Description	As	Ba	Be	Cd	Cl	Co	Cr	Cr
AT-15-126	VFPR- 4, Product Coal	4.4	23	3	< 1.0	1412	9	9	11
AT-15-192	VFPR-14, Product Coal	9.5	21	3	< 1.0	1320	9	14	10
AT-15-213	VFPR-16, Product Coal	24.1	21	3	< 1.0	1632	11	12	11
AT-15-243	VFPR-23, Product Coal	7.2	22	3	< 1.0	1251	10	13	12
Average		11.3	22	3	< 1.0	1404	10	12	11
Standard Deviation		7.6	1	0	0	144	1	2	1

Test Method		ASTM D3761	ASTM D3683	ASTM D3684	ASTM D3683	ASTM D3684	ASTM D3683	SLURRY /GFAAS	ASTM D3683
SCS SN	Sample Description	F	Pb	Hg	Mn	Ni	Ni	Se	Zn
AT-15-126	VFPR- 4, Product Coal	95	14	< 0.02	13	7	19	< 2	22
AT-15-192	VFPR-14, Product Coal	89	14	0.04	13	7	17	< 2	22
AT-15-213	VFPR-16, Product Coal	74	21	0.02	13	7	19	< 2	23
AT-15-243	VFPR-23, Product Coal	100	15	0.02	16	5	20	< 2	22
Average		90	16	0.03	14	7	19	< 2	22
Standard Deviation		10	3	0.01	1	1	1	0	0

Samples marked with (*) are samples that have QC analyses directly underneath.
(<) Indicates result below instrument detection limits.

Illinois No. 5 Demonstration Run
 Vacuum Filter Product Analyses
 All Analyses Reported On A Dry Basis
 Results % By Weight Unless Otherwise Noted
 Short Proximate, Ultimate, Sulfur Forms

SCS SN	Sample Description	Ash	Vol	FC	BTU/lb
AT-15-126	VFPR- 4, Product Coal	4.66	37.32	58.02	13848
AT-15-192	VFPR-14, Product Coal	4.39	37.22	58.39	13891
AT-15-213	VFPR-16, Product Coal	4.56	37.08	58.36	13888
AT-15-243	VFPR-23, Product Coal	4.7	37.8	57.5	13930
Average		4.58	37.36	58.07	13889
Standard Deviation		0.12	0.27	0.36	29

SCS SN	Sample Description	C	H	N	O	Sulfur Forms			
						Tot	Org	Sulf	Pyr
AT-15-126	VFPR- 4, Product Coal	78.78	5.11	1.75	8.24	1.46	0.73	0.13	0.6
AT-15-192	VFPR-14, Product Coal	79.09	5.09	1.77	8.27	1.39	0.73	0.1	0.56
AT-15-213	VFPR-16, Product Coal	79.16	5.08	1.77	8.05	1.38	0.71	0.12	0.55
AT-15-243	VFPR-23, Product Coal	79.22	5.18	1.77	7.74	1.39	0.72	0.13	0.54
Average		79.06	5.12	1.77	8.08	1.41	0.72	0.12	0.56
Standard Deviation		0.17	0.04	0.01	0.21	0.03	0.01	0.01	0.02

Samples marked with (*) are samples that have QC analyses directly underneath.
 (<) Indicates result below instrument detection limits.

Illinois No. 5 Demonstration Run
System Water Analyses
All Analyses Reported On An As Received Basis
All Results In mg/L

Trace Elements

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-15-119,120	CLWT- 4, Recycle Water	09/09/92	0001	0.005	0.03	< 0.01	< 0.01	164.6	< 0.01	< 0.01
AT-15-135,136	CLWT- 6, Recycle Water	09/09/92	0800	0.008	0.03	< 0.01	< 0.01	175.3	< 0.01	< 0.01
AT-15-171,172	CLWT-11, Recycle Water	09/10/92	0800	0.007	0.03	< 0.01	< 0.01	193.9	< 0.01	< 0.01
AT-15-177,178	CLWT-13, Recycle Water	09/10/92	1600	0.003	0.03	< 0.01	< 0.01	212.6	< 0.01	< 0.01
AT-15-193,194	CLWT-15, Recycle Water	09/11/92	0001	0.002	0.03	< 0.01	< 0.01	184.6	< 0.01	< 0.01
AT-15-208,209	CLWT-17, Recycle Water	09/11/92	0800	0.003	0.03	< 0.01	< 0.01	227.2	< 0.01	< 0.01
AT-15-230,231	CLWT-20, Recycle Water	09/11/92	2200	0.005	0.03	< 0.01	< 0.01	244.6	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-15-119,120	CLWT- 4, Recycle Water	0.31	0.872	< 0.01	< 0.0002	0.80	< 0.02	0.003	0.04
AT-15-135,136	CLWT- 6, Recycle Water	0.31	1.520	< 0.01	< 0.0002	0.80	< 0.02	0.002	0.02
AT-15-171,172	CLWT-11, Recycle Water	0.39	2.460	< 0.01	< 0.0002	0.83	< 0.02	0.003	0.02
AT-15-177,178	CLWT-13, Recycle Water	0.31	0.932	< 0.01	< 0.0002	0.91	< 0.02	0.003	0.02
AT-15-193,194	CLWT-15, Recycle Water	0.39	0.864	< 0.01	< 0.0002	0.92	< 0.02	0.003	0.01
AT-15-208,209	CLWT-17, Recycle Water	0.45	1.160	< 0.01	< 0.0002	1.01	< 0.02	0.002	0.01
AT-15-230,231	CLWT-20, Recycle Water	2.13	1.640	< 0.01	< 0.0002	1.15	< 0.02	0.003	0.01

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-15-121,122	Laboratory Deionized Water	09/09/92	0001	< 0.002	< 0.01	< 0.01	< 0.01	2.00	< 0.01	< 0.01
AT-15-204,205	MW-17, Makeup Water	09/11/92	0800	< 0.002	0.05	< 0.01	< 0.01	34.00	< 0.01	< 0.01
AT-15-206,207	PW-17, Potable Water	09/11/92	0800	0.002	0.01	< 0.01	< 0.01	80.60	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-15-121,122	Laboratory Deionized Water	< .01	-	< 0.01	< 0.0002	< 0.01	< 0.02	< 0.0002	0.02
AT-15-204,205	MW-17, Makeup Water	0.15	-	< 0.01	< 0.0002	0.01	< 0.02	< 0.0002	0.02
AT-15-206,207	PW-17, Potable Water	0.03	-	< 0.01	< 0.0002	0.03	< 0.02	< 0.0002	0.05

(<) Indicates that the result is below instrument detection limits

Illinois No. 5 Demonstration Run
Product Filtrate Water Analyses
All Analyses Reported On An As Received Basis
All Results In mg/L

Trace Elements

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-15-127,128	VFFLP- 4, Product Filtrate	09/08-09/92	2315-0315	0.006	0.03	< 0.01	< 0.01	217.9	< 0.01	< 0.01
AT-15-161,162	VFFLP- 9, Product Filtrate	09/09-10/92	2315-0315	0.003	0.03	< 0.01	< 0.01	206.6	< 0.01	< 0.01
AT-15-199,200	VFFLP-15, Product Filtrate	09/10-11/92	2315-0315	0.003	0.03	< 0.01	< 0.01	233.7	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-15-127,128	VFFLP- 4, Product Filtrate	0.39	-	< 0.01	< 0.0002	1.09	0.02	0.004	0.02
AT-15-161,162	VFFLP- 9, Product Filtrate	0.31	-	< 0.01	< 0.0002	1.07	0.03	0.002	0.02
AT-15-199,200	VFFLP-15, Product Filtrate	0.47	-	< 0.01	0.0003	1.30	0.02	0.002	0.02

Test Method				EPA 206.2	EPA 200.7	EPA 200.7	EPA 200.7	EPA 300.0	EPA 200.7	EPA 200.7
SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-15-129,130	VFFLT- 4, Tailings Filtrate	09/08-09/92	2315-0315	0.003	0.03	< 0.01	< 0.01	211.2	< 0.01	< 0.01
AT-15-163,164	VFFLT- 9, Tailings Filtrate	09/09-10/92	2315-0315	0.004	0.03	< 0.01	< 0.01	227.9	< 0.01	< 0.01
AT-15-201,202	VFFLT-15, Tailings Filtrate	09/10-11/92	2315-0315	0.004	0.03	< 0.01	< 0.01	242.2	< 0.01	< 0.01

Test Method		EPA 300.0	EPA 236.1	EPA 200.7	EPA 245.1	EPA 200.7	EPA 200.7	EPA 270.2	EPA 200.7
SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-15-129,130	VFFLT- 4, Tailings Filtrate	0.39	-	< 0.01	< 0.0002	0.97	< 0.02	0.002	0.02
AT-15-163,164	VFFLT- 9, Tailings Filtrate	0.47	-	< 0.01	0.0005	1.04	< 0.02	0.002	0.02
AT-15-201,202	VFFLT-15, Tailings Filtrate	0.51	-	< 0.01	< 0.0002	1.12	< 0.02	0.002	0.02

(<) Indicates that the result is below instrument detection limits

Illinois No. 5 Demonstration Run
 No. 2 Diesel Analysis
 All Analyses Reported On An As Received Basis
 All Results In mg/L

Trace Elements

SCS SN	Sample Description	Date	Time	As	Ba	Be	Cd	Cl	Co	Cr
AT-15-123	No. 2 Diesel Fuel	09/09/92	0001	< 1	< 1	< 1	< 1	< 150	< 1	< 1

SCS SN	Sample Description	F	Fe	Pb	Hg	Mn	Ni	Se	Zn
AT-15-123	No. 2 Diesel Fuel	657	1.000	< 1	< 0.02	< 1	< 1	< 1	< 1

(<) Indicates that the value is less than instrument detection limits

2 of 2

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Illinois No. 5 Feed Coal Analyses

	Alabama Power Company Laboratory	Standard Laboratories	Southern Research Institute
Arsenic	26.1*	6.8*	52.4
Barium	80*	62*	69.9
Beryllium	5*	2*	1.67
Cadmium	1.1*	BD	.074
Chlorine	1123*	2400*	2027
Cobalt	7*	16.4*	4.73
Chromium (D3684)	11*	ND	ND
Chromium (D3683)	18*	19.5*	22.9
Fluorine	88*	127*	41.9
Lead	31*	27*	18.2
Mercury	.08*	.19*	BD
Manganese	46*	42*	31.1
Nickel (D3684)	8*	ND	ND
Nickel (D3683)	25*	28*	20.7
Selenium	BD	1.1*	BD
Zinc	88*	49*	34.7

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Illinois No. 5 100M Flotation Product

	Alabama Power Company Laboratory	Standard Laboratories	Southern Research Institute
Arsenic	10.7*	7.88	24.8
Barium	19*	17	17.8
Beryllium	3*	1.48	1.63
Cadmium	BD	BD	.063
Chlorine	1448*	2100	2356
Cobalt	9*	7	3.46
Chromium (D3684)	16*	ND	ND
Chromium (D3683)	10*	11	12.9
Fluorine	51*	55.7	BD
Lead	19*	18.57	9.77
Mercury	.03*	.15	BD
Manganese	12*	10	8.88
Nickel (D3684)	13*	ND	ND
Nickel (D3683)	18*	19	16.1
Selenium	BD	.8	BD
Zinc	23*	15.16	24.4

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Illinois No. 5 100M Tailings

	Alabama Power Company Laboratory	Standard Laboratories	Southern Research Institute
Arsenic	56.3*	10.8	124
Barium	200*	126.8	213*
Beryllium	13*	2.7	1.95*
Cadmium	3.74*	BD	.45*
Chlorine	968*	1400	1022
Cobalt	32*	45	9.3
Chromium (D3684)	17*	ND	ND
Chromium (D3683)	79*	79	101*
Fluorine	159*	213.8	BD
Lead	42*	49	41.9*
Mercury	.05*	.18	.05
Manganese	171*	137	117*
Nickel (D3684)	18*	ND	ND
Nickel (D3683)	54*	60	34*
Selenium	BD	1.9	BD
Zinc	212*	173	191*

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons Illinois No. 5 200M Flotation Product

	Alabama Power Company Laboratory	Standard Laboratories
Arsenic	12*	5.94
Barium	20*	18
Beryllium	3*	1.33
Cadmium	BD	BD
Chlorine	1590*	2000
Cobalt	7*	6
Chromium (D3684)	22*	ND
Chromium (D3683)	12*	13
Fluorine	43*	50
Lead	19*	15
Mercury	BD	.17
Manganese	16*	12
Nickel (D3684)	7*	ND
Nickel (D3683)	17*	19
Selenium	BD	.72
Zinc	27*	21.8

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

TRACE ELEMENT REMOVAL STUDY

Laboratory Results Comparisons

Illinois No. 5 200M Tailings

	Alabama Power Company Laboratory	Standard Laboratories
Arsenic	57.3*	13.3
Barium	228*	169.5
Beryllium	15*	3.21
Cadmium	5.5*	BD
Chlorine	670*	1100
Cobalt	33*	53.03
Chromium (D3684)	22*	ND
Chromium (D3683)	113*	125
Fluorine	159*	211.2
Lead	51*	58
Mercury	.03*	.33
Manganese	217*	201.5
Nickel (D3684)	12*	ND
Nickel (D3683)	68*	77
Selenium	BD	2
Zinc	272*	242.66

* = Average Values

BD= Below Reported Laboratory Detection Limit

ND= Not Determined

Alabama Power Company

Coal
Method 2-6.1
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Test Codes:
Antimony Oxide 4310
Zinc Oxide 4670
Beryllium Oxide 4350
Cadmium Oxide 4380
Chromium Oxide 4420
Cobalt Oxide 4430
Copper Oxide 4440
Manganese Oxide 4515
Molybdenum Oxide 4525
Nickel Oxide 4530
Vanadium Oxide 4660

TRACE ELEMENTS IN COAL ASH (Ash Mineral)

1.0 PURPOSE

This procedure may be used to document the determination of the trace constituents in coal and coal ash.

3.0 Scope

This test method covers the determination of antimony, beryllium, cadmium, chromium, cobalt, copper, lead, lithium, manganese, molybdenum, nickel, lead, strontium, vanadium, and zinc in coal and coal ash.

3.0 References

3.1 ASTM Method D3683, "Test Method for Trace Elements in Coal and Coke Ash by the Atomic Absorption Method."

4.0 Definitions

Concentrations determined in this method are in units of milligrams per kilogram of the element in coal ash. Calculations may be used to convert to dry basis coal.

5.0 Summary

The coal to be analyzed is ashed under standard conditions and ignited to constant weight. The ash is dissolved in aqua regia and hydrofluoric acid. Beryllium, cadmium, chromium, copper, manganese, nickel, strontium, vanadium, and zinc are then analyzed by argon plasma emission. Antimony, cobalt, lead, and molybdenum are analyzed by graphite furnace atomic absorption. Lithium and strontium are analyzed by flame atomic absorption.

6.0 Sample Handling and Preservation

A representative air-dried whole coal or coke sample is pulverized to pass through a No. 60 sieve.

7.0 Apparatus

7.1 Steam bath.

7.2 Bottles, plastic, 125 ml. (able to withstand 130°C.)

- 7.3 Volumetric flasks, 100 ml.
- 7.4 Ion coupled argon plasma unit
- 7.5 Atomic absorption spectrophotometer with associated gases, and associated EDL lamps. (Hollow cathode tubes are acceptable).
- 7.6 Ion Coupled Argon Plasma Spectrophotometer with associated gases.

8.0 Reagents

8.1 Aqua regia

8.1.1 Mix one part conc. nitric acid, three parts conc. hydrochloric acid, and one part deionized water.

8.2 Hydrofluoric acid, concentrated

8.3 Boric acid, saturated (60 gms. per liter)

8.4 Stock solutions for all the listed metals prepared in the same matrix as the samples.

9.0 Safety Precautions

9.1 Safety glasses, gloves, and labcoat should be worn at all times.

9.2 High temperatures will be encountered in this procedure. Use of long tongs and high temperature gloves should be used.

9.3 Hydrofluoric acid is used in this procedure. Gloves should be worn at all times when in use. Do not pick up the reagent bottle without gloves. This acid is a bone seeker. It will penetrate skin.

9.4 Analyst should be familiar with safety data sheets of chemicals and reagents used in this procedure.

10.0 Interferences

Principle interferences are given in ICAP or AA procedures given in section 2-2.1, "Trace Metal Analysis of Water Samples," which follows this procedure.

11.0 Equipment calibration and setup

See set-up and calibration in ICAP procedure in water section.

12.1 Procedure

12.1 Ash approximately 6 grams of the sample in an open crucible.

12.2 Place in a cold muffle furnace and set furnace controls such that temperature of 300° C is reached in one hour. Ramp to 500° C and hold for two hours.

12.3 Allow to cool to room temperature.

12.4 Transfer to an agate mortar and crush to 200 mesh.

12.5 Rapidly re-ignite to 500° C and continue heating for 1 hour.

12.6 Weigh out approximately 0.2 gram ash to the nearest 0.1 mg and place in 125 ml plastic bottles.

12.7 Add 3.0 mls of aqua regia and 5 mls of Hydrofluoric Acid.

12.8 Tighten screw caps and place on the steam bath for at least 2 hours.

12.9 Add 50 mls of boric acid. If residue remains, sample may be re-heated for about one hour to help dissolve it.

12.10 Cool to room temperature and dilute to 100 mls. with deionized water.

12.11 Analyze per ICAP or AA procedures found in Laboratory Procedures Manual Section Water.

13.0 Quality Control

13.1 Precision

On every tenth sample or if less than ten, the last sample, analyze a second aliquot.

13.2 Accuracy

After every tenth sample or if less than ten, the last sample, a Certified Reference Material should be analyzed.

14.0 CALCULATIONS

$$M_O = A * B * f / W$$

where

A is the conc. of sample solution after digestion in milligrams per liter

B is the sample solution volume (typically 0.1 liter)

M_O is the metal oxide concentration in the ash in mg/kg

W is the weight of ash sample in kilograms

f is the weight factor of element to element oxide
(See Table 1)

=====

Table 1 - Laboratory Determined Metals and Oxide Forms

Element	Symbol	Atomic Weight	¹ Oxide Form	Oxide Mol Wt	Ratio Metal/Oxide
Aluminum	Al	26.982	Al ₂ O ₃	101.96	1.89
Antimony	Sb	121.750	Sb ₂ O ₅	323.50	1.33
Arsenic	As	74.922	As ₂ O ₅	229.84	3.07
Barium	Ba	137.340	BaO	153.00	1.12
Beryllium	Be	9.012	BeO	27.00	3.00
Boron	B	10.810	B ₂ O ₃	70.00	3.18
Cadmium	Cd	112.400	CdO	128.40	1.14
Calcium	Ca	40.080	CaO	56.08	1.40
Chromium	Cr	51.996	Cr ₂ O ₃	152.00	1.46
Cobalt	Co	58.933	Co ₂ O ₂	165.86	1.41
Copper	Cu	63.546	CuO	79.54	1.25
Iron	Fe	55.847	Fe ₂ O ₃	159.69	1.43
Lead	Pb	207.200	PbO ₂	239.20	1.15
Lithium	Li	6.941	Li ₂ O	30.00	2.14
Magnesium	Mg	24.305	MgO	40.31	1.66
Manganese	Mn	54.938	MnO	70.90	1.29
Mercury	Hg	200.590	HgO	216.59	1.08
Molybdenum	Mo	95.940	Mo ₂ O ₅	271.88	1.42
Nickel	Ni	58.700	NiO	74.71	1.27
Phosphorus	P	30.974	P ₂ O ₅	141.94	2.29
Potassium	K	39.098	K ₂ O	94.20	1.20
Selenium	Se	78.960	SeO	127.96	1.61
Silicon	Si	28.086	SiO ₂	60.09	2.14
Silver	Ag	107.868	Ag ₂ O	231.74	1.07
Sodium	Na	22.990	Na ₂ O	61.98	1.35
Strontium	Sr	87.620	SrO	104.00	1.18
Sulfur	S	32.060	SO ₃	80.06	2.50
Thallium	Tl	204.370	Tl ₂ O	424.74	1.04
Tin	Sn	118.690	SnO ₂	150.69	1.27
Titanium	Ti	118.690	TiO ₂	79.90	1.67
Tungsten	W	183.850	WO ₃	231.85	1.26
Vanadium	V	50.941	V ₂ O ₅	181.88	1.79
Zinc	Zn	65.380	ZnO	81.37	1.25

¹Highest Oxidation State.

TRACE METAL ANALYSIS OF WATER SAMPLES

(Approved for N.P.D.E.S.)

1.0 Purpose

1.1 This method may be used for the determination of dissolved, suspended, or total elements in drinking water, surface water, domestic and industrial waste waters.

2.0 References

- 2.1 Methods for Chemical Analysis of Water and Wastes, EPA Manual 600/4-79-020, Method 200.7.
- 2.2 Federal Register, Vol. 51, No. 125, Monday, June 30, 1986, page 23693.

3.0 Summary of Method

3.1 The method describes a technique for the simultaneous or sequential multielement determination of trace elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio-frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines on samples during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interference and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases of line broadening where a background correction measurement would actually degrade the analytical result. The possibility of additional interferences named in 5.1 (and tests for their presence as described in 5.2) should also be recognized and appropriate corrections made.

4.0 Sample Handling and Preservation

4.1 See appropriate digestion procedure

5.0 Interferences

5.1 Several types of interference effects may contribute to inaccuracies in the determination of trace elements.

They can be summarized as follows:

5.1.1 Spectral interferences can be categorized as:

5.1.1.1 Overlap of spectral line from another element;

5.1.1.2 Unresolved overlap of molecular band spectra;

5.1.1.3 Background contribution from continuous or recombination phenomena;

5.1.1.4 Background contribution from stray light from the line emission of high concentration elements.

The first of these effects can be compensated by utilizing a computer correction of the raw data, requiring the monitoring and measurement of the interfering element. The second effect may require the selection of an alternate wavelength. The third and fourth effects can usually be compensated by a background correction adjacent to the analyte line. In addition, users of simultaneous multielement instrumentation must assume the responsibility of verifying the absence of spectral interference from an element that could occur in a sample but for which there is no channel in the instrument array. Listed in Table 2 are some interference effects for the recommended wavelengths given in Table 1. The data in Table 2 are intended for use only as a rudimentary guide for the indication of potential spectral interferences. For this purpose, linear relations between concentration and intensity for the analytes and the interferents can be assumed.

The interference information is expressed at analyte concentration equivalents (i.e. false analyte concentrations) arising from 100 mg/L of the interferent element. The suggested use of this information is as follows: Assume that arsenic (at 193.696 nm) is to be determined in a sample containing approximately 10 mg/L of aluminum. According to Table 2, 100 mg/L of aluminum would yield a false signal for arsenic equivalent to approximately 1.3 mg/L. Therefore, 10 mg/L of aluminum would result in a false signal for arsenic equivalent to approximately 0.13 mg/L. Use caution that other analytical systems may

exhibit somewhat different levels of interference than those shown in Table 2, and that the interference effects must be evaluated for each individual system.

Only those interferents listed were investigated and the blank spaces in Table 2 indicate that measurable interferences were not observed for the interferent concentrations listed in Table 3. Generally, interferences were discernible if they produced peaks or background shifts corresponding to 2.5% of the peaks generated by the analyte concentrations also listed in Table 3.

At present, information on the listed silver and potassium wavelength are not available but it has been reported that second order energy from the magnesium 383.231 nm wavelength interferes with the listed potassium line at 766.491 nm.

5.1.2 Physical interferences are generally considered to be effects associated with the sample nebulization and transport processes. Such properties as change in viscosity and surface tension can cause significant inaccuracies especially in samples which may contain high dissolved solids and/or acid concentrations. The use of a peristaltic pump may lessen these interferences. If these types of interferences are operative, they must be reduced by dilution of the sample and/or utilization of standard addition techniques. Another problem which can occur from high dissolved solids is salt buildup at the tip of the nebulizer. This affects aerosol flow-rate causing instrumental drift. Wetting the argon prior to nebulization, the use of a tip washer, or sample dilution have been used to control this problem. Also, it has been reported that better control of the argon flow rate improves instrument performance. This is accomplished with the use of mass flow controllers.

5.1.3 Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not pronounced with the ICP technique, however, if observed they can be minimized by careful selection of operating conditions (that is, incident power, observation position, and so forth), by buffering of the sample, by matrix matching, and by standard addition procedures.

These types of interferences can be highly dependent on matrix type and the specific analyte element.

6.0 Apparatus

6.1 Inductively Coupled Argon Plasma Spectrometer

6.2 Peristaltic Pump

6.3 10 ml test tubes

6.3.1 Wash in 1 + 1 nitric acid

6.3.2 Rinse with deionized water

6.3.3 Wash in 1 + 1 hydrochloric acid

6.3.4 Rinse in deionized water

6.3.5 Store filled with deionized water.

6.4 Disposable 10 ml. test tubes with teflon lined caps.

7.0 Reagents

7.1 Deionized Water

7.2 Ultrex Nitric Acid

7.3 Ultrex Hydrochloric Acid

7.4 Liquid Argon Supply

7.5 Stock Solutions - commercially available

The following stock solutions are prepared to order by Spex Industries. The names utilized indicate a custom mix for Alabama Power Company. The various solutions contain various analytes of interest.

7.5.1 APC1

7.5.2 APC2

7.5.3 APC3

7.5.4 APC4

7.5.5 APC5

7.6 Analytical Standards

7.6.1 APC1 - deionized water blank

7.6.2 APC2 - dilute 1 ml stock to 100 ml. -> 10 mg/l

7.6.3 APC3 - dilute 1 ml stock to 100 ml. -> 10 mg/l

7.6.4 APC4 - dilute 1 ml stock to 100 ml. -> 10 mg/l

7.6.5 APC5 - dilute 2.5 ml stock to 100 ml. -> 25 mg/l

8.0 Procedure

8.1 System Check-Out

8.1.1 Check for Gas Leaks

8.1.1.1 Turn on the argon, set the pressure regulator to about 2.8 g/cm² (50 lb/sq in.).

8.1.1.2 Flip up the one toggle valve beneath the coolant and sample flow meters.

8.1.1.3 Rotate the control knob beneath the coolant gas flow meter counter clockwise two complete turns to open the gas lines.

- 8.1.1.4 Adjust the coolant knob so that the argon flows at the valve indicated on the log sheet (20 liters/min).
- 8.1.1.5 Check the gas connections to the coolant gas flow meter for leaks.
- 8.1.1.6 Turn off the coolant gas by rotating the coolant knob completely clockwise.
- 8.1.1.7 Rotate the control knob beneath the sample flow meter counter clockwise two complete turns to open the gas lines.
- 8.1.1.8 Check the gas connections to the sample flow meter for leaks.
- 8.1.1.9 Turn off the sample gas by rotating the sample knob completely clockwise.
- 8.1.1.10 Flip up the toggle valve beneath the plasma gas flow meter.
- 8.1.1.11 Rotate the control knob beneath the plasma gas flow meter until the flow registers 1 liter/min.
- 8.1.1.12 Check for gas leaks.
- 8.1.1.13 Turn off the plasma gas by rotating the plasma knob completely clockwise.
- 8.1.1.14 Flip down both toggle valves.
- 8.1.2 Check the Nebulizer
 - 8.1.2.1 Flip up the coolant toggle valve and turn the knob beneath the sample gas flow completely counter-clockwise.
 - 8.1.2.2 Adjust the pressure controller beneath the plasma unit so that the aspirator pressure 287 psi for aqueous.
 - 8.1.2.3 Place a beaker of distilled water on the sample stand and insert the capillary tubing in the water, to aspirate the samples. Set uptake rate to 1.5 to 2.5 ml/min. If the needle is damaged or broken, do not attempt to use it. Return it to Jarrell-Ash for a replacement immediately.
 - 8.1.2.4 If the nebulizer is aspirating at the proper rate, push the teflon holder over the rim of the glass expansion chamber and allow the nebulizer to run for a few minutes to check for leaks around the O-ring seal. (You can often detect a leak by a spot of water beneath the O-ring.)
 - 8.1.2.5 Recheck the torch position.

8.1.3 Check the discharge

8.1.3.1 Push the igniter button and check that a discharge occurs through the argon flow (a buzzing sound). Look inside the chassis to see if the spark grounds to the chassis. If it does, adjust the position of Tesla or reposition torch argon feeds.

8.1.3.2 Operate the Tesla coil at a minimum power by rotating the knurled knob on the base of the Tesla coil counter-clockwise while the ignitor button is depressed until the Tesla no longer discharges. Then rotate the control knob one-half turn clockwise and press the ignitor button to verify that a discharge occurs.

8.2 Profile

NOTE: The spectrometer should be profiled before beginning analyses and checked as conditions warrant.

8.2.1 From main menu select Run ICAP 1100.

8.2.2 From next menu select Operation.

8.2.3 From Operation menu select Analysis and enter Method name.

8.2.4 Select profile

8.2.5 Select manual or automatic profile (Automatic profile can be used to check manual profile)

8.2.6 Follow screen prompts.

8.2.7 When complete select done.

8.2.8 Check that the 1.0 ppm solution of cadmium gives a response on the monitor meter. Scan the image across the entrance slit using the horizontal adjustment of the mirror. Maximize the signal.

8.2.10 Repeat step 26 using the vertical adjust.

8.2.11 Repeat steps 26 and 27 to best settings.

8.3 Standardization

8.3.1 From analysis screen press select standardize.

8.3.2 Aspirate standards and select run and done/keep after each standard.

8.3.3 When complete select done/keep.

8.4 Sample Analysis

8.4.1 Autosampler setup (optional)

8.4.1.1 Ensure switching box is on "A".

8.4.1.2 Enter "IA" to initiate the autosampler.

8.4.1.3 Press ESC until main menu is displayed.

8.4.5.4 Press "S" for setup.

8.4.5.5 Press "A" for autosampler.

8.4.5.6 Enter autosampler name "AS2".

8.4.5.7 Press "F5" to delete the current set.

- 8.4.5.8 Enter "1" to delete set 1.
- 8.4.5.9 Press "F3" to add a set.
- 8.4.5.10 Press "F1" to edit the set.
 - 8.4.5.10.1 Enter "TREL" for the method name.
 - 8.4.5.10.2 Enter default values until rinse time, then enter "60" seconds.
 - 8.4.5.10.3 Enter "#" of sample positions needed. (Count the number of samples on the autosampler tray(s) and enter this number.
 - 8.4.5.10.4 Enter the first sample number under default sample name.
 - 8.4.5.10.5 Press "F4" for *no check under default table check.
 - 8.4.5.10.6 Press "F1" to edit the sample names.
 - 8.4.5.10.7 Press "F9" twice to return to store changes.
- 8.4.5.11 Press "ESC" to get to the main menu.
- 8.4.5.12 Press "O" for operation.
- 8.4.5.13 Press "A" for analysis.
- 8.4.5.14 Enter "TREL" for the method name.
- 8.4.5.15 Press "F1" for analyze.
- 8.4.5.16 Press "F2" to change output.
- 8.4.5.17 Enter "C" for concentration.
- 8.4.5.18 Press "F9" to start the autosampler.
- 8.4.5.19 Press F2 to change output mode to concentration.
- 8.4.5.20 Press C for concentration.
- 8.4.5.21 Press F9.
- 8.4.5.22 Enter (sample number) for sample name.
- 8.4.5.23 Aspirate sample and press F1.
- 8.4.5.24 When analysis is complete rinse aspirator in xylene for - 40 seconds.
- 8.4.5.25 Press F1
- 8.4.5.26 Report steps 5.2.5.4 thru 5.2.5.7 for each sample.

8.5. Shutdown

- 8.5.1 Open torch door to extinguish plasma.
- 8.5.2 Turn automatic power control switch to manual.
NOTE: Wait at least 5 min between steps 3 and 4.
- 8.5.3 Turn argon supply off.
- 8.5.4 Turn coolant water supply off.
- 8.5.5 Remove disks from disk drives and turn terminal off.

9.0 Reporting

9.1 Sorting

- 9.1.1 From analysis menu press **ESC** to get back to Main Menu.
- 9.1.2 From main menu select IMS.
- 9.1.3 Select enable and end.
- 9.1.4 From enable menu select Use System.
- 9.1.5 Select DBMS/Graphics.
- 9.1.6 Select interact.
- 9.1.7 Select sort.
- 9.1.7 Type database name results were stored under e.g. "ANAD2".
- 9.1.8 Under Where prompt type SYS:RECORDS> XXX Note: (The system record should be recorded in the log book for the last batch of samples sent. Use default number)
- 9.1.9 For Field 1 type "ELEM,A".
- 9.2 Transferring
 - 9.2.1 When report is sorted press **ESC**.
 - 9.2.2 Select 9=Report.
 - 9.2.3 Enter database name and form PERPT.
 - 9.2.4 Select Printer (If it is desired to view the report on the monitor at this time select screen.
 - 9.2.5 Select pgdn = next page.
 - 9.2.6 Select File for type of printer interface.
 - 9.2.7 While holding **ALT** down press **F2**.
 - 9.2.8 When screen prompts press **ENTER** to reuse file.
 - 9.2.9 Press **ESC**.
 - 9.2.10 To return to main menu press **Q** for quit then **R** for return.
 - 9.2.11 From the main menu, select exit and quit to DOS.
 - 9.2.12 From master menu select 2. DATA TRANSFER.
 - 9.2.13 When transfer is complete select 3. LABMAN from main menu and enter command OFF.
 - 9.2.14 Select **F1** to get the LABMAN screen.
 - 9.2.15 Select the ICAP menu.
 - 9.2.16 Select file and option. The report is now being read by the main computer.
- 9.0 Quality Control
 - 9.1 Precision
 - 9.1.1 After every tenth sample, reanalyze a high standard. Tolerance is $\pm 10\%$.
 - 9.1.2 On every tenth sample, or if less than ten the last sample, analyze a second aliquot.
 - 9.2 Accuracy
 - 9.2.1 Add 1 ml of APC2, APC3, APC4 and A 100 PPM Solution of APC5 to a third, fourth, fifth and sixth aliquot of the last sample.
 - 9.2.2 Reanalyze and record emission.

9.3 Standards

9.3.1 All samples must be preceded with the intensity of the standards.

9.3.2 Standards must be of the concentration of standards listed in section 7.6.

10.0 Permit Violation Verification

10.1 Standards shall be verified by analysis of a freshly prepared standard at the approximate permit value. Upon verification of standard concentrations, the sample concentration shall be confirmed by analysis of a second aliquot. Confirmed violations should be reported at once.

TABLE 1. Recommended Wavelengths, Estimated Instrumental Detection Limits, and Test Codes

<u>Element</u>	<u>Test Code</u> <u>Total</u>	<u>Test Code</u> <u>Dissolved</u>	<u>Test Code</u> <u>Dissolved</u> <u>Low Level</u>	<u>Test Code</u> <u>Total</u> <u>Low Level</u>	<u>Wavelength</u> <u>nm</u>	<u>Est det</u> <u>limit,</u> <u>ug/L</u>
Aluminum	1300				308.215	45
Arsenic	1320	1321	1323	1322	193.696	53
Antimony	1310				206.833	32
Barium	1340	1341			455.403	2
Beryllium	1350				313.042	0.3
Boron	1360				249.774	5
Cadmium	1380	1381	1383	1382	226.502	4
Calcium	1390				317.933	10
Chromium	1420	1421	1423	1422	267.716	7
Cobalt	1430				228.616	7
Copper	1440	1441	1443	1442	324.754	6
Iron	1470	1471	1473	1472	259.940	7
Lead	1480	1481			220.353	42
Magnesium	1500	1501			279.079	30
Manganese	1510	1511			257.610	2
Molybdenum					202.030	8
Nickel	1530	1531			231.604	15
Selenium	1580	1581	1583	1582	196.026	75
Silica (SiO ₂)	1591				288.158	58
Silver	1600	1601			328.068	7
Sodium	1610				588.995	29
Thallium					190.864	40
Vanadium	1660				292.402	8
<u>Zinc</u>	<u>1670</u>	<u>1671</u>			<u>213.856</u>	<u>2</u>

Note 1: The wavelengths listed are recommended because of their sensitivity and overall acceptance. Other wavelengths may be substituted if they can provide the needed sensitivity and are treated with the same corrective techniques for spectral interference. (See 5.1.1.).

Note 2: The estimated instrumental detection limits as shown are taken from "Inductively Coupled plasma-Atomic Emission Spectroscopy-Prominent Lines," EPA-600/4-79-017. They are given as a guide for an instrumental limits. The actual method detection limits are sample dependent and may vary as the sample matrix varies.

TABLE 2. Analyte Concentration Equivalents (mg/L) Arising From Interferents at the 100 mg/L

Analyte	Wavelength, nm	Interferent									
		Al	Ca	Cr	Cu	Fe	Mg	Mn	Ni	Ti	V
Al	308.215	--	--	--	--	--	--	0.21	--	--	1.4
An	206.833	50.47	--	2.9	--	0.08	--	--	--	0.25	0.45
Ar	193.696	1.3	--	0.44	--	--	--	--	--	--	1.1
Ba	455.403	--	--	--	--	--	--	--	--	--	--
Be	313.042	--	--	--	--	--	--	--	--	0.04	0.05
Bo	249.774	0.04	--	--	--	0.32	--	--	--	--	--
Cd	226.502	--	--	--	--	0.03	--	--	0.02	--	--
Ca	317.933	--	--	0.08	--	0.01	0.01	0.04	--	0.03	0.03
Cr	267.716	--	--	--	--	0.003	--	0.04	--	--	0.04
Co	228.616	--	--	0.03	--	0.005	--	--	0.03	0.15	--
Cu	324.754	--	--	--	--	0.003	--	--	--	0.05	0.02
Fe	259.940	--	--	--	--	--	--	0.12	--	--	--
Pb	220.353	0.17	--	--	--	--	--	--	--	--	--
Mg	279.079	--	0.02	0.11	--	0.13	--	0.25	--	0.07	0.12
Mn	257.610	0.005	--	0.01	--	0.002	0.002	--	--	--	--
Mo	202.030	0.05	--	--	--	0.03	--	--	--	--	--
Ni	231.604	--	--	--	--	--	--	--	--	--	--
Se	196.026	0.23	--	--	--	0.09	--	--	--	--	--
Si	288.158	--	--	0.07	--	--	--	--	--	--	0.01
Na	588.995	--	--	--	--	--	--	--	--	0.08	--
Th	190.864	0.30	--	--	--	--	--	--	--	--	--
V	292.402	--	--	0.05	--	0.005	--	--	--	0.02	--
Zn	213.856	--	--	--	0.14	--	--	--	0.29	--	--

TABLE 3. Interferent and Analyte Elemental Concentrations Used for Interference
Measurements in Table 2.

Analytes	(mg/L)	Interferents	(mg/L)
Al	10	Al	1000
As	10	Ca	1000
B	10	Cr	200
Ba	1	Cu	200
Be	1	Fe	1000
Ca	1	Mg	1000
Cd	10	Mn	200
Co	1	Ni	200
Cr	1	Ti	200
Cu	1	V	200
Fe	1		
Mg	1		
Mn	1		
Mo	10		
Na	10		
Ni	10		
Pb	10		
Sb	10		
Se	10		
Si	1		
Tl	10		
V	1		
Zn	10		

Mercury in Coal

Mercury, Dry 4520
Mercury, A.D. 4521
Mercury, A.R. 4522

1.0 Purpose

This procedure is for the determination of mercury content in coal.

2.0 References

- 2.1 Trace Elements in Fuel, American Chemical Society, Suresh P. Babu, 1975
- 2.2 American Society for Testing Materials, Method D3684, Total Mercury in Coal by the Oxygen Bomb Combustion Method.
- 2.3 Laboratory Guidelines and Procedures: Trace Elements in Coal, Volume 5: Analytic Procedures for Trace Elements., EPRI, November 1990.

3.0 Summary

A one gram sample is burned in an oxygen bomb calorimeter that has a nitric acid collection solution added. The bomb is left undisturbed for ten minutes. Mercury is analyzed using flameless atomic absorption.

4.0 Sample Handling and Preservation

5.0 Interferences

Principle interferences are referenced in ICAP procedure given in section Water.

6.0 Apparatus

- 6.1 Atomic Absorption Spectrophotometer
- 6.2 Mercury Hydride System
- 6.3 Mercury Hollow Cathode Tube
- 6.4 Oxygen bomb
- 6.5 Quartz liner for oxygen bomb
(Liner is required only when chromium and nickel are to be determined by this method.)

7.0 Safety Precautions

- 7.1 Safety glasses, gloves, and labcoat should be worn at all times.
- 7.2 Oxygen bomb pressure is at 24 atmospheres.
- 7.3 Analyst should be familiar with safety data sheets of all reagents used.

8.0 Reagents

- 8.1 Nitric acid solution, 10%

- 8.2 Oxygen bomb pressure is at 24 atmospheres. All safety precautions associated with pressure bombs should be followed.
- 8.3 Stock solutions for all mercury analysis is given in mercury procedure listed in water section.

9.0 Procedure

- 9.1 Place 10 ml. 10% nitric acid in an oxygen bomb.
- 9.2 Place approximately one gram (weighed to the nearest 0.1 mg) coal in the bomb crucible. Attach fuse wire.
- 9.3 Assemble the bomb and pressurize to 24 atmospheres.
- 9.4 Submerge bomb in water and ignite sample.
Note: Bomb may be placed in sink for ignition. Verify that there are no leaks from the bomb before firing. Should leakage be noted, discontinue the test. Repair the bomb and start over with another test sample.
- 9.5 Allow the sample to remain undisturbed for at least 10 minutes.
- 9.6 Remove the bomb from the bath, open and transfer the contents to a 100 ml. volumetric flask. Wash the bomb with small portions of 10% nitric acid. Collect all washings in the volumetric flask.
- 9.7 Dilute to volume with 10% nitric acid.
- 9.8 Analyze per procedure 2-4.1 in water section. Method is Mercury in Water.

10.0 Calculations

10.1 As determined concentration

$$\text{Metal, mg/kg} = A * B * 1000 / C$$

Where:

A= concentration of parameter in
aqueous sample in mg/kg
B= volume aqueous sample in liters
C= weight coal sample in grams

10.2 Dry basis

$$\text{Metal, mg/kg} = Hg_{AD} * 100 / (100 - M)$$

Where:

Hg_{AD} = as determined mercury concentration
M = 60 mesh moisture

10.3 As received basis

$$\text{Metal, mg/kg} = Hg_{AD} * 100 - TM / 100$$

Where:

Hg_{AD} = as determined mercury concentration
 TM = 60 mesh moisture

11.0 Quality Control

11.1 Precision

On every tenth sample or if less than ten, the last sample, analyze a second aliquot.

11.2 Accuracy

Analyze a mercury in coal standard to assure proper digestion, calibration, and analysis.

Test Codes
Chlorine Dry 4410
A.D. 4411
A.R. 4412
Fluorine Dry 4450
A.D. 4451
A.R. 4452

Chlorine and Fluorine in Coal

1.0 Purpose

This procedure may be used for the determination of total chlorine and fluorine content of coal.

2.0 References

- 2.1 ASTM Method D3761, "Total fluorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method.
- 2.2 ASTM Method D5085, "Standard Test Method for the Determination of Chloride, Nitrate, and Sulfate in Atmospheric Wet Deposition by Chemically Suppressed Ion Chromatography."
- 2.2 Trace Elements in Fuel, American Chemical Society series 141, August 30, 1973.
- 2.3 ASTM Method D4208, "Test Method for Total Chlorine in Coal by the Oxygen Bomb/ Ion Selective Electrode Method."

3.0 Summary

A one quarter gram sample of pulverized coal is burned in a tube furnace with oxygen purge. The oxygen is passed through a sodium carbonate scrubber solution. The resulting solution is analyzed by ion chromatography.

4.0 Sample Handling and Preservation

- 4.1 Every precaution should be taken to prevent contamination of the test sample. Gloves should be worn during sample preparation.
- 4.2 Polished Distilled Water should be used for all reagent preparation and washings.
- 4.3 All surfaces that will be exposed to the coal or released gases should be preignited to prevent contamination.

5.0 Interferences

Principle interferents are referenced in Ion Chromatography procedure given in section "Water."

6.0 Apparatus

- 6.1 Ion Chromatograph
- 6.2 Tube furnace
- 6.3 Ceramic combustion boats.

- 6.4 Polycarbonate sample bottle, 125 ml.
- 6.5 Oxygen cylinder and related regulators.
- 7.0 Safety Precautions
 - 7.1 Safety glasses and labcoat should be worn at all times.
 - 7.2 Analyst should be familiar with safety data sheets of all reagents used.
 - 7.3 Extremely high temperatures are used in the procedure. Care must be exercised to prevent burns.
- 8.0 Reagents
 - 8.1 Sodium Carbonate
 - 8.2 Oxygen
- 9.0 Procedure

Note: Combustion boats should be ignited prior to use.

 - 9.1 Set furnace temperature to 1200° C. Allow temperature to stabilize.
 - 9.2 Place approximately 75 ml. deionized water in a 125 ml. polycarbonate flask.
 - 9.3 Set oxygen flow such that bubbler does not splash sample from container.
 - 9.4 Place 0.1 gm. sodium carbonate in 125 ml. polycarbonate bottle.
 - 9.5 Weigh container weight to nearest gram.
 - 9.6 Add approximately 75 grams water to a 125 ml. polycarbonate bottle..
 - 9.7 Place 0.25 grams (weighed to the nearest 0.1 gram) pulverized coal in a furnace ignition boat.
 - 9.8 Place bubbler in carbonate solution.
 - 9.9 Slide boat into furnace heated zone.
 - 9.10 Allow to purge for 3 minutes.
 - 9.11 Analyze per procedure Water 3-5.2 or other approved methods listed in water section.
- 10.0 Calculations
 - 10.1 As determined concentration

$$\text{Halogen, mg/kg} = A * B * 1000 / C$$

Where:

A= concentration of parameter in
aqueous sample in mg/l

B= volume aqueous sample in liters (100 ml.)

C= weight of coal test sample in grams (0.25 g)

10.2 Dry basis

$$\text{Halogen, mg/kg} = Cl_{AD} * 100 / (100 - M)$$

Where:

Cl_{AD} = as determined halogen concentration
 M = 60 mesh moisture in test sample in %

10.3 As received basis

$$\text{Halogen, mg/kg} = Cl_{\text{DRY}} * (100 - TM) / 100$$

Where:

Cl_{DRY} = dry basis halogen concentration
 TM = % Total Moisture of laboratory sample

11.0 Quality Control

11.1 Precision

On every tenth sample or if less than ten, the last sample, analyze a second aliquot.

11.2 Accuracy

Following the duplicate, analyze an NBS standard to assure proper recovery.

ARSENIC IN COAL AA SLURRY

- 1.0 Purpose
This method covers the determination of arsenic in coal samples.
- 2.0 References
 - 2.1 USS - 100 Perkin Elmer Manual
 - 2.2 Rapid slurry analysis of solid coal and fly ash samples - paper by Deborah Bradshaw and Walter Slavin.
 - 2.3 Fast furnace analyses and slurry sampling - paper by W. Slavin, N. J. Miller-Ihli and G. R. Carnrick.
- 3.0 Summary
60 mesh coal is weighed into autosampler cups and then analyzed using a slurry sampler on the furnace atomic absorption spectroscopy.
- 4.0 Sample Handling and Preservation
- 5.0 Interferences
 - 5.1 There are no observed interferences in either sampling or analysis.
- 6.0 Apparatus
 - 6.1 Atomic absorption spectrophotometer equipped with a furnace. Perkin Elmer 4100 ZL.
 - 6.2 USS-100 slurry attachment to the 4100 ZL.
 - 6.3 EDL Arsenic lamp with power supply.
 - 6.4 Polyethylene autosampler cups
- 7.0 Safety Precautions
 - 7.1 Since metallic elements and other toxic substances are vaporized and vented into the atmosphere during graphite furnace operation, it is imperative that a fume hood be used.
 - 7.2 Analyst should be familiar with safety data sheets of all reagents used in this procedure.
- 8.0 Reagents
 - 8.1 Liquid argon supply
 - 8.2 Triton X-100 surfactant
 - 8.3 Nitric Acid, concentrated
 - 8.4 Diluent solution
 - 8.4.1 Place 500 ml. deionized water in a 1000 ml. volumetric flask.
 - 8.4.2 Add 50 ml. conc. Nitric Acid.
 - 8.4.3 Add 50 ul. Triton X-100
 - 8.4.4 Dilute to volume with deionized water.

- 8.3 Arsenic standard, 1000 mg/l.
 - 8.3.1 Commercially available stock standard.
- 8.4 Intermediate stock, 10 mg/l.
 - 8.4.1 Place 50 ml. deionized water in a 100 ml. volumetric flask.
 - 8.4.2 Add 1 ml. Nitric Acid.
 - 8.4.3 Add 1 ml. Arsenic stock standard.
 - 8.4.4 Dilute to volume with deionized water.
- 8.5 Working standard, 0.100 mg/l (0.1 ppm).
 - 8.5.1 Place 50 ml. deionized water in a 100 ml. volumetric flask.
 - 8.5.2 Add 1 ml. Nitric Acid.
 - 8.5.3 Add 1 ml intermediate standard stock.
 - 8.5.4 Dilute to volume with deionized water.

Note: The 4100 ZL instrument utilizes this working standard to produce additional working standards. These additional standards are 0.020 ppm, 0.050 ppm, 0.080 ppm and 0.100 ppm.

9.0 Procedure

- 9.1 Sample preparation
 - 9.1.1 For the coal digestion, place 0.005g coal into a polyethylene autosampler cup.
 - 9.1.2 Put 1 ml of diluent solution into each vial. Do not place diluent solution in standards.
- 9.2 Instrument preparation
 - 9.2.1 Set bypass/USS100 to USS100.
 - 9.2.2 Vibro cell settings.
 - 9.2.2.1 Power on.
 - 9.2.2.2 Output Control - 20
 - 9.2.2.3 25 - 50 setting on 25
 - 9.2.2.4 15 seconds for mix time seconds box.
 - 9.2.3 Turn power on to 4100 ZL AA, terminal, screen, and printer.
- 9.3 Turn on Power supply for EDL lamp, push reset button, and set to 400mA.
- 9.4 Insert As EDL lamp into turret 2.
- 9.5 Insert As labeled pin into HCL connector panel 2.
- 9.6 Using mouse, select menu "AA Inst Execute". Note: To select a menu, double click on mouse.
- 9.7 Wait for icons to illuminate and select "Auto"
- 9.8 Select "Element File".
- 9.9 Select "As Slur. Gel"
- 9.10 Select "Untitled" to bring up file name. Select "As Air".

- 9.11 From windows select and modify "ID/WT FILE" as follows:
- 9.11.1 Set sample volume to 1.0.
 - 9.11.2 Set nominal wt. to 0.005
 - 9.11.3 List diluent in position 0.
 - 9.11.4 List standard in position 1.
 - 9.11.5 Enter sample number and weight (in grams) in succeeding positions beginning with position 2.
 - 9.11.6 Save file by using windows menu "save as..."
File should be saved using the Julian date as a file name.
- 9.12 From windows menu, select "lamp alignment" to check lamp energy. Lamp energy should be between 60 to 70. Use mouse to select "AGC/AIC" to update lamp energy reading.
- 9.13 Select "Read" and "Autozero" on keyboard.
- 9.14 Select "HGA/FIAS Read" on keyboard.
- 9.15 From windows select "Calibration" and "Set Fixed Exp". Set expansion to 1 and enter.
- 9.16 Select "Calibrate" to run standards
- 9.17 After standards are run select "Run Samples".

10.0 Instrument parameters

- 10.1 Wavelength 193.7
- 10.2 Slit 0.7 nm low
- 10.3 Dry
- 10.3.1 Temperature 100° C
 - 10.3.2 Ramp time 1 second
 - 10.3.3 Hold time 20 seconds
 - 10.3.4 Gas flow 250
- 10.4 Dry
- 10.4.1 Temperature 130° C
 - 10.4.2 Ramp time 3 seconds
 - 10.4.3 Hold time 20 seconds
 - 10.4.4 Gas flow 250
- 10.5 Char
- 10.5.1 Temperature 200° C
 - 10.5.2 Ramp time 3 seconds
 - 10.5.3 Hold time 10 seconds
 - 10.5.4 Gas flow 250
- 10.6 Atomize
- 10.6.1 Temperature 2000° C
 - 10.6.2 Ramp time 0 seconds
 - 10.6.3 Hold time 5 seconds
 - 10.6.4 Gas flow 0

10.7 Purge

10.7.1 Temperature 2300° C
10.7.2 Ramp time 1 second
10.7.3 Hold time 2 seconds
10.7.4 Gas flow 250

10.8 Autosampler injection volume 23 ul

11.0 Calculations

11.1 Manual

$As, \text{mg/kg} = [As] * 0.001 / \text{sample weight}$

11.2 Auto

If weights and volumes were entered into the "ID/WT." file, enter data as listed on the printout.

12.0 Quality Control

12.1 After every ten samples or if less than ten after last sample, analyze a NIST 1632b coal standard. Five milligrams of coal standard is weighed out.

SELENIUM IN COAL AA SLURRY

- 1.0 Purpose
This method covers the determination of selenium in coal samples.
- 2.0 References
 - 2.1 USS - 100 Perkin Elmer Manual
 - 2.2 Rapid slurry analysis of solid coal and fly ash samples - paper by Deborah Bradshaw and Walter Slavin.
 - 2.3 Fast furnace analyses and slurry sampling - paper by W. Slavin, N. J. Miller-Ihli and G. R. Carnrick.
- 3.0 Summary
60 mesh coal is weighed into autosampler cups and then analyzed using a slurry sampler on the furnace atomic absorption spectroscopy.
- 4.0 Sample Handling and Preservation
- 5.0 Interferences
 - 5.1 There are no observed interferences in either sampling or analysis.
- 6.0 Apparatus
 - 6.1 Atomic absorption spectrophotometer equipped with a furnace. Perkin Elmer 4100 ZL.
 - 6.2 USS-100 slurry attachment to the 4100 ZL.
 - 6.3 EDL Selenium lamp with power supply.
 - 6.4 Polyethylene autosampler cups
- 7.0 Safety Precautions
 - 7.1 Since metallic elements and other toxic substances are vaporized and vented into the atmosphere during graphite furnace operation, it is imperative that a fume hood be used.
 - 7.2 Analyst should be familiar with safety data sheets of all reagents used in this procedure.
- 8.0 Reagents
 - 8.1 Liquid argon supply
 - 8.2 Triton X-100 surfactant
 - 8.3 Nitric Acid, concentrated
 - 8.4 Diluent solution
 - 8.4.1 Place 500 ml. deionized water in a 1000 ml. volumetric flask.
 - 8.4.2 Add 50 ml. conc. Nitric Acid.
 - 8.4.3 Add 50 ul. Triton X-100
 - 8.4.4 Dilute to volume with deionized water.

- 8.3 Selenium standard, 1000 mg/l.
 - 8.3.1 Commercially available stock standard.
- 8.4 Intermediate stock, 10 mg/l.
 - 8.4.1 Place 50 ml. deionized water in a 100 ml. volumetric flask.
 - 8.4.2 Add 1 ml. Nitric Acid.
 - 8.4.3 Add 1 ml. Selenium stock standard.
 - 8.4.4 Dilute to volume with deionized water.
- 8.5 Working standard, 0.100 mg/l (0.1 ppm).
 - 8.5.1 Place 50 ml. deionized water in a 100 ml. volumetric flask.
 - 8.5.2 Add 1 ml. Nitric Acid.
 - 8.5.3 Add 1 ml intermediate standard stock.
 - 8.5.4 Dilute to volume with deionized water.

Note: The 4100 ZL instrument utilizes this working standard to produce additional working standards. These additional standards are 0.020 ppm, 0.050 ppm, 0.080 ppm and 0.100 ppm.

9.0 Procedure

- 9.1 Sample preparation
 - 9.1.1 For the coal digestion, place 0.005g coal into a polyethylene autosampler cup.
 - 9.1.2 Put 1 ml of diluent solution into each vial. Do not place diluent solution in standards.
- 9.2 Instrument preparation
 - 9.2.1 Set bypass/USS100 to USS100.
 - 9.2.2 Vibro cell settings.
 - 9.2.2.1 Power on.
 - 9.2.2.2 Output Control - 20
 - 9.2.2.3 25 - 50 setting on 25
 - 9.2.2.4 15 seconds for mix time seconds box.
 - 9.2.3 Turn power on to 4100 ZL AA, terminal, screen, and printer.
- 9.3 Turn on Power supply for EDL lamp, push reset button, and set to 400mA.
- 9.4 Insert Se EDL lamp into turret 2.
- 9.5 Insert Se labeled pin into HCL connector panel 2.
- 9.6 Using mouse, select menu "AA Inst Execute". Note: To select a menu, double click on mouse.
- 9.7 Wait for icons to illuminate and select "Auto"
- 9.8 Select "Element File".
- 9.9 Select "Se Slur. Gel"
- 9.10 Select "Untitled" to bring up file name. Select "As Air".

- 9.11 From windows select and modify "ID/WT FILE" as follows:
- 9.11.1 Set sample volume to 1.0.
 - 9.11.2 Set nominal wt. to 0.005
 - 9.11.3 List diluent in position 0.
 - 9.11.4 List standard in position 1.
 - 9.11.5 Enter sample number and weight (in grams) in succeeding positions beginning with position 2.
 - 9.11.6 Save file by using windows menu "save as..."
File should be saved using the Julian date as a file name.
- 9.12 From windows menu, select "lamp alignment" to check lamp energy. Lamp energy should be between 60 to 70. Use mouse to select "AGC/AIC" to update lamp energy reading.
- 9.13 Select "Read" and "Autozero" on keyboard.
- 9.14 Select "HGA/FIAS Read" on keyboard.
- 9.15 From windows select "Calibration" and "Set Fixed Exp". Set expansion to 1 and enter.
- 9.16 Select "Calibrate" to run standards
- 9.17 After standards are run select "Run Samples".

10.0 Instrument parameters

- 10.1 Wavelength 196.0
- 10.2 Slit 2.0 nm low
- 10.3 Dry
- 10.3.1 Temperature 100° C
 - 10.3.2 Ramp time 1 second
 - 10.3.3 Hold time 20 seconds
 - 10.3.4 Gas flow 250
- 10.4 Dry
- 10.4.1 Temperature 130° C
 - 10.4.2 Ramp time 3 seconds
 - 10.4.3 Hold time 30 seconds
 - 10.4.4 Gas flow 250
- 10.5 Char
- 10.5.1 Temperature 200° C
 - 10.5.2 Ramp time 3 seconds
 - 10.5.3 Hold time 10 seconds
 - 10.5.4 Gas flow 250
- 10.6 Atomize
- 10.6.1 Temperature 1600° C
 - 10.6.2 Ramp time 0 seconds
 - 10.6.3 Hold time 5 seconds
 - 10.6.4 Gas flow 0
 - 10.6.5 Read *

10.7 Purge

10.7.1 Temperature 2300° C
10.7.2 Ramp time 1 second
10.7.3 Hold time 2 seconds
10.7.4 Gas flow 250

10.8 Autosampler injection volume 23 ul

11.0 Calculations

11.1 Manual

Se, mg/kg = [Se] * 0.001 / sample weight

11.2 Auto

If weights and volumes were entered into the "ID/WT."
file, enter data as listed on the printout.

12.0 Quality Control

12.1 After every ten samples or if less than ten after last
sample, analyze a NIST 1632b coal standard. Five
milligrams of coal standard is weighed out.

Iowa State University

ANALYTICAL PROCEDURES FOR THE DETERMINATION OF CHLORINE AND FLUORINE IN COAL

Chlorine

One gram samples of coal were combusted in a bomb as described in ASTM D4208-88 Option A procedure. After combustion, the pressure in the bomb was released more slowly than required by the ASTM procedure. A fifteen minute release time was used. During release of pressure from selected samples, the gases were bubbled through a silver nitrate solution to assure that no chloride was aspirated from the bomb. After the pressure was released, the bomb was opened and its contents washed into a beaker with approximately 80 ml deionized distilled water. This solution was brought to a boil, cooled and diluted to exactly 100 ml with deionized distilled water. Samples of these solutions were then analyzed by ion chromatography for chloride using a Dionex 2020i instrument, standard Dionex anion exchange columns, and a carbonate/bicarbonate eluent. Peak areas were determined using a 4270 integrator and concentrations calculated based on comparison with a standard calibration curve.

Several procedures were used to check the accuracy of the determinations. First, an NBS standard coal sample was analyzed several times during the course of this work. This sample does not have a certified chlorine value, and we typically obtained recoveries of about 60 percent of the non-certified chlorine content (Alabama also obtains low recoveries on this standard - chlorine apparently has been lost during size reductions prior to distribution of the sample). Second, known amounts of chloride were added to the bomb during combustion and to in other cases to the solutions during analyses. We typically obtained recoveries of 95 to 105 percent of these internal standards. The boiling step adopted after combustion led to increased resolution of the chloride peak and reduced uncertainties in the measurement of its area, but did not appear to have any effect on the area of peaks.

We also evaluated all results on the basis of whether or not they were reasonable. With the Pittsburgh coal, the raw coal, feed coal, product coal and tailing were found to contain approximately the same levels of chlorine, which was what we would have expected. Different feed and product coal samples also had about the same chlorine levels, which was what we expected. There was a large difference in the chlorine content of two to the Pittsburgh tailing samples, which we attributed to sample inhomogeneity.

Fluorine

The procedures for combustion and analysis of combusted sampled for fluorine was identical to that for chlorine except for the different absorbing solutions used in the bomb as specified in ASTM D3761-91 Option A procedure.

There is no NBS standard for fluorine, but we did obtain recoveries of 90 to 110 percent of standard additions of fluoride to the bomb.

In contrast with chlorine results, there were some significant differences amount different feed, raw, and product coals. In the case of the Pittsburgh coal, all the raw, feed and product coals except one were found to contain about 80 to 100 ppm fluorine. That one exception was a feed coal found to contain 40 ppm. However, results obtained by Alabama exhibited just as large of differences. Alabama found from 50 to 80 parts per million fluoride in product and feed coal samples, but only 17 ppm in the raw coal.

Arsenic and Selenium

Dissolutions

Dissolutions were performed in accordance with procedures specified in ASTM Method 4606, with the exceptions listed below.

- Samples were vacuum-dried at room temperature for 20 to 24 hours, rather than air drying.
- Analyses were performed using the vacuum dried samples rather than performing a moisture determination on a separate air-dried sample and then correcting analytical values for the moisture content.
- Cleaning of glassware was performed by washing with soap and water followed by leaching with ten percent HNO_3 for several days and then rinsing with water. ASTM specifies using ten percent HCl for the acid leaches.
- A one-gram sample of coal was mixed with two grams of Eschka mixture (rather than the specified 1.5 grams of Eschka) and was then covered with an additional three grams of Eschka mixture (rather than the specified 1.5 grams of additional Eschka).
- Solutions for analysis were stored in glass volumetric flasks rather than the specified polypropylene flasks.
- Blank solutions were prepared by repeating the exact dissolution procedures without any sample. ASTM procedures specify that blanks be prepared by scaling everything up by a factor of five.

Analyses

Analyses were performed using conventional hydride generation atomic absorption techniques. However, the analytical procedures described in the ASTM method were not used. Instead, recommended procedures from the instrument manufacturer were employed.

For arsenic, 0.75 ml of sample solution and 40 ml of 1.5 percent HCl were added to the reaction flask. A solution of three percent NaOH_4 in one percent NaOH was then introduced into the reaction flask, and arsenic was subsequently determined at a wavelength of 193.7 mm.

For selenium, five ml of sample solution plus five ml of concentrated HCl were heated on a hot plate at 60 to 90°C for 45 minutes. The solutions were then cooled and diluted to ten ml in a volumetric flask. Three ml of that diluted solution and 37 ml of water were then added to the reaction flask. A solution of three percent NaOH_4 in one percent NaOH was then introduced into the reaction flask, and selenium was subsequently determined at a wavelength of 196.0 nm.

Mercury

Dissolutions

Mercury dissolutions were performed using a combustion bomb in accordance with procedures specified in ASTM Method 3684, with the exceptions listed below.

- All glassware was leached in ten percent HNO_3 for several days. ASTM does not specify the acid leaching procedure.
- Fused silica crucibles were used instead of quartz crucibles.
- Solutions for analysis were transferred to glass volumetric flasks rather than the specified biochemical oxygen demand bottles.
- KMnO_4 was added to the final sample solutions until the permanganate color persisted for days, which took only a few drops. The ASTM procedure states that the color should persist for 60 seconds.

Analyses

Analyses were performed using conventional cold vapor atomic absorption techniques. However, the ASTM procedure involving the use of hydroxylamine hydrochloride and stannous chloride solutions was not used. Instead, recommended analytical procedures from the instrument manufacturer were employed. This procedure involved adding ten ml of sample solution and 600 microliters of 1.1 HNO_3 to the reaction flask, adding KMnO_4 if the solutions were not already purple, and then introducing three percent NaOH_4 in a one percent solution of NaOH into the reaction flask. Determinations were made at a wavelength of 253.6 nm.

ANALYTICAL PROCEDURES FOR THE DETERMINATION OF Ba, Be, Co, Cr, Mn, Ni, Zn, Pb, AND Cd - Robert Dawson, Ames Laboratory, Ames IA, February 25, 1993

To obtain representative samples, the raw and feed coal samples were riffled using a rotating riffler into 16 representative samples, then recombined into eight samples of about 50 grams each. One of these aliquots was then ground using a corundum shatter box (to avoid trace metal contamination) and riffled using a rotating riffler into eight portions. All other samples were riffled using a rotating riffler into representative samples of about 5 grams each. Sample bottles and riffler parts were first acid washed with 1+1 HNO₃ (one part water to one part acid) to remove any adsorbed trace metals. The acid wash was followed by successive rinsings with deionized-distilled water.

For the analyses of the above elements, the samples were first ashed following ASTM method D3683. Approximately 6 g of sample were ashed in open 50-ml fused silica crucibles. The crucibles were placed in a cold muffle furnace, set to attain 300°C in about 1 h, and then 500°C for an additional 2 h. The samples were stirred each hour until no carbonaceous materials remained. After cooling and weighing, the samples were finely ground in a clean agate mortar and then reignited at 500°C for 1 h, cooled, and reweighed to calculate the percent ash. Ashing was done in triplicate to determine the precision of analyses.

The ashed samples were digested and concentrated by heating hydrofluoric and nitric acids in teflon crucibles. HCl was not used to avoid interferences during graphite furnace atomic absorption spectroscopy. The method used was an extension of the rapid method of Shapiro (1975) with several modifications. The sample is digested with HF and HNO₃ employing multiple evaporations. The resulting clear solution is diluted to 50 ml with 5% nitric acid. This method yields values for elemental concentrations of equal or better quality to the values obtained by other "rapid" techniques with a considerable savings of time and effort. The exceptions are the alkali metals (Na, K, Li, etc.) which may be more accurately and precisely determined using other instrumental methods. The method and references follow this summary.

Digested samples were analyzed as described below. The elemental composition of the coal was determined by "back-calculation" using the percentage of ash in the original coal samples. The accuracy of all analytical techniques was monitored using standards, run as unknowns, and the analyzed values were compared to published values. The standards used were NIST flyash and coal standards and US Geologic Survey silicate rock standards. These

were used in addition to coal standards because coal standards have to first be ashed then digested, necessitating a further QA check.

The trace elements Ba, Be, Co, Cr, Mn, Ni, and Zn were determined by inductively coupled plasma - atomic emission spectroscopy (ICP-AES). The instrument used was an ARL 3410 ICP Spectrometer. The lines used were as follows:

<u>Element</u>	<u>Wavelength (nm)</u>
Ba	455.353
Be	313.113
Co	236.355 (found Fe interference) 231.138
Cr	267.677
Mn	293.284
Ni	227.000
Zn	213.833

Supporting information for the ICP accompanies this summary.

Lead and cadmium (Pb and Cd) were determined using graphite furnace atomic absorption spectroscopy (Perkin-Elmer 5000) on digested samples. Lead was determined at a wavelength of 283.3 nm and Cd at 228.8 nm. The slit was set at "Low 0.7 nm". All samples were hand injected with 20 microliters of sample as received. Ten microliters of PO₄ was added to Pb determinations but was omitted from Cd determinations for reasons of reproducibility. Rather than adding PO₄, the "charring temperature" was reduced to 500°C. Also, peak area was used rather than peak height. Difficulty was found with the read-out peak sometimes becoming negative. This was resolved by shortening the read time from 5 to 1.8 seconds. Heat up, charring, atomization, and clean-out temperatures are given below.

Cadmium

<u>Step Temperature</u>	<u>Ramp Time (sec)</u>	<u>Hold Time (sec)</u>
Step 1 = 120°	2	25
Step 2 = 700°	20	20
Step 3 = 1600°	0	5
Step 4 = 2700°	1	5

Lead

<u>Step Temperature</u>	<u>Ramp Time (sec)</u>	<u>Hold Time (sec)</u>
Step 1 = 120°	2	25
Step 2 = 500°	20	20
Step 3 = 1700°	0	5
Step 4 = 2700°	1	3

Notes regarding the operation of the graphite furnace are attached.

Conclusions: Based on analyses of the standards run as unknowns, I think most of the results within 10% relative error. We seem to have a problem with nickel being low in the tailings samples. This may have been due to iron interference. Overall I think there was good agreement, for these elements, between the laboratories involved in this study.

DEPARTMENT OF GEOLOGICAL AND ATMOSPHERIC SCIENCES
IOWA STATE UNIVERSITY
ANALYTICAL GEOCHEMISTRY LABORATORY

RECOMMENDED ANALYTICAL PROCEDURE # WR-2

Revision: 0

Date: 4/1/92

Supersedes: This is the original issuance.

Title: Procedure for the determination of major, minor and selected trace elements in silicate and carbonate rocks.

1. Scope

This procedure describes the digestion of silicate rock powders and the manipulation of the resulting solutions in preparation for determination of major element constituents (except SiO_2 , Al_2O_3 , FeO , H_2O and CO_2) and selected trace elements (not including the REE) by ICP spectroscopy.

2. Summary of Method

This method is an extension of the rapid method of Shapiro, 1975, with several modifications. This method commonly uses HCl for the digestions; although, for the trace elements in coal study we substituted nitric acid for HCl. This was done so the digested samples could be analyzed using both ICP and graphite furnace atomic absorption (GFAA). HCl tends to interfere with GFAA. The sample is digested with HF and Nitric acid, employing multiple evaporations. The resulting clear solution is diluted to 100ml with 5% Nitric acid, yielding a stock solution which may be further diluted for analysis. Elemental concentrations are determined using the ICP-AES spectrophotometer, using international silicate reference samples or high-purity elemental solutions as standards.

3. Significance

This method yields values for elemental concentrations of equal or better quality to the values obtained by other "rapid" techniques with a considerable savings of time and effort. The exceptions are the alkali metals (Na, K, Li, etc.) which may be more accurately and precisely determined using other instrumental methods.

4. Interferences

There are no spectral interferences for any of the major elements at the wavelengths currently used. Spectral interferences for other elements (notably Ti on V and Fe on Co) have been investigated and are corrected empirically (see discussion below).

5. Apparatus

- a. ICP spectrophotometer
- b. 100ml Teflon beakers
- c. Teflon watch glasses to fit beakers
- d. Hot plate with bead bath
- e. Analytical balance
- f. Wash bottle
- g. Teflon stirring rod
- h. 25ml plastic graduated cylinder
- i. 10ml pipette
- j. 100ml volumetric flask with stopper

k. 125ml Nalgene bottles.

6. Reagents and materials

- a. Concentrated HF (HNO_3). Reagent grade or in-house distilled.
- b. Concentrated Nitric acid. Reagent grade or in-house distilled.
- c. 50% (1:1) Nitric acid. Dilute 250ml of concentrated reagent to 500ml in a volumetric flask. Mix well and transfer to a clean container for storage.
- d. 5% Nitric acid. Dilute 100ml of concentrated reagent to 2000ml in a volumetric flask. Mix and store as above.

7. Procedure

a. Using a glass watch glass, accurately weigh 0.4000g of sample powder. Handle the glass with forceps at all times during a weighing. Label a Teflon beaker with the sample number and add a few milliliters of water to the beaker. Quantitatively transfer the weighed powder to the beaker. Rinse all of the powder from the watch glass into the beaker using a wash bottle filled with water. Rinse any powder clinging to the beaker wall to the bottom of the beaker. Cover the beaker with a Teflon watch glass. Carefully swirl the beaker to ensure all of the sample powder is wet. Repeat this step for all samples in the set.

b. In a fume hood, transfer 10ml of concentrated Nitric acid to each sample beaker. This may be accomplished using either a graduate or a glass pipette; however, the pipette is preferred since the walls of the beaker may be rinsed down again as the pipette drains.

c. In the hood, add 5-7ml of concentrated HF to each sample beaker while stirring the mixture with a Teflon stirring rod. The stirring prevents the formation of a "cake" which may prevent complete digestion of the powder.

d. Place the covered beakers in the bead bath on a hotplate set for low to medium heat. Allow the samples to digest for 4-6 hours.

e. Remove the covers, increase the heat and allow the solutions to evaporate to near but not complete dryness. Avoid baking of the residue at this point to prevent the formation of insoluble fluorides. Allow the beakers to cool.

f. Add 10ml of 1:1 Nitric acid to each beaker, place them in the bead bath and heat to near dryness.

g. Add an additional 10ml of 1:1 Nitric acid and evaporate to dryness. Repeat this step.

h. With a pipette, add 10ml of 1:1 Nitric acid to each beaker, cover with a watch glass and heat gently to dissolve the residue. It is sometimes helpful to add 10-15ml of water to the beakers to promote dissolution. A clear solution should be obtained at this point. Allow the beakers to cool.

i. Transfer the solution to a 100ml volumetric flask and rinse the beaker with water.

j. Pipette an additional 10ml of 1:1 Nitric acid to each beaker and return it to the hotplate for a few minutes. Remove and cool.

k. Police the bottom of each beaker with the Teflon stirrer, if necessary. Transfer the liquid to the 100ml volumetric flask containing the remainder of the sample solution. Dilute to the mark. Stopper and mix well by inverting the flask several times, while agitating the solution.

l. Transfer this solution to a clean 125ml Nalgene bottle. This solution contains the equivalent of 4000 ppm TDS of the sample in it (400mg/100ml). This is not strictly true, since the silica in the sample was volatilized during the evaporations.

m. Using a pipette, transfer 20ml of the "4000TDS" solution to a clean 100ml volumetric flask. Dilute to the mark with 5% Nitric acid, mix and transfer this solution to a clean 125ml Nalgene bottle. This 1:5 dilution results in an "800TDS" solution.

n. If international reference samples are to be used as standards, they should be brought into solution in exactly the manner described above. Standards made from elemental solutions should be made up in 5% Nitric acid, so as to match the unknown sample matrix (see Potts, 1987 for more on this subject).

o. The concentrations of the major and minor elements, as oxides, and of trace elements are determined using either the 4000TDS or 800TDS solutions.

8. Quality assurance/control

a. Solutions of international reference samples or of in-house standards should be used for quality assurance purposes. Several should be available in the laboratory. Pick reference samples that are similar in composition to your unknowns. Reference samples should be run after every 4-5 unknowns to monitor possible instrument drift. Calcium and iron concentrations are particularly prone to drift and should be monitored carefully.

b. Precision and accuracy estimates vary from element to element. Highly sensitive elements, such as Sr and Mg, commonly exhibit values in the range 0.5% to 1.0%. Less sensitive elements and those elements subject to memory effects (i.e. drift) may show values between 1.5% and 3.0%.

9. Spectral interference corrections

At this point the only interferences detected or inferred are that of Ti on V and Fe on Co. The empirical correction for V is:

$$\text{Actual ppm V} = \text{Measure ppm V} - (\% \text{TiO}_2 \times 44.13)$$

The results we have obtained for Co to date have been unimpressive. Literature references suggest an interference of Fe on Co. Scans on our instrument suggest that Fe is not an interfering element on Co. Further investigation is required and Co values obtained at the present time should be considered suspect.

10. References

Potts, P. J. 1987. A handbook of silicate rock analysis. Chapman and Hall, New York. 622p.

Shapiro, Leonard. 1975. Rapid analysis of silicate, carbonate and phosphate rocks--revised edition. United State Geological Survey Bulletin 1401.

Southern Research Institute

ANALYTICAL METHOD

Method No.: ACD-3291

Title: Preparation of Solid Samples for Total Chloride Analysis by Eschka fusion.

1.0 PRINCIPLE

In this procedure, a sample is weighed into a porcelin crucible. Eschka mixture is then added and heated in a muffle furnace. After cooling, the contents are washed into a 100-mL volumetric flask for analysis.

2.0 REAGENTS

See Section 6.3 of ASTM D4606-86.

3.0 INSTRUMENTS AND APPARATUS

See Section 5.5 and 5.6 of ASTM D4606-86.

4.0 PROCEDURE

4.1 Prepare as in Section 8.3.1 of ASTM D4606-86.

4.2 Sample Solution: Wash sample from crucible into a 100-mL volumetric flask with about 50 mL of hot DI water. Stopper, shake well and dilute to mark with DI water.

5.0 QUALITY CONTROL

For each set of samples, prepare a blank consisting of 5 times the amount of Eschka mixture used in samples. Carry through same process as samples, but washing into a 500-mL volumetric flask and diluting to the mark.

ANALYTICAL METHOD

Method No.: ACD-CSL

Title: μ Wave Digestion of Coal and Ash Samples

1.0 SUMMARY

This method provides for the acid digestion of anthracite coal (60 mesh) in a closed vessel using pressure controlled microwave heating in multiple steps for the determination of metals by spectroscopic methods. NOTE: This procedure is a reference point for sample digestion using the CEM Microwave Sample Preparation System and may need to be modified or changed to obtain the required results on your samples.

2.0 REQUIRED EQUIPMENT

CEM Microwave Sample Preparation System, Lined Digestion Vessel Accessory Set

3.0 REAGENTS

HNO₃ (70%), HF (48%), HCl (37%)

4.0 NUMBER OF VESSELS

6. NOTE: This procedure is outlined for 6 samples. To increase or decrease the number of samples, increase or decrease the power approximately 30 watts/sample (5% increments in a 630 watt system or 3% increments in a 950 watt system).

5.0 PROCEDURE

5.1 Weigh 1.0 gram of sample into each vessel. Add 10 mL of HNO₃ to each vessel.

5.2 Seal all vessels except the one to be used for pressure control.

5.3 Seal the control vessel with a modified cap assembly. NOTE: The control vessel must contain the largest and most reactive sample.

5.4 Place the vessels into the turntable. Connect the vent tubes from the vessels to the collection vessel.

5.5 Place the turntable into the system. Connect the pressure sensing line to the control vessel.

ANALYTICAL METHOD

Method No.: ACD-CSL

Title: μ Wave Digestion of Coal and Ash Samples

5.6 Program as follows (or recall the stored program).

FILENAME: COALI

stage	(1)	(2)	(3)	(4)	(5)
%POWER*	50	50	50	50	0
PSI	40	80	120	160	0
TIME	10:00	10:00	10:00	25:00	0:00
TAP	5:00	5:00	5:00	15:00	0:00
TEMP					
FAN SPEED	100	100	100	100	100

*This is for a nominal 630 watt system. For a nominal 950 watt system, multiply the % power by 0.66.

5.7 Run heating program to completion.

5.8 Cool the samples for a minimum of 5 min, vent the control vessel and remove the pressure sensing line. Remove the turntable from the system.

5.9 Manually vent and open the vessels. NOTE: Manual venting of CEM closed vessels should only be performed when wearing hand, eye and body protection and only when the vessel contents are at or below room temperature to avoid the potential for chemical burns. Always point the vent hole away from the operator and toward the back of a fume hood.

5.10 Add 5 mL of HF and 1 mL of HCl to each vessel and reseal.

5.11 Place turntable in system. Connect the pressure sensing line to the control vessel.

ANALYTICAL METHOD

Method No.: ACD-CSL

Title: μ Wave Digestion of Coal and Ash Samples

5.12 Program as follows (or recall the stored program).

FILENAME: COALII

stage	(1)	(2)	(3)	(4)	(5)
%POWER*	60	0	0	0	0
PSI	150	0	0	0	0
TIME	20:00	0:00	0:00	0:00	0:00
TAP	15:00	0:00	0:00	0:00	0:00
TEMP					
FAN SPEED	100	100	100	100	100

*This is for a nominal 630 watt system. For a nominal 950 watt system, multiply the % power by 0.66.

5.13 Run the heating program to completion.

5.14 Cool the samples for a minimum of 5 min, vent the control vessel and remove the pressure sensing line. Remove the turntable from the system.

5.15 Manually vent and open the vessels. NOTE: Manual venting of CEM closed vessels should only be performed when wearing hand, eye and body protection and only when the vessel contents are at or below room temperature to avoid the potential for chemical burns. Always point the vent hole away from the operator and toward the back of the fume hood.

5.16 Add 30 mL of H₃BO₃ to each vessel and reseal.

5.17 Place the turntable into the system. Connect pressure sensing line to the control vessel.

ANALYTICAL METHOD

Method No.: ACD-CSL

Title: μ Wave Digestion of Coal and Ash Samples

5.18 Program as follows (or recall the stored program).

FILENAME: BORIC

stage	(1)	(2)	(3)	(4)	(5)
%POWER*	100	0	0	0	0
PSI	50	0	0	0	0
TIME	5:00	0:00	0:00	0:00	0:00
TAP	5:00	0:00	0:00	0:00	0:00
TEMP					
FAN SPEED	100	100	100	100	100

*This is for a nominal 630 watt system. For a nominal 950 watt system, multiply the % power by 0.66.

5.19 Run the heating program to completion.

5.20 Cool the samples for a minimum of 5 min, vent the control vessel and remove the pressure sensing line. Remove the turntable from the system.

5.21 Manually vent and open the vessels. NOTE: Manual venting of CEM closed vessels should only be performed when wearing hand, eye and body protection and only when the vessel contents are at or below room temperature to avoid the potential for chemical burns. Always point the vent hole away from the operator and toward the back of the fume hood. Transfer the solution into a flask, with a filtration step, if needed, prior to analysis.

QA/SOP No.: ACD-3182
Revision No.: 0

Page: 1 of 2
Date: 07/23/91

ANALYTICAL METHOD

Title: Mercury in Solid or Semisolid Waste: Manual Cold-Vapor Technique

1.0 Principle

This method, a cold-vapor atomic absorption technique, is based on the absorption of radiation at 253.7-nm by mercury vapor. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

2.0 Reagents

See Section 5.0 Reagents, Method 7471, SW-846.

3.0 Instruments and Apparatus

See Section 4.0 Apparatus and Materials, Method 7471, SW-846.

4.0 Procedure

See Section 7.0 Procedure, Method 7471, SW-846.

5.0 Data Reduction

The analyst determines the mercury value from a calibration curve constructed by plotting the absorbance of mercury standards versus the concentration of mercury (ng/mL) in the standards.

6.0 Quality Control

6.1 Employ a minimum of one blank per sample batch to determine if contamination or any memory effects are occurring.

6.2 A matrix spike and a matrix spike duplicate are analyzed for each batch of samples.

6.3 Calibration curves in the range of 0.3 ng/mL to 10 ng/mL must be composed of a minimum of three standards.

6.4 Dilute samples if they are more concentrated than the highest standard or if they fall on the plateau of a calibration curve.

QA/SOP No.: ACD-3182
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Title: Mercury in Solid or Semisolid Waste: Manual Cold-Vapor Technique

7.0 Reporting

7.1 The results of the analyses are transmitted by the analyst to the project manager for review.

7.2 Results of all analyses are kept or crossed referenced in the appropriate notebook specifically assigned to the project.

7.3 All record-keeping and reporting are maintained by the analyst or designee and are archived in the Analytical Chemistry Files.

8.0 Reference

8.1 Method 7471, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, SW-846. Office of Solid Waste and Emergency Response, U.S. Environmental Protection Agency, Washington, DC. November 1986.

8.2 Method 245.5, Method for Chemical Analysis of Water and Wastes, EPA-600/4-82-055, Environmental Monitoring and Support Laboratory, U.S. Environmental Protection Agency, Cincinnati, Ohio. December 1982.

Approved by:

Kim Bangman
Head, Environmental Analytical
Chemistry Section

7/26/91

Date

Standard Laboratories

STANDARD LABORATORIES INC./GOULD ENERGY DIVISION

STANDARD TEST PROCEDURE - 01.39

METHOD:

Chlorine in Coal, Ion-Selective Electrode Method

REFERENCE:

ASTM Annual Book of Standards, Volume 05.05, Method D-4208
D-1193 Specification for Reagent Water
D-3173 Test for Moisture in the Analysis Sample of Coal and Coke
D-3180 Calculating Coal and Coke Analysis from As-determined to
Different Bases
E-144 Recommended Practice for Safe Use of Oxygen Combustion
Bombs

SUMMARY OF METHOD:

Total chlorine is determined in this method by combusting a weighed sample in an oxygen bomb with a dilute base (sodium carbonate) absorbing the chlorine vapors. The bomb is rinsed into a beaker with distilled water and, following the addition of an ionic strength adjustor, the chlorine is determined by ion-selective electrode.

The chlorine content of coals may be useful in the evaluation of slagging problems, corrosion in engineering processes, and in the total analysis of coal and coke.

When coal samples are combusted in accordance with this method, the chlorine is quantitatively retained and is representative of the total chlorine content of the whole coal.

EQUIPMENT:

Balance - Mettler Model AE-160 single pan electronic balance, 160 gram capacity, 0.1 mg sensitivity.

Oxygen Bomb - Parr 1108 oxygen bombs.

Crucible - Parr base metal alloy crucibles are used exclusively.

Bucket - Parr A391DD oval bucket.

Chloride Ion-Selective Electrode - Orion Model 9417B

Double Junction Reference Electrode - Orion Model 90-02-00

PA STP 01.39

Automatic Pipets
Electrode Polishing Strips - Orion
Specific Ion Meter - Orion Research Model 407A

Magnetic Stirrer

Stir Bars - Teflon Coated
Volumetric Flasks - Class "A" - Nitric acid wash prior to use

REAGENTS:

Distilled Water - Reagent water conforming to D-1193.

Tempered Bucket Water - Tap water, cool.

Fuse Wire - Emerson .004 gauge annealed TC-FE wire 1.08 BTU/cm

Sodium Carbonate Solution (2%) - Dissolve 2.0 g of reagent grade sodium carbonate in 100 ml of distilled water.

Oxygen - Must be manufactured from liquid air, 99.5% pure free of combustible matter.

Ionic Strength Adjuster "ISA" - (5 M NaNO₃) - Dissolve 42.5 g of reagent grade sodium nitrate in 100 ml of distilled water.

Chloride, Standard Stock Solution (1000 ppm) - Dissolve 1.65 g of reagent grade sodium chloride in distilled water and dilute to 1 liter. The NaCl should be dried for 1 hour at 105 °C and cooled to room temperature in a dessicator before weighing.

PROCEDURE:

- 1.0 Place a dry stainless steel crucible on balance, tare. Place approximately 0.8 grams of -60 mesh coal into a crucible and weigh to the nearest 0.1 mg.
- 2.0 Transfer 5 ml of 2% Sodium Carbonate solution into the combustion bomb. Attach the fuse wire to the bomb electrodes. Place the crucible with the sample into the electrode support of the bomb, and insert the fuse wire into the coal sample.
- 3.0 Assemble the bomb in the normal manner and charge it with oxygen to a pressure of 30 atmospheres.
 - 3.1 If the oxygen should exceed the specified pressure, do not proceed with the combustion.

4.0 Place the bomb in a cooling water bath, with water flowing. Attach the ignition wires from the firing circuits, and ignite the sample. Allow the bomb to remain in the cooling water for 15 minutes to allow cooling and absorption of soluble vapors.

4.5 Remove the bomb and release the pressure at a uniform rate. (Overall time should be greater than 2 minutes).

5.0 Check for complete combustion and rinse out the bomb with distilled water. Dilute the final solution to 100 ml volume.

NOTE: Discard test if unburned or sooty deposits are found.

6.0 Take 50 ml of solution and transfer to a beaker. Add 1.00 ml of ISA (Ionic Strength Adjuster) to the solution. Set on stirrer and mix.

7.0 Set the specific-ion meter to the X+ mode. Adjust sample reading to infinity mark on green scale. Allow to stabilize.

8.0 Add 1.00 ml of 1000 ppm Cl - standard to the solution.

9.0 Take reading off the green (X+) scale. Record the reading.

10.0 The chlorine "value" is equal to:

$$\frac{[\text{final reading}] \times [\text{conc. of standard (1000 ppm)}]}{\text{sample volume (50 ml)}} = \text{ppm Cl}$$

$$\frac{[\text{ppm Cl}] \times [100 \text{ ml}]}{\text{sample wt. (g)}} \times .0001 = \% \text{ Chlorine in coal}$$

10.1 Use the percent moisture to calculate the results of the analysis to a dry basis.

10.2 Procedures for converting the value obtained on the analysis sample to other bases are described in Method D-3180.

QUALITY ASSURANCE:

Blanks are to be run with each set of samples in order to effect a background correction, if necessary. The blank consists of distilled water diluted to 100 ml and treated exactly as a sample (Steps 6 - 9). A duplicate should be analyzed twice weekly. Records are to be entered into the logbook designated for ion-selective electrode.

PA STP 01.39

SAFETY:

Reference Method 01.34, Heating Value, regarding handling and safety of combustion bombs.

NEVER fire a bomb containing greater than 30 atmospheres of oxygen.

Compressed gas tanks should be moved by dolly only - not by hand-rolling.

PROCEDURE 01.39

Approval *Don't* Title *Technical Mgr.* Date *12/7/92*

Authorization for Use *Thomas G. Right* Title *Division Mgr.* Date *1/25/93*

STANDARD LABORATORIES INC./GOULD ENERGY DIVISION

STANDARD TEST PROCEDURE - 01.50

METHOD:

Total Mercury in Coal

REFERENCE:

ASTM Annual Book of Standards, Volume 05.05, Method D-3684
ASTM E-144 Recommended Practice for Safe Use of Oxygen Combustion Bombs

SUMMARY OF METHOD:

Total mercury is quantitatively determined by combusting a weighed sample in an oxygen bomb with dilute nitric acid as the absorbent. The bomb washings are diluted to a known volume utilizing dilute nitric acid. Final results are obtained by cold vapor atomic absorption techniques.

EQUIPMENT:

Balance - Mettler Model AE-160 single pan electronic balance, 160 gram capacity, 0.1 mg sensitivity

Oxygen Bomb - Parr 1108 oxygen bombs

Crucible - Parr Base metal alloy crucibles are used exclusively.

Bucket - Parr A391DD oval bucket

Atomic Absorption Spectrophotometer - IL 951

Cold Vapor Accessory - IL

Fleakers - 50 ml capacity

Automatic Pipets

Stir Bars - Teflon Coated

Volumetric Flasks - Class "A"

PA STP 01.50

REAGENTS:

Distilled Water - Reagent water conforming to D-1193

Tempered Bucket Water - Tap water, cool

Fuse Wire - Emerson .004 guage annealed TC-FE Wire

Oxygen - Must be manufactured from liquid air, 99.5% pure

Hydroxylamine Hydrochloride Solution-dissolve 1.5g of $\text{NH}_2\text{OH}\cdot\text{HCl}$ in distilled water and dilute to 100 ml.

Nitric Acid (1:9) - dilute 100 ml of concentrated nitric acid to 1 liter with distilled water.

Stannous Chloride Solution - dissolve 10 g of $\text{SnCl}_2\cdot 2\text{H}_2\text{O}$ in 45 ml of concentrated hydrochloric acid and cautiously dilute to 100 ml with distilled water.

Potassium Permanganate Solution - dissolve 5g of KMnO_4 in distilled water and dilute to 100 ml.

Mercury Standard Stock Solution (1000 ppm) - Banco

Nitrogen - purge gas

PROCEDURE:

- 1.0 Place a dry stainless steel crucible on balance, tare. Place approximately 0.8 grams of -60 mesh coal into the crucible and weigh to the nearest 0.1 mg.
- 2.0 Transfer 10 ml of HNO_3 (1:9) solution into the combustion bomb. Attach the fuse wire to the bomb electrodes. Place the crucible with the sample into the electrode support of the bomb, and insert the fuse wire into the coal sample.
- 3.0 Assemble the bomb in the normal manner and charge it with oxygen to a pressure of 30 atmospheres.
 - 3.1 If the oxygen should exceed the specified pressure, do not proceed with the combustion.
- 4.0 Place the bomb in a cooling water bath, with water flowing. Attach the ignition wires from the firing circuits and ignite the sample. Allow the bomb to remain in the cooling water for 15 minutes to allow cooling and absorption of soluble vapors.

PA STP 01.50

4.5 Remove the bomb and release the pressure at a uniform rate (overall time should be greater than 2 minutes).

5.0 Check for complete combustion and rinse out the bomb with distilled water. Complete the final dilution to 100 ml utilizing HNO_3 (1:9). Add KMnO_4 solution dropwise until the color persists for 60 seconds.

NOTE: Discard test if unburned or sooty deposits are found.

6.0 Align the optical cell in the beam path of the atomic absorption spectrophotometer and optimize the instrument using normal operating conditions as per manufacturer's directions. (Peak Height = 50 sec.).

7.0 Prepare Hg standard, as usual, allowing for a five point calibration curve. When preparing the standards, the final dilution should utilize HNO_3 (1:9) as diluent. Add KMnO_4 dropwise until the color persists for 60 seconds.

8.0 Add 2.5 ml of Hydroxylamine Hydrochloride ($\text{NH}_2\text{OH}\cdot\text{HCL}$) to 50 ml of standard. Place reduction flask in cold vapor accessory. Press start.

8.5 Allow the system to go through the Flush cycle (20 sec.).

9.0 After the pink color fades, add 2.5 ml of stannous chloride (SnCl_2).

10.0 As the instrument continues through the REACTION cycle, time it for 40 seconds, hit READ.

11.0 Repeat this procedure for unknown sample solutions.

12.0 A reagent blank shall be prepared according to steps 7.0, 8.0 - 10.0 but omit the Mercury standard solution. This blank will be used to auto-zero the instrument.

13.0 A method blank shall be prepared according to step 1.0 - 5.0 and 8.0 - 10.0. This blank will be recorded and subtracted from final reading.

14.0 Calculation:

$$\text{A. } \frac{(\text{Absorbance of sample} - \text{absorbance of method blank})}{\text{Absorbance of standard}} \times \text{standard value (ppb)} = \text{ppb of Hg in solution}$$

$$\text{B. } \frac{\text{ppb of Hg in solution}}{\text{sample weight (g)}} \times \frac{100 \text{ ml}}{1000} = \text{ppm of Hg in coal}$$

PA STP 01.50

QUALITY ASSURANCE:

Blanks - Refer to step 12.0 and 13.0 for procedure.

Duplicates - Attempt to run at least twice weekly or with a weekly sample set.

Standard - NIST 1630 or 1633a should be run with each sample set.

Glassware - All glassware and bottles used should be washed with detergent and tap water and rinsed with 1:1 nitric acid, distilled water, 1:1 Hydrochloric Acid and distilled water, respectively.

SAFETY:

Acid Handling: Always remember to add Acid to Water, SLOWLY, using a fume hood, goggles, labcoat and safety glasses.

Reference Method 01.34, Heating Value, regarding handling and safety of combustion bombs.

NEVER fire a bomb containing greater than 30 atmospheres of oxygen.

Compressed gas tanks should be moved by dolly only - not by hand rolling.

PROCEDURE 01.50

Approval By Tom [Signature] Title Technical Mgr Date: 12/7/92

Authorization for Use Thomas A. Right Title Division Mgr Date: 1/05/93

STANDARD LABORATORIES INC./GOULD ENERGY DIVISION

STANDARD TEST PROCEDURE - 01.51

METHOD:

Trace Elements in Coal by Atomic Absorption

REFERENCE:

ASTM Annual Book of Standards, Volume 05.05, Method D-3683

SUMMARY OF METHOD:

Whole coal or coke is ashed at low temperature (500 deg. C.) and digested with an Aqua Regia/hydrofluoric acid mixture on a steam bath. The solution is neutralized with boric acid, reheated, filtered and diluted to a known volume. The resultant solution can now be analyzed for individual elements by atomic absorption spectrophotometry. The ASTM test reference covers the determination of Beryllium, Chromium, Copper, Manganese, Nickel, Lead, Vanadium, and Zinc.

EQUIPMENT:

Balance - Mettler Model AE-160 single pan electronic balance, 160 gram capacity, 0.1 mg sensitivity

Atomic Absorption Spectrophotometer - IL 951

Hollow Cathode Lamps - single element

Ashing Furnace - Thermolyne Type 6000 Programmable Furnace

Teflon Flasks - Preiser #4106-0050 Erlenmyer Flasks with screw closure

Steam Bath - Preiser Model # 16-4305-02

Volumetric Flasks - Class "A"

Crucibles - Coors porcelain Model # 60053

Acetylene

Nitrous Oxide

PA STP 01.51

REAGENTS:

Deionized Water - Reagent water conforming to D-1193

Aqua Regia Solution - Mix 100 ml of concentrated nitric acid and 300 ml of Hydrochloric acid with 100 ml of deionized water in a 500 ml volumetric flask

Boric Acid Solution, Saturated - dissolve 120 grams of boric acid in 500 ml volumetric flask

Hydrofluoric Acid - CAUTION EXTREMELY DANGEROUS

Stock Standard Solutions - prepare 100 ppm stock solutions for each element being analyzed utilizing the following ratios:

100 ml final volume: 1.5 ml Aqua Regia solution
3 ml Boric Acid

PROCEDURE:

- 1.0 Ash enough sample to allow for 0.2000 g of ash to be used for final dissolution. The number of crucibles to be used per sample can roughly be assessed by knowledge of the as determined ash value as prompted by the computer.
- 2.0 Place approximately 1 g of coal into each crucible (weigh to the nearest 0.1 mg). Use only the middle 16 slots for each tray. Both top and bottom levels in the furnace can be used.
- 3.0 Place the trays in a cold ashing furnace. The furnace programmer should be set to attain 300 deg. C. in an hour and then 500 deg. C. for an additional 2 hours. Stir the sample once each hour.

4.0	Furnace Settings	<u>Program</u>	<u>Setting</u>
		Ramp 1	4 deg C./min.
	Air Flow	Level 1	300 deg. C.
	35 PSIG	Dwell 1	0.0 min.
		Ramp 2	4 deg. C./min.
		Level 2	500 deg. C.
		Dwell 2	180 min.

- 5.0 At the end of the cycle, remove the tray from the furnace and cover with lids. Place entire tray in dessicator.

- 6.0 After cooling, weigh each crucible to the nearest 0.1 mg. Record weights and calculate ash content.
- 7.0 Weigh 0.2 grams of ash to the nearest 0.1 mg. Place in teflon erlenmyer flask. Add 3 ml aqua regia solution and 5 ml hydrofluoric acid, tighten screw caps, and place the bottles on the steam bath for at least 2 hours.
- 8.0 Add 30 ml of saturated boric acid solution to each flask. If a residue remains, the mixture may be reheated for an hour to dissolve it.
- 9.0 Cool the solution to room temperature. Filter the solution and make a final dilution to 50 ml.
- 10.0 Align the burner head in the beam path of the atomic absorption spectrophotometer and optimize the instrument as per manufacturer's instructions.
- 11.0 Use the following instrumental parameters:

<u>Elements</u>	<u>Wavelength, nm</u>
Beryllium	234.9
Cadmium	228.8
Chromium	357.9
Copper	324.8
Manganese	279.5
Nickel	232.0
Lead	283.3
Vanadium	318.0
Zinc	213.9

Other elements may also be analyzed using this same methodology.

- 12.0 A five-point calibration graph, at a minimum, should be prepared for each element. The graph should be verified with at least 2 standards for each sample set analyzed.

PA STP 01.51

13.0 Calculations:

$$\text{A. ppm in ash} = \frac{(\text{absorbance of sample} - \text{absorbance of blank})}{\text{absorbance of standard}} \times \text{ppm of standards} \times \frac{50 \text{ ml}}{0.2000 \text{ g}}$$

$$\text{B. ppm in coal} = \frac{\text{ppm in ash} \times \text{low temp. ash \%}}{100}$$

QUALITY ASSURANCE:

Blanks - A method blank should be prepared with each sample set.

Duplicates - Analyze one per every 10 samples

Standard - Utilize one or more of the following standards with each sample set:

NIST 1632b - Ba, Co, Cu, Pb, Mn, Ni, Zn

NIST 1633a - Sr, Tl, V, Be, Mo

NIST 1635 - For subbituminous (Lignite) -
Cr, Cu, Pb, Mn, Ni, V, Zn

Glassware - All glassware and bottles used should be washed with detergent and tap water and rinsed with 1:1 Nitric Acid, distilled water, 1:1 Hydrochloric Acid and distilled water, respectively.

- Store volumetric flasks in Nitric Acid (1:7) and cover with Parafilm.
- Oven temperature checks - weekly

SAFETY:

Acid Handling: Always remember to add Acid to Water - SLOWLY - using a fume hood, goggles, lab coat and safety glasses. Refer to appropriate material safety data sheets.

Hydrofluoric Acid - Extreme care should be taken when handling this acid. All proper safety equipment should be used. If you come into contact with this acid, get help IMMEDIATELY !!!! A hydrofluoric acid spill kit is located in the work area. This kit is specific for HF only.

PA STP 01.51

Compressed gas tanks should be moved by dolly only - not by hand rolling

PROCEDURE 01.51

Approval By Loni Patney Title Technical Mgr. Date: 12/7/92
Authorization for Use Thomas A. Right Title Division Mgr. Date: 1/05/93

STANDARD LABORATORIES INC./GOULD ENERGY DIVISION

STANDARD TEST PROCEDURE - 01.52

METHOD:

Total Arsenic and Selenium in Coal

REFERENCE:

ASTM Annual Book of Standards, Volume 05.05, Method D-3684
Procedure for bomb combustion

Consolidation Coal Co. Bomb Combustion Method

ASTM E-144 Recommended Practice for Safe Use of Oxygen Combustion
Bombs

SUMMARY OF METHOD:

Arsenic & Selenium are determined by combusting a weighed sample in a platinum-fitted bomb. A quartz liner and lid are utilized to contain sample and washings in a non-contaminating environment. The sample is diluted to a known volume with final results obtained by graphite furnace atomic absorption spectroscopy.

EQUIPMENT:

Balance - Mettler Model AE-160 single pan electronic balance, 160
gram capacity, 0.1 mg sensitivity

Oxygen Bomb - PARR 1108 oxygen bombs

Bucket - PARR A391DD oval bucket

Quartz Liner with cover - PARR A513A

Crucible - PARR fused Silica combustion capsules part #43A3

Fuse Wire - PARR platinum fuse wire part #45C3

Platinum Electrode Conversion Kit-PARR part #'s 4A, 5A, and 68 AC

Atomic Absorption Spectrophotometer - Perkin Elmer Model 460

Graphite Furnace - Perkin Elmer HGA-400

Volumetric Flasks - Class "A"

Automatic Pipets

Graphite Tubes

PA STP 01.52

REAGENTS:

Distilled Water - Reagent water conforming to D-1193

Tempered Bucket Water - tap water, cool

Oxygen - Must be manufactured from liquid air, 99.5% pure

Arsenic Stock Solution (1000 ppm) - Banco

Selenium Stock Solution (1000 ppm) - Banco

Nitric Acid (1:9) - prepare by cautiously adding 100 ml of concentrated nitric acid and diluting to 1 L with distilled water.

Potassium Permanganate Solution - dissolve 5g KMnO_4 in distilled water and dilute to 100 ml.

Argon - purge gas

PROCEDURE:

- 1.0 Place a dry silica crucible on balance, tare. Place approximately 0.8 grams of -60 Mesh coal into the silica crucible and weigh to the nearest 0.1 mg.
- 2.0 The bomb referred to in this procedure should be modified with platinum electrodes, quartz liner and cover.
- 3.0 Transfer 10 ml of HNO_3 (1:9) into the quartz liner. Attach platinum fuse wire to the bomb electrodes. Place the silica crucible with the sample into the electrode support of the bomb, and insert the platinum fuse wire into the coal sample.
- 4.0 Assemble the bomb in the normal manner and charge it with oxygen to a pressure of 30 atmospheres.
 - 4.1 If the oxygen should exceed the specified pressure, do not proceed with combustion.
- 5.0 Place the bomb in a cooling water bath, with water flowing. Attach the ignition wires from the firing circuits, and ignite the sample. Allow the bomb to remain in the cooling water for 15 minutes to allow cooling and absorption of soluble vapors.

5.5 Remove the bomb and release the pressure at a uniform rate (Overall time should be greater than 2 minutes).

6.0 Check for complete combustion and rinse out the bomb with distilled water. Dilute the final solution to 100 ml volume. Add KMnO₄ solution dropwise until the color persists for 60 seconds.

NOTE: Discard test if unburned or sooty deposits are found.

7.0 Align the graphite furnace assembly in the beam path of the atomic absorption spectrophotometer and optimize the instrument using normal operating conditions as per manufacturer's instructions.

8.0 Prepare appropriate working standards within the linear range of each element. A minimum 5-point calibration curve should be prepared and verified prior to each analysis.

9.0 Graphite Furnace Parameters:

<u>Selenium</u>	<u>STEP</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
	Temp. C.	120	200	1800	2650
	Ramp (s)	10	5	0	0
	Hold (s)	20	10	3	1

<u>Arsenic</u>	<u>STEP</u>	<u>1</u>	<u>2</u>	<u>3</u>	<u>4</u>
	Temp. C.	120	300	1900	2650
	Ramp (s)	10	5	0	0
	Hold (s)	20	10	4	1

10.0 A 25 microliter sample size is used. After manual sample introduction, record final reading.

11.0 CALCULATION:

$$\frac{\text{ppb reading}}{\text{sample weight (g)}} \times \frac{100 \text{ ml}}{1000} = \text{ppm of As/Se in coal}$$

PA STP 01.52

QUALITY ASSURANCE:

Blanks - A reagent blank should be used to auto-zero the instrument. A method blank should be used to correct the sample reading.

Duplicates - Analyze one per every 10 samples

Standard - NIST 1632b should be analyzed with each sample set.

Glassware - All glassware and bottles used should be washed with detergent and tap water and rinsed with 1:1 Nitric Acid, distilled water, 1:1 Hydrochloric Acid and distilled water, respectively.

SAFETY:

Reference Method 01.34, Heating Value, regarding handling and safety of combustion bombs.

NEVER fire a bomb containing greater than 30 atmospheres of oxygen.

Compressed gas tanks should be moved by dolly only - not by hand rolling.

Acid Handling - always remember to add Acid to Water-SLOWLY-using a fume hood, goggles, lab coat and safety glasses.

PROCEDURE

Approval *Lori P. Huey* Title *Technical Mgr.* Date *12/7/92*
Authorization for Use *Thomas A. Light* Title *Division Mgr.* Date *1/15/93*

STANDARD LABORATORIES INC./GOULD ENERGY DIVISION

STANDARD TEST PROCEDURE - 01.53

METHOD:

Total Fluorine in Coal

REFERENCE:

ASTM Annual Book of Standards, Volume 05.05, Method D-3761
ASTM E144 - Recommended Practice for Safe Use of Oxygen Combustion
Bombs
ASTM D1193 - Specification for Reagent Water

SUMMARY OF METHOD:

Total fluorine is determined by combusting a weighed sample in an oxygen bomb utilizing a dilute base (NaOH) to absorb the fluorine vapors. The bomb washings are rinsed into a beaker with distilled water. A volumetric dilution is made with subsequent analysis for fluorine by ion-selective electrode. When coal samples are combusted according to this method, the fluorine is quantitatively retained and is representative of the total fluorine concentration in whole coal.

EQUIPMENT:

Balance - Mettler Model AE-160 single pan electronic balance, 160 gram capacity, 0.1 mg sensitivity

Oxygen Bomb - PARR 1108 oxygen bombs

Crucibles - PARR metal alloy crucibles are used exclusively

Fluoride Ion-Selective Electrode - Corning Model 34121-403

Double Junction Reference Electrode - Orion Model 90-02-00

Specific Ion Meter - Orion Model 407A

Magnetic Stirrer

Teflon Coated Stir Bars

Volumetric Flasks - Class A

Bucket - PARR A391DD oval bucket

PA STP 01.53

REAGENTS:

Distilled Water - Reagent water conforming to D-1193

Tempered Bucket Water - tap water, cool

Fuse Wire - Emerson .004 guage annealed TC-FE Wire

Fluoride, Standard Stock Solutions (1000 ppm) - Dissolve 2.2100 grams sodium fluoride in water and dilute to 1 L. The NaF should be dried for 1 hour at 105 deg. C. and cooled to room temperature in a dessicator before weighing.

Oxygen - 99.5 % pure

Sodium Hydroxide (1N) - dissolve 40g NaOH in distilled water and dilute to 1 L.

TISAB III - Ionic Strength Adjustor - Orion Model # 940911

Polishing Strips - Orion

PROCEDURE:

- 1.0 Place a dry stainless steel crucible on balance, tare. Place approximately 0.8 grams of -100 Mesh coal into crucible and weigh to the nearest 0.1 mg.
- 2.0 Transfer 5 ml of 1N NaOH solution into the combustion bomb. Attach the fuse wire to the bomb electrodes. Place the crucible with the sample into the electrode support of the bomb, and insert the fuse wire into the coal sample.
- 3.0 Assemble the bomb in the normal manner and charge it with oxygen to a pressure of 30 atmospheres.
 - 3.1 If the oxygen should exceed 30 atmospheres, do not proceed with the combustion.
- 4.0 Place the bomb in a cooling water bath, with water flowing. Attach the ignition wires from the firing circuits and ignite the sample. Allow the bomb to remain in the cooling water for 15 minutes to allow cooling and absorption of soluble vapors.

PA STP 01.53

- 4.5 Remove the bomb and release the pressure at a uniform rate (overall time should be greater than 2 minutes).
- 5.0 Check for complete combustion and rinse out the bomb with distilled water. Dilute the final solution to 100 ml volume.

NOTE: Discard test if unburned or sooty deposits are found.

- 6.0 Take 50 ml of solution and transfer to a beaker.
- 7.0 Measure out 50ml of a 10 ppm and 100 ppm fluoride standards prepared from the Standard Stock Solution.
- 8.0 Add 5.0 ml of TISAB III to all standards and samples.
- 9.0 Set the specific-ion meter to the x - mode. Calibrate the meter on the red scale using the 10 and 100 ppm standards.
- 10.0 After calibration is complete, place sample on stirrer. Allow reading to stabilize prior to recording result. Always rinse electrodes with distilled water between runs.
- 11.0 Calculation:

$$\frac{\text{ppm reading} \times \text{sample volume (100ml)}}{\text{sample weight (g)}} = \text{ppm Fluorine}$$

QUALITY ASSURANCE:

Blanks - analyze distilled water according to Steps 5.0 - 10.0

Duplicates - Analyze on a twice - weekly basis

Standard - Alpha AR1800 should be analyzed with each sample set.

SAFETY:

Reference Method 01.34, Heating Value, regarding handling and safety of combustion bombs.

NEVER fire a bomb containing greater than 30 atmospheres of oxygen.

Compressed gas tanks should be moved by dolly only - not by hand-rolling.

PA STP 01.53

PROCEDURE 01.53

Approval *L. J. [Signature]* Title *Technical Mgr.* Date *12/7/92*

Authorization for Use *Thomas G. Kubit* Title *Division Mgr.* Date *1/25/93*

Huffman Laboratories

Element: As
Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Second Recheck</u>	<u>Run Average</u>
AT-15-143	16.0	17.6		26.1
AT-15-195/SL	6.02	6.35		26.1
AT-15-223/SL	7.24	7.26		26.1
AT-15-116	16.4	28.3	5.7	10.7
AT-15-191/SL	7.88	4.42		10.7
AT-15-212	15.7	6.0		10.7
AT-15-228/SL	5.94	4.17		12
AT-15-115	30.8	46.2		56.3
AT-15-190/SL	10.80	7.67		56.3
AT-15-227/SL	13.30	9.64		57.3

Pittsburgh #8

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-P8-017	7.6	7.3	14.5
AT-P8-078	9.2	3.7	14.5
AT-P8-105	7.8	3.3	14.5
AT-P8-127	9.2	3.7	14.5

Upper Freeport

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-UF-011	22.1	28.4	15.8
AT-UF-050	5.2	3.3	6.8
AT-UF-088	13.0	21.6	6.8

Element: Ba
Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-217	97	94	80
AT-15-233/SL	61.57	57.7	80
AT-15-237	59	67	80
AT-15-190/SL	126.83	113.2	200
AT-15-211	163	197	200
AT-15-227/SL	165.52	169.5	228

Element: Be
Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	1.90	2.00	5
AT-15-217	7	4	5
AT-15-233/SL	1.81	1.94	5
AT-15-237	6	4	5
AT-15-191/SL	1.48	1.29	3
AT-15-228/SL	1.26	1.33	3
AT-15-190/SL	2.73	2.54	13
AT-15-227/SL	3.21	2.67	13

Element: Cd
Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	< 0.05	< 0.05	1.1
AT-15-190/SL	< 0.50	< 0.50	3.74
AT-15-227/SL	< 0.05	< 0.05	3.74

Element: Cl			
Illinois #5			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195	1350	1954	1494
AT-15-195/SL	2700	2500	1494
AT-15-217	623	1981	1494
AT-15-233	650	2419	1494
AT-15-233/SL	2300	2700	1494
AT-15-237	714	2034	1494
AT-15-191/SL	2100	2400	1448
AT-15-212	1159	1465	1448
AT-15-228/SL	2000	2000	1448
AT-15-190/SL	1400	2000	968
AT-15-227/SL	1100	1300	670

Upper Freeport			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-UF-050	1736	1298	1900

Element: Co			
Illinois #5			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	16.12	20.4	7
AT-15-233/SL	16.75	21.8	7
AT-15-190/SL	45.00	58.3	32
AT-15-227/SL	53.03	55.8	33

Element: Cr			
Illinois #5			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	21.8	19.0	18

Upper Freeport			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-UF-127	8	18	13
AT-UF-128	186	146	129

Element: F			
Illinois #5			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	136.2	120.8	88
AT-15-233	58	136	88
AT-15-233/SL	133.45	133.8	88
AT-15-116	66	70	51
AT-15-191/SL	55.67	59.5	51
AT-15-115	113	134	159
AT-15-190/SL	214.8	213.8	159
AT-15-227/SL	236.22	211.2	159

Upper Freeport			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	
AT-UF-050	79	65	35
AT-UF-088	74	64	35

Element: Pb			
Illinois #5			
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-186	47	40	31
AT-15-223/SL	23.54	26.7	31
AT-15-191/SL	15.92	18.57	19

Upper Freeport				
<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Second Recheck</u>	<u>Run Average</u>
AT-UF-036	60	39	45	30

Element: Hg

Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-149	0.05	0.06	0.08
AT-15-195/SL (ppb)	426.7	219.55	80
AT-15-233/SL (ppb)	442.41	163.4	80
AT-15-191/SL (ppb)	707.6	152.65	30
AT-15-228/SL (ppb)	542.8	173.55	30
AT-15-190/SL (ppb)	373.8	180.25	50
AT-15-227/SL (ppb)	331.2	327.6	50

Pittsburgh #8

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-P8-127	0.17	0.18	0.15

Element: Mn

Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	39.34	43.5	46
AT-15-217	60	49	46
AT-15-233/SL	36.67	40.8	46
AT-15-190/SL	133.65	137.0	171
AT-15-227/SL	194.45	201.5	217

Upper Freeport

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-UF-128	171	157	140

Element: Ni

Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	30.81	28.0	25
AT-15-233/SL	30.33	28.15	25

Element: Se

Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-195/SL	0.98	1.37	< 2
AT-15-233/SL	1.18	1.49	< 2
AT-15-191/SL	0.80	0.88	< 2
AT-15-228/SL	0.72	0.95	< 2

Element: Zn

Illinois #5

<u>Sample #</u>	<u>Original #</u>	<u>First Recheck</u>	<u>Run Average</u>
AT-15-149	66	56	88
AT-15-195	110	97	88
AT-15-195/SL	50.24	54.5	88
AT-15-233/SL	39.84	43.72	88
AT-15-191/SL	14.44	15.16	23
AT-15-228/SL	17.34	21.8	27
AT-15-115	240	243	212
AT-15-214	177	179	212
AT-15-227/SL	220.16	242.66	272

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