

**Characterization of a Fluidized-Bed combustion Ash to
Determine Potential for Environmental Impact**

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LIST OF ACRONYMS

AFBC	atmospheric fluidized-bed combustion
AMD	acid mine drainage
ASTM	American Society for Testing and Materials
CCBs	coal combustion by-products
CCSEM	computer-controlled scanning electron microscopy
CCSRs	coal combustion solid residues
CFBC	circulating fluidized-bed combustion
CLSM	controlled low-strength material
DOE	U.S. Department of Energy
EERC	Energy & Environmental Research Center
EPA	U.S. Environmental Protection Agency
EPMA	electron probe microanalysis
EPRI	Electric Power Research Institute
FBC	fluidized-bed combustion
FBR	fluidized-bed reactor
IMMR	Institute for Mining and Minerals Research
JSRP	Jointly Sponsored Research Program
LLQ	lower level of quantitation
LOI	loss on ignition
LTL	long-term leaching
MDU	Montana-Dakota Utilities
MP	Malcolm Pirnie

ND	North Dakota
PIXE	proton-induced x-ray emission
RCRA	Resource Conservation and Recovery Act
SEM	scanning electron microscopy
SGLP	synthetic groundwater leaching procedure
SGV	superficial gas velocity
SNCR	selective noncatalytic reduction
SPLP	synthetic precipitation leaching procedure
TCLP	toxicity characteristic leaching procedure
TGA	thermogravimetric analysis
XRD	x-ray diffraction

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CHARACTERIZATION OF A FLUIDIZED-BED COMBUSTION ASH TO DETERMINE POTENTIAL FOR ENVIRONMENTAL IMPACT

INTRODUCTION

A 440-megawatt, circulating fluidized-bed combustion (CFBC), lignite-fired power plant is planned for construction in Choctaw County north of Ackerman, Mississippi. This power plant will utilize Mississippi lignite from the first lignite mine in that state. Malcolm Pirnie, Inc., is working with the power plant developer in the current planning and permitting efforts for this proposed construction project. In order to accommodate Mississippi state regulatory agencies and meet appropriate permit requirements, Malcolm Pirnie needed to provide an indication of the characteristics of the by-products anticipated to be produced at the proposed plant. Since the Mississippi lignite is from a newly tapped mine and the CFBC technology is relatively new, Malcolm Pirnie contracted with the Energy & Environmental Research Center (EERC) to develop and perform a test plan for the production and characterization of ash similar to ash that will be eventually produced at the proposed power plant.

The work performed at the EERC included two primary phases:

- Production of by-products in a bench-scale CFBC unit using lignite provided by Malcolm Pirnie with test conditions delineated by Malcolm Pirnie to represent expected operating conditions for the full-scale plant
- An extensive characterization of the by-products produced, focusing on Mississippi regulatory requirements for leachability, with the understanding that return of the by-product to the mine site was an anticipated by-product management plan

In order to meet the project objectives and time schedule, representatives of Malcolm Pirnie and the EERC worked together to develop the project work plan and to evaluate preliminary results as available. In addition to the funds provided by Malcolm Pirnie for this work, funds were made available through the U.S. Department of Energy (DOE) Jointly Sponsored Research Program (JSRP) at the EERC, which allowed an effort to perform a preliminary evaluation of the utilization potential of the CFBC by-products. There are numerous environmental and economic advantages to the utilization of coal combustion by-products (CCBs), and DOE has a commitment to work with industry to increase utilization of CCBs in the government and commercial sectors.

The overall focus of this project was the environmental assessment of the by-product expected to be produced at the proposed power plant. Emphasis was placed on the leachability of potentially problematic trace elements in the by-products. The leaching research documented in this report was performed to determine trends of leachability of trace elements under leaching conditions appropriate for evaluating land disposal in monofills, such as returning the by-products to the mine site. The assumption for the validity of this research was based on the development of leachate under conditions most likely to be present at the site and to take into consideration mineralogical changes that ash will undergo after contact with water. The EERC

has performed numerous research projects in the past 20 years that have focused on the environmental aspects of CCB disposal and utilization. As a result of these projects, a generic test of leachability, the synthetic groundwater leaching procedure (SGLP) (1) was designed to evaluate leachate formation under conditions representative of actual monofill disposal. The EERC has also developed a detailed characterization scheme that includes leaching studies and provides a broad base of information to facilitate CCB management decisions.

For this project, it was assumed that the leaching solution most likely to contact this type of ash would be groundwater or rainwater after percolation through soil or soil placed over the by-product. While an infinite number of management scenarios exist for any by-product, the assumption made for this study was that artificial conditions, such as acetic acid leaching and any other unlikely scenarios, would be avoided except for regulatory leaching tests such as the toxicity characteristic leaching procedure (TCLP). The SGLP was selected as the appropriate test for the formulation of objective, informed decisions concerning the potential for environmental impact from leachate from solid CCBs.

The ash evaluated was produced in a bench-scale CFBC system at the EERC using lignite provided by Malcolm Pirnie. Two combustion tests were performed with and without selective noncatalytic reduction (SNCR) achieved through the addition of ammonia. The test burns were designed to produce ash similar to that expected at the commercial-scale CFBC unit under development in Mississippi. The goal was to duplicate the mineralogy that is expected in the full-scale plant and thus produce a material with leaching properties similar to those of the ash that will eventually be produced.

This report contains the following:

- A description of the equipment and procedure used in the test burn to prepare the ash
- Characterization results and discussion for the ash produced during the test burn, including a detailed interpretation and discussion of the environmental data.
- Appendices including detailed test burn, analytical, and characterization data.

BACKGROUND

Management of industrial wastes is of general concern to all, and the potential for environmental harm through disposal, although minimal, is real. Because of this, the proper testing of materials to evaluate the potential for environmental harm must be carried out in a manner that is scientifically valid, legally defensible, accurate, precise, and relevant to the disposal conditions anticipated. Often, materials are subjected to the TCLP (2) and, for the most part, some waste materials tested in this manner can be at least partially evaluated for their potential environmental impact. It is recognized that nearly any disposed material has the potential to generate leachate with characteristics different from those of local groundwater. This does not always imply degradation of the environment. Some waste materials disposed in environments where local sediments produce groundwater of relatively high ionic strength may

generate leachates from the infiltration of rainwater that are of higher quality than native groundwater. These waste materials are of little concern, and it is the potentially problematic substances for which proper testing is imperative.

A limitation of the TCLP that appears to be often overlooked is that the application for which it was intended was the evaluation of leaching under codisposal conditions in a sanitary landfill. There are numerous materials that are highly unlikely to be disposed of in sanitary landfills and, under expected monofill disposal, are highly unlikely to encounter an acidic environment. Rather, an alkaline environment will be maintained for long-duration leaching because of the nature of these wastes. The coal combustion solid residues (CCSRs) evaluated in this research are a prime example of materials that should not be evaluated on the basis of TCLP leaching results if scientifically valid and legally defensible data are to be generated.

The TCLP uses an acidic solution containing acetic acid to simulate leaching in a sanitary landfill under codisposal conditions. However, groundwater or rainwater is extremely unlikely to contain acetic acid, and the TCLP would be inappropriate in cases where materials such as CCSRs are being disposed of in a monofill. Even in the event that acid rain infiltrates a landfill, the actual pH and chemistry of the leaching fluid likely to contact placed material must be considered. The percolation of rainwater through the top layer of sediment and placed material would often render the water alkaline from its initial weak acid rain composition. Additionally, rainwater would likely acquire significant concentrations of dissolved constituents upon contact with ash. In the case of exposed material, the amount of material affected by acidic water would be minuscule compared with the amount that would be leached by a highly alkaline solution formed from the interaction of disposed material and water. If it is the case that an alkaline leaching solution will be doing the actual field leaching, the solution used in the laboratory must be alkaline with a similar chemistry. Even in the event that a neutral solution would contact the ash, the bulk or major element solution concentrations of leachate would be determined primarily by the composition of the fly ash being leached. The use of a leaching solution with the proper chemistry and pH cannot be overemphasized, although in the case of low-rank coal ash and ash from many advanced combustion systems, leachate chemistry, especially major ion makeup, is defined and controlled by the ash itself, and the makeup of the leaching solution used initially is not a significant factor unless there are high concentrations of select elements or other unusual conditions such as acid mine drainage (AMD).

An additional reason why certain regulatory leaching tests may be inappropriate is that ash materials from a combustion process can be assumed to change mineralogical composition as a result of hydration reaction upon contact with water and through interaction with different minerals and ash constituents. Many of these reactions are slow and can take up to 30 days or more to exert a measurable effect, as demonstrated in a previous research project at the EERC. The effect of hydration reactions can be in excess of an order of magnitude with respect to solution concentration reductions of several potentially hazardous trace elements such as boron, chromium, and selenium, as well as a number of similar trace elements that exist in solution as oxyanions. Additionally, there are several cationic constituents with solubilities that can be significantly influenced by the formation of various secondary hydrated phases. Notable among these materials is the mineral ettringite, which forms in many ash types upon hydration. Ettringite is both an individual mineral, calcium aluminosulfate hydroxide hydrate

($\text{Ca}_6\text{Al}_2[\text{SO}_4]_3[\text{OH}]_{12} \cdot 26\text{H}_2\text{O}$), and the group name for a series of similar compounds. Ettringite formation is often associated with the reduction of solution concentrations of trace elements that exist as oxyanions in aqueous solution. These elements include but are not limited to arsenic, boron, chromium, molybdenum, selenium, and vanadium (3, 4). Reductions of solution concentrations have been observed for most of these elements in ash-leaching experiments (5) and all of these trace elements have been found to form fully or partially substituted ettringites (5). This mineral can also exert an influence on the solution concentration of several major groundwater constituents such as calcium and sulfate. Since all that is required for ettringite formation is a source of soluble calcium, a source of soluble aluminum, an appropriate oxyanion such as sulfate, and a pH in the range of 11.5 to 12.5, coal ash, particularly FBC ash, provides all of the necessary starting materials and reaction conditions.

The SGLP incorporates long-term leaching (LTL), generally carried out for 30 and 60 days. The TCLP is best used for materials to be disposed in sanitary landfills or to provide comparative data for regulatory purposes. LTL is especially important for alkaline CCSRs, such as FBC ash, which are known to form ettringite as a primary hydration product on contact with water. Ettringite has the ability to incorporate trace elements that exist as oxyanions into its structure, thus lowering leachate concentration with respect to time as the ettringite forms. The results of ettringite formation can be dramatic, with solution concentrations of select trace elements decreasing by up to 2 orders of magnitude. The trace elements most often affected by ettringite formation include arsenic, boron, chromium, molybdenum, selenium, and vanadium. When being used to evaluate highly alkaline and often reactive material, SGLP and LTL are generally performed using either distilled deionized water or a site-specific synthetic groundwater as the leaching solution.

Interpretation of leachate data requires high-quality analytical data with detection limits low enough to assure numeric data points wherever possible. Because of the use of LTL to demonstrate the formation of secondary hydrated phases, such as ettringite, real data points are necessary to identify leaching trends, such as anomalous leaching. Anomalous leaching is defined as leaching results where solution concentrations are decreasing with respect to time. These are often associated with the formation of the mineral ettringite. For this reason, analytical techniques with detection limits well below RCRA (Resource Conservation and Recovery Act) limits were chosen.

Mineralogical characterization using x-ray diffraction (XRD) and computer-controlled scanning electron microscopy (CCSEM) is generally performed on ash before and after LTL. This facilitates the identification of mineralogical changes and aids the interpretation of leaching results.

PROJECT GOAL

The goal of this project was to produce and characterize CFBC by-products that represent the materials anticipated at a proposed commercial CFBC power plant using available bench-scale combustion equipment and the best analytical and interpretation techniques. A complete

description of the equipment and test conditions used to prepare the ash tested in this program can be found in the Experimental section.

WORK PLAN

In order to achieve the project goal, the following work plan was developed with approval from Malcolm Pirnie. The characterization protocol performed on each sample type was as follows:

- Coal characterization
 - Proximate analysis
 - Ultimate analysis
 - Proton-induced x-ray emission (PIXE) for selection of trace elements of concern
 - Bulk inorganic analysis (major, minor, and trace)
 - Leaching (synthetic precipitation leaching procedure [SPLP] U.S. Environmental Protection Agency [EPA] Method 1312)

- Limestone characterization
 - PIXE for selection of trace elements of concern
 - Bulk inorganic analysis (major, minor, and trace)
 - XRD
 - Reactivity (on all samples, with selection of one for complete characterization), maximum of four samples

- Ash characterization

Two sequential runs were conducted with and without SNCR, with complete characterization of both types of ash plus composite samples produced for each run.

- PIXE
- Bulk chemistry (major, minor, and trace)
- Leaching (SGLP, LTL, TCLP 2)
- Permeability ASTM (American Society for Testing and Materials) D 5084-90
- Mineralogy (before and after leaching)
- CCSEM
- SEM morphology
- Sulfate:sulfite
- Particle-size distribution
- Moisture and loss on ignition

CCSEM was not performed on the samples generated during the course of this project. CCSEM, which was initially proposed for the characterization, was not included because of the particle-size distribution and general particle morphology determined during the examination of samples of combustion residue using SEM. The particles were generally too large and irregular

for effective CCSEM characterization. Additionally, the information gained from XRD provided sufficient information for interpretation of the leaching phenomenon observed.

Numerous investigations of the leachability of trace elements from coal combustion solid by-products have been conducted at the EERC using several leaching procedures. The primary objectives of these investigations and the approach that was used in this project can be summarized as follows:

- Identify trace elements of environmental significance, to include currently regulated trace elements and others present in significant total concentrations.
- Determine the total amounts of all identified trace elements. Measure and compare the leachability (mobility) of the identified trace elements using several leaching tests.
- Solid combustion residues were evaluated in this research project. These solid residues were subjected to a comprehensive chemical, physical, and mineralogical characterization scheme that met the objectives listed above.
 - Qualitative screening for identification of elements present. PIXE was chosen as the screening tool in this research project. PIXE determines the elements from sodium to uranium in the periodic chart.
 - Quantitation of total concentrations of selected elements in the bulk sample and determination of mineral phases present. Standard analytical techniques, including atomic absorption and inductively coupled argon plasma spectroscopy, were used to generate most of the elemental analytical data.
 - Leaching of the solid by the selected leaching procedures, determination of concentrations of all selected elements in resulting leachates, and identification of mineral phases present in leached solids.

EXPERIMENTAL

Two combustion tests were completed, one using normal conditions and one with SNCR NO_x control achieved by the addition of ammonia. Limestone was analyzed for major, minor, and trace components but was not leached. Results for the limestone include a calculated maximum concentration described below. Coal was leached using the SPLP because coal would likely be leached by rainwater. In separate experiments, it was demonstrated that pyrite present in the coal could oxidize, thus forming acidic conditions. Combustion by-products were leached using TCLP, SGLP, and LTL. This provides data relevant to likely disposal conditions and also provides TCLP data to evaluate the regulatory status of the ash.

The determination of elemental concentrations in solids and in generated leachates was done with appropriate spike recoveries and duplicate determinations performed at least every ten samples or with every varying sample type. Solid samples were digested using a lithium

metaborate flux fusion followed by acid dissolution for determining major and minor components. A closed-bomb microwave digestion using mixed acids was used for digestion of solids for the determination of bulk trace constituents. A closed system was used to minimize the chances for sample contamination and analyte loss during the digestion procedure. A nitric-sulfuric acid mixture followed by hydrofluoric acid was used for the digestion of coal, while a nitric-hydrofluoric acid mixture was used for ash materials.

Ash characterization was done primarily as a means to predict potential for environmental impact of disposed combustion residues. CCSRs, which consisted of spent bed material and fly ash, were characterized for chemical, mineralogical, and leaching characteristics. The primary effort was focused on composite ash samples consisting of spent bed material and fly ash. This composite sample was prepared in the laboratory by combining spent bed material with fly ash. Bulk chemical composition of the solid residues was performed using standard analytical techniques such as atomic absorption and atomic emission spectroscopy. Major and minor constituents were determined by inductively coupled argon plasma spectrometry and included silicon, aluminum, iron, calcium, magnesium, sodium, potassium, titanium, strontium, sulfur, phosphorus, and manganese. Trace elements included the eight RCRA elements, arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver, as well as boron, copper, molybdenum, nickel, vanadium, and zinc identified as a result of screening analyses using PIXE. Boron and molybdenum were included in addition to the RCRA elements and trace elements identified by PIXE. These two elements are of high interest in coal conversion residue chemistry and are, as is the case with boron, not detected by PIXE, which determines the elements sodium through uranium. In addition to the complete characterization of the ash materials, a determination will be made for potential reuse options. A list of potential reuse options will be compiled for the individual ash streams based on information from the characterization effort.

Leaching was performed using a variety of techniques to generate comparative data and to determine changes in leachate composition with respect to time. The various leaching tests utilized were as follows:

- TCLP (6)
- SGLP developed at the EERC with a LTL component where samples were equilibrated for 30 and 60 days (7).
- The SPLP, EPA Method 1312 (2).

Leaching was carried out in 220-mL polysulfone bottles using 10 g of combustion residue and 200 mL of the appropriate leaching solution. Leaching was carried out using end-over-end agitation at 30 rpm as specified in the TCLP protocol. Leaching characterization of the composite ash samples was performed in triplicate.

All leachate concentrations have been reported as milligrams per liter for consistency. Bulk analyses of trace elements are reported in micrograms per gram, and pH is reported in standard pH units. Bulk composition of major and minor elements is reported as weight percent of oxides using the following convention: Al_2O_3 , CaO , Fe_2O_3 , MgO , MnO , P_2O_5 , K_2O , SiO_2 ,

Na₂O, SrO, TiO₂, and SO₂, (SO₄²⁻ in leachate). In order to adequately address environmental concerns and achieve the project goal, the objective of the analytical team was to achieve detection limits below primary drinking water levels or below other regulatory limits such as secondary drinking water or irrigation levels where applicable. These detection limits also allow the principal scientist to interpret data with more confidence and identify leaching trends, such as anomalous leaching, which has been described previously. In the SGLP and LTL experiments, leachate pH was over 11.5, thus providing conditions ideal for ettringite formation (4).

Bench-Scale Testing

Bench-Scale Test Objectives

The overall objectives of the bench-scale testing were to generate ash for characterization, and to determine the feasibility of using SNCR for control of NO_x emissions.

Description of Fluidized-Bed Reactor

A fluidized-bed reactor (FBR) has been constructed to simulate the bed chemistry, ash interactions, and emissions from a fluidized-bed combustor under closely controlled conditions. This reactor is used for sorbent characterization, gaseous emissions including trace elements, agglomeration, and hot-gas cleanup testing in a cost-effective manner over a wide range of operational conditions. The 55-in.-tall reactor is constructed of 3-in. Schedule 80 pipe and is externally heated with three ceramic heaters. A hot cyclone collects the ash and bed material that is carried out of the reactor. The preheated fluidizing gas can be a mixture of air and nitrogen or just air; in addition, one additional gas such as carbon dioxide, carbon monoxide, sulfur dioxide, or a nitrogen oxide can be added to result in a fuel gas similar to that generated in a full-scale fluidized-bed combustor. Preheated gas at temperatures up to 1400°F and pressures up to 200 psig are supplied at the bottom of the reactor through a 1-in. Schedule 40 pipe. The fluidizing gas is supplied at sufficiently high velocities to prevent the sized bed material from dropping out during operation.

The fluidizing gas enters into the 3-in. Schedule 80 main section of the reactor through a conical transition. This conical section was designed without a distributor plate to allow quick removal and quench of the bed material after completion of a test. Bed material can be sampled or collected using a lock hopper system located at the bottom of the reactor. Figure 1 is a side view of the reactor and cyclone. Figure 2 is a photograph of the actual reactor vessel, cyclone, air preheater, reactor collection pot and fuel feed hopper. Figure 3 is a photograph of the final system after the external heaters and other auxiliaries have been installed.

Dry coal and sorbent are premixed, then metered with a variable-speed auger that feeds into a high-speed, water-cooled auger, which in turn carries the material into the reactor. Two coal hoppers ensure that fuel feed is not interrupted while the coal hopper is being refilled. A bed material hopper empties directly into the water-cooled auger, without flow control. Each hopper is maintained at a pressure slightly higher than that in the combustor during operation. The hoppers can be isolated from the pressurized system so that they can be refilled during a test. Fly ash is collected in a hot cyclone and a bag filter. A data acquisition and control system is used to monitor and record all critical pressures, temperatures, flow rates, and emissions, and to remotely

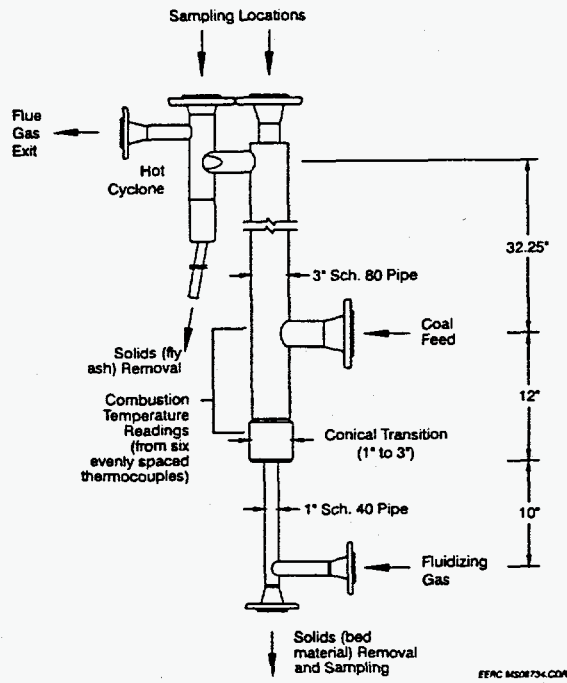


Figure 1. Side view of FBR.

EERC MS09968.CDR

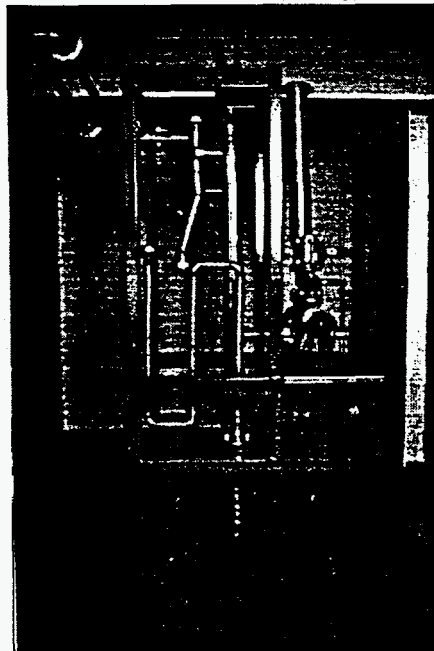


Figure 2. Photograph of FBR.



Figure 3. Photograph of FBR in external heat jacket with auxiliaries installed.

control the numerous valves distributed throughout the system. These critical data include the gas flow rates, bed static and differential pressures across the bed and cyclone, eight different internal reactor temperatures, and coal feed rate, as well as information monitored from other operating conditions and gaseous emissions. Critical data are saved every 30 s throughout the test. Ports for gas-sampling probes and ammonia injection are located at the top of the reactor and the top of the cyclone. The ammonia flow rate is controlled with a mass flow controller.

Test Matrix

Two tests were performed with this coal and limestone. The first test was run at a bed temperature of 1550°F, an excess air level of 20%, superficial velocity of 3.8 ft/s, and a limestone addition to obtain 90% sulfur retention. The second test employed ammonia injection at the top of the reactor to reduce NO_x emissions, and was to operate at the same velocity, excess air, and sulfur retention, but at a freeboard temperature of 1700°F. This temperature is higher than typical CFB operating conditions, but lower than the optimum temperature for SNCR, which is about 1750°F (8); the higher temperature was chosen not to optimize the FBC operating conditions, but to improve NO_x reduction. The increased temperature did adversely affect the sulfur capture reaction, requiring a much higher limestone add rate. A decision was made during the pretest period of the second test to limit the amount of limestone added and run at a sulfur capture of less than 90%.

Fuel and Limestone Preparation and Analysis

The fuel was provided by Malcolm Pirnie in the form of core samples shipped directly from the mine site. The cores arrived sealed in plastic bags, with considerable condensation

inside the bags; the core samples were broken up and allowed to air dry before being crushed and sized to $\frac{1}{8}$ -in. As part of the crushing process, the core samples from each bag were carefully mixed to obtain a product as homogeneous as possible.

Four limestone samples were obtained prior to testing. Thermogravimetric analysis (TGA) was performed on each sample to determine reactivity with SO_2 . The results of the TGA tests are shown in Figure 4. The Ocean Cay and Lime Co. Inc sorbents had the highest reactivity, but the Ocean Cay material appeared to be rather soft and friable, suggesting it may quickly elutriate from the bed of a CFB. The Lime Co. Inc sorbent was chosen over the Ocean Cay because of its greater physical stability. Both coal and limestone were analyzed prior to testing; the results are shown in Table 1.

The as-received limestone contained a very broad size distribution, with a top size of about $\frac{1}{4}$ in. In order to obtain a suitable particle size for the start-up bed material, the limestone was passed through a 14-mesh (1.4-cm) screen. The large size fraction was used for the bed material for this test, although the size of the material was reduced by passing through the delivery auger twice. Because of the relatively short duration of the tests, the start-up material was sulfated prior to the start of the test. This was done in the FBR without any fuel using the ceramic heaters to maintain a bed temperature of about 1400°F and adding a high concentration of SO_2 to the fluidizing gas. This procedure took several hours, during which the downstream gas analyzers measured virtually no SO_2 emissions. The sulfation was considered complete when the SO_2 emissions reached 3000 ppm; at this point the SO_2 was increasing rapidly and further sulfation was expected to be minimal. The smaller size fraction (which was not presulfated) was premixed with the coal for sulfur capture without removing any of the fines.

Results from the Bench-Scale Tests

Operability

Overall operability of the FBR during these tests was very good. In spite of the high moisture content of the coal, which had proved to be a problem with coals burned in this unit previously, the coal fed very uniformly without bridging in the hopper. Temperature was controlled by maximizing coal feed rate, then using the three ceramic heaters to maintain each reactor zone at a given gas temperature. The excess air level specified in the proposal was 25%; however, it was determined that 20% excess air would be more representative of a full-scale CFB system. This minor change in air input was not expected to impact the characteristics of the ash. Limestone was premixed with the coal in small batches of about 2 lb at a time (corresponding to about an hour of operation) during the pretest period, so that frequent adjustments to the rate of limestone addition could be made. When the appropriate ratio of limestone to coal was determined, batches equivalent to about one hopper of mixture were prepared, and the ratio was maintained for the duration of the test.

When it was determined by the sulfur emissions that the bed had reached equilibrium and the correct limestone:coal ratio had been reached, the cyclone and bed material collection pots were emptied and replaced. This marked the beginning of steady-state operation. The material collected during the pretest period was set aside and not included with the material submitted for analysis. Bed material was drained periodically to maintain a uniform pressure drop across the bed.

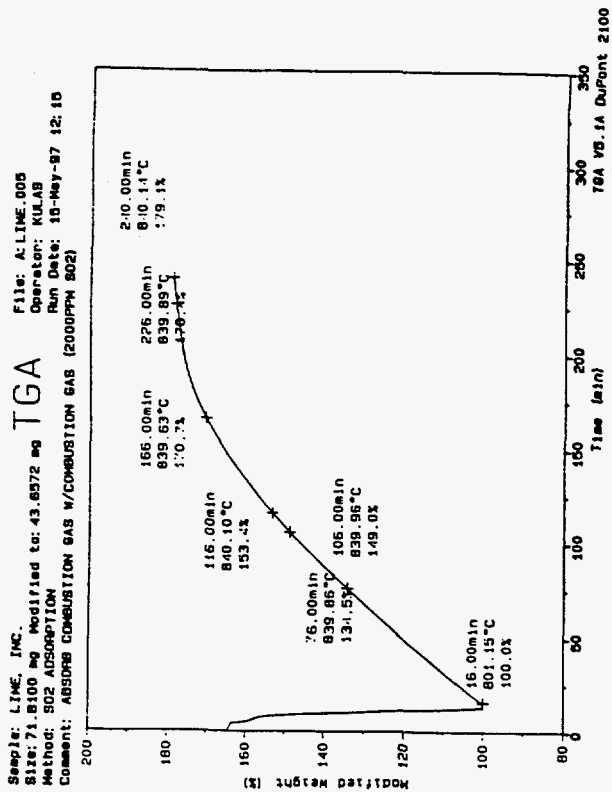
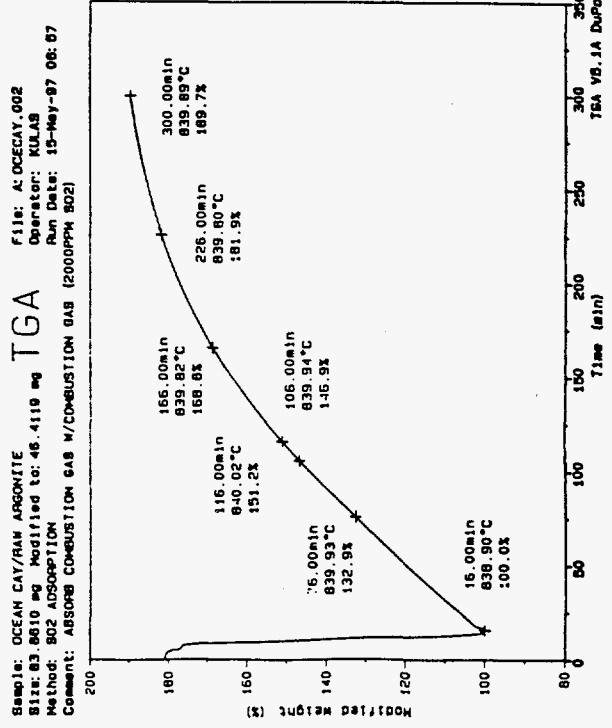
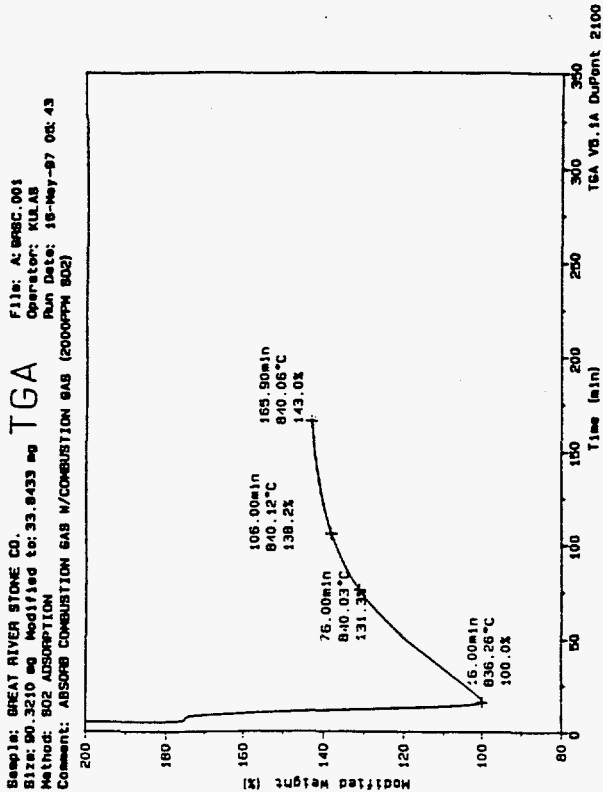
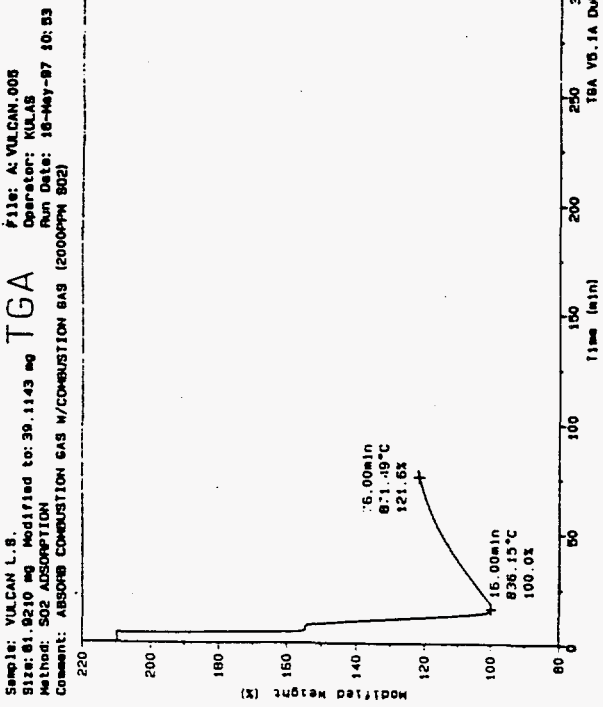


Figure 4. Thermogravimetric analyses of the four limestones tested for SO₂.

TABLE 1

Fuel Analyses for Bench-Scale Tests		
	As-Received Coal	As-Received Limestone
Proximate Analysis, wt%		
Moisture	39.50	NA
Volatile Matter	30.74	
Fixed Carbon	18.39	
Ash	11.37	
Ultimate Analysis, wt%		
Carbon	34.93	NA
Hydrogen	7.31	
Nitrogen	0.62	
Sulfur	0.54	
Oxygen	45.23	
Ash	11.37	
Moisture	39.50	
Ash Composition, % as oxides		
Calcium, CaO	11.27	44.16
Magnesium, MgO	2.80	0.69
Sodium, Na ₂ O	0.32	0.20
Silica, SiO ₂	52.94	12.15
Aluminum, Al ₂ O ₃	19.99	4.67
Ferric, Fe ₂ O ₃	5.62	1.58
Titanium, TiO ₂	0.90	0.16
Phosphorous, P ₂ O ₅	0.03	0.20
Potassium, K ₂ O	1.17	0.90
Sulfur, SO ₃	4.40	0.37
High Heating Value		
Moisture-Free, Btu/lb	10,364	NA
As-Received, Btu/lb	6270	

Test 1

During the first test (without SNCR), steady-state operation was achieved after about 9.4 hr; this is how long it took to stabilize the sulfur retention at 90%. Even though the limestone bed material was presulfated, the higher temperature during actual operation allowed for the bed material to take up more sulfur. Consequently, the bed material had to reach equilibrium before the appropriate limestone add rate could be determined. The superficial gas velocity in the reactor was 3.57 ft/s.

Test 2

The actual test was performed in much the same way as the first test, except that the temperature was increased from 1550° to 1700°F. At this temperature, the sulfur capture reaction is quite inefficient, requiring a much higher calcium-to-sulfur ratio to achieve the same level of

sulfur retention. In fact, under the conditions tested here, 90% sulfur retention could not be attained, even with excessive limestone addition. Steady-state operation was achieved in about 6.9 hr of operation, at which time the bed material and cyclone pots were emptied and ammonia injection was started.

Ammonia injection was from a cylinder of 9% NH_3 in nitrogen, controlled with an ammonia mass flow controller which was calibrated in the lab prior to the test. A stoichiometric ratio of 1 was chosen for the ammonia injection rate, i.e., one ppm NH_3 per ppm NO_x .

In order to gain more information on the effect of ammonia injection on emissions, a second flue gas sample line was added near the top of the reactor (well above the dense bed and below the ammonia injection port). This sample line had dedicated O_2 and SO_2 analyzers, and the NO_x analyzer could be switched between the two sample lines. In theory, this should have allowed frequent monitoring of the NO_x reduction taking place; in practice, it was impossible to keep the reactor sample line open and functioning.

Summary of Results

Upon completion of the tests, data for each steady-state period were averaged. A summary of the process data for each test is shown in Table 2. Complete summaries of test data are included in Appendix C. Under both sets of conditions, the temperature profile in the unit was fairly uniform.

Material Balance

The solids balances for each test are shown in Table 3. The total weights reported are from initial start-up to final shutdown of each test, including pretest periods. The sorbent weight is adjusted to reflect the weight of reactant, CaSO_4 , and does not include the CO_2 in the limestone, which is quickly driven off during calcination and treated as a gas rather than as a solids input. The calculated values for CaO and CaSO_4 are based on the calcium utilization. The closure for both tests was very good.

Flue Gas Emissions

Average flue gas emissions for each steady-state test period, as well as the pretest period prior to Test 2, are presented in Table 4 and discussed in detail in the following sections.

SO_2 Emissions

The average concentration of SO_2 in the flue gas (corrected to 3% O_2) was 112 ppm for Test 1, and 279 ppm for Test 2, corresponding to sulfur retention values of 89% and 74%, respectively. In Test 1, the limestone addition was adjusted until the sulfur retention reached 90%; the Ca:S for this test was 3.6. This may be somewhat higher than what would be expected in a full-scale boiler, for two reasons. First, the temperature at which the test was performed may not be the temperature at which sulfur retention is optimized. Previous studies at the EERC (9) demonstrated the relationship between sulfur capture performance and temperature. Figure 5 shows the sulfur retention for several tests performed with North Dakota lignite on the EERC-1 MWth pilot-scale CFB; as average furnace temperature exceeds 1500°F, sulfur capture drops off

TABLE 2

Summary of Process Data		
Test Number:	MP1-0197	MP-0297
Start Time:	1700, May 21	2137, June 4
Stop Time:	1607, May 23	1955, June 5
Fuel Feed Rate, lb/hr	1.87	1.78
Sorbent Feed Rate, lb/hr	0.10	0.53
Ammonia addition, ppm	NA	340
Reactor Pressure Drop, in. H ₂ O	15.21	17.9
Cyclone Pressure Drop, in. H ₂ O	0.7	0.8
Air Flow Rate, scfm	2.2	2.1
Excess Air, %	21.56	21.91
FG SGV, ¹ ft/s	3.57	3.65
Air and Gas Temperatures, °F		
Preheater Exit	670	642
Plenum	1330	1437
0.25 in.	1473	1588
1.75 in.	1529	1675
3.5 in.	1549	1692
5.0 in.	1554	1692
7.0 in.	1561	1688
9.0 in.	1567	1687
11.0 in.	1576	1683
15.0 in.	1557	1685
23.0 in.	1607	1716
31.0 in.	1582	1710
43.25 in.	1517	1693
Average	1557	1685

¹ Flue gas superficial gas velocity.

dramatically. While excess air and Ca:S varied somewhat between these tests with the North Dakota lignite, temperature was by far the most influential parameter. From the data in this figure it can be seen that the operating temperature for both Malcolm Pirnie tests was higher than the optimum sulfur capture temperature. The resultant Ca:S ratios were therefore higher than industry standards for CFBCs operating at optimal temperatures. Differences in Ca:S ratios between tests are normalized by comparing calcium utilization (calculated by dividing sulfur retention by Ca:S). In Figure 6, the data generated during the Malcolm Pirnie tests showed good agreement with the 1-MW_{th} CFBC data generated using the North Dakota lignite, where maximum calcium utilization occurred at about 1450°F. For higher-rank coals, the optimum temperature is 100° to 150°F higher, and the curve does not drop as dramatically as for a lignite; that is, there is less penalty for operating at a higher temperature. The importance of optimizing operating temperature is evident in Figure 6. A second reason for higher-than-expected Ca:S is that the small scale of this unit results in a fairly short gas residence time, limiting the extent of the sulfur capture reactions. Previous test results at the EERC have shown this effect to be minor compared to the effects of temperature and limestone feed rate.

TABLE 3

Solids Balance		
	MP1-0197	MP1-0297
Input, g		
Coal Ash	4896	3348
Sorbent:		
CaO	815	3172
CaSO ₄	423	466
MgO	15	53
Inerts	449	1526
Bed Material	1352	1350
Total Solids In	7950	9914
Output, g		
Cyclone Ash	4814	7234
Baghouse Ash	1421	673
Bed Material	1595	1337
Misc. Ash	337	649
Total Solids Out	8167	9892
Closure, %	102.7	99.78

An expanded test matrix on a larger test unit, such as the EERC 1 MWth CFBC, could help to determine optimum operating conditions of temperature, excess air, and Ca/S for this fuel, and result in significant savings in capital and operating costs for the full-scale design.

The sulfur capture efficiency for the second test, operated at a higher bed temperature, was much lower than for the first test. The limestone add rate was increased hourly, with minimal effect on sulfur retention; eventually it was decided to stop increasing the ratio of limestone to coal and operate at less than 90% sulfur capture. Like the North Dakota lignite described above (see Figures 5 and 6), it appears that this fuel has a very sharp decrease in sulfur capture performance at increased temperatures, making a sulfur capture of 90% difficult to achieve at a bed temperature of 1700°F.

NO_x Emissions

Flue gas emissions of NO_x (corrected to 3% O₂) measured during Test 1 were 342 ppm, somewhat higher than expected for a relatively low-temperature test. It is not unusual in a unit of this size for NO_x emissions to be significantly higher than for a larger unit operating at the same conditions. One reason is that wall effects (caused by a high surface-to-volume ratio) adversely impact NO_x emissions; another reason is the absence of secondary or overfire air at the bench scale. Table 5 shows NO_x emissions recorded on the EERC FBR (used for the Malcolm Pirnie tests); the EERC-1 MWth pilot-scale CFB; and the Montana-Dakota Utilities (MDU) Heskett Station Unit 2, an 80-MW fluidized-bed boiler. The fuel for all the tests shown was a North Dakota lignite. The data in Table 5 clearly show the impact of bench scale on NO_x emissions,

while the much larger pilot-scale unit with secondary air capabilities provides more realistic emissions levels. While NO_x emissions from the bench-scale tests can safely be used for comparison between tests, they should not be relied on as representing the full scale; to more accurately predict full-scale emissions, a pilot-scale test burn is recommended.

TABLE 4

Emissions Data		
	MP1-0197	MP1-0297
O ₂ , %	3.52	3.73
Excess Air, %	19.52	21.85
CO Content, ppm	721	419
CO Content, corrected ppm ¹	742	437
CO Emission, lb/MM Btu	0.597	0
CO ₂ Content, %	15.6	17.9
CO ₂ Content, corrected % ¹	16.1	18.6
NO _x Content, ppm	326	266
NO _x Content, corrected ppm ¹	337	277
NO _x Emission, lb/MM Btu	0.444	0
N ₂ O Content, ppm	55	46
N ₂ O Content, corrected ppm ¹	57	48
N ₂ O Emission, lb/MM Btu	0.072	0
SO ₂ Content, ppm	97	268
SO ₂ Content, corrected ppm ¹	100	279
SO ₂ Emission, lb/MM Btu	0.184	0
SO ₂ Retention, %	89.3	74
Ca:S Ratio (limestone only)	3.60	14.00
Ca Utilization (limestone only)	24.70	5.30
Ca:S Ratio (total)	5.00	15.40
Ca Utilization (total)	18.00	4.80
Alkali-to-Sulfur (total)	5.00	15.40
Alkali Utilization (total)	17.80	4.80
Avg. Comb. Temp., °F	1557	1685

¹ Corrected to 3% O₂.

For Test 2, baseline NO_x emissions measured during the pretest (before the addition of ammonia) were 371 ppm (corrected to 3% O₂); during ammonia injection, the NO_x dropped to 277 ppm, a 25% reduction. However, when compared to Test 1, the reduction in NO_x was only 19%. A wet-chemistry test performed downstream of the ammonia injection port indicated that 92% of the ammonia was present in the ash, while 8% remained in the gas stream.

Near the end of Test 2, the ammonia injection rate was doubled to a stoichiometric ratio of 2 for 38 min. During this time, the NO_x emissions dropped from 277 to 221 ppm (corrected to 3% O₂). Then the ammonia injection was discontinued, resulting in NO_x emissions of 373 ppm, virtually the same as those measured during the pretest period.

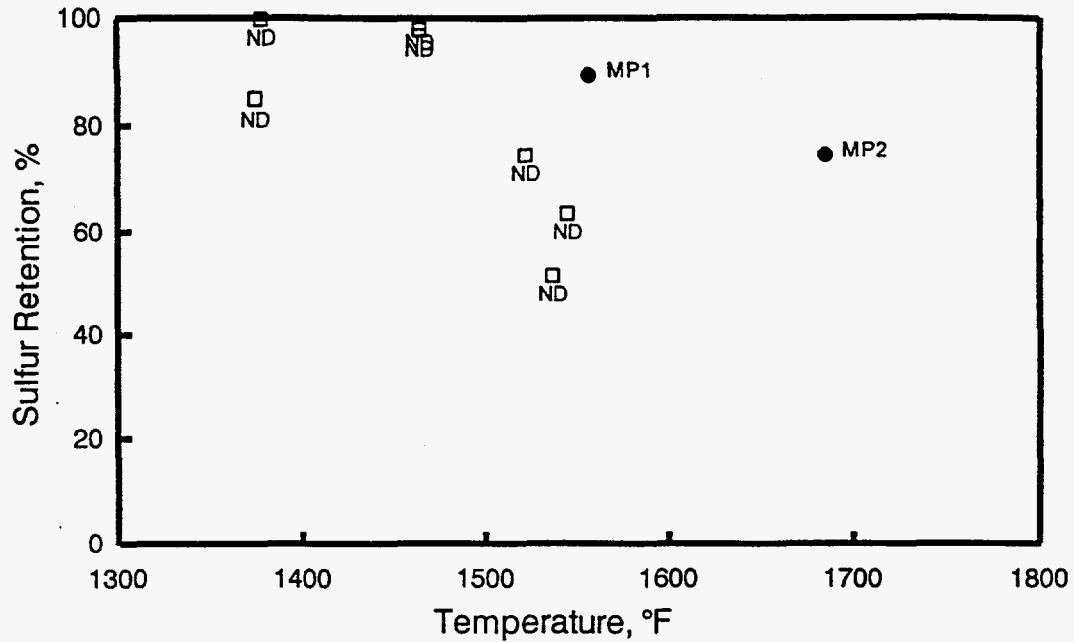


Figure 5. Effect of temperature on sulfur retention present Malcolm Pirnie tests (MP), and a North Dakota lignite (ND). The ND data were generated on a 1-MWth CFBC, with Ca:S ranging from 0.9 to 2.0.

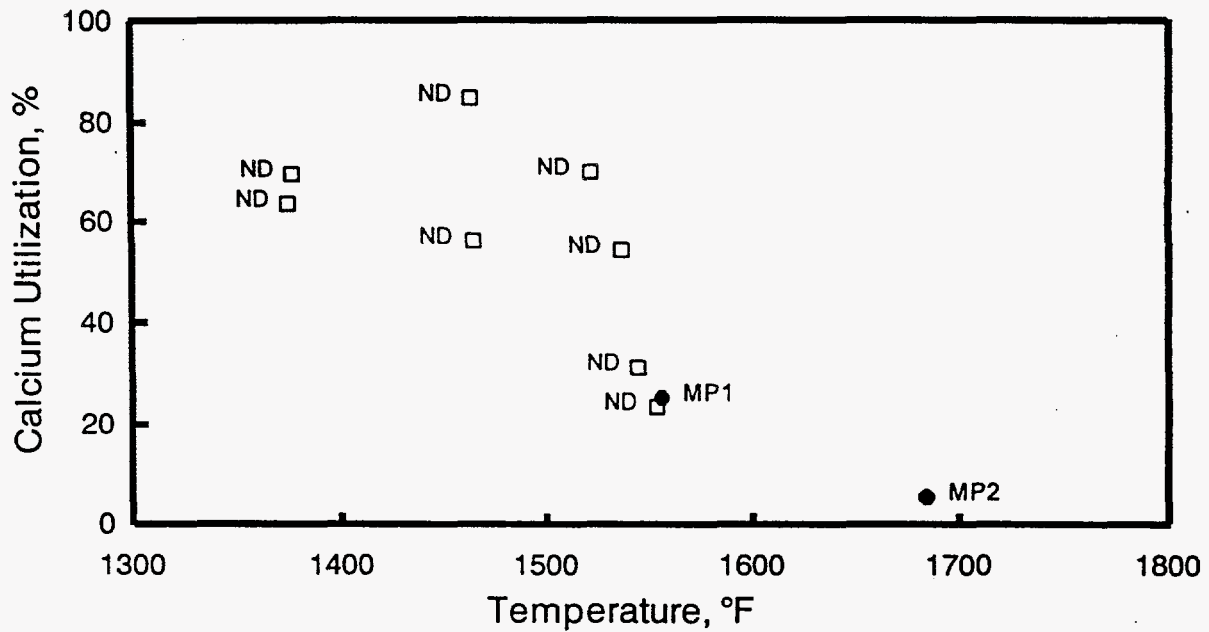


Figure 6. Calcium utilization for limestone used with a North Dakota lignite during a 1-MWth CFBC test compared to current Malcolm Pirnie tests.

TABLE 5

Effect of Boiler Size on NO_x Emissions

Boiler	Heskett Unit 2 ¹	1-MW _{th} CFB ²	1-MW _{th} CFB ³	EERC FBR ⁴
Temperature, °F	1464	1465	1554	1531
Excess Air, %	21.5	23.4	25.7	28.8
NO _x , ppm ¹	150	97	201	363

¹ Personal communication with Paul Eslinger, Results Engineer, MDU Heskett Station.

² EPRI TR-100829 (9).

³ Mann and others, 1996 (10).

⁴ Corrected to 3% O₂.

Because sulfur capture with this fuel decreases dramatically as temperature increases and SNCR requires higher temperatures for effective NO_x reduction, it appears as though an operating temperature chosen to optimize one emission will severely compromise the other. The limited number of test points in this program was not sufficient to optimize sulfur capture performance; the desired operating temperature for this fuel may be even lower than the 1550°F of Test 1. Other NO_x reduction strategies should be considered; lower temperature and lower Ca:S will result in reduced NO_x emissions, and optimizing overfire air could further reduce NO_x.

N₂O Emissions

Average N₂O emissions for Test 1 were 53 ppm (corrected to 3% O₂). At the higher temperature of Test 2, N₂O dropped to 39 ppm prior to NH₃ injection, as expected. However, the addition of ammonia increased N₂O emissions to 48 ppm, resulting in very little change from Test 1.

Leaching Characterization

A number of leaching tests were selected for the leaching characterization. A summary of the leaching procedures used follows.

Toxicity Characteristic Leaching Procedure

The TCLP (1) is the EPA regulatory leaching procedure, through RCRA, for the determination of the characteristic of hazardousness of wastes. Land disposal of materials identified as hazardous by this leaching procedure is prohibited by EPA. The TCLP has also been adopted by many state regulatory agencies to provide leaching information on solid wastes (nonhazardous) that are not federally regulated. This test uses end-over-end agitation and a 20-to-1 liquid-to-solid ratio with an 18-hr equilibration time. Two leaching solutions are specified for use with this test. Leaching Solution 1 is an acetate buffer prepared with 5.7 mL of glacial acetic acid per liter of distilled deionized water, which is adjusted to pH 4.93 with 1N sodium hydroxide solution. Leaching Solution 2 is an acetic acid solution prepared by diluting 5.7 mL of glacial acetic acid to 1 liter with distilled deionized water. This solution will have a pH of 2.88. The TCLP specifies a test to determine the alkalinity of the waste to be leached which, in turn, determines which leaching solution should be used. More alkaline materials utilize Solution

No. 2, while less alkaline materials are leached with Solution No. 1. The CCSRs evaluated in this project were highly alkaline in nature and required TCLP leaching Solution No. 2.

Synthetic Precipitation Leaching Procedure

The SPLP (2) is a test that was developed to simulate leaching from contact of a material by acid precipitation. Acid precipitation for use in this leaching test is prepared in the laboratory using one of two formulations, depending on whether precipitation to be simulated is east or west of the Mississippi River. Extraction fluid No. 1 to simulate conditions east of the Mississippi River was chosen for use in this project. This is the more acidic of the two solutions and is prepared by adding 60:40 wt% sulfuric-nitric acid to distilled deionized water to achieve a pH of 4.20 ± 0.05 . Extraction fluid No. 2 is prepared in the same way except to a final pH of 5.00 ± 0.05 . Extraction conditions are the same as for TCLP or SGLP using end-over-end agitation and an 18-hr equilibration time with a 20-to-1 liquid-to-solid ratio.

Synthetic Groundwater Leaching Procedure

The SGLP (1) was developed as a generic leaching test to be applied to materials to simulate actual field leaching conditions. Since the TCLP was designed to simulate leaching in a sanitary landfill under codisposal conditions, it is not appropriate to evaluate leaching of coal conversion by-products in typical disposal or utilization scenarios. To provide more appropriate and predictive information for coal conversion by-products and other unique materials, a leaching test was developed using the same basic protocol as the TCLP, but allowing for the appropriate leaching solution chemistry. Test conditions are end-over-end agitation, a 20-to-1 liquid-to-solid ratio, and an 18-hr equilibration time.

Long-Term Leaching

A LTL procedure using distilled water was included to identify effects associated with any mineralogical changes that may occur in the waste forms upon long-term contact with water. Separate samples were analyzed after 18 hours, 30 days, and 60 days for each of the five materials analyzed. It has been found, in a previous research project (12), that on long-term contact with water, certain coal conversion solid waste materials form secondary hydrated phases with mineralogical and chemical compositions different from any of the material in the original ash. In another research project, it was demonstrated that the formation of these hydrated phases was often accompanied by dramatic decreases in solution concentrations of oxyanionic species such as borate, chromate, selenate, and vanadate (4). The decrease in concentration of these elements would not be predicted from the results of short-term leaching tests.

Morphology was determined using SEM. Sulfate:sulfite ratios were also determined using an iodometric titration for the determination of sulfite.

RESULTS AND DISCUSSION

PIXE Results

Six materials were characterized using PIXE. These were coal, limestone, non-SNCR spent bed material, SNCR spent bed material, non-SNCR fly ash, and SNCR fly ash. PIXE

characterization revealed the presence of several trace elements in the samples. On discussion with Malcolm Pirnie, it was decided that boron, copper, molybdenum, nickel, vanadium, and zinc would be added to the list of RCRA elements to be determined in leaching experiments. A complete summary of PIXE characterization is included in Appendix A.

General Ash Characterization

Samples of SNCR and non-SNCR spent bed material and fly ash were analyzed for sulfite to determine the relative sulfite:sulfate ratio. Sulfur content was determined using a combustion technique followed by infrared detection of SO₂. This technique provides a determination of total sulfur content. Sulfite was determined using an iodometric titration with visual end-point determination using starch indicator. Moisture and LOI were determined gravimetrically using standard ASTM techniques. Moisture determination was modified slightly to account for any water of hydration that might be associated with calcium sulfate. Calcium sulfate water of hydration requires a temperature of 230°C for complete release. The standard ASTM C-311 determination of moisture in ash utilizes a temperature of 105° to 110°C. LOI was determined at 750 ± 50°C. Results of these determinations are shown in Table 6.

TABLE 6

Sulfate, Sulfite, Moisture, and LOI Determination

Sample	SO ₃	SO ₂	Moisture	LOI
Spent Bed Material, non-SNCR	27.56	<0.05	0.24	0.32
Fly Ash, non-SNCR	5.99	<0.05	0.56	5.37
Spent Bed Material, SNCR	18.40	<0.05	0.57	1.80
Fly Ash, SNCR	4.77	<0.05	0.40	6.16

All values are in weight percent.

XRD Characterization

Mineralogical characterization was carried out using XRD, which measures the interplanar spacing of crystalline compounds. The resulting pattern is unique to each crystalline compound, which allows its identification. Complex materials, such as coal combustion fly ash, often contain several crystalline phases that can be identified by XRD, as well as an amorphous (noncrystalline) component that cannot be identified by XRD.

The samples analyzed by XRD include the limestone to be used as the bed material, non-SNCR fly ash, non-SNCR fly ash after 60 days of leaching, non-SNCR spent bed material before and after 60 days of leaching, non-SNCR composite ash after 60 days of leaching, SNCR fly ash before and after 60 days of leaching, SNCR spent bed material before and after 60 days of leaching, and SNCR composite ash after 60 days of leaching. The diffractograms can be found in Appendix B. Non-SNCR results are shown in Table 7, and SNCR matter is shown in Table 8. There were only minor differences between the dry products from both the SNCR and the non-SNCR combustion tests. Dry spent bed material contained large amounts of calcium sulfate, identified as anhydrite, as well as minor amounts of lime. Dry ash from both combustion tests

TABLE 7

Non-SNCR By-Products XRD Results

Limestone	Leached Fly Ash Non-SNCR	Leached Spent Bed Material Non-SNCR
Calcite (major)	Quartz (minor)	Gypsum (major)
Quartz (minor)	Ettringite (minor)	Ettringite (minor)
Lime (questionable)	Calcite (minor)	Quartz (minor)
	Anhydrite (minor)	

Fly Ash Non-SNCR	Spent Bed Material Non-SNCR	Leached Composite Ash
Quartz (minor)	Anhydrite (major)	Ettringite (minor)
Anhydrite (minor)	Quartz (minor)	Quartz (minor)
Calcite (minor)	Hematite (minor)	Calcite (minor)
Lime (minor)	Lime (minor)	Gypsum (minor)
Sodalite (questionable)	Thenardite (questionable)	Lime (minor)
Tricalcium aluminate (questionable)	Clinopyroxene (questionable)	
	Iron (questionable)	

TABLE 8

SNCR By-Products XRD Results

Fly Ash SNCR	Spent Bed Material SNCR	Leached Composite Ash SNCR
Quartz (minor)	Anhydrite (major)	Ettringite (minor)
Lime (minor)	Quartz (minor)	Gypsum (minor)
Anhydrite (minor)	Lime (minor)	Calcite (minor)
Calcite (minor)	Portlandite (minor)	Quartz (minor)
Sodalite (minor)	Sodalite (minor)	Lime (questionable)
Clinopyroxene (questionable)	Plagioclase (questionable)	
Tricalcium Aluminate (questionable)	Clinopyroxene (questionable)	
Portlandite (questionable)	Tricalcium aluminate (questionable)	
Iron (questionable)		

Leached Fly Ash SNCR	Leached Spent Bed Material SNCR
Quartz (minor)	Gypsum (minor)
Ettringite (minor)	Ettringite (minor)
Calcite (minor)	Quartz (minor)
Gypsum (minor)	Calcite (minor)
Unidentified Peak at 8.13 and 4.07Å	

were similar. The spent bed material was deficient in sources of aluminum. This is reflected in the mineralogy of the leached material where ettringite, normally the primary hydrated phase, is identified as a minor constituent. Leached materials also show parallel trends, with gypsum, a hydrated form of anhydrite, present in both samples. Ettringite was formed in all of the leached samples and was likely still forming at the end of the test at 60 days. The presence of lime in the leached composite ash as well as in the leached spent bed material is due to the presence of some of the original calcined bed material that had not yet reacted with water. There was a striking similarity between SNCR and non-SNCR samples, as would be expected. Ammonia addition was expected to impact only the trace elements if there was even a measurable effect. As can be seen in the figures in Appendix B, the diffractograms were rather cluttered and complex. This is expected in combustion residues. Questionable phases have been listed as questionable because of the large number of phases that can be fit into the diffraction patterns of these materials. The questionable phases have been selected on the basis of their likelihood to form under either combustion or leaching conditions.

Permeability and Density Determination

Permeability was determined using a standard test called the falling head method. Permeability was conducted on composite ash, which consisted of a mixture of spent bed material and fly ash in the ratio produced during the combustion test. The results of this determination are summarized in Table 9. Wet density was determined using a Harvard miniature compaction apparatus. For wet compacted densities, 30% water was added to the samples. A measured moisture determination was made after the actual densities were determined. The compacted dry density was performed by vibrating a given mass of ash in a graduated cylinder until no additional settling could be observed. No additional force was applied. The results of density determinations are shown in Table 10.

TABLE 9

Permeability

Sample	Moisture Content, %	Wet Density, pcf	Permeability, cm/s
Ash, non-SNCR	34.2	82.8	2.63×10^{-4}
Ash, SNCR	39.8	101.7	6.57×10^{-5}

TABLE 10

Density (non-SNCR)

	Fly Ash	Spent Bed Material
Dry, Loose Density	33.0	53.0
Dry, Compacted Density	45.1	61.4
Wet, Compacted Density at 100% Compaction	68.1	83.0
Wet, Compacted Density at 70% Compaction	64.1	78.2
Moisture Content	23.8	27.0

All values are pounds per cubic foot.

Particle-Size Distribution

Particle-size was determined using standard sieves by measuring the amount of material remaining on each of a series of calibrated sieves. The results of this determination are shown in Tables 11 and 12 and graphically in Figures 7-10.

Ash Morphology and Mineralogy

Two samples, referred to in this section as the non-SNCR and the SNCR samples, were examined by SEM to determine ash morphology and point chemistry by means of EPMA (electron probe microanalysis). EPMA data were not quantified and were used qualitatively to identify the various mineral grains in the sample.

TABLE 11

Particle-Size Distribution of Spent Bed Material

Screen Mesh	Spent Bed Material non-SNCR, % retained	Spent Bed Material SNCR, % retained
8 (2360 microns)	0.8	2.6
10 (2000 microns)	1.9	1.6
12 (1700 microns)	4.1	4.2
20 (850 microns)	39.0	39.4
30 (600 microns)	27.8	27.6
50 (300 microns)	23.5	19.2
<50	2.9	5.3
Total	100.0	99.9

TABLE 12

Particle-Size Distribution of Fly Ash

Screen Mesh	Fly Ash, non-SNCR	Fly Ash, SNCR
20 (850 microns)	1.3	1.2
30 (600 microns)	2.8	2.8
50 (300 microns)	21.0	16.1
100 (150 microns)	7.2	6.6
140 (106 microns)	15.8	17.8
170 (90 microns)	12.5	0.5
200 (75 microns)	12.8	5.0
270 (53 microns)	2.3	6.9
325 (45 microns)	12.8	14.2
400 (38 microns)	11.0	19.9
< 400	0.5	2.1
Total	100.0	100.0

non-SNCR Spent Bed Material Particle Size Distribution

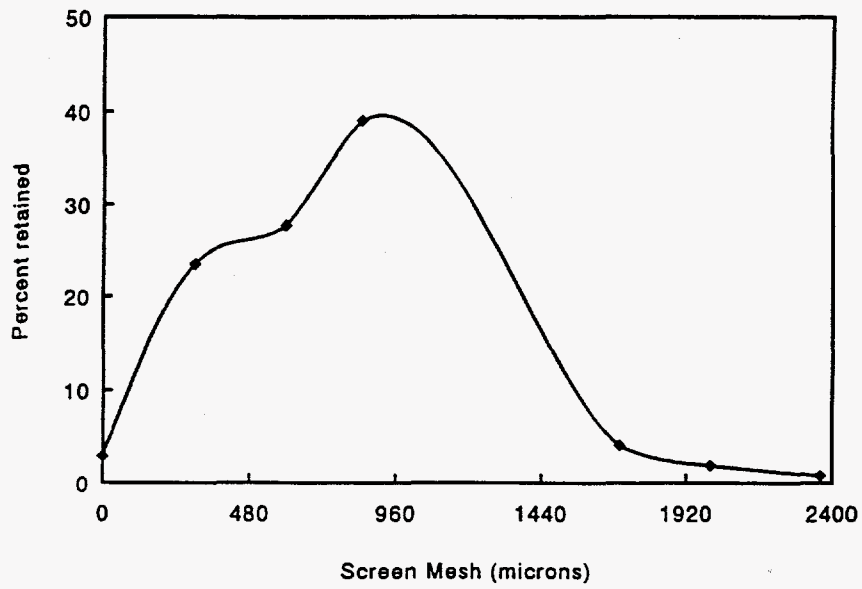


Figure 7. Non-SNCR spent bed material – particle-size distribution.

SNCR Spent Bed Material Particle Size Distribution

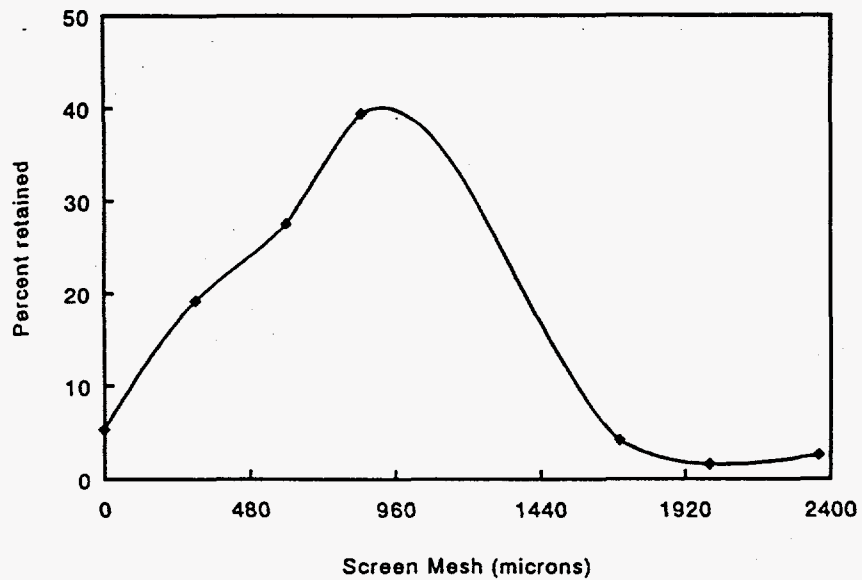


Figure 8. SNCR spent bed material – particle-size distribution.

non-SNCR Fly Ash Particle Size Distribution

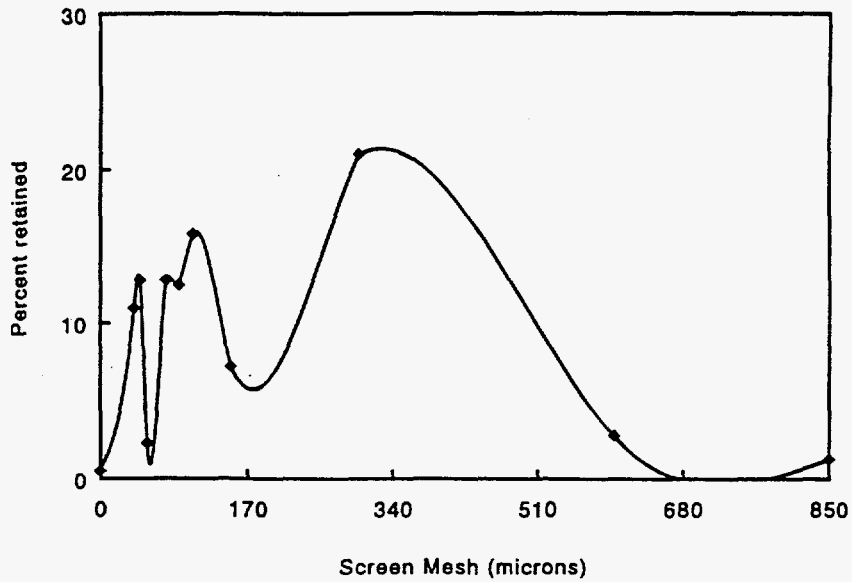


Figure 9. Non-SNCR fly ash – particle-size distribution.

SNCR Fly Ash Particle Size Distribution

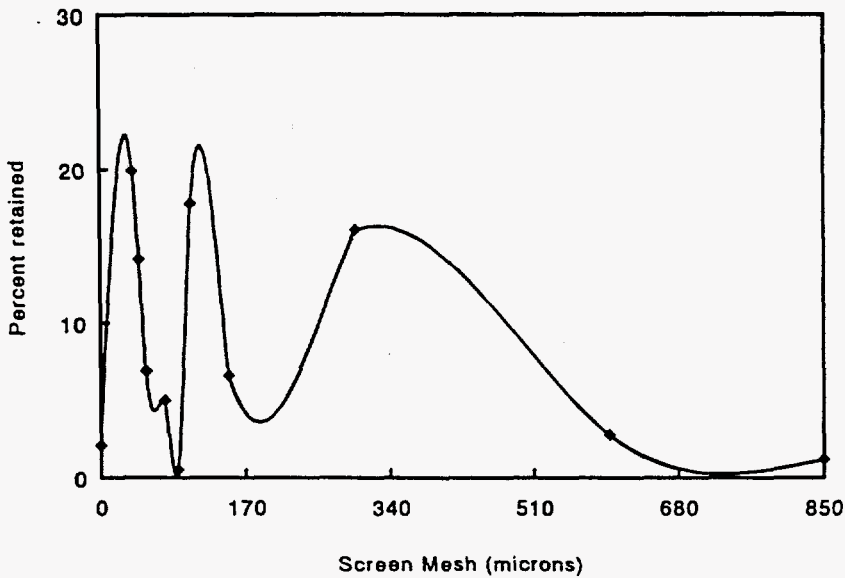


Figure 10. SNCR fly ash – particle-size distribution.

The most prominent feature of the two samples supplied is the angularity of the ash particles. This is consistent with other FBC ashes derived from lignite (13). Conventional pulverized coal fly ash is generally spherical, with a majority of particles less than 50 μm . The SNCR and non-SNCR samples both contain angular particles that are generally less than 0.5 mm (500 μm). There appears to be no difference in particle size and degree of angularity between the SNCR and non-SNCR samples.

The difference between this ash and pulverized coal fly ash is primarily because of the temperature at which the ash is formed. Pulverized coal fly ash is formed at temperatures often exceeding 1600°C, at which point most of the inorganic compounds have either melted or have been incorporated into a melt. The temperatures attained for these samples were not high enough to cause much melting. There was likely very little liquid phase, if any, associated with the formation of these samples. Temperatures were high enough to cause out-gassing, as evidenced by bubbles in some grains.

Figure 11 is a SEM photomicrograph of the non-SNCR sample showing a wide size distribution and the angularity of the samples examined. The large, solid, grey grains are quartz grains. The porous grains were clay agglomerates that have been heated to temperatures high enough to dehydrate and deform the clay structures, but not high enough to cause melting. Figure 12 is a backscattered electron image of the same area. A backscattered image shows compositional differences as different levels of grey, with lower-atomic-number compounds darker and higher-atomic-number compounds brighter. The epoxy that the grains are mounted in is the lowest-atomic-number compound and shows up as the darkest grey. Several chemical point analyses were taken to determine the chemistry associated with the grey levels. The elements were listed in order of highest concentration to lowest. Point 1 was the only iron-

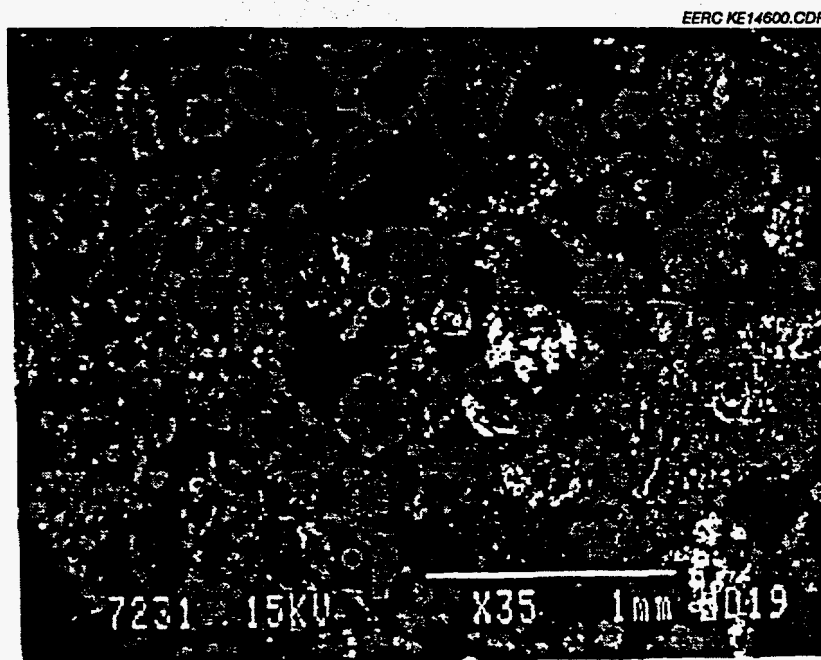


Figure 11. SEM secondary image of the non-SNCR sample.

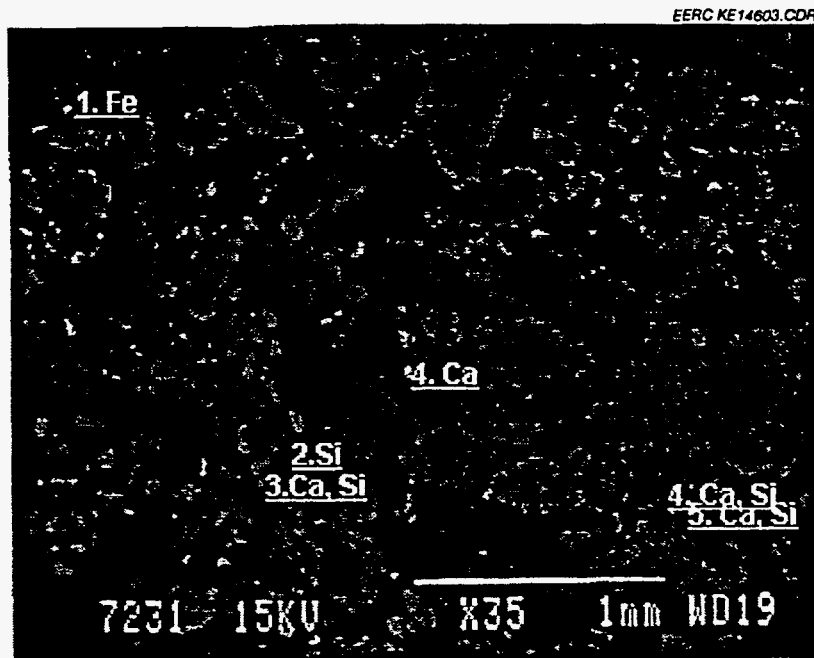


Figure 12. SEM backscattered image of the same area as Figure 11. The elements are listed from greatest concentration to least.

bearing grain observed in these samples. It was likely derived from pyrite but is thermally dissociated at about 450°C. Point 2 is a quartz grain, and Point 3 is a calcium silicate coating on the quartz grain. Points 4 and 5 are calcium silicates derived from the thermal dissociation of calcite releasing CaO, which can react with silicates freed from the destruction of clay minerals. This reaction can take place at temperatures below 800°C (13).

Figure 13 is a SEM image of the SNCR sample showing the same features as the non-SNCR sample. No differences in particle size or angularity were noted. Figure 14 is a backscattered electron image of the same area as Figure 13. Point 1 on Figure 14 is a composite grain composed primarily of clay minerals. Point 2 was on a coating surrounding the composite grain. The coating is primarily calcium sulfate, and the Si and Al are from the grain being coated rather than part of the coating. Point 3 is primarily carbon that was uncombusted. Some Ca was measured in the particle, but the x-ray counts were very low, and the detector cannot detect elements lower than Na (Atomic Number 11). Point 4 contained iron only, and Point 5 contained Si and Al only. These grains were likely derived from pyrite and clay minerals, respectively.

Leaching

Leaching data have been reported in table format. Tables 13 through 20 contain data for bulk composition and leaching of trace elements, including the eight RCRA elements arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver, in the first section of the tables and non-RCRA elements, including boron, copper, molybdenum, nickel, vanadium, and zinc, in the second section of the tables. Tables 21 through 27 contain data for bulk composition and leaching of major and minor elements, including aluminum, calcium, iron, magnesium, manganese, phosphorus, potassium, silicon, sodium, strontium, titanium, and sulfur. Bulk

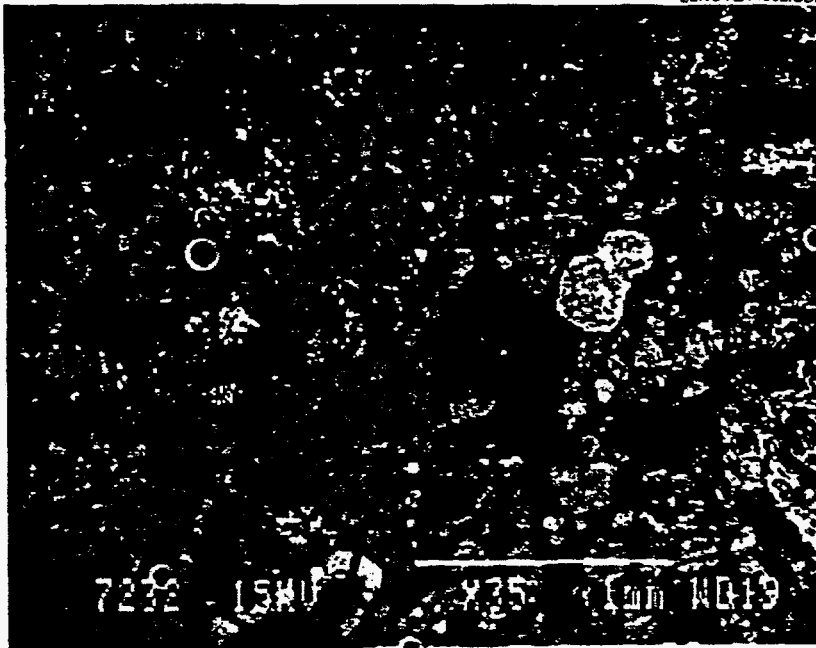


Figure 13. SEM secondary image of the SNCR sample.

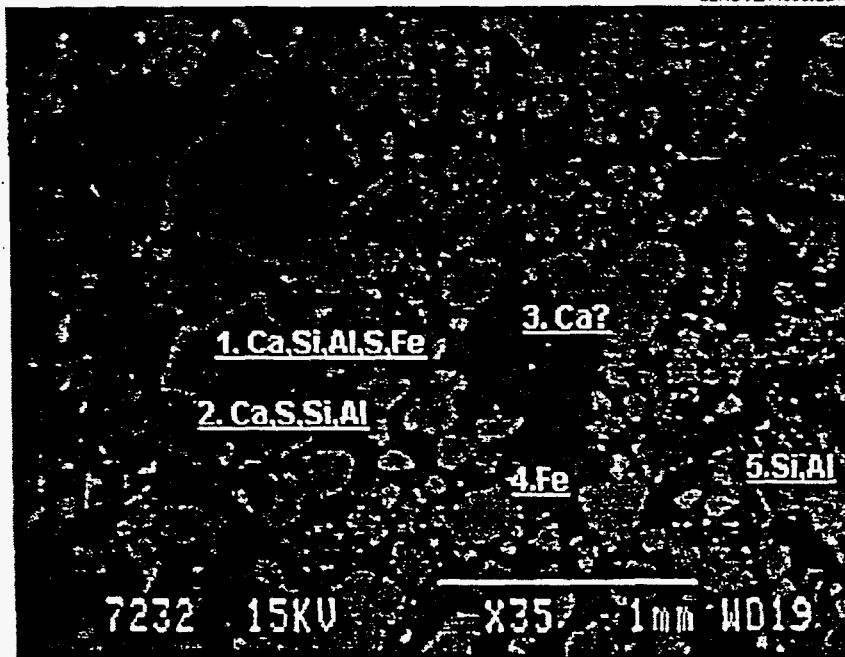


Figure 14. SEM backscattered image of the same area as Figure 13.

concentration, in the first column, represents the total content of the material as received by the laboratory. Maximum, the second column, represents the theoretical maximum possible solution concentration and is a calculated value representing the maximum solution concentration that could be achieved during leaching at a 20:1 liquid-to-solid ratio. This maximum calculated value would be achieved if the ash or related analyte were totally dissolved and represents a true worst-case scenario. In reality, this value would be difficult to achieve because of the limited solubility of most combustion by-products and because of factors related to the solubility of many trace elements in natural systems where solution concentration is often limited by the formation of carbonates or other minerals, as trace elements combine with naturally occurring constituents of groundwater. Calculated maximum concentrations were all well below RCRA limits except for chromium and selenium in the non-SNCR fly ash. For these two elements in this sample, the calculated maximum for chromium was just at 5.0 mg/L, which is the RCRA limit, and selenium was at 1.10, with a RCRA limit of 1.0. It must be kept in mind, however, that these are calculated maximum levels and represent total dissolution of the ash or total mobilization of the analyte. Primary drinking water limits were exceeded for barium and chromium in some of the leachates. Generally, concentrations of regulated trace elements were well below drinking water limits. There is some sodium, potassium, and strontium present as would be expected by the generalization that sodium and potassium compounds are soluble, with strontium behaving somewhat like calcium. Much of the sodium and potassium that are available were deposited on the surface of ash particles during the combustion process. This is partially due to the fact that some of the sodium and potassium is present in the coal as salts of carboxylic acid groups and during combustion forms inorganic vapors that condense on the surface of the cooling ash particles. In LTL, it was generally observed that sodium and potassium concentrations continued to increase with respect to time, while calcium and sulfate concentrations decreased.

A maximum concentration was not calculated for the major and minor elements. The remaining columns reflect the concentrations of various constituents as determined using available laboratory techniques. Values in the tables reflect elemental concentrations. This is simply a reporting convention, since it is known that several of the elements, most notably sulfur, is actually present in leachates as sulfate. The data for leaching of composite ash samples were generated in triplicate. Data in Tables 13 through 27 contain average values for these samples. A complete set of data for these leachings, including the calculated average and standard deviation, are shown in Appendix C.

It can be seen from the data that there is no significant variation between replicate samples in leaching results. This is true for both trace and major/minor elements. It was interesting to note the difference in sulfur values for SPLP leaching of coal. It had been found that there was oxidizable pyrite in the coal, and this is likely the reason for the difference in sulfur for this particular sample. Limestone used as the bed material contained low concentrations of trace elements, as indicated in Table 19. Even in a calculated worst-case scenario assuming total dissolution, calculated concentrations would be below RCRA limits by an order of magnitude or more for most of the RCRA trace elements. In Tables 13 through 27, less-than numbers are lower levels of quantitation (LLQ) rather than detection limits. Actual detection limits are generally 10 times lower than the LLQ.

TABLE 13

Fly Ash (non-SNCR) Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, mg/L	SGLP, 30-day, mg/L	SGLP, 60-day, mg/L
Arsenic	26	1.3	<0.008	<0.004	<0.004	<0.004
Barium	824	41.2	0.92	0.32	1.9	0.26
Cadmium	0.42	0.02	<0.0003	<0.0003	<0.0003	<0.0003
Chromium	100	5.00	0.22	0.18	0.063	0.24
Lead	36.9	1.85	<0.002	<0.002	<0.002	<0.002
Mercury	0.292	.0146	<0.0001	<0.0001	0.0028	<0.0001
Selenium	22	1.10	0.0420	0.0340	0.0098	0.031
Silver	<0.4	<.02	<0.0003	<0.0003	<0.0003	<0.0003
Boron	507	25.35	<0.2	<0.2	<0.2	<0.2
Copper	148	7.40	<0.1	<0.1	<0.1	<0.1
Molybdenum	10	0.5	0.20	0.17	0.11	0.24
Nickel	62	3.10	<0.07	<0.07	<0.07	<0.07
Vanadium	215	10.75	<0.04	<0.04	<0.04	0.12
Zinc	62.2	3.11	<0.03	<0.03	<0.03	<0.03
pH			11.7	12.4	11.6	11.8

TABLE 14

Spent Bed Material (non-SNCR) Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, mg/L	SGLP, 30-day, mg/L	SGLP, 60-day, mg/L
Arsenic	18	0.9	<0.008	<0.004	<0.004	<0.004
Barium	367	18.4	0.12	0.31	<0.1	<0.1
Cadmium	0.78	0.04	0.00087	<0.0003	<0.0003	<0.0003
Chromium	36	1.8	0.20	0.11	0.53	0.53
Lead	42.5	2.13	<0.002	<0.002	<0.002	<0.002
Mercury	<.01	<0.020	<0.0001	<0.0001	<0.0001	<0.0001
Selenium	<0.8	<0.04	<.008	<0.008	<0.008	<0.008
Silver	<0.4	<0.02	<.0003	<0.0003	<0.0003	<0.0003
Boron	140	7.00	1.4	<0.2	<0.2	<0.2
Copper	66	3.30	<0.1	<0.1	<0.1	<0.1
Molybdenum	5	0.25	0.067	0.065	0.15	0.15
Nickel	22	1.10	<0.07	<0.07	<0.07	<0.07
Vanadium	82	4.10	0.20	<0.04	0.082	0.16
Zinc	68.6	3.43	0.15	<0.03	<0.03	<0.03
pH			6.05	12.2	10.6	10.6

TABLE 15

Composite Ash (non-SNCR) Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, Avg., mg/L	SGLP 30-day, Avg., mg/L	SGLP, 60-day Avg., m/L
Arsenic			<0.008	<0.004	<0.004	<0.004
Barium			0.64	0.26	<0.1	<0.1
Cadmium			<0.0003	<0.0003	<0.0003	<0.0003
Chromium			0.21	0.21	0.44	0.32
Lead			<0.002	0.0022	<0.0026	0.0044
Mercury			<0.0001	<0.0001	<0.0001	<0.0001
Selenium			0.042	0.036	0.033	0.023
Silver			<0.0003	<0.0003	<0.0003	<0.0003
Boron			<0.2	<0.2	<0.2	<0.2
Copper			<0.1	<0.1	<0.1	<0.1
Molybdenum			0.19	0.17	0.27	0.27
Nickel			<0.07	<0.07	<0.07	<0.07
Vanadium			0.075	<0.04	0.21	0.26
Zinc			<0.03	<0.03	<0.03	<0.03
pH			12.3	12.3	11.0	11.03

TABLE 16

Fly Ash SNCR Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, mg/L	SGLP, 30-day, mg/L	SGLP, 60-day, mg/L
Arsenic	17	0.85	<0.008	<0.004	<0.004	<0.004
Barium	510	25.5	2.84	0.90	6.80	7.54
Cadmium	0.3	0.02	<0.0003	<0.0003	<0.0003	<0.0003
Chromium	78	3.90	0.10	0.11	0.0054	0.015
Lead	15.5	0.775	<0.002	<0.002	0.0021	0.0022
Mercury	0.105	0.0053	<0.0001	<0.0001	<0.0001	<0.0001
Selenium	6.2	0.31	<0.008	<0.008	<0.008	<0.008
Silver	<0.4	<0.02	<0.0003	<0.0003	<0.0003	<0.0003
Boron	270	13.50	<0.2	<0.2	<0.2	<0.2
Copper	78	3.90	<0.1	<0.1	<0.1	<0.1
Molybdenum	7.2	0.36	0.071	0.064	0.014	0.024
Nickel	45	2.25	<0.07	<0.07	<0.07	<0.07
Vanadium	123	6.15	<0.04	<0.04	<0.04	<0.04
Zinc	58	2.90	<0.03	<0.03	<0.03	<0.03
pH			12.3	12.6	12.2	12.6
Ammonia	11.2	0.56				

TABLE 17

Spent Bed Material SNCR Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, mg/L	SGLP, 30-day, mg/L	SGLP, 60-day, mg/L
Arsenic	29	1.45	<0.008	<0.004	<0.004	<0.004
Barium	376	18.8	0.99	0.47	<0.1	0.11
Cadmium	0.43	0.02	<0.0003	<0.0003	<0.0003	<0.0003
Chromium	67	3.35	0.50	0.14	0.89	1.07
Lead	33.6	1.68	<0.002	<0.002	<0.002	<0.002
Mercury	<0.01	<0.0005	<0.0001	<0.0001	<0.0001	<0.0001
Selenium	<0.8	<0.04	<0.008	<0.008	<0.008	<0.008
Silver	<0.4	<0.02	<0.0003	<0.0003	<0.0003	<0.0003
Boron	160	8.00	<0.2	<0.2	<0.2	<0.2
Copper	45	2.25	<0.1	<0.1	<0.1	<0.1
Molybdenu	4.8	0.24	0.088	0.054	0.086	0.085
Nickel	23	1.15	<0.07	<0.07	<0.07	<0.07
Vanadium	69.3	3.47	<0.04	<0.04	<0.04	<0.04
Zinc	85.2	4.26	<0.03	<0.03	<0.03	<0.03
pH			12.1	12.4	12.2	12.6
Ammonia	2.8	0.14				

TABLE 18

Composite Ash SNCR Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	TCLP, mg/L	SGLP, 18-hr, Avg., mg/L	SGLP, 30-day Avg., mg/L	SGLP, 60-day, Avg., mg/L
Arsenic			<0.008	<0.004	<0.004	<0.004
Barium			2.68	1.17	6.44	6.61
Cadmium			<0.0003	<0.0003	<0.0003	<0.0003
Chromium			0.076	0.085	<0.0095	0.021
Lead			0.0036	0.0032	<0.0023	0.0031
Mercury			<0.0001	<0.0001	<0.0001	<0.0001
Selenium			<0.008	<0.008	<0.008	<0.008
Silver			<0.0003	<0.0003	<0.0003	<0.0003
Boron			<0.2	<0.2	<0.2	<0.2
Copper			<0.1	<0.1	<0.1	<0.1
Molybdenu			0.060	0.0593	0.0217	<0.02
Nickel			<0.07	<0.07	<0.07	<0.07
Vanadium			<0.04	<0.04	<0.04	<0.04
Zinc			<0.03	<0.03	<0.03	<0.03
pH			12.3	12.5	12.3	12.6

TABLE 19

Coal Trace Elements

Element	Bulk, µg/g	Maximum, mg/L	SPLP 1, mg/L	SPLP 2, mg/L
Arsenic	4.5	0.22	<0.004	<0.004
Barium	985	49	<0.1	<0.1
Cadmium	0.15	0.075	<0.0003	<0.0003
Chromium	14	0.70	<0.05	<0.05
Lead	6.56	0.328	<0.002	<0.002
Mercury	0.096	0.0048	<0.0001	<0.0001
Selenium	5.8	0.29	0.019	0.020
Silver	<0.4	<0.02	<0.0003	<0.0003
Boron	83	4.15	1.0	1.0
Copper	25.1	1.26	<0.1	<0.1
Molybdenum	1.7	0.09	0.0078	0.0073
Nickel	6.0	0.30	<0.07	<0.07
Vanadium	35.5	1.78	<0.04	<0.04
Zinc	9.09	0.45	<0.03	<0.03
pH			7.62	7.15

TABLE 20

Limestone Trace Elements

Element	Bulk, µg/g	Maximum, mg/L
Arsenic	3.8	0.19
Barium	<3600	<180
Cadmium	0.31	0.02
Chromium	24	1.20
Lead	4.83	0.24
Mercury	0.014	0.0007
Selenium	<0.8	<0.04
Silver	<0.4	<0.02
Boron	52	2.60
Copper	6.3	0.32
Molybdenum	3.37	0.17
Nickel	11	0.55
Vanadium	26	1.30
Zinc	36	1.80

TABLE 21

Fly Ash (non-SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr, mg/L	SGLP, 60-day, mg/L
Aluminum	15.4	<0.7	13.7
Calcium	23.9	1070	77.5
Iron	3.71	<0.08	<0.08
Magnesium	2.26	<0.03	<0.03
Manganese	0.15	0.011	<0.01
Phosphorus	<0.4	<0.3	<0.3
Potassium	0.93	7.4	66
Silicon	36.1	0.59	4.4
Sodium	0.29	4.66	27.0
Strontium	0.23	25.4	7.89
Titanium	0.72	<0.02	<0.02
Sulfur	5.99	260	16.7

TABLE 22

Spent Bed Material (non-SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr, mg/L	SGLP, 60-day, mg/L
Aluminum	11.1	<0.7	1.8
Calcium	27.5	1190	620
Iron	2.76	<0.08	0.63
Magnesium	1.02	<0.03	0.52
Manganese	0.049	<0.01	0.026
Phosphorus	0.13	<0.3	<0.3
Potassium	0.68	4.8	44
Silicon	27.9	0.58	21.2
Sodium	0.24	3.89	14.5
Strontium	0.13	9.00	15.3
Titanium	0.41	<0.02	0.060
Sulfur	27.56	523	482

TABLE 23

Composite Ash (non-SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr Avg., mg/L	SGLP, 60-day Avg., mg/L
Aluminum		<0.7	1.23
Calcium		1130	211
Iron		<0.08	<0.08
Magnesium		<0.03	0.14
Manganese		0.01	<0.01
Phosphorus		<0.3	<0.3
Potassium		6.8	67.7
Silicon		0.36	10.0
Sodium		4.5	27.5
Strontium		23.9	12.6
Titanium		<0.02	<0.02
Sulfur		354	194

TABLE 24

Fly Ash (SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr, mg/L	SGLP, 60-day, mg/L
Aluminum	9.62	<0.7	1.2
Calcium	39.2	1170	688
Iron	2.69	<0.08	<0.08
Magnesium	1.59	<0.03	<0.03
Manganese	0.11	<0.01	<0.01
Phosphorus	0.20	<0.3	<0.3
Potassium	0.96	18	197
Silicon	24.1	<0.5	<0.5
Sodium	0.23	4.33	35.2
Strontium	0.21	25.7	52.2
Titanium	0.53	<0.02	<0.02
Sulfur	4.77	82.6	0.76

TABLE 25

Spent Bed Material (SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr, mg/L	SGLP, 60-day, mg/L
Aluminum	8.64	<0.7	<0.7
Calcium	36.5	1020	1230
Iron	2.70	<0.08	<0.08
Magnesium	1.04	<0.03	<0.03
Manganese	0.057	<0.01	<0.01
Phosphorus	0.19	<0.3	<0.3
Potassium	0.87	4.2	65
Silicon	21.2	<0.5	<0.5
Sodium	0.18	2.05	11.9
Strontium	0.15	8.63	28.2
Titanium	0.36	<0.02	<0.02
Sulfur	18.40	189	403

TABLE 26

Composite Ash (SNCR) Major/Minor Elements

Element	Bulk, as % oxide	SGLP, 18-hr Avg., mg/L	SGLP, 60-day Avg., mg/L
Aluminum		<0.7	0.86
Calcium		1090	666
Iron		<0.08	<0.08
Magnesium		<0.03	<0.03
Manganese		<0.01	<0.01
Phosphorus		<0.3	<0.3
Potassium		16	184
Silicon		<0.5	<0.5
Sodium		3.86	30.3
Strontium		23.7	47.5
Titanium		<0.02	<0.02
Sulfur		64	0.81

TABLE 27

Coal Major/Minor Elements (in ash)

Element	Bulk, as % oxide	SPLP 1, 18-hr, mg/L	SPLP 2, 18-hr, mg/L
Aluminum	18.8	<0.7	<0.7
Calcium	10.6	13.5	10.6
Iron	5.29	<0.08	<0.08
Magnesium	2.62	3.39	3.26
Manganese	0.19	0.043	0.042
Phosphorus	<0.4	<0.3	<0.3
Potassium	0.98	1.7	1.6
Silicon	49.8	0.63	0.56
Sodium	0.30	6.92	6.71
Strontium	0.22	0.35	0.273
Titanium	0.84	<0.02	<0.02
Sulfur	10.34	23.5	8.80

CFBC By-Product Utilization Potential

The characteristics of FBC by-products depend on the bed material, fuel and ash compositions, unburnt carbon, desulfurization products, and unreacted sorbents. The by-products generated in this study provided several pieces of information that are critical to making preliminary evaluations of the utilization potential on a specific by-product. The environmental evaluation performed answered a key question regarding the decision to use the by-products as part of the ash management plan for the proposed plant. Additionally, the bulk composition of both by-product types was determined, and a limited number of physical characteristics were determined. The following discussion of utilization potential is based on these laboratory results; however, responsible ash management requires a program of quality control/quality assurance for both disposal and utilization purposes. Further, prior to any large-scale utilization of the CFBC by-product to be produced, laboratory- and/or field-scale demonstrations of the selected applications and appropriate performance testing on the demonstrated applications should be performed.

General FBC By-Product Characteristics

Fluidized-bed combustion systems operate at low temperatures, typically less than 900°C, which prevents significant fusion and melting of the ash particles. The FBC fly ash particles are, therefore, angular and very different from the spherical fused ash particles produced in pulverized coal firing (15, 16). Entrained bed material also influences the physical and chemical properties of the fly ash collected in the particulate control devices. The characteristics of the

spent bed material depend on the properties of the coal ash, the bed material, and the sorbent, and the degree of sulfation due to sulfur capture. High-calcium materials used for sulfur capture (i.e., limestone or dolomite) produce by-products containing high levels of calcium sulfate, free lime, and coal ash, which reflect the chemical characteristics of the sorbent and coal used. Selection of coal and sorbent combinations may provide an opportunity to adjust by-product compositions to meet a particular utilization specification. The particle size of the fines collected from an FBC baghouse is similar to that of pulverized coal fly ash. The respective particle sizes of the bed offtake, cyclone, and baghouse by-products have been shown to be similar for eight test coals burned under similar conditions (17). Comparison of atmospheric fluidized-bed combustion (AFBC) and CFBC by-products indicates finer particle-size distributions for the CFBC because of the high degree of solid recycle (18). Potential problems arise in the disposal of FBC by-products due to the high levels of CaO and CaSO₄, the alkalinity of leachate, and dust associated with by-products that contain high levels of CaO. Rapid exothermic reactions and solidification occurring with the addition of water require that care be used in handling, utilizing, and disposing of the FBC residuals (16), and high-calcium and -sulfur materials can provide a source of calcium and sulfate for cementitious reactions in numerous applications where controlled cementation is required. The properties and chemistry exhibited by FBC by-products give them a high utilization potential in many of the new application technologies, including specialty cements, aggregates, lightweight block, brick, waste stabilization, mine reclamation, agriculture, and soil stabilization. Many of these applications take advantage of the unique chemistry and cementitious properties of FBC by-products. FBC by-products, which have been shown to be environmentally acceptable for many use applications and disposal by conventional methods (19–21).

The Environmental Protection Agency has solicited input on the characteristics and management practices for FBC by-products and will begin the review of submitted information in October, 1997. Following this review, EPA will make a determination of the RCRA status of FBC by-products. Pure streams of conventional CCBs (fly ash, bottom ash, boiler slag, and FGD material) have already been placed under Subtitle D of RCRA, designating them as solid wastes and therefore under the jurisdiction of individual states. This determination provides added opportunity for these types of materials to be utilized. Several states have encouraged utilization by exempting utilized by-products from solid waste rules (22). As the determination is under consideration for FBC by-products, it is important for industry to work with both EPA and state regulatory agencies to facilitate development of rules that will allow the environmentally sound utilization of FBC by-products.

General FBC By-Product Utilization

FBC by-products have been used for a variety of applications (see Figure 15). Although Figure 15 refers to AFBC by-products, it can be assumed that CFBC by-products can be used in many of these applications because the chemical composition, mineralogy, and physical properties are similar. FBC by-products have often been used to make cements, mortars, and concretes, but are very slow to develop good strength (23, 24). As controlled, low-strength materials, FBC by-products have several useful construction applications because of self-leveling properties (25). Roadbed stabilization tests have had positive results, but there is concern over the long-term durability and the performance under freeze–thaw conditions (25). Cementitious

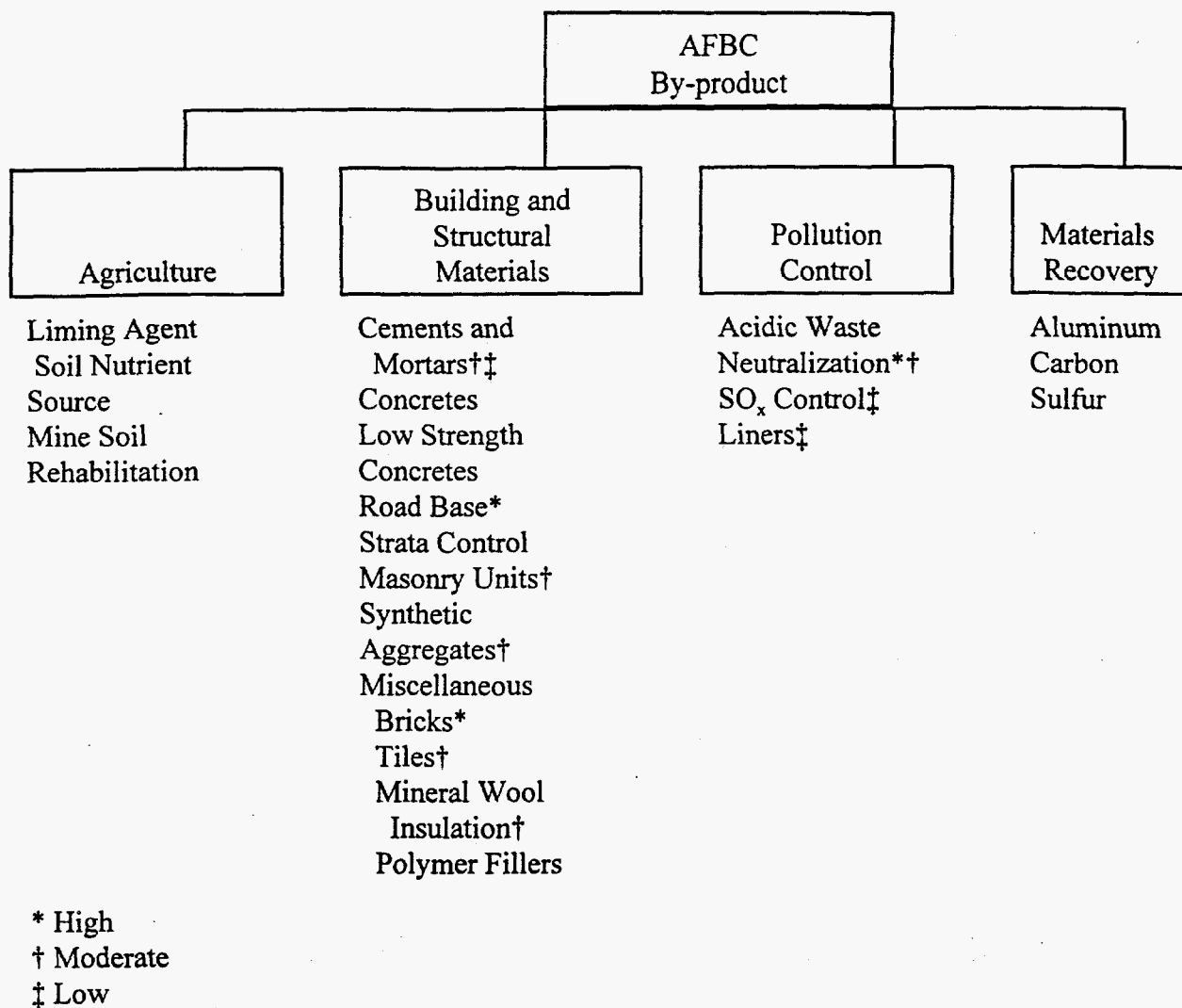


Figure 15. Potential utilization options for AFBC by-products (14).

properties, free lime, and absorbing capacities make FBC by-products useful for the treatment of acidic wastes and sludges, as well as for control of acid mine drainage (27, 28). FBC by-products have been utilized in a variety of small-scale applications (16). The major concern with the utilization of FBC by-products is the compositional variability found in the by-products themselves. Agricultural use as a liming agent causes concern over the possible release of harmful trace elements. Any future improvements in sulfur capture will also reduce the amount of free lime (CaO) that would be available to the soil. This would reduce the desirability of the by-product as a liming agent for agricultural purposes (29-31).

In the realm of construction materials, it has become increasingly clear that no single type of cementing system can provide for all the technical and cost requirements of modern cement and concrete products. For FBC by-product utilization in this growing marketplace, it is necessary to demonstrate by-product attributes and limitations and the mechanics by which by-products function. In addition, these products will need to be evaluated for potential durability

under various typical environments and for the extent to which by-product properties might be modified to broaden the range of applications or to increase durability.

Uses of pulverized coal ashes frequently exploit the pozzolanic activity, a term used to distinguish long-term strength development. Some confusion exists as to whether FBC by-products are pozzolanic or cementitious. Recent studies (24) suggest that FBC by-product compositions (which contain unreacted lime and calcium sulphate, both of which exhibit exothermic hydration reactions and are potential sources of expansion in cement systems) can provide the lime and sulfate components necessary to induce "sulfopozzolanic" reactivity in a fly ash or natural pozzolan rather than showing pozzolanic action. Recognizing the limitations of the materials, a novel approach to the use of FBC by-products involves the production of what is termed "no-cement" concretes from co-utilization of FBC by-products in combination with conventional pulverized coal fly ash. Three main requirements need to be met for FBC by-product utilization: that the by-product contain at least some CaO, that it have a granular texture, and that it be somewhat cementitious.

Some potential utilization options that have been identified for FBC by-products are described briefly below:

- Controlled low-strength material (CLSM)

CLSM is mixed to a wet, flowable slurry used as an economical fill or backfill material placed by pouring into a desired cavity. Typically, it is designed as a low-strength material, but with small mixture modifications, much higher strengths are attainable. Applications include backfill (sewer conduit and utility trenches, building excavations, and bridge abutments), structural fill (road and floor slab base, subfooting, and pipe bending), underground storage tanks, slope stabilization, soil erosion control, and abandoned sewers. Examined parameters include compressive strength, density, and flow characteristics.

- Brick

Both fly ash and bottom ash are used to manufacture building brick. The required mechanical and physical properties that would need to be determined include compressive strength, flexural strength, unit weight, and water absorption characteristics. The required specifications for masonry brick are listed in ASTM C62.

- Soil stabilization

The high pH and chemistry/mineralogy of FBC by-products are generally beneficial for stabilization and applications. These applications include road base stabilization and other soil stabilization. Mine land reclamation is a distinct soil stabilization application with particularly high potential in areas where acid mine drainage is an environmental issue.

- Structural fill

FBC by-products can be substituted for soil borrow to construct embankments, dikes, or as general site fills. Structural fills utilize high volumes of materials in many regions of the United States annually. ASTM has approved a standard practice to determine the applicability of the use of CCBs, including FBC by-products for structural fills.

- Waste stabilization

FBC by-products may be used to neutralize acid waste such as acid mine drainage (AMD) or to facilitate the solidification and/or chemical stabilization of a variety of wastes, including those containing organic and inorganic contaminants. The water-absorbing capacity of these by-products is also advantageous in waste stabilization applications.

- Production of expansive cement and other specialty cements

Expansive cements are designed to expand for grouting in mining and drilling applications. The expansive nature of this type of product presents other use opportunities in minefilling and other applications requiring low strength. Cements with unique or specific chemical compositions are of scientific interest and have commercial potential for the future. These will be investigated on a limited basis.

- Agricultural applications

FBC bed by-products have been used at high rates of over 100 tons per acre as a mulching agent applied directly to cap the soil surface in orchards and raised-bed tomato rows (31). An extensive study on the land application of AFBC and PFBC by-products was performed in Ohio (21). Selected by-products, alone or in combination with sewage sludge, were mixed with acid soils and mine spoils and tested in greenhouse growth studies. Interactions of different materials gave somewhat different results. For example, growth of tall fescue was enhanced in overburden spoil, but was suppressed in acid underclay. Sulfite-bearing material did not harm seed germination. The conclusion reached from the greenhouse tests was that the by-products tested, when used appropriately, are suitable substitutes for traditional soil-liming materials for acid soils. Field tests supported these findings.

- Mining and mine land reclamation applications

Mine applications have previously been considered disposal but, due to the relatively benign nature of coal by-products, should more appropriately be considered reuse for reclamation of mined land because of the benefits derived in these applications. By-products from the combustion of low-rank coals which generates leachate at extremely high pH tend to form the mineral ettringite. The alkaline nature of some coal by-products (including duct injection by-products/FBC by-products, and low-rank coal fly ash) can be capitalized on for abatement of AMD and spoils (32, 33, 34). Return of ash to the mined settings is a sound high-volume use of this versatile engineering material. Not only can land be reclaimed, but, in the case of underground mines, the setting can be stabilized to prevent future subsidence. Treatment of and spoils has high

potential, especially for high-volume, alkaline by-products from advanced coal processes. Impacts from trace elements, the primary concern, have been minimal or unmeasurable in almost all instances where monitoring has been carried out. There have been examples where groundwater quality has been shown to actually improve from the placement of coal by-products in the environment (34, 35).

These applications have high potential for CFBC by-products because of the alkaline nature of the by-product, the physical properties of the material, and the generally low concentrations of potentially hazardous constituents. Any commercial utilization of a by-product in a utilization application should be demonstrated and tested on the laboratory- and/or demonstration-scale first.

Discussion of Utilization Potential for Bench-Scale CFBC By-Products

The by-products generated in this study were characterized with the primary objective of evaluating the potential for environmental impact, and the results indicated that these by-products exhibit low concentrations of any potentially hazardous constituents. The results further indicated that the mobility of any constituents is extremely limited. For all practical purposes, these by-products were found to be environmentally benign. The environmental assessment is the critically important first step to determining the utilization potential of any industrial by-product, and the results of that assessment are favorable for selecting utilization as a viable management option. EPA has not yet made a final determination on the status of FBC by-products under RCRA. These by-products currently are exempt from RCRA Subtitle C (hazardous solid waste), so individual states are responsible for rules on the disposition of these materials. Several approaches to regulating utilization have been reported throughout the United States. In Mississippi, CCBs are regulated as solid wastes, and there are no specific rules addressing use. Requests for use are addressed on a case-by-case basis (22). The Mississippi state solid waste regulations provide an exemption from the definition of solid waste for by-products used in concrete, concrete blocks, and similar applications. Part of every assessment of utilization potential should include an evaluation of appropriate regulations at state and local levels.

The bulk chemical composition (major constituents) indicate a strong possibility that both the fly ash and bed materials for both non-SNCR and SNCR would exhibit some cementitious reactions on hydration. In fact, the non-SNCR fly ash nearly meets the chemical requirements for a Class C fly ash under ASTM C 618, Standard Specification for Coal fly Ash and Raw or Calcined Natural Pozzolan for Use as a Mineral Admixture in Concrete, failing only by exceeding the limit of 5% SO₃ by less than 0.5%. The ASTM C618 chemical specifications are noted in Table 28. Physical and engineering tests prescribed in ASTM C311, Standard Test Methods for Sampling and Testing Fly Ash or Natural Pozzolans for Use as a Mineral Admixture in Portland-Cement Concrete, should be performed to provide a better indication of the usefulness of this material, but preliminary data suggest that the non-SNCR fly ash might be used successfully as an admixture in concrete. More important though, these results indicate that the material might be best suited for applications where cementitious reactions are desirable but the ASTM C618 requirements need not be strictly adhered to, such as CLSM, soil and waste

TABLE 28

ASTM C618 Specifications

Chemical Composition (%)	Class F	Class C
Silicon Dioxide (SiO ₂)		
Aluminium Oxide (Al ₂ O ₃)		
Iron Oxide (Fe ₂ O ₃)		
Total (SiO ₂ + Al ₂ O ₃ + Fe ₂ O ₃)	70.0 Min	50.0 Min
Sulfur Trioxide (SO ₃)	5.0 Max	5.0 Max
Calcium Oxide (CaO)		
Moisture Content	3.0 Max	3.0 Max
Loss on Ignition	6.0 Max	6.0 Max
Available Alkalies as Na ₂ O	1.5 Max	1.5 Max

stabilization, aggregate, masonry products, and brick. The non-SNCR bed material and the SNCR fly ash and bed material have similar bulk composition and similar potential, except that they do not as closely approximate the composition of a Class C fly ash. It is therefore more difficult to make a preliminary judgement on their usefulness in these applications. Additional testing would provide the information to pinpoint the best potential use applications. The coarseness of the bed materials is greater than the fly ash, as expected, and it is anticipated the reactivity of the bed materials would be lower than the fly ash as a result of the difference in particle size. Generally speaking, finer grain size can be related to increased reactivity in conventional CCBs, and the authors anticipate a similar trend with CFBC by-products. Based on the summary of Mississippi solid waste exemptions for by-products, it is logical that any utilization that takes advantage of the cementitious nature of the by-product would be allowed.

The analytical data indicated that the by-products contain calcium and sulfate and trace element constituents that exhibit extremely limited mobility. These characteristics would mean a recommendation that all by-products be evaluated further for use in agricultural applications. The chemical and physical characteristics indicate that these materials could provide advantages in improving soil tilth, availability of calcium, and neutralization of acid soils.

The same characteristics that give these CFBC by-products high potential for use in agricultural applications also make them desirable for mine reclamation projects. Since returning these by-products to the mine site has already been identified as a potential by-product management strategy, it will be beneficial to the mine placement process to understand the neutralization capacity of the by-products, the potential for cementitious reactions to occur, and the density and permeability of the materials. These parameters and other physical tests should be monitored at the power plant to facilitate effective mine reclamation procedures. There are generally state regulations for mine reclamation activities.

Summary of Utilization Potential for Bench-Scale CFBC By-Products

The CFBC by-products generated during bench-scale combustion tests indicated good utilization potential. The following utilization applications were identified as applications that warrant further evaluation from a technical perspective:

- Nonconcrete cementitious applications such as CLSM, soil and waste stabilization, aggregate, masonry products, and brick
- Agricultural applications
- Mine reclamation applications

Technical evaluations should include physical and engineering material testing and laboratory- and field-scale demonstration and performance testing. It is important to note that demonstration and performance testing may require some long-term procedures. It is important to work with local and state regulatory agencies in identifying the utilization applications and the requirements for testing and demonstration that they may require. In addition to the technical assessment and working with regulators to evaluate these applications, a market assessment is recommended to provide the best fit to local and regional needs and facilitate an economic evaluation of the various applications. In many cases, power plants work jointly with marketers to perform these evaluations and to market the materials.

The critical point that can be made based on the environmental data presented on the bench-scale CFBC by-products is that these materials are environmentally benign, and it is recommended that a power plant by-product management strategy include the appropriate evaluations to include materials utilization as a management option.

CONCLUSIONS AND RECOMMENDATIONS

Because of the low concentrations of potentially problematic trace elements found in these ash leachate samples, it is highly unlikely that this ash could present any adverse risk or threat to the environment. Concentrations in leachate were for the most part low enough that interpretation concerning LTL trends were difficult to make. Most LTL results were in a low enough range that expected experimental error was greater than some of the measurements, many of which were just above LLQs. The following can be stated:

- In spite of a relatively high moisture content, the fuel fed very well, maintaining a very uniform temperature distribution throughout the reactor. While the calcium-to-sulfur ratio was higher than expected for Test 1, optimization of the operating temperature could decrease limestone requirements.
- The NO_x emissions generated during the combustion tests are higher than would be generated at full scale. Wall effects are the absence of secondary air in the bench-scale reactor resulting in higher NO_x; testing at a larger scale may be necessary to accurately predict full-scale NO_x.

- The results of the SNCR test suggest that this method of NO_x reduction may be inappropriate for this fuel under CFB conditions. The higher temperatures required for effective NO_x reduction have a dramatic negative impact on sulfur retention, so that optimizing one parameter will seriously compromise the other. Because of the strong effect of temperature on sulfur capture for this fuel, operating at a "middle ground" condition may result in excessive levels of both NO_x and SO₂. SNCR could be effective if some type of reburn were used to raise flue gas temperatures prior to SNCR, while maintaining the furnace at a lower temperature to optimize sulfur capture. Other methods of NO_x reduction, including air staging and optimization of Ca:S and excess air, may provide acceptable emission levels.
- RCRA standards were not exceeded in any of the leachates. Concentrations of RCRA constituents were nearly all below drinking water standards.
- Leachate solution concentrations of major and minor constituents are very low, except for calcium and sulfate. Since calcium and sulfate are present in relatively high concentrations, with sulfate concentrated on the surface of spent bed material as well as ash particles, it would be expected that the solubility of calcium sulfate, which is relatively high, would be a major factor in determining the chemical composition of the leachate.
- While ettringite was a primary hydration product in all of the leached materials, the low concentrations of oxyanionic trace elements, along with the lack of significant documentable trends in leachate concentration evolution, made it difficult to determine exactly what role ettringite formation played in trace element leaching. It is known that ettringite formation can result in what is referred to as anomalous leaching, resulting in a decrease in leachate concentration of select trace elements with respect to time. For this to be documented, however, it is necessary to have significant concentrations of the trace elements, which were simply not present in these samples.
- Overall, the large number of extremely low or less-than values further attest to the benign nature of these as well as most other CCBs.

Interaction of leachates with the local geologic material was an aspect of environmental impact that was not a part of this project, but must be considered in the interpretation of these data. After leachate migrates from a disposal site, varying factors will continue to influence the concentrations of leached materials. Under normal geological and hydrogeological conditions, dispersion, diffusion, and dilution with groundwater will all lead to lower constituent concentrations. Additionally, many geologic materials have attenuating properties that can further reduce the concentrations of trace constituents. Cation and anion exchange as well as precipitation and sorption effects will likely even further reduce constituent concentrations in leachate.

ACKNOWLEDGMENTS

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APPENDIX A
PIXE RESULTS

Samples in this section are identified by laboratory numbers. For reference, the samples are as follows:

1. 49441 Coal
2. 49442 Limestone
3. 49443 Non-SNCR Composite Ash
4. 49444 Non-SNCR Bed Material
3. 49445 SNCR Composite Ash
4. 49446 SNCR Bed Material

Name: Energy & Environmental Research Center

Project: Solids/Charlene Crocker

Date: 06-19-1997

Job: 1980-97

Dear Client;

We would like to thank you for using our analytical services, and hope that we have been able to assist you in meeting your requirements for element analysis. We take a great deal of pride in trying to provide the best possible service to our clients at the lowest possible costs. If you are a new client and have not used our services before we have prepared a brief explanation of this report which should be useful as an aid in evaluating your results. If, after reading this explanation, you should have additional questions or require further clarification, please don't hesitate to contact us.

This report is composed of multiple sections, each of which describes some aspect of your data which we feel is important to your understanding of the results. Briefly stated, pages 1 and 2 that follow are a tabular listing of all the elements that we are capable of analyzing. Page 3 is a graphical representation of the elements found to be present in your sample and their relative concentrations. Finally, the last page is a plot of the actual x-ray spectrum that your sample generated. Atop of each page of this report is the client header information that tracked the analysis of this/these samples. The client provided information, such as client name, project, and sample label track the sample from time of login to time of shipment. This is also true of job number, which is our internal identifier, which tracks your job. The analysis date, and the archive code are provided at the time of irradiation and allow us to reference your data for each sample back to you as the client. Always check this information for correctness and immediately report to us any discrepancies that you might find. By doing this, you will be helping us to provide better service to you, our clients.

Pages 1 and 2 contain analysis results listed in the following six (6) columns:

- **COLUMN 1** shows the **ELEMENT SYMBOL** or **NAME**. If the name appears, the element is present. If the symbol appears, the element is not present at the specified detection limit (Column 3).

If elements below Sodium, (C,H,N,O,etc) appear in the report, they represent a synthetic component to the analysis. The amounts listed for these elements are either based upon stoichiometric calculations or are present to normalize the sample to 100% mass. This procedure is sometimes necessary, to account for scattering of the impinging proton and absorption of the emitted X-Rays when thick or intermediate target analysis is performed.

- **COLUMN 2** shows the energy of the **MAJOR X-RAY** line (KEV) used for quantification of that element.
- **COLUMN 3** provides the **DETECTION LIMIT** achieved expressed as a weight fraction of the sample.

Detection limits are inversely proportional to the square root of the irradiation time. In order to achieve lower detection limits, a longer analysis is required. For pricing information concerning extended irradiation procedures, please call or write.

- **COLUMN 4** is the **WEIGHT FRACTION** of the element present in the sample expressed as either as % or ppm. One ppm is equal to 0.0001% or 10,000 ppm equal 1%.

Sometimes you will find an amount that is below the calculated detection limit. This is

because the detection limit is conservatively calculated at 95% confidence, but we allow concentrations to be determined as low as 65% confidence (1 sigma). It is at your discretion to evaluate the validity of such values as they may be the result of statistical noise.

- COLUMN 5 lists the ERROR (listed as the uncertainty in the element mass fraction) has been converted from a fractional error. For example, a 10% error in a concentration of 100 ppm would be listed as 10 ppm in the error column.
- COLUMN 6, when appropriate, is used to list the calculated OXIDE concentrations for the elements present. These calculations are done for the major and minor elements assuming the most common valence states.

Page 3 contains the following information:


On this page is a graphical representation of the concentration of the elements determined by the analysis are shown in a bar graph format. The data is graphed only for those elements that are determined to be present in the sample. The data will be graphed either as a linear or square root plot depending on what best represents the data. In addition, the data may either be internally normalized and therefore self-scaling, or scaled to a fixed value if requested by the client. In either case, the options are presented in an information box located at the top right hand corner of the page.

Page 4 contains the following information:

Page 4 is a display of the actual X-Ray spectrum of your sample. The x-axis is in units of energy (KEV), while the y-axis represents intensity or counts for each channel of the spectrum.

After reviewing your results, if you should have further questions, please do not hesitate to contact us. We look forward to continuing to work with you in the future and, again, would like to thank you for choosing PAL.

Results verified by:



Sene Bauman Ph.D.
Chief Scientist

Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49441

Analysis Date: 06-19-1997
Archive Code: 361 361
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	
* H	-----		1.000 % ±		
* C	-----		89.976 % ±	0.027 %	
Na	1.041	937.000 ppm			
Mg	1.254	448.400 ppm			
Aluminum	1.487	258.400 ppm	1.935 % ±	0.131 %	3.505 %
Silicon	1.740	152.600 ppm	3.796 % ±	0.250 %	1.049 %
P	2.014	139.600 ppm			
Sulfur	2.308	70.390 ppm	0.695 % ±	0.046 %	2.399 %
Cl	2.622	99.950 ppm			
Potassium	3.314	30.730 ppm	0.152 % ±	0.010 %	9.577 %
Calcium	3.692	26.120 ppm	1.671 % ±	0.110 %	1.741 %
Sc	4.091	47.130 ppm			
Titanium	4.511	8.853 ppm	0.117 % ±	0.008 %	2.278 %
Vanadium	4.952	11.010 ppm	53.103 ppm ±	6.282 ppm	1.319 %
Chromium	5.415	5.068 ppm	12.908 ppm ±	2.548 ppm	4.021 %
Manganese	5.899	3.887 ppm	274.702 ppm ±	18.43 ppm	9.892 %
Iron	6.399	4.605 ppm	0.579 % ±	0.038 %	2.409 %
Co	6.925	15.710 ppm			
Nickel	7.472	3.359 ppm	8.935 ppm ±	1.211 ppm	4.387 %
Copper	8.041	1.822 ppm	36.565 ppm ±	2.757 ppm	1.771 %
Zinc	8.631	1.818 ppm	17.226 ppm ±	1.531 ppm	8.412 %
Gallium	9.243	1.726 ppm	6.318 ppm ±	0.907 ppm	2.915 %
Ge	9.876	1.967 ppm			
As	10.532	2.011 ppm			
Selenium	11.208	1.752 ppm	6.760 ppm ±	0.972 ppm	2.376 %
Bromine	11.924	2.582 ppm	3.242 ppm ±	0.830 ppm	6.677 %
Rb	13.395	4.722 ppm			
Strontium	14.165	4.723 ppm	320.444 ppm ±	21.63 ppm	3.816 %
Yttrium	14.958	4.760 ppm	8.888 ppm ±	1.701 ppm	8.648 %
Zirconium	15.775	14.870 ppm	23.775 ppm ±	4.142 ppm	1.848 %
Nb	16.615	7.207 ppm			
Mo	17.479	8.762 ppm			
Tc	18.367	9.325 ppm			
Ru	19.279	10.850 ppm			
Rh	20.216	13.440 ppm			
Pd	21.177	13.360 ppm			
Ag	22.163	17.190 ppm			
Cd	23.174	21.130 ppm			
In	24.210	24.000 ppm			
Sn	25.271	29.760 ppm			
Sb	26.359	42.930 ppm			
Te	3.768	363.300 ppm			
I	3.936	209.200 ppm			
Cs	4.285	78.840 ppm			
Ba	4.465	81.210 ppm			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49441

Analysis Date: 06-19-1997
Archive Code: 361 361
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error
La	4.649	68.780 ppm		
Ce	4.838	41.610 ppm		
Pr	5.032	34.090 ppm		
Nd	5.228	21.250 ppm		
Pm	5.430	17.140 ppm		
Sm	5.632	22.260 ppm		
Eu	5.842	31.310 ppm		
Gd	6.054	43.720 ppm		
Tb	6.270	117.800 ppm		
Dy	6.492	114.300 ppm		
Ho	6.716	53.690 ppm		
Er	6.944	49.480 ppm		
Tm	7.180	51.920 ppm		
Yb	7.416	18.480 ppm		
Lu	7.655	7.270 ppm		
Hf	7.899	9.523 ppm		
Ta	8.146	9.980 ppm		
W	8.398	6.965 ppm		
Re	8.653	7.873 ppm		
Os	8.912	7.289 ppm		
Ir	9.175	6.313 ppm		
Pt	9.442	6.141 ppm		
Au	9.713	5.873 ppm		
Hg	9.989	5.865 ppm		
Tl	10.269	6.043 ppm		
Lead	10.552	5.593 ppm	13.288 ppm ±	2.644 ppm 1.669 %
Bi	10.839	5.691 ppm		
Th	12.969	8.291 ppm		
U	13.615	11.070 ppm		

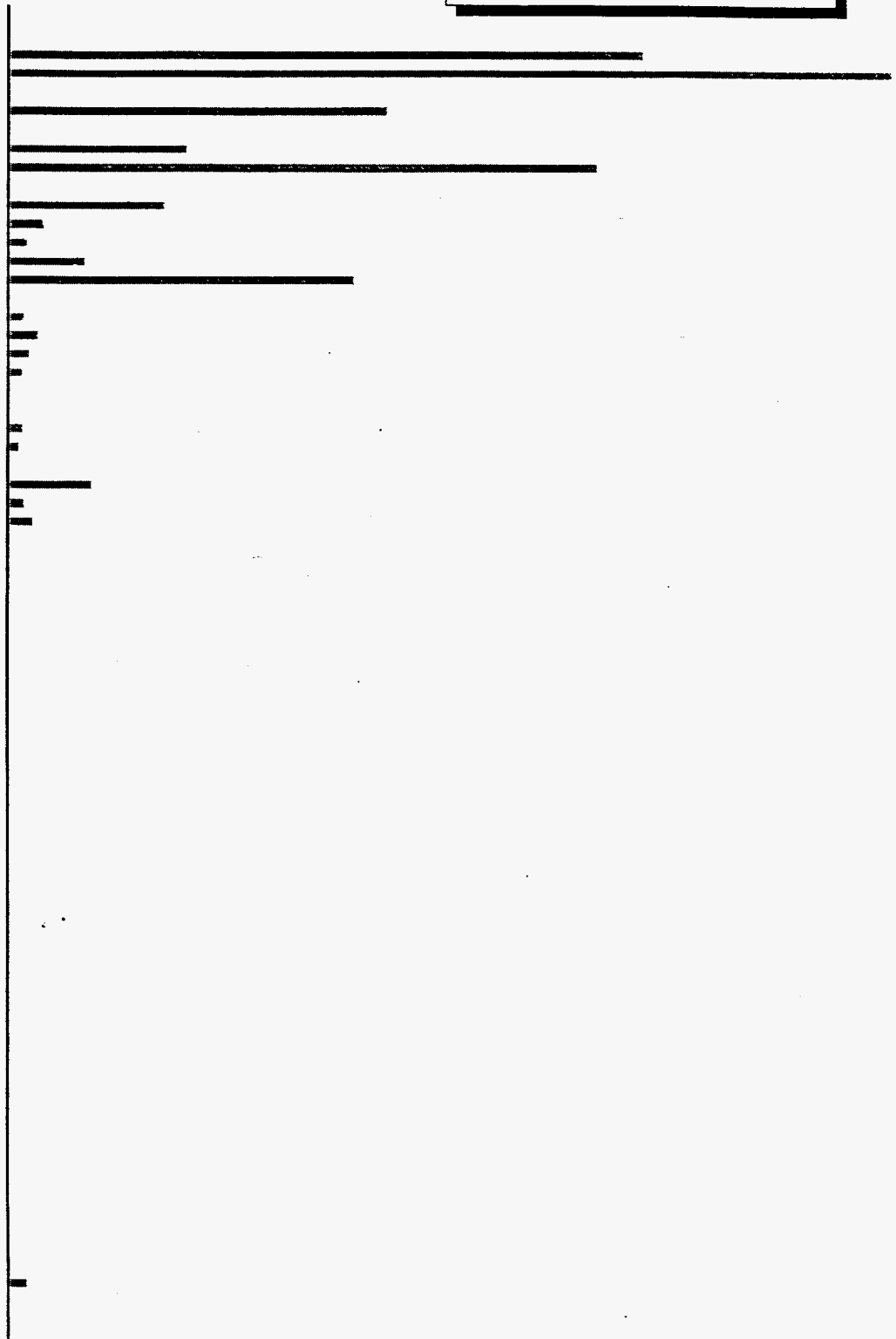
Name: Energy & Environmental Research Center
 Project: Solids/Charlene Crocker
 Target: 49441

Archive Code: 361 361
 Analysis Date: 06-19-1997
 Job: 1980-97

Concentration Graph

Scaling: Square Root
 Normalization: Internally Self-Scaling

Aluminum	1.9349 % ±	0.131
Silicon	3.7959 % ±	0.250
Sulfur	0.6952 % ±	0.046
Potassium	0.1522 % ±	0.010
Calcium	1.6710 % ±	0.110
Titanium	0.1170 % ±	0.008
Vanadium	53.1033 ppm ±	6.282
Chromium	12.9078 ppm ±	2.548
Manganese	274.702 ppm ±	18.433
Iron	0.5795 % ±	0.038
Nickel	8.9350 ppm ±	1.211
Copper	36.5649 ppm ±	2.757
Zinc	17.2258 ppm ±	1.531
Gallium	6.3177 ppm ±	0.907
Selenium	6.7598 ppm ±	0.972
Bromine	3.2415 ppm ±	0.830
Strontium	320.444 ppm ±	21.630
Yttrium	8.8877 ppm ±	1.701
Zirconium	23.7752 ppm ±	4.142



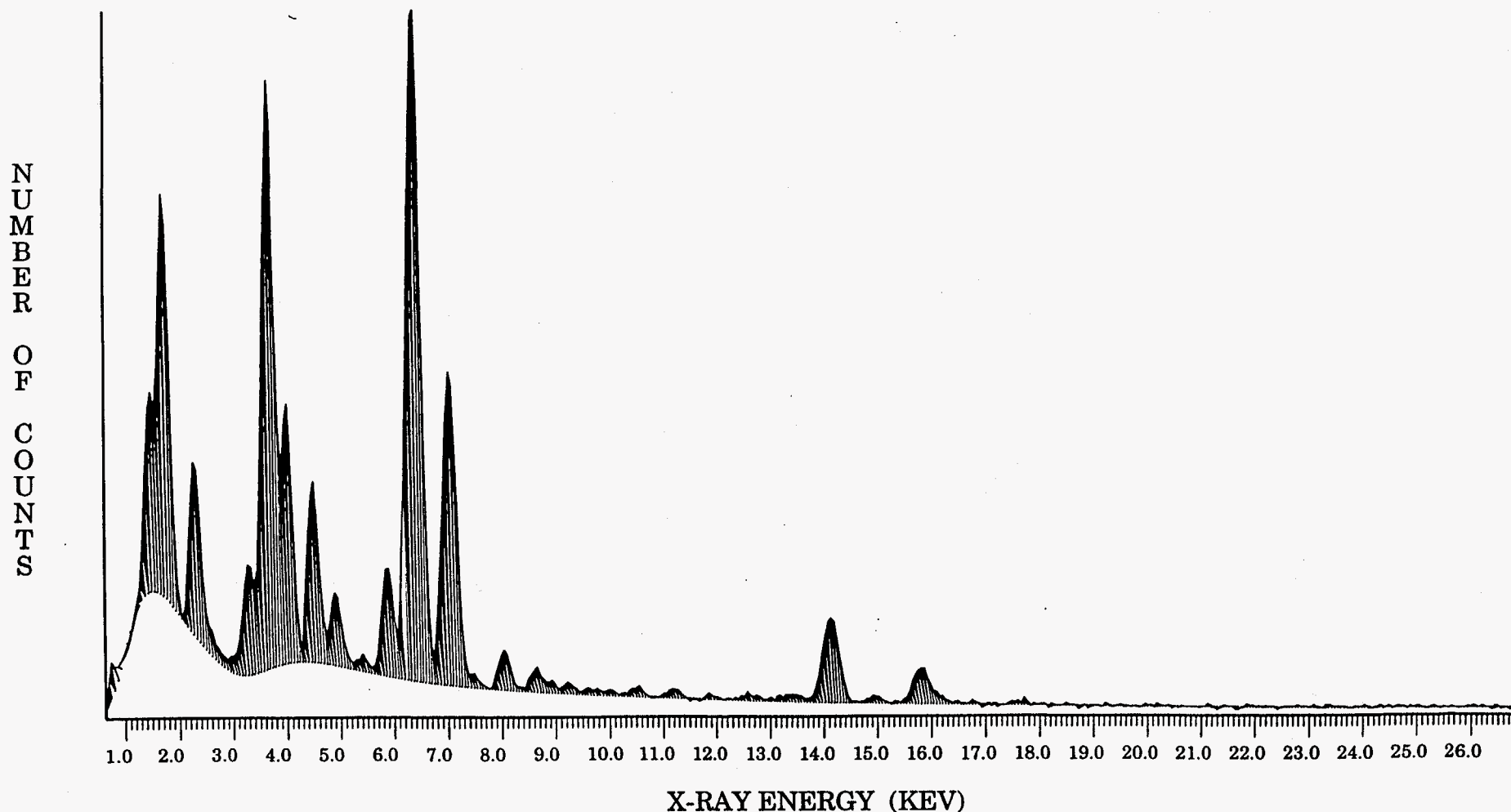
Lead 13.2875 ppm ± 2.644

Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49441

Analysis Date: 06-19-1997
Archive Code: 361 361
Job: 1980-97

Fit Analysis

Total Error(%)= .631
Sum Of Neg Error(%)= -1.961 Points= 425
Sum Of Pos Error(%)= 2.592 Points= 599



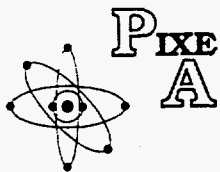
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49442

Analysis Date: 06-19-1997
Archive Code: 362 362
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
* C	-----		9.839 % ±		
* O	-----		48.056 % ±	0.082 %	
Na	1.041	0.365 %			
Mg	1.254	0.139 %			
Aluminum	1.487	796.200 ppm	2.231 % ±	0.172 %	4.216 %
Silicon	1.740	457.500 ppm	4.921 % ±	0.329 %	10.528 %
Phosphorus	2.014	406.100 ppm	0.118 % ±	0.022 %	0.271 %
Sulfur	2.308	195.000 ppm	0.233 % ±	0.020 %	0.584 %
Chlorine	2.622	204.800 ppm	676.621 ppm ±	113.4 ppm	
Potassium	3.314	365.200 ppm	0.437 % ±	0.032 %	0.527 %
Calcium	3.692	222.100 ppm	32.795 % ±	2.151 %	45.887 %
Sc	4.091	964.500 ppm			
Titanium	4.511	62.330 ppm	0.122 % ±	0.010 %	0.204 %
V	4.952	55.770 ppm			
Chromium	5.415	33.410 ppm	51.727 ppm ±	14.46 ppm	0.008 %
Manganese	5.899	26.040 ppm	316.577 ppm ±	26.34 ppm	0.048 %
Iron	6.399	20.590 ppm	1.018 % ±	0.067 %	1.456 %
Co	6.925	73.600 ppm			
Nickel	7.472	18.880 ppm	51.668 ppm ±	7.358 ppm	0.007 %
Cu	8.041	11.660 ppm			
Zinc	8.631	10.240 ppm	32.211 ppm ±	5.263 ppm	0.004 %
Ga	9.243	9.408 ppm			
Ge	9.876	9.517 ppm			
As	10.532	9.065 ppm			
Se	11.208	7.322 ppm			
Br	11.924	11.720 ppm			
Rb	13.395	23.610 ppm			
Strontium	14.165	18.310 ppm	0.102 % ±	0.007 %	0.120 %
Yttrium	14.958	18.480 ppm	19.575 ppm ±	6.554 ppm	0.002 %
Zr	15.775	82.170 ppm			
Nb	16.615	24.510 ppm			
Mo	17.479	32.140 ppm			
Tc	18.367	33.560 ppm			
Ru	19.279	42.430 ppm			
Rh	20.216	47.940 ppm			
Pd	21.177	55.380 ppm			
Ag	22.163	62.070 ppm			
Cd	23.174	106.500 ppm			
In	24.210	87.770 ppm			
Sn	25.271	124.700 ppm			
Sb	26.359	223.200 ppm			
Te	3.768	0.491 %			
I	3.936	0.288 %			
Cs	4.285	0.123 %			
Ba	4.465	364.000 ppm			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49442

Analysis Date: 06-19-1997
Archive Code: 362 362
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
La	4.649	308.400 ppm			
Ce	4.838	204.400 ppm			
Pr	5.032	171.100 ppm			
Nd	5.228	133.200 ppm			
Pm	5.430	116.500 ppm			
Sm	5.632	121.300 ppm			
Eu	5.842	138.700 ppm			
Gd	6.054	204.800 ppm			
Tb	6.270	554.300 ppm			
Dy	6.492	525.800 ppm			
Ho	6.716	242.900 ppm			
Er	6.944	231.200 ppm			
Tm	7.180	239.100 ppm			
Yb	7.416	95.820 ppm			
Lu	7.655	50.310 ppm			
Hf	7.899	39.140 ppm			
Ta	8.146	34.740 ppm			
W	8.398	34.510 ppm			
Re	8.653	39.930 ppm			
Os	8.912	34.340 ppm			
Ir	9.175	29.620 ppm			
Pt	9.442	30.120 ppm			
Au	9.713	29.610 ppm			
Hg	9.989	29.150 ppm			
Tl	10.269	32.270 ppm			
Pb	10.552	27.510 ppm			
Bi	10.839	22.400 ppm			
Th	12.969	34.520 ppm			
U	13.615	57.310 ppm			

Name: Energy & Environmental Research Center
 Project: Solids/Charlene Crocker
 Target: 49442

Archive Code: 362 362
 Analysis Date: 06-19-1997
 Job: 1980-97

Concentration Graph

Scaling: Square Root
 Normalization: Internally Self-Scaling

Aluminum	2.2314 % ±	0.172
Silicon	4.9213 % ±	0.329
Phosphorus	0.1185 % ±	0.022
Sulfur	0.2335 % ±	0.020
Chlorine	676.621 ppm ±	113.402
Potassium	0.4374 % ±	0.032
Calcium	32.7954 % ±	2.151
Titanium	0.1224 % ±	0.010
Chromium	51.7267 ppm ±	14.463
Manganese	316.577 ppm ±	26.339
Iron	1.0184 % ±	0.067
Nickel	51.6679 ppm ±	7.358
Zinc	32.2111 ppm ±	5.263
Strontium	0.1017 % ±	0.007
Yttrium	19.5749 ppm ±	6.554

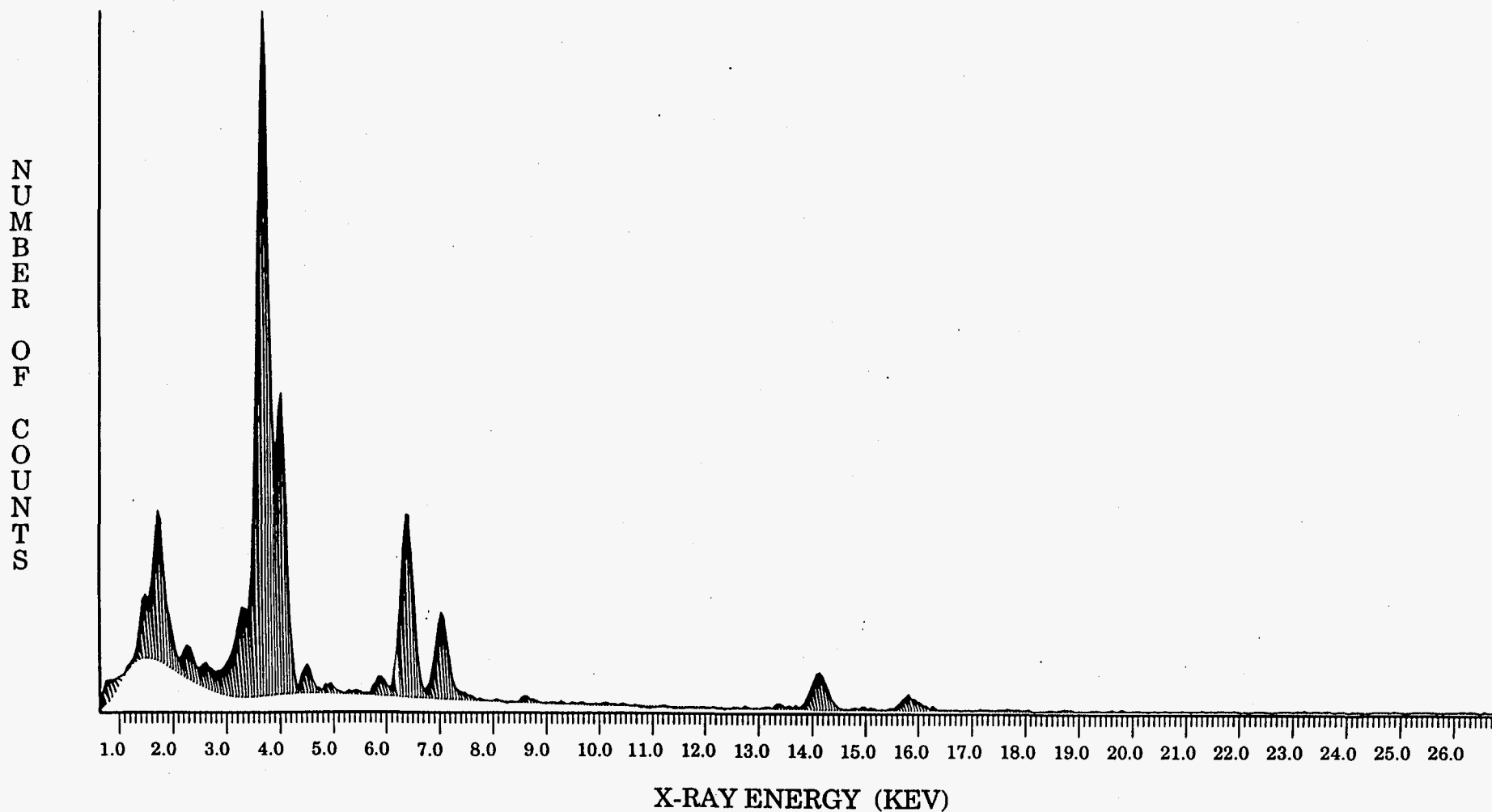


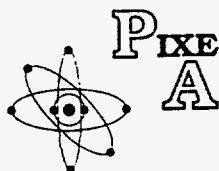
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49442

Analysis Date: 06-19-1997
Archive Code: 362 362
Job: 1980-97

Fit Analysis

Total Error(%)= .497
Sum Of Neg Error(%)= -1.383 Points= 433
Sum Of Pos Error(%)= 1.880 Points= 591





Analytical

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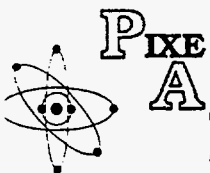
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49443

Analysis Date: 06-19-1997
Archive Code: 363 363
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
* O	-----		43.482 % ±	0.113 %	
Na	1.041	0.385 %			
Mg	1.254	0.197 %			
Aluminum	1.487	0.104 %	9.427 % ±	0.633 %	17.812 %
Silicon	1.740	695.700 ppm	18.129 % ±	1.195 %	38.783 %
P	2.014	738.700 ppm			
Sulfur	2.308	311.600 ppm	2.505 % ±	0.168 %	6.263 %
Cl	2.622	369.000 ppm			
Potassium	3.314	308.100 ppm	0.650 % ±	0.045 %	0.783 %
Calcium	3.692	187.900 ppm	21.629 % ±	1.419 %	30.263 %
Sc	4.091	587.400 ppm			
Titanium	4.511	66.530 ppm	0.675 % ±	0.045 %	1.126 %
Vanadium	4.952	78.350 ppm	310.854 ppm ±	42.56 ppm	0.055 %
Chromium	5.415	37.170 ppm	235.671 ppm ±	24.56 ppm	0.034 %
Manganese	5.899	29.040 ppm	0.158 % ±	0.011 %	0.242 %
Iron	6.399	33.380 ppm	2.998 % ±	0.197 %	4.287 %
Co	6.925	95.530 ppm			
Nickel	7.472	21.270 ppm	78.041 ppm ±	8.920 ppm	0.010 %
Copper	8.041	12.510 ppm	180.890 ppm ±	14.36 ppm	0.023 %
Zinc	8.631	11.350 ppm	60.924 ppm ±	6.903 ppm	0.008 %
Gallium	9.243	10.770 ppm	28.518 ppm ±	4.959 ppm	0.004 %
Ge	9.876	11.340 ppm			
Arsenic	10.532	10.810 ppm	12.813 ppm ±	5.776 ppm	0.002 %
Selenium	11.208	9.161 ppm	23.991 ppm ±	4.611 ppm	0.003 %
Br	11.924	13.870 ppm			
Rb	13.395	23.740 ppm			
Strontium	14.165	22.460 ppm	0.224 % ±	0.015 %	0.265 %
Yttrium	14.958	19.840 ppm	73.125 ppm ±	10.01 ppm	0.009 %
Zirconium	15.775	96.630 ppm	138.690 ppm ±	23.74 ppm	0.019 %
Nb	16.615	34.330 ppm			
Mo	17.479	36.360 ppm			
Tc	18.367	32.340 ppm			
Ru	19.279	42.330 ppm			
Rh	20.216	45.560 ppm			
Pd	21.177	49.020 ppm			
Ag	22.163	51.790 ppm			
Cd	23.174	72.590 ppm			
In	24.210	82.780 ppm			
Sn	25.271	106.500 ppm			
Sb	26.359	155.600 ppm			
Te	3.768	0.350 %			
I	3.936	0.205 %			
Cs	4.285	817.400 ppm			
Ba	4.465	605.200 ppm			
La	4.649	498.500 ppm			
Ce	4.838	299.800 ppm			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49443

Analysis Date: 06-19-1997
Archive Code: 363 363
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
Pr	5.032	244.200 ppm			
Nd	5.228	165.400 ppm			
Pm	5.430	141.000 ppm			
Sm	5.632	161.100 ppm			
Eu	5.842	214.300 ppm			
Gd	6.054	288.800 ppm			
Tb	6.270	741.000 ppm			
Dy	6.492	704.300 ppm			
Ho	6.716	326.600 ppm			
Er	6.944	301.000 ppm			
Tm	7.180	315.300 ppm			
Yb	7.416	115.500 ppm			
Lu	7.655	50.600 ppm			
Hf	7.899	59.020 ppm			
Ta	8.146	61.160 ppm			
W	8.398	41.520 ppm			
Re	8.653	43.750 ppm			
Os	8.912	41.610 ppm			
Ir	9.175	36.760 ppm			
Pt	9.442	37.020 ppm			
Au	9.713	34.890 ppm			
Hg	9.989	34.140 ppm			
Tl	10.269	34.460 ppm			
Lead	10.552	32.550 ppm	49.428 ppm ± 12.70 ppm		0.005 %
Bi	10.839	30.500 ppm			
Th	12.969	39.470 ppm			
U	13.615	60.000 ppm			



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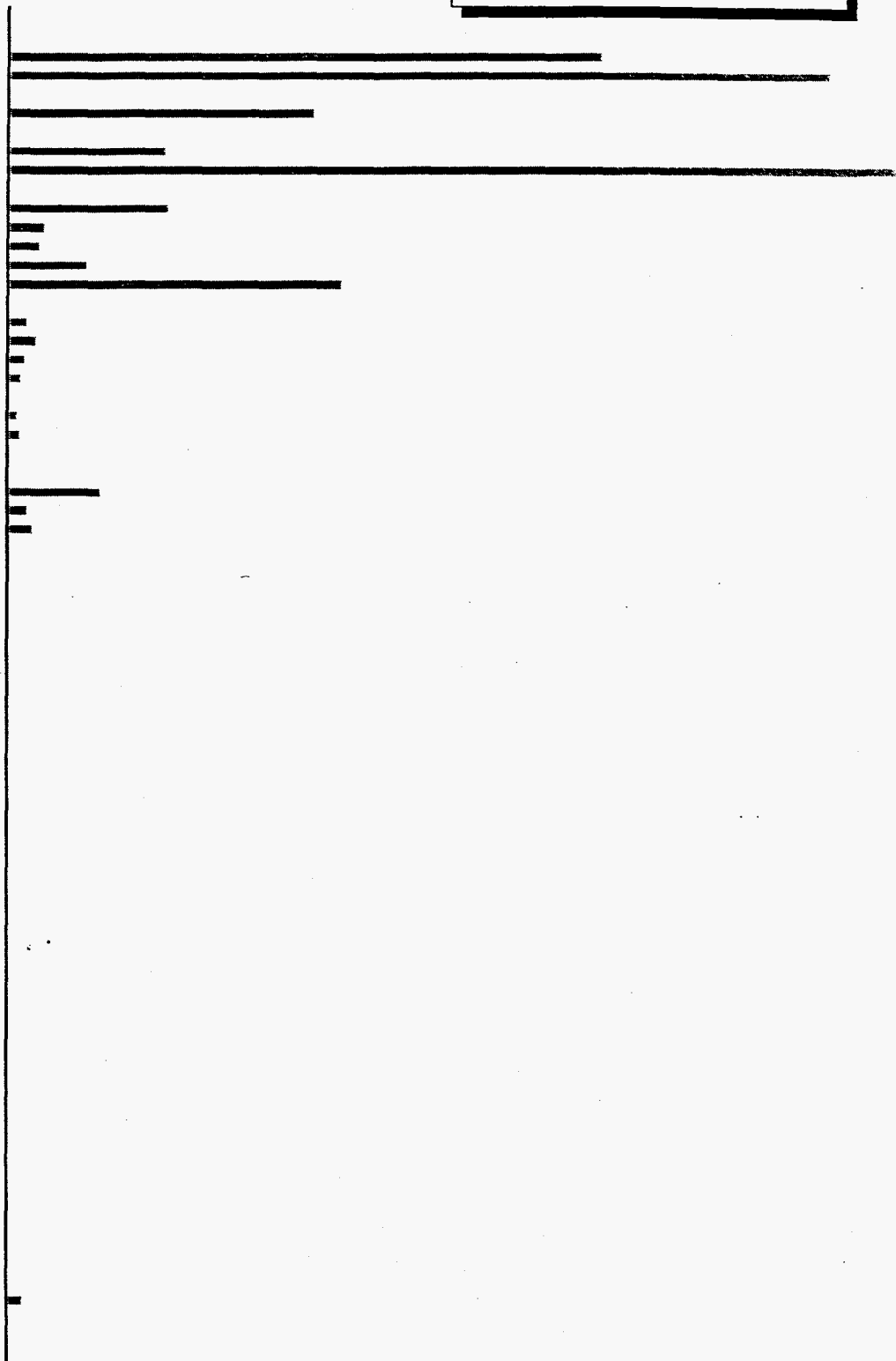
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49443

Archive Code: 363 363
Analysis Date: 06-19-1997
Job: 1980-97

Concentration Graph

Scaling: Square Root
Normalization: Internally Self-Scaling

Aluminum	9.4269 % ±	0.633
Silicon	18.1288 % ±	1.195
Sulfur	2.5053 % ±	0.168
Potassium	0.6497 % ±	0.045
Calcium	21.6287 % ±	1.419
Titanium	0.6748 % ±	0.045
Vanadium	310.854 ppm ±	42.556
Chromium	235.671 ppm ±	24.557
Manganese	0.1585 % ±	0.011
Iron	2.9983 % ±	0.197
Nickel	78.0409 ppm ±	8.920
Copper	180.890 ppm ±	14.363
Zinc	60.9245 ppm ±	6.903
Gallium	28.5176 ppm ±	4.959
Arsenic	12.8134 ppm ±	5.776
Selenium	23.9909 ppm ±	4.611
Strontium	0.2244 % ±	0.015
Yttrium	73.1253 ppm ±	10.011
Zirconium	138.690 ppm ±	23.744



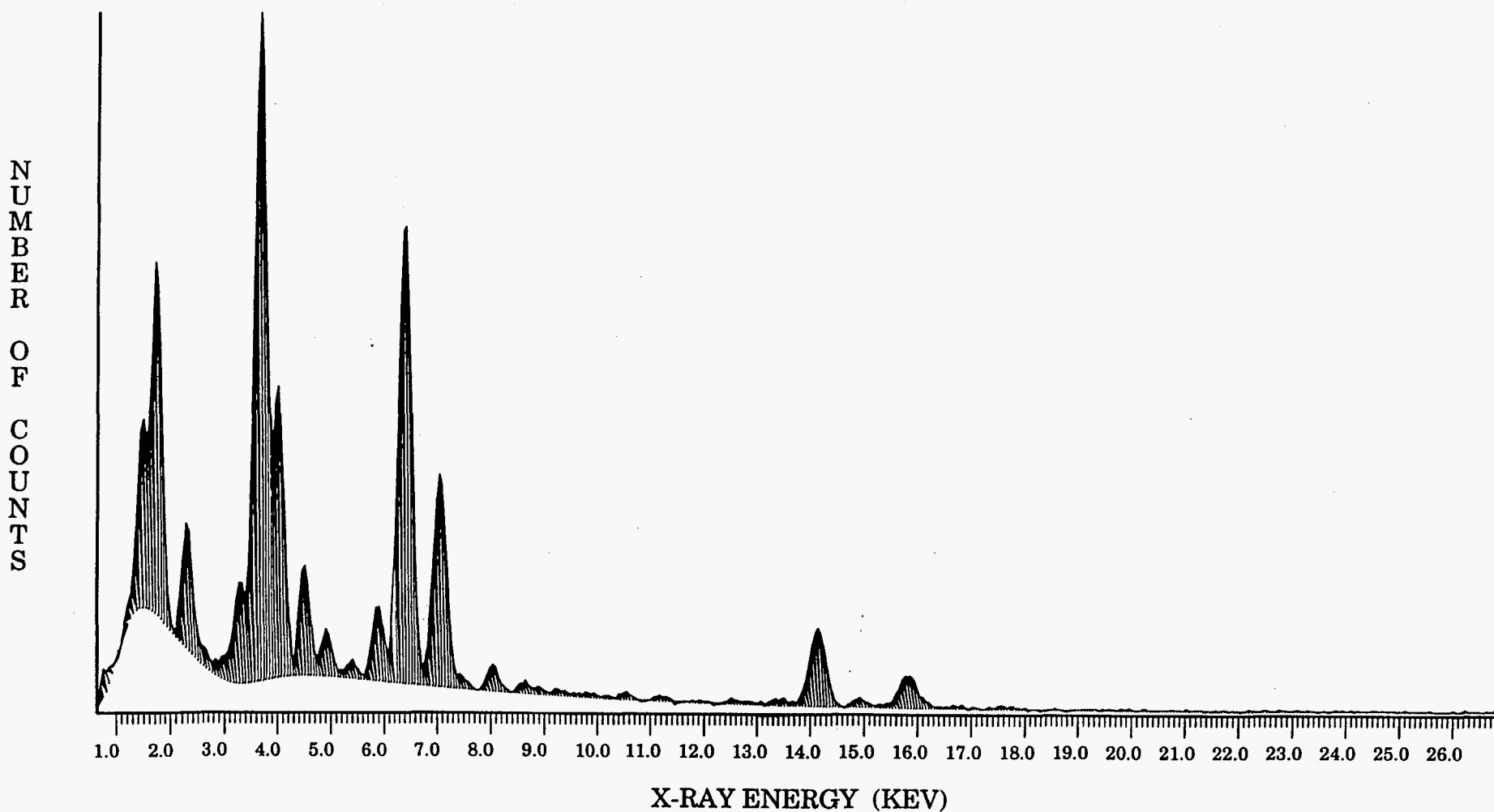
Lead 49.4282 ppm ± 12.698

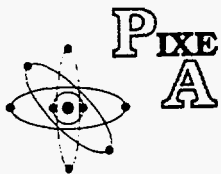
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49443

Analysis Date: 06-19-1997
Archive Code: 363 363
Job: 1980-97

Fit Analysis

Total Error(%)= .624
Sum Of Neg Error(%)= -1.696 Points= 415
Sum Of Pos Error(%)= 2.320 Points= 609





Name: Energy & Environmental Research Center
 Project: Solids/Charlene Crocker
 Target: 49444

Analysis Date: 06-19-1997
 Archive Code: 364 364
 Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
* H	-----		1.000 % ±		
* C	-----		5.000 % ±		
* O	-----		44.509 % ±	0.134 %	
Na	1.041	0.436 %			
Mg	1.254	0.216 %			
Aluminum	1.487	0.121 %	7.876 % ±	0.537 %	14.882 %
Silicon	1.740	776.500 ppm	15.666 % ±	1.034 %	33.515 %
Phosphorus	2.014	905.500 ppm	0.284 % ±	0.041 %	0.651 %
Sulfur	2.308	417.100 ppm	8.191 % ±	0.541 %	20.478 %
Cl	2.622	713.900 ppm			
Potassium	3.314	309.600 ppm	0.545 % ±	0.039 %	0.657 %
Calcium	3.692	157.300 ppm	14.767 % ±	0.969 %	20.662 %
Sc	4.091	449.300 ppm			
Titanium	4.511	45.910 ppm	0.358 % ±	0.024 %	0.597 %
Vanadium	4.952	54.080 ppm	110.992 ppm ±	23.55 ppm	0.020 %
Chromium	5.415	24.310 ppm	72.378 ppm ±	11.52 ppm	0.011 %
Manganese	5.899	19.010 ppm	340.506 ppm ±	25.23 ppm	0.052 %
Iron	6.399	18.630 ppm	1.633 % ±	0.107 %	2.334 %
Co	6.925	68.540 ppm			
Nickel	7.472	14.530 ppm	34.191 ppm ±	4.787 ppm	0.004 %
Copper	8.041	7.921 ppm	55.139 ppm ±	5.575 ppm	0.007 %
Zinc	8.631	7.531 ppm	87.018 ppm ±	7.240 ppm	0.011 %
Gallium	9.243	7.277 ppm	22.267 ppm ±	3.360 ppm	0.003 %
Ge	9.876	7.874 ppm			
As	10.532	9.658 ppm			
Se	11.208	5.360 ppm			
Br	11.924	8.852 ppm			
Rb	13.395	19.070 ppm			
Strontium	14.165	14.280 ppm	762.417 ppm ±	52.84 ppm	0.090 %
Yttrium	14.958	16.090 ppm	26.253 ppm ±	5.560 ppm	0.003 %
Zirconium	15.775	54.670 ppm	100.181 ppm ±	14.42 ppm	0.014 %
Nb	16.615	22.910 ppm			
Mo	17.479	29.160 ppm			
Tc	18.367	31.010 ppm			
Ru	19.279	25.860 ppm			
Rh	20.216	39.160 ppm			
Pd	21.177	43.600 ppm			
Ag	22.163	60.320 ppm			
Cd	23.174	51.520 ppm			
In	24.210	69.480 ppm			
Sn	25.271	83.850 ppm			
Sb	26.359	133.500 ppm			
Te	3.768	0.300 %			
I	3.936	0.169 %			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.

Name: Energy & Environmental Research Center
 Project: Solids/Charlene Crocker
 Target: 49444

Analysis Date: 06-19-1997
 Archive Code: 364 364
 Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Error Mass	Oxide(%)
Cs	4.285	595.500 ppm		
Ba	4.465	420.300 ppm		
La	4.649	343.600 ppm		
Ce	4.838	203.200 ppm		
Pr	5.032	163.100 ppm		
Nd	5.228	103.300 ppm		
Pm	5.430	87.190 ppm		
Sm	5.632	88.840 ppm		
Eu	5.842	105.000 ppm		
Gd	6.054	182.000 ppm		
Tb	6.270	525.600 ppm		
Dy	6.492	499.900 ppm		
Ho	6.716	229.100 ppm		
Er	6.944	215.500 ppm		
Tm	7.180	224.600 ppm		
Yb	7.416	79.760 ppm		
Lu	7.655	32.920 ppm		
Hf	7.899	35.190 ppm		
Ta	8.146	36.000 ppm		
W	8.398	32.800 ppm		
Re	8.653	39.550 ppm		
Os	8.912	32.120 ppm		
Ir	9.175	26.490 ppm		
Pt	9.442	28.180 ppm		
Au	9.713	25.620 ppm		
Hg	9.989	23.590 ppm		
Tl	10.269	26.460 ppm		
Lead	10.552	22.100 ppm	87.642 ppm ± 10.90 ppm	0.009 %
Bi	10.839	24.430 ppm		
Th	12.969	29.910 ppm		
U	13.615	44.280 ppm		



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49444

Archive Code: 364 364
Analysis Date: 06-19-1997
Job: 1980-97

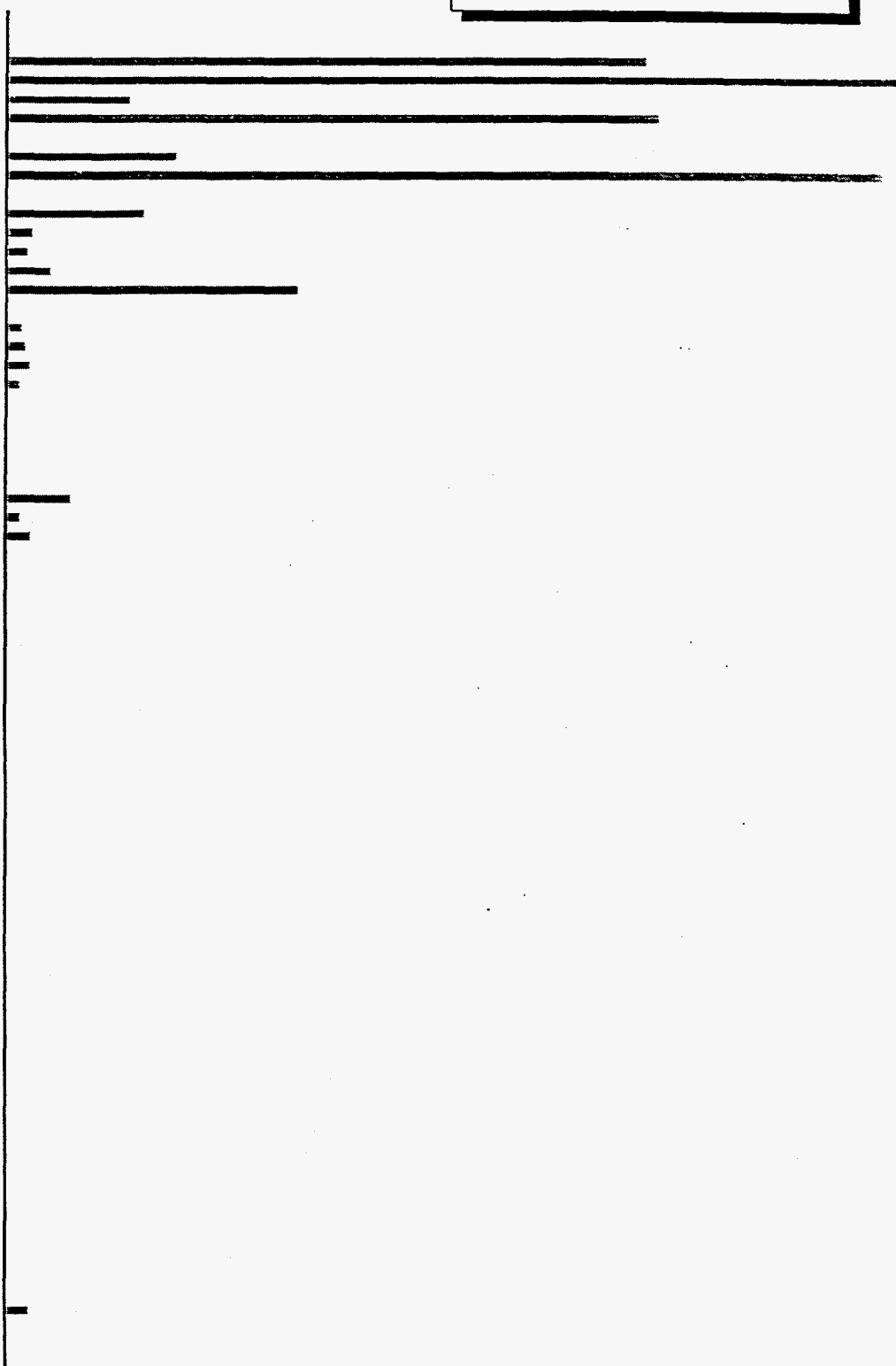
Concentration Graph

Scaling: Square Root
Normalization: Internally Self-Scaling

Aluminum	7.8759 % ±	0.537
Silicon	15.6663 % ±	1.034
Phosphorus	0.2843 % ±	0.041
Sulfur	8.1913 % ±	0.541
Potassium	0.5452 % ±	0.039
Calcium	14.7668 % ±	0.969
Titanium	0.3576 % ±	0.024
Vanadium	110.992 ppm ±	23.553
Chromium	72.3779 ppm ±	11.515
Manganese	340.506 ppm ±	25.231
Iron	1.6328 % ±	0.107
Nickel	34.1912 ppm ±	4.787
Copper	55.1389 ppm ±	5.575
Zinc	87.0184 ppm ±	7.240
Gallium	22.2675 ppm ±	3.360

Strontium	762.417 ppm ±	52.835
Yttrium	26.2534 ppm ±	5.560
Zirconium	100.181 ppm ±	14.416

Lead 87.6423 ppm ± 10.903





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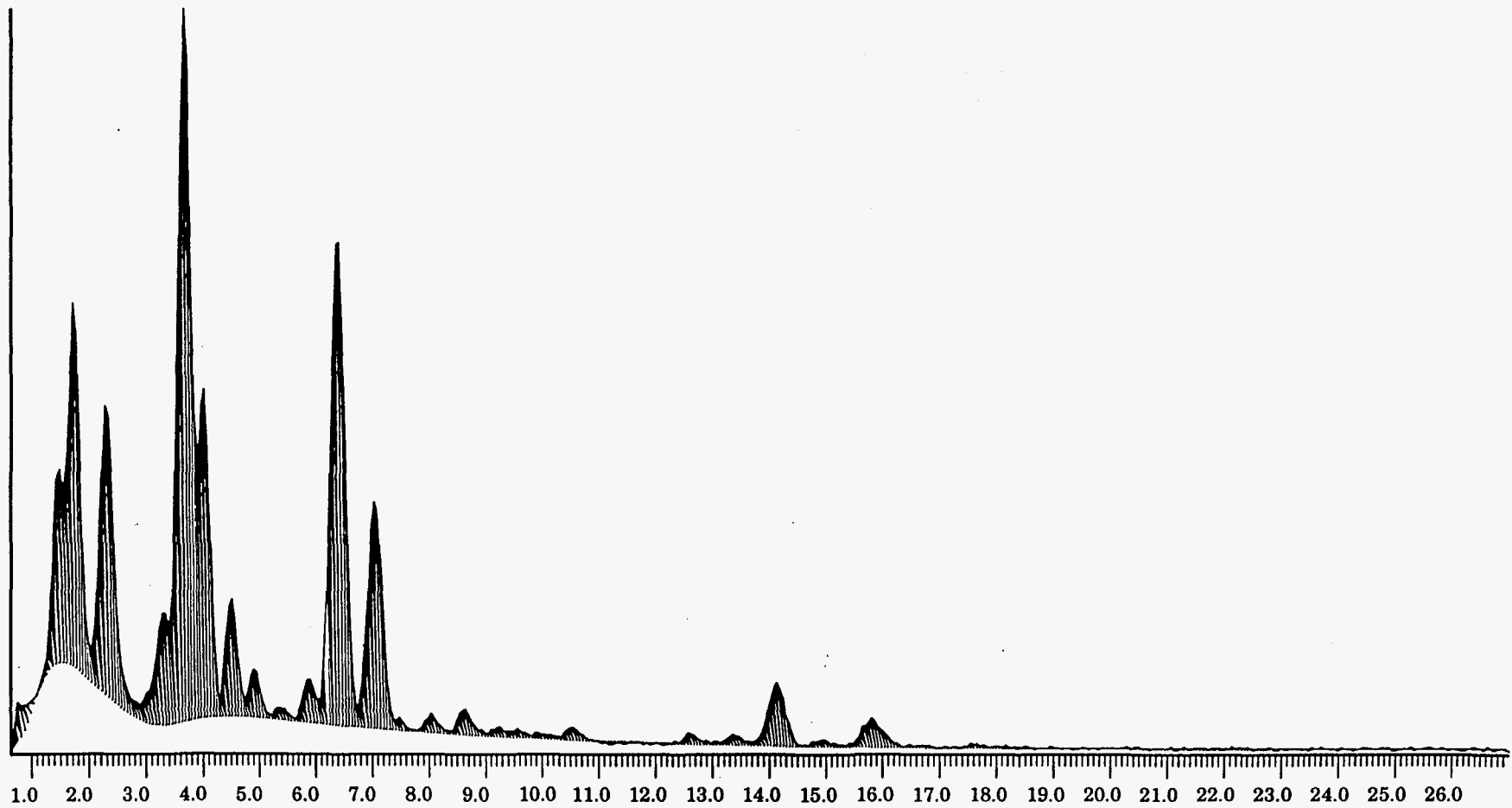
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49444

Analysis Date: 06-19-1997
Archive Code: 364 364
Job: 1980-97

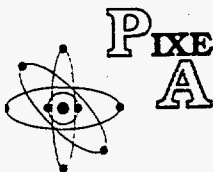
Fit Analysis

Total Error(%)= .768
Sum Of Neg Error(%)= -1.463 Points= 455
Sum Of Pos Error(%)= 2.230 Points= 569

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X-RAY ENERGY (KEV)



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49445

Analysis Date: 06-19-1997
Archive Code: 365 365
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
* O			39.313 % ±	0.134 %	
Na	1.041	0.526 %			
Mg	1.254	0.238 %			
Aluminum	1.487	0.135 %	6.404 % ±	0.450 %	12.100 %
Silicon	1.740	835.400 ppm	13.244 % ±	0.877 %	28.334 %
P	2.014	866.500 ppm			
Sulfur	2.308	387.000 ppm	1.888 % ±	0.129 %	4.719 %
Cl	2.622	457.300 ppm			
Potassium	3.314	470.000 ppm	0.536 % ±	0.040 %	0.646 %
Calcium	3.692	273.200 ppm	35.440 % ±	2.325 %	49.588 %
Sc	4.091	0.112 %			
Titanium	4.511	84.390 ppm	0.446 % ±	0.031 %	0.743 %
Vanadium	4.952	88.260 ppm	178.131 ppm ±	42.15 ppm	0.032 %
Chromium	5.415	44.690 ppm	158.818 ppm ±	23.68 ppm	0.023 %
Manganese	5.899	34.040 ppm	0.102 % ±	0.007 %	0.156 %
Iron	6.399	33.510 ppm	2.375 % ±	0.156 %	3.395 %
Co	6.925	108.900 ppm			
Nickel	7.472	24.930 ppm	74.362 ppm ±	9.652 ppm	0.009 %
Copper	8.041	15.090 ppm	92.550 ppm ±	9.894 ppm	0.012 %
Zinc	8.631	13.570 ppm	80.156 ppm ±	8.729 ppm	0.010 %
Gallium	9.243	12.190 ppm	17.592 ppm ±	4.952 ppm	0.002 %
Ge	9.876	13.270 ppm			
As	10.532	12.290 ppm			
Se	11.208	9.988 ppm			
Br	11.924	12.940 ppm			
Rb	13.395	28.930 ppm			
Strontium	14.165	30.480 ppm	0.172 % ±	0.012 %	0.203 %
Yttrium	14.958	22.050 ppm	37.173 ppm ±	8.516 ppm	0.005 %
Zirconium	15.775	100.900 ppm	126.968 ppm ±	25.03 ppm	0.017 %
Nb	16.615	37.580 ppm			
Mo	17.479	46.120 ppm			
Tc	18.367	47.630 ppm			
Ru	19.279	42.530 ppm			
Rh	20.216	49.470 ppm			
Pd	21.177	66.370 ppm			
Ag	22.163	79.490 ppm			
Cd	23.174	87.400 ppm			
In	24.210	121.400 ppm			
Sn	25.271	122.000 ppm			
Sb	26.359	161.300 ppm			
Te	3.768	0.548 %			
I	3.936	0.314 %			
Cs	4.285	0.139 %			
Ba	4.465	658.900 ppm			
La	4.649	544.600 ppm			
Ce	4.838	334.600 ppm			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.

Name: Energy & Environmental Research Center
 Project: Solids/Charlene Crocker
 Target: 49445

Analysis Date: 06-19-1997
 Archive Code: 365 365
 Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Error Mass	Oxide(%)
Pr	5.032	274.800 ppm		
Nd	5.228	190.300 ppm		
Pm	5.430	165.000 ppm		
Sm	5.632	178.500 ppm		
Eu	5.842	227.300 ppm		
Gd	6.054	321.500 ppm		
Tb	6.270	842.800 ppm		
Dy	6.492	796.400 ppm		
Ho	6.716	366.400 ppm		
Er	6.944	342.900 ppm		
Tm	7.180	357.300 ppm		
Yb	7.416	132.400 ppm		
Lu	7.655	62.360 ppm		
Hf	7.899	63.330 ppm		
Ta	8.146	61.770 ppm		
W	8.398	50.990 ppm		
Re	8.653	56.830 ppm		
Os	8.912	50.190 ppm		
Ir	9.175	40.270 ppm		
Pt	9.442	39.070 ppm		
Au	9.713	39.590 ppm		
Hg	9.989	40.330 ppm		
Tl	10.269	39.330 ppm		
Pb	10.552	34.890 ppm		
Bi	10.839	32.490 ppm		
Th	12.969	42.510 ppm		
U	13.615	69.820 ppm		

Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49445

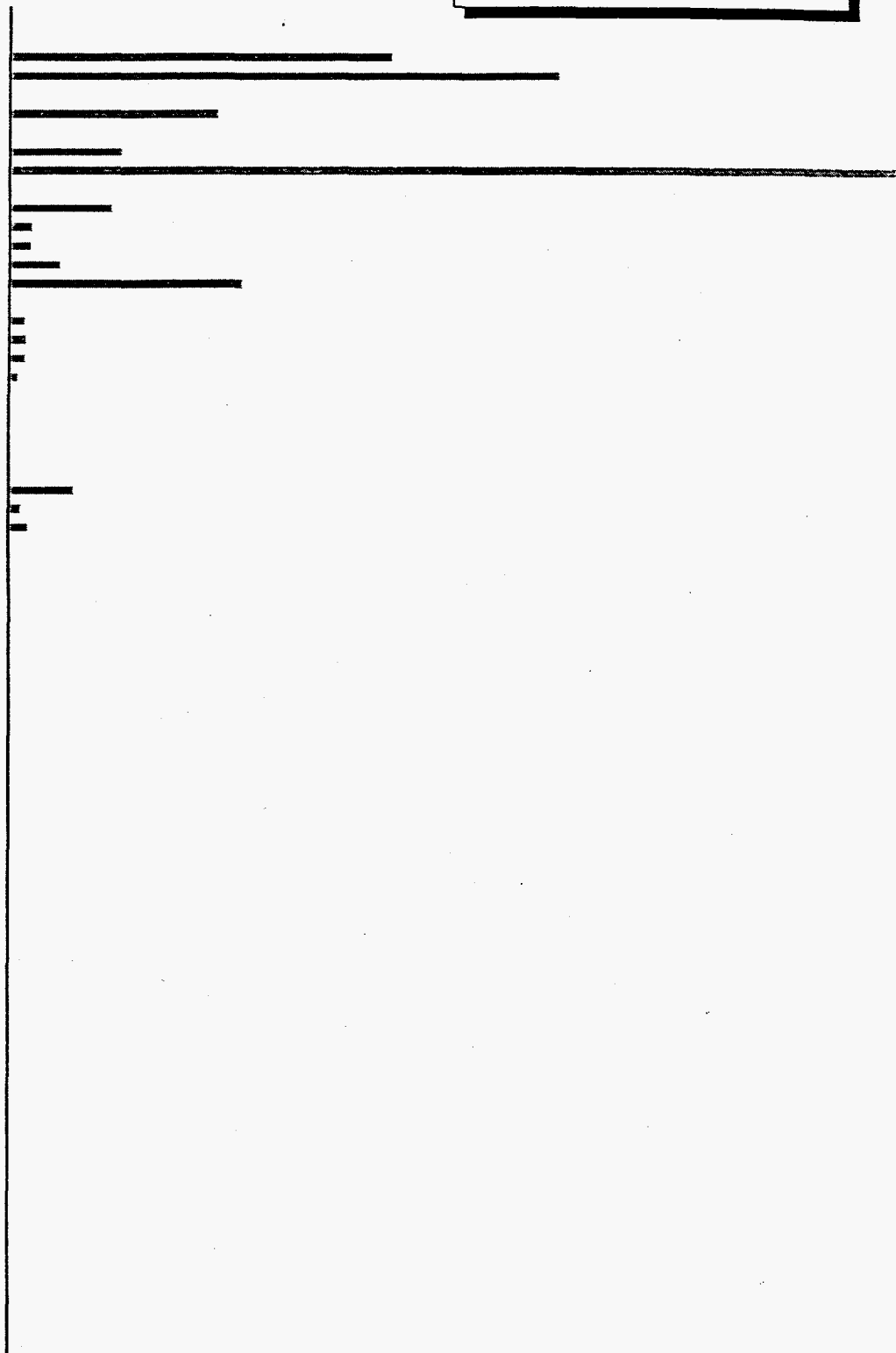
Archive Code: 365 365
Analysis Date: 06-19-1997
Job: 1980-97

Concentration Graph

Scaling: Square Root
Normalization: Internally Self-Scaling

Aluminum	6.4038 % ±	0.450
Silicon	13.2445 % ±	0.877
Sulfur	1.8875 % ±	0.129
Potassium	0.5359 % ±	0.040
Calcium	35.4403 % ±	2.325
Titanium	0.4455 % ±	0.031
Vanadium	178.131 ppm ±	42.146
Chromium	158.818 ppm ±	23.680
Manganese	0.1024 % ±	0.007
Iron	2.3750 % ±	0.156
Nickel	74.3623 ppm ±	9.652
Copper	92.5502 ppm ±	9.894
Zinc	80.1559 ppm ±	8.729
Gallium	17.5923 ppm ±	4.952

Strontium	0.1716 % ±	0.012
Tritium	37.1725 ppm ±	8.516
Zirconium	126.968 ppm ±	25.025



Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49445

Analysis Date: 06-19-1997
Archive Code: 365 365
Job: 1980-97

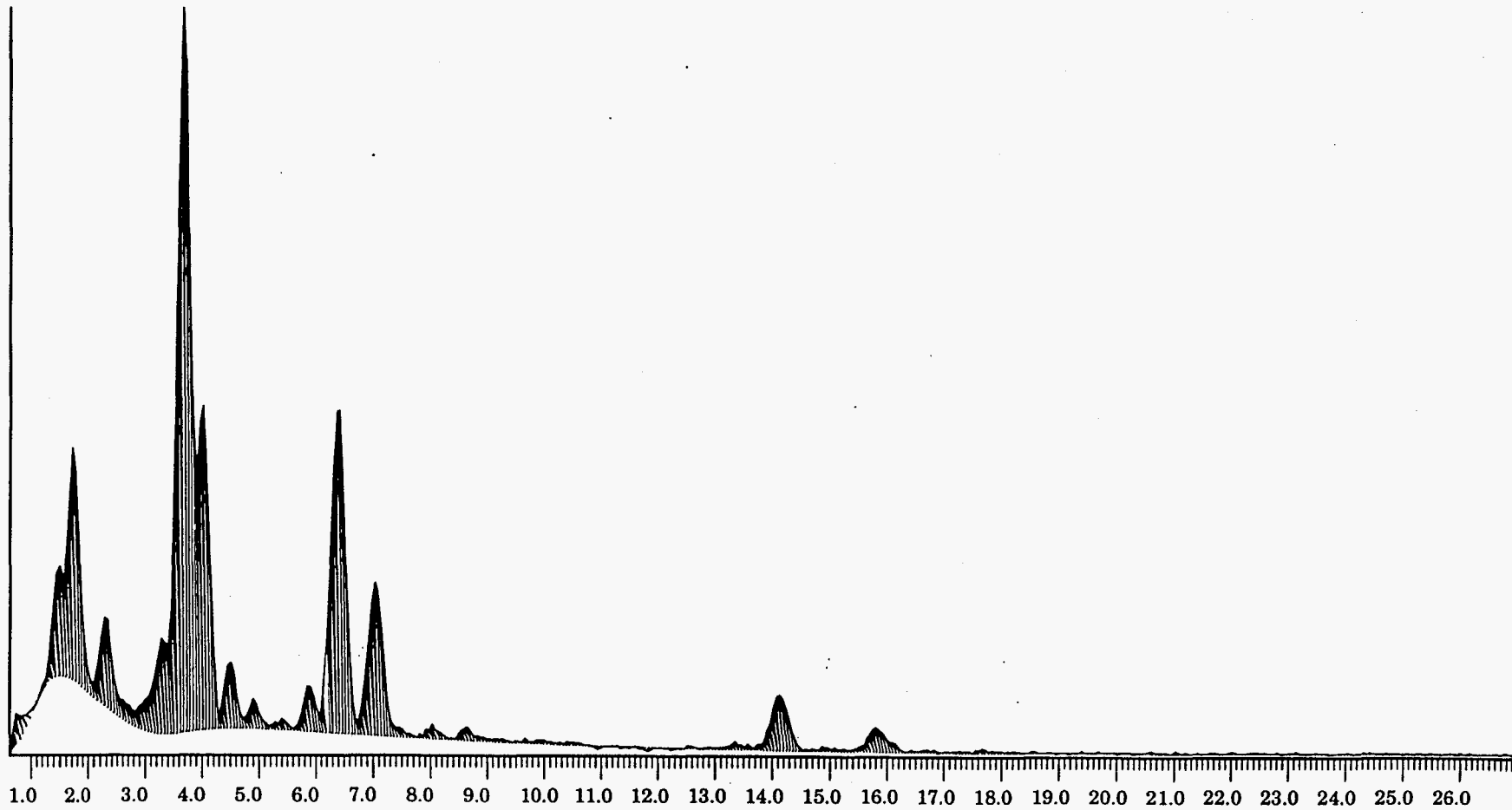
Fit Analysis

Total Error(%)= .611
Sum Of Neg Error(%)= -1.586 Points= 421
Sum Of Pos Error(%)= 2.197 Points= 603

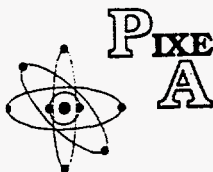
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X-RAY ENERGY (KEV)



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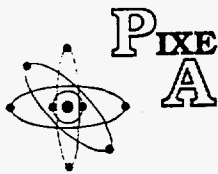
Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49446

Analysis Date: 06-19-1997
Archive Code: 366 366
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
* O			43.869 % ±	0.145 %	
Na	1.041	0.579 %			
Mg	1.254	0.237 %			
Aluminum	1.487	0.135 %	6.113 % ±	0.434 %	11.550 %
Silicon	1.740	838.600 ppm	12.841 % ±	0.851 %	27.471 %
Phosphorus	2.014	928.400 ppm	0.431 % ±	0.053 %	0.988 %
Sulfur	2.308	430.100 ppm	7.778 % ±	0.514 %	19.445 %
Cl	2.622	766.800 ppm			
Potassium	3.314	409.700 ppm	0.486 % ±	0.036 %	0.586 %
Calcium	3.692	199.900 ppm	26.353 % ±	1.729 %	36.873 %
Sc	4.091	704.000 ppm			
Titanium	4.511	52.010 ppm	0.287 % ±	0.020 %	0.479 %
Vanadium	4.952	54.950 ppm	88.555 ppm ±	24.99 ppm	0.016 %
Chromium	5.415	27.870 ppm	79.474 ppm ±	13.59 ppm	0.012 %
Manganese	5.899	21.770 ppm	399.674 ppm ±	29.82 ppm	0.061 %
Iron	6.399	20.130 ppm	1.639 % ±	0.108 %	2.344 %
Co	6.925	70.830 ppm			
Nickel	7.472	16.160 ppm	43.332 ppm ±	5.924 ppm	0.006 %
Copper	8.041	10.030 ppm	46.012 ppm ±	5.636 ppm	0.006 %
Zinc	8.631	8.575 ppm	131.608 ppm ±	10.36 ppm	0.016 %
Gallium	9.243	8.534 ppm	15.873 ppm ±	3.468 ppm	0.002 %
Ge	9.876	9.650 ppm			
Arsenic	10.532	9.308 ppm	14.769 ppm ±	4.471 ppm	0.002 %
Se	11.208	6.074 ppm			
Br	11.924	10.430 ppm			
Rb	13.395	19.510 ppm			
Strontium	14.165	15.990 ppm	0.104 % ±	0.007 %	0.123 %
Yttrium	14.958	15.680 ppm	28.348 ppm ±	5.851 ppm	0.004 %
Zirconium	15.775	63.690 ppm	72.791 ppm ±	14.94 ppm	0.010 %
Nb	16.615	22.660 ppm			
Mo	17.479	25.840 ppm			
Tc	18.367	28.420 ppm			
Ru	19.279	30.990 ppm			
Rh	20.216	34.740 ppm			
Pd	21.177	40.960 ppm			
Ag	22.163	49.070 ppm			
Cd	23.174	66.430 ppm			
In	24.210	83.460 ppm			
Sn	25.271	92.570 ppm			
Sb	26.359	109.300 ppm			
Te	3.768	0.402 %			
I	3.936	0.225 %			
Cs	4.285	853.000 ppm			
Ba	4.465	408.600 ppm			
La	4.649	336.000 ppm			
Ce	4.838	204.700 ppm			

1.* Those elements below Sodium in atomic number (i.e. C,O,H,N,etc) represent an unanalyzed component of the sample. See your interpretation sheet for a more detailed explanation.

2. Concentration and Detection Limit units are expressed as either % or ppm (parts per million) A ppm is equivalent to 0.0001 % and is used in order to avoid having to format many needless zeros after the decimal place.



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Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49446

Analysis Date: 06-19-1997
Archive Code: 366 366
Job: 1980-97

Element Name	Energy (KeV)	Det. Limit 95% Conf.	Concentration Mass	Error	Oxide(%)
Pr	5.032	166.000 ppm			
Nd	5.228	114.900 ppm			
Pm	5.430	99.690 ppm			
Sm	5.632	102.700 ppm			
Eu	5.842	119.300 ppm			
Gd	6.054	195.000 ppm			
Tb	6.270	546.400 ppm			
Dy	6.492	515.800 ppm			
Ho	6.716	235.700 ppm			
Er	6.944	222.700 ppm			
Tm	7.180	230.700 ppm			
Yb	7.416	85.560 ppm			
Lu	7.655	39.080 ppm			
Hf	7.899	39.680 ppm			
Ta	8.146	38.350 ppm			
W	8.398	38.800 ppm			
Re	8.653	48.000 ppm			
Os	8.912	36.560 ppm			
Ir	9.175	27.950 ppm			
Pt	9.442	31.850 ppm			
Au	9.713	31.760 ppm			
Hg	9.989	28.080 ppm			
Tl	10.269	29.450 ppm			
Lead	10.552	27.910 ppm	52.784 ppm ±	9.718 ppm	0.006 %
Bi	10.839	25.520 ppm			
Th	12.969	29.810 ppm			
U	13.615	45.220 ppm			



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Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49446

Archive Code: 366 366
Analysis Date: 06-19-1997
Job: 1980-97

Concentration Graph

Scaling: Square Root
Normalization: Internally Self-Scaling

Aluminum 6.1128 % ± 0.434
Silicon 12.8411 % ± 0.851
Phosphorus 0.4314 % ± 0.053
Sulfur 7.7780 % ± 0.514

Potassium 0.4861 % ± 0.036
Calcium 26.3530 % ± 1.729

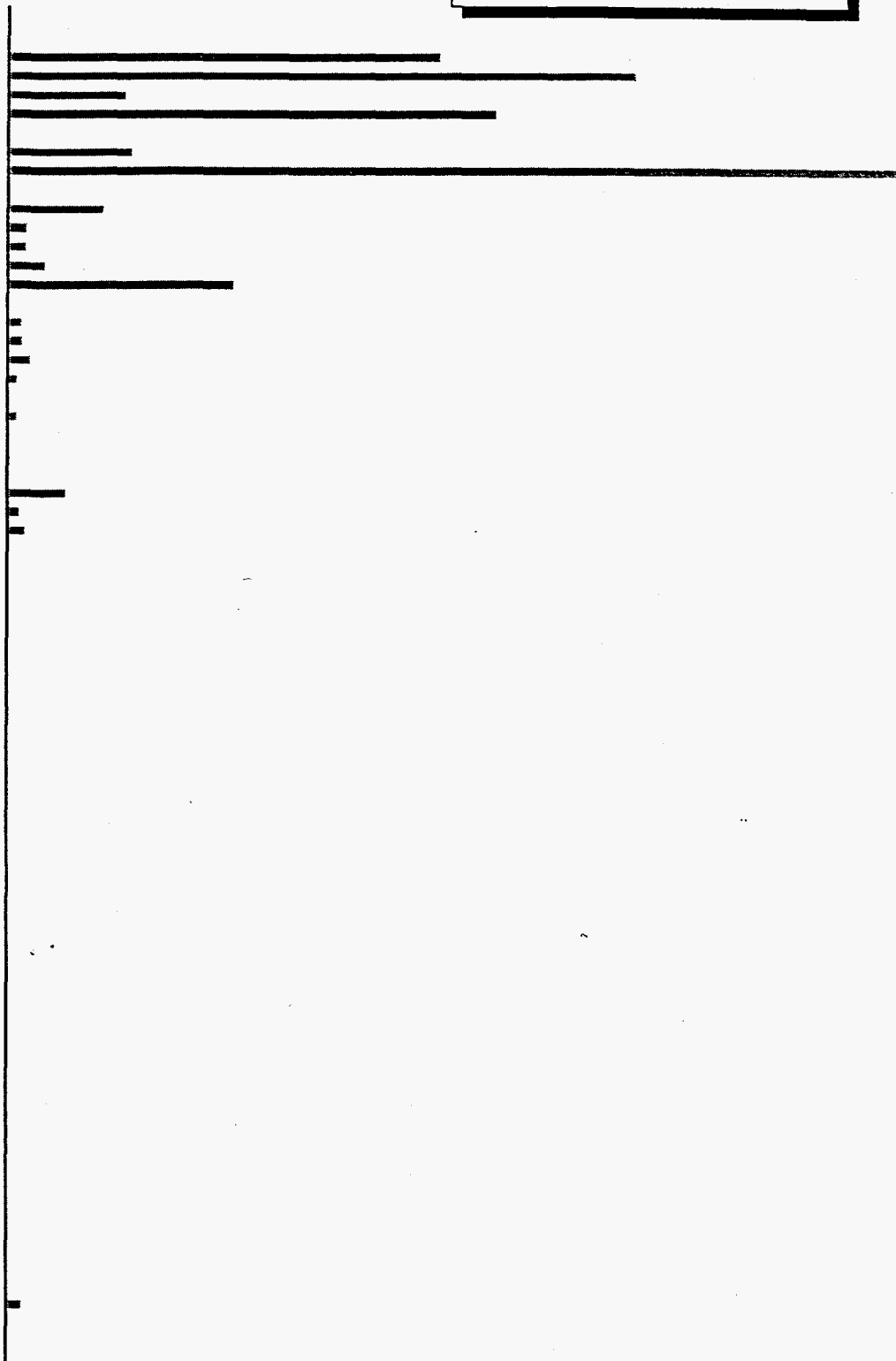
Titanium 0.2870 % ± 0.020
Vanadium 88.5548 ppm ± 24.990
Chromium 79.4739 ppm ± 13.590
Manganese 399.674 ppm ± 29.816
Iron 1.6395 % ± 0.108

Nickel 43.3324 ppm ± 5.924
Copper 46.0118 ppm ± 5.636
Zinc 131.608 ppm ± 10.358
Gallium 15.8729 ppm ± 3.468

Arsenic 14.7691 ppm ± 4.471

Strontium 0.1044 % ± 0.007
Yttrium 28.3481 ppm ± 5.851
Zirconium 72.7907 ppm ± 14.944

Lead 52.7842 ppm ± 9.718

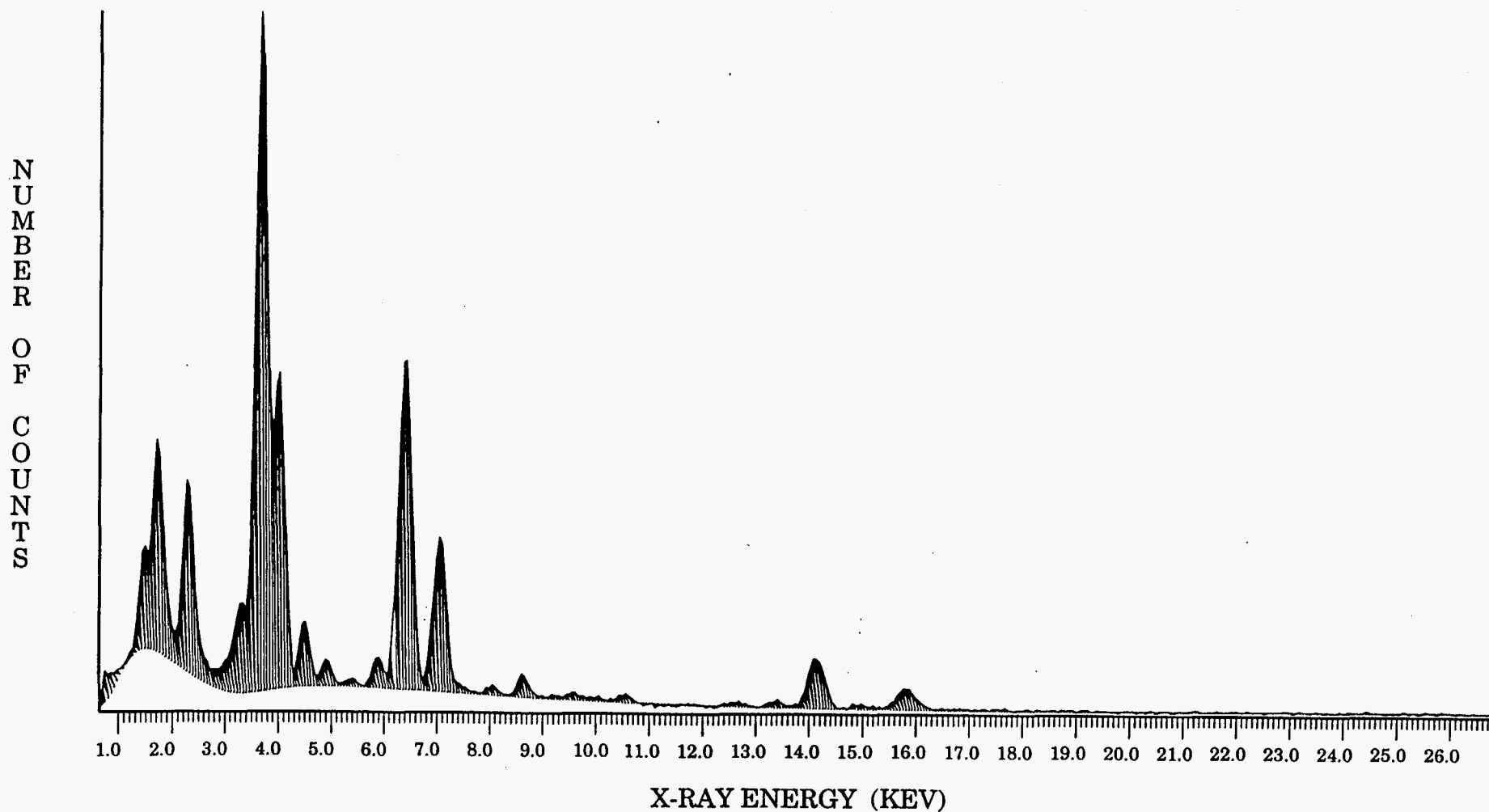


Name: Energy & Environmental Research Center
Project: Solids/Charlene Crocker
Target: 49446

Analysis Date: 06-19-1997
Archive Code: 366 366
Job: 1980-97

Fit Analysis

Total Error(%)= .551
Sum Of Neg Error(%)= -1.544 Points= 421
Sum Of Pos Error(%)= 2.095 Points= 603

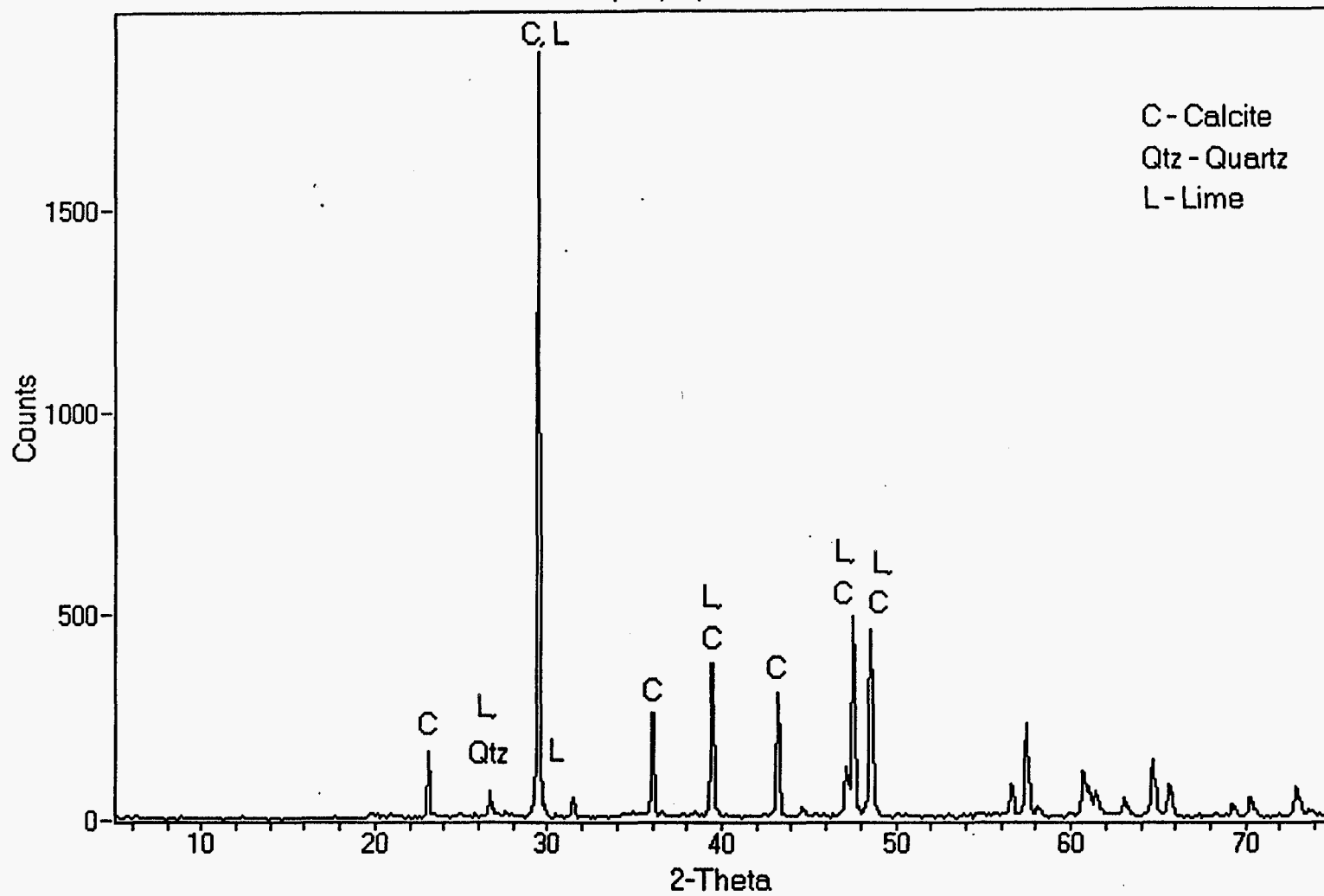


APPENDIX B

X-RAY DIFFRACTOGRAMS

ID: Limestone
File: 08.DIF

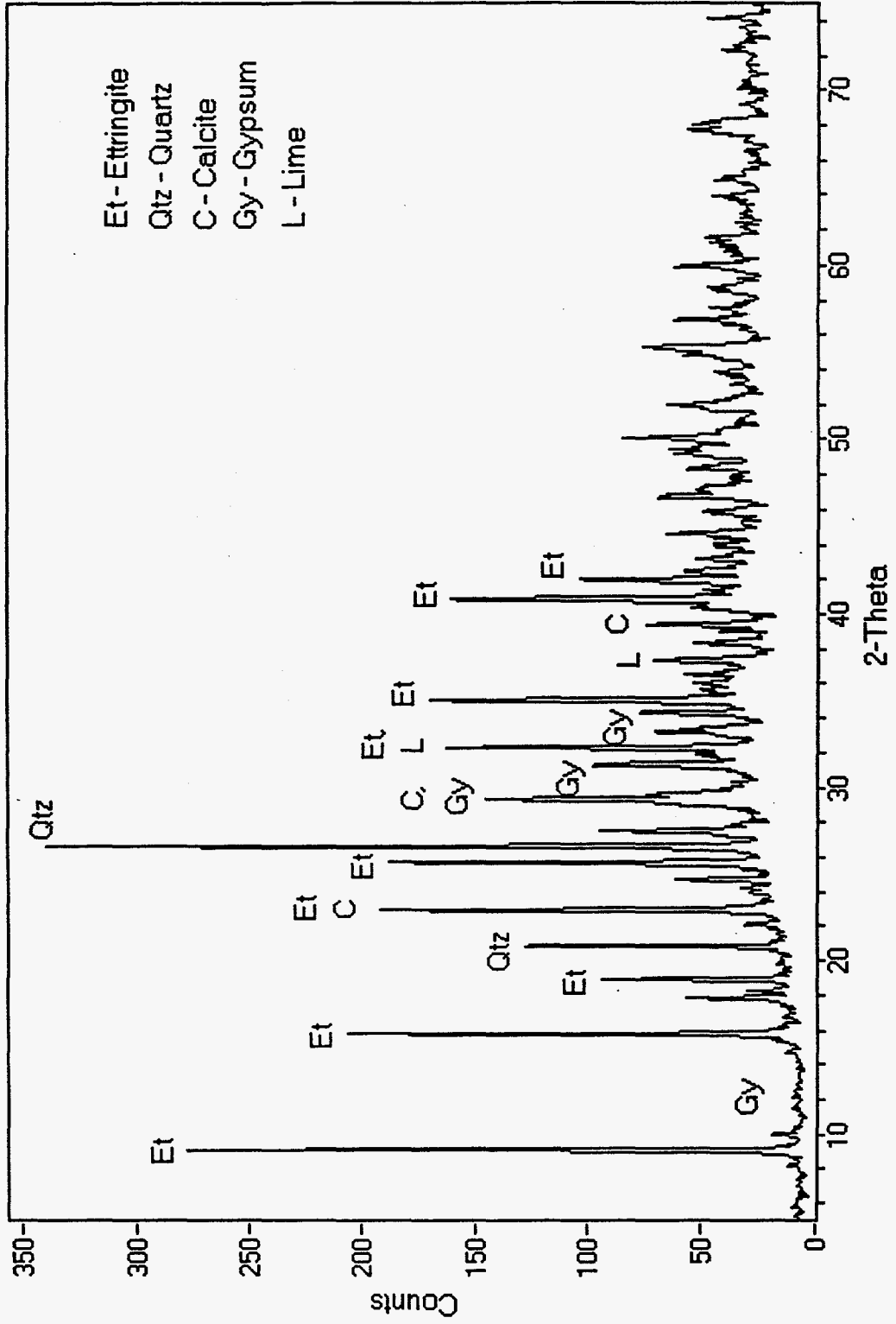
Scan: 5-75/.02/.5/#3501, Anode: CU



ID: Leached Composite Ash - Non-SNCR

File: 09.DIF

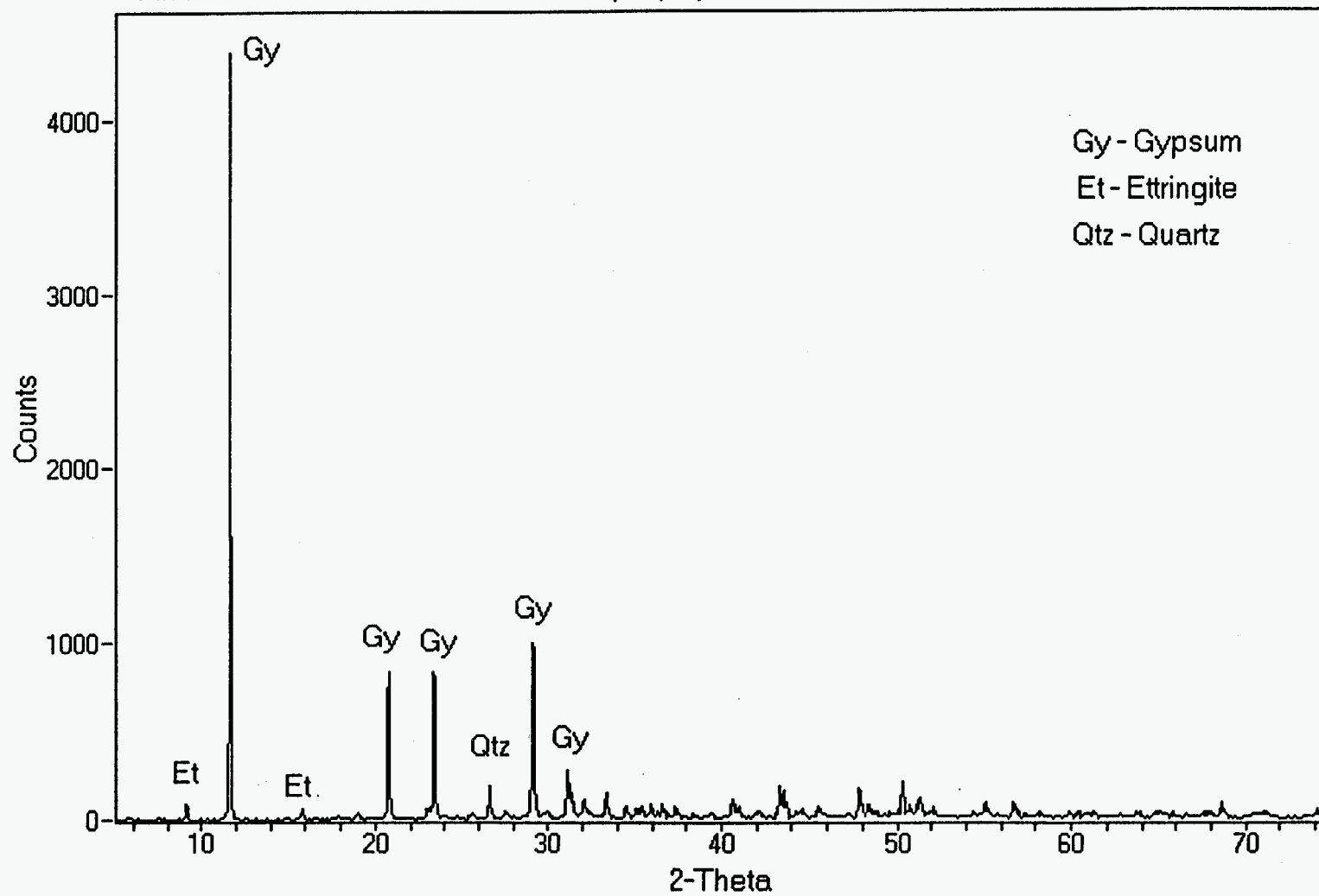
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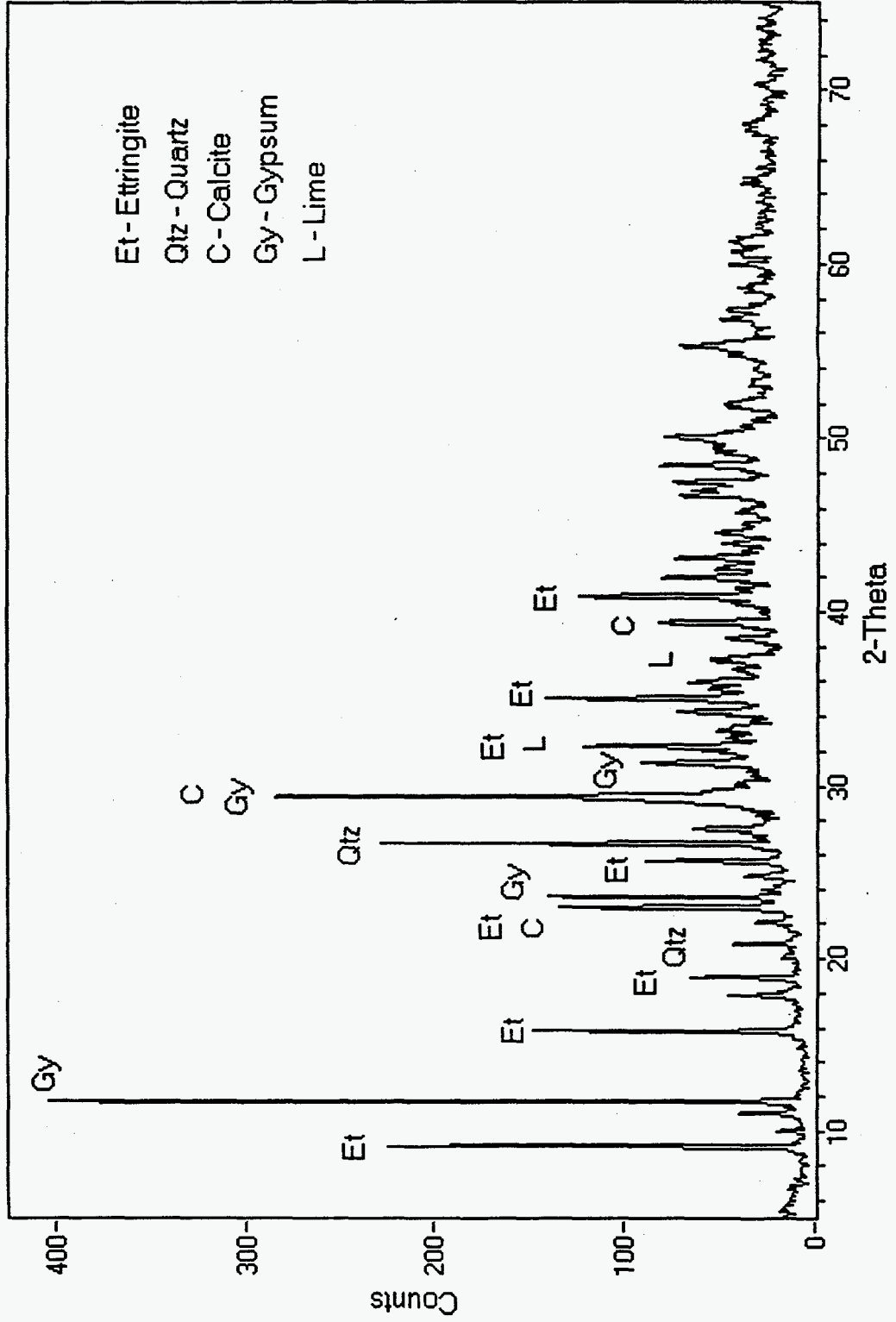
ID: Leached Spent Bed - Non SNCR

File: 11.DIF

Scan: 5-75/.02/.5/#3501, Anode: CU



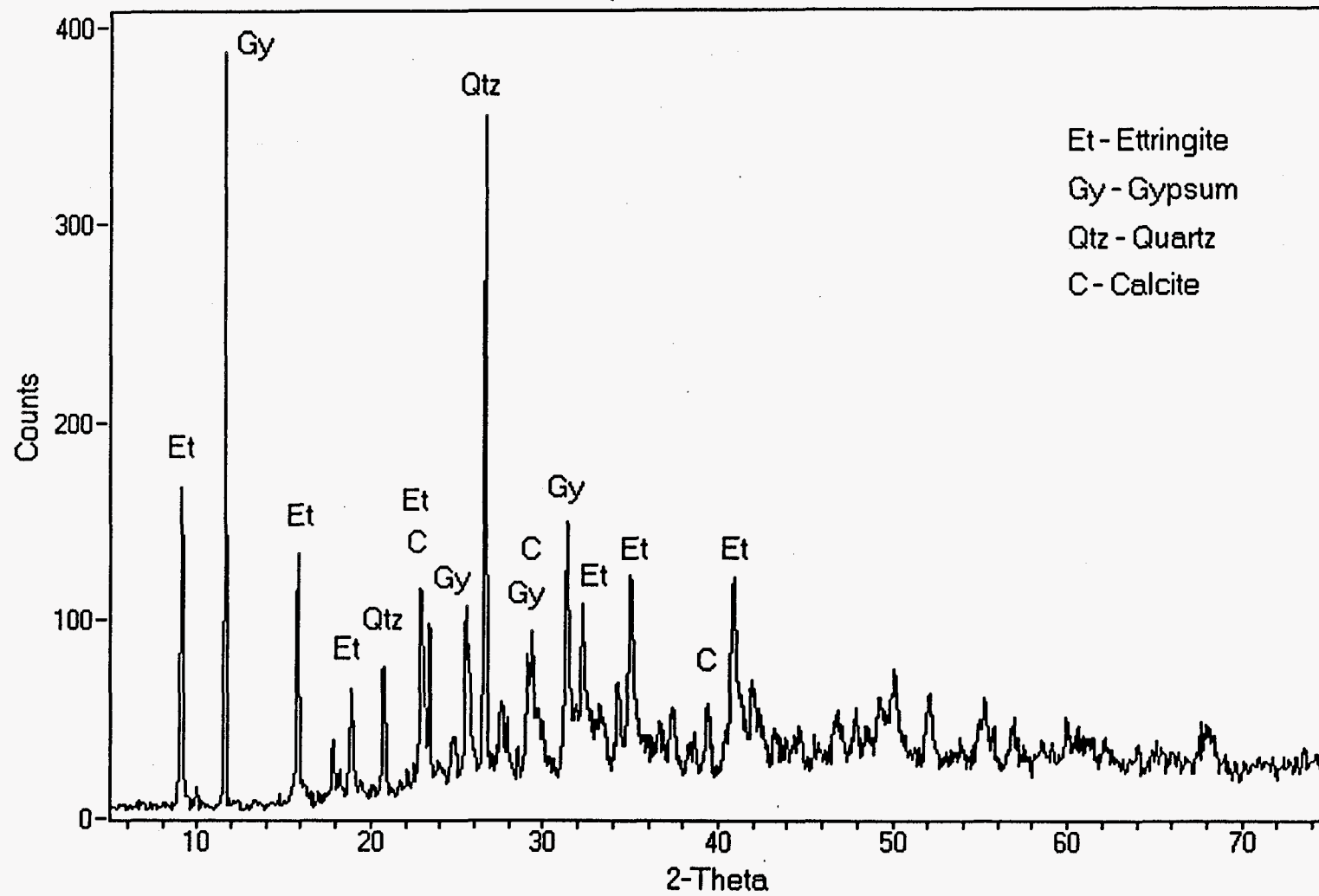
ID: Leached Composite Ash - SNCR
File: 10.DIF Scan: 5-75/02/ 5/#3501, Anode: CU



ID: Leached Spent Bed - SNCR

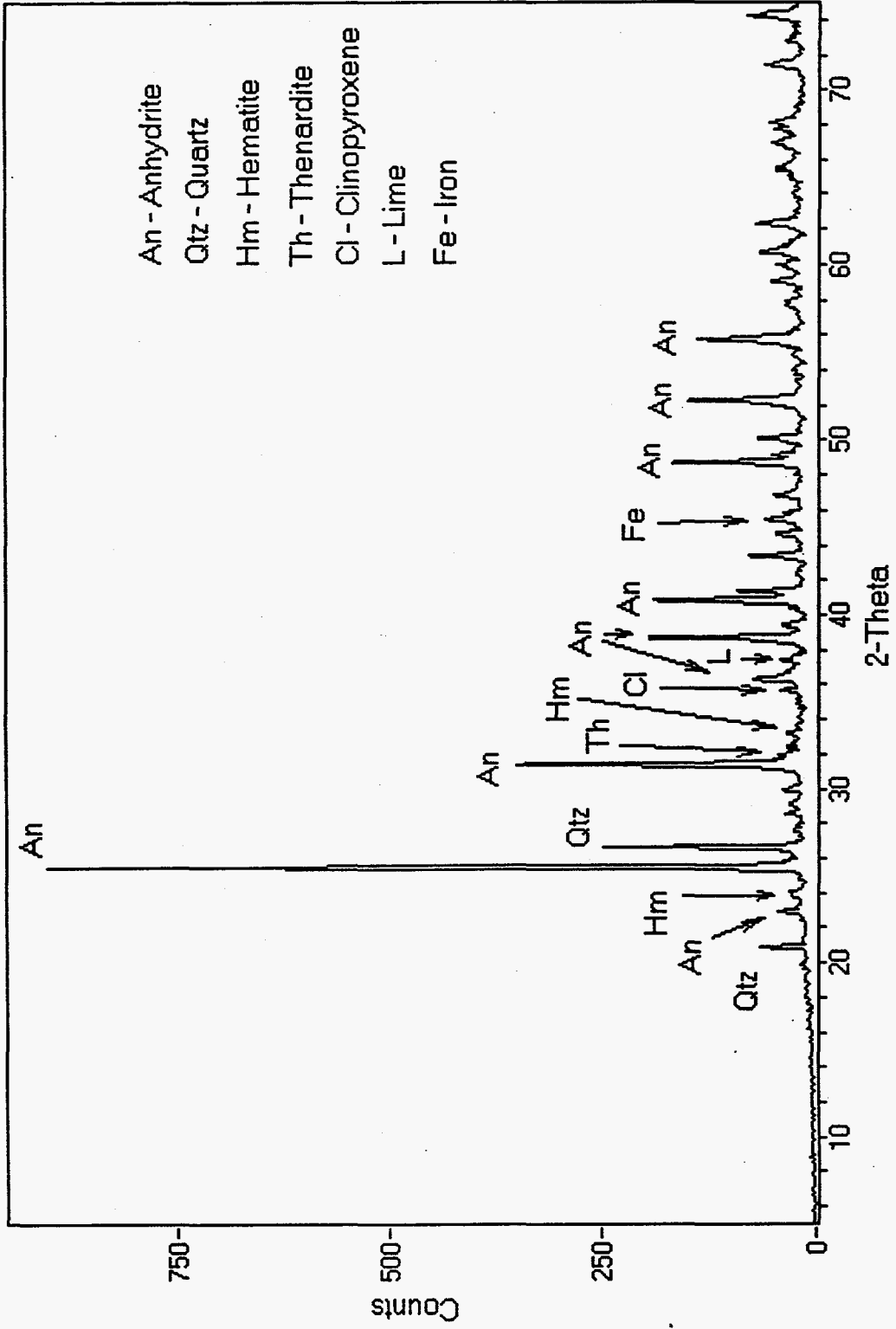
File: 12.DIF

Scan: 5-75/02/.5/#3501, Anode: CU



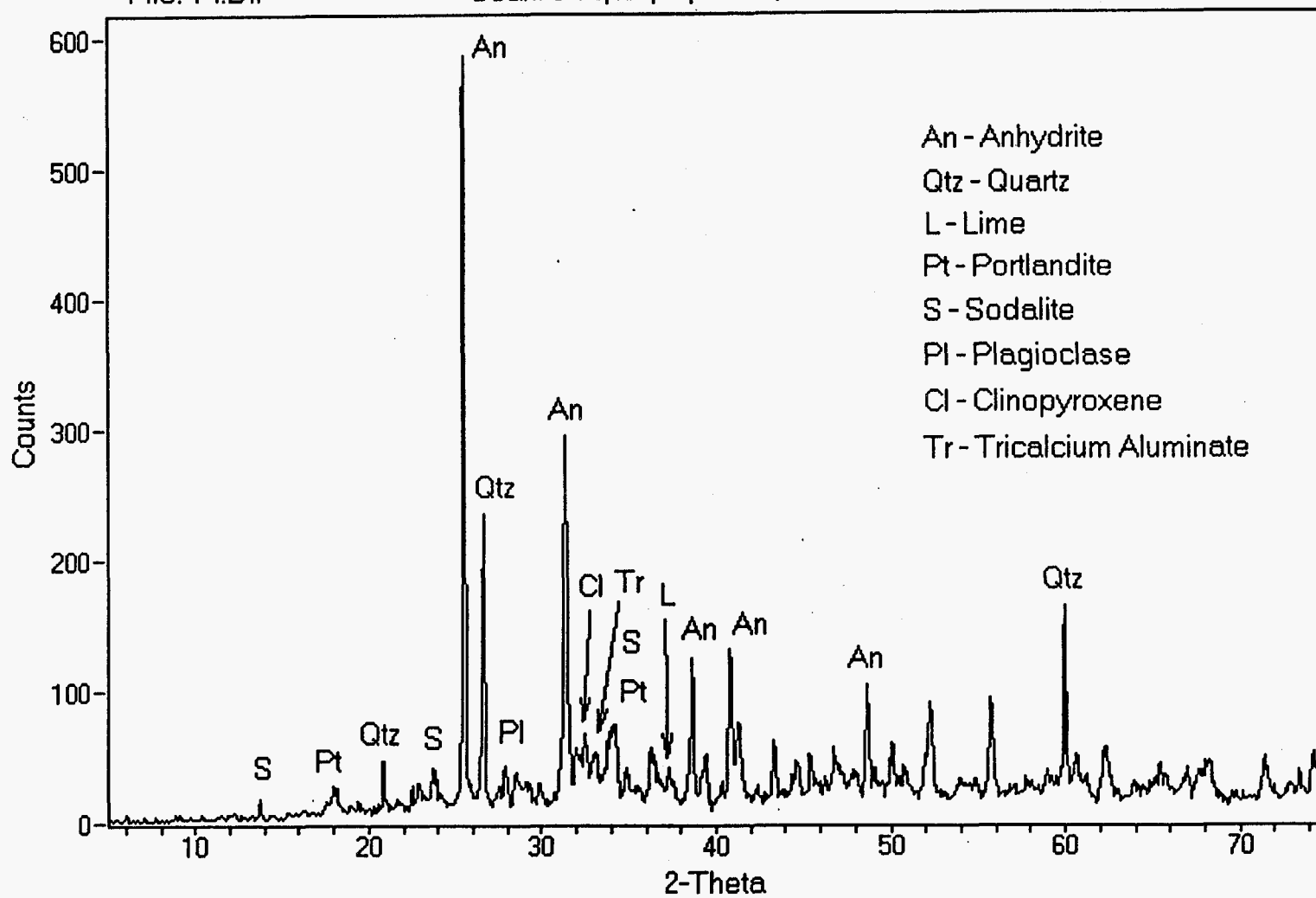
ID: Spent Bed - Non-SNCR
File: 13.DIF

Scan: 5-75/02/ 5/#3501, Anode: CU



ID: Spent Bed - SNCR
File: 14.DIF

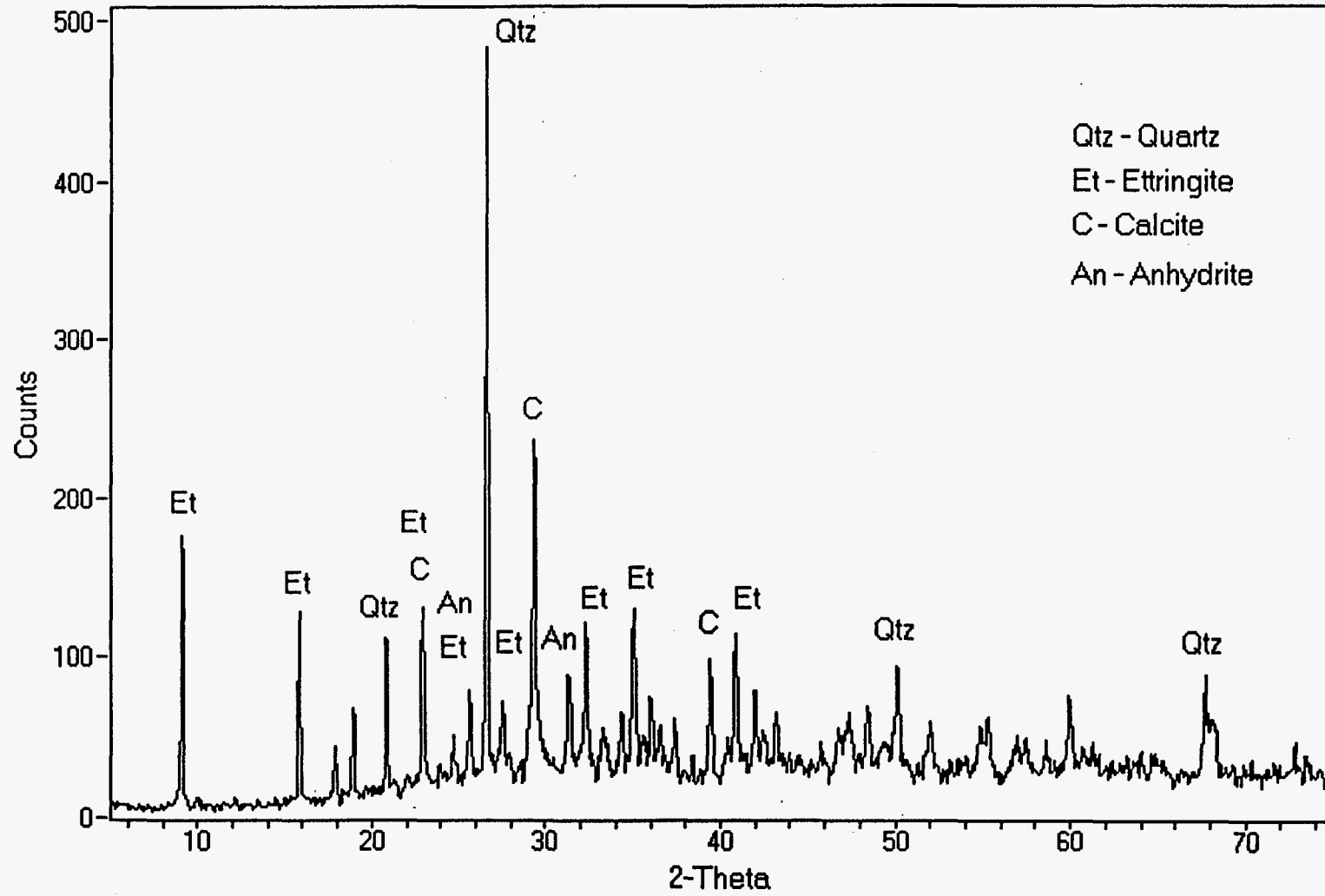
Scan: 5-75/02/.5/#3501, Anode: CU



ID: Leached Fly Ash - Non-SNCR

File: 15.DIF

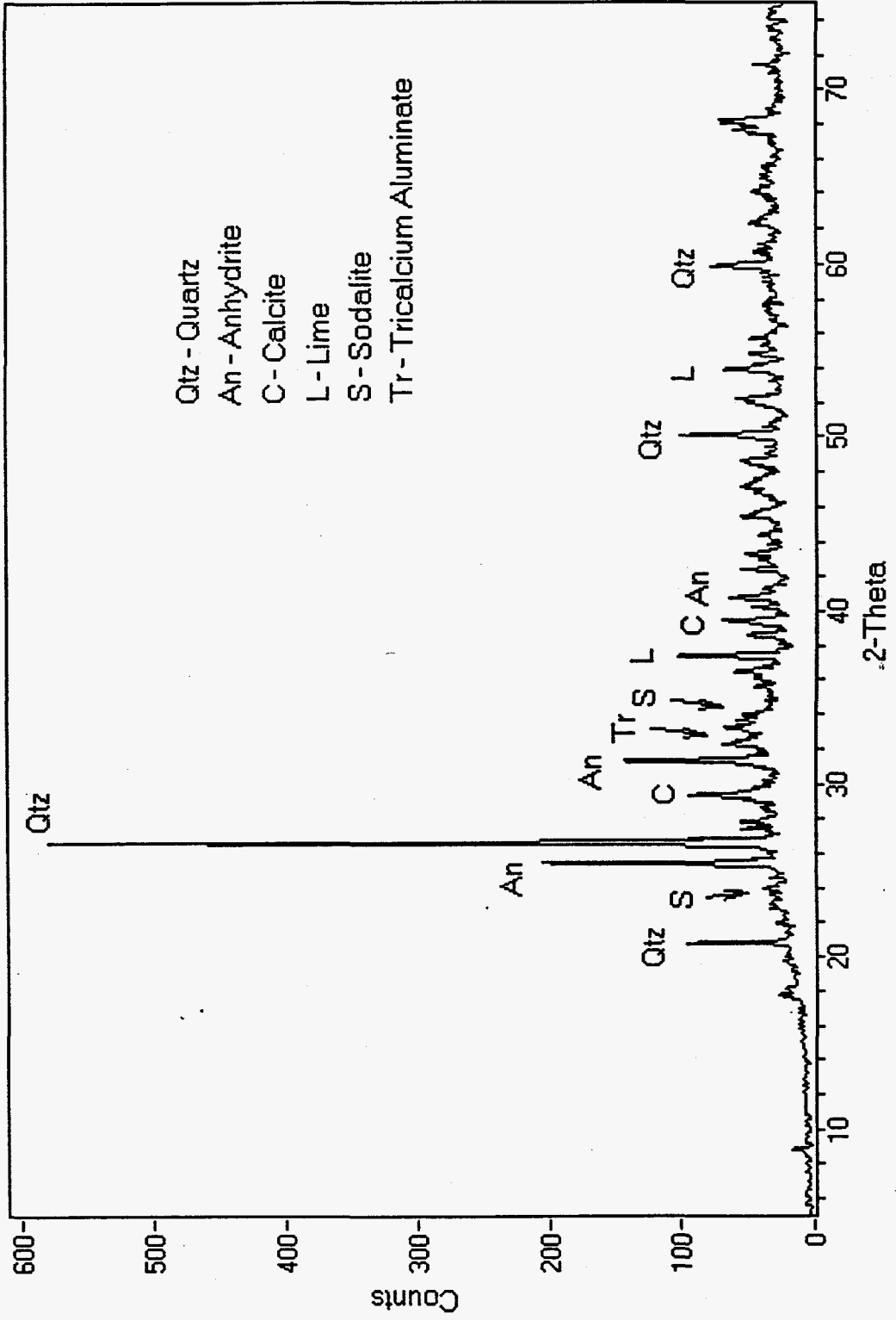
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ID: Fly Ash - Non-SNCR

File: 17.DIF

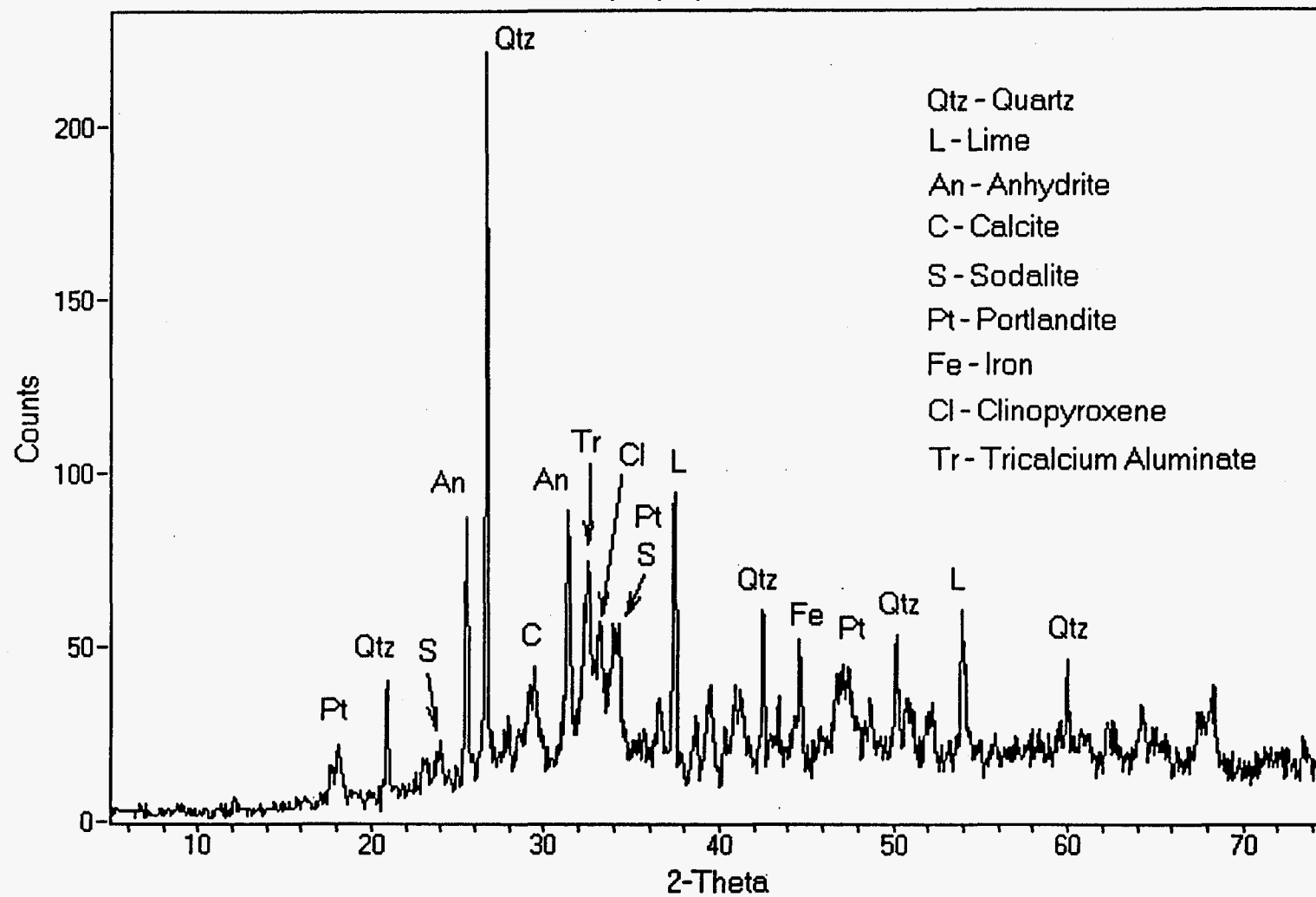
Scan: 5-75/02/ .5/#3501, Anode: CU



ID: Fly Ash - SNCR

File: 18.DIF

Scan: 5-75/.02/.5/#3501, Anode: CU



APPENDIX C
COMPLETE LEACHING RESULTS

Composite ash Non-SNCR SGLPs

	<u>18hr #1</u>	<u>18hr#2</u>	<u>18hr#3</u>	<u>18hr Avg</u>	<u>18hr Std. Deviation</u>
As	<0.004	<0.004	<0.004	<0.004	
Ba	0.27	0.27	0.25	0.26	0.0094
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.22	0.20	0.21	0.21	0.0082
Pb	0.0027	0.0020	0.0020	0.0022	0.0003
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	0.035	0.038	0.036	0.036	0.0012
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.17	0.17	0.16	0.17	0.0047
Ni	<0.07	<0.07	<0.07	<0.07	
V	<0.04	<0.04	<0.04	<0.04	
Zn	<0.03	<0.03	<0.03	<0.03	
Al	<0.7	<0.7	<0.7	<0.7	
Ca	1080	1160	1150	1130	36
Fe	<0.08	<0.08	<0.08	<0.08	
Mg	<0.03	<0.03	<0.03	<0.03	
Mn	<0.01	<0.01	<0.01	<0.01	
P	<0.3	<0.3	<0.3	<0.3	
K	6.8	6.7	6.9	6.8	0.08
Si	0.55	0.52	0.55	0.36	0.25
Na	4.76	4.41	4.47	4.55	0.15
Sr	23.6	23.6	24.5	23.9	0.15
Ti	<0.02	<0.02	<0.02	<0.02	
S	340	371	351	354	13

all measurements in mg/L

Composite ash Non-SNCR SGLPs

	<u>30 day #1</u>	<u>30 day #2</u>	<u>30 day #3</u>	<u>30 day Avg</u>	<u>30 day Std. Deviation</u>
As	<0.004	<0.004	<0.004	<0.004	
Ba	<0.1	<0.1	<0.1	<0.1	
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.43	0.44	0.44	0.44	0.0047
Pb	<0.0020	<0.0020	0.0039	0.0013	0.0018
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	0.032	0.033	0.033	0.033	0.0005
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.27	0.26	0.28	0.27	0.0082
Ni	<0.07	<0.07	<0.07	<0.07	
V	0.20	0.22	0.20	0.21	0.0094
Zn	<0.03	<0.03	<0.03	<0.03	

all measurements in mg/L

Composite ash Non-SNCR SGLPs

	<u>60 day #1</u>	<u>60 day #2</u>	<u>60 day #3</u>	<u>60 day Avg</u>	<u>60 day Std. Deviation</u>
As	<0.004	<0.004	<0.004	<0.004	
Ba	<0.1	<0.1	<0.1	<0.1	
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.45	0.47	0.44	0.45	0.0125
Pb	0.0072	<0.002	0.0039	0.0037	0.0029
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	0.022	0.024	0.023	0.023	0.0008
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.27	0.28	0.27	0.27	0.0047
Ni	<0.07	<0.07	<0.07	<0.07	
V	0.26	0.26	0.26	0.26	0.0000
Zn	<0.03	<0.03	<0.03	<0.03	
Al	1.2	1.2	1.3	1.2	0.05
Ca	214	217	203	211	6
Fe	<0.08	<0.08	<0.08	<0.08	
Mg	0.14	0.14	0.13	0.14	0.005
Mn	<0.01	<0.01	<0.01	<0.01	
P	<0.3	<0.3	<0.3	<0.3	
K	67	69	67	68	1
Si	9.60	9.29	11.10	10.00	0.79
Na	27.9	27.8	26.9	27.5	0.45
Sr	12.8	12.8	12.1	12.6	0.33
Ti	<0.2	<0.2	<0.2	<0.2	
S	199	196	188	194	5

all measurements in mg/L

Composite ash SNCR SGLPs

	<u>18hr #1</u>	<u>18hr#2</u>	<u>18hr#3</u>	<u>18hr Avg</u>	<u>18hr Std. Deviation</u>
As	<0.004	<0.004	<0.004	<0.004	
Ba	1.2	1.2	1.1	1.2	0.0471
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.084	0.079	0.093	0.085	0.0058
Pb	<0.0020	<0.0020	0.0057	0.0019	0.0027
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	<0.008	<0.008	<0.008	<0.008	
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.059	0.060	0.059	0.059	0.0005
Ni	<0.07	<0.07	<0.07	<0.07	
V	<0.04	<0.04	<0.04	<0.04	
Zn	<0.03	<0.03	<0.03	<0.03	
Al	<0.7	<0.7	<0.7	<0.7	
Ca	1110	1080	1080	1090	14
Fe	<0.08	<0.08	<0.08	<0.08	
Mg	<0.03	<0.03	<0.03	<0.03	
Mn	<0.01	<0.01	<0.01	<0.01	
P	<0.3	<0.3	<0.3	<0.3	
K	16	16	16	16	0.00
Si	<0.5	<0.5	<0.5	<0.5	
Na	3.94	3.80	3.84	3.86	0.06
Sr	23.5	23.4	24.3	23.7	0.40
Ti	<0.02	<0.02	<0.02	<0.02	
S	66.1	61.3	64.6	64.0	2.00

all measurements in mg/L

Composite ash SNCR SGLPs

	<u>30 day #1</u>	<u>30 day #2</u>	<u>30 day #3</u>	<u>30 day Avg</u>	<u>30 day Std. Deviation</u>
As	<0.004	<0.004	<0.004	<0.004	
Ba	6.60	6.44	6.28	6.44	0.1306
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.0093	0.0090	0.0101	0.0095	0.0005
Pb	<0.0020	<0.0020	0.0030	0.0010	0.0014
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	<0.008	<0.008	<0.008	<0.008	
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.0204	0.0200	0.0247	0.0217	0.0021
Ni	<0.07	<0.07	<0.07	<0.07	
V	<0.04	<0.04	<0.04	<0.04	
Zn	<0.03	<0.03	<0.03	<0.03	

all measurements in mg/L

Composite ash SNCR SGLPs

	60 day #1	60 day #2	60 day #3	60 day Avg	60 day Std. Deviation
As	<0.004	<0.004	<0.004	<0.004	
Ba	6.66	6.76	6.42	6.61	0.14
Cd	<0.0003	<0.0003	<0.0003	<0.0003	
Cr	0.022	0.022	0.019	0.021	0.001
Pb	0.0025	0.0021	0.0048	0.0031	0.0012
Hg	<0.0001	<0.0001	<0.0001	<0.0001	
Se	<0.008	<0.008	<0.008	<0.008	
Ag	<0.0003	<0.0003	<0.0003	<0.0003	
B	<0.2	<0.2	<0.2	<0.2	
Cu	<0.1	<0.1	<0.1	<0.1	
Mo	0.0278	0.0279	0.0246	0.0268	0.0015
Ni	<0.07	<0.07	<0.07	<0.07	
V	<0.04	<0.04	<0.04	<0.04	
Zn	<0.03	<0.03	<0.03	<0.03	
Al	0.91	0.83	0.84	0.86	0.04
Ca	661	671	666	666	4
Fe	<0.08	<0.08	<0.08	<0.08	
Mg	<0.03	<0.03	<0.03	<0.03	
Mn	<0.01	<0.01	<0.01	<0.01	
P	<0.3	<0.3	<0.3	<0.3	
K	187	184	180	184	3
Si	<0.5	<0.5	<0.5	<0.5	
Na	33.2	29.3	28.5	30.3	2.1
Sr	47.4	47.8	47.3	47.5	0.2
Ti	<0.02	<0.02	<0.02	<0.02	
S	0.76	0.87	0.79	0.81	0.05

all measurements in mg/L