

Contract No. DE-AC21-94MC31160--32

**PARTICULATE HOT GAS STREAM CLEANUP  
TECHNICAL ISSUES**

**QUARTERLY REPORT**

January 1999 - March 1999

Prepared for

UNITED STATES DEPARTMENT OF ENERGY  
Federal Energy Technology Center - Morgantown  
Post Office Box 880, 3610 Collins Ferry Road  
Morgantown, West Virginia 26505

# **PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES**

## **QUARTERLY REPORT**

January 1999 - March 1999

SRI-ENV-99-8484-Q18

June 1, 1999

SOUTHERN RESEARCH INSTITUTE  
2000 NINTH AVENUE SOUTH  
Post Office Box 55305  
BIRMINGHAM, ALABAMA 35255-5305  
Principal Investigator: D. H. Pontius

for

UNITED STATES DEPARTMENT OF ENERGY  
Federal Energy Technology Center - Morgantown  
Post Office Box 880, 3610 Collins Ferry Road  
Morgantown, West Virginia 26505

Contract No. DE-AC21-94MC31160  
Project Manager: Thomas P. Dorchak

## Disclaimer

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

Available to the public from the National Technical Information Service, U.S. Department of Commerce, 5285 Port Royal Road, Springfield, VA 22161; phone orders accepted at (703) 487-4650.

## Abstract

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. The analyses of hot gas stream cleanup (HGCU) ashes and descriptions of filter performance studied under Task 1 of this contract are designed to address problems with filter operation that are apparently linked to characteristics of the collected ash. This report includes summaries of analyses performed on particulate samples from Sierra Pacific Power Company's Piñon Pine Power Project. This report also reviews the status of the HGCU data bank of ash and char characteristics, and plans for enhancing the data bank with interactive querying of measured particulate properties. Task 1 plans for the remainder of the project include completion and delivery of the HGCU data bank.

Task 2 of this project concerns the testing and failure analyses of new and used filter elements and filter materials. Task 2 work during the past quarter included preliminary testing of two materials. One material tested was the soft candle filter manufactured by CGC and supplied by ABB. The other material was N610/mullite manufactured by Albany International (AIT).

## TABLE OF CONTENTS

	<u>PAGE</u>
EXECUTIVE SUMMARY .....	1
INTRODUCTION .....	2
OBJECTIVES .....	3
TASK 1 ASSESSMENT OF ASH CHARACTERISTICS .....	4
PIÑON PINE PARTICULATE SAMPLE ANALYSES .....	4
ASH DATA BANK DEVELOPMENT .....	19
FILTER CAKE ENCAPSULATION .....	24
TASK 2 FILTER MATERIAL CHARACTERIZATION .....	26
FUTURE WORK .....	27

## EXECUTIVE SUMMARY

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. The analyses of Hot Gas Stream Cleanup (HGCU) ashes and descriptions of filter performance studied under this contract are designed to address problems with filter operation that are apparently linked to characteristics of the collected ash. Task 1 is designed to generate a data bank of the key characteristics of ashes collected from operating advanced particle filters (APFs) and to relate these ash properties to the operation and performance of these filters and their components. APF operations have also been limited by the strength and durability of the ceramic materials that have served as barrier filters for the capture of entrained HGCU ashes. Task 2 concerns testing and failure analyses of ceramic filter elements currently used in operating APFs and the characterization and evaluation of new ceramic materials.

Task 1 research activities during the past quarter included analyses of particulate samples from Sierra Pacific Power Company's Piñon Pine Power Project. A draft version of the HGCU data bank of ash and char characteristics was submitted to DOE/FETC for review. This report reviews the status of this data bank, and plans for enhancing it with interactive querying of measured particulate properties. Other work performed during the past quarter included hardening of the filter cake on the surface of a filter element collected from the PSDF in January 1999, followed by impregnation of the filter cake with low-viscosity epoxy.

Task 2 work during the past quarter included preliminary testing of two materials. One material tested was the soft candle filter manufactured by CGC and supplied by ABB. The other material was N610/mullite manufactured by Albany International (AIT).

## INTRODUCTION

This quarterly report describes technical activities performed under Contract No. DE-AC21-94MC31160. Task 1 of this contract concerns analyses of HGCU ashes and descriptions of filter performance that are designed to address problems with filter operation linked to characteristics of the collected ash. Much of the work planned for Task 1 builds directly on work performed under a prior contract (No. DE-AC21-89MC26239) with the Department of Energy's Federal Energy Technology Center in Morgantown, WV (DOE/FETC-MGN). Task 2 of this contract includes characterization of new and used filter elements. Some of the problems observed at PFBC facilities include excessive filtering pressure drop, the formation of large, tenacious ash deposits within the filter vessel, and bent or broken candle filter elements. These problems have been attributed to ash characteristics, durability of the ceramic filter elements, and specific limitations of the filter design. In addition to the problems related to the characteristics of PFBC ashes, laboratory characterizations of gasifier and carbonizer particulates have shown that these ashes and chars also have characteristics that might negatively affect filtration. Specifically, gasifier particulates may form filter cakes that accumulate in thickness quite rapidly and also may reentrain following cleaning pulses. Recent measurements suggest that these gasifier char filter cakes may be compressed by the filtering pressure drop across their structure, thereby increasing their strength and the pressure losses incurred during filtration.

To identify which particulate characteristics can lead to problems with filtration, 384 particulate samples from fourteen facilities involved in FETC's HGCU program have been assembled. Three samples from gasification studies being carried out by Herman Research Pty. Ltd. (HRL) of Melbourne, Australia have also been included in the data bank. The most recent facility included in the data bank is the Westinghouse filter at Sierra Pacific Power Company's Piñon Pine Power Project. Many of the samples in the data bank have been analyzed with a variety of laboratory tests. Physical attributes of the particles that have been examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. A range of chemical analyses of these samples, as well as characterizations of agglomerates of particles removed from filter vessels at Tidd, Karhula and Foster Wheeler's pilot-scale combustion facility located in Livingston, New Jersey have also been performed. The data obtained in these studies are being assembled into an interactive data bank which will help the manufacturers and operators of high-temperature barrier filters tailor their designs and operations to the specific characteristics of the particulate materials they are collecting.

Two soft candles manufactured by CGC were supplied by ABB and tested under Task 2. One candle was tested as-manufactured and one after exposure in the ABB PTF. Conditions of exposure (temperature, time, chemical, etc.) were not supplied to SRI. Information from ABB indicated that the chemical composition of the material was 43.7%  $\text{Al}_2\text{O}_3$  and 56.3%  $\text{SiO}_2$  and the porosity was 89%. The average bulk density measured at SRI on eight virgin specimen blanks was  $0.42 \text{ gr/cm}^3$ . One virgin N610/mullite candle manufactured by AIT was tested. This candle was manufactured by a layer-to-layer interlock method which AIT hopes will solve problems with loss of through thickness fibers experienced at Karhula. The average bulk density measured at SRI was  $1.73 \text{ gr/cm}^3$ . One of these candles is in operation

at the Southern Company Services PSDF and will be tested at SRI upon its removal from operation.

## OBJECTIVES

Task 1 of this project is explicitly designed to address aspects of filter operation that are linked to the characteristics of the collected particles. This task has two primary objectives. The first is the generation of an interactive computerized data bank of the key characteristics of HGCU ashes collected from operating high-temperature, high-pressure, particle filters. The data bank is structured to identify, when possible, relationships between HGCU particulate properties and the operation and performance of these filters. Construction of the data bank is intended to help manufacturers and operators of high-temperature barrier filters tailor process design and operation to the specific characteristics of the particulate materials they are collecting. The second objective is to relate these measured properties and the contents of the data bank to the operation and performance of the advanced particle filters and filter components. The first objective includes formatting the data bank and collecting, analyzing, and maintaining particulate samples from operating HGCU facilities. The second objective of this task involves the collection of operating histories from advanced particle filters, correlating these histories with sample characteristics, interpreting these correlations, and communicating results in the various venues prescribed by DOE/FETC-MGN.

The objectives of the Task 2 test program at Southern Research are as follows:

- Provide material characterization to develop an understanding of the physical, mechanical, and thermal behavior of hot gas filter materials.
- Develop a material property data base from which the behavior of materials in the hot gas cleanup environment may be predicted.
- Perform testing and analysis of filter elements after exposure to actual operating conditions to determine the effects of the thermal and chemical environments in hot gas filtration on material properties.
- Explore the glass-like nature of the matrix material.

## TASK 1 ASSESSMENT OF ASH CHARACTERISTICS

Task 1 research activities during the past quarter included analyses of particulate samples from Sierra Pacific Power Company's Piñon Pine Power Project. A draft version of the HGCU data bank of ash and char characteristics was submitted to DOE/FETC for review. This report reviews the status of this data bank, and plans for enhancing it with interactive querying of measured particulate properties. Other work performed during the past quarter included hardening of the filter cake on the surface of a filter element collected from the PSDF in January 1999, followed by impregnation of the filter cake with low-viscosity epoxy.

### PIÑON PINE PARTICULATE SAMPLE ANALYSES

Leo Leighton of Sierra Pacific Power Company provided two particulate samples collected from the Piñon Pine facility in January, 1999. The first sample, ID# 4349, was collected from the system of hoppers that receive the particulate matter collected in the Westinghouse filter vessel. The second sample, ID# 4350, was collected from the surface of the filter elements during a filter inspection on January 26, 1999.

Figures 1 through 8 present representative micrographs of these two samples. Each sample comprises a wide size distribution of irregularly-shaped particles. The measured size distributions of these two samples are compared in Figure 9, and this comparison is the basis for calculation of the degree of inertial collection (settling) of relatively coarse particles in the filter vessel (Figure 10). In this calculation, a scaling factor of 0.80 was applied to the filter cake particulate size distribution to cause the finest portion of the two distributions shown in Figure 9 to coincide. Consequently, this analysis assumes 20 % of the entrained mass entering the vessel settles into the hopper without ever reaching the filter cake.

Mr. Leighton described the filter cake sample as containing nodules prior to shipment. Although most of the nodules were apparently broken up during shipping to the Southern Research Institute laboratory, the filter cake sample still contained some small, thin nodules (0.01 to 0.02 g, about 1 mm thick) that were characterized with porosity and SEM analyses. The physical analyses performed on these nodules and on loose bulk particulate matter from these two samples are summarized in Table 1.

It is important to note the measured porosity of the filter cake nodules from sample # 4350. These values (63 to 68 %) are significantly lower than the uncompacted bulk porosity value of 85 %. Because it is believed that filter cakes initially form with a porosity near the uncompacted bulk porosity value, it must be assumed that the portion of the filter cake represented by these small nodules has been compacted during filtration. The most likely mechanism for this compaction is the force of the cumulative filtering pressure applied across the filter cake. Although it may only be a small, thin (1 mm) portion of the cake that is compressed to this degree, the effect of this compression on overall filtering pressure drop is quite significant. Filter cake compaction from an assumed initial value of 85 % to a final value of 63 % would cause the filtering pressure drop to increase by a factor of more than 20. Although the values for specific gas-flow resistance shown in Table 1 were measured for the hopper char, the magnitude of pressure drop increase would be the same for filter cake char. There was insufficient sample to perform the measurement of specific gas-flow resistance on

the filter cake char sample, but it is reasonable to assume that it would be about the same as the value measured for the hopper char sample (at an equivalent cake porosity). This assumption is based on the fact that although the median diameter of the filter cake char sample is somewhat smaller than that of the hopper char sample, the specific surface areas of these two samples are similar. The drag-equivalent diameter of the hopper char sample is not so small as to indicate excessive pressure drop for uncompacted filter cakes. However, at a cake porosity equal to that of the filter cake nodules that were analyzed, pressure drops would become excessive.

The chemical compositions of the two samples from Piñon Pine are given in Table 2. These data demonstrate the similarity between these samples. Each sample has a high LOI value, with a large proportion of calcium, silicon, sulfur, aluminum, and iron in the ignited sample residue. In both samples, the sulfur has apparently been converted from its reduced state to sulfate. This conversion is to be expected because of the nearly inevitable exposure of the char samples to air as they are collected from the gasifier facility, and as they are subsequently handled during analysis. (The values of  $\text{SO}_3$  measured in the ignited samples are comparable with the soluble  $\text{SO}_4^{\pm}$  values in the as-received samples. When the  $\text{SO}_3$  values are corrected for LOI, the sulfur content of hopper char is 3.3 %, and the sulfur content of the filter cake char is 3.1 %. These values agree with the sulfate values presented in Table 2.)

SEM analyses were performed on a fresh fracture surface of one of the filter cake nodules. The micrographs and EDX spectra produced in these analyses are presented in Figures 11 through 20. (In addition to the micrographs shown in this report, a stereographic SEM image was made of this fracture surface to demonstrate its structure. Because proper viewing of these stereographic images requires special equipment, these images are not included in this report. The appearance of this micrograph shows irregularly-shaped particles with a wide range of sizes. A large proportion of these appear to be submicron in size. The apparent packing density of the particles in the nodule qualitatively agrees with the measured porosity of the nodule.) . Because of the nature of EDX measurements, the locations of the EDX measurements (labeled P1 through P7 in Figure 13) do not guarantee that the elemental distributions shown in Figures 14 through 20 represent the single particle under the labels. The elemental composition of nearby or attached particle structures are likely to influence the overall EDX spectra. The EDX spectra indicate that the larger particles (locations P3 and P4 in Figure 13) in the filter cake char sample are predominately carbon (Figures 16 and 17). The one particle in Figure 13 that appeared spherical (location P1) exhibited a high proportion of silicon, aluminum, and calcium (Figure 14). Calcium and carbon were concentrated (Figures 19 and 20) in two other relatively small particles (locations P6 and P7). At locations P2 and P5, the spectra in Figures 15 and 18 indicate that the particles at these locations are high in zinc and titanium, presumably as a result of carryover of sorbent material from the absorber. None of the larger particles that were examined contained a high proportion of either zinc or titanium.

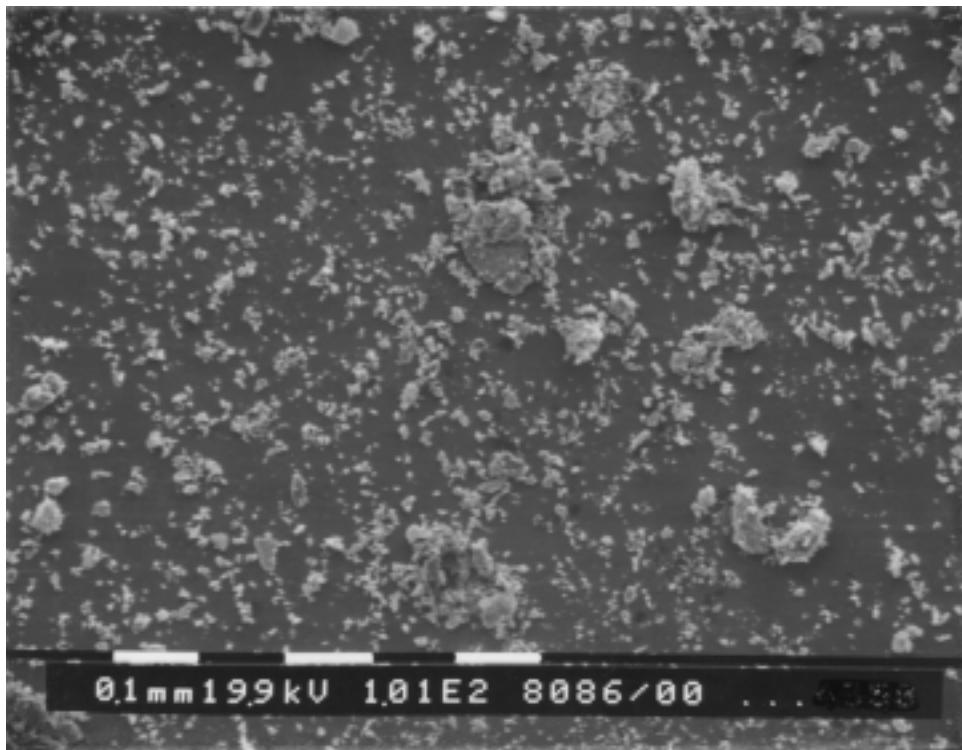


Figure 1. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4349) collected in January 1999 from the filter hopper. The black and white bars at the bottom of the micrograph represent lengths of 100  $\mu\text{m}$ .

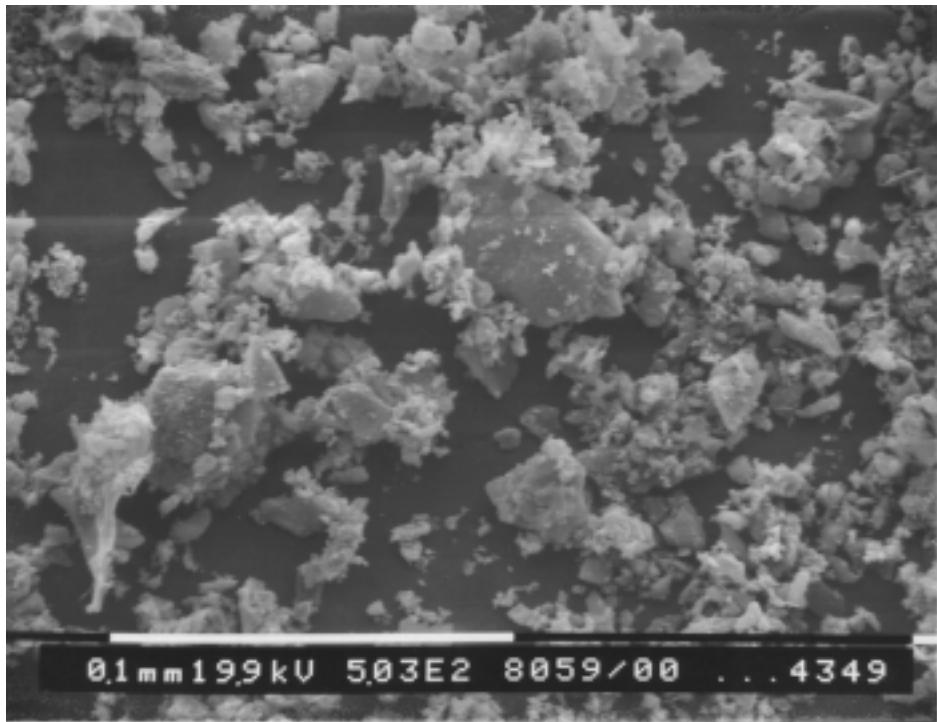


Figure 2. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4349) collected in January 1999 from the filter hopper. The black and white bars at the bottom of the micrograph represent lengths of 100  $\mu\text{m}$ .

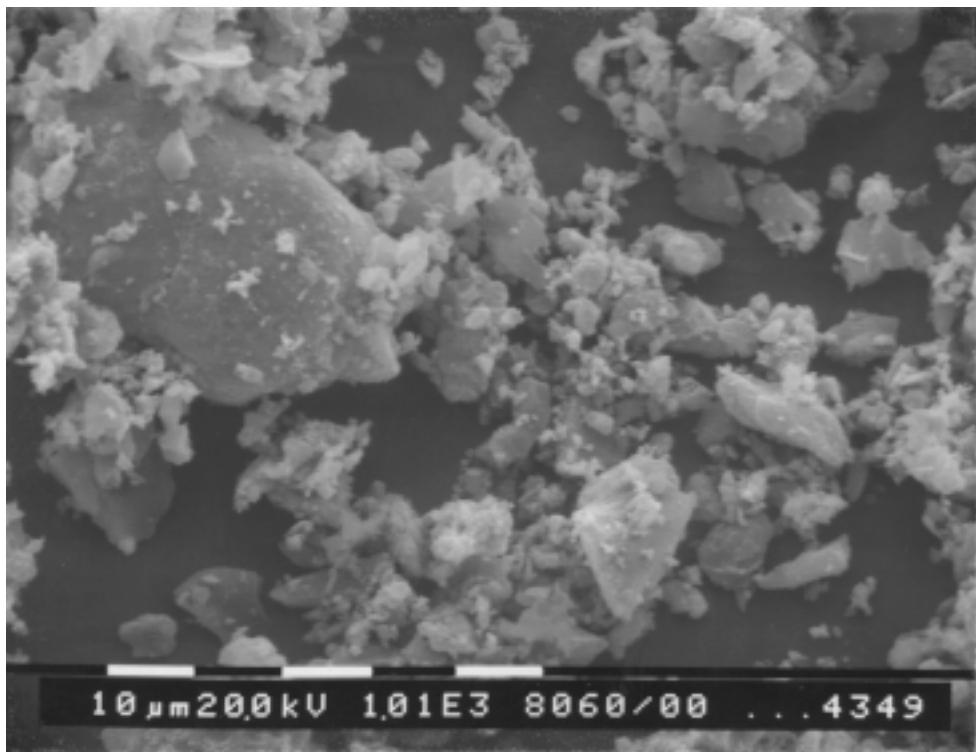


Figure 3. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4349) collected in January 1999 from the filter hopper. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu$ m.

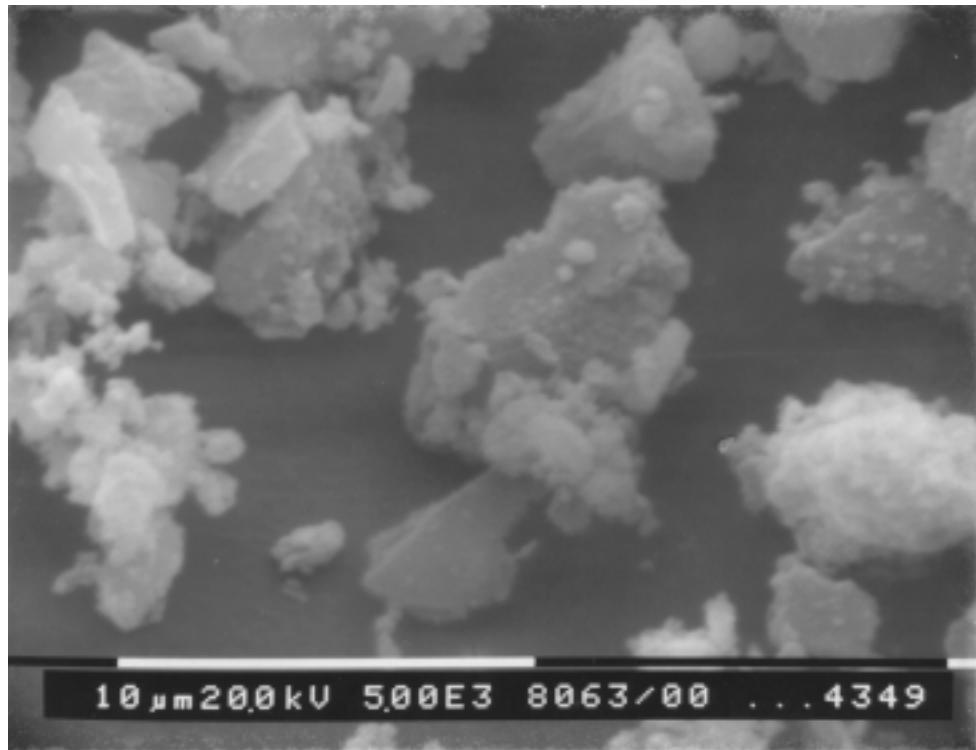


Figure 4. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4349) collected in January 1999 from the filter hopper. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu$ m.

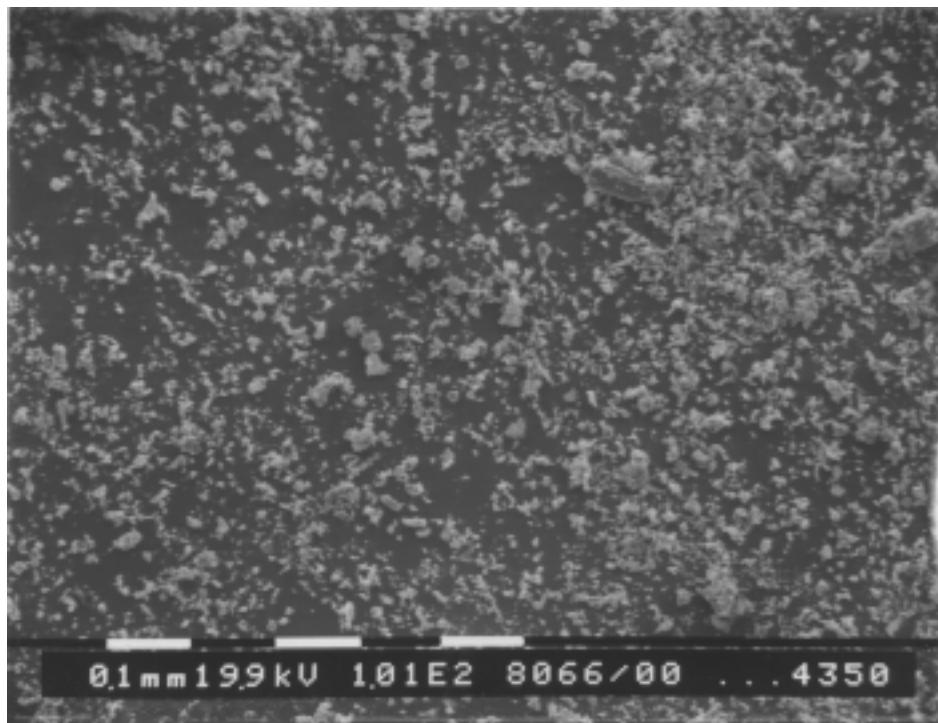


Figure 5. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4350) collected in January 1999 as part of the filter cake. The black and white bars at the bottom of the micrograph represent lengths of 100  $\mu\text{m}$ .

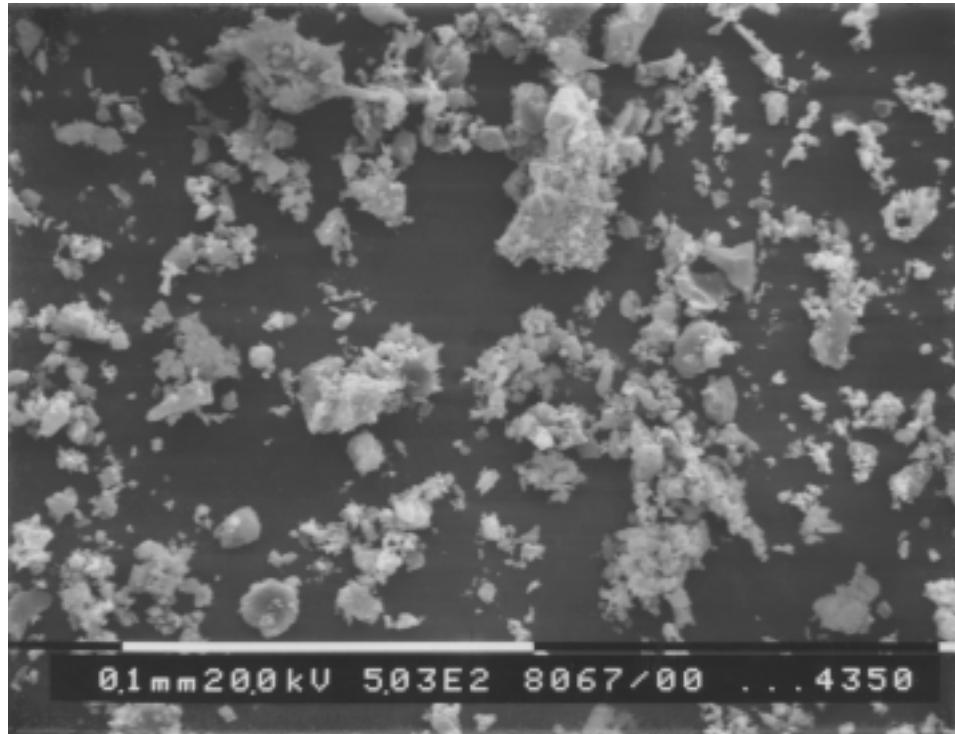


Figure 6. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4350) collected in January 1999 as part of the filter cake. The black and white bars at the bottom of the micrograph represent lengths of 100  $\mu\text{m}$ .

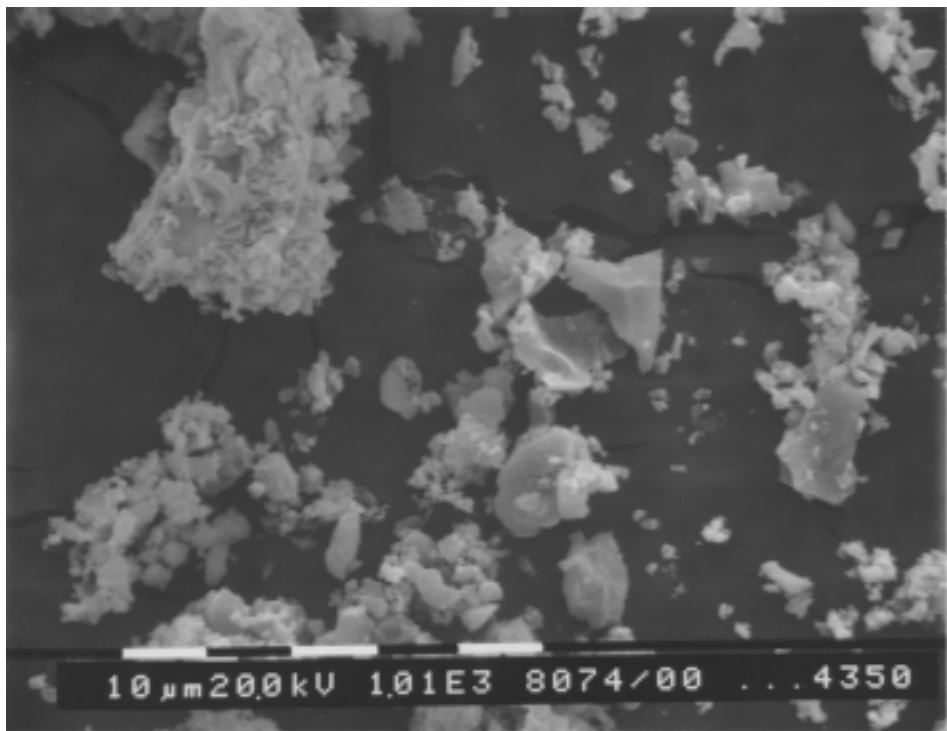


Figure 7. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4350) collected in January 1999 as part of the filter cake. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu\text{m}$ .

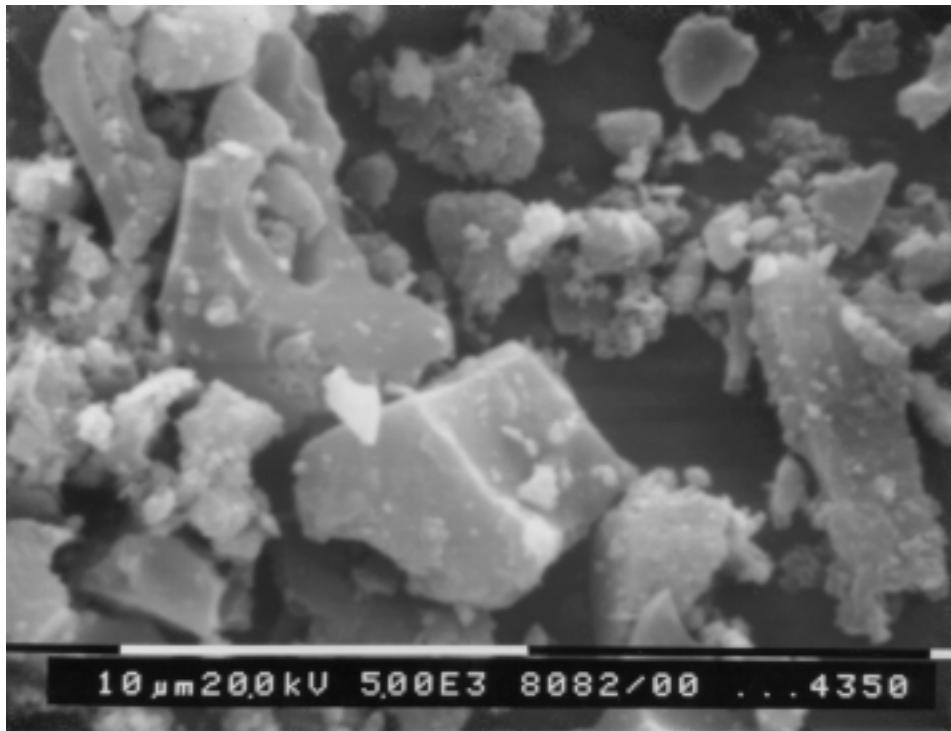


Figure 8. Representative scanning electron micrograph of Piñon Pine gasifier char (ID # 4350) collected in January 1999 as part of the filter cake. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu\text{m}$ .

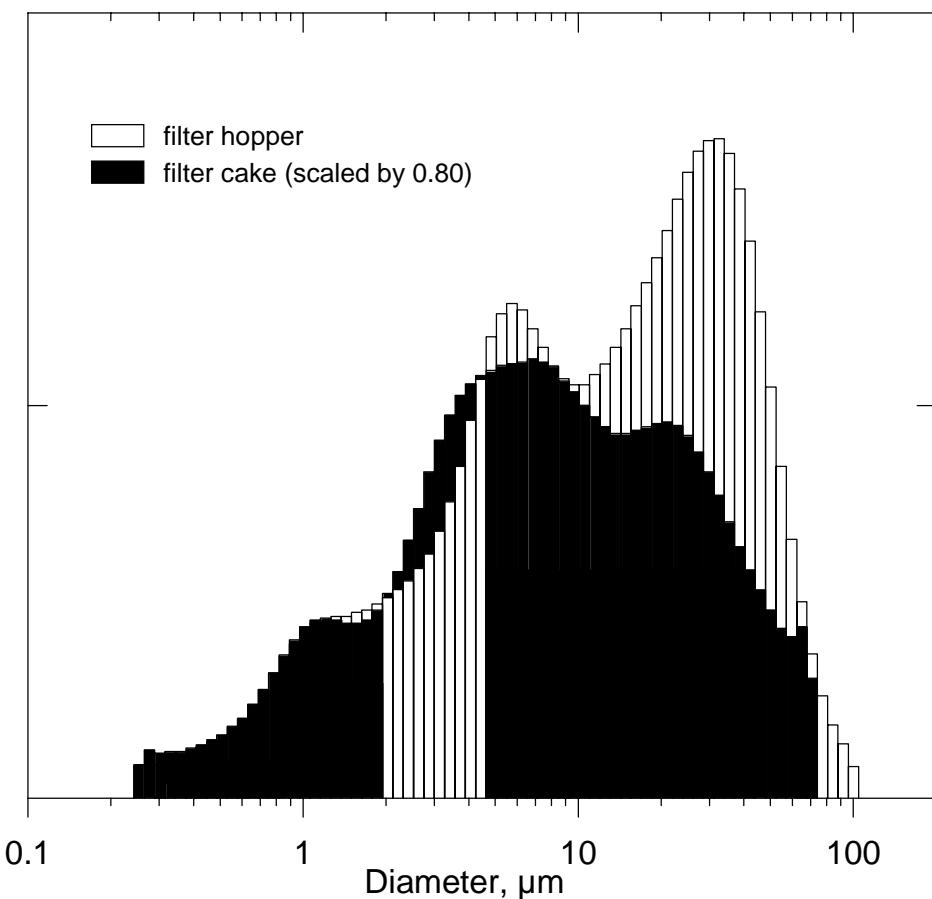


Figure 9. Differential size distribution data measured for Piñon Pine particulate matter collected in January 1999. The size distribution of the material collected in the filter hopper (ID # 4349) is overlaid with the size distribution of particulate matter collected as part of the filter cake (ID# 4350). A scaling factor of 0.80 was applied to the filter cake particulate size distribution to cause the finest portion of these two distributions to coincide.

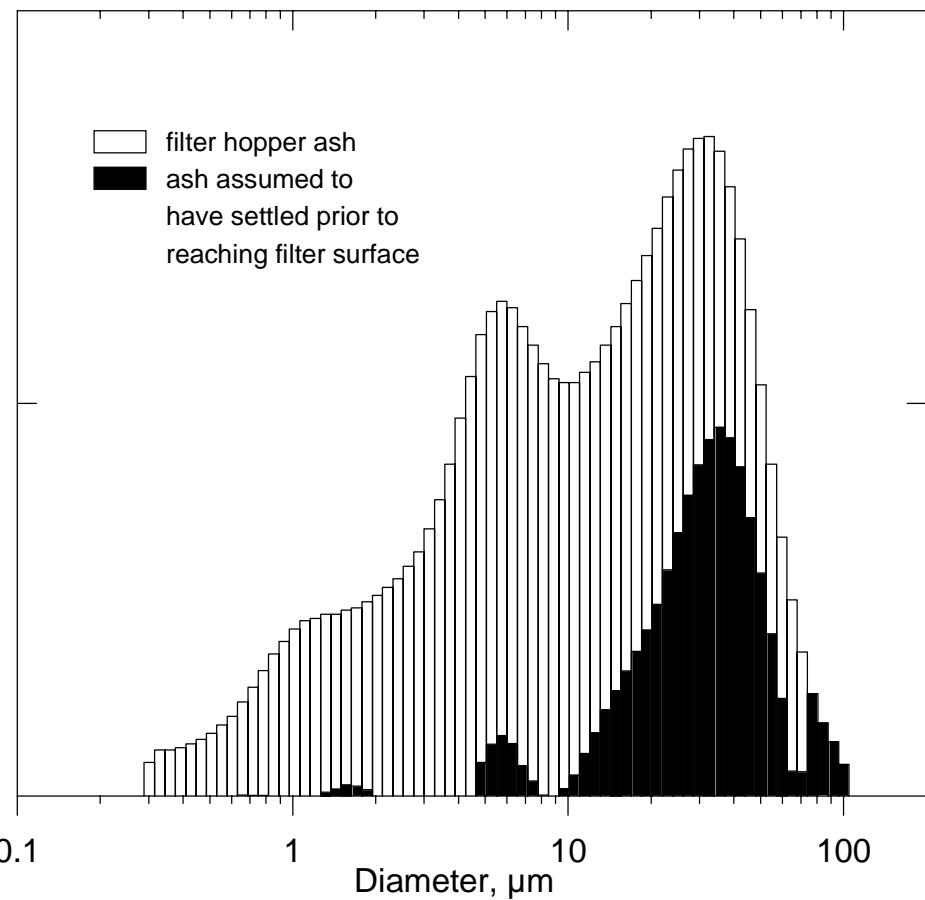


Figure 10. Differential size distribution data measured for Piñon Pine particulate matter collected in January 1999. The size distribution of the filter hopper char is overlaid with the size distribution of particulate matter assumed to have settled into the hopper without ever reaching the surface of the filter cake.

Table 1  
Physical Analyses of Piñon Pine Samples Generated in January 1999

quantity	sample location	hopper	filter cake
mass median diameter, $\mu\text{m}$		12	7.4
specific surface area, $\text{m}^2/\text{g}$		35.1	28.2
filter cake nodule porosity, %		--	63 to 68
uncompacted bulk porosity, %		78	85
drag-equivalent diameter, $\mu\text{m}$		1.91	--
true particle density, $\text{g}/\text{cm}^3$		2.19	--
specific gas-flow resistance, in $\text{H}_2\text{O}\cdot\text{min}\cdot\text{ft}/\text{lb}$ (at cake porosity of 85 %)		3.5	--
specific gas-flow resistance, in $\text{H}_2\text{O}\cdot\text{min}\cdot\text{ft}/\text{lb}$ (at cake porosity of 78 %)		12	--
specific gas-flow resistance, in $\text{H}_2\text{O}\cdot\text{min}\cdot\text{ft}/\text{lb}$ (at cake porosity of 68 %)		43	--
specific gas-flow resistance, in $\text{H}_2\text{O}\cdot\text{min}\cdot\text{ft}/\text{lb}$ (at cake porosity of 63 %)		72	--

Table 2  
Chemical Analyses of Piñon Pine Samples Generated in January 1999, % wt.\*

Constituent	hopper	filter cake
$\text{Li}_2\text{O}$	0.02	0.02
$\text{Na}_2\text{O}$	1.2	1.2
$\text{K}_2\text{O}$	0.38	0.39
$\text{MgO}$	1.3	1.3
$\text{CaO}$	46.2	47.5
$\text{Fe}_2\text{O}_3$	4.8	4.9
$\text{Al}_2\text{O}_3$	11	10.6
$\text{SiO}_2$	20.4	20.1
$\text{TiO}_2$	1.9	1.8
$\text{P}_2\text{O}_5$	0.2	0.2
$\text{SO}_3$	8.9	7.7
LOI	63	59.6
soluble $\text{SO}_4^=$	3.2	2.7

\* all constituents were measured for ignited char  
except for LOI, and soluble  $\text{SO}_4^=$ , which were  
measured on as-received samples

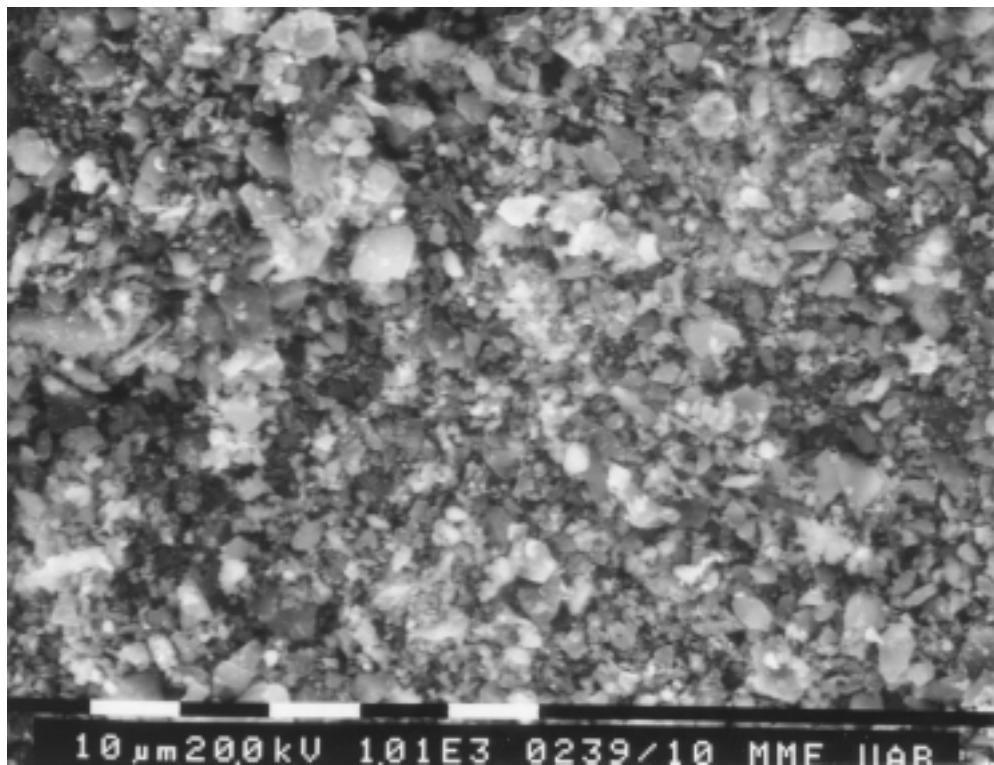


Figure 11. Representative scanning electron micrograph of a fresh fracture surface of a Piñon Pine gasifier char filter cake nodule (ID # 4350) collected in January 1999. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu$ m.

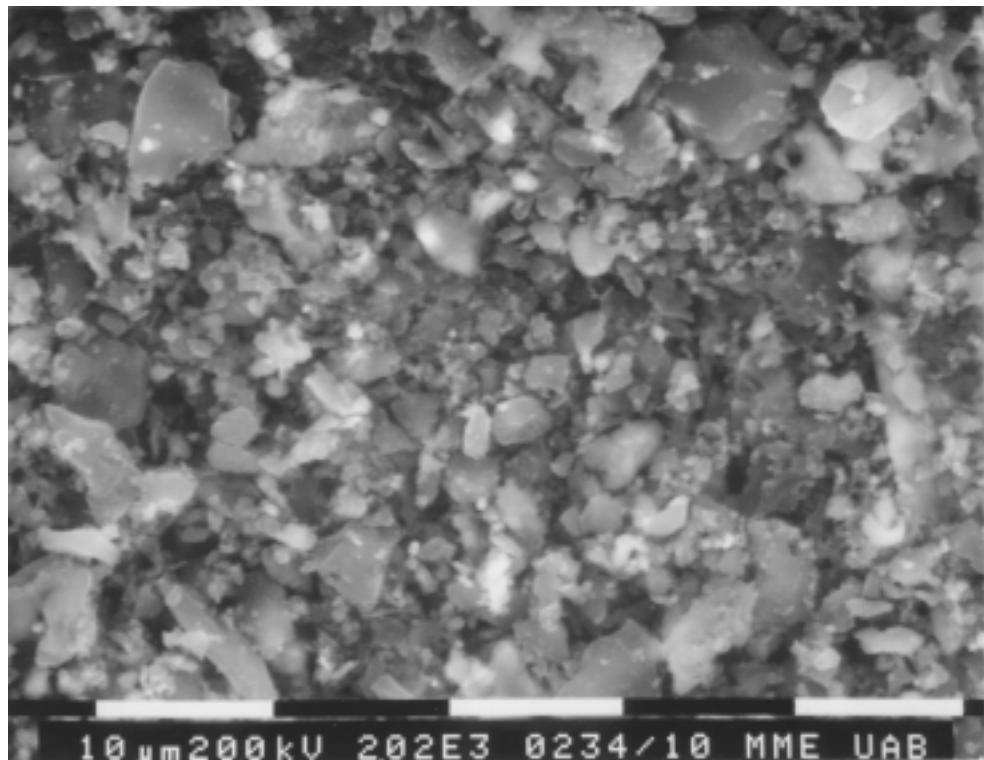


Figure 12. Representative scanning electron micrograph of a fresh fracture surface of a Piñon Pine gasifier char filter cake nodule (ID # 4350) collected in January 1999. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu$ m.

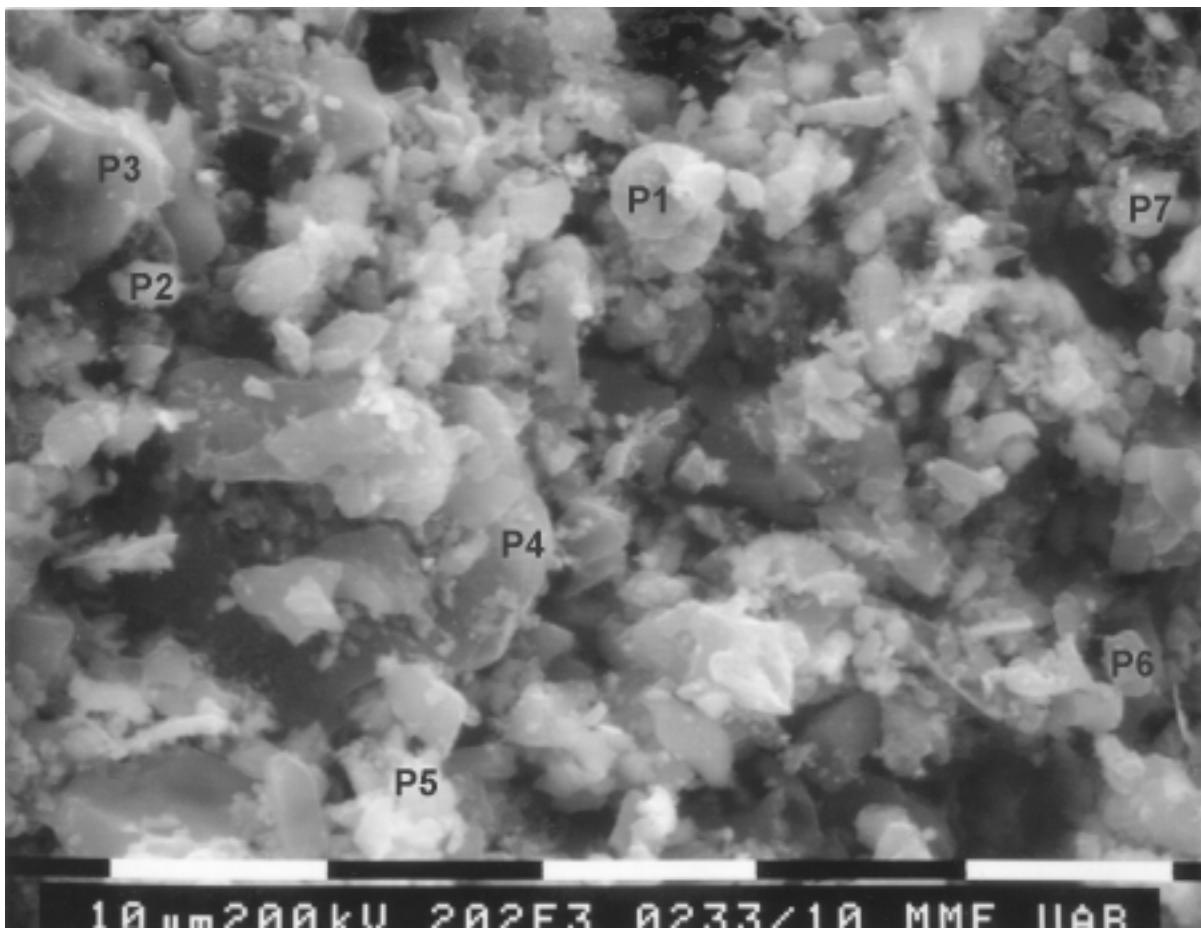


Figure 13. Representative scanning electron micrograph of a fresh fracture surface of a Piñon Pine gasifier char filter cake nodule (ID # 4350) collected in January 1999. The black and white bars at the bottom of the micrograph represent lengths of 10  $\mu\text{m}$ . The locations where X-ray diffraction spectra (Figure 14 through 20) were measured are labeled P1 through P7.

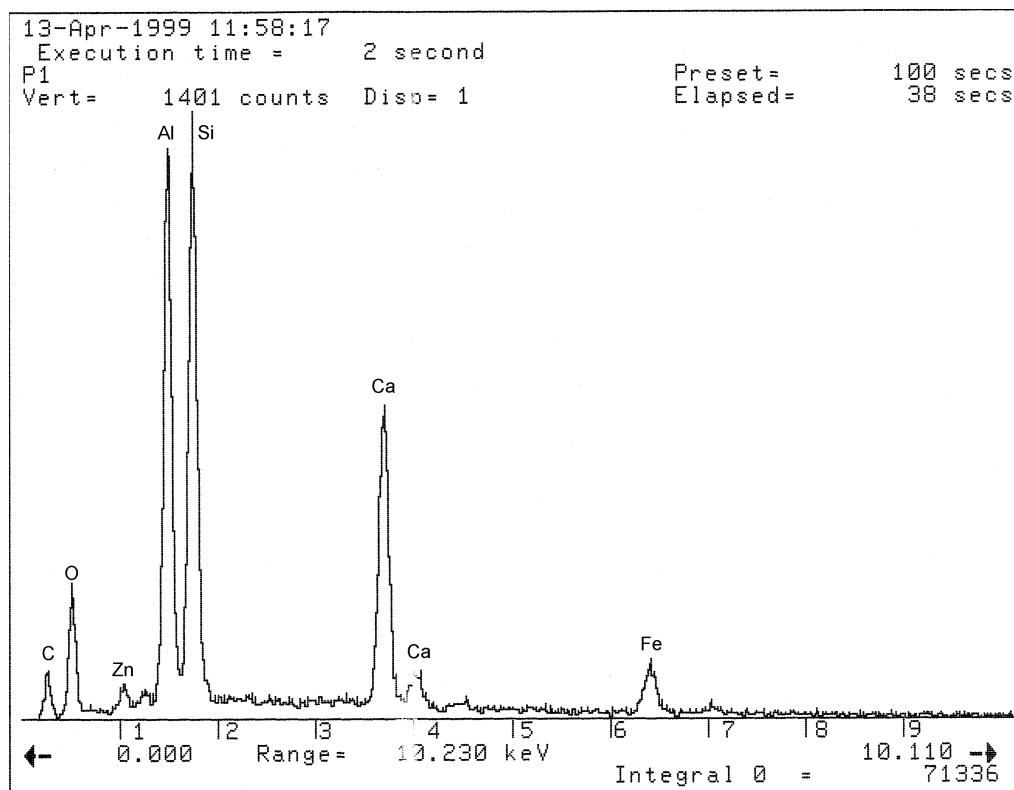


Figure 14. EDX spectrum taken at location P1 in the Piñon Pine filter cake nodule shown in Figure 13.

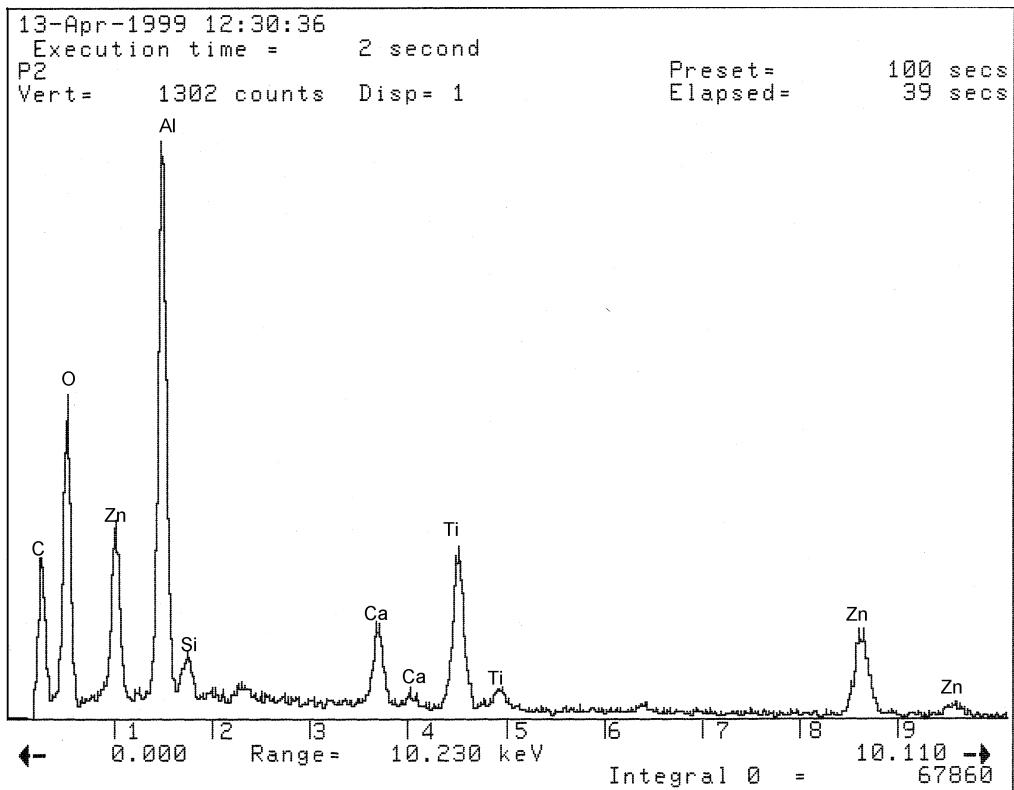


Figure 15. EDX spectrum taken at location P2 in the Piñon Pine filter cake nodule shown in Figure 13.

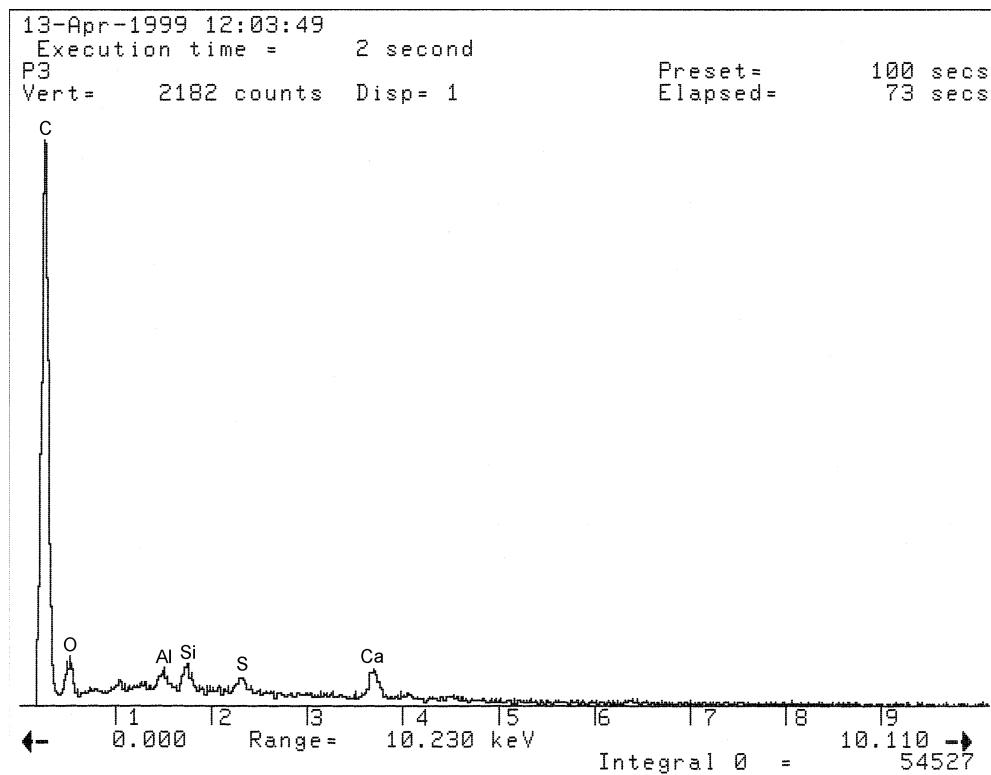


Figure 16. EDX spectrum taken at location P3 in the Piñon Pine filter cake nodule shown in Figure 13.

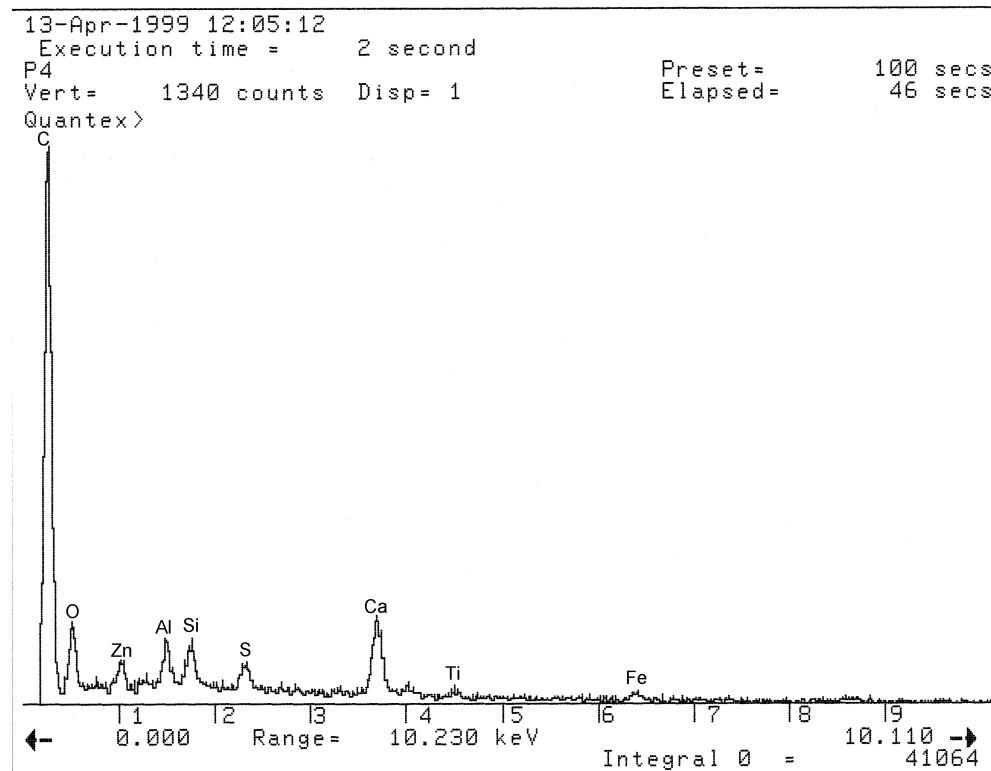


Figure 17. EDX spectrum taken at location P4 in the Piñon Pine filter cake nodule shown in Figure 13.

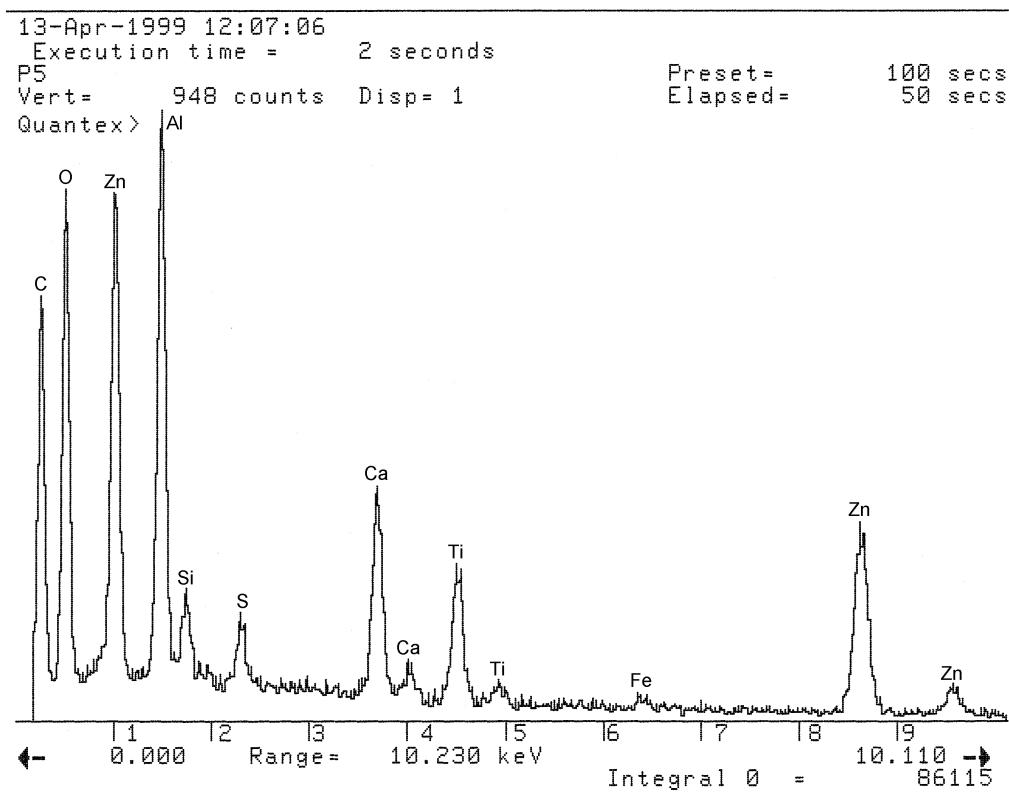


Figure 18. EDX spectrum taken at location P5 in the Piñon Pine filter cake nodule shown in Figure 13.

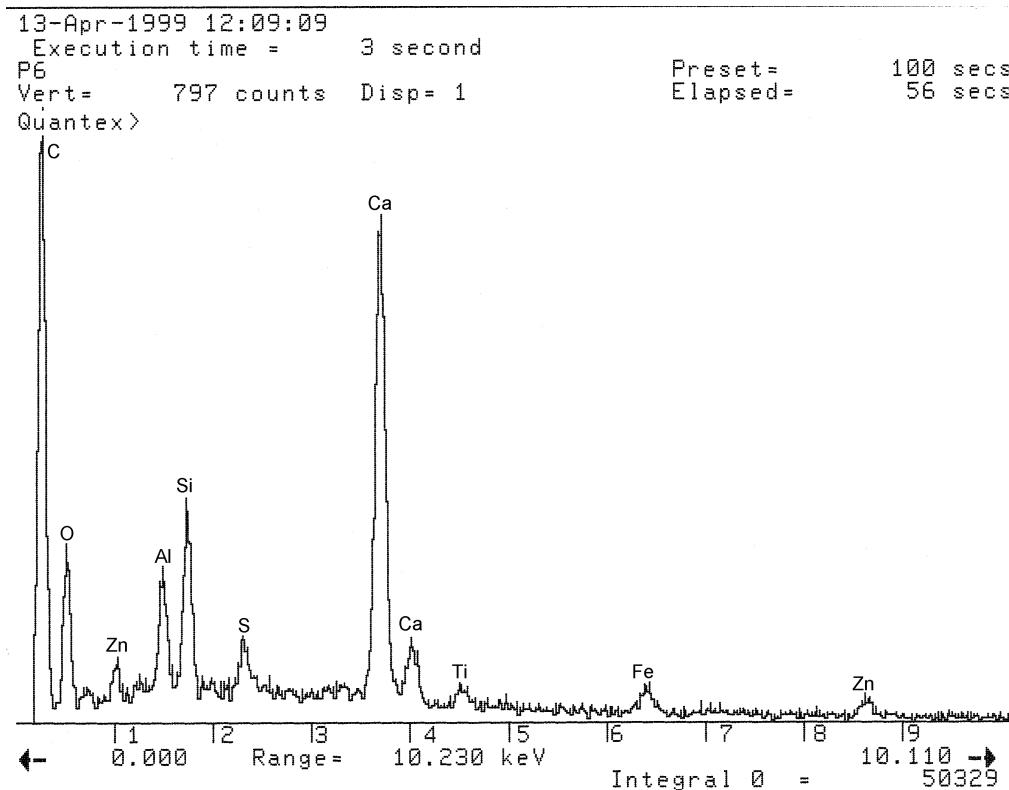


Figure 19. EDX spectrum taken at location P6 in the Piñon Pine filter cake nodule shown in Figure 13.

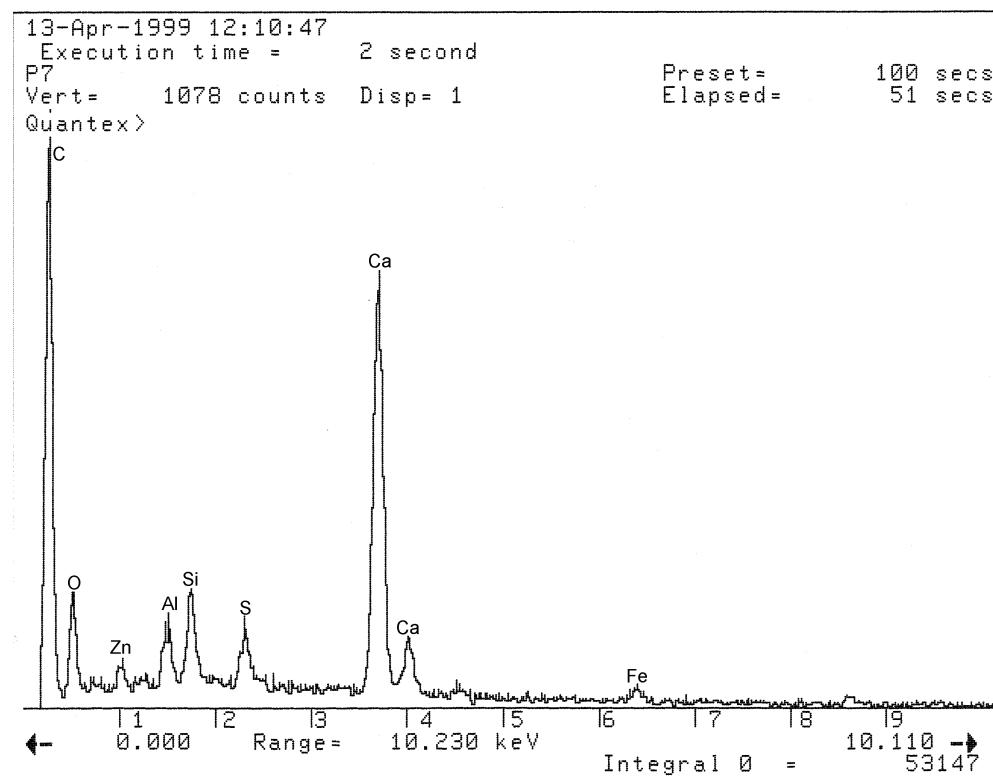


Figure 20. EDX spectrum taken at location P7 in the Piñon Pine filter cake nodule shown in Figure 13.

## ASH DATA BANK DEVELOPMENT

The data bank is currently being modified to include the features described below. Although many of these features have not yet been fully implemented, the final form of the data bank should closely resemble this discussion.

To date, SRI has accumulated 387 HGCU particulate samples and has performed various analyses on over 150 of these samples. The results of all of these analyses are included in the data bank. The data bank accomplishes two principal functions. In addition to archiving the results of laboratory analyses of HGCU samples for interactive access (shown schematically in Figure 21), the data bank is structured to identify relationships between the HGCU particulate properties that have been measured and the performance of the HGCU filters. Upon activating the data bank, the user initially views a title page and then a page that provides some instructions for properly using and interpreting the information and constructing comparisons between data. (On-line help for the various options is available throughout the data bank.) The user is then directed to a screen that both serves as a main navigation screen, and as an invitation for the user to contribute samples and/or data to the data bank. The first of the principal functions of the data bank is accessed through this main navigation screen and allows the user to perform interactive queries on the collection of numerical analysis data that was measured to physically and chemically characterize the HGCU samples. Parameters available to limit the sample population in this user-controlled querying include the HGCU facility where the sample was generated, the conversion process used, the date when the sample was generated, the location in the process stream where the sample was obtained, and type of ash sample (nodular deposits vs. loose bulk particulate samples). During querying, the user assembles the limiting parameters in a stepwise process. As the limits of each parameter are set, the data bank displays the number of samples satisfying the limiting criteria. If the user desires, previously enacted limits on parameters can be removed or restructured. In addition to allowing the user to build custom sets of parameters to limit the data population, the data bank also offers the user several preexisting sets of parameters from which analytical data can be drawn and plotted.

After defining all the parameters that will be used to limit the samples that will be included in the data comparison, the user has three options: outputting the limited population with its associated data set to a Windows clipboard, examining all available data for any specific sample in the population (including scanning electron micrographs, and any specialized analyses performed on the sample), or proceeding to select analytical quantities for a plotted display of the data. The analytical quantities that have been measured and can be plotted are listed in Table 3. When the user selects the analytical quantities that will be plotted, a running summary is provided of the number of samples in the sample population that contain numerical values for the quantities the user has selected. Depending on whether one or two quantities are selected for the data population, this plot will be displayed either as the distribution and range of values measured for a single quantity, or as data pairs for two different quantities. Once data from the limited sample population is selected and graphed on the screen by the data bank, the user has the option of obtaining a hard copy of the plot, or of returning to the data bank for further comparisons.

The second principal function of the data bank is utilized when sufficient operating data, samples, and sample analyses were available for SRI to draw conclusions about system or process behavior. This portion of the data bank is shown schematically in Figure 22. In this case, the data bank includes prepackaged discussions of these conclusions. The user can select from a list of in-depth discussions of ash behavior and/or analyses procedures. The first of these discussions presents one of the principal findings of Contract No. DE-AC21-94MC31160 - a coherent mechanism describing how and why consolidated ash deposits form in PFBC filter vessels. This description is based on site observations made at the Tidd PFBC, field and laboratory analyses of ashes and nodules collected from Grimethorpe, Tidd and Karhula, and a review of literature describing eutectic formation, sintering, and consolidation of boiler tube deposits. The next three in-depth discussions review the factors in a PFBC that contribute to filter system failure, inertial particle collection in barrier filter vessels, and the potential for rapid increases in the thickness of transient IGCC filter cakes. The fifth and sixth discussions accessible for review from this screen detail the procedures and sampling protocol used during site visits, and the techniques used in the laboratory to characterize particulate samples. The seventh discussion describes laboratory equipment constructed to allow fragile filter cakes to be hardened with cyanoacrylate vapor while still on the surface of the filter element.

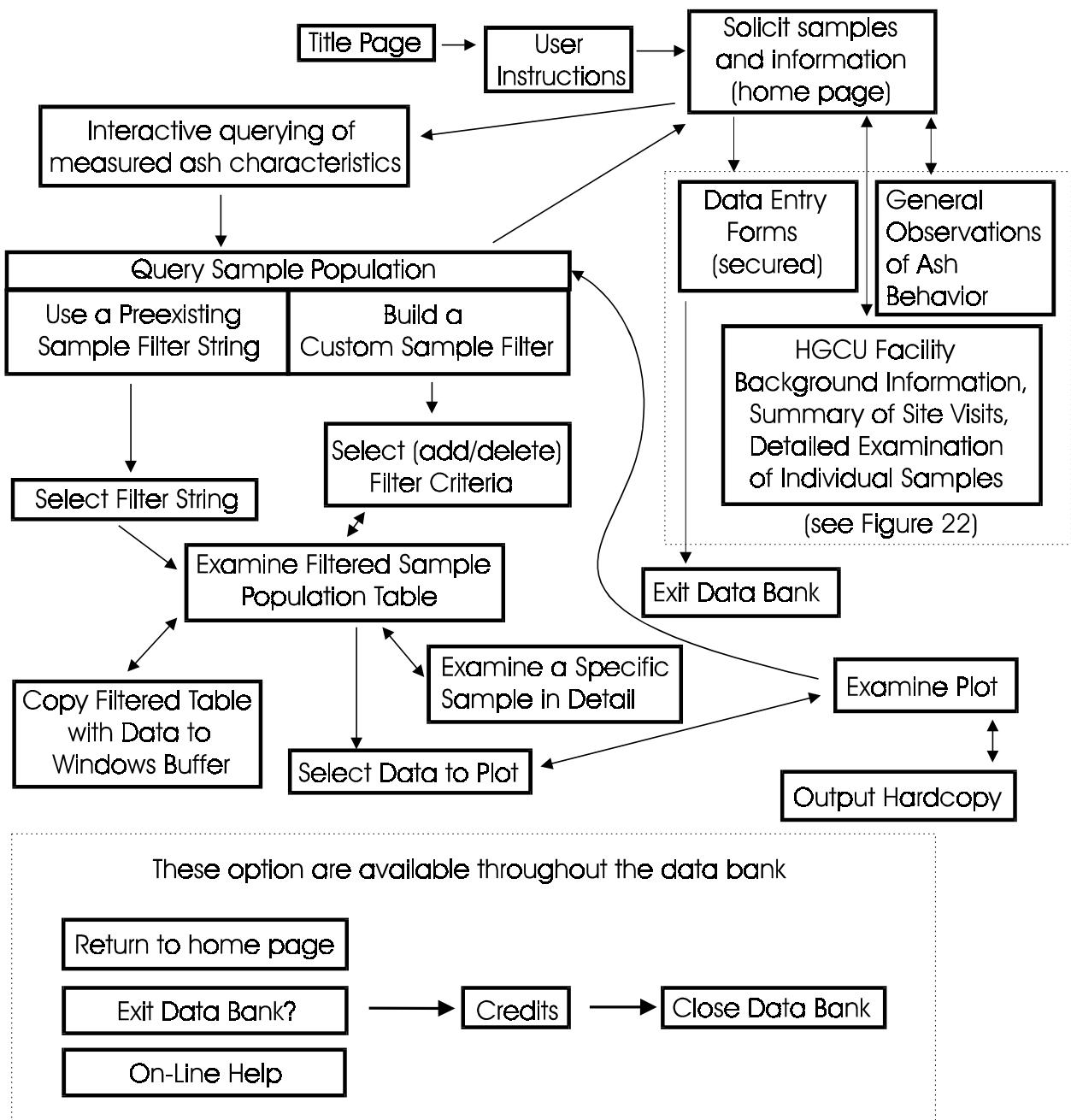


Figure 21. Schematic representation of the portion of the data bank controlling the interactive querying and plotting of sample analyses data.

Table 3  
Analytical Quantities that can be Selected and Plotted as part of Interactive Querying

Analytical Quantity	units
Stokes' Mass Median Diameter	$\mu\text{m}$
true particle density	$\text{g}/\text{cm}^3$
specific surface area	$\text{m}^2/\text{g}$
uncompacted bulk porosity	%
morphology factor	dimensionless
drag-equivalent diameter	$\mu\text{m}$
specific gas-flow resistance	in $\text{H}_2\text{O}\cdot\text{min}\cdot\text{ft}/\text{lb}$
tensile strength	$\text{N}/\text{m}^2$
$\text{Li}_2\text{O}$ content in ashed sample	% wt.
$\text{Na}_2\text{O}$ content in ashed sample	% wt.
$\text{K}_2\text{O}$ content in ashed sample	% wt.
$\text{MgO}$ content in ashed sample	% wt.
$\text{CaO}$ content in ashed sample	% wt.
$\text{Fe}_2\text{O}_3$ content in ashed sample	% wt.
$\text{Al}_2\text{O}_3$ content in ashed sample	% wt.
$\text{SiO}_2$ content in ashed sample	% wt.
$\text{TiO}_2$ content in ashed sample	% wt.
$\text{P}_2\text{O}_5$ content in ashed sample	% wt.
$\text{SO}_3$ content in ashed sample	% wt.
loss-on-ignition during ashing of sample	% wt.
soluble sulfate content of as-received sample	% wt.
equilibrium pH of as-received sample	dimensionless
porosity of ash deposit	%

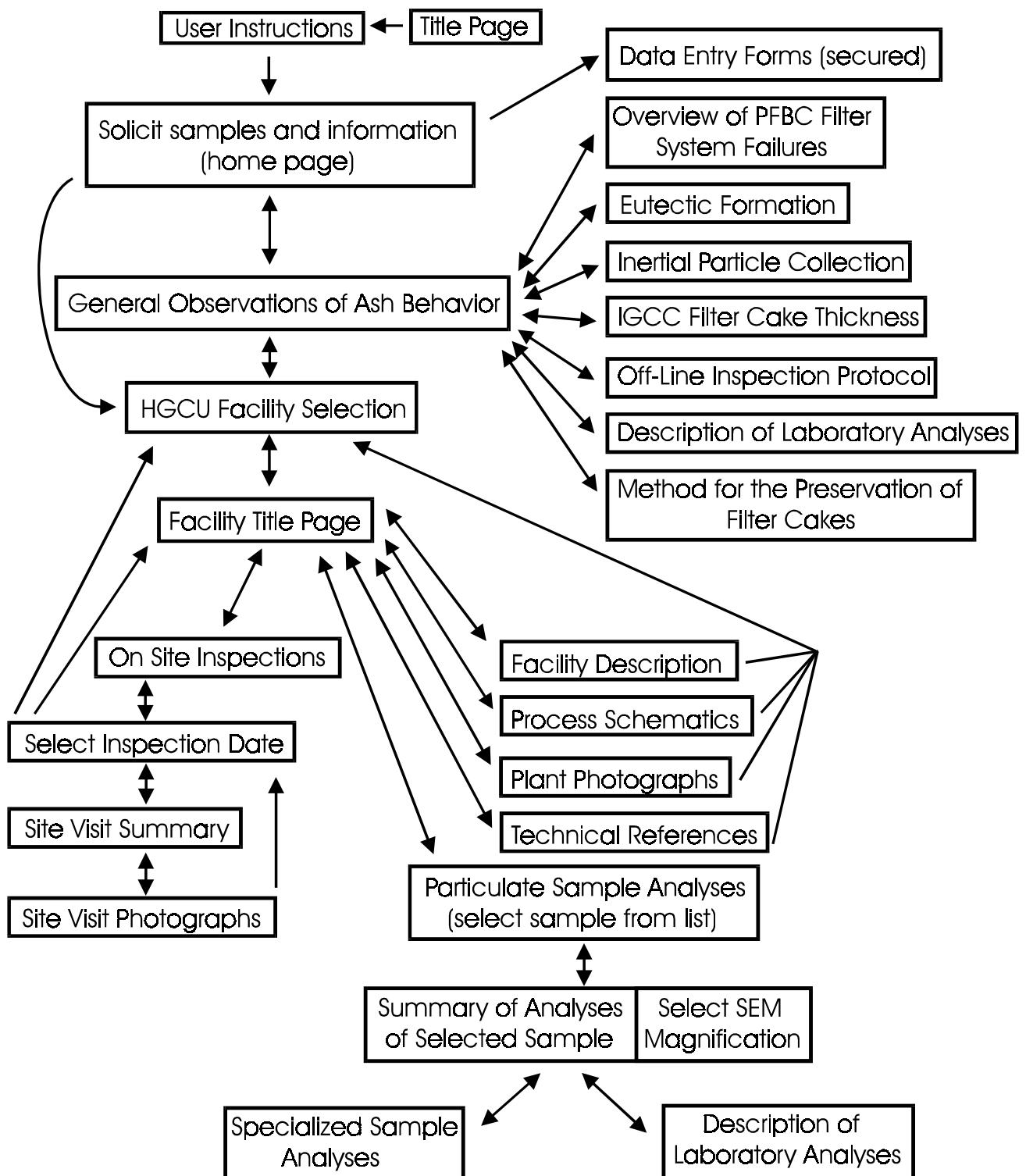


Figure 22. Schematic representation of the portion of the data bank controlling the review of project findings, site visits, background information on the HGCU facilities, and detailed analyses of individual samples.

From the main navigation screen, the user may also proceed to examine data and samples for specific facilities. If this option is selected, the user chooses one of the fifteen HGCU facilities to examine. Once a facility has been selected, the data bank lists the primary participating organizations and principal contact personnel for the facility. The user can then select and review one of the six categories listed: brief description of the facility; process schematics; plant photographs; technical references; on-site inspections; or particulate sample analyses. Under the first category, brief descriptions (up to two pages of text) are provided for each of the facilities in the data bank from which the various particulate samples were obtained. Series of process schematics and plant photographs can be scrolled through by selecting the second or third category. The fourth category provides the user with references to more detailed information about the facility. The category for on-site inspections contains information gathered during filter inspection and sampling trips made by Southern Research Institute personnel. Information in this category covers four site visits to the Tidd PFBC, one visit to the MGCR at Morgantown, and seven inspection and sampling trips to the PSDF. After selecting a particular site visit to review, the data bank provides a brief summary of the condition of the filter, the sampling procedures and the particulate samples obtained, and some of the key data obtained during the visit. A series of photographs of the filter cakes and ash deposits observed during the visit can also be reviewed.

When the user wishes to review the analyses of samples obtained from a particular facility, a scroll-down list of the samples from that facility is displayed. Included with this listing are brief descriptions of the samples, and where and when they were obtained. After a sample is selected to examine in detail, a screen is displayed that summarizes the physical and chemical analyses that have been performed on that sample. Physical attributes that have been measured and are included in this display include median particle size, specific surface area, particle morphology, bulk ash cohesivity, permeability, and tensile strength. This screen also provides access to scanning electron micrographs of many of the samples in the data bank. In general, these micrographs were obtained and can be viewed at four different magnifications. Chemical analyses of the selected sample are also summarized on this screen. Some of the samples collected which have unusual histories or unique characteristics have been analyzed with specialized techniques. When specialized analyses have been performed on the selected sample, the results of these analyses can also be accessed from this screen. This screen also provides a direct link to descriptions and explanations of the various analyses used to characterize the samples.

Another option available through the main navigation screen is the entry of additional data obtained during the analysis of particulate samples. This option is password-protected so the integrity of data included in the data bank can be maintained. The entry of additional data into the data bank can be accomplished using forms customized to accept sample identification information and the results of the analyses listed in Table 6.

## FILTER CAKE ENCAPSULATION

Filter cakes that have been observed on-site at the PSDF (and most of the filter cakes from other facilities) are quite fragile. Although relatively strong nodular deposits obtained at the Tidd PFBC were successfully encapsulated and preserved for analysis by the infiltration of low-viscosity epoxy, efforts to apply this epoxy to more fragile cakes has resulted in their

destruction. Therefore, a laboratory setup was developed to strengthen these fragile cakes with cyanoacrylate “super” glue vapor prior to the introduction of the low-viscosity epoxy. This method was described in the quarterly report covering the first quarter of FY 1999. The hardening and encapsulation processes were successfully applied to two filter elements and their attached filter cakes during this reporting quarter. The first of these elements was a Pall 442T element removed from the Westinghouse FL0301 filter vessel at the PSDF on August 1, 1997. Following encapsulation of the bottom 16 inches of the filter element, a portion of the preserved specimen covering about one inch of the candle’s length was polished and given to Howard Hendrix at the PSDF. A second element with its attached cake was obtained during the site visit to the PSDF on January 26, 1999. This filter element was a McDermott Ceramic Composite design taken from location T-10 in the top plenum. This element was newly installed just prior to the run ending on January 22, 1999. The bottom 16 inches of this sample was also successfully hardened and encapsulated. Several sections of this preserved sample are being polished for study. One of these sections that has already been polished has been given to the staff at the PSDF for examination.

## TASK 2 FILTER MATERIAL CHARACTERIZATION

All results obtained from the testing described above were supplied in data packages dated March 25, 1999 (CGC-ABB material) and March 26, 1999 (AIT material). Conclusions from the testing are summarized here.

### CGC – ABB Material:

- Thermal conductivity is low, which will lead to large temperature gradients; however, thermal stress failures are unlikely because of the low thermal expansion.
- Failures are likely during operation because of low mechanical strength.
- Vibrations are also a possible source of problems because of low Young's modulus.
- Surface damage is likely during installation and handling because of the soft outer membrane.

### AIT N610/mullite:

- Hoop tensile strength in as-manufactured condition is greater than other composite candles tested to date.
- Young's modulus is high enough that system induced vibrations are not likely to cause failures.
- Like other aluminum oxide materials this material has low thermal conductivity; however, the thin wall will help keep temperature gradients relatively low during thermal transients.
- Need axial tensile stress-strain measurements to complete the strength analysis and to calculate the temperature gradients required for thermal stress induced cracking.

Based on these initial results from the AIT N610/mullite, key material issues appear to be:

- Are properties retained after operation?
- Does the material become brittle during operation?
- Will the membrane survive handling, installation, and operation?
- What are the elevated temperature properties?

Some of these issues will be addressed in testing the N610/mullite candle after operation in the SCS - PSDF.

## FUTURE WORK

Efforts under Task 1 during the next quarter will include further development of the interactive data bank including completion of the querying and plotting capabilities, user instructions, on-line help, and the inclusion of additional data.

Under Task 2, hoop tensile testing of Schumacher TF20 with 1-inch long specimens, 3-inch long specimens, 6-inch long specimens, and 10-inch long specimens has been conducted to evaluate machining effects, if any. A data package describing these results is currently in preparation and will be provided during the upcoming quarter. Testing of McDermott ceramic composite and AlliedSignal PRD-66C candles after operation in the SCS - PSDF will also begin this quarter.

PARTICULATE HOT GAS STREAM CLEANUP TECHNICAL ISSUES

QUARTERLY REPORT

January 1999 - March 1999

SRI-ENV-99-8484-Q18

Contract No. DE-AC21-94MC31160

June 1, 1999

Approved by

A handwritten signature in black ink, appearing to read "Duane H. Pontius". The signature is fluid and cursive, with a small dot at the end.

---

Duane H. Pontius, Principal Investigator