



**Particulate Hot Gas Stream Cleanup
Technical Issues
Task 1.0 -- Assessment of Ash Characteristics
Quarterly Report
January - March 1995**

D.H. Pontius

Work Performed Under Contract No.: DE-AC21-94MC31160

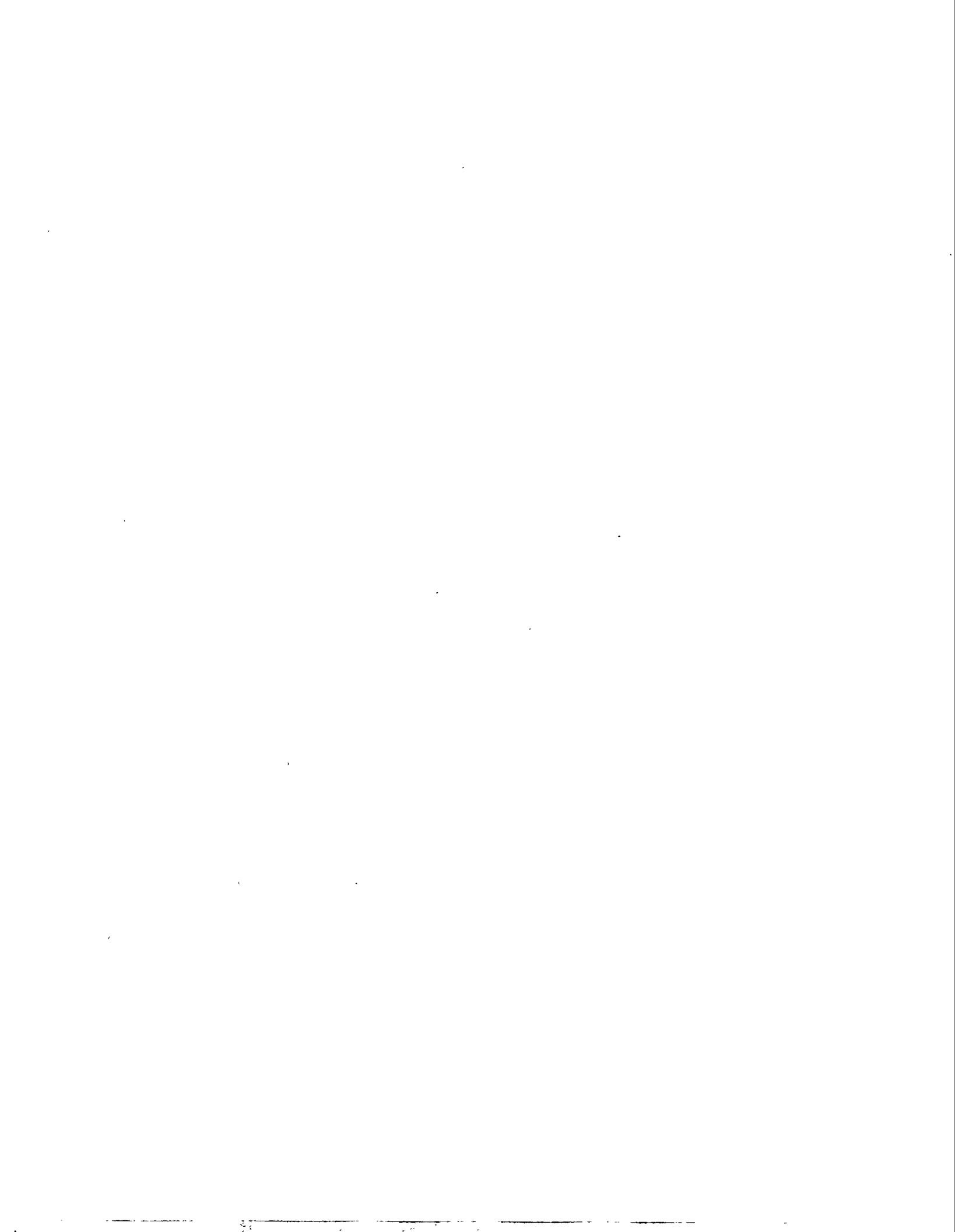
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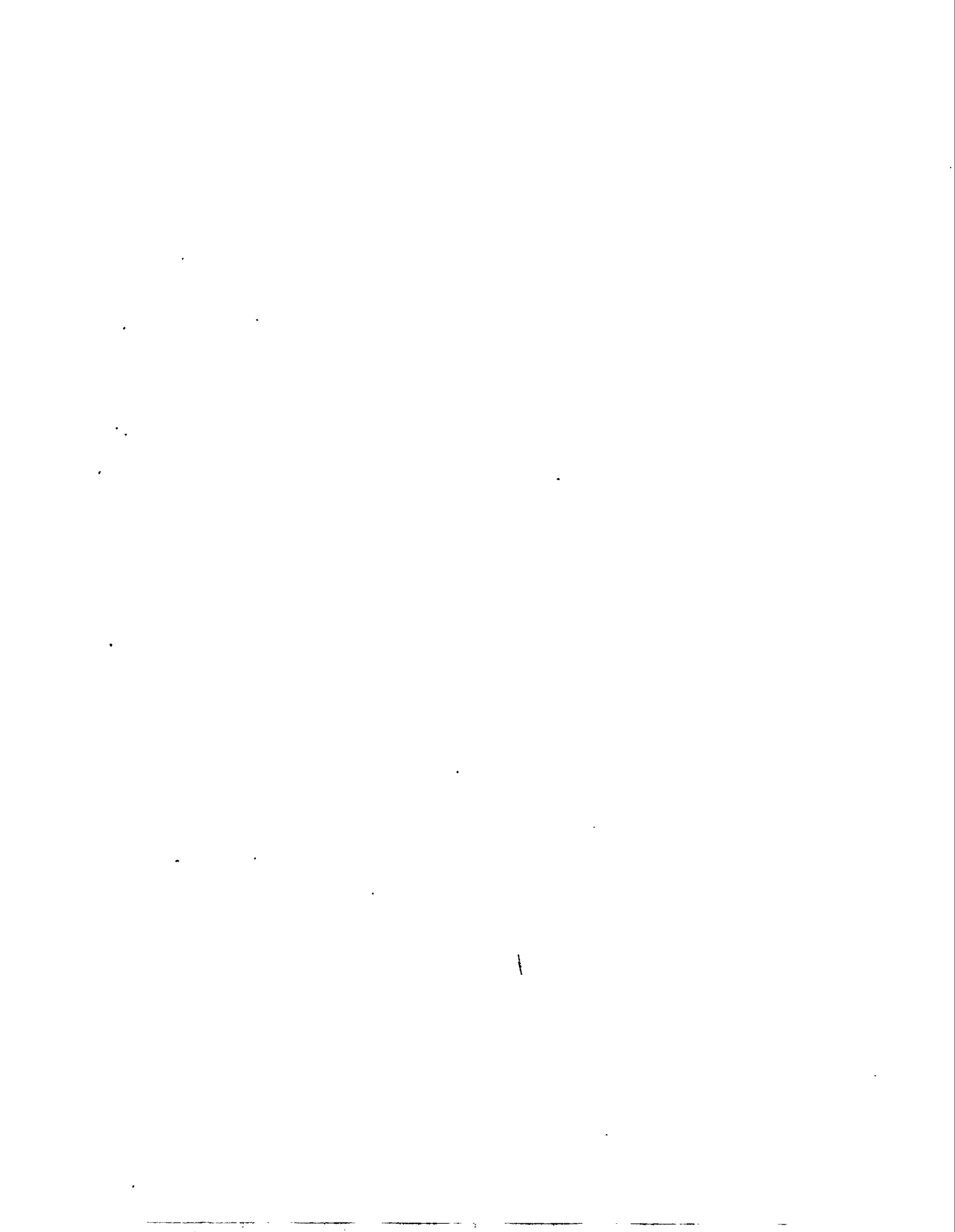


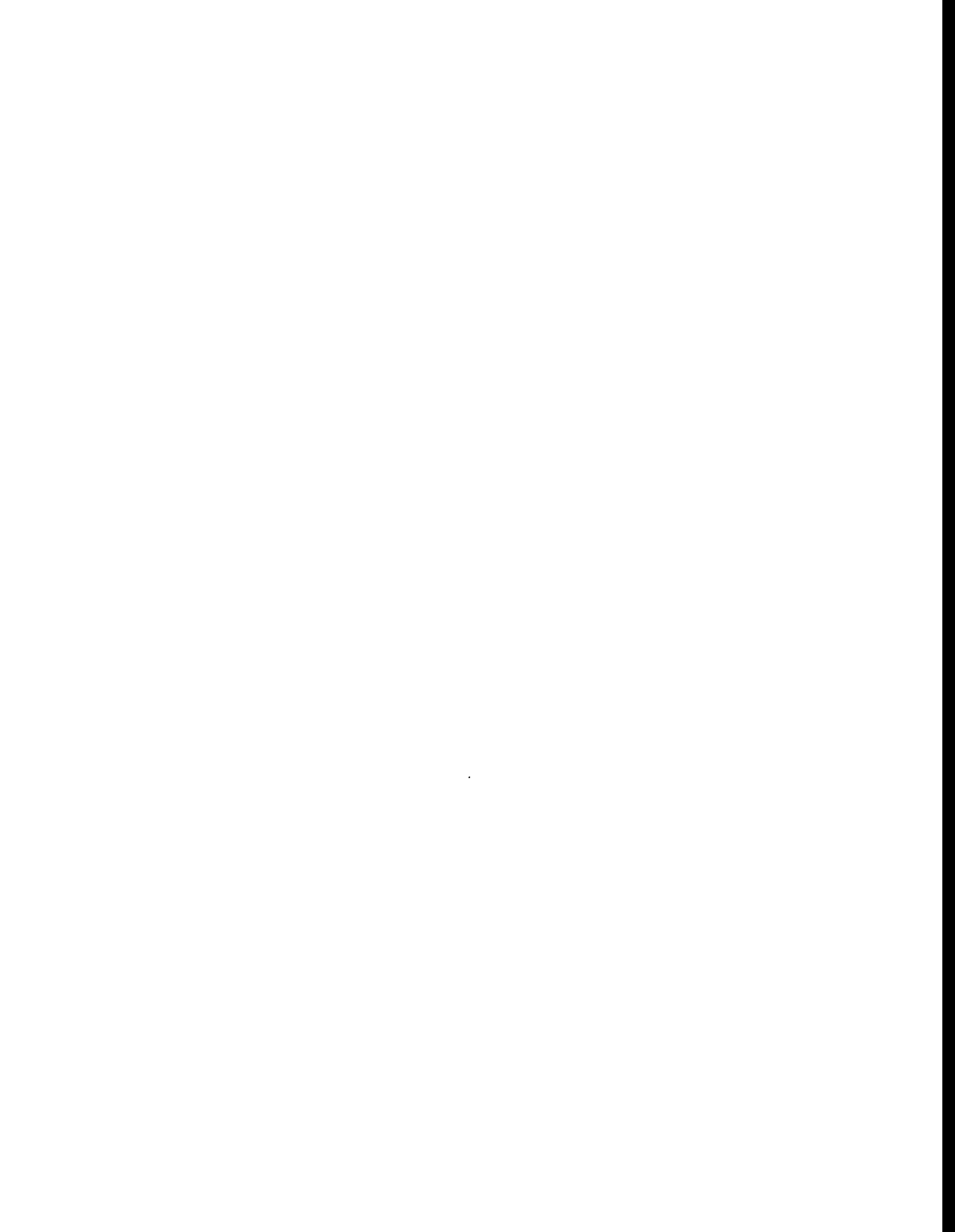
**PARTICULATE HOT GAS STREAM CLEANUP
TECHNICAL ISSUES
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The sample will also be examined to determine if it consolidated after being deposited within the volume of the ring. In the currently envisioned design, this device will operate only at ambient pressures.

FUTURE WORK

Plans for the next quarter include completion of the design of the uncompacted bulk porosity test device mentioned above, continued work on the interactive data base of HGCU ash characteristics, and a site visit to Tidd to collect additional samples for analysis and to observe and characterize the condition of the ash deposits within the APF.

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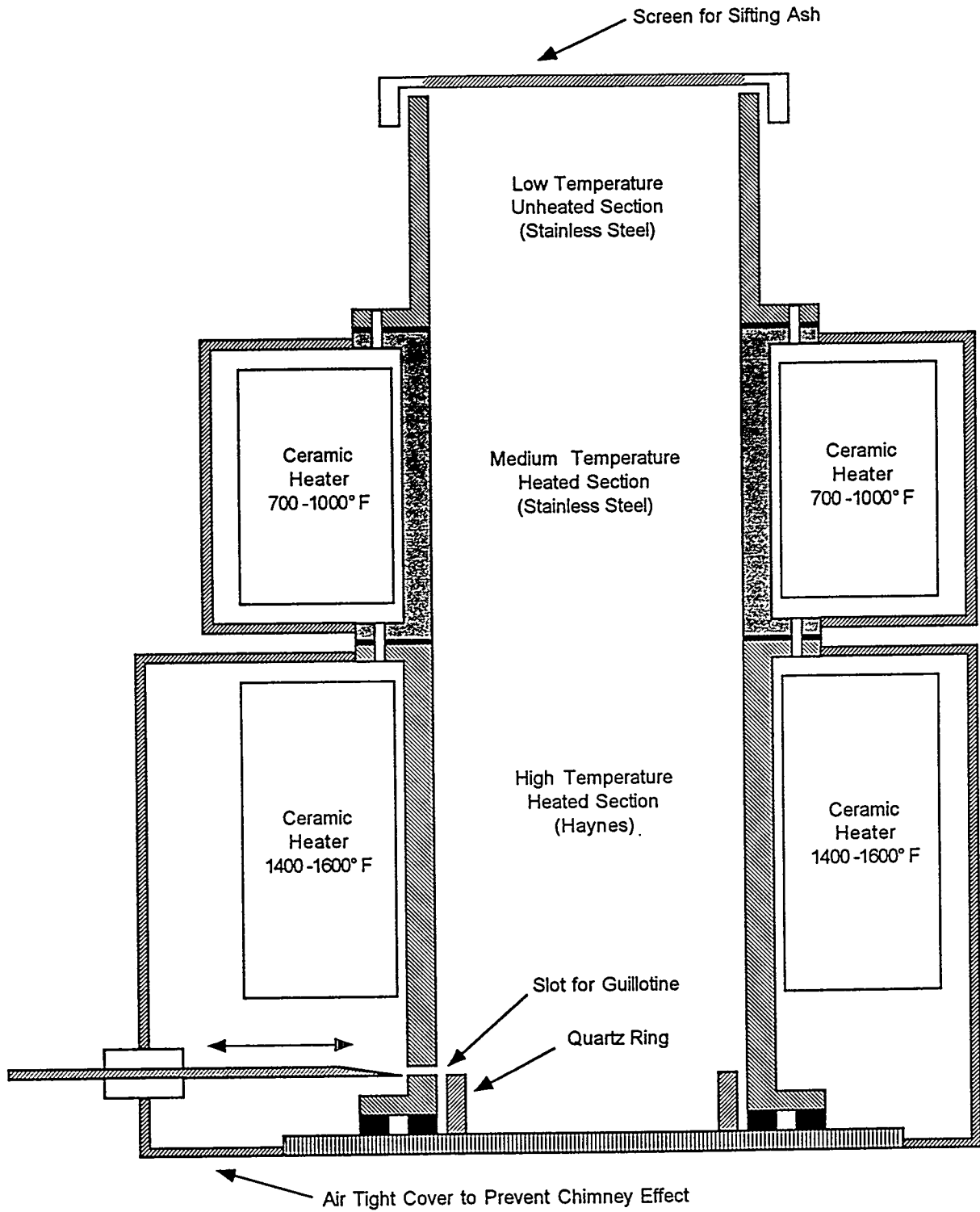


Figure 3. Preliminary conceptual design of the elevated temperature uncompacted bulk porosity test device.

Table 3
Overview of Ash Samples in the HGCU Data Base

# of samples	source	process
7	New York Univ.	Bubbling Bed PFBC
8	M.W. Kellogg	Circulating PFBC
14	M.W. Kellogg	gasification
2	Texaco	gasification
11	Grimethorpe	Circulating PFBC
9	KRW	gasification
2	Allison	coal-fired combustion turbine
10	Foster Wheeler	carbonizer
7	Foster Wheeler	Circulating PFBC
2	Iowa State University	AFBC
45	Karhula	Circulating PFBC
92	Tidd	Bubbling Bed PFBC

DESIGN OF A HIGH-TEMPERATURE UNCOMPACTED BULK POROSITY TEST DEVICE

As part of our effort to characterize HGCU ashes, we are designing a laboratory-test device that we will be able to use to form uncompacted beds of ash at temperatures up to 1700 °F. Measurement of the uncompacted bulk porosity of the HGCU ashes is highly desirable for two reasons. Because the permeability of a filter cake is strongly dependent on its porosity, the influence of temperature on the porosity of HGCU aggregates will help predict and explain filtering pressure drop encountered in HGCU filters. Furthermore, the physical tensile strength of aggregates obtained from the Tidd APF is strongly correlated with aggregate porosity. As with permeability, it is crucial to determine the effects of temperature on the strength of ash deposits such as those found in operating APFs.

A sketch of our preliminary design is shown in Figure 3. The device basically consists of a heated tube, open at the top, with a porous disk forming the bottom of the tube. Ash will be sifted or elutriated into the open top of the tube, and the particles will be heated to 1700 °F by the time they fall (or are transported by gas flow) through two heated zones of the tube and reach its bottom. After enough ash has been sifted into the preheated device to fill and cover the quartz ring, a guillotine-type blade will be slid across the top of the ring to isolate the amount of ash sample that was required to exactly fill the volume of the ring. After the blade is securely positioned over the top surface of the ring, the device will be allowed to cool down. (For some tests, the isolated ash sample may be allowed to remain at the test temperature for extended periods to determine the relationship between the time of exposure to high temperatures and the porosity of the aggregate.) The device will then be partially disassembled to allow the ash in the ring to be separately weighed.



HGCU filter vessel cause changes in gas volume and gas viscosity. In turn, these two factors affect the filtering pressure drop. Based on the conditions included in Table 2, equation (2) demonstrates the translation from laboratory to HGCU conditions for a typical HGCU facility operating at 10 atmospheres and 1600 °F.

Table 2
Comparison of Laboratory with HGCU Filter Conditions

condition	Laboratory conditions	HGCU conditions	effect on pressure drop
temperature, °K	295	1144	X 3.88
gas viscosity, poise	184	456 *	X 2.48
pressure , Atm	1.0	10	X 0.1

* These calculations use the values associated with air to approximate flue gas.

$$\Delta p(\text{HGCU conditions}) = \Delta p(\text{laboratory conditions}) \times 3.88 \times 2.48 \times 0.1 \quad (2)$$

Coincidentally, these multiplicative factors to combine to an overall factor of approximately 1.0. Therefore, when we measure the permeability of a simulated filter cake at laboratory conditions, the values we obtain apply directly to filter cakes in operating HGCU filters.

DATA BASE DEVELOPMENT AND FORMATTING

SRI currently maintains a collection of 209 ashes obtained from ten facilities involved in METC's HGCU program. An overview of the sources of these samples is presented in Table 3. Approximately half of these samples have been subjected to selected laboratory analyses. We are in the process of structuring the data describing these samples into an Excel spreadsheet. Almost all of the simple data (such as chemical analyses, specific surface area, uncompacted bulk porosity, etc.) have already been entered. There are a number of analyses and tests we have performed for a very limited number of samples of special interest that may be difficult to present in a conventional data base. Examples of these kinds of data include SEM photographs, SEM analyses (elemental and X-ray mapping) of individual ash particles, and baking and consolidation tests performed on bulk ash samples prepared in the laboratory. We have not yet decided how to include these types of specialized data in the data base.



$$\Delta p = (\mu U/D^2) \cdot (W/\rho) \cdot [111 - 211\varepsilon + 100\varepsilon^2]^2 \quad (1)$$

where:

Δp = pressure drop across the porous bed, μbar

μ = gas viscosity, poise

U = face velocity of the gas, cm/s

D = drag-equivalent diameter of the ash, cm

W = areal mass loading of the porous bed, g/cm^2

ρ = average true density of the ash particles, g/cm^3

ε = porosity of the porous bed, dimensionless ($0 < \varepsilon < 1$).

This new, adjusted form of the SRI permeability model was then used to recalculate the drag-equivalent diameters and relative gas flow resistances of all of the HGCU ashes for which we had measured permeability. These new values have been entered in the interactive data base.

TRANSLATING LABORATORY PERMEABILITY DATA TO HGCU CONDITIONS

Measuring the permeability of simulated filter cakes formed from HGCU ashes is a key component of our ash characterizations. These permeability measurements are used to estimate or rank the on-line filtering pressure drop associated with actual filter cakes formed from these ashes. To generate reliable estimates from our laboratory measurements, it is crucial that the key characteristics of the filter cake and flue gas as they exist during high temperature filtration are accurately known.

As discussed in the preceding section, the permeability of the filter cakes depends in part on the morphology of the filter cake (expressed by the drag-equivalent diameter and the porosity of the cake). The amount (areal loading) of filter cake and the true density of the ash particles also influence the overall pressure drop associated with a given filter cake. The other factors that determine the pressure drop (face velocity and gas viscosity) are dependent on the characteristics of the flue gas. Our laboratory measurements of bulk ash permeability and the porosity of filter cakes removed from HGCU filters in combination with SEM observations of actual HGCU filter cakes have allowed us to accurately characterize the morphology of these cakes. Assessments of areal loading must be performed on site. True particle density has been accurately determined in the laboratory for many ash samples obtained from HGCU filters and is unchanged at HGCU flue gas conditions. Face velocity and flue gas viscosity are significantly different in HGCU filters than in the laboratory. However, the overall effect of increased temperature and pressure can be accurately estimated based on filtration theory and basic theories of fluid dynamics.

Because we have accurate characterizations of the morphology of the filter cakes in operating HGCU filters, translating our laboratory permeability data (acquired at ambient temperature and pressure) to HGCU operating conditions is relatively simple. The



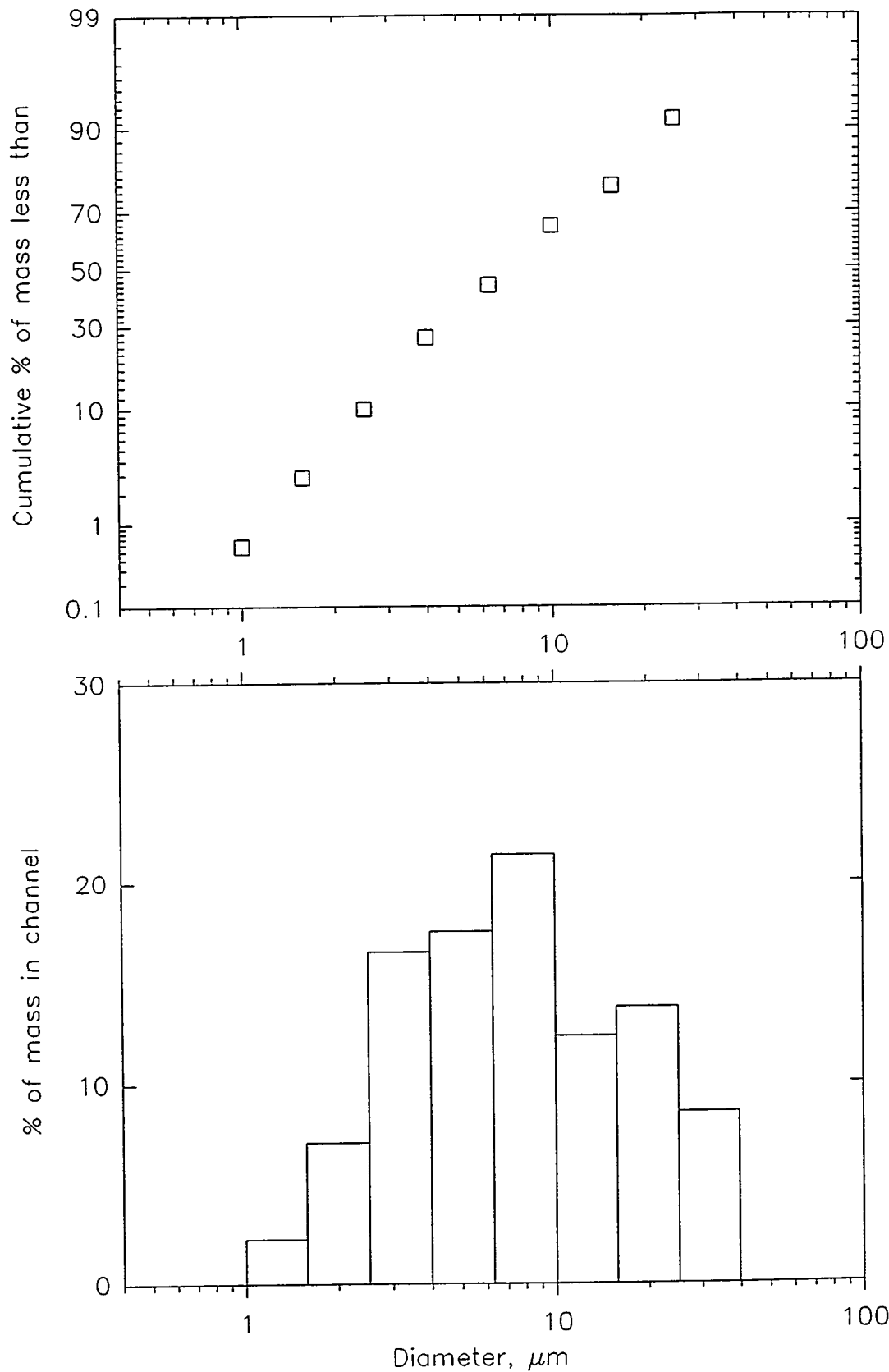


Figure 2. Cumulative and differential size distributions of the ash from the Tidd APF (ID # 4133) measured with a sedigraph on the basis of Stokes' diameters. The D_{50} of this distribution is $7.2 \mu\text{m}$, the $D_{16} = 3.0 \mu\text{m}$; and the $D_{84} = 20 \mu\text{m}$. For this analysis, the ash particles were assumed to have a density of 2.9 g/cm^3 .



ANALYSIS OF TIDD ASH SAMPLE

We measured the size distribution of a sample of ash from the Tidd APF that was sent to us by E. E. Smeltzer of Westinghouse Electric Corporation. Cumulative and differential presentations of the size distribution data were obtained with a centrifugally augmented sedigraph are shown in Figure 2. The D_{50} of this distribution is $7.2 \mu\text{m}$, the $D_{16} = 3.0 \mu\text{m}$; and the $D_{84} = 20 \mu\text{m}$. For this analysis, the ash particles were assumed to have a density of 2.9 g/cm^3 . This distribution is similar to other ashes we have analyzed from the Tidd APF. A sample information form has been forwarded to E. E. Smeltzer so that all available descriptive information corresponding to this sample can be entered in the HGCU data base.

MODIFICATIONS TO THE SRI EMPIRICAL PERMEABILITY MODEL

The mathematical permeability model SRI developed in 1986 was based almost exclusively on empirical data measured for pulverized-coal ashes. One ash produced by atmospheric fluidized-bed combustion was also included in this modeling. The SRI model, which relates the characteristic gas flow resistance of a porous bed of ash particles with the porosity of the bed, was based on the work of Kozeny⁴, Carman⁵, Langmuir⁶, and Davies⁷.

We have used this model in our analyses of bulk samples of HGCU ashes to estimate the characteristic gas flow resistance of filter cakes formed from the ash being analyzed. Two key parameters determine this characteristic flow resistance: porosity and particle diameter. However, during the development of the model, we found that simple measurements of physical diameter obtained with various devices (Coulter Counter, Bahco Classifier, or Shimadzu centrifugally augmented sedigraph) do not correlate well enough with measured permeability data to accurately predict gas flow resistance. Therefore, we used our empirical permeability data to define the drag-equivalent diameter, a value we calculate for each ash tested, which accurately ranks the gas flow resistance of different ashes at equal filter cake porosities.

As our data base of HGCU ashes expanded, we found that the permeability data we measured for these ashes comprising small, irregular particles was not satisfactorily fit by the model developed in 1986 primarily for larger, spherical particles. In almost every case, the 1986 model would tend to overestimate the gas flow resistance values of uncompacted filter cakes. To correct these estimates, we used permeability data measured for 34 ashes composed of small, irregular particles to develop an adjusted permeability model. Most of these 34 ashes were generated at HGCU facilities. The new adjusted equation is presented below:



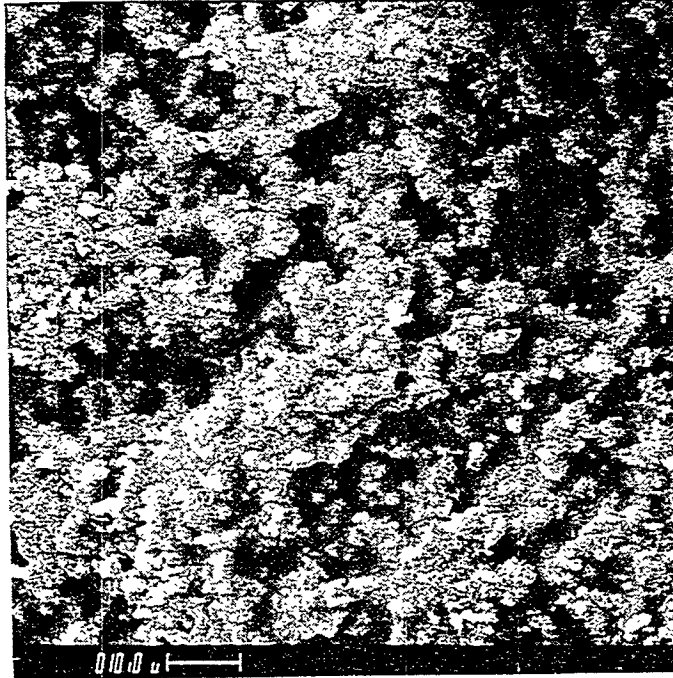


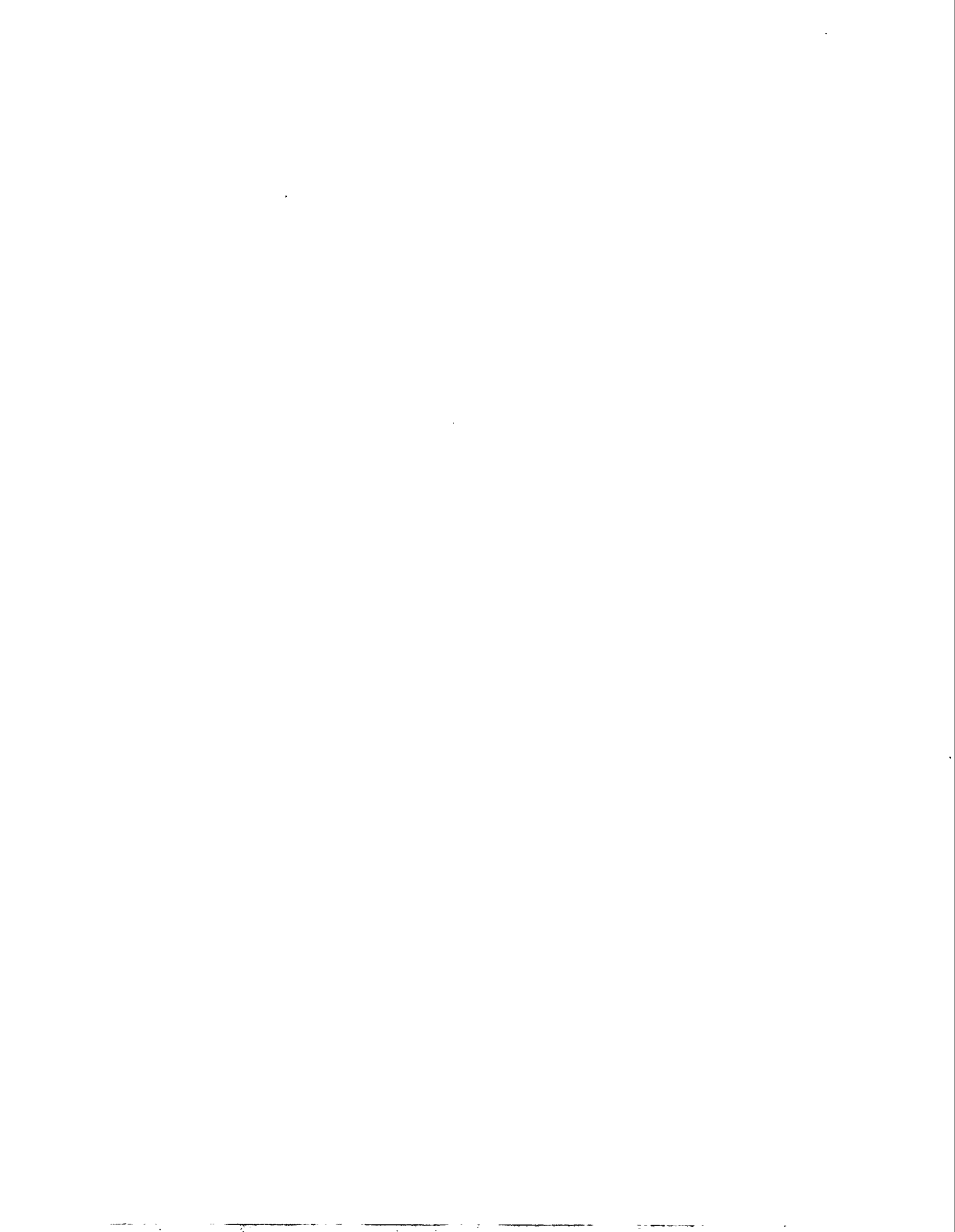
Figure 1. Representative SEM photograph of a fresh fracture surface of an aggregate of ash removed from the ash shedding cone below the middle plenum at Tidd on October 27, 1994 (ID # 4114) taken at 1000X.

Table 1
Chemical Characteristics of Tidd Ashes and Sorbents, % wt.

constituent	hopper ashes		filter cake ashes	
	ID # 2889	ID # 2988	ID # 4011	ID # 4012
Na ₂ O	0.30	0.31	0.30	0.27
K ₂ O	1.6	1.42	1.73	1.77
MgO	9.9	9.63	8.77	7.96
CaO	15.3	15.48	14.16	13.67
Fe ₂ O ₃	5.3	6.49	4.79	5.63
Al ₂ O ₃	13.4	11.80	13.01	13.63
SiO ₂	19.2	21.29	23.03	22.89
TiO ₂	0.50	0.56	0.61	0.07
P ₂ O ₅	0.08	0.12	0.11	0.10
SO ₃	33.6	31.10	31.06	31.38
LOI	1.5	2.96	1.45	1.84
soluble SO ₄ ⁼	48.3	--	--	40.2

By examining the SEM photograph of an aggregate of ash taken from the ash shedding cone below the middle plenum at Tidd (Figure 1), it is apparent that a significant proportion of the mass of the ash particles has been transformed into the amorphous material mentioned above. The physical appearance of the amorphous mass is clearly distinct from the appearance of the small ash particles. The appearance of the amorphous mass can best be described as concretion formed from individual ash particles embedded in what appears to be a large, interconnected molten mass. This appearance supports our contention that eutectics have formed in the ash aggregate.

It is doubtful that process changes such as slightly lowering the temperature in the APF, sorbent switching, or addition of a conditioning agent will be able to significantly affect the formation of these eutectics and the subsequent consolidation and strengthening of the ash aggregates. The minimum operating temperature of the APF is strictly limited by the economics of the PFBC process. Past operation at reduced temperatures around 1250 °F have not been able to prevent the formation of consolidated ash aggregates. Since magnesium and calcium are both excellent fluxing agents, altering the type of sorbent used in the PFBC process is not likely to alter the tendency for eutectic formation. Finally, the addition of any conditioning agent to the eutectic system is only likely to lower its melting point even further. Therefore, the optimum solution to the problems caused by the ash aggregates that have been consolidated and strengthened by pervasive eutectic formation is the removal of ash aggregates from the APF before these eutectics have had enough time to develop.



eventually consolidating the structure of the entire ash agglomerate. The same consolidating mechanism would apply to any eutectic mixture that melted or significantly softened at the temperatures within the filter vessel. Consolidation may be further enhanced by the relatively small size of the ash particles in the agglomerate.

As we have seen in SEM photographs of aggregates of ash removed from the Tidd APF, the primary ash particles are nearly completely imbedded in a pervasive amorphous mass. Based on various observations of the behavior of these aggregates, we believe that the amorphous mass in which the particles are embedded is derived directly from the primary coal ash particles and sorbent particles originally deposited on the surface of the aggregate. The first observation that supports this contention is based on the difference between the porosity of newly-deposited regions of ash aggregates (85 % or higher) and the porosity of portions of the aggregates that have been exposed to the temperatures in the APF for extended periods (around 74 %). In other words, the newly-deposited regions of the agglomerates are no more than 15 % solid, whereas the solid content of older portions is at least 26 %. This means that as the aggregates age in the APF, either the amount of mass has been nearly doubled from some source other than the primary particles (condensation or adsorption from the flue gas), or the primary particles have rearranged themselves to occupy about 58 % of their original total volume.

If the flue gas contributed large amounts of mass to the aggregate through condensation or adsorption, the chemical constituents of this added mass would be limited to compounds that could exist as a vapor at the normal operating conditions of the APF. Although many compounds could satisfy these requirements, some of the major constituents found in the fly ash do not. Three major constituents that will not be found in a gaseous state in the APF are iron, aluminum and silicon. When we compare the mineral analyses of Tidd APF hopper ashes (ID #s 2889 and 2988) with aged Tidd filter cake ashes (ID #s 4011 and 4012), the iron, aluminum, and silicon contents of the two types of samples are very similar (see Table 1). Since the amounts of these three non-volatile elements are not significantly lower in filter cake ash than in hopper ash, it is apparent that essentially all of the mass of the filter cake is due to the original ash particles, and not to any significant deposition of gas-phase constituents from the flue gas.



OBJECTIVES

This task has two primary objectives. The first is to generate a readily accessible data base of the key characteristics of ashes collected from operating advanced particle filters. The second objective is to relate these ash properties and the contents of the data base to the operation and performance of the advanced particle filters and filter components. The first objective includes formatting the data base and collecting, analyzing, and maintaining ashes from operating HGCU facilities. To expand the usefulness of the data base we are also designing a device for direct measurement of the uncompacted bulk porosity of ashes from HGCU facilities at temperatures comparable to those experienced in HGCU filters. The second objective of this task involves the collection of operating histories from advanced particle filters, correlating these histories with ash characteristics, interpreting these correlations, and communicating our conclusions in the various venues prescribed by the U.S. Department of Energy's Morgantown Energy Technology Center (DOE/METC).

RESEARCH ACTIVITIES DURING THE REPORTING PERIOD

IDENTIFYING PARTICLE-TO-PARTICLE BONDS IN TIDD ASH AGGREGATES

Studies of the buildup of boiler tube deposits in conventional pulverized-coal fired boilers describe a mechanism which may account for the apparent sintering of PFBC ash deposits found in the Tidd APF. Many of the ash particles collected in HGCU filter assemblies are derived directly from coal particles. These ash particles often contain a large percentage of aluminosilicate compounds. The other main source of ash particles is the sorbent used in the PFBC process. Sorbent-derived ash particles contain relatively large amounts of magnesium and/or calcium. Once these two types of ash particles come in contact with each other in the agglomerates formed in the filter vessel, the aluminosilicate compounds in the coal fly ash tend to react with alkali and alkaline metals in the sorbent ash particles to form eutectics that melt at relatively low temperatures¹. The progress of these reactions is supported by the intimate contact of the ash particles in the agglomerate and by long-term exposure of the ash particles to the temperatures in the filter vessel.

Most of the research into the formation of these eutectics has examined the formation of calcium aluminosilicate compounds (e.g. $2\text{Ca}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ or $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$). Although pure forms of these two compounds do not melt at the temperatures encountered in HGCU filter vessels (pure $2\text{Ca}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ and $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ melt at around 2800 °F), impurities that would almost certainly be present in these compounds due to the heterogeneous nature of coal fly ash particles would lower their melting points. It is likely that this reduction in melting points could combine with long-term exposure to the temperatures in the filter vessel to create relatively soft, sticky layers on the surfaces of the ash particles^{2,3}. As the viscosity of the outer layer of the ash particles decreases, the bonds between the particles become stronger. Also, the surface tension of the liquid or near-liquid layer on the particles tends to pull adjacent ash particles closer together, thereby



INTRODUCTION

This is the second in a series of quarterly reports describing the activities performed under Task 1 of Contract No. DE-AC21-94MC31160. The analyses of HGCU ashes and descriptions of filter performance presented in this report are designed to address the problems with filter operation that are apparently linked to the characteristics of the collected ash. Some of the problems observed at the Tidd and Karhula 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. (Task 2 of this contract includes characterization of new and used filter elements.) In addition to these problems related to the characteristics of PFBC ashes, our previous laboratory characterizations of gasifier and carbonizer ashes have shown that these ashes also have characteristics that might negatively affect filtration.

In order to identify which ash characteristics can lead to problems with filtration, we have assembled over 200 ash samples from ten facilities involved in METC's HGCU program. We have analyzed many of these ashes with a variety of laboratory tests. Physical attributes of the particles that we have examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. We have also performed a range of chemical analyses on these ashes, as well as characterizations of agglomerates of ash removed from various places in the filter vessels at Tidd and Karhula. We are in the process of assembling the data obtained in these studies into an interactive data base which will help the manufacturers and operators of high-temperature barrier filters tailor their designs and operations to the specific characteristics of the ashes they are collecting.

Much of the work planned for Task 1 builds directly on work performed under a prior contract (No. DE-AC21-89MC26239). Two visits to the 70 MWe Pressurized Fluidized-Bed Combustion (PFBC) Facility at the Tidd Plant were made during this prior contract to collect samples and observe and record the condition of the APF. Two topical reports covering the collection and analyses of HGCU ashes were also issued under Contract No. DE-AC21-89MC26239. The first report, *Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, covered samples obtained and analyzed between October 1992 and September 1993. The second report, *Updated Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, is still in draft form, and covers samples obtained and analyzed between October 1993 and August 1994. Because operating experiences with HGCU facilities continue to accumulate, Task 1 of this current contract will continue many of the same types of analyses described in these two topical reports.



EXECUTIVE SUMMARY

This is the second in a series of quarterly reports describing the activities performed under Task 1 of 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 the problems with filter operation that are apparently linked to the characteristics of the collected ash. This task is designed to generate a data base 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.

Research activities during this reporting period include initial formatting of the data base and data entry, modification of the permeability model used to predict the filtering pressure drop associated with ashes in the data base, and initial design of a high-temperature test device for the measurement of the uncompacted bulk porosity of aggregates of ash formed at temperatures commonly encountered in operating APFs. (Uncompacted bulk porosity is a good indicator of the relative cohesivity of the ash, and also can be used to help estimate filter cake porosity and permeability.)

During the past quarter, we also reviewed the chemical analyses of hopper and filter cake ashes from Tidd to assess whether additional material that might be condensed out of the flue gas or adsorbed onto the collected particles could account for the apparent consolidation of the various agglomerates of ash present in the filter vessel. Our data show that the degree of consolidation of these agglomerates can not be accounted for by condensation and/or adsorption of materials from the flue gas. The mechanism responsible for the extreme consolidation of these agglomerates of ash is most likely a physical rearrangement of the ash particles due to the surface tension of melted or partially melted alkali-aluminosilicate eutectic mixture(s) that form at the contact points between adjacent particles after long-term exposure to the temperatures in the APF.

Plans for the next quarter include completion of the design of the uncompacted bulk porosity test device mentioned above, continued work on the interactive data base of HGCU ash characteristics, and a site visit to Tidd to collect additional samples for analysis and to observe and characterize the condition of the ash deposits within the APF.



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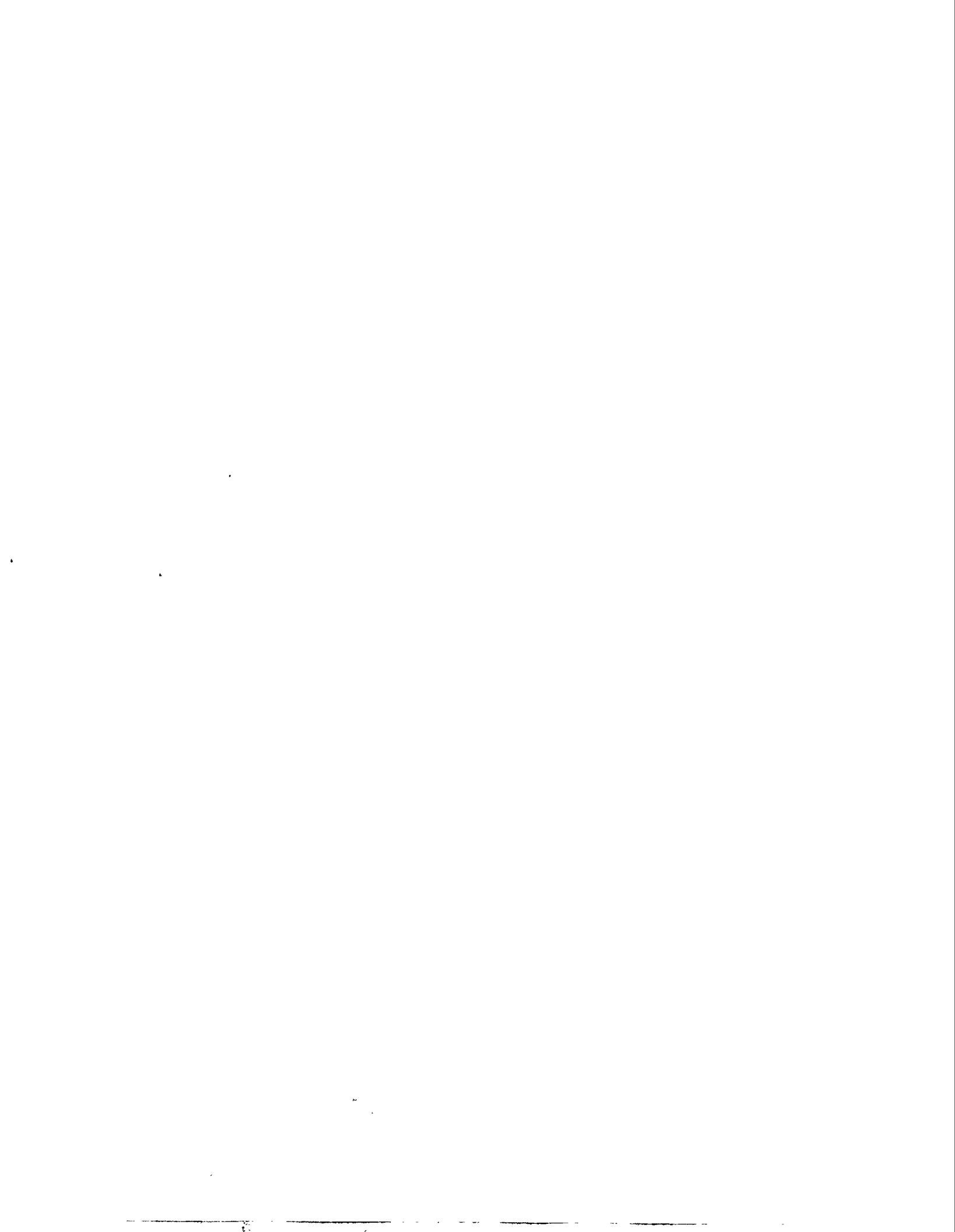
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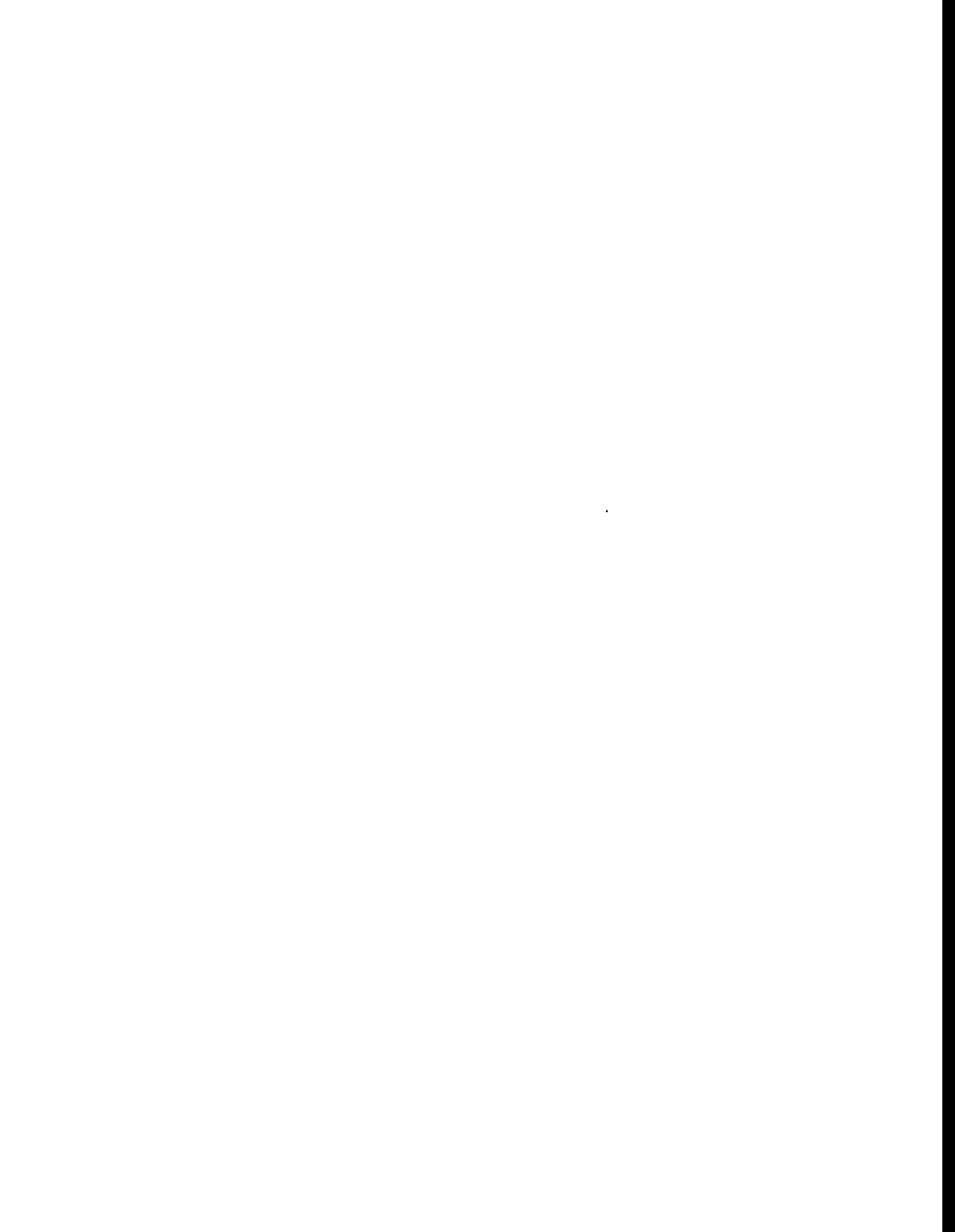


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EXECUTIVE SUMMARY

This is the second in a series of quarterly reports describing the activities performed under Task 1 of 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 the problems with filter operation that are apparently linked to the characteristics of the collected ash. This task is designed to generate a data base 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.

Research activities during this reporting period include initial formatting of the data base and data entry, modification of the permeability model used to predict the filtering pressure drop associated with ashes in the data base, and initial design of a high-temperature test device for the measurement of the uncompacted bulk porosity of aggregates of ash formed at temperatures commonly encountered in operating APFs. (Uncompacted bulk porosity is a good indicator of the relative cohesivity of the ash, and also can be used to help estimate filter cake porosity and permeability.)

During the past quarter, we also reviewed the chemical analyses of hopper and filter cake ashes from Tidd to assess whether additional material that might be condensed out of the flue gas or adsorbed onto the collected particles could account for the apparent consolidation of the various agglomerates of ash present in the filter vessel. Our data show that the degree of consolidation of these agglomerates can not be accounted for by condensation and/or adsorption of materials from the flue gas. The mechanism responsible for the extreme consolidation of these agglomerates of ash is most likely a physical rearrangement of the ash particles due to the surface tension of melted or partially melted alkali-aluminosilicate eutectic mixture(s) that form at the contact points between adjacent particles after long-term exposure to the temperatures in the APF.

Plans for the next quarter include completion of the design of the uncompacted bulk porosity test device mentioned above, continued work on the interactive data base of HGCU ash characteristics, and a site visit to Tidd to collect additional samples for analysis and to observe and characterize the condition of the ash deposits within the APF.



INTRODUCTION

This is the second in a series of quarterly reports describing the activities performed under Task 1 of Contract No. DE-AC21-94MC31160. The analyses of HGCU ashes and descriptions of filter performance presented in this report are designed to address the problems with filter operation that are apparently linked to the characteristics of the collected ash. Some of the problems observed at the Tidd and Karhula 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. (Task 2 of this contract includes characterization of new and used filter elements.) In addition to these problems related to the characteristics of PFBC ashes, our previous laboratory characterizations of gasifier and carbonizer ashes have shown that these ashes also have characteristics that might negatively affect filtration.

In order to identify which ash characteristics can lead to problems with filtration, we have assembled over 200 ash samples from ten facilities involved in METC's HGCU program. We have analyzed many of these ashes with a variety of laboratory tests. Physical attributes of the particles that we have examined include size distribution, specific surface area, particle morphology, and bulk ash cohesivity and permeability. We have also performed a range of chemical analyses on these ashes, as well as characterizations of agglomerates of ash removed from various places in the filter vessels at Tidd and Karhula. We are in the process of assembling the data obtained in these studies into an interactive data base which will help the manufacturers and operators of high-temperature barrier filters tailor their designs and operations to the specific characteristics of the ashes they are collecting.

Much of the work planned for Task 1 builds directly on work performed under a prior contract (No. DE-AC21-89MC26239). Two visits to the 70 MWe Pressurized Fluidized-Bed Combustion (PFBC) Facility at the Tidd Plant were made during this prior contract to collect samples and observe and record the condition of the APF. Two topical reports covering the collection and analyses of HGCU ashes were also issued under Contract No. DE-AC21-89MC26239. The first report, *Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, covered samples obtained and analyzed between October 1992 and September 1993. The second report, *Updated Assessment of Ash Characteristics from Gas Stream Cleanup Facilities*, is still in draft form, and covers samples obtained and analyzed between October 1993 and August 1994. Because operating experiences with HGCU facilities continue to accumulate, Task 1 of this current contract will continue many of the same types of analyses described in these two topical reports.



OBJECTIVES

This task has two primary objectives. The first is to generate a readily accessible data base of the key characteristics of ashes collected from operating advanced particle filters. The second objective is to relate these ash properties and the contents of the data base to the operation and performance of the advanced particle filters and filter components. The first objective includes formatting the data base and collecting, analyzing, and maintaining ashes from operating HGCU facilities. To expand the usefulness of the data base we are also designing a device for direct measurement of the uncompacted bulk porosity of ashes from HGCU facilities at temperatures comparable to those experienced in HGCU filters. The second objective of this task involves the collection of operating histories from advanced particle filters, correlating these histories with ash characteristics, interpreting these correlations, and communicating our conclusions in the various venues prescribed by the U.S. Department of Energy's Morgantown Energy Technology Center (DOE/METC).

RESEARCH ACTIVITIES DURING THE REPORTING PERIOD

IDENTIFYING PARTICLE-TO-PARTICLE BONDS IN TIDD ASH AGGREGATES

Studies of the buildup of boiler tube deposits in conventional pulverized-coal fired boilers describe a mechanism which may account for the apparent sintering of PFBC ash deposits found in the Tidd APF. Many of the ash particles collected in HGCU filter assemblies are derived directly from coal particles. These ash particles often contain a large percentage of aluminosilicate compounds. The other main source of ash particles is the sorbent used in the PFBC process. Sorbent-derived ash particles contain relatively large amounts of magnesium and/or calcium. Once these two types of ash particles come in contact with each other in the agglomerates formed in the filter vessel, the aluminosilicate compounds in the coal fly ash tend to react with alkali and alkaline metals in the sorbent ash particles to form eutectics that melt at relatively low temperatures¹. The progress of these reactions is supported by the intimate contact of the ash particles in the agglomerate and by long-term exposure of the ash particles to the temperatures in the filter vessel.

Most of the research into the formation of these eutectics has examined the formation of calcium aluminosilicate compounds (e.g. $2\text{Ca}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ or $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$). Although pure forms of these two compounds do not melt at the temperatures encountered in HGCU filter vessels (pure $2\text{Ca}\cdot\text{Al}_2\text{O}_3\cdot\text{SiO}_2$ and $\text{CaO}\cdot\text{Al}_2\text{O}_3\cdot 2\text{SiO}_2$ melt at around 2800 °F), impurities that would almost certainly be present in these compounds due to the heterogeneous nature of coal fly ash particles would lower their melting points. It is likely that this reduction in melting points could combine with long-term exposure to the temperatures in the filter vessel to create relatively soft, sticky layers on the surfaces of the ash particles^{2,3}. As the viscosity of the outer layer of the ash particles decreases, the bonds between the particles become stronger. Also, the surface tension of the liquid or near-liquid layer on the particles tends to pull adjacent ash particles closer together, thereby



eventually consolidating the structure of the entire ash agglomerate. The same consolidating mechanism would apply to any eutectic mixture that melted or significantly softened at the temperatures within the filter vessel. Consolidation may be further enhanced by the relatively small size of the ash particles in the agglomerate.

As we have seen in SEM photographs of aggregates of ash removed from the Tidd APF, the primary ash particles are nearly completely imbedded in a pervasive amorphous mass. Based on various observations of the behavior of these aggregates, we believe that the amorphous mass in which the particles are embedded is derived directly from the primary coal ash particles and sorbent particles originally deposited on the surface of the aggregate. The first observation that supports this contention is based on the difference between the porosity of newly-deposited regions of ash aggregates (85 % or higher) and the porosity of portions of the aggregates that have been exposed to the temperatures in the APF for extended periods (around 74 %). In other words, the newly-deposited regions of the agglomerates are no more than 15 % solid, whereas the solid content of older portions is at least 26 %. This means that as the aggregates age in the APF, either the amount of mass has been nearly doubled from some source other than the primary particles (condensation or adsorption from the flue gas), or the primary particles have rearranged themselves to occupy about 58 % of their original total volume.

If the flue gas contributed large amounts of mass to the aggregate through condensation or adsorption, the chemical constituents of this added mass would be limited to compounds that could exist as a vapor at the normal operating conditions of the APF. Although many compounds could satisfy these requirements, some of the major constituents found in the fly ash do not. Three major constituents that will not be found in a gaseous state in the APF are iron, aluminum and silicon. When we compare the mineral analyses of Tidd APF hopper ashes (ID #s 2889 and 2988) with aged Tidd filter cake ashes (ID #s 4011 and 4012), the iron, aluminum, and silicon contents of the two types of samples are very similar (see Table 1). Since the amounts of these three non-volatile elements are not significantly lower in filter cake ash than in hopper ash, it is apparent that essentially all of the mass of the filter cake is due to the original ash particles, and not to any significant deposition of gas-phase constituents from the flue gas.



Table 1
Chemical Characteristics of Tidd Ashes and Sorbents, % wt.

constituent	hopper ashes		filter cake ashes	
	ID # 2889	ID # 2988	ID # 4011	ID # 4012
Na ₂ O	0.30	0.31	0.30	0.27
K ₂ O	1.6	1.42	1.73	1.77
MgO	9.9	9.63	8.77	7.96
CaO	15.3	15.48	14.16	13.67
Fe ₂ O ₃	5.3	6.49	4.79	5.63
Al ₂ O ₃	13.4	11.80	13.01	13.63
SiO ₂	19.2	21.29	23.03	22.89
TiO ₂	0.50	0.56	0.61	0.07
P ₂ O ₅	0.08	0.12	0.11	0.10
SO ₃	33.6	31.10	31.06	31.38
LOI	1.5	2.96	1.45	1.84
soluble SO ₄ ⁼	48.3	--	--	40.2

By examining the SEM photograph of an aggregate of ash taken from the ash shedding cone below the middle plenum at Tidd (Figure 1), it is apparent that a significant proportion of the mass of the ash particles has been transformed into the amorphous material mentioned above. The physical appearance of the amorphous mass is clearly distinct from the appearance of the small ash particles. The appearance of the amorphous mass can best be described as concretion formed from individual ash particles embedded in what appears to be a large, interconnected molten mass. This appearance supports our contention that eutectics have formed in the ash aggregate.

It is doubtful that process changes such as slightly lowering the temperature in the APF, sorbent switching, or addition of a conditioning agent will be able to significantly affect the formation of these eutectics and the subsequent consolidation and strengthening of the ash aggregates. The minimum operating temperature of the APF is strictly limited by the economics of the PFBC process. Past operation at reduced temperatures around 1250 °F have not been able to prevent the formation of consolidated ash aggregates. Since magnesium and calcium are both excellent fluxing agents, altering the type of sorbent used in the PFBC process is not likely to alter the tendency for eutectic formation. Finally, the addition of any conditioning agent to the eutectic system is only likely to lower its melting point even further. Therefore, the optimum solution to the problems caused by the ash aggregates that have been consolidated and strengthened by pervasive eutectic formation is the removal of ash aggregates from the APF before these eutectics have had enough time to develop.



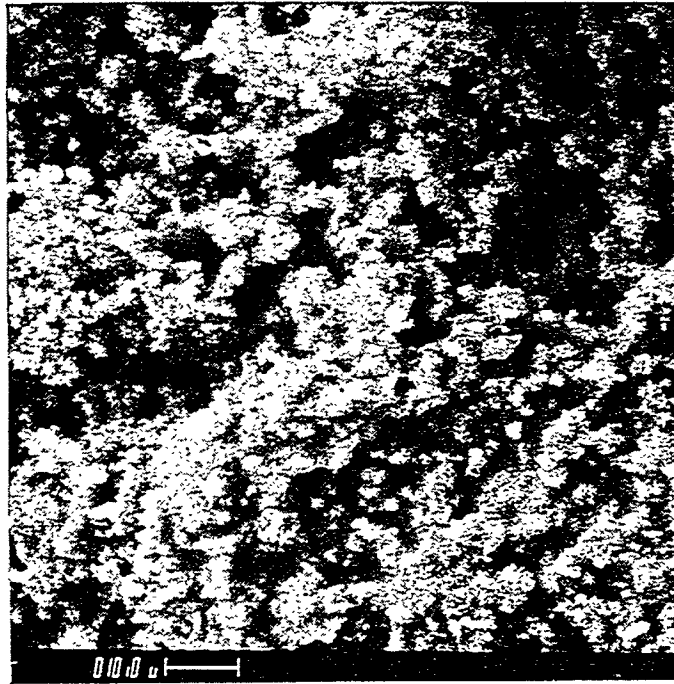


Figure 1. Representative SEM photograph of a fresh fracture surface of an aggregate of ash removed from the ash shedding cone below the middle plenum at Tidd on October 27, 1994 (ID # 4114) taken at 1000X.



ANALYSIS OF TIDD ASH SAMPLE

We measured the size distribution of a sample of ash from the Tidd APF that was sent to us by E. E. Smeltzer of Westinghouse Electric Corporation. Cumulative and differential presentations of the size distribution data were obtained with a centrifugally augmented sedigraph are shown in Figure 2. The D_{50} of this distribution is $7.2 \mu\text{m}$, the $D_{16} = 3.0 \mu\text{m}$; and the $D_{84} = 20 \mu\text{m}$. For this analysis, the ash particles were assumed to have a density of 2.9 g/cm^3 . This distribution is similar to other ashes we have analyzed from the Tidd APF. A sample information form has been forwarded to E. E. Smeltzer so that all available descriptive information corresponding to this sample can be entered in the HGCU data base.

MODIFICATIONS TO THE SRI EMPIRICAL PERMEABILITY MODEL

The mathematical permeability model SRI developed in 1986 was based almost exclusively on empirical data measured for pulverized-coal ashes. One ash produced by atmospheric fluidized-bed combustion was also included in this modeling. The SRI model, which relates the characteristic gas flow resistance of a porous bed of ash particles with the porosity of the bed, was based on the work of Kozeny⁴, Carman⁵, Langmuir⁶, and Davies⁷.

We have used this model in our analyses of bulk samples of HGCU ashes to estimate the characteristic gas flow resistance of filter cakes formed from the ash being analyzed. Two key parameters determine this characteristic flow resistance: porosity and particle diameter. However, during the development of the model, we found that simple measurements of physical diameter obtained with various devices (Coulter Counter, Bahco Classifier, or Shimadzu centrifugally augmented sedigraph) do not correlate well enough with measured permeability data to accurately predict gas flow resistance. Therefore, we used our empirical permeability data to define the drag-equivalent diameter, a value we calculate for each ash tested, which accurately ranks the gas flow resistance of different ashes at equal filter cake porosities.

As our data base of HGCU ashes expanded, we found that the permeability data we measured for these ashes comprising small, irregular particles was not satisfactorily fit by the model developed in 1986 primarily for larger, spherical particles. In almost every case, the 1986 model would tend to overestimate the gas flow resistance values of uncompacted filter cakes. To correct these estimates, we used permeability data measured for 34 ashes composed of small, irregular particles to develop an adjusted permeability model. Most of these 34 ashes were generated at HGCU facilities. The new adjusted equation is presented below:



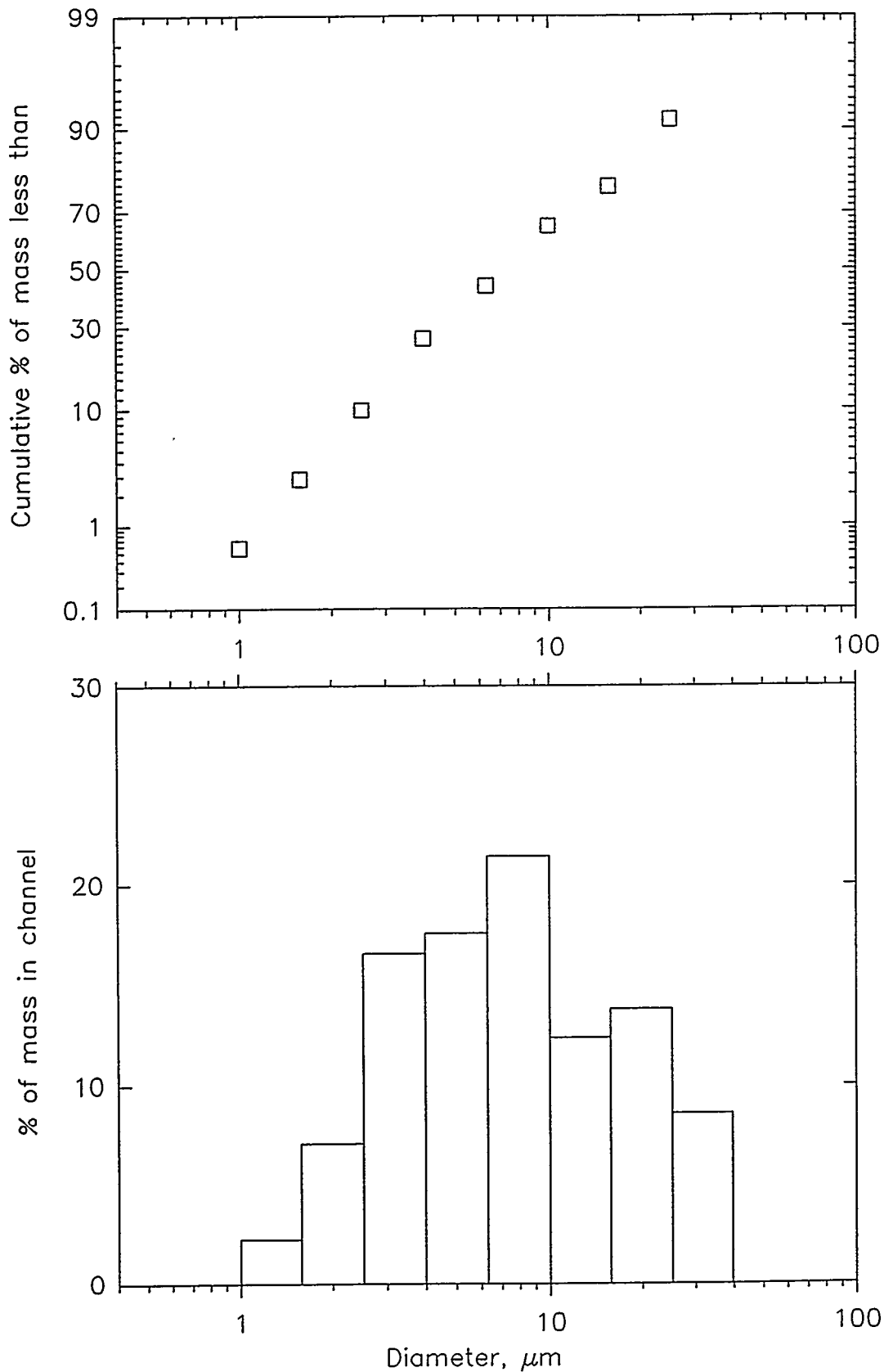


Figure 2. Cumulative and differential size distributions of the ash from the Tidd APF (ID # 4133) measured with a sedigraph on the basis of Stokes' diameters. The D_{50} of this distribution is 7.2 μm , the $D_{16} = 3.0 \mu\text{m}$; and the $D_{84} = 20 \mu\text{m}$. For this analysis, the ash particles were assumed to have a density of 2.9 g/cm^3 .



$$\Delta p = (\mu U/D^2) \cdot (W/\rho) \cdot [111 - 211\varepsilon + 100\varepsilon^2]^2 \quad (1)$$

where:

Δp = pressure drop across the porous bed, μbar

μ = gas viscosity, poise

U = face velocity of the gas, cm/s

D = drag-equivalent diameter of the ash, cm

W = areal mass loading of the porous bed, g/cm^2

ρ = average true density of the ash particles, g/cm^3

ε = porosity of the porous bed, dimensionless ($0 < \varepsilon < 1$).

This new, adjusted form of the SRI permeability model was then used to recalculate the drag-equivalent diameters and relative gas flow resistances of all of the HGCU ashes for which we had measured permeability. These new values have been entered in the interactive data base.

TRANSLATING LABORATORY PERMEABILITY DATA TO HGCU CONDITIONS

Measuring the permeability of simulated filter cakes formed from HGCU ashes is a key component of our ash characterizations. These permeability measurements are used to estimate or rank the on-line filtering pressure drop associated with actual filter cakes formed from these ashes. To generate reliable estimates from our laboratory measurements, it is crucial that the key characteristics of the filter cake and flue gas as they exist during high temperature filtration are accurately known.

As discussed in the preceding section, the permeability of the filter cakes depends in part on the morphology of the filter cake (expressed by the drag-equivalent diameter and the porosity of the cake). The amount (areal loading) of filter cake and the true density of the ash particles also influence the overall pressure drop associated with a given filter cake. The other factors that determine the pressure drop (face velocity and gas viscosity) are dependent on the characteristics of the flue gas. Our laboratory measurements of bulk ash permeability and the porosity of filter cakes removed from HGCU filters in combination with SEM observations of actual HGCU filter cakes have allowed us to accurately characterize the morphology of these cakes. Assessments of areal loading must be performed on site. True particle density has been accurately determined in the laboratory for many ash samples obtained from HGCU filters and is unchanged at HGCU flue gas conditions. Face velocity and flue gas viscosity are significantly different in HGCU filters than in the laboratory. However, the overall effect of increased temperature and pressure can be accurately estimated based on filtration theory and basic theories of fluid dynamics.

Because we have accurate characterizations of the morphology of the filter cakes in operating HGCU filters, translating our laboratory permeability data (acquired at ambient temperature and pressure) to HGCU operating conditions is relatively simple. The

HGCU filter vessel cause changes in gas volume and gas viscosity. In turn, these two factors affect the filtering pressure drop. Based on the conditions included in Table 2, equation (2) demonstrates the translation from laboratory to HGCU conditions for a typical HGCU facility operating at 10 atmospheres and 1600 °F.

Table 2
Comparison of Laboratory with HGCU Filter Conditions

condition	Laboratory conditions	HGCU conditions	effect on pressure drop
temperature, °K	295	1144	X 3.88
gas viscosity, poise	184	456 *	X 2.48
pressure , Atm	1.0	10	X 0.1

* These calculations use the values associated with air to approximate flue gas.

$$\Delta p(\text{HGCU conditions}) = \Delta p(\text{laboratory conditions}) \times 3.88 \times 2.48 \times 0.1 \quad (2)$$

Coincidentally, these multiplicative factors to combine to an overall factor of approximately 1.0. Therefore, when we measure the permeability of a simulated filter cake at laboratory conditions, the values we obtain apply directly to filter cakes in operating HGCU filters.

DATA BASE DEVELOPMENT AND FORMATTING

SRI currently maintains a collection of 209 ashes obtained from ten facilities involved in METC's HGCU program. An overview of the sources of these samples is presented in Table 3. Approximately half of these samples have been subjected to selected laboratory analyses. We are in the process of structuring the data describing these samples into an Excel spreadsheet. Almost all of the simple data (such as chemical analyses, specific surface area, uncompacted bulk porosity, etc.) have already been entered. There are a number of analyses and tests we have performed for a very limited number of samples of special interest that may be difficult to present in a conventional data base. Examples of these kinds of data include SEM photographs, SEM analyses (elemental and X-ray mapping) of individual ash particles, and baking and consolidation tests performed on bulk ash samples prepared in the laboratory. We have not yet decided how to include these types of specialized data in the data base.



Table 3
Overview of Ash Samples in the HGCU Data Base

# of samples	source	process
7	New York Univ.	Bubbling Bed PFBC
8	M.W. Kellogg	Circulating PFBC
14	M.W. Kellogg	gasification
2	Texaco	gasification
11	Grimethorpe	Circulating PFBC
9	KRW	gasification
2	Allison	coal-fired combustion turbine
10	Foster Wheeler	carbonizer
7	Foster Wheeler	Circulating PFBC
2	Iowa State University	AFBC
45	Karhula	Circulating PFBC
92	Tidd	Bubbling Bed PFBC

DESIGN OF A HIGH-TEMPERATURE UNCOMPACTED BULK POROSITY TEST DEVICE

As part of our effort to characterize HGCU ashes, we are designing a laboratory-test device that we will be able to use to form uncompacted beds of ash at temperatures up to 1700 °F. Measurement of the uncompacted bulk porosity of the HGCU ashes is highly desirable for two reasons. Because the permeability of a filter cake is strongly dependent on its porosity, the influence of temperature on the porosity of HGCU aggregates will help predict and explain filtering pressure drop encountered in HGCU filters. Furthermore, the physical tensile strength of aggregates obtained from the Tidd APF is strongly correlated with aggregate porosity. As with permeability, it is crucial to determine the effects of temperature on the strength of ash deposits such as those found in operating APFs.

A sketch of our preliminary design is shown in Figure 3. The device basically consists of a heated tube, open at the top, with a porous disk forming the bottom of the tube. Ash will be sifted or elutriated into the open top of the tube, and the particles will be heated to 1700 °F by the time they fall (or are transported by gas flow) through two heated zones of the tube and reach its bottom. After enough ash has been sifted into the preheated device to fill and cover the quartz ring, a guillotine-type blade will be slid across the top of the ring to isolate the amount of ash sample that was required to exactly fill the volume of the ring. After the blade is securely positioned over the top surface of the ring, the device will be allowed to cool down. (For some tests, the isolated ash sample may be allowed to remain at the test temperature for extended periods to determine the relationship between the time of exposure to high temperatures and the porosity of the aggregate.) The device will then be partially disassembled to allow the ash in the ring to be separately weighed.



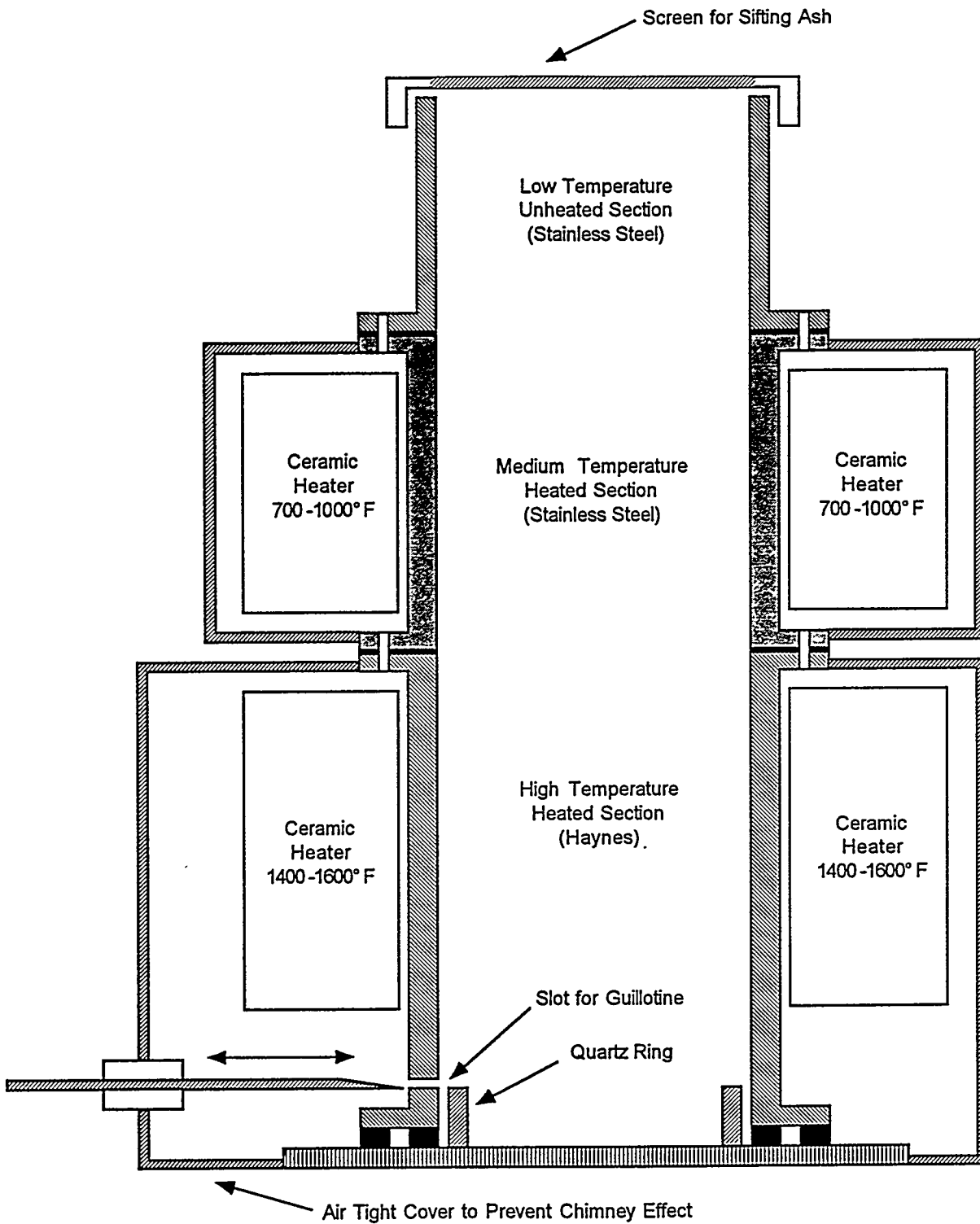


Figure 3. Preliminary conceptual design of the elevated temperature uncompact bulk porosity test device.



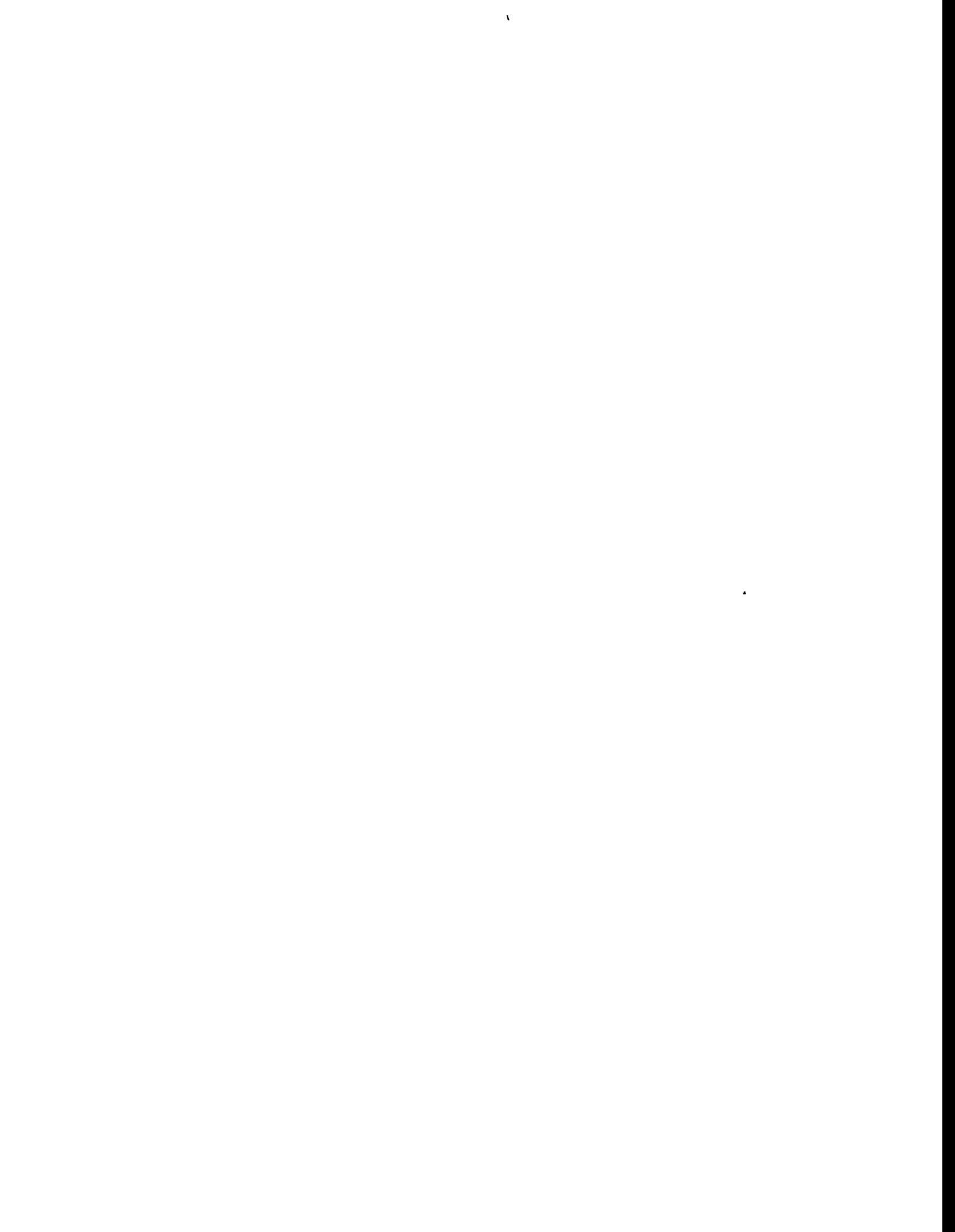
The sample will also be examined to determine if it consolidated after being deposited within the volume of the ring. In the currently envisioned design, this device will operate only at ambient pressures.

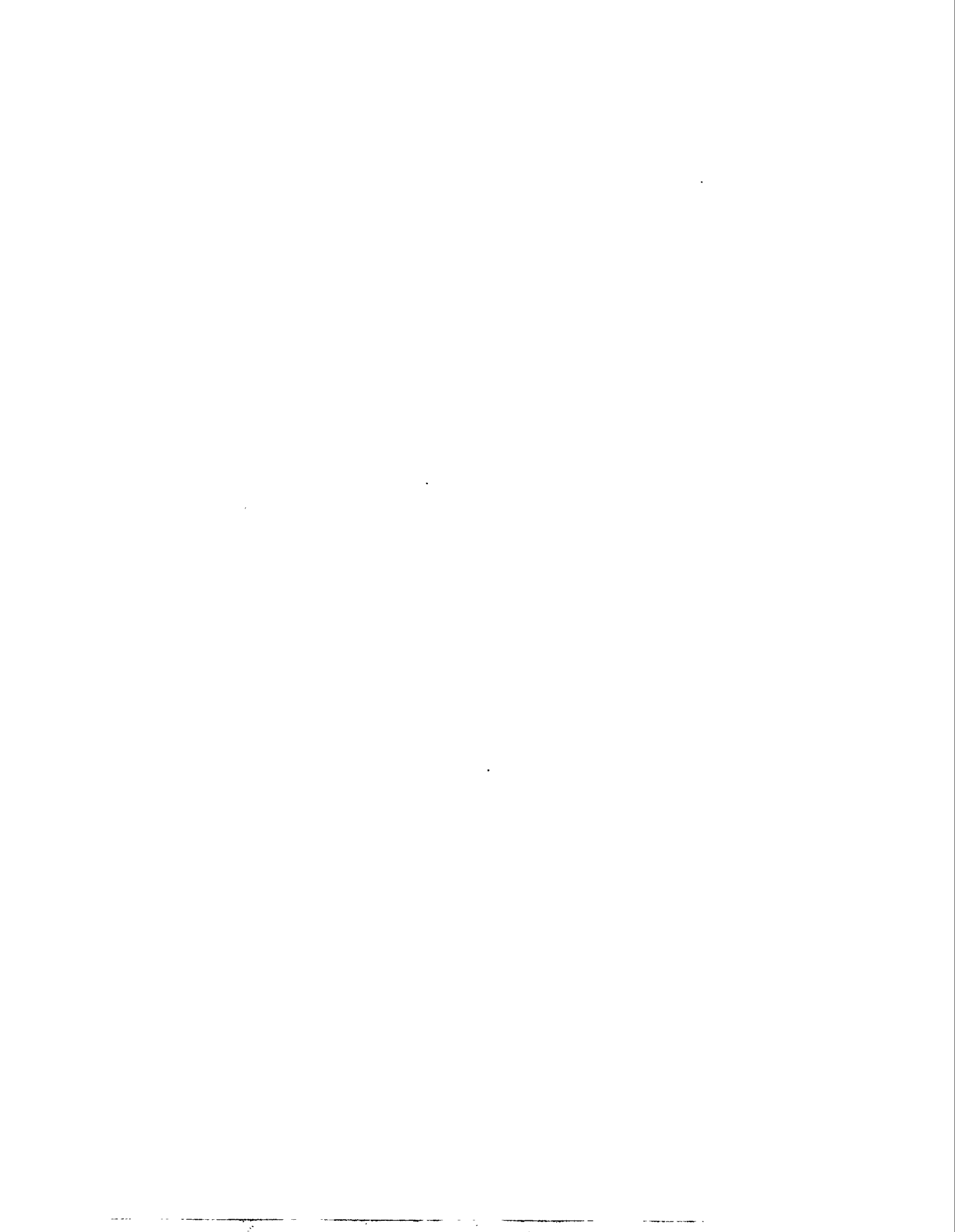
FUTURE WORK

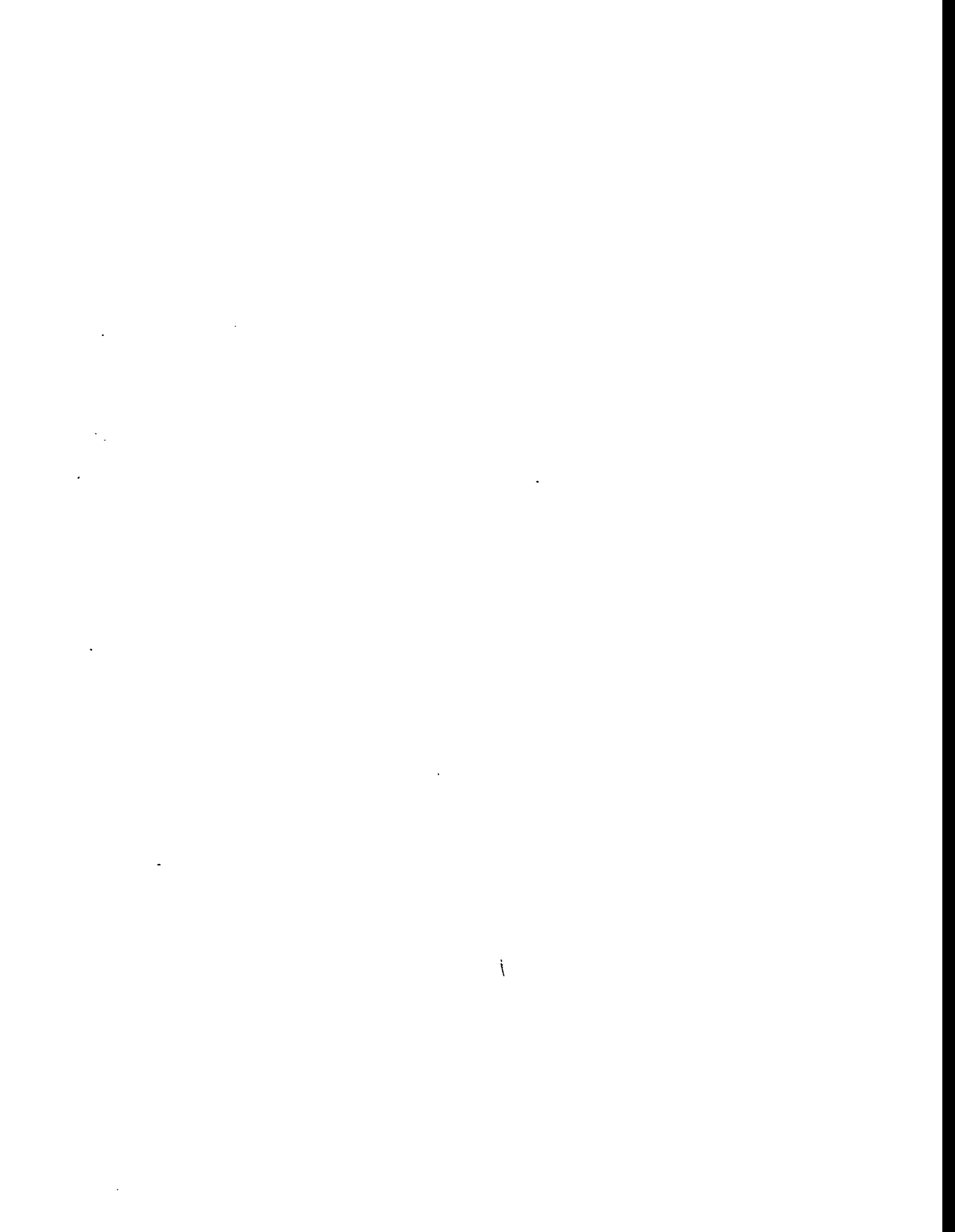
Plans for the next quarter include completion of the design of the uncompacted bulk porosity test device mentioned above, continued work on the interactive data base of HGCU ash characteristics, and a site visit to Tidd to collect additional samples for analysis and to observe and characterize the condition of the ash deposits within the APF.

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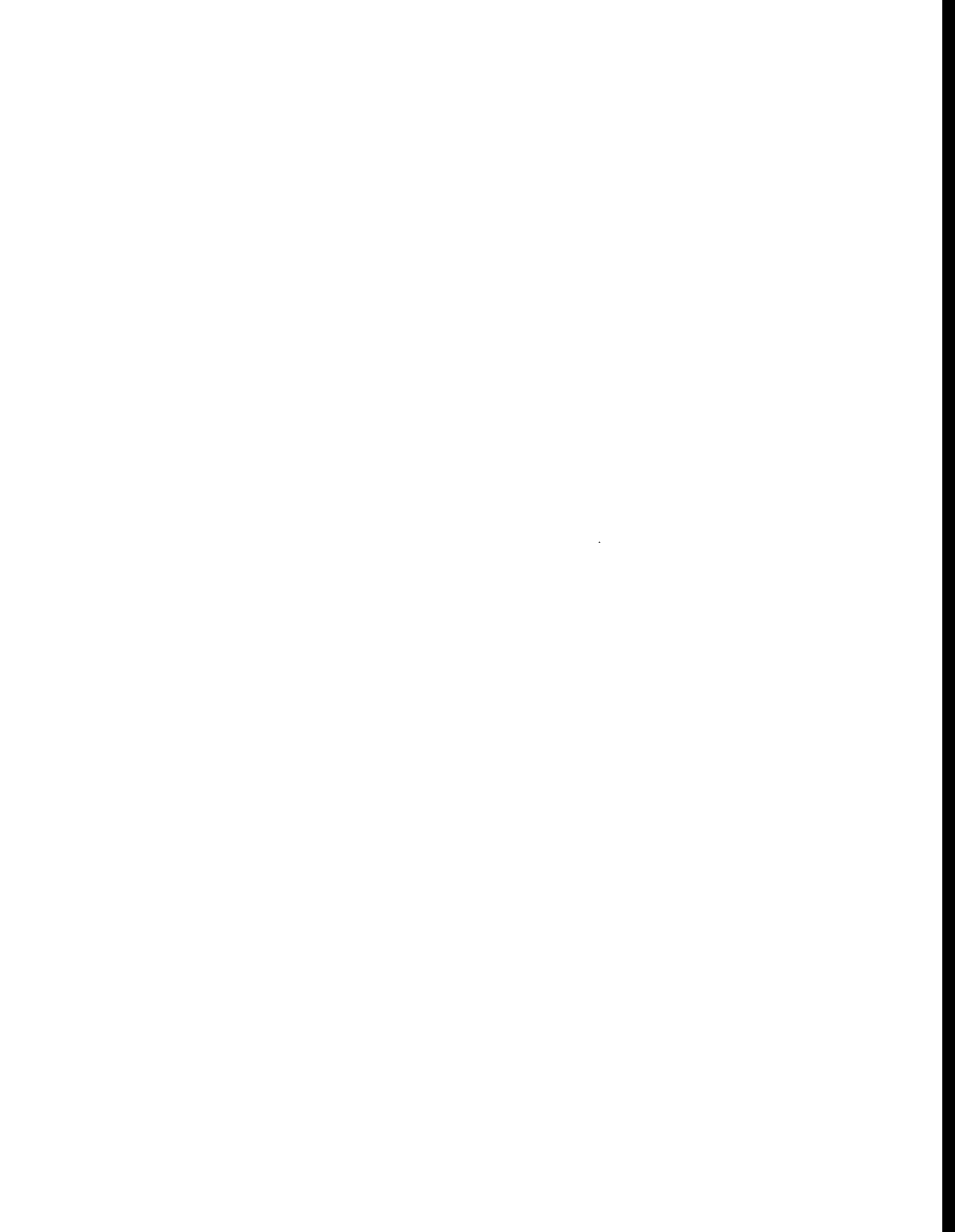








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**PARTICULATE HOT GAS STREAM CLEANUP
TECHNICAL ISSUES
Task 1.0 -- Assessment of Ash Characteristics**



**Particulate Hot Gas Stream Cleanup
Technical Issues
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**Quarterly Report
January - March 1995**

D.H. Pontius

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