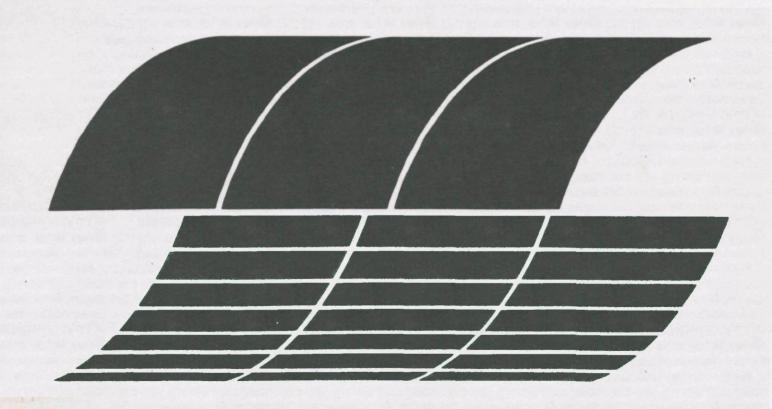
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# Miniplant and Bench Studies of Pressurized Fluidized-bed Coal Combustion: Final Report

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# Miniplant and Bench Studies of Pressurized Fluidized-bed Coal Combustion: Final Report

by

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Office of Research and Development
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### **ABSTRACT**

The pressurized fluidized bed combustion (PFBC) of coal and regeneration of spent sorbent were studied in the continuous 480 lb coal/hr (220 kg/hr) "miniplant" and the 28 lb coal/hr (13 kg/hr) bench PFBC units. The ability of the PFBC system to reduce SO2 emissions by 90% or more was demonstrated using both dolomite and limestone sorbents. The dynamic response of SO2 emissions to sudden changes in the sulfur content of the coal, and in the dolomite to coal feed ratio, was also measured. NO $_{\rm X}$  control tests carried out in the bench scale unit indicated that either two stage combustion or ammonia injection could reduce NO $_{\rm X}$  emissions below the already low levels normally measured in PFBC.

Particulate emissions were studied using a number of particulate control devices. A three stage cyclone system was shown to be unexpectedly efficient. However, it could not consistently reduce particulate emissions to the level required by the current New Source Performance Standard for utility boiler. A high temperature/pressure ceramic fiber filter was successfully tested, giving collection efficiencies high enough to meet current emission standards. Further testing of a granular bed filter was unsuccessful due to poor removal efficiencies and various other operating problems. Conventional low temperature/low pressure electrostatic precipitator and bag house systems were also tested. Both were shown to be capable of reducing particulate emissions, following the three stages of cyclones, to levels meeting the current New Source Performance Standard.

Regeneration of spent sorbent was studied in the miniplant and bench units. In the miniplant, a series of extended tests were completed which confirmed the operability of the system and the large reduction in fresh limestone resulting from the use of regeneration. An approximate four fold reduction in fresh limestone feed requirements results from the use of regeneration. SO2 retentions were in excess of 90% at all times. No loss in sorbent activity due to regeneration was observed. Bench scale tests indicated that coal could probably be used as the fuel for regeneration in place of natural gas used in the miniplant program.

A series of sampling campaigns was completed which generated samples used by another contractor for Level I and Level II comprehensive analyses of emissions from PFBC.

This report is submitted in fulfillment of Contract Number 68-02-1312 by Exxon Research and Engineering Company under sponsorship of the Environmental Protection Agency. Work was completed in August 1979.

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### SECTION I

### SUMMARY

The pressurized fluidized bed combustion of coal (PFBC) and regeneration of sulfated SO2 sorbent were studied in the continuous miniplant unit and the semi-continuous bench unit. In the miniplant combustion program, a series of runs were made to verify that PFBC could attain the 90% SO2 retention level using dolomite or limestone sorbents. The dynamic response in SO2 emissions to a sudden change in the sulfur content of the coal, and in the dolomite to coal feed ratio, was also measured. Emission of  $NO_X$ , other gases and particulates was measured. Combustion efficiency results were updated.

The measurement and control of particulate emissions was studied. Various particulate measurement methods were employed and cross checked. Particulate emission control using cyclones, and using cyclones in series with a high temperature/pressure ceramic fiber filter and with a granular bed filter was studied. Particulate removal from the flue gas at low temperature/pressure was also studied, using a conventional electrostatic precipitator and bag house.

Regeneration of sulfated limestone was studied in a series of extended tests. A series of sampling campaigns was also completed in which samples were taken which were used to conduct a comprehensive analysis of emissions with and without regeneration.

Combustion and regeneration studies were also carried out in the bench unit.  $NO_X$  control methods were evaluated in a series of combustion tests. The use of coal as the regenerator fuel was also studied.

### COMBUSTION STUDIES

The miniplant combustor consists of a refractory lined vessel 10 m (33 ft) high with an inside diameter of 32 cm (12.5 in). A number of vertical water-cooled tubes are mounted in the combustor to remove the heat of combustion.

Premixed coal and sorbent are injected into the combustor a single point 28 cm (11 in) above the fluidized bed support grid. The combustor normally operates at pressures up to 950 kPa (9 atm), at temperatures up to the ash agglomeration temperature of the coal (usually less than  $960^{\circ}\text{C}$ ), at superficial velocities of up to 2 m/s (7 ft/sec) and with expanded beds of up to 3.3 m (12 ft). The coal feed rate is normally less than 160 kg/hr (350 lb/hr). Flue gas leaving the combustor passes through three cyclones in series to remove most of the particulate matter. Particulates captured in the first cyclone are recycled to the combustor to improve combustion efficiency. Particulates captured in the second and third stage cyclones are rejected through lock hoppers. Spent sorbent is also rejected from the combustor through a lock hopper system to maintain a constant bed level in the combustor.

Runs were made with: an Eastern bituminous Pittsburgh seam coal (Champion) containing about 1.5% sulfur with a particle size distribution of 300 to 2400 microns; an Illinois No. 6 coal containing about 3.5% sulfur screened to a particle size runge of 400 to 3400 microns; and an Ohio coal (Valley Camp) containing 2.5% sulfur screened to a size range of 300 to 3400 microns. A Virginia limestone (Grove No. 1359) and an Ohio dolomite (Pfizer No. 1337) were used. Both were screened to a size range of 700 to 2400 microns.

Operational performance of the combustor was very good. At the completion of the program in August 1979, over 3700 hours of coal combustion time was accumulated. Twelve runs of 100 to 250 hours duration were included in the total. The ability of the miniplant to conduct short daily runs was also demonstrated. In one period, fourteen 8 to 10 hour runs were completed in fifteen working days. Multiple purpose runs were also conducted. In some cases, as many as three EPA contractors, in addition to Exxon, were present at the same time, conducting sampling and equipment evaluation tests during extended combustion runs. During this reporting period, a total of 1117 hours of test time was dedicated to a materials evaluation program sponsored by the Department of Energy (DOE), under a cooperative agreement between DOE and EPA. Gas turbine and boiler tube materials were evaluated under realistic PFBC operating conditions. Evaluation of test results will be made by General Electric and Westinghouse, the DOE contractors involved in the materials test program.

SO<sub>2</sub> retention studies for EPA were conducted to confirm the sorbent requirements, indicated in earlier tests, needed to assure 90% or higher SO<sub>2</sub> retention. The work was done in support of the most recent New Source Performance Standards for large coal fired boilers. A number of runs were made with Ohio and Illinois coals, dolomite and limestone sorbents at various calcium to sulfur (Ca/S) molar ratios. It was found that 90% and higher SO<sub>2</sub> retention levels could be reached with either sorbent. In fact, retention levels as high as 99<sup>+</sup>% were measured. As expected, dolomite was shown to be more reactive. A Ca/S ratio of 1.5 will assure 90% SO<sub>2</sub> retention at a gas superficial residence time of 2 s while limestone use will require a Ca/S ratio of between 3.5 and 4.0.

The recent SO2 retention results obtained with dolomite sorbent were analyzed using the simple first order kinetic expression developed previously (2). The recent data did not show the expected effect of gas residence time on SO2 retention. However, the lack of a gas residence time effect may have been due to the fairly narrow range of residence time variations. It may also indicate that the reaction rates may be affected in larger FBC units such as the miniplant, by other operating parameters such as solids recycle and rejection. These factors may make it difficult to obtain satisfactory results using simple rate expressions, the rate expression must be refined to account for these factors. However, this rate expression can be used as an approximate guide until a better understanding of the FBC system is developed.

The dynamic response of the SO<sub>2</sub> emissions to sudden changes in the sulfur content of the coal, and in the Ca/S ratio, was also studied. A new auxiliary coal/sorbent feed system was built and installed to permit rapid switching

from the current feed system to the new one to cause the rapid change in coals and coal/sorbent ratios. The time to switch from one system to the other was about 40 s. The work was done in cooperation with General Electric, another EPA contractor, who is developing a dynamic  $SO_2$  response model for assessing automatic control options. It was found that the  $SO_2$  emissions were stabilized in 7 to 8 minutes following a step change in the sulfur content of the coal from 1.9 to 3.4%. However, when the Ca/S ratio was changed suddenly from 1.4 to 0.4, the time to stabilize was 110 min. The change back to the higher Ca/S ratio required 300 min to reach a stable emission level. The reason for the difference in the two response times (110 and 300 min) is not apparent. However, it is clear that the response to a change in the Ca/S ratio has a time constant much greater than that for a change in the coal sulfur content. These results will be used by General Electric to develop the emission response model model. The model will be described in a separate report by General Electric.

 $\rm NO_X$  emissions measured recently follow the same trend line with excess air developed in earlier studies. At an excess level of 20%,  $\rm NO_X$  emissions averaged 0.2  $\pm$  0.1 lb/MBTU. Even at excess levels in the range of 60 to 100%, emissions are no greater than 0.4 lb/MBTU. Therefore,  $\rm NO_X$  emissions are still well within the New Source Performance Standard even with the recent reduction in the NSPS to 0.6 lb/MBTU.

SO3 emissions were measured using the controlled condensation method in place of the older Method 8 method. Method 8 results averaged 12 + 12 ppm, controlled condensation results averaged 6 + 9 ppm. In both cases the range of values was 0 to 30 ppm. Although the controlled condensation method gave an average value one half that of the Method 8 average, the uncertainty ranges were so wide that the difference may not be significant. No positive conclusions could be drawn from the results regarding the cause or the factors affecting the degree of  $SO_3$  formation. The reduced sulfur compounds,  $H_2S$ , COSand CS2, were found to be less than the detectability level of 1 ppm in the flue gas. Methane averaged  $7 \pm 5$  ppm, ethane  $4 \pm 4$  ppm,  $C_3$  through  $C_6$  hydrocarbons were generally less than 1 ppm, the detectability limit. Sodium and potassium present in the flue gas were measured by quenching and filtering a flue gas sample, then extracting the collected particulates and sample system with hot water. Sodium concentration in the extract was equivalent to 2 to 3 wppm in the flue gas. Potassium was found to be between 0.3 and 0.5 wppm in the flue gas. No vanadium was detected. Chloride levels of about 50 wppm in the flue gas were also measured.

Particulate emissions in the flue gas, after passage through three conventional cyclones in series, generally ranged from 0.03 to 0.15 g/Nm³, compared to the NSPS of about 0.035 g/Nm³ (13 ng/J) for particulates. The particulates generally had a median particle size of 1 to 2  $\mu m$ , with 80 to 90% smaller than 5  $\mu m$ .

Carbon combustion efficiency measurements averaged 99.3% for the runs made during this period, about 0.8% higher than expected based on the correlation published in the previous report (1).

### PARTICULATE MEASUREMENT AND CONTROL

An important technical issue to be resolved before pressurized fluidized bed combustion can be applied commercially is the degree of particulate removal needed to protect the gas turbine. A related issue is which technology to use to achieve the needed degree of the particulate control. In addition to meeting the particulate removal requirements set by the gas turbine, the environmental New Source Performance Standards published by the Environmental Protection Agency must also be met. Although the EPA is not directly concerned with studying particulate control to protect gas turbines. it is responsible for the evaluation of such particulate control devices to the extent that these devices could determine the level, size and composition of particulate matter emitted from a PFBC system. Therefore a particulate control program was begun in which pre-turbine devices such as granular bed filters, a ceramic fiber filter and high efficiency cyclones were evaluated. In addition, post-turbine devices were also tested, based on the realization that the degree of pre-turbine cleanup may not be sufficient to meet the more stringent New Source Performance Standards for particulates. In this case, a trailer mounted electrostatic precipitator (ESP) and a trailer mounted bag house were connected to the miniplant flue gas system and tested under typical low pressure, low temperature conditions.

In addition to evaluating the particulate control devices, it was also necessary to develop and improve particulate measurement systems. Such systems were used to determine particulate concentrations in the flue gas before and after the control devices and to measure particulate size distributions. For most of the current program, particulate concentrations were measured by passing isokinetic samples of the flue gas through high temperature total fil-Particle size distribution was measured using a Coulter Counter on a sample of particulate captured on the filter. A question arose as to whether the size distribution measured by the Coulter Counter was the same as that occurring in the flue gas. The degree of particulate agglomeration on the filter and redispersion during the Coulter Counter measurement were unknown. and could affect the measured particle size. The primary concern was that redispersion in the Coulter Counter resulted in a particle size distribution significantly finer than that actually existing in the flue gas. To answer the question, a series of samples were obtained by Southern Research Institute and Air Pollution Technology, Inc. using a high temperature, pressure cascade impactor developed by Air Pollution Technology. This is a different particulate sampling system which would not cause possible agglomeration and redispersion effects. The impactor results indicated a larger concentration of fine particles and a mass median size of about 0.8 m in the flue gas following the third cyclone, compared to 1.6 m measured by the Coulter Counter. Electron micrographs of the material captured on the impactor stages also showed very little sign of agglomeration. These findings indicated that the results of the filter/Coulter Counter method differed somewhat from cascade impactor results, but to the degree expected, based on measurements made in other particulate systems. The Coulter Counter is definitely not biased toward the finer particles by breakdown of agglomerates in the Coulter Counter during sample preparation. A cross check was also made between the Coulter Counter and a Bahco inertial system. Both devices gave very similar particle size distributions. An attempt was also made to use a miniature five stage cyclone

train to obtain particulate concentration and size distributions. Satisfactory particle size distributions could not be obtained with this system due to overlapping size differentiation on the cyclone stages. A continuous particle monitor was also tested but did not perform satisfactorily. This device measured the rate of transfer of electrical charge from particles in the duct to a stationary rod as the charged particles impinged on the rod. The system was very sensitive to temperature changes and was not reliable. It was concluded that either the filter/Coulter Counter system or a cascade impactor system was satisfactory. The filter/Coulter Counter system is easier to use and probably gives a better measure of particulate concentration, but gives a coarser size distribution.

Particulate control studies included the testing of the three-cyclone system installed on the miniplant in addition to a number of other systems. The second cyclone was found to be 90 to 95% efficient, reducing the concentration of particulates entering the cyclone with a mass median diameter of 20 to 25 µm to 0.4 to 1.2 g/Nm<sup>3</sup> in the cyclone outlet. The outlet particulate stream had a median size of 3 to 5 um. The third cyclone was found to be about 90% efficient, reducing the concentration of particulates entering it with a mass median diameter of 3 to 5  $\mu$ m to 0.03 to 0.15 g/Nm<sup>3</sup> in the outlet. Particulate in the flue gas leaving the third cyclone had a mass median particle size of 1 to 3  $\mu m$ . The efficiency of third stage cyclone was independently verified by a team from Southern Research Institute and Air Pollution Technology, Inc., using the same high temperature/pressure cascade impactor system mentioned previously. The impactor results verified those measured using total filters and Coulter Counter particle size measurements. Both methods indicated the cyclone cut diameter (particle size captured with 50% collection efficiency) to be about 0.7 to 0.9  $\mu m$ . The measured efficiency is significantly greater than that expected from theoretical efficiency models. The reason for the difference is not known.

Although the cyclone system, at times, did just meet the EPA New Source Performance Standard for particulates (0.035 g/Nm³), it did not do so consistently. Further studies were carried out with cyclones of differing designs to see if the performance could be optimized and higher efficiencies obtained. Two new cyclones were built similar to those currently being tested at the National Coal Board PFBC facility in Leatherhead, U.K. They were tested on the miniplant flue gas in place of the existing conventional third stage cyclone. Contrary to expectations, the new cyclones were found to be no more efficient than the existing third stage cyclone. Also, no significant effects of pressure, inlet velocity, temperature or coal type on cyclone performance were found. An increase in inlet particulate concentration did increase overall efficiency significantly. Comparisons will be made with the performance of the similar cyclones at the National Coal Board PFBC unit as the data become available.

Tests were also conducted on the performance of a high temperature/pressure ceramic fiber filter provided by Acurex Corporation. A single cylindrical filter element consisting of a mat of Saffil alumina, was mounted in a heated pressure vessel and used to filter a slipstream of flue gas extracted between the second and third cyclones. The filter was cleaned periodically by a combination of reverse flow of compressed air and short, high pressure pulses.

Pressure drops were continuously recorded and were found to be 0.2 to 2 kPa after cleaning, rising to a maximum of 14 kPa before cleaning. Filtration efficiencies were high, ranging from 96 to 99.5%. Outlet particulate loadings averaged 0.013 g/Nm<sup>3</sup>, well under the EPA emission standard for particulates and significantly lower than that obtained with the three stage cyclone system. An extended test was attempted but was terminated after 19 hours due to failure of the filter. The failure was caused by a combination of corrosion of the very fine filter support screen and excessively high reverse pressure drops during bag cleaning. Corrosion of the support screen was probably accelerated by a failure in the temperature control system during this test. The support screen used in these tests was the only type available at the time and it was recognized that it would have a short lifetime at test conditions. The tests did indicate the ceramic filter was operable under PFBC conditions. High collection efficiency at high face velocities was measured. In general, the test filter exhibited performance similar to that of a conventional bag filter. However, additional work is needed to develop a better mechanical support system to prevent bag failure.

A granular bed filter was also evaluated as a high temperature, pressure particulate control device. This is a system in which particulates are removed by passage through a bed of granular material. A number of small beds operated in parallel are used to reduce the pressure drop. The beds are periodically cleaned by the reverse flow of clean gas. The "blow back" gas fluidizes the granular filter media at a velocity sufficient to blow the collected dust off, but low enough to prevent blowing the filter media itself out of the filter vessel. The removed dust settles outside the filter and is collected at the bottom of a containment vessel. Previous studies with this system were beset with operating problems, primarily caused by plugging of the filter inlet sections with particulates, poor bed cleaning and loss of filter media during cleaning. Filtration efficiency was low and furthermore, decreased with time. Modifications were made to the system in an attempt to improve performance. This included testing a number of inlet screen sizes, using baffles to prevent the loss of filter media and testing filter media of various sizes and densities, also to prevent loss during the cleaning step. However. the test program was again unsuccessful due to poor filtration efficiency, loss of filter media and other serious operating problems which persisted to the end of the program. The lowest outlet particulate concentrations measured were 0.07 to 0.11  $g/Nm^3$ , but the concentrations increased with time during the tests by as much as a factor of three. At no time did the filter performance equal that required by the particulate emission standards. Since the filter was installed after the second stage cyclone, the outlet loadings could be compared to those from the third stage cyclone. The comparison indicated that the cyclone was more efficient and moreover, the efficiency was maintained. The poor filter performance was believed to be due to poor cleaning. Filter beds examined after a test program were generally found to contain high concentrations of dust intimately mixed with the filter media. Tests made with transparent models at ambient conditions showed that the dust was adhesive and only partly removed during blow back. Some of the dust adhered to the filter media and was mixed into the filter bed by the motion of the fluidized filter media. Attempts to improve bed cleaning by modifying the blow back conditions were partly successful. However, outlet concentrations were still high and

increased with time. Attempts to prevent filter media loss were also unsuccessful. Screens were found to plug, even when screen opening sizes as large as 10 mesh were used. Larger filter media and very dense filter media were tested but did not prevent the loss. Baffles were also unsuccessful. Transparent model tests were made and indicated that the loss was probably caused by a surge in the blow back gas flow caused by the sudden opening of the blow back valve. It was also suggested that the bed height was too low, not allowing enough space for disengagement of the fluidized filter media. In addition to these problems, the operation of the filter caused periodic upsets in the miniplant pressure control system, which in turn caused coal feeding problems. Pressure drops across the filter were also high, probably due to poor bed cleaning.

A final attempt was made to determine if an operable granular bed system could be developed. A small single bed filter was installed and tested on a flue gas slipstream after the second stage cyclone. The blow back system was modified to bring in some of the reverse flow gas at the interface between the filter media and the dust layer. This blow back gas was directed horizontally across the top of the bed and was intended to shear off the dust layer without disturbing the filter media enough to cause its loss. A single test was made with the modified filter but was unsuccessful. Pressure drops were very high after the first few minutes of operation. Increasing the severity of the cleanup step again resulted in loss of filter media. Unfortunately, the testing of this filter was terminated due to lack of time before all operability questions could be answered. An unresolved issue was whether the loss of filter media could have been prevented by modifying the blow back procedure, possibly by introducing the blow back gas more gradually. The filter face velocity was also very high in the test and the effect of lowering the velocity on filter performance was also unresolved.

A conventional low temperature, low pressure electrostatic precipitator (ESP) and bag house were also tested on the flue gas from the miniplant in a series of long term tests. The purpose of the tests was to determine if an ESP or bag house could be used after expansion of the flue gas through the gas turbine, to meet particulate emission standards. This assumed that cyclones may be sufficient to protect the gas turbine from excessive wear but would not be sufficient to meet environmental standards. The tests were performed using mobile, trailer mounted systems operated by Acurex Corporation for the EPA. Both systems appeared to be applicable in this service. The ESP overall efficiency was 87%, corresponding to an emission level of about 0.02 g/Nm $^3$ . The bag house overall filtration efficiency was 99.3%, corresponding to an emission level of about 0.001 g/Nm $^3$ .

### REGENERATION STUDIES

A series of extended runs was made in which sulfated limestone was continuously regenerated and recirculated to the combustor. The primary objective of the runs was to determine the reduction in fresh limestone requirement resulting from regeneration of the sulfated limestone.

The regenerator consists of a refractory lined vessel with an inside diameter of 22 cm (8.5 in) and an overall height of 6.7 m (22 ft). Gaseous fuel is burned in a plenum below the fluidized bed to achieve the reaction temperature. Additional fuel is injected directly into the fluidized bed just above the fluidizing grid to create a reducing zone in which the  $CaSO_4$  reduction reaction occurs. Supplementary air is injected directly into the bed at a higher elevation to create an oxidizing zone. The oxidizing environment at the top of the bed assures high selectivity to  $CaO_*$ , the desired product of the regeneration reaction, by minimizing the formation of  $CaS_*$ , an undesired byproduct.

Three extended runs totalling 370 hours were completed in the test series. Operations were generally good. Fresh limestone, at a Ca/S ratio ranging from 0.7 to 1.5 was fed to the combustor. SO2 emissions from the combustor were low, the SO<sub>2</sub> retention exceeded 90% at all times and was normally above 97%. In a once through system, a Ca/S ratio of 3 to 4 would have been necessary to achieve the same level of SO2 retention. The recirculating regenerated sorbent rates were much higher than the fresh limestone rates. giving total Ca/S ratios entering the combustor usually between 6 and 12 (one was 54). The regenerated sorbent appeared to have the same activity of fresh limestone with no sign of diminished activity due to regeneration. There was also no evidence of loss in activity during the runs. Loss of sorbent by attrition and entrainment from the combustor and regenerator was somewhat greater than the corresponding loss from a once through operation. In fact, the fresh limestone Ca/S ratios fed to the combustor were usually those required to maintain constant sorbent levels in the combustor and regenerator. It had been planned to reduce fresh limestone feed rates to the point where the SO<sub>2</sub> retention in the combustor was about 90%. This could not be done, since sorbent feed would have had to be reduced to a point where it would have been inadequate to make up for attrition/entrainment losses, and bed depth would have dropped. In a once through operation, the entrainment losses are about 1% of the bed/hr. In the regeneration runs, the entrainment losses averaged about 1.8%. SO2 concentration in the regenerator off gas was low. ranging from 0.2 to 0.5% equivalent to 6 to 16% of the calculated equilibrium concentration. These levels are lower than what might reasonably be expected in a commercial system. As pointed out in previous reports (1,2), due to the size of the miniplant regenerator, the SO2 level in the miniplant regenerator off gas is determined by energy and mass balance requirements rather than chemical equilibria. It had been planned to use a higher sulfur coal in these tests to maximize the SO2 concentrations. Unfortunately, a prepared high sulfur coal could not be obtained and a lower sulfur coal was used, contributing to the low SO2 concentrations.

However, the runs did establish the minimum degree to which the sorbent requirement would be decreased by regeneration. This is a factor of 3 to 4. The runs also indicated that regenerated sorbent activity was high and activity loss due to regeneration was low.

### COMPREHENSIVE ANALYSIS OF EMISSIONS

The comprehensive analysis of emissions program initiated previously was concluded. In the earlier tests, a Level I sampling and analytical program was conducted in cooperation with Battelle Columbus Laboratory, the EPA contractor responsible for the program at that time. In the current series of tests, a set of samples was taken during a run with Illinois No. 6 coal and analyzed for inorganic elements by spark source mass spectroscopy (SSMS). The results were forwarded to Battelle to be included with the Level I results obtained by them in the earlier tests which used Champion (Eastern) coal. Also, in the current series of tests, sampling campaigns were conducted in cooperation with GCA Corp., the current EPA contractor. In this case, Exxon assisted in sampling but essentially all analytical work will be done, interpreted and reported by GCA. In the recent tests, a Level I sampling campaign was conducted during an extended run in which the regenerator was in operation. A Level II sampling campaign was also conducted with the combustor operating in a once-through fashion.

A brief investigation into the formation of the double salt Mg3Ca(SO4)4 or MgSO4 in the PFBC solid waste products was conducted. These materials, if present, could cause environmental problems upon disposal since they are water soluble. No MgSO4 was found in any of the samples. Mg3Ca(SO4)4 was found in solid samples collected in the third stage cyclone and the gas turbine test section downstream of the cyclone. However, SO2 concentrations in the run from which samples were taken was considerably higher than normal. Also, the residence time in the miniplant ducting is probably greater than that which will occur in a commercial plant and the average temperature somewhat lower. All of these factors promoted the formation of the double salt. Additional tests should be conducted in newer PFBC units to determine the extent of the double salt formation under more realistic conditions.

### BENCH UNIT STUDIES

Programs were carried out in both the bench combustor and regenerator sections. The combustor had been modified to permit continuous solids feeding and removal. The combustor was then used to evaluate three  $\text{NO}_X$  control methods: two stage combustion, NH3 injection and simulated flue gas recirculation. The regenerator was used in a series of tests using sulfated sorbent produced in the miniplant, studying the use of natural gas and coal to fuel the regeneration section.

The bench combustion unit consists of a refractory lined combustor vessel which normally operates at temperatures of 840 to 950°C, pressures of five to eight atm, superficial velocities of 1 to 2 m/s and coal feed rates of 1 to 12 kg/hr. The inside diameter is 11.4 cm, the interior height is about 4.9 m. Three sets of vertically mounted water cooled coils are located inside the combustor to remove heat of combustion. Coal is fed by a pneumatic injection system similar to that used on the miniplant. Sorbent is fed separately by means of a transfer line lock hopper consisting of two cycling valves mounted in the sorbent feed line. Provisions were also added to inject supplementary air into the combustor at various locations to study two stage combustion, and to inject NH3 and N2 to study the effects of NH3 injection and simulated flue gas recirculation on  $\mathrm{NO}_{\mathrm{X}}$  emissions.

Two sets of runs were made studying the effect of two stage combustion. The first was made at 8 atm pressure. The runs were subject to frequent temperature excursions and bed agglomeration caused by low superficial velocities in the bed below the point where the supplementary air was injected. As a result, the test series was not completed. The pressure was reduced to 5 atm to increase the velocity and the second set of tests was run. The location of the supplementary air injector was varied during the initial tests at 8 atm. Injection of the air into the bed, 15 cm above the grid, rather than above the bed, was necessary to promote CO burnout. Despite the problems, some data were obtained during the 8 atm tests which indicated that NOv emissions could be reduced about 30% by using two stage combustion. The test series at 5 atm pressure was completed with no serious operating problems. The supplementary air injection point was raised to 33 cm above the grid to increase the residence time in the lower reducing zone thereby promoting NO reduction reactions. However, the runs at 5 atm resulted in only a 10 to 20% reduction in NO<sub>2</sub> emissions. The increase from 10 to 20% reduction occurred as the amount of primary combustion air was decreased from 90 to 75% of the stoichiometric amount at a fixed level of total combustion air (primary plus supplementary). The reason for the apparent effect of pressure on the effectiveness of two stage combustion is not known. NO<sub>X</sub> emissions were also shown to increase with overall excess air and temperature as had been noted in previous studies (1.2).

Staged combustion increased SO<sub>2</sub> emission levels about 20%. The effect was believed to be caused by low oxygen concentrations in the reducing zone, depressing the calcium sulfation reaction which requires the presence of oxygen. CO emissions were increased up to 20% by staging, but only at low excess air levels. The increased SO<sub>2</sub> and CO emissions could be offset by increasing the gas residence time in the oxidizing zone slightly. A more serious concern is the effect of two stage combustion on boiler tube materials. This potential problem must be addressed if two stage combustion is to be studied further.

NH3 injection was also evaluated as a means of reducing  $NO_x$  emissions from PFBC. The use of NH3 to reduce NO<sub>X</sub> emissions was developed by Exxon Research and Engineering Company and successfully applied to conventional oil, gas and coal fired furnaces. The current program was designed to test the effectiveness of NH3 injection under PFBC conditions. A series of tests was conducted by injecting NH<sub>3</sub> at NH<sub>3</sub>/NO ratios ranging from 1 to 8 at three locations, below the grid into the combustion air, into the combustor at the bed overflow port (168 cm above the grid) and into the combustor freeboard region, 290 cm above the grid. Combustor pressure was approximately 7 atm during the tests. The effect on  $NO_x$  emissions was found to be a strong function of location and the NH<sub>3</sub>/NO $_{\rm X}$  ratio. Injection below the grid into the combustion air resulted in a 50% increase in NO $_{\rm X}$  emissions. Injection at the bed overflow port had no effect on emissions. Injection in the freeboard zone decreased  $NO_X$  emissions 30 to 50%. Increasing the  $NH_3/NO_X$  ratio increased  $NO_X$ emissions at the below-grid location, but decreased emissions at the freeboard zone location. The results are probably related to local oxygen concentrations and temperatures. Injecting below the grid exposed  $NH_3$  to high  $0_2$  concentrations and high temperatures near the coal feed point. These conditions

promoted NH $_3$  oxidation rather than the NO/NH $_3$  reaction. In the freeboard zone, the lower temperatures (~700°C) and  $0_2$  concentrations favored NO reduction. At the intermediate location, the temperature, and possibly  $0_2$  concentration conditions, promoted each of the competing reactions equally, resulting in no significant effect on NO $_{\rm X}$  emissions. The optimum temperature for the NH $_3$ /NO reaction found in this study (~700°C) is lower than expected based on previous studies and further work may be needed to determine if it is the true optimum. If such a temperature is required, NH $_3$  could be injected ahead of the gas turbine. The desired temperature would occur within the turbine itself, where the NO $_{\rm X}$  removal reactions would take place.

The effect of flue gas recirculation on  $NO_X$  emissions was simulated by adding nitrogen to the combustion air inlet stream. The ratio of  $N_2$  to air was set at 0.10 vs 0.2. Excess air and temperature were also varied. The use of  $N_2$  addition was found to cause an apparent decrease in  $NO_X$  emissions. However, the addition of  $N_2$  always caused a decrease in the excess air level since more coal had to be fed to heat up the cold  $N_2$  added to the inlet air. When the results were plotted against excess air and compared to results obtained without  $N_2$  addition, no significant effect of  $N_2$  addition was seen. Therefore, it is concluded that flue gas recirculation would have no effect on controlling  $NO_X$  emissions.  $SO_2$  and CO emissions were also increased significantly, probably because of lowered oxygen partial pressure and gas residence time.

A series of tests was conducted to determine if a combination of two stage combustion and NH $_3$  injection would result in further reductions in NO $_{\rm X}$  emissions. The runs were made by adding the control techniques one at a time and then in combination to a run made under conditions typical of those used in the NO $_{\rm X}$  control program. It was found that either two stage combustion or ammonia injection decreased NO $_{\rm X}$  emissions 25 to 30%. However, the combination of the two techniques resulted in a reduction of only 26%. Therefore, the combination did not offer any advantages over the use of either method alone. It should be mentioned that only one run was made and that additional testing might be needed to determine if a positive interaction exists.

Regeneration studies were carried out in the modified bench regenerator. The unit now is equipped for continuous feeding of sulfated sorbent and withdrawal of regenerated sorbent. The regenerator vessel is refractory lined to an inside diameter of 9.5 cm and has an interior height of 4.6 m. The air and fuel addition systems are similar to those used in the miniplant regenerator. A below-grid grid burner provides most of the heat required by the system. Additional fuel is added above the grid to create a reducing zone where CaSO4 reduction occurs. Supplementary air is added higher in the bed to create the oxidizing zone needed to oxidize the undesirable CaS byproduct and complete combustion of the fuel. The test program was divided into two segments, natural gas fueled regeneration and coal fueled regeneration. Most of the work concentrated on developing satisfactory operating techniques. The work done with natural gas-fueled regeneration included a study of the most effective way to introduce fuel to the regenerator and a limited study of operating variables and their effect on sorbent regeneration. Studies made

with coal-fueled regeneration were preliminary in nature and were intended to determine the feasibility of operating a regeneration unit with coal. The incentive to use coal instead of natural gas of course, is to reduce operating costs and minimize dependence on natural gas supplies.

Attempts were made to add all the natural gas fuel directly to the bed. using the below-grid burner only to preheat the bed to temperatures high enough to initiate methane combustion. This was done after it was found that about 30% of the natural gas fed to the burner was used to overcome heat losses from the burner plenum. The attempts to add all the fuel directly to the bed were not completely successful due to the occurrence of frequent high temperature excursions. The temperature excursions were believed to be caused by unstable combustion conditions in the regenerator. Operations with natural gas addition to the burner as well as to the bed were more successful. A series of six runs was made. However, because of the limited amount of data obtained it was not possible to draw conclusions concerning the relationship between operating variables and the extent of regeneration. The SO<sub>2</sub> concentration in the off gas varied from 0.2 to 0.6%, equivalent to 15 to 30% of the calculated equilibrium concentrations. These low SO2 concentrations occurred since the concentrations were not limited by kinetics or thermodynamics, but rather by heat and material balance requirements.

A series of runs was made with coal added to the bed instead of natural gas. The results of the few runs were promising. The only serious operating problem was maintaining good temperature control. Some of the runs were subject to high and erratic bed temperature. Modifications, such as the use of nitrogen instead of air as the coal transport medium, improved temperature control significantly. However, control problems persisted until the end of the program, possibly due to poor solids and gas mixing caused by the small regenerator diameter. Solids regeneration levels (the percentage of the sulfated sorbent converted to CaO in the regenerator) were around 40%, somewhat lower than the levels of 40 to 80% measured with natural gas fueled runs. SO2 concentrations in the off gas were about 17% of the calculated equilibrium concentrations, roughly comparable to the levels measured with natural gas fuel. More work is needed before coal-fueled regeneration can be completely evaluated.

### SECTION II

### INTRODUCTION

The pressurized fluidized bed combustion of coal is a new combustion technique which can reduce the emission of  $SO_2$  and  $NO_\chi$  from the burning of sulfur-containing coals to levels meeting EPA emission standards. This is done by using a suitable  $SO_2$  sorbent such as limestone or dolomite as the fluidized bed material. In addition to emissions control, this technique has other potential advantages over conventional coal combustion systems which could result in a more efficient and less costly method of electric power generation. By immersing steam generating surfaces in the fluidized bed, the bed temperature can be maintained at low and uniform temperatures in the vicinity of 800 to 950°C. The lower temperatures allow the use of lower grade coals (since these temperatures are lower than ash slagging temperatures), and also decrease  $NO_\chi$  emissions. Operation at elevated pressures, in the range of 600 to 1000 kPa, offers further advantages. The hot flue gas from a pressurized system can be expanded through a gas turbine, thereby increasing the power generating efficiency. Operation at the higher pressure also results in a further decrease in  $NO_\chi$  emissions.

In the fluidized bed boiler, limestone or dolomite is calcined and reacts with  $SO_2$  and oxygen in the flue gas to form  $CaSO_4$  as shown in reaction (1).

$$CaO + SO_2 + 1/2O_2 \rightarrow CaSO_4$$
 (1)

Fresh limestone or dolomite sorbent feed rates to the boiler can be reduced by regeneration of the sulfated sorbent to CaO and recycle of the regenerated sorbent back to the combustor. One regeneration system, studied by Exxon Research and Engineering Company in the past, is the so-called one step regeneration process in which sulfated sorbent is reduced to CaO in a separate vessel at a temperature of about  $1100^{\circ}$ C according to equation (2). The goal is to produce  $SO_2$  in the regenerator off gas at a sufficiently high concentration to be recovered in a by-product sulfur plant.

$$C_{a}S_{4} + H_{2} \rightarrow C_{a}O + S_{2} + H_{2}O$$
 (2)

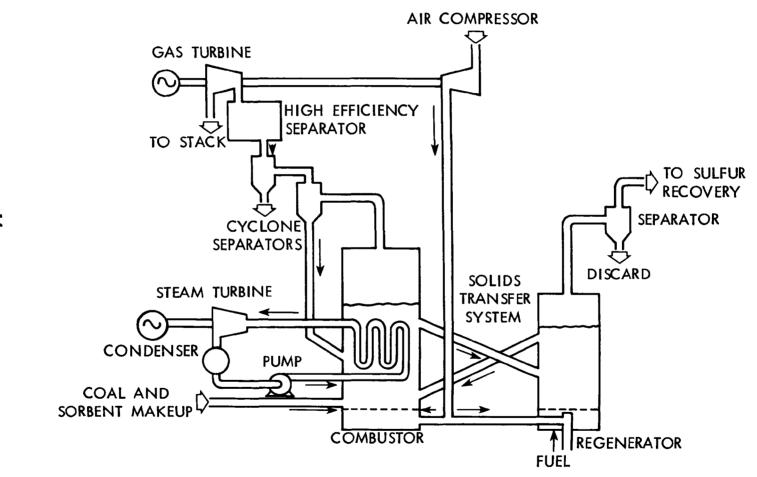
A diagram of the pressurized fluidized bed combustion and regeneration process is shown in Figure II-1.

Exxon Research and Engineering Company, under contract to the EPA, has built two pressurized fluidized bed combustion units to study the combustion and regeneration processes. The smaller of the two units, the bench scale unit, was built under contract CPA 70-19 and was described in previous reports (1,2,3,4). Those reports also described regeneration and combustion studies carried on in the bench unit. This report describes work done in the

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FIGURE II-I

PRESSURIZED FLUIDIZED BED COAL COMBUSTION SYSTEM



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bench combustor evaluating various  $NO_X$  emission control methods. Regeneration studies were conducted in the bench regenerator evaluating fuel injection methods and the use of coal as the regenerator fuel. These results are also included in this report.

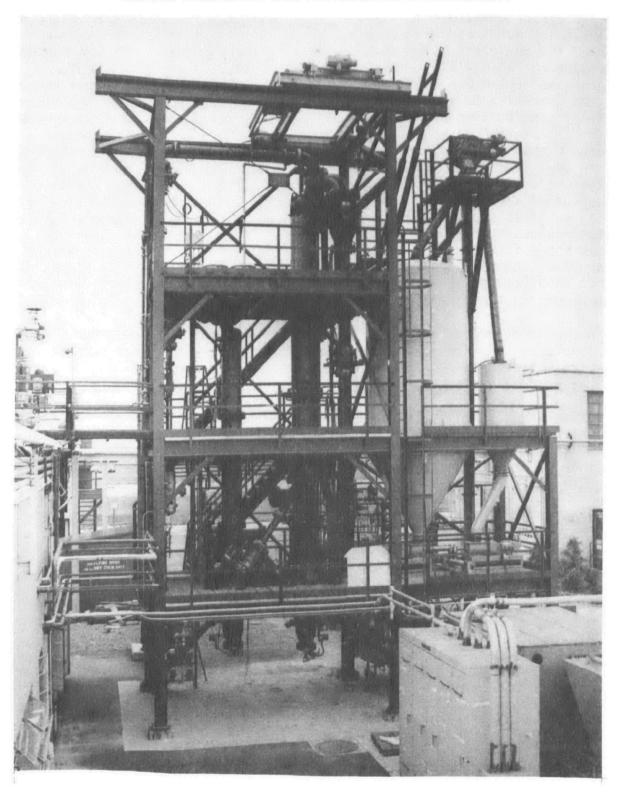
The larger unit, called the miniplant, was designed under EPA Contract CPA 70-19 and built under Contract 68-02-0617. Figure II-2 shows a photograph of the miniplant. The shakedown and operation of the unit was funded under Contract 68-02-1312. Previous reports (1,2,3,5) described design, shakedown and operation of the unit. This report includes additional results from the operation of the combustion and regeneration sections of the miniplant. The combustion program included tests to verify and document further, the ability of PFBC to reduce SO<sub>2</sub> emissions by 90% or more. The dynamic response of SO<sub>2</sub> emissions to a rapid change in coal sulfur content and dolomite to coal feed ratio was also measured. This was done to provide the basis for the evaluation of an SO<sub>2</sub> emission control concept by General Electric Company under a separate EPA contract. Additional data on the emission of  $NO_{x}$ , other gases and particulates were also obtained. Particulate control studies evaluating high efficiency cyclones, a high temperature/pressure ceramic fiber filter, a granular bed filter and a low temperature/pressure electrostatic precipitator and bag house were also completed. This work was done in cooperation with a number of other EPA contractors including Acurex Corporation, Southern Research Institute and Air Pollution Technology, Inc.

A series of regeneration tests was also conducted. Three extended runs were completed in which activity maintenance of the regenerated sorbent,  $S0_2$  concentrations in the regenerator off gas and  $S0_2$  retention in the combustor were measured.

A series of sampling campaigns was conducted in cooperation with another EPA contractor, GCA/Technology Division, to provide samples for Level I and Level II comprehensive analysis test programs. Samples were taken during operation of the combustor alone and during regeneration tests in which the combustor and regenerator were both in operation.

The period of performance discussed in this report is August 12, 1977 to August 7, 1979.

FIGURE 11-2
EXXON FLUIDIZED BED COMBUSTION MINIPLANT



### SECTION III

### MINIPLANT COMBUSTION STUDIES

Combustion studies have been carried out in the EPA/Exxon pressurized fluidized bed combustor referred to as the miniplant. The miniplant is shown schematically in Figure III-1. This unit has provisions for continuous addition of coal and sorbent and continuous withdrawal of sulfated sorbent. It normally operates at pressures of about 9 atm, temperatures of 840 to  $950^{\circ}$ C, with superficial velocities of about 1 to 2.5 m/s, feeding 100 to 160 kg/hr of coal and  $15 \text{ to } 20 \text{ Nm}^3/\text{min}$  of combustion air. As of August 1979, the combustor has been operated for a total of approximately 3700 hours in a series of individual runs up to 250 hours duration. This section of the report describes the combustor equipment, operating procedures, combustor performance and combustion results. A discussion of the regeneration work is given in Section V.

### EQUIPMENT, MATERIALS, PROCEDURES

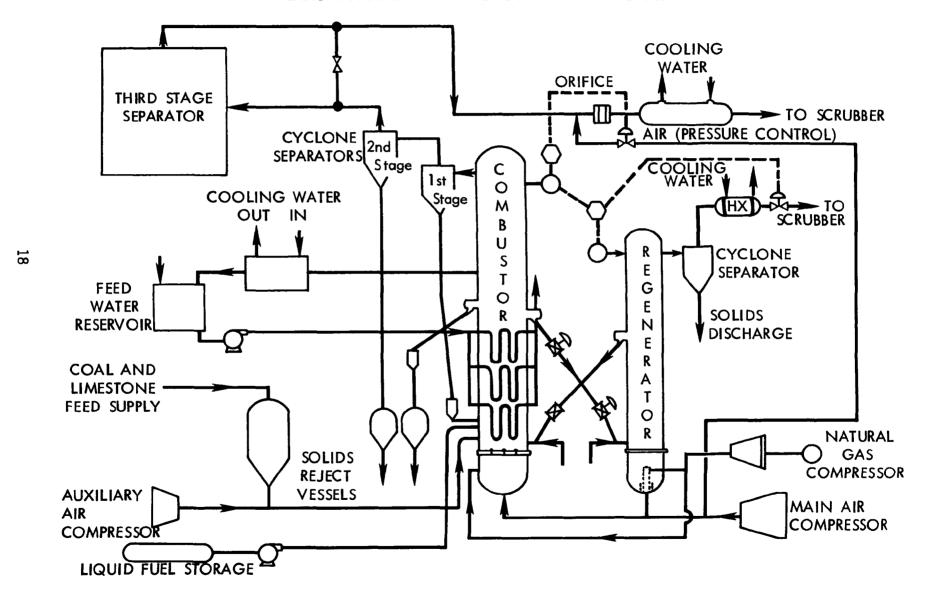
This section will focus on the major system components which include: (1) solids feeding system, (2) combustor with internal subcomponents, (3) flue gas system, (4) temperature and pressure control, (5) flue gas sampling and analytical system, (6) process monitoring and data generation system, (7) combustor safety and alarm system, (8) coal and sorbent properties, and (9) operating procedures. A detailed description of each of these systems can be found in earlier reports (1,2) and only a brief discussion will be included here. Changes in the equipment made since the last report are also included.

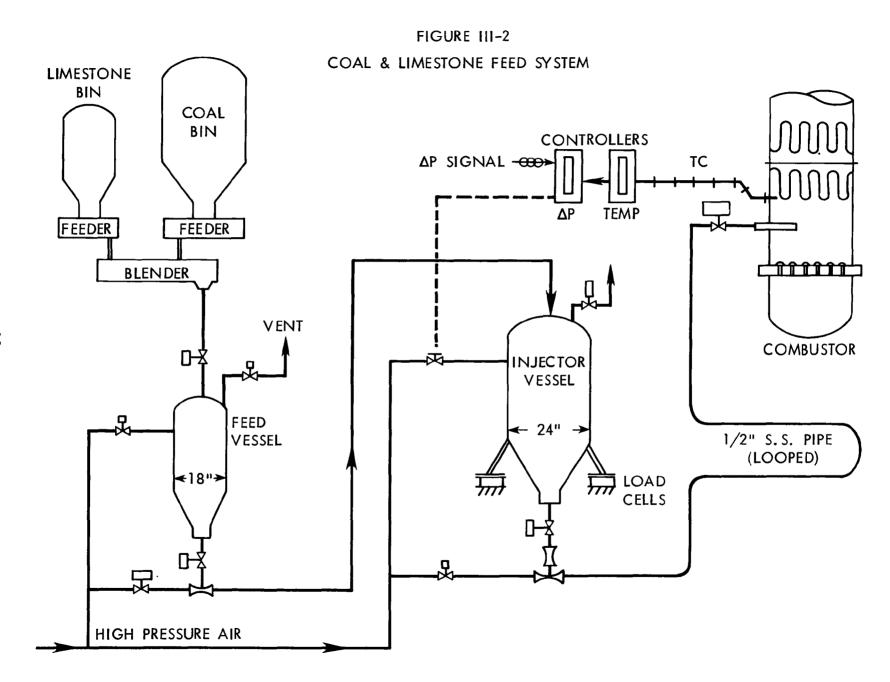
### Solids Feeding System

Figure III-2 displays a schematic of the miniplant coal and sorbent feeding system. Crushed and sized coal and limestone or dolomite are held in separate storage bins (20 tonnes for coal and 3 tonnes for sorbent) under atmospheric conditions. On demand, the solids from the bins are proportioned to a specific coal/sorbent ratio. Inverters control the motor speeds of separate coal and sorbent screw feeders and volumetrically control the coal/sorbent ratio. A blending screw transports the mixture into a solids feed vessel. The coal/sorbent mixture is held in this vessel until refill of the injector vessel is required.

The solids feeding system provides for continuous solids delivery (coal and sorbent) from the injector vessel to the pressurized combustor, while allowing intermittent refilling of the injector vessel (193 kg operating capacity). Load cells located under the injector vessel monitor the solids feed rate and actuate control signals for the refill cycle. Prior to initiation of a refilling operation, the injector vessel, feed vessel, and the pair of solids storage bins remain isolated from each other. When the load cell under the injector vessel detects a solids loading of less than 102 kg, 91 kg of solids are automatically transferred pneumatically from the feed vessel

FIGURE III-1
EXXON FLUIDIZED BED COMBUSTION MINIPLANT





to the pressurized injector vessel without interrupting feed to the combustor. Refilling is usually completed in about 5 minutes. After refilling, the feed vessel is again isolated from the injector vessel, vented, and filled with solids from the storage bins. The feed vessel is then isolated and repressurized to await another cycle.

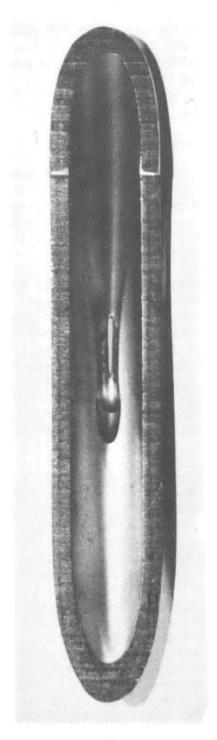
Solids in the injector vessel are continuously aerated by the pressurized air stream, and the vessel is automatically controlled at a pressure level slightly above that in the combustor. Originally the port used to sense combustor pressure was near the top of the combustor where plugging with bed material would not occur. Maintenance of the proper pressure differential between the combustor and the injector vessel during minor bed upsets was difficult. These upsets sometimes resulted in hot solids back flow into the injector vessel. For this reason, the combustor pressure tap was relocated to a port very near the coal injection nozzle. A large purge flow rate in the pressure sensing line and a high coefficient of damping in the measurement system were used to control plugging and oscillations. This system has worked without incident for over one year.

The coal and sorbent mixture is discharged from the injector vessel through a 1.3 cm diameter orifice and pneumatically conveyed by a stream of dried transport air through a 1.6 cm ID stainless steel pipe. Originally the pipe was S-shaped. To increase the pressure differential between the combustor and the injector a longer conduit with one 180° and four 90° bends was installed. The conduit is 17 m long with 30 cm radius bends. The pressure differential between the injector vessel and the combustor was increased from about 20 kPa to 70 kPa. The erosion of the coal transport conduit at the location of the four bends consistently occurred after about 200 hours of operation when the conduit was made of 0.37 cm thick, type 304 or 316 stainless steel heavy wall pipe. Heat treated, type 410 stainless steel pipe bends although much harder than 304 or 316 stainless steel, did not produce any better results. Carbonized steel pipe failed after only 60 hours of operation. Limited data were obtained with type 304 stainless steel pipe that had been alonized. After approximately 150 hours of operation, the alonized conduits were still functioning.

A comparison of the locations where the holes were eroded in the conduit bends of various materials showed that in all cases the failure occurred when the angle of solids impingement upon the wall was between 15 and 20 degrees. A segment from a type 304 stainless steel bend showing the hole location after 200 hours of operation is shown in Figure III-3.

An auxiliary solids feed system was constructed to allow changing from one coal/sorbent feed to another instantaneously. The capacity of this new vessel is equivalent to about 8 hours of operating inventory. It is situated on load cells to monitor feed rate. There is no provision for on-line refilling. The coal/sorbent blend exits the auxiliary vessel through a 1.3 cm orifice and is pneumatically conveyed by a stream of dried transport air through a 1.5 cm ID polyurethane tube. This material was chosen to minimize line erosion, because of the excellent resilience of polyurethane. This tube is shielded in the event of rupture. The tube is 12.5 m long and winds once around the 2.4 m diameter vessel before it reaches the combustor.

FIGURE III-3
CROSS SECTION OF COAL/SORBENT FEED LINE



Final entry of solids into the combustor is through a 1.3 cm ID nozzle located 28 cm above the fluidizing grid and horizontally extending about 2.5 cm beyond the reactor wall. Both the primary and the auxiliary feed systems have their own identical entry ports located 180° apart on the combustor. The tip of the probes include ten 0.79 mm diameter holes which surround the solids feed opening. They are used to continuously inject an annular stream of sonic-velocity air to assist penetration of the solids feed into the fluidized bed and to protect the feed nozzles from blockage with bed solids. Only one probe is used at any one time. The probe not in use is continuously purged to prevent blockage of the openings with solids. The flow of solids into the combustor is controlled to maintain constant temperature in the combustor.

### Combustor

The combustor consists of a 61 cm ID steel shell, refractory lined to an inside diameter of 33 cm. The 9.75 m high unit is fabricated in flanged sections to allow insertion and removal of the cooling coils. Various ports are strategically located to allow for material entry and discharge. Numerous taps are also provided for monitoring both pressure and temperature. A schematic of the combustor is shown in Figure III-4.

Heat removal from the combustor is provided by cooling coils located in discrete vertical zones above the grid. Each coil has a total surface area of 0.55 m² and consists of vertically-oriented loops constructed of 1/2-inch Schedule 40 316 stainless steel pipe. The number of coils normally varies from one to four depending on the combustor operating conditions and the amount of cooling required. A high pressure pump is used to pump the cooling water through a closed-loop arrangement consisting of a demineralized feed water reservoir, cooling coils, and a heat exchanger. The flow rate and exit temperature from each coil can be separately controlled and monitored. Baffles were installed on the cooling coils to prevent excessive erosion by the bed solids (2).

Only one failure of the baffled cooling coils has been recorded after 1200 hours of operation. A hole (Figure III-5) developed on the lower portion of the coil closest to the bed support grid, facing the center of the combustor, approximately 45° of arc along a bend. The lowest portion of the coil was only 23 cm above the fluidizing grid. The section of coil was analyzed and the failure was attributed to erosion. Very little oxidation or sulfidation was present. The lack of sulfidation is not surprising, since the coil water outlet temperature is controlled at 140°C. At this internal temperature, little sulfidation is expected. Most of the 180° bends in the lower section of the coil also exhibited a decided sharpening of the leading This is also attributed solely to erosion. The entire coil is shown in Figure III-6. A similar coil located further from the fluidizing grid was exposed over the same time period with no significant erosion. The erosion problem was apparently solved by welding small rectangular steel "guards" horizontally at the bottom of the coils. These should prevent erosion but have been used for only 500 hours of operation to date.

FIGURE III-4
COMBUSTOR VESSEL

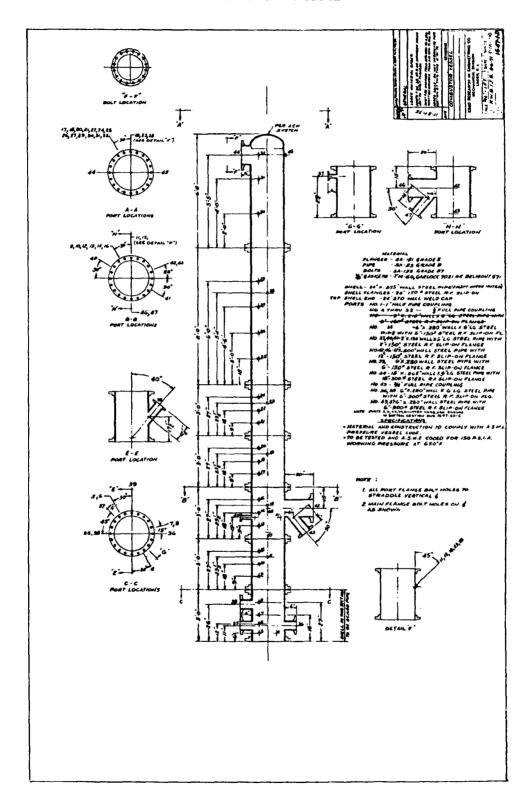


FIGURE III-5

MINIPLANT COMBUSTOR COOLING COIL 1A

AFTER 1200 HOURS OPERATION (CLOSE UP)

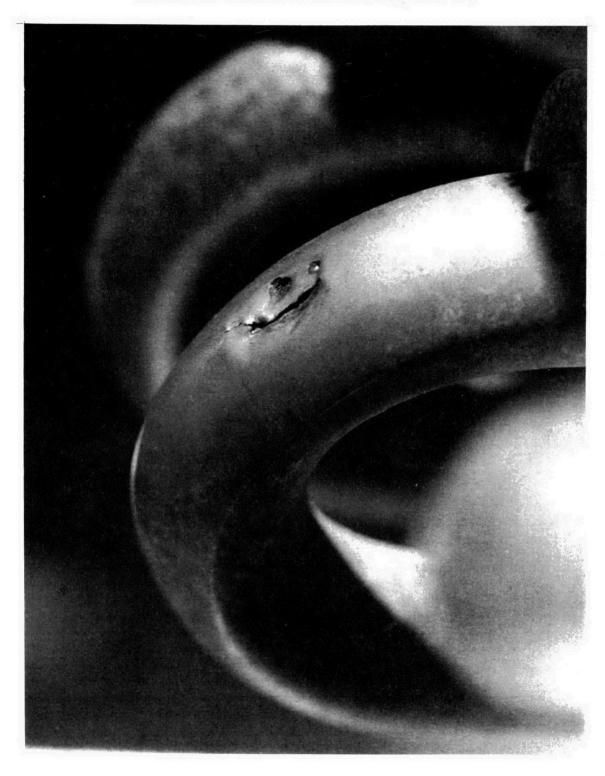
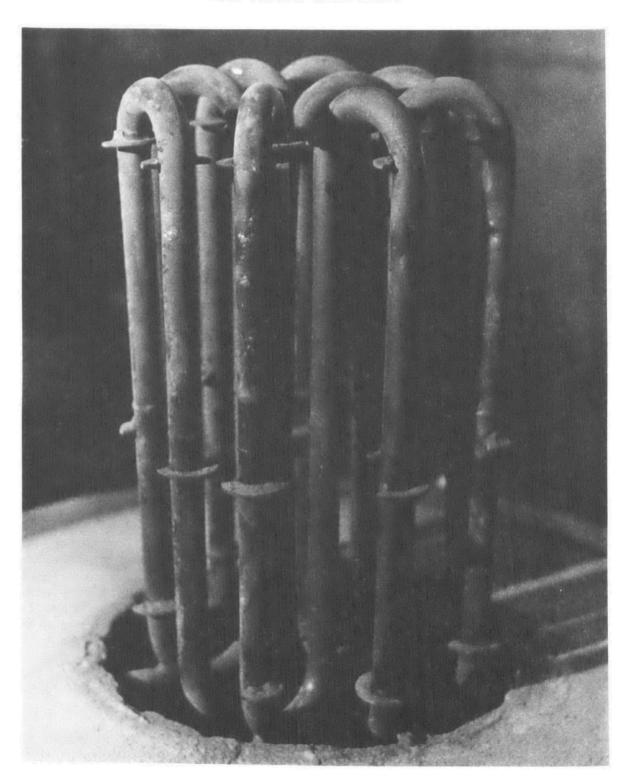


FIGURE III-6

MINIPLANT COMBUSTOR COOLING COIL 1A AFTER 1200 HOURS OPERATION



The combustion air to the unit is provided by a main air compressor having a capacity of 40  $Nm^3/min$  at 1030 kPa (1400 SCFM at 150 psig). Preheat of the combustor during startup is made possible by a natural gas burner which is housed in the bottom plenum section of the combustor. Once the fluidized bed temperature reaches approximately 430°C, a liquid fuel system is used to heat the bed further to the coal ignition temperature. Both systems are more completely described in a previous report (1).

With continuous addition of solids to the combustor, there must be provision for continuous removal to control combustor expanded bed height. The expanded bed height can be controlled at any level above 2.3 m by the continuous withdrawal of bed solids through a port located 2.3 m above the fluidizing grid. Originally, solids flowed by gravity through a refractory lined pipe into a "pulse pot" from where they were pneumatically transported by controlled nitrogen pulses to a pressurized lock hopper. This system did not give precise solids flow control and was replaced with a dual valve system. Solids now flow by gravity to a refractory lined sliding gate valve. This valve and a ball valve behind it alternately open and close an adjustable number of times per hour. Each cycle rejects 4 kg of bed material, the amount of material that fills the volume between the two valves. From the ball valve, the solids empty into a lock hopper which is manually emptied at regular intervals.

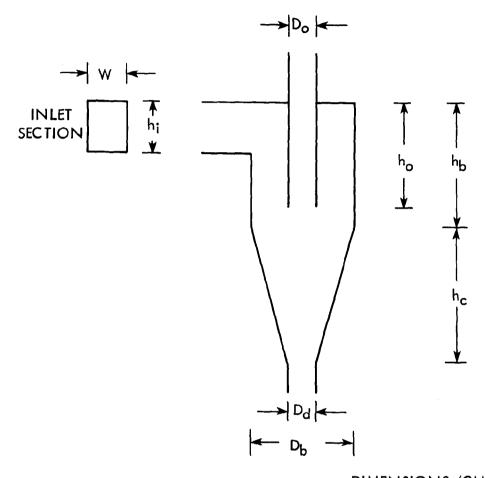
The combustor fluidizing grid consists of 332 3/32 inch holes and four independent cooling water loops consisting of five channels each. It has performed since run 50, without incident. A more complete description is contained in a previous report (1).

## Flue Gas System

Combustion gases exit the combustor and immediately pass through two cyclones in series. From the second cyclone, the gas passes through a long duct to a large pressure vessel. This vessel was designed to contain a Ducon granular bed filter or other high efficiency hot gas cleanup device. After completion of the granular bed filter tests (run 66), the filter was replaced with a third cyclone installed within the vessel. When the third cyclone is used, no flow enters the vessel; it is merely a pressure containment device. After passing through a third cyclone (or the granular bed filter in the earlier tests), the flue gas is sampled, expanded and sent to a wet scrubber for final cleanup. The dimensions of all three cyclones are shown in Figure III-7. The dimensions of the second stage cyclone have been modified since it was originally built. Originally, the barrel diameter was 31.8 cm and the inlet section sized to give an inlet velocity of about 10 m/s. After run 18.3, the inlet dimensions were changed to increase the inlet velocity to about 30-35 m/s. The outlet diameter was decreased. The final change was made after run 47 to the dimensions in Figure III-7. These changes decreased the barrel diameter but maintained the inlet section dimensions to keep the inlet velocity range fixed.

The granular bed filter, used in tests prior to run 67, is described in Section IV.

FIGURE III-7
CYCLONE DESIGN DIMENSIONS



		DIMENSIONS (CM)						
SYMBOL	DESCRIPTION	CYCLONE NO. 1	CYCLONE NO. 2	NO. 3				
	Inlet Type	Tangential	Tangential	Tangential				
$D_{\mathbf{b}}$	Barrel Diameter	32.4	17.8	15.2				
W	Inlet Width	7.62	4.45	3.81				
h;	Inlet Height	25.4	10.2	7.62				
ho	Outlet Pipe Insertion	20.3	13.3	11.4				
hb	Barrel Height	43.2	35.6	40.6				
h	Cone Height	45.7	35.6	20.3				
h Do	Outlet Pipe Diameter	14.6	8.89	6.2				
0/1	Outlet/Inlet Ratio	0.87	1.37	1.04				
$D_d$	Diameter of Cone at Bottom	12.7	9.0	9.0				
	Function	Recycle	Cleanup	Cleanup				

The primary intent of the first cyclone is to recirculate larger unburned carbon particles back to the combustor to improve combustion efficiency. The particles fall down the dipleg and are reinjected with a nitrogen pulse about 66 cm above the fluidizing grid.

Particulates collected in the second stage cyclone are removed through a lock hopper. The cyclone, which was originally cast with Grefco Litecast 75-28 refractory, was recast after run 47 with Resco RS-17-E, and is still in service after 2-1/2 years. None of the severe pitting that was present with the earlier refractory was found.

From the second cyclone, the flue gas enters the large pressure vessel which contains the third cyclone. This cyclone is of conventional design and constructed of 316 stainless steel. Particulates captured by the third cyclone are removed through a lock hopper.

Following the third cyclone, the flue gas may either be expanded through a pressure control nozzle or a turbine test section supplied by General Electric under contract with the Department of Energy (DOE Fireside Corrosion Contract No. EX-76-C-01-2452). The test section contains 24 stationary blades of various materials that simulate both the impulse and reaction vanes of a gas turbine. The turbine section was tested for 1100 hours of operation with three stage cyclone particulate cleanup as part of the DOE Fireside Corrosion program. A more complete description is given in a report to DOE (6).

In order to simulate gas turbine operation, care must be taken to prevent the gas temperature from dropping below 843°C (1550°F). Below this temperature, gas phase alkali will condense on duct walls or on particulate matter. This would unrealistically reduce the vapor phase alkali concentration to which the turbine blades are exposed. For this purpose, four natural gas injection ports were located between the second and third cyclones. The locations of the four ports were staggered to maintain gas temperatures between 857°C and 900°C. The total amount of natural gas (typically 0.6 to 0.9 Nm³/min) is controlled to maintain a set third cyclone inlet temperature. The flow distribution between the four probes is manually controlled. The injection probe consists of a closed end 3/8 inch alonized tube projecting to the duct centerline with a 0.10 cm diameter hole facing downstream. The probes are purged with nitrogen when not in use.

The natural gas injection system is frequently used to hasten thermal equilibrium in the miniplant off gas system. Heating the refractory to steady state would otherwise require 8 to 12 hours. With natural gas injection, this is reduced to two hours. The effects of natural gas injection will be reported in a following section.

#### Temperature Control

The rate of solids feed to the combustor is automatically controlled in order to maintain a specific operating temperature within the combustor. This is accomplished, through a series of controls which adjust the pressure

differential between the combustor and the injector vessel, thereby varying the coal feed rate in a way to maintain the proper temperature. This system is described in detailed in a previous report (1).

#### Pressure Control

The FBC miniplant combustor has the capability of operating at pressure levels of up to 10.5 atmospheres (140 psig). Normally, pressure control is achieved by restricting the discharge flow of all the gas from the combustor, so as to achieve an increase in back pressure. This is done by use of a silicon carbide converging nozzle inserted in the discharge line. Adjustment of the combustor pressure is accomplished by metering high pressure air into the discharge line just upstream of the flow nozzle. A 2 inch-ball valve equipped with a pneumatic positioner and actuator regulates the amount of air added in response to a signal from the pressure controller. A more complete description is contained in a previous report (1).

During the time that the combustor gas passed through the General Electric turbine test section, this turbine section acted as the back pressure regulator for most of the gas. The converging nozzle was disconnected during these tests. However, to maintain proper pressure control, a small slip stream of gas ( $\sim$ 10%) was diverted prior to the test section and expanded through a smaller variable nozzle. Makeup air was added just upstream of this nozzle through the same valves and controllers that controlled pressure in the normal operating mode. This variable nozzle (Figure III-8) was adjusted to maintain 5 to 9 Nm³/min makeup air flow. The nozzle was constructed of 316 stainless steel and performed satisfactorily for over 700 hours of testing.

## Sampling and Analytical Systems

Gas Sampling System--

Flue gas is sampled at a point about 20 m downstream of the third stage cyclone. The system is designed to produce a solids-free, dry stream of flue gas at approximately ambient temperature and atmospheric pressure whose composition, except for moisture, is essentially unaltered from that of the original flue gas. The conditioned gas is analyzed in a series of continuous, on-line analyzers for SO2, CO, CO2, O2 and NO $_{\rm X}$  concentration. The flue gas sampling and continuous analytical system was described in a previous report (2). Another sample of flue gas can be extracted which has been filtered, cooled and depressured, but not dried. This system is used to obtain batch samples of flue gas for analysis of SO2 and SO3 by wet chemistry methods. The method to determine SO3 concentration was modified since the publication of the previous report.

Previous measurement of  $SO_3$  from the FBC miniplant consisted of flowing the flue gas through an 80% isopropyl alcohol (IPA) impinger. Because of potential problems with this method, such as possible contamination of IPA lots with small quantities of peroxides which oxidizes  $SO_2$  to  $SO_3$ , collection of  $SO_3$  is now accomplished by using a controlled condensation coil. The coil is maintained at 60%C, which is well above the water dew point and low enough to remove most of the  $SO_3$  from the gas phase.

FIGURE III-8

VARIABLE PRESSURE REDUCING NOZZLE

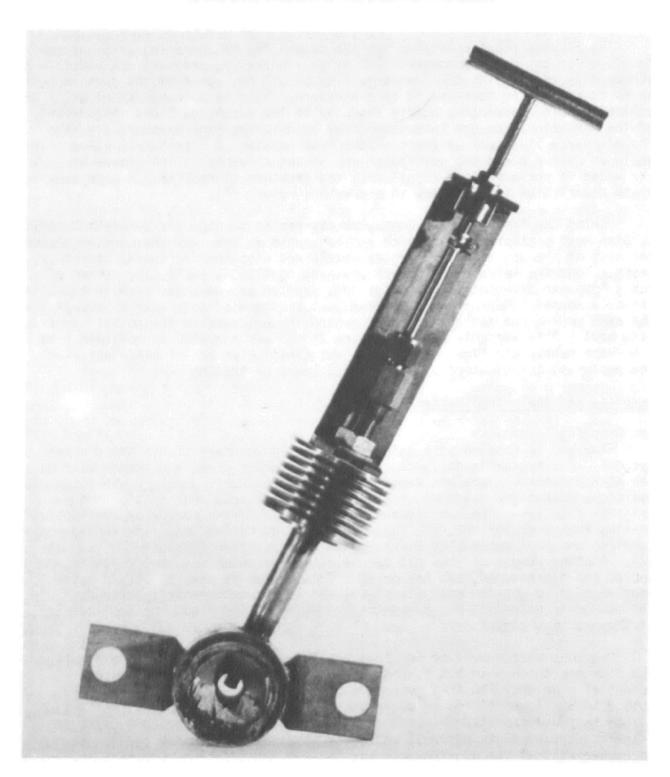


Figure III-9 shows the sampling train as it is now used. Particulate material is removed at 9 atm by a Balston filter heated to  $288^{\circ}$ C, well above the SO<sub>3</sub> dew point under FBC conditions. The cleaned gas is delivered to the gas sampling train through a heated ( $288^{\circ}$ C) stainless steel line followed by heated teflon tubing. Collection of SO<sub>3</sub> as H<sub>2</sub>SO<sub>4</sub> is accomplished by using a Goksoyr-Ross controlled condensation coil. The 80% IPA impinger is used as a backup for SO<sub>3</sub> collection with IPA known to have a negligible peroxide contamination.

 $\rm H_2SO_4$  collected in the condensation coil and the IPA impinger is titrated with barium perchlorate reagent and thorin indicator. Comparable results were obtained with an acid/base titration using bromphenol indicator. Two  $\rm H_2O_2$  impingers are used to trap  $\rm SO_2$  which is also determined using barium perchlorate/thorin titration.

## Particulate Sampling Systems

The miniplant has three particulate sampling systems. These systems were installed or modified as the need for more and different particulate sampling information arose. The oldest system referred to as the "Balston Filter Particulate Sampling System" is described in a previous report (1). The Balston filter elements consist of borosilicate glass fibers bonded with an epoxy resin. A newer system is capable of sampling at high temperature and high pressure with an in-situ size distribution measuring device. This system is referred to as the high temperature high pressure (HTHP) particulate sampling system. It was constructed during this reporting period to sample the flue gas with a 5-stage cyclone train developed by the Southern Research Institute or a University of Washington 7-stage impactor. Both the Balston filter and the HTHP sampling systems sample particulates in the gas leaving the tertiary The third system samples the particulates in the gas entering the tertiary cyclone. This system, referred to as the Upstream Balston Filter Particulate Sampling System, was only installed after run 94. It was used to determine cyclone capture efficiency. The locations of all of these systems is shown in Figure III-10.

The old Balston Filter Particulate Sampling System has been modified since its description in a previous report (1). Originally the system contained three 18 cm Balston total filters in series followed by coolers, a water knockout, flow control valve and rotameter. The secondary and tertiary Balston filters were removed from the system. During the sampling it was found that their weight gain contributed only a negligible amount to the total loading and what was captured had the appearance of wet corrosion products. A wet test meter was also added after the rotameter. This allowed for a more accurate measurement of sample flow than was previously possible with a rotameter and a timer. A schematic of the system as modified at the end of this reporting period is shown in Figure III-11. This system has provided a large data base of particulate measurements. It has been used with probe sizes between 1.09 and 0.46 cm and isokinetic flow rates between 20 and 200 Ndm<sup>3</sup>/min, all with good results.

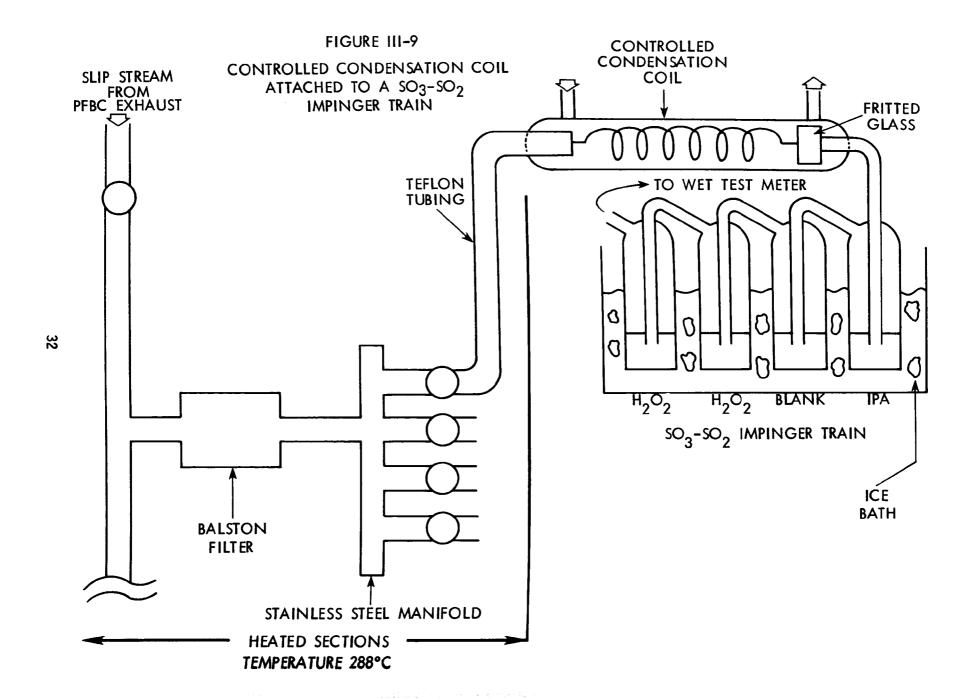


FIGURE III-10

MINIPLANT FLOW SCHEMATIC

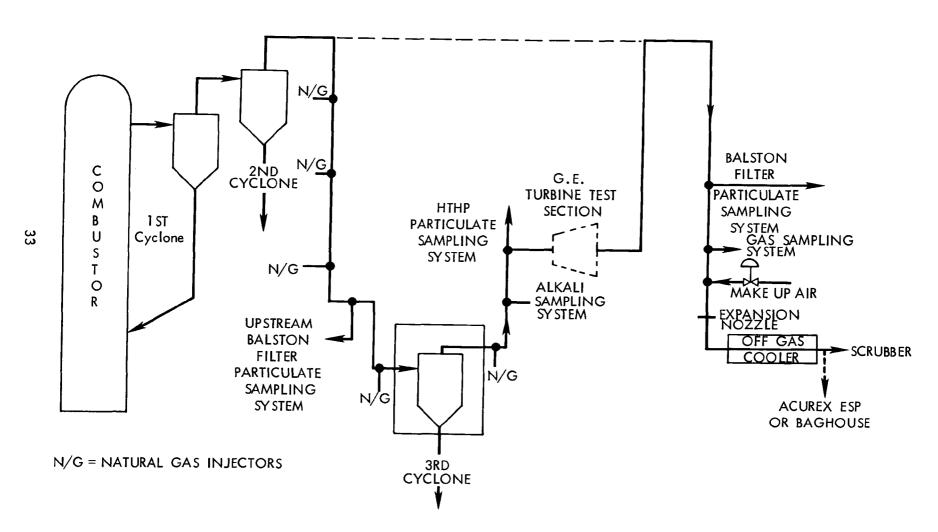
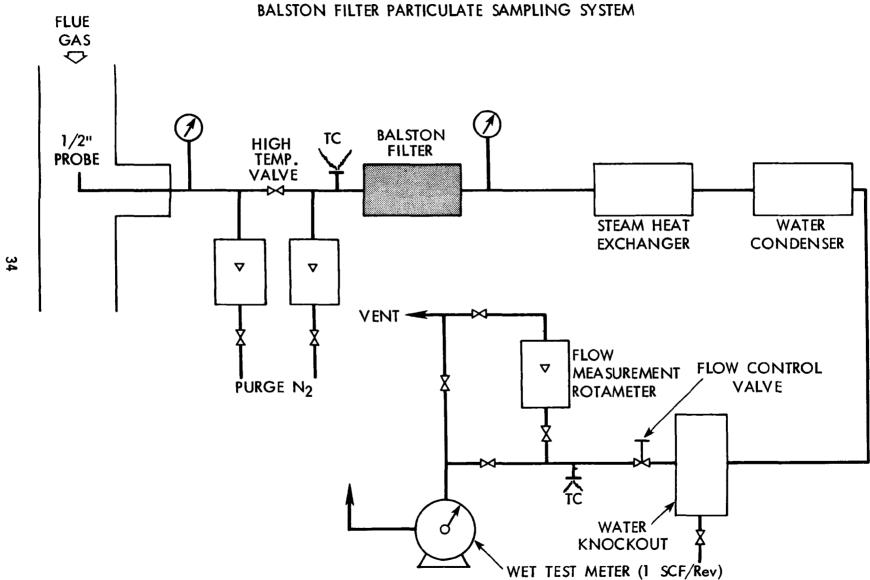


FIGURE III-11



The HTHP sampling system is designed to obtain isokinetic, isothermal samples of particulate laden flue gas. The system consists of a long 0.95 cm (3/8 inch) diameter tube projecting into the flue gas stream along the duct centerline. Once outside the flue gas duct, the tube is heated with electric heaters to maintain a set gas temperature. A high temperature (Kamyr) ball valve provides system isolation during combustor operations. Following the valve, the gas enters a pressure furnace which contains the sampling device. This furnace is large enough for most in-stack sampling devices. It is electrically heated to a controlled temperature (usually 1600°F) and pressurized with helium to prevent sample leakage or high temperature corrosion of the steel. After the furnace, the gas passes through a backup Balston filter and some gas coolers. Then the gas passes through a water knockout; another filter to remove the condensed acid mist; and the flow control system. Initially, the flow control system was automated to maintain a set flow rate over a span of sampling device pressure drops. This automatic system was prone to plugging and corrosion failure. The automatic flow controllers were replaced with a manual valve. This valve has performed satisfactorily for many samples. Following pressure let down, the gas passes through a gas saturator and a wet test meter that measures total flow. Figure III-12 is a schematic of the original system. Figure III-13 is a schematic of the system as modified at the end of the reporting period.

One additional modification that was made to the HTHP sampling system, that could not easily be shown in the figure, is the provision to bypass the furnace and the small (7 cm) backup Balston filter and install an 18 cm Balston total filter in their place. In this way, the probe and flow control system can be used for sampling at 500°C with one 18 cm Balston filter as well as at 850°C with the other devices. The advantage of the Balston filter is rapid and reliable particulate concentration measurements.

The in-situ size distribution measuring devices used in the HTHP particulate sampling system were a Southern Research Institute 5-cyclone train and a University of Washington 7-stage impactor. The 5-cyclone train (Figure III-14) was housed in the pressure furnace in the vertical position. Two cyclones were upside down during sampling. A complete description of the cyclones is given in a separate report by SoRI which also details cold flow calibrations (7). The 7-stage impactor was also installed in a vertical position, with a precutter cyclone before the impactor. The cyclone was there to prevent overloading of the first stage due to large concentrations of large particles. The impactor is described in a technical bulletin (8), a picture is shown in Figure III-15.

The third particulate sampling system was installed after run 94 to measure particulate loadings entering the tertiary cyclone. This system was built mainly to verify the high collection efficiency of the tertiary cyclone. This efficiency was previously obtained by completing a mass balance with the outlet filter sample and the captured lock hopper material. This sampling system consists of a 6 mm probe projecting to the duct centerline. Filter isolation is provided by two bellows seal valves (650°C temperature limit). Immediately after the valves is the 18 cm Balston total filter. Following the filter a large cooling coil provides natural convective cooling. This is followed by a water knockout, manual flow control valve, gas saturator and

FIGURE 111-12

ORIGINAL HTHP PARTICULATE SAMPLING SYSTEM

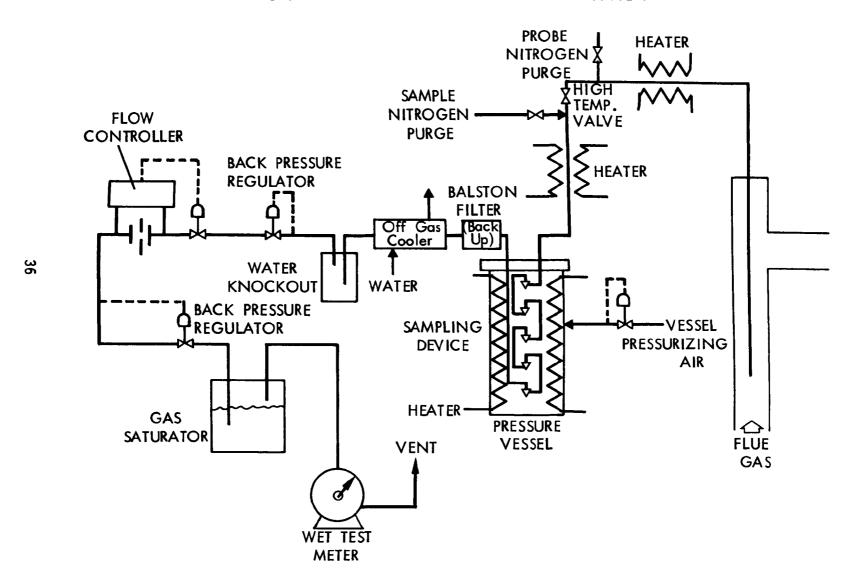
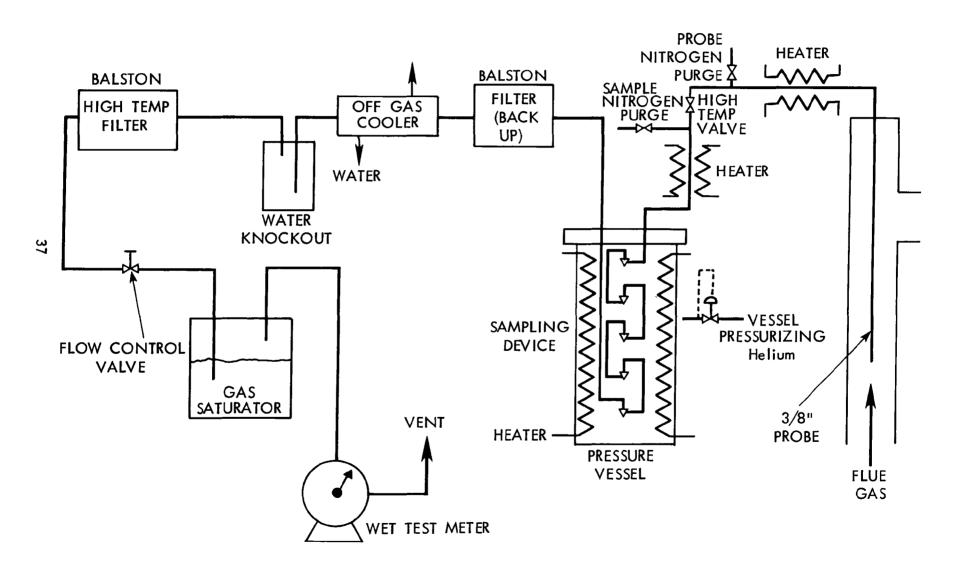
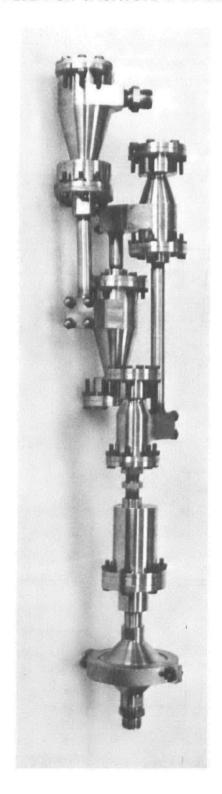


FIGURE III-13

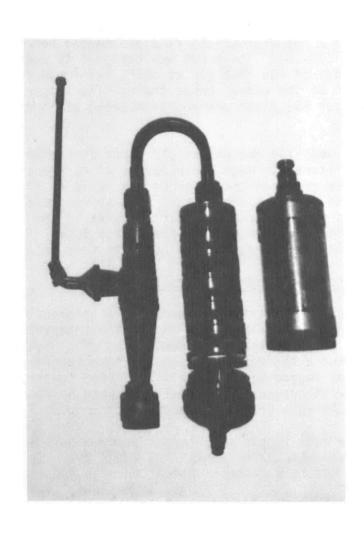
HTHP PARTICULATE SAMPLING SYSTEM AS MODIFIED



# FIGURE III-14 SOUTHERN RESEARCH INSTITUTE 5-CYCLONE TRAIN



# FIGURE III-15 UNIVERSITY OF WASHINGTON 7-STAGE IMPACTOR



a wet test meter. The flow is set to isokinetic conditions by timing the gas flow through the wet test meter. This system has verified efficiencies obtained earlier by the mass balance technique. Low flow rates are required due to the limited natural convective cooling.

#### Alkali Probe Train

An alkali probe train was designed and constructed by Exxon Research to enable acquisition of a hot pressurized flue gas sample before the turbine test section (Figure III-10). The probe was designed to measure vapor phase Na and K concentrations of the flue gas at 840°C and 9 atm pressure. Figure III-16 is a schematic of the alkali probe train. The temperatures shown are those for test 4 of run 78; these are representative of normal operating conditions.

The hot pressurized flue gas enters the probe at system temperature and pressure and is prefiltered through three layers of astroquartz. Na and K vapors in the flue gas are then condensed on the walls of an air cooled quartz tube, which lowers the flue gas temperature from approximately 840°C to 200°C. Alkali metals which condense on particulates in the flue gas are collected in a Balston filter after exiting the quartz tube. Flow is manually controlled by a needle valve and measured by a wet test meter.

## Process Monitoring and Data Generation System

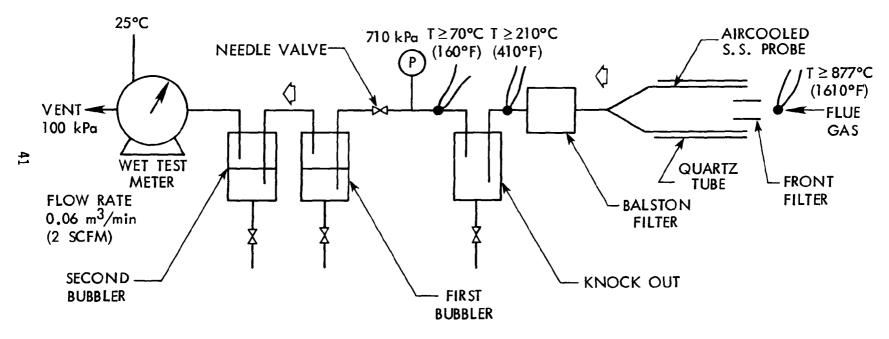
Data characterizing the miniplant operating conditions are recorded on 5 multipoint recorders. In addition, at one minute intervals, the same output is recorded by a data logger system consisting of a Digitrend 210 data logger with printer and a Kennedy 1701 magnetic tape recorder. Approximately 100 pieces of data are logged with one-half involving temperature measurement while the rest deal with pressure, material flow rate or flue gas composition. The system is described in a previous report (1).

Signals from the data logger are scanned every minute and are stored on magnetic tape. The magnetic tape, containing about 6000 items of data per hour of run time, is fed to a computer which converts the logger output to flow rates, pressures, etc. with the proper dimensions. The data are then averaged and standard deviations are calculated over preselected time intervals (usually 10 to 30 min.). Other quantities are also calculated. This includes average bed temperature, based on four thermocouple readings covering the 15-114 cm interval above the fluidizing grid, superficial gas velocity, excess air, as well as the important gas concentrations.

## Combustor Safety and Alarm System

A process alarm system was designed to warn of impending operational problems. Two general alarm categories exist. The first, dealing with less critical situations, alerts the operator of the problem so that appropriate corrective action can be taken. The second class of more critical alarms results in the immediate or time delayed shutdown of the complete system or specific subsystems. An alarm condition is brought to the attention of the

FIGURE 111-16
SCHEMATIC OF ALKALI PROBE TRAIN\*



\* TYPICAL TEMPERATURES AND CONDITIONS DURING TEST 4 OF RUN 78

operators by a flashing light above the control panel accompanied by a high pitch sound. The sensitivity of the individual alarms is controlled by potentiometers located beneath the control panel. The system is described in a previous report (1).

### Coal and Sorbent Properties

#### Coal --

Coals used in the miniplant variables study were a high volatile bitum-inous coal from the Consolidation Coal Company's "Champion" preparation plant in Pennsylvania, an Illinois No. 6 seam coal obtained from Carter Oil Company's Monterey No. 1 mine, and an Ohio coal obtained from the Valley Camp mine.

The Champion coal was partly classified to remove fines smaller than 50 U.S. Mesh. The Illinois coal was screened to 6 x 40 U.S. Mesh to prevent plugging of the primary injector feed vessel. Particulate size distribution and composition data for the coals are shown in Figure III-17 and Table III-1 respectively. The Valley Camp coal was only used on a very limited basis during a coal strike. This coal was purchased from Curtiss-Wright and no size analysis was obtained. The coal was prescreened 6 x 50 U.S. Mesh.

#### Sorbent

#### Sorbent --

Grove limestone (BCR No. 1359) and Pfizer dolomite (BCR No. 1337) were the primary sorbents used in the miniplant variables study. The composition of these stones is given in Table III-2. Most of the runs were made with the stone screened to  $8\times25$  U.S. Mesh to give the distribution of dolomite shown in Figure III-18. The limestone was only used on a limited basis, no size distribution was obtained.

#### Operating Procedures

Prior to initiating a run, a detailed checkout procedure is followed to insure that the system is ready for operation. This includes various equipment checks, alarm system checks, calibration of flue gas analyzers, activation of process monitoring and control systems, and the turning on of all cooling water systems. All runs are begun with an initial bed of sorbent in the combustor. This consists of either a charge of sulfated limestone or dolomite, or the bed material from the previous run. Fresh, uncalcined limestone has been used, but is not preferred.

The first operation of start-up involves heating the gently fluidized sorbent bed by burning natural gas in the burner plenum followed by injection of kerosene into the bed. Once the bed temperature is  $\sim 650\,^{\circ}\text{C}$ , coal injection is begun. As soon as coal ignition is confirmed by a sharp temperature increase, the other fuels are shut off. Combustor temperature, gas flow rate and pressure are than rapidly increased to their operating values. A more complete procedure is contained in a previous annual report (1).

FIGURE III-17 COAL PARTICLE SIZE DISTRIBUTION 100 90 **-**|80 WT. % FINER THAN -60 43 **-**|50 ILLINOIS 🗘 CHAMPION 40 -30 20 -110

600

700

500

PARTICLE SIZE,  $\mu m$ 

400

300

200

900 1000

3000

2000

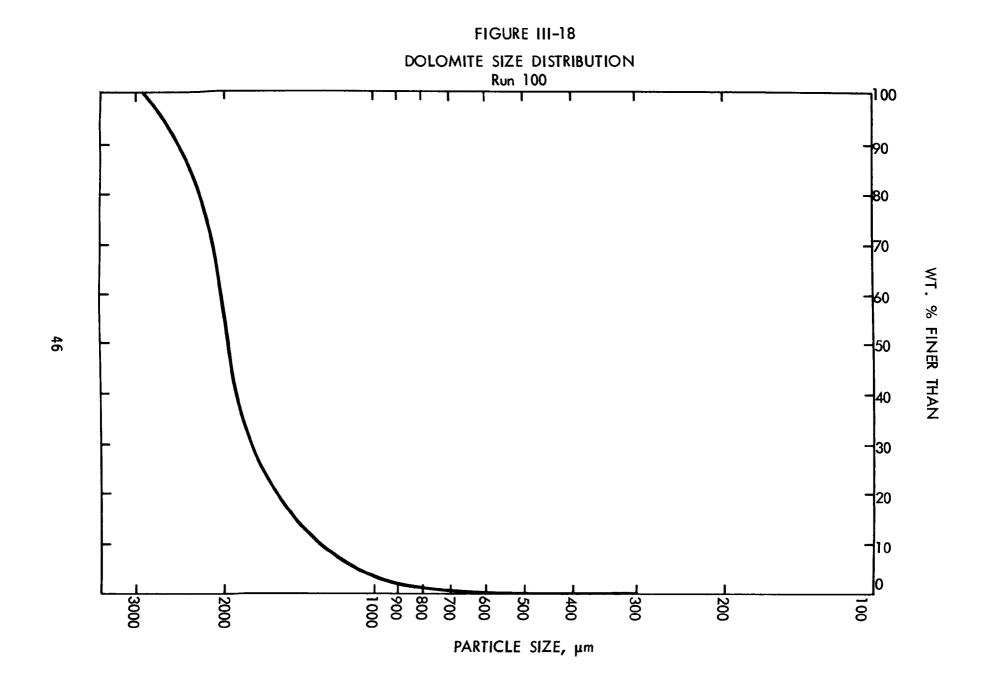
TABLE III-1, COAL COMPOSITION

		Ultimate Anlysis F								Heating
Run Number	Coal Type	Moisture	Ash	Carbon	<u>H</u>	<u>_s</u>	<u>N</u>	_C1_	02	Value 1b/BTU
60-65	Champion	2.19	12.50	71.35	4.74	1.40	1.54	0.07	6,31	12,514
66-70	Illinois	1.96	9,68	67.35	5.40	4.00	1.24		10.37	12,575
71	Illinois	1.60	9.47	68.73	5.41	4,00	1.24		9.58	12,645
72	Ohio	1.76	8.76	73.41	5.23	2.84	1.35		6.70	13,171
73-75	Ohio	1 .82	7.27	74.33	5.35	2.63	1.29		7.93	13,351
76 and 77	Ohio	1.8	6.77	75.13	5.52	2.47	1.16		7.18	13,711
78.1	Illinois	3.16	11.57	65.58	5.63	4.17	2,41		7.55	12,042
78.2-78.10	Illinois	12.82	8.88	57.76	5.02	3,52	2,26		11.0	10,988
79	Illinois	12.5	7.98	60.63	5.48	3,33	1.17		10.11	11,043
80	Illinois	12.86	8.54	67.67	5.81	3,53	1.29		11.99	11,528
81 -88	Illinois	12.9	8.12	58.81	4.78	3.37	1.09		10.94	10,892
89-96	Champion	2.3	9.82	71.91	5.24	1.65	1.43		7.70	12,809
97,98,99.1, 99.2,99,5-99.7	Champion	3,12	12,20	68.04	4.76	1.92	1.42		8.63	12,581
99.3 and 99.4	Illinois	13.0	9.18	60.34	4.70	3.4	1.02		9.65	10,912
100	Champion	3.12	8.91	70.44	4.88	1.87	1.48		9.41	12,984
101 and 102	Champion	2,28	6.64	74.35	5.23	1.38	1.58		8.58	13,728
103 and 104	Champion	2.94	8.61	72.43	5.20	1.61	1.49		7.81	13,151
105	Champion	2.52	6.97	79.41	5.87	1.64	1.47		2.04	13,612
106	Illinois	11.36	7.89	67.39	5.10	3.52	1.33		4,43	11,969
107-115	Champion	2.40	8.34			1.84				12,874

45

TABLE III-2. SORBENT COMPOSITION

		Weight Percent							
Quarry	Sorbent Type	Ca O	MgO	S10 <sub>2</sub>	A1 <sub>2</sub> 0 <sub>3</sub>	Fe <sub>2</sub> 0 <sub>3</sub>			
Grove	Limestone	94.64	1.01	0.04	0.46	0.117			
Pfizer	Dolomite	55.78	37.04	0.59	0.45	0.23			



#### EXPERIMENTAL RESULTS AND DISCUSSION

### Combustor Operations

During the month of August 1979, experimental miniplant studies under the present contract were completed with run 115. The combustor was shut down after completing over 3700 hours of coal combustion operation. Twelve runs were made of over 100 hours duration; five of these were over 200 hours duration. The longest continuous run of the combustor was 250 hours long.

The miniplant also made many short runs in rapid succession. During the ceramic bag filtration studies, fourteen 8-10 hour runs were completed in fifteen working days. This was possible because of the rapid startup procedure developed for the miniplant. Coal was typically burned 1 to 1-1/2 hours after the start of the first shift.

The ability to turn the unit around rapidly was also demonstrated. For example, during the month of June 1979, the combustor was operated for nearly 400 hours in 3 run segments for two different test series. These runs were coordinated with two other EPA contracts (Acurex and GCA Corp.). Several runs were also made in a "piggyback" fashion with up to three other EPA contractors participating.

Although the operation of the miniplant became rather routine, some operating problems still occurred. The impact of these problems was usually minimized by employing a number of operating procedures which were developed over a period of time.

Initially coal feeding was one of the more serious operating problems. After a number of equipment modifications described in an earlier report (3), the system performed satisfactorily. Some problems still persisted and were dealt with by modified operating procedures. The erosion of the coal feed line described in an earlier section of this report was such a problem. The 316 stainless steel line eroded through after about 200 hours operation at a bend in the line. The impact of this problem was minimized by replacing all bends in the coal feed system as soon as one bend eroded. This required only 10-15 minutes, during which time the combustor was fed liquid fuel. Before long runs, entirely new bends were installed, further reducing run interruptions.

Coal feed plugging was only a minor problem during this period. Plugging of the coal feeder would occur occasionally if coal was fed with a combination of high moisture and high fines content. Feeding of coal with a moisture content as high as 11% was possible if fines smaller than 420  $\mu m$  (40 U.S. Mesh) were removed. Drier coals with moisture contents of 1 to 3% were fed without incident when fines less than 300  $\mu m$  (50 U.S. Mesh) were removed. The drier coals were easier to process and generated less waste as fines.

The availability of properly processed coal was much more a problem than coal feed plugs. No supplier of dry Illinois No. 6 coal was found for the quantities needed. The high moisture coal was used because of this. Unprepared Illinois coal was dried by spreading it in a parking lot and raking it.

Coal prepared in this manner contained 11% moisture. The supplier who did this work elected to discontinue after processing only our most urgent supply needs. Regeneration runs were made with low sulfur Champion coal because no supplier of prepared Illinois could be found.

Problems associated with the back flow of hot bed solids through the coal transfer line into the coal injector vessel became severe during the testing of the granular bed filter. During the filter blow back cycle, a high pressure pulse would upset the pressure differential between the combustor and the injector vessel. Hot solids would back flow to the injector vessel and ignite the coal in the vessel. Several runs were terminated for this reason. The problem, however, disappeared when testing of the granular bed filter stopped.

Corrosion of the wet scrubber used to ensure compliance with EPA emission standards was a continual problem. It required frequent repairs during turnaround times. The severity of the problem was lessened with the injection of NH3 into the scrubber to neutralize the acids formed.

Operations during winter months were difficult. Because of the start/stop nature of many of the miniplant runs, frequent draining and blowing of water systems was necessary. Even with these precautions, freeze ups still occurred. Productivity during the winter was much lower for these reasons.

Small problems with the main fluidizing air compressor frequently required attention during a run. One failure during a run gave a chance to observe the effect of bed slumping and hot start. When the compressor failed, it stopped all fluidizing air to the bed. The temperature profiles during this period are shown in Figure III-19. The unit was restarted within 1/2 hour after repairs were made to the compressor. The restart was completely successful with no bed agglomeration.

Hot Corrosion/Erosion Testing of Materials--

During this reporting period, a total of 1117 hours of combustor operation were devoted to "Hot Corrosion/Erosion Testing of Materials for Application to Advanced Power Conversion Systems Using Coal-Derived Fuels." This work was sponsored by DOE, under a cooperative agreement with EPA, to test the effect of exposing gas turbine blade and boiler tube materials to real PFB conditions. The gas turbine specimen test section was designed and built by General Electric. The air cooled boiler tube probes were designed and built by Westinghouse. The evaluation of results from these two systems is the responsibility of General Electric and Westinghouse. The combustor flue gas system was modified as shown in Figure III-20 to permit good pressure control in the presence of the turbine test section. Most of the flue gas was expanded through the turbine test section; 10% was diverted for pressure control.

A picture of the blade specimens after 1000 hours of exposure is shown in Figure III-21. As can be seen in the photograph, little visible erosion is present. Further metallographic examination is being done by General Electric. Results will be reported by them in the future.

FIGURE III-19
SELECTED COMBUSTOR TEMPERATURES DURING CRASHDOWN AND RESTART

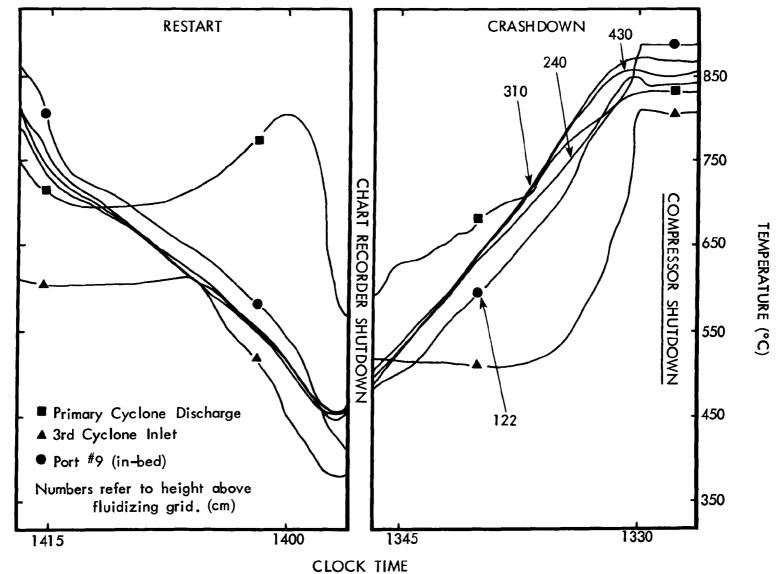
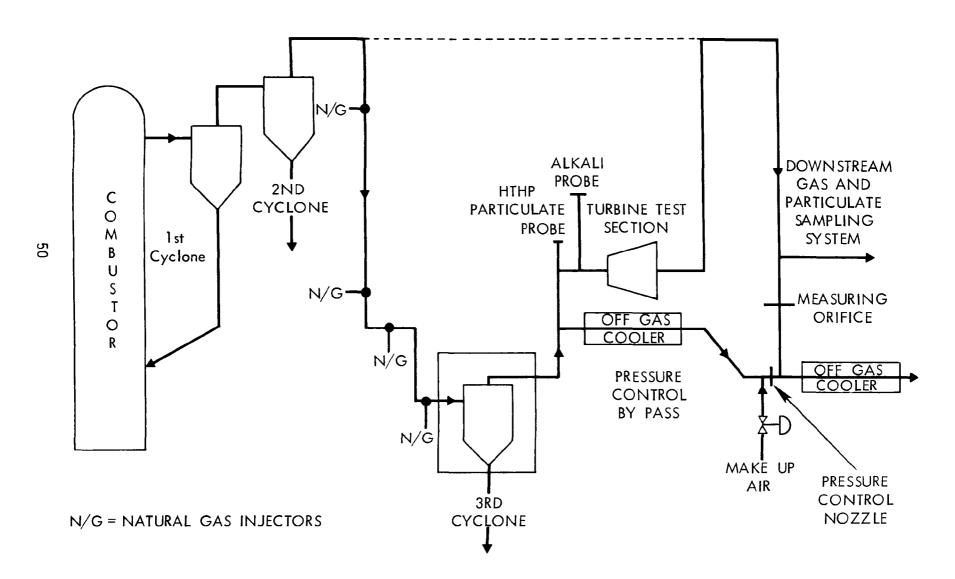
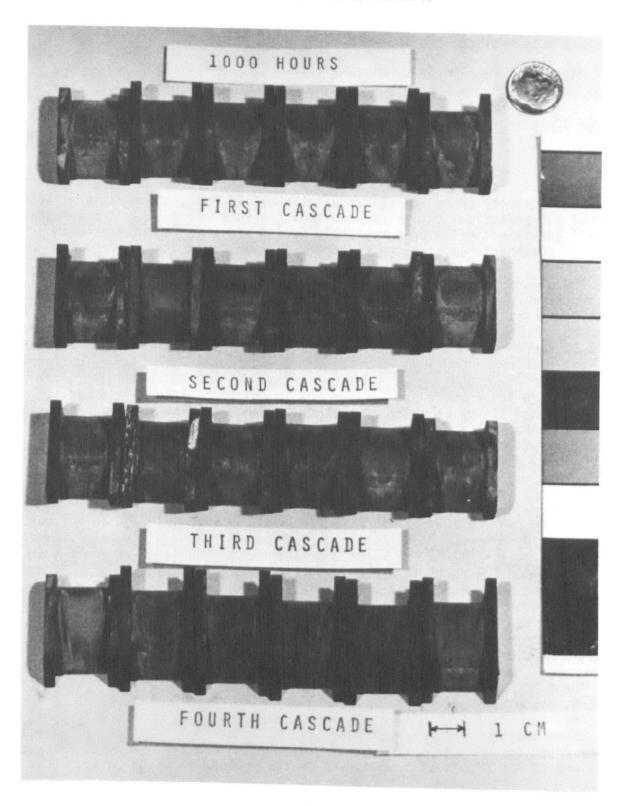


FIGURE III-20

MINIPLANT FLOW SCHEMATIC
HOT CORROSION/EROSION TEST CONFIGURATION



# FIGURE III-21 GE TURBINE BLADE SPECIMENS



Twenty-eight of the air cooled boiler tube probes designed by Westinghouse were exposed for up to 1117 hours. These were controlled at specified temperatures, and located both in and above the fluidized bed. Each probe contained two test materials. A picture of two probes is shown in Figure III-22. Results of the metallographic examination of the specimens will be reported separately by Westinghouse in the future.

Exxon will report run conditions including particulate loadings and other exposure conditions in a report to DOE later in 1979.

Effect of Natural Gas Injection --

Natural gas injection is used in the miniplant to maintain temperatures above  $840^{\circ}\text{C}$  (1550°F) for the DOE corrosion tests as well as to shorten the time necessary for thermal equilibrium in downstream components. Without natural gas injection, the temperatures at the tertiary cyclone would only reach their steady operating values after about 12 hours of operation. With natural gas injection this is reduced to 1-1/2 to 2 hours. For these reasons, natural gas injection is used routinely during miniplant operations when tests of components outside the combustor are run.

The effects of natural gas injection on flue gas composition and particulate characteristics have been investigated. One concern is that instantaneous combustion of the gas would cause hot spots, which could increase formation of  $NO_X$ , or alter particulate characteristics. Temperature measurements have shown that the gas does not begin to burn until it is several centimeters downstream of the injection point. Combustion then occurs uniformly over the next 2 meters before it is complete as indicated by a temperature decline. The temperature profile for the injection of natural gas can be seen in Figure III-23. During this study the amount of natural gas normally distributed among 4 probes was injected in only the first probe. During normal operations, the temperature rise is no more than  $20^{\circ}\text{C}$ , however, the shape of the profile for each probe is similar.

The only effect of natural gas injection on flue gas composition that has been measured besides a 0.5 to 1.0% reduction in flue gas oxygen is a drastic reduction in CO. During run 81, CO emissions fell from 525 ppm 1-1/2 hours after coal injection was begun to 30 ppm after natural gas injection into the flue gas lines was started. Reduction in CO emissions was also observed in runs 79 and 80 with natural gas injection. CO emissions observed in the past for miniplant runs without natural gas injection were in the range of 100 to 200 ppm. For the runs with natural gas injection, the residence time of the flue gas in the piping at the temperature range of 815 to 940% was about twice as long as for runs without natural gas injection. Thus, natural gas injection could provide increased opportunity for burnout of CO.

The effect of natural gas injection on flue gas particulates is also of concern, especially if the gas is responsible for the high collection efficiencies measured with the tertiary cyclone. Studies in the miniplant have shown that cyclone collection efficiencies are not significantly affected by the injection of natural gas. The slight effect that is measured is usually explained by the effect of temperature on the cyclone inlet velocity.

## FIGURE III-22

## WESTINGHOUSE BOILER TUBE PROBES After 1117 Hours Exposure (850°C In-Bed)

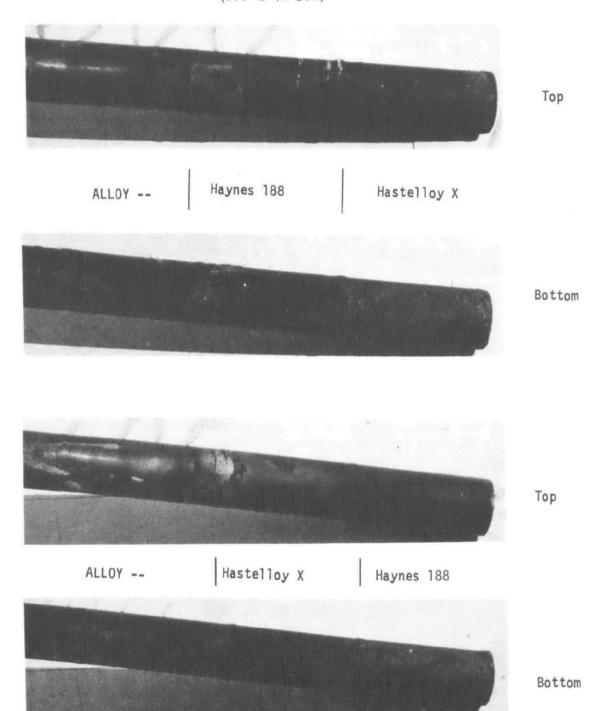
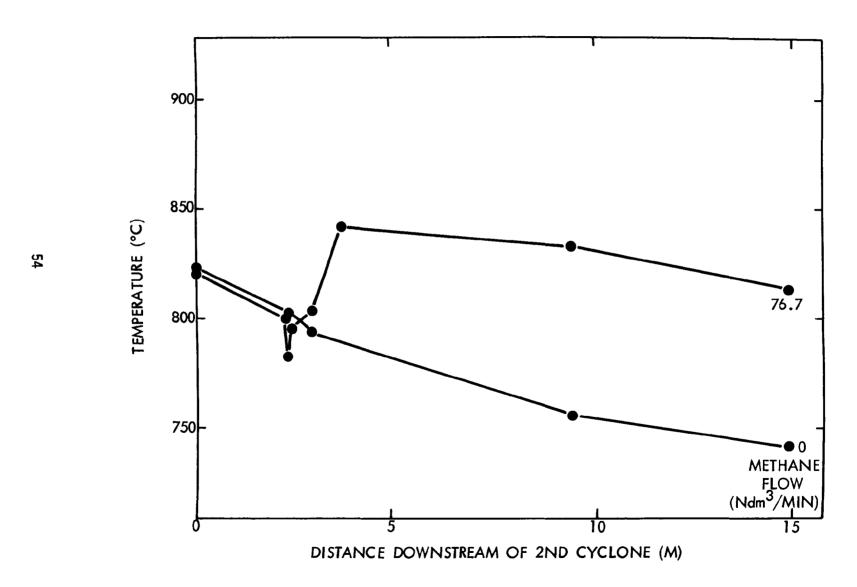


FIGURE 111-23
NATURAL GAS INJECTION - TEMPERATURE PROFILES



#### SO<sub>2</sub> Emission Control

As a result in a change in the New Source Performance Standards for coal fired utility boilers, the allowable SO<sub>2</sub> emission level was decreased from 1.2 lb/MBTU to a sliding scale which, for higher sulfur coals, requires 90% SO<sub>2</sub> retention. A series of runs were made to demonstrate that PFBC could attain the 90% SO<sub>2</sub> retention level using dolomite or limestone sorbents. Ohio and Illinois coals, containing 2.5 and 4% sulfur respectively were tested. Ca/S molar ratio was the primary variable in the study. With dolomite sorbent, the Ca/S ratio was varied from 1.5 to 2.1. With limestone, it was varied from 3.7 to 7.6. Superficial gas phase residence time in these tests was varied slightly from 1.6 to 2.2 s. Other conditions are summarized in Table III-3, which also tabulates the SO<sub>2</sub> retention results. As seen, 90% SO<sub>2</sub> retention can easily be reached with either sorbent. Retention levels as high as 99<sup>+%</sup> were measured. As expected, dolomite is more active. A Ca/S ratio of 1.5 will assure 90% SO<sub>2</sub> retention at a gas residence time of about 2s while limestone use will require a Ca/S ratio of between 3.5 and 4.0.

In subsequent runs, additional data were obtained in the high  $SO_2$  retention area. All  $SO_2$  retention data were then plotted against Ca/S ratio for both dolomite and limestone sorbents and analyzed. In the plots, the Ca/S ratio set on the coal/sorbent blender was used as the correlating parameter. In earlier runs, a Ca/S ratio calculated by mass balance was used because of blender speed control problems. Since the problems were solved by installing an electronic speed control system on the blender, it was decided to use the "set" Ca/S ratio since it appeared to be more reproducible, possibly because it did not require reliance on a series of chemical analyses of the solid and gaseous products.

Results with Dolomite Sorbent --

Figure III-24 gives SO<sub>2</sub> retention results using Pfizer dolomite sorbent, Champion, Illinois and Ohio coals, at gas residence times from 1.5 to 2.5s. Retention results shown in the figure were corrected to a residence time of 2s using the simple first order rate expression described in the previous report (2). Figure III-25 gives results of runs made with a residence time of 2.5 to 3.5s, corrected to a residence time of 3s. Since a significant amount of data scatter occurs on each plot, values from the smooth curves were used to construct a curve relating the first order rate constant for the SO<sub>2</sub> sorbent reaction, k, to the sorbent utilization. A set of values for k was calculated from the first order rate equation by using a range of retentions at constant residence times. The sorbent utilization was obtained from the smooth curves by using the expression for a sulfur balance:

% Ca Utilization = 
$$\frac{SO_2 \text{ retention (\%)}}{Ca/S \text{ ratio}}$$

If the  $SO_2$  reaction rate is indeed first order, then the values from the curves in Figures III-24 and III-25 would overlay on a k vs. utilization curve, all other variables being equal. However, when this was done, two k vs. utilization curves were obtained, one for each residence time. The reason for this was that the 3s residence time data did not show a significant improvement in  $SO_2$  retention over the 2s data, as would be predicted by the first

TABLE III-3. RUN SUMMARY 90% SO2 RETENTION STUDY

Nominal Operating Conditions	Run 68	Run 69	Run 70	Run 71	Run 72	Run 73	Run 74.1	Run 74.2	<u>Run 75</u>
Run Length (hrs)	10	11	11	13	13	11.5	4.5	5.75	9
Pressure (kPa)	930	930	930	930	930	930	930	930	930
Temperature (°C)	946	947	936	933	943	939	933	937	936
Superficial Velocity (m/s)	1.7	1.8	1.9	1.7	1.8	1.8	1.7	1.7	1.7
Expanded Bed Height (m)	2.8	3.0	3.0	3.8	3.7	3.8	3.7	3.7	3.8
Gas Residence Time (s)	1.6	1.7	1,6	2.2	2.1	2.1	2.2	2.2	2.2
Excess Air (%)	17	14	23	13	11	10	16.5	18.9	17
Coal Feed Rate (kg/hr)	127	132	127	122	116	117	113	112	115
Ca/S Molar Feed Ratio	2.0	1.5	1.5	1.5	1.8	2.1	7.6	4.8	3.6
Sorbent	PD	PD	PD	PD	PD	PD	GL	GL	GL
Coal	IL	IL	IL	IL	ОН	ОН	OH	ОН	ОН
Emissions									
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) O <sub>2</sub> (%) CO <sub>2</sub> (%) CO <sup>2</sup> (ppm)*	50	197	94	60	60	4	1	30	170
NO <sub>x</sub> (ppm)	79	56	81	40	30	50	80	60	50
02^ (%)	3.2	2.6	4.0	2.4	2.1	2.0	3.3	3.7	3.4
CÖ <sub>2</sub> (%)	16	16	17	17	22	18	19.5	19	15.2
CO <sup>2</sup> (ppm)*									
SO <sub>2</sub> Retention	98.5	94.2	96.9	98,1	99.0	99.7	99.9	98.2	90.9

Notes: PD = Pfizer 1337 Dolomite

GL = Grove 1359 Limestone
IL = Illinois Coal - 4% S
OH = Ohio Coal - 2.5% S
\* CO Analyzer Malfunctioned

FIGURE III-24

SO<sub>2</sub> RETENTION ADJUSTED TO 2 S. GAS RESIDENCE TIME (tg).
DOLOMITE RUNS HAVING ACTUAL tg BETWEEN 1.5 AND 2.5 S.

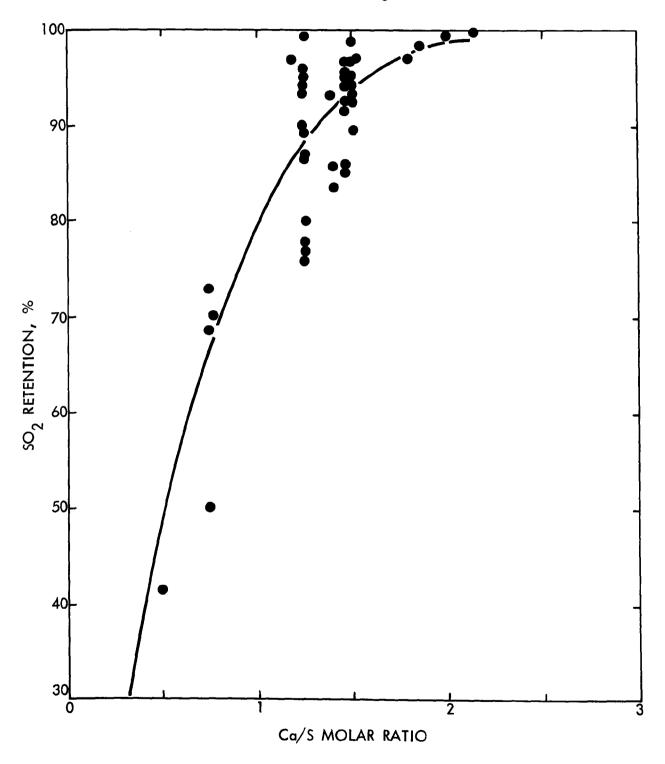
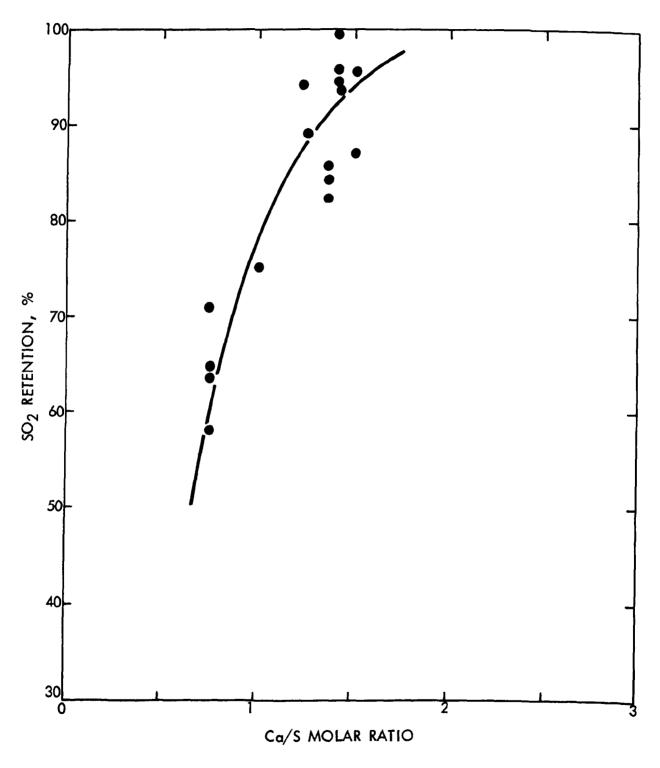


FIGURE 111-25
SO<sub>2</sub> RETENTION ADJUSTED TO 3 S. GAS RESIDENCE TIME (tg).
DOLOMITE RUNS HAVING ACTUAL tg BETWEEN 2.5 AND 3.5 S.



order expression. A plot of the mean k vs. utilization curve derived from the two retention curves is presented in Figure III-26. This curve is a compromise between the upper and lower limits formed by the 2s and 3s residence time data.

At present, there is no explanation for the lack of uniformity in the two groups of data, but one obvious observation would be that the reaction does not strictly comply with first order kinetics. This may be a problem inherent in large scale FBC operations where additional operating parameters, such as solids recycle and rejection, compound the difficulty of obtaining representative samples necessary to characterize the system.

To study the effect of residence time on SO<sub>2</sub> retention, the k vs. utilization curve in Figure III-26 was used to construct retention curves for four residence times. 2s and 3s retention curves were calculated and plotted with the miniplant data curve in Figure III-27. As seen, the two calculated surves straddle the data curve reasonably well, indicating that effects of gas residence time on SO<sub>2</sub> retention become more difficult to discern as residence time increases. This can be seen more clearly in Figure III-28, where retention curves for all four residence times were drawn. Here it becomes obvious that below 2s, residence time has a significant effect on SO<sub>2</sub> retention, while above 3s this effect would necessarily become less substantial, particularly at Ca/S ratios greater than 1.

Additional work is obviously needed before the effect of gas phase residence time as well as other kinetic parameters can be accurately predicted. Figure III-28 can be used as an approximate guide until a better understanding is developed.

Results with Limestone Sorbent --

Figure III-29 gives SO<sub>2</sub> retention results obtained with limestone sorbent at temperatures high enough to calcine the limestone substantially. As seen in the previous figures with dolomite, the data scatter is significant. With limestone, the degree of calcination and porosity of the calcined stone are other parameters in addition to the kinetic effects discussed above. Again, more work is needed to improve understanding of the desulfurization system.

It should also be mentioned that limestone not only is less active than dolomite under normal PFB combustor operating conditions, but becomes completely inactive at temperatures at or below  $760^{\circ}\text{C}$  (2). This must be considered if limestone is to be used and if low temperatures are expected, for example, to aid in decreasing the steam output from the combustor.

#### SO2 Dynamic Response

The new SO<sub>2</sub> emission standards call for 90% retention of the SO<sub>2</sub> from new coal fired utility boilers. This retention must be averaged over 30 days. It is expected that utilities would operate at slightly higher retentions to avoid exceeding the new limit and avoid costly violations. The incentive therefore exists to develop a system to control SO<sub>2</sub> retention close to the desired level without large safety margins. With this goal in mind, General Electric has set out, under contract to the EPA, to develop a control system that would adjust sorbent feed rates to assure a specified SO<sub>2</sub> retention despite variations in

FIGURE 111-26

FIRST ORDER RATE CONSTANT VS. CALCIUM UTILIZATION
MINIPLANT DOLOMITE RUNS
(APPROXIMATE ONLY)

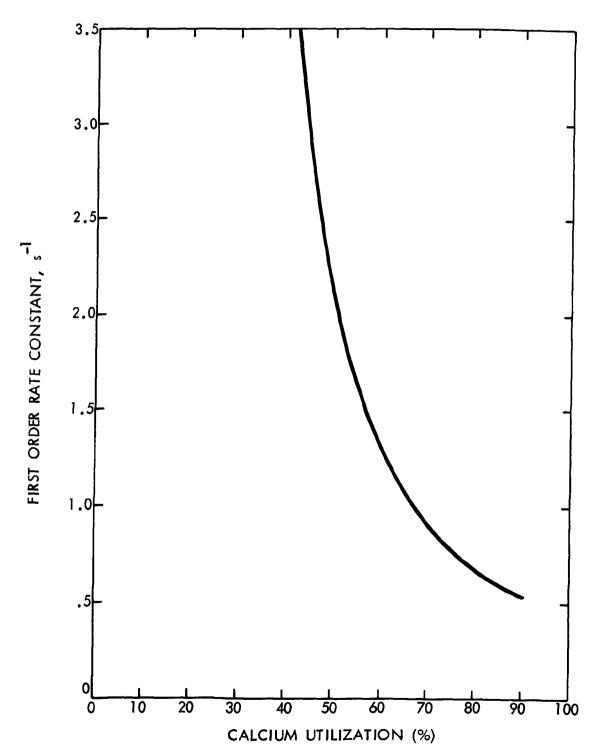


FIGURE III-27

MEASURED AND CALCULATED SO<sub>2</sub> RETENTION VS. Ca/S FOR MINIPLANT DOLOMITE RUNS

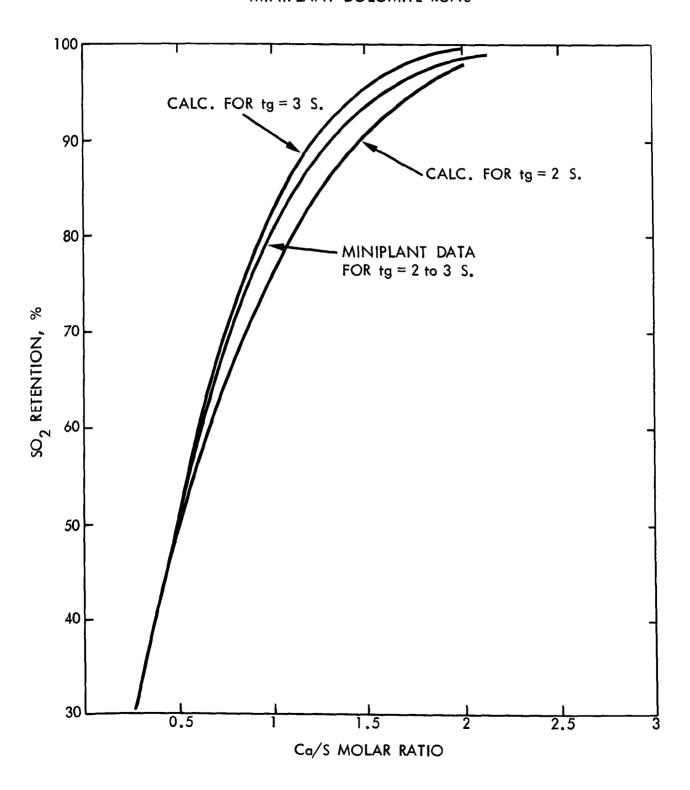


FIGURE 111-28

CALCULATED SO<sub>2</sub> RETENTION VS. Co/S

MINIPLANT DOLOMITE RUNS

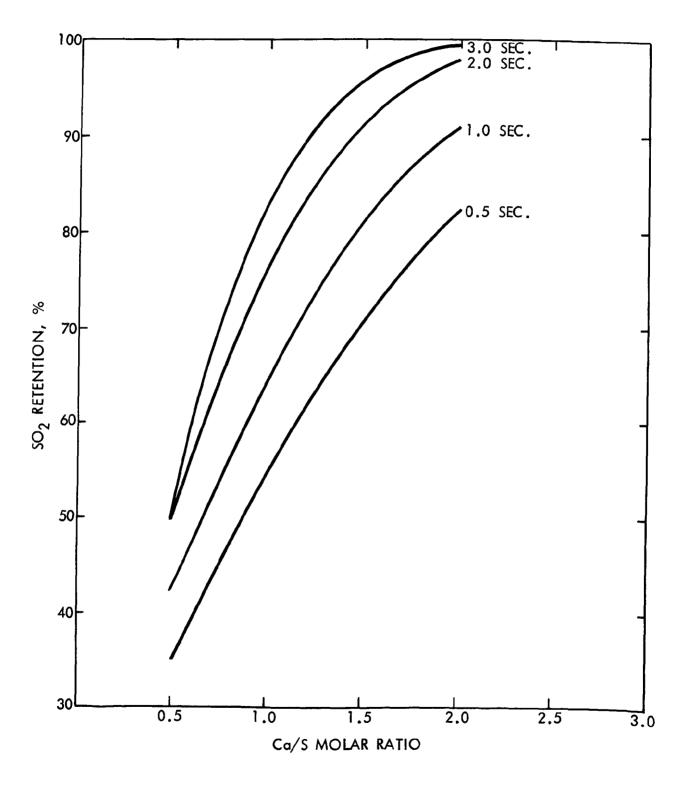
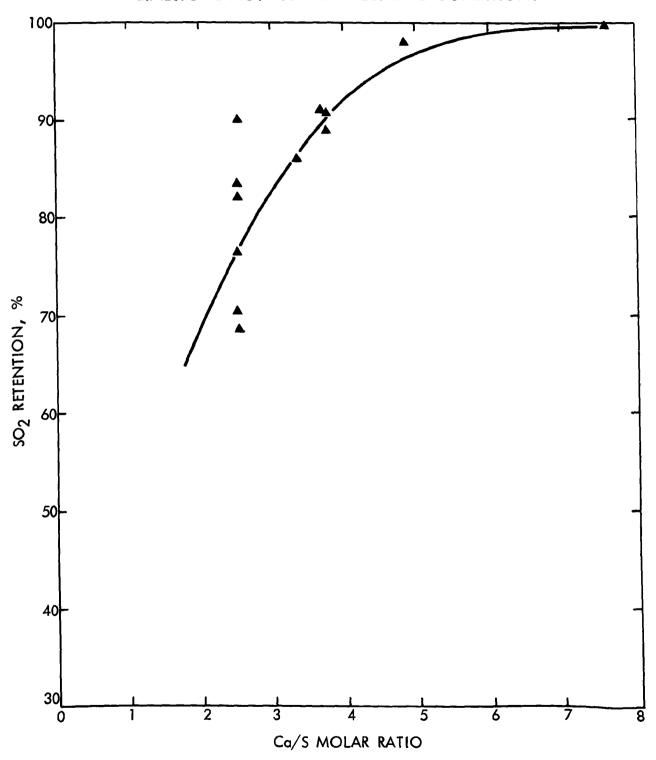


FIGURE III-29

SO<sub>2</sub> RETENTION VS. Ca/S RATIO FOR LIMESTONE NO. 1359 AT CALCINING CONDITIONS



the sulfur content of the coal, and in the reactivity of the sorbent. Tests were carried out on the miniplant in cooperation with GE, to determine the SO<sub>2</sub> emissions response to a step change in either coal sulfur or dolomite feed rate, with other variables held constant.

Experimental Equipment and Procedures--

At GE's request, the miniplant was run with no primary cyclone recycle during the SO2 response tests. The combustor was allowed to reach a steady state for 4 to 5 hours before a change of condition was made. During this period, the old, continuous, primary feed system was used. Once steady state emissions were achieved, the change was made to the auxiliary feed system containing a different coal/sorbent blend. The auxiliary feeder was used for 8 hours, the approximate capacity of the vessel. After 8 hours on the auxiliary feeder, the change was made back to the primary feeder and the original coal/sorbent blend. The combustor was then run for 18 to 20 hours with the primary feeder. The 18 hours were required to assure two changes of the bed material inventory.

The procedure for changing from one feeder to another consisted of:

- 1. Calibrating the analyzers 30 minutes before the change.
- 2. Closing the valve at the bottom of the active feeder.
- 3. Allowing the coal transport line to clear of solids (~20 sec.)
- 4. Changing over  $\Delta P$  taps, transport air and electronic controls to the other vessel.
- 5. Opening the valve at the bottom of the other vessel.
- 6. Confirming coal feed by non-zero SO<sub>2</sub> emissions and combustor temperature increase.

The time required for the change of feed vessels was 40 to 80 sec, depending on crew experience and operating conditions.

Results and Discussions--

Shakedown-Shakedown of the auxiliary feed system went smoothly during runs  $\overline{97}$  and  $\overline{98}$ . Several modifications made after run  $\overline{97}$  allowed the time required for the change to be reduced from 4 minutes to  $\overline{40}$  sec during run  $\overline{98}$ . The resultant combustor temperature drop was reduced from  $\overline{260}$  to  $70^{\circ}\text{C}$ . Table III-4 lists the results of step changes in the coal source during shakedown as well as in the actual runs. The time listed is the time required from the confirmation of coal feed by non-zero  $\overline{502}$  emissions to the first time the  $\overline{502}$  emission reached the equilibrium value. A large, short  $\overline{502}$  "spike" was ignored in this summary since it was due to the rapid combustor temperature drop and subsequent coal feed rate increase caused by the action of the coal feed controllers. These temperature fluctuations may be unique to the miniplant and may not appear in a commercial installation.

Change in Coal Sulfur--The first actual response test conducted on the miniplant (run 99) was a step change in coal sulfur content at a constant dolomite mass feed rate. The miniplant was started with Champion coal and dolomite (1.9% sulfur, Ca/S = 1.40). After 4 hours, the combustor was switched to the auxiliary feeder which contained Illinois No. 6 coal and dolomite (3.4% sulfur, Ca/S = 0.76). Then, after 8 hours, the combustor was switched back to

Run #	Type of <u>Change</u>	From (2)	То	SO <sub>2</sub> Before (ppm)	SO <sub>2</sub> at Steady State (ppm)	Time from Feed Change to Steady State (min)
97 (1)	% S	Champion	Illinois	90	675	6-3/4
97 (1)	% ·S	Illinois	Champion	675	225	7
<sub>98</sub> (1)	% S	Champion	Illinois	225	675	6
98(1)	% S	Illinois	Champion	675	225	4
98(1)	% S	Champion	Illinois	225	675	5
<sub>98</sub> (1)	% S	Illinois	Champion	675	225	7-1/2
99	% S	Champion	Illinois	225	675	7-1/2
99	% \$	Illinois	Champion	675	225	8-1/4
100 <sup>(3)</sup>	Ca/S	Ca/S = 1.43	Ca/S = 0.38	375	500	110
100 <sup>(3)</sup>	Ca/S	Ca/S = 0.38	Ca/S = 1.43	500	300	200

<sup>(1)</sup> Shakedown Runs

55

<sup>(2)</sup> Champion Coal -1.92% Sulfur (Ca/S = 1.40 during tests where % S changed) Illinois No. 6 coal -3.4% Sulfur (Ca/S = 0.76 during tests where % S changed)

<sup>(3)</sup> Champion Coal Used
Pfizer dolomite used in all tests

the Champion coal, dolomite blend for 20 hours. The SO<sub>2</sub> emission, averaged over 10 minute intervals, over the entire run is shown in Figure III-30. Figures III-31 and III-32 show the continuous SO<sub>2</sub> analyzer output immediately before and after the two changes in coal sulfur. The response times of 7.5 and 8.2 minutes were determined from these figures. During the change to the higher sulfur coal, the dolomite mass feed rate actually increased 19% although it was intended to hold it constant. The effect of a 19% increase in dolomite mass feed rate was superimposed on the 104% increase in coal sulfur mass feed rate occurring because of the change. The change back to the lower sulfur coal also had a 19% decrease in dolomite mass feed rate. The unintended change in the dolomite mass feed rate was caused by slight changes in the sulfur content and heating value of the coal actually used, versus prior analyses of other batches used to set conditions.

During the combustion of Champion coal,  $SO_2$  emissions were close to the predicted level of 170 ppm. This prediction comes from Figure III-25 of the previous section. The predicted  $SO_2$  emission for the Illinois No. 6 coal is 1080 ppm, much higher than the 725 ppm measured.

Change in Sorbent Feed Rate--During run 100, the SO2 emission response to a setp change in Ca/S from 1.43 to 0.38 using Champion coal and dolomite was investigated. The combustor was started with the higher Ca/S which was then rapidly changed to the lower Ca/S by switching to the auxiliary feed system. The first attempt to switch to the auxiliary feed vessel failed due to a plug in the transport line. The plug was cleared while the combustor was maintained on liquid fuel. The low Ca/S coal was fed from the auxiliary vessel for 20 minutes to determine the likelihood of another plug. The combustor was switched back to the high Ca/S blend until the SO<sub>2</sub> emission stabilized at the preswitch level (about 2 hours). The combustor was again switched to the low Ca/S blend in the auxiliary feeder. After 8 hours of operation on the low Ca/S blend. the change was made back to the higher Ca/S blend. During this final run segment, coal was fed for 19 hours to assure that steady state had been attained. From Figure III-33 the response time for the step from Ca/S = 1.43 to 0.38 is shown to be 110 minutes. The reverse step required 300 minutes to achieve steady state. It is unexplained why the original steady emission, at startup (about 375 ppm), was not the same as the final segment emission (300 ppm). At no time during the run was the average SO2 emission as close to the predicted emission as expected. The predicted emission for Ca/S = 1.40 is 170 ppm. actual average emission during the 19 hour final portion of the run was 300 ppm. During the low Ca/S portion (mid portion) of the run, the actual average emission was only 500 ppm compared to 750 ppm predicted. This may be due to the fact that the low Ca/S operating region has not been well defined experimentally. The time required during the second change (to the higher Ca/S) to reach the "startup emission" of 375 ppm was approximately 120 minutes. This suggests that there is a reversibility in the response times for these changes. The SO2 emission just prior to and just after the coal sorbent blend changes can be seen in Figures III-34 and III-35. The immediate effect of both changes is not noticeable since the response times are fairly long.

#### Conclusions --

For the typical 90% SO<sub>2</sub> retention levels expected to be used in most pressurized fluidized bed combustors, the response to 100% increase in coal sulfur

FIGURE III-30

CHANGE IN COAL SULFUR/SO<sub>2</sub> EMISSIONS (RUN 99)

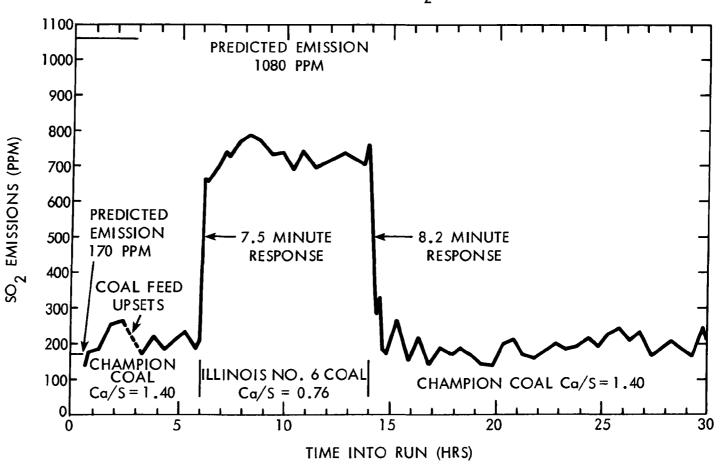
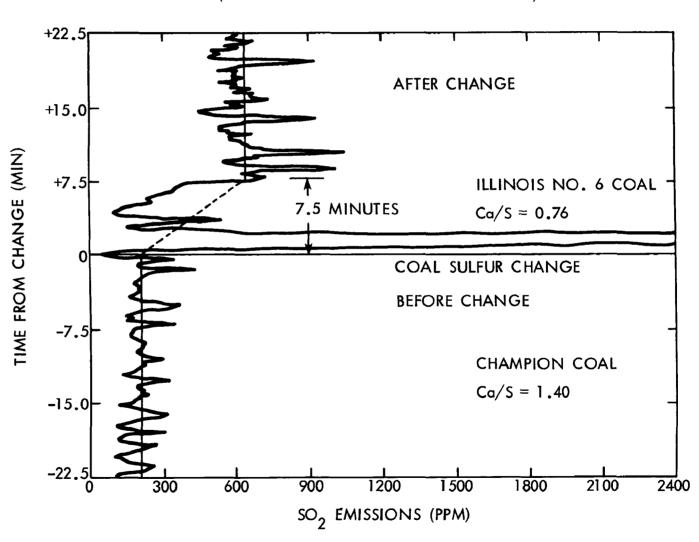
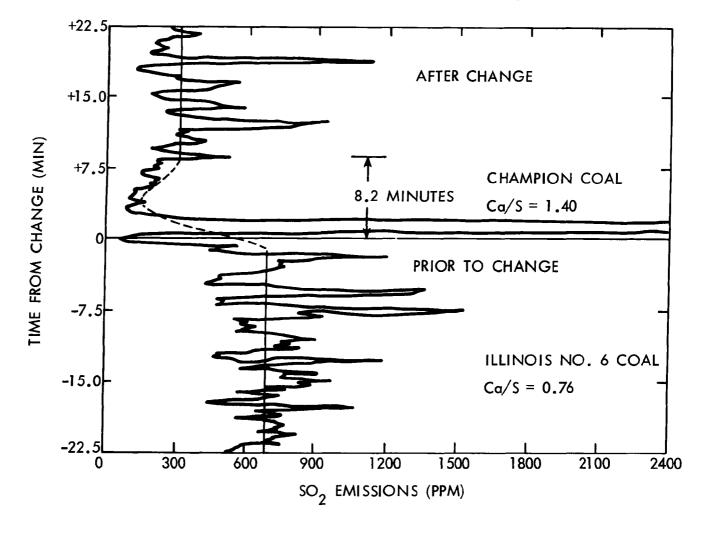


FIGURE III-31
INSTANTANEOUS SO<sub>2</sub> RESPONSE (RUN 99)
(CHAMPION TO ILLINOIS NO. 6 COAL)

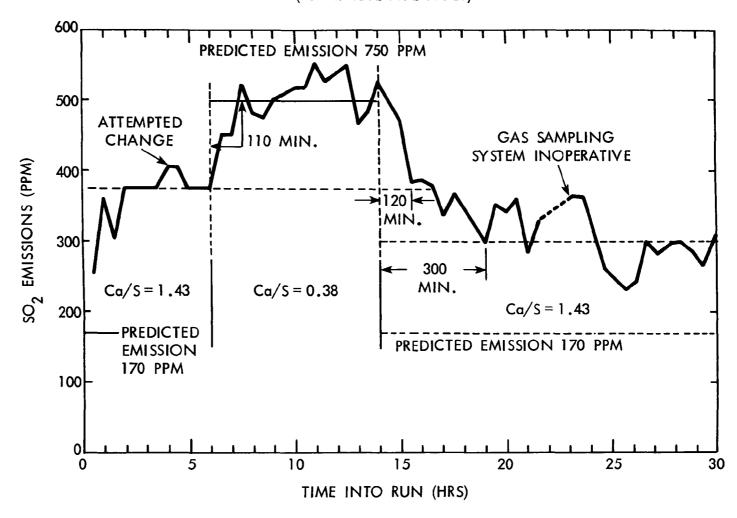


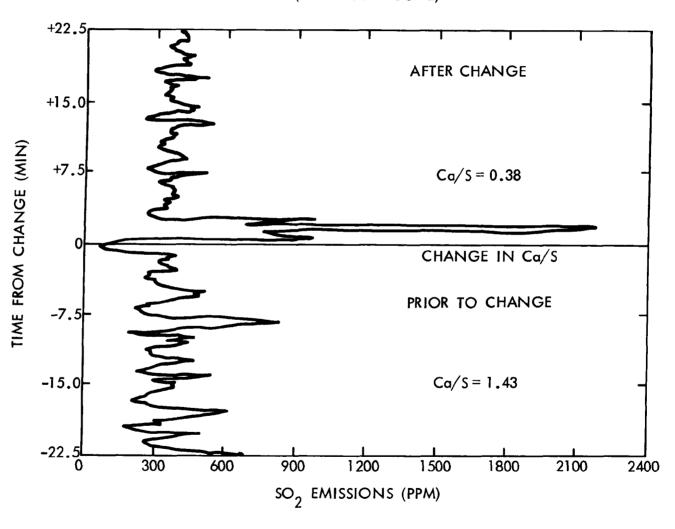


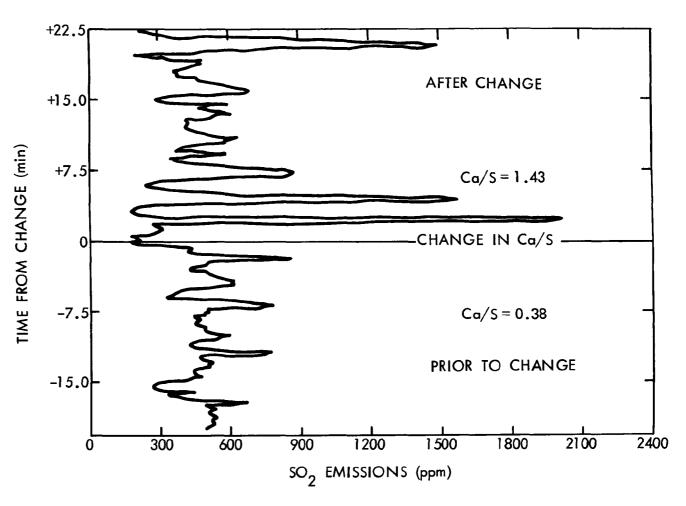
70

FIGURE III-33

# RUN 100 SO<sub>2</sub> EMISSIONS (10 MINUTE AVERAGES)







was fairly rapid (~8 minutes). The response to a 73% change in dolomite feed rate required 2 to 5 hours to reach a stable emission. This would indicate that it may not be possible to prevent sudden changes in SO<sub>2</sub> emissions which respond rapidly to variations in coal sulfur content with changes in sorbent feed rate which require much longer response times.

The fact that most emissions were far from the predicted emission levels, may suggest that there is an initial short response time coupled with a very much longer response time. Chemical analyses of the sorbent, bed extract material, and cyclone dumps have been sent to GE. They are incorporating these data into their SO<sub>2</sub> emission response model and will issue a separate report with their conclusions.

#### NO<sub>X</sub> Emissions

 $NO_X$  emissions measured in all miniplant runs are plotted in Figure III-36 as a function of percent excess air. Data follow the same trend line shown in previous reports (1).  $NO_X$  emissions are well below the recently modified New Source Performance Standard of 0.6 lb/MBTU for coal fired utility boilers. Most PFBC design studies have indicated the excess air level will most likely be around 20%. At this excess air level,  $NO_X$  emissions are expected to be 0.2  $\pm$  0.1 lb/MBTU. Emissions are expected to be below 0.4 lb/MBTU, even at excess air levels in the range of 60 to 100%.

An evaluation of methods which could decrease  $NO_X$  emissions below those in Figure III-36 was carried out in the bench PFB combustion unit. The results of this study are given in Section VII.

#### Other Gaseous Emissions

SO3 emissions in the combustor flue gas were measured for most runs. For runs up to run 75, the EPA Method 8 method was used. For subsequent runs, the controlled condensation method described in Section III was used. Results for all measurements is given in Appendix Table G. Two measurements out of a total of 40 appeared to be in error and were discarded. These were readings of 213 ppm from run 71 (Method 8), and 73 from run 100 (controlled condensation). Excluding these two measurements, 16 Method 8 measurements averaged 12 + 12 ppm, 22 controlled condensation measurements averaged 6 + 9 ppm. All measurements averaged 9 + 11 ppm. In both cases, the range of measurements was 0 to 30 ppm. This is the same range reported previously when only the Method 8 technique was used (1). Aside from the fact that the controlled condensation method gave an average reading exactly one half of the Method 8 average, no positive conclusions could be drawn from the results. Even the difference between the average measurements is not statistically significant due to the large degree of uncertainty. In both cases, the degree of uncertainty was approximately + 100% of the average values. Large variations were also measured within a single run. Therefore, the variation appears to be random and no conclusions could be drawn regarding the cause of SO3 formation or the factors affecting the degree of SO3 formation.

CO emissions were again in the range of 50 to 200 ppm as reported previously (1). A few measurements were in the 300 to 500 ppm range, but could have been caused by analyzer problems.

FIGURE III-36 CORRELATION OF NO  $_{\rm X}$  EMISSIONS 0.8 0.7 0.6 NO (Ib/M BTU) 0.5 0.4 0.2 120 140 100 ol O 80 60 40 20 EXCESS AIR, %

Reduced sulfur compounds, H<sub>2</sub>S, COS and CS<sub>2</sub> in the flue gas were measured by gas chromatography (GC) a number of times and were consistently less than the detectability limit of 1 ppm. Hydrocarbons were also measured by GC. Methane averaged  $7 \pm 5$  ppm, ethane  $4 \pm 4$  ppm, C<sub>3</sub> through C<sub>6</sub> hydrocarbons were generally below the detectability limit of 1 ppm. GC results are given in Appendix Table F.

Emission of sodium, potassium, chlorine and vanadium in the flue gas was also analyzed using the system described previously. In this system, a sample of high temperature, pressure flue gas is extracted from the ducting following the third stage cyclone, partly filtered, cooled, filtered again, cooled further and bubbled through an impinger train (see Figure III-37). Figure III-37 also shows the different locations where the four elements are condensed, deposited or absorbed and subsequently analyzed. The filters are extracted with boiling water and the extract analyzed. Unused filters are also extracted to give a blank for the filter material itself. The quartz tube is washed and the wash solution analyzed. Table III-5 gives the distribution of the four elements and particulates found in the six locations in the train. Sodium totalled 2 to 3 wppm in the inlet flue gas, potassium 0.3 to 0.5 wppm. Generally, over 90% of these elements were found on the front and final filters (sampling locations 1 and 3 in Figure III-37). The front filter was not retaining all the particulates impinging on it, as seen by the large fraction (60 to 70%) of particulates captured by the final filter in samples 78 and 79-1. The front filter media was removed for sample 79-2. However, the fraction of total alkali captured on the final filter was greater than the fraction of particulates captured on the final filter. Therefore, sodium and potassium compounds were condensing in the quartz tube on the surface of particulates which were removed by the final filter. Very little alkali compounds were condensing and collecting on the surface of the quartz tube itself.

The concentration of chlorides measured in the flue gas was about 50 wppm. This represents about 60% of the total Cl entering with the coal and dolomite feeds. As seen in Table III-5 chlorides were not detected on the particulates but only in the knockout condensate. Therefore, chlorides were probably all present as HCl at the low temperature conditions in the sampling probe. Sodium and potassium were then probably present as the sulfates in the sampling system, although they probably occurred as chlorides in the high temperature flue gas prior to sampling.

No vanadium was detected in any of the samples.

## Particulate Emissions

Particulate emissions from the miniplant depend heavily upon the particulate cleanup system used. The emission when the flue gas is cleaned by a granular bed filter is very different from that when it is cleaned with cyclones. During this reporting period almost all runs were made with 3 stages of cyclones in series. For this reason, this section will deal mostly with emissions measured in the flue gas cleaned in this manner. Emissions using other control technology are contained in Section IV.

FIGURE III-37 SCHEMATIC OF THE ALKALI PROBE TRAIN

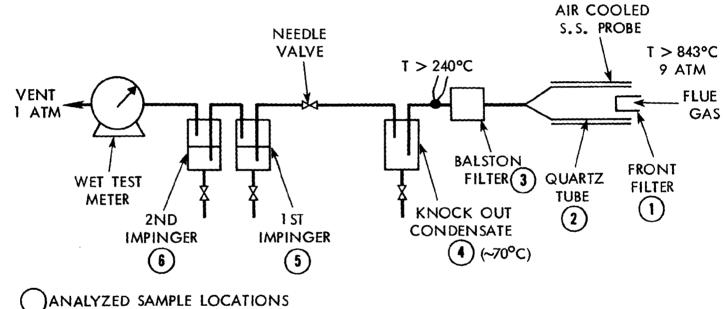


TABLE III-5. EMISSION OF SODIUM, POTASSIUM, CHLORINE, VANADIUM IN FLUE GAS

Run/Sample No.		78/	4				79/	'1				79/	2	
Element	Na	<u>K</u>	<u>v</u>	Part.	Na	<u>K</u>	<u>v</u>	<u>C1</u>	Part.	Na_	<u>K</u>	<u>v</u>	<u>C1</u>	Part.
Concentration (wppm)	2.06	0.54	0		1.84	0.28	0	47 .4		3.23	0.38	0	53.8	
Distribution (wt. %) <pre>Location</pre>														
1 - Front Filter	7	37	-	39	1	5	-	0	31	< 1	6	-	0	3
2 - Quartz Tube	< 1	3	-	0	2	14	-	0	0	2	8	-	0	0
3 - Final Filter	89	59	-	61	96	80	-	0	69	95	83	-	0	97
4 - Condensate	3	1	-	0	1	1	-	100	0	2	3	-	100	0
5 - 1st Impinger	< 1	0	-	0	0	0	-	0	0	0	0	-	0	0
6 - 2nd Impinger	< 1	0	-	0	0	0	-	0	0	0	0	-	0	0

Particulate emissions during this reporting period generally ranged from 0.03 to 0.15  $g/Nm^3$  (0.013 to 0.065 gr/SCF) with 3 stages of cyclone cleanup. These gas particulate concentrations were measured with Balston total filters in either the Balston filter sampling system or the HTHP sampling system. The size distribution of particulates in the filter cake was obtained with the Coulter Counter utilizing techniques described in Appendix D. The size distributions of most samples are recorded in Appendix M. Table III-6 gives particle size distributions in the flue gas leaving the third cyclone for two runs which span the expected range.

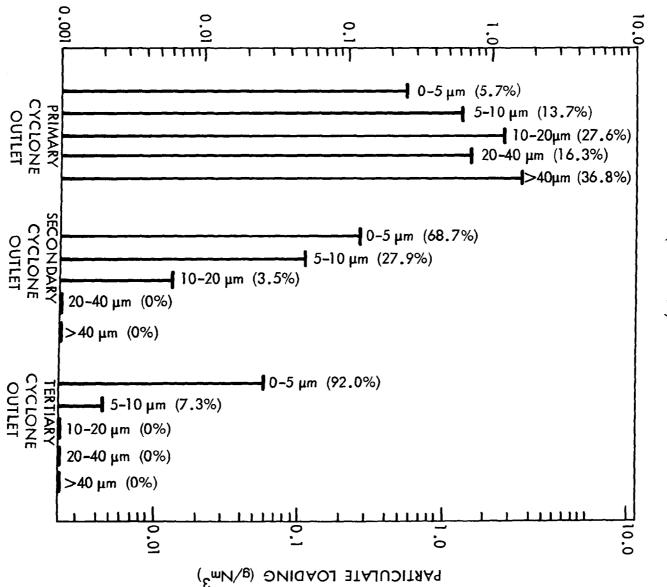
TABLE III-6. PARTICULATE EMISSION PARTICLE SIZE DISTRIBUTION

			Par	rticle Size			
Run	5%	10%	25%	50%	75%	90%	95%
No.	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than
78	0.58	0.66	0.98	1.9	4.6	8.5	11.0
80	0.56	0.66	0.85	1.2	1.8	3.4	5.4

The particulate loading of the PFBC effluent at various locations within the off gas system during a typical run (run 80) is shown in Figure III-38. Run 80 is typical of good long term operations. The concentrations are broken down into five principal size ranges for material leaving each cyclone in the gas stream. These loadings were obtained from mass balance calculations around the cyclones. Size analysis was performed on a Balston filter catch, as well as second and third cyclone dump material taken at a similar time as the Balston filter flue gas particulate samples were being collected. These size analyses together with the mass collected by each cyclone and on the Balston filter were used to calculate the loadings and size distributions leaving the primary and secondary cyclones. The only assumption made was that the particles did not change size significantly during their pass through the cyclone. Total particulate concentrations and median particulate sizes in the flue gas at the same three points, and median particulate sizes in material captured in the secondary and tertiary cyclones, are given in Table III-7. These data cover the range of values measured in a large number of individual samples from many runs. A comparison of the data in Figure III-38 and the range of values in Table III-7 shows that run 80 was a typical run.

TABLE III-7. PARTICULATE CONCENTRATION AND SIZE RANGES REPRESENTING A NUMBER OF RUNS

Cyclone	Part. Conc. (g/m <sup>3</sup> ) <u>Passing</u>	Mass Median Captured	Size (µm) Passing	Cyclone Efficiency (%)
Recycle (Primary)	8-12	-	20-25	-
Second	0.4-1.2	20-25	3-5	90-95
Third	0.03-0.15	3-5	1-3	85-94



PARTICULATE LOADING (gr/SCF)

PARTICLE CONCENTRATION IN CYCLONE GAS OUTLETS (RUN 80.2)

FIGURE III-38

The primary cyclone was intentionally designed with a low efficiency. Its primary function is to return the larger particles back to the bed. During normal operations, the material in the cyclone dipleg has the size distribution shown in Figure III-39. At times this dipleg has become plugged and operations have continued as in run 81. During this type of operation, there is a large increase in the material captured in the second cyclone, a slight increase in the third cyclone capture and a large reduction in the bed overflow solids rate. This change in the mass balance envelope reflects the greater amount of material entrained from the combustor and recycle cyclone and captured in the last two cyclones. At the exit of the third cyclone, the only significant difference from normal operations when the primary cyclone dipleg plus is a 3-5 percentage point increase in particles over 10  $\mu$ m. The particulate concentration and average particle size are not changed significantly. A further discussion of run 81 is contained in another report (6).

The secondary cyclone is in reality the first cleanup cyclone. As shown in Table III-7 this cyclone captures 90 to 95% of the particulates in the flue gas exiting the recycle cyclone. The average particle size of the particulates captured by and passing through the cyclone is 20-25  $\mu$ m and 3-5  $\mu$ m respectively.

The third cyclone in the train -- the second cleanup cyclone -- captures approximately 90% of the particulates which exit the second cyclone. The particulates exiting this cyclone with the flue gas have a mean diameter of 1 to 3  $\mu m$ . Of the particulates leaving the cyclone with the flue gas, normally 80 to 90% are smaller than 5  $\mu m$ .

During the course of particulate sampling, many samples of particulates were found to have magnetic particles in them. These samples required special techniques for Coulter Counter size analysis, these techniques are described in Appendix D. The possibility of enhanced particulate collection by magnetic interactions was investigated. Figure III-40 is a magnetization curve for particles captured by the third cyclone during runs 99 and 100. Figure III-41 shows the effect of temperature on magnetic induction. Clearly this effect is not useful in the temperature range of interest (800-900°C). Furthermore based on the type of response curves seen in the two figures, it is evident that the magnetization is primarily due to the presence of ferrites in the ash.

## Combustion Efficiency

Carbon combustion efficiencies for runs made since the last report were consistently above 99%. Only three runs out of a total of 39 were below 99%. The correlation published in the previous report (1) which relates combustion efficiency to the average bed temperature, excess air level and gas residence time, was used to predict combustion efficiencies for these runs. The calculated efficiencies were consistently lower than the measured efficiencies. The average calculated efficiency was 98.5% compared to 99.3% measured, an average miss of 0.8 combustion efficiency units. The standard error for the correlation was previously reported to be 0.6 units but without the bias shown in the recent runs. If a consistent measurement error occurred

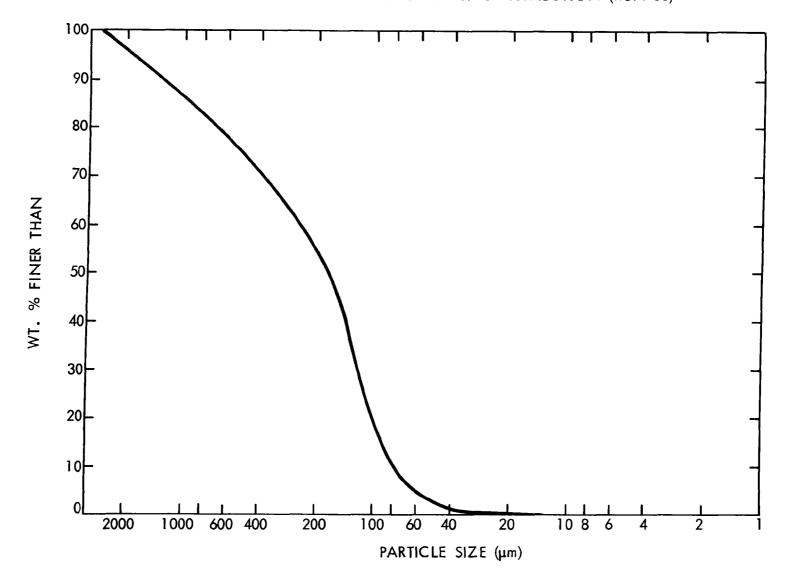


FIGURE III-40

MAGNETIZATION CURVE FOR THE MINIPLANT FLYASH SAMPLE AT 25°C

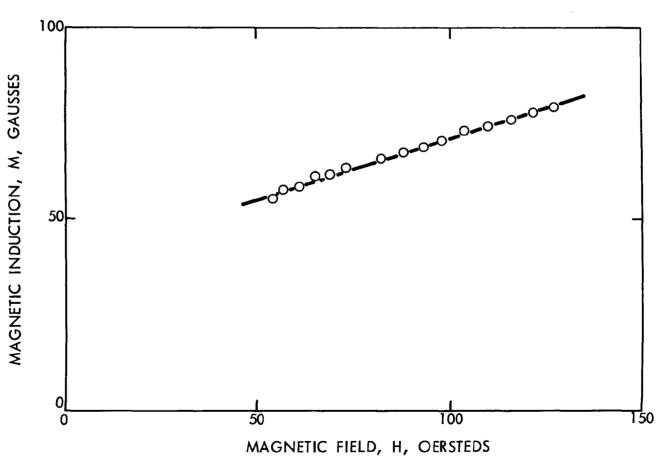
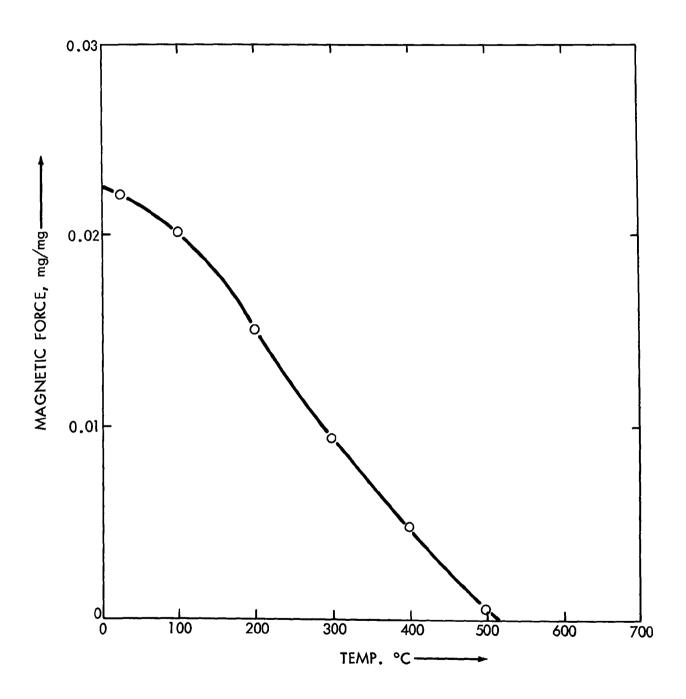


FIGURE 111-41

EFFECT OF TEMPERATURE ON THE MAGNETIC INDUCTION
FORCE OF THE MINIPLANT FLYASH SAMPLES



in the recent runs, the error was apparently less than 1%. Therefore, a conservative estimate of the average combustion efficiency from recent runs, obtained from the correlation, would be 98.5%. This is within the targeted range for a commercial PFBC facility.

#### SECTION IV

#### PARTICULATE MEASUREMENT AND CONTROL

An important technical issue to be resolved before pressurized fluidized bed combustion can be applied commercially is the degree of particulate removal needed to protect the gas turbine. A related issue is which technology to use to achieve the needed degree of the particulate control. In addition to meeting the particulate removal requirements set by the gas turbine, the environmental New Source Performance Standards published by the Environmental Protection Agency must also be met.

The degree of particulate removal needed to protect the gas turbine from erosion, hot corrosion and deposition has not been defined as yet. Studies, including the 1000 hour turbine cascade exposure test recently completed in the miniplant for the Department of Energy, are now underway to define the needed degree of particulate control. Although the EPA is not directly concerned with studying particulate control to protect gas turbines, it is responsible for the evaluation of such particulate control devices to the extent that these devices could determine the level, size and composition of particulate matter emitted from a PFBC system. Therefore a particulate control program was begun in which pre-turbine devices such as granular bed filters, a ceramic fiber filter and high efficiency cyclones were evaluated. In addition, post-turbine devices were also tested, based on the realization that the degree of pre-turbine cleanup may not be sufficient to meet the more stringent New Source Performance Standards for particulates. In this case, a trailer mounted electrostatic precipitator (ESP) and a trailer mounted bag house were connected to the miniplant flue gas system and tested under typical low pressure, low temperature conditions. A number of these tests were conducted in cooperation with other EPA contractors. A ceramic fiber filter, built and tested by Acurex Company, was provided by Acurex and tested jointly with them. The mobile ESP and bag house were also tested in cooperation with Acurex. These devices are operated by Acurex, for the EPA, on a number of industrial gas sources.

The cyclone tests were carried out primarily by Exxon. However, Southern Research Institute and Air Pollution Technology, both EPA contractors, conducted a series of particulate sampling tests to confirm cyclone performance results obtained by Exxon using a different particulate sampling system.

In addition to evaluating the particulate control devices, it was also necessary to develop and improve particulate measurement systems. Such systems were used to determine particulate concentrations in the flue gas before and after the control devices and to measure particulate size distributions.

The results of the particulate measurement and control programs are reported in this section.

#### PARTICULATE MEASUREMENT

Particulate measurements in the high pressure, high temperature flue gas are difficult to make. A lot of data have been obtained with 18 cm Balston high temperature total filters. These filters were used on all three sampling systems described earlier. The particles captured on the filters were sized with a Coulter Counter Model TAll as described in Appendix D. However, there is some question as to whether the particulate size distribution measured by the Coulter Counter is the same as that actually occurring in the flue gas before sampling. For this reason sampling was attempted with the Southern Research Institute (SoRI) 5-cyclone train and the University of Washington (UoW) 7-stage impactor using the HTHP sampling system for an in-situ size distribution measurement. Verification of results was also made by other EPA contractors (Southern Research Institute and Air Pollution Technology) with impactors designed for the high temperature, high pressure conditions. In order to understand better the instantaneous particulate emission, an on-line particle concentration monitor was used. This device was developed by IKOR. Inc. of Gloucester, Massachusetts. It is based on a charge transfer principle

#### Balston Filter Measurements

Balston filter samples have been used to build a large data base of cyclone, granular bed filter and ceramic barrier filter performance. This total filter gives a good measurement of the particulate concentration provided good sampling procedure is followed. Obtaining the size distribution of this material can be a problem. The Coulter Counter method used to size the particulates requires that a small amount of solids be dispersed in an agitated electrolyte (Isoton II). The particles are then drawn through a precision orifice. A small current continuously passing through the orifice is interrupted each time a particle passes through. The degree of interruption is proportional to the particle volume. An accumulator records the number of these interruption pulses whose magnitude falls in each of the 16 accumulator channels. The particle diameter reported is for a sphere of the same volume as the particle.

One question that arises is whether the particulates are agglomerated in the flue gas or on the filter and to what degree they are redispersed in the agitated electrolyte. Depending on the relative extent of agglomeration and redispersion, the measured particle size distribution could be either coarser or finer than that occurring in the flue gas. Also sufficient particulate must be captured to form a filter cake. If there is no cake formed, filter material may contaminate the sample. Filter material consists of fine, long cylindrical particles with a volume mean diameter of about 9  $\mu m$ .

A cyclone or other cleanup device sees the aerodynamic equivalent diameter. This is different from the volumetric diameter measured by the Coulter Counter. In order to determine the validity of Coulter Counter measurements for approximating aerodynamic size, some material was sent to General Electric for size analysis with a Bahco particle size analyzer. A comparison between the Coulter Counter and the Bahco results for both second and third stage cyclone capture material is shown in Figures IV-1 and IV-2. The distributions

FIGURE IV-1

COMPARISON OF PARTICLE SIZE DISTRIBUTION VIA BAHCO VS. COULTER COUNTER

OF SECONDARY CYCLONE CAPTURED MATERIAL (RUN #78)

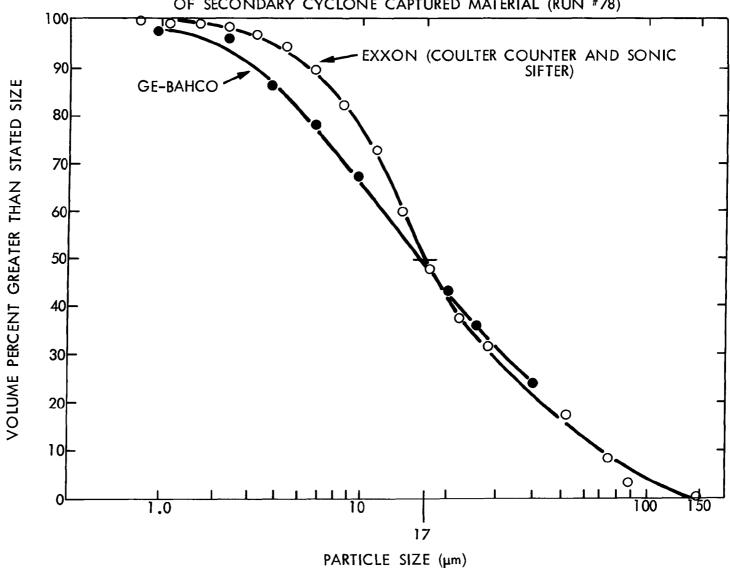
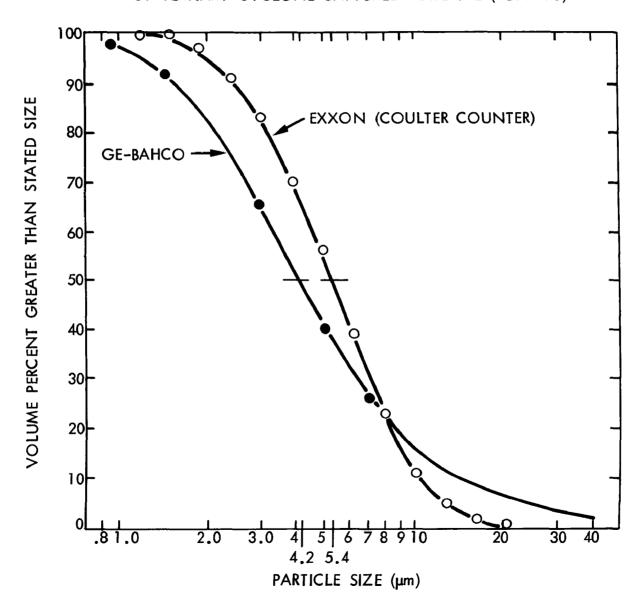


FIGURE IV-2

COMPARISON OF PARTICLE SIZE DISTRIBUTION VIA BAHCO VS. COULTER COUNTER OF TERTIARY CYCLONE CAPTURED MATERIAL (RUN #78)



are remarkably similar for two such different techniques. The second cyclone material was preclassified with a sonic sifter for the size distribution above 45  $\mu m$  and combined with the Coulter Counter size distribution to give the Exxon result. Balston filter captured particulates cannot be analyzed with the Bahco, because there is an insufficient quantity of material available for this instrument.

## High Temperature High Pressure Particulate Sampling

The question as to the difference between the size distribution measured by the Coulter Counter and the true size of particulates in the flue gas before sampling was also addressed. It was answered as part of a joint program with Southern Research Institute (SoRI) and Air Pollution Technology, Inc. (APT), both EPA contractors. The joint program was intended to resolve the question of the validity of the filter/Coulter Counter method and thereby confirm cyclone efficiencies based on this method.

The minimum particle size detectable by the Coulter Counter in this application was estimated to be approximately 0.6  $\mu m$ . Thus, the possibility existed that the fraction efficiency results might have been biased by using the mass balance technique with incomplete size distributions. Further, the ultrasonic deagglomeration of particles prior to performing the Coulter analyses may have resulted in aerodynamically large agglomerates being measured by the Coulter Counter as individual primary particles. The magnitude of the possible errors in the fractional efficiency curves resulting from these biases are difficult to assess.

Cascade impactors have been used for a number of years for determinations of control device fractional efficiencies over the size range from approximately 0.3  $\mu m$  to 10  $\mu m$ . Recent work at APT (9), has shown that cascade impactor performance at temperatures and pressures like those in the miniplant flue gas lines can be predicted to good accuracy by current theories. A pair of cascade impactors which were designed by APT, to operate at high pressures and temperatures was made available by the EPA for a series of independent tests of the miniplant tertiary cyclone outlet size distribution and fractional collection efficiency.

Inconel shim substrates were used for particle collection surfaces for each stage. Ceramic fiber backup filters were used to collect those particles which were not removed by the impaction stages. Qualitative verification of the performance of the impactors was obtained by Coulter Counter analyses (where applicable) and by electron microscopy of the various stage catches from typical runs. Previous experience by Exxon and the samples obtained during this joint program by Southern Research and APT showed that the particles, at the sampling conditions, were highly adhesive. This permitted valid impactor results to be obtained even though bare metal substrates were used. (Ceramic fiber substrates were on hand for use had this not been the case.)

Figure IV-3 shows comparative results of size distributions of the particles in the tertiary cyclone exhaust stream obtained by Coulter analyses of filter catches from miniplant run 105 by Exxon and those obtained by means of cascade impactors during the same run by SoRI and APT.

As seen, the cascade impactor results indicate a larger concentration of fine particles and a mass median particle size of about 0.8 micron, where the Coulter Counter mass median particle size averaged 1.6 microns.

Electron micrographs of material captured on the various impactor stages indicated that, with the exception of the first stages, the particulate matter collected in the impactor stages was fine, non-agglomerated particulates. Electron microscopy also revealed that much of the aerodynamically large particulate matter on the first impactor stage at the cyclone inlet was agglomerates of smaller particles.

These findings indicated that the results of the Balston filter/Coulter Counter method used by Exxon differed somewhat from cascade impactor results but to the degree expected, based on measurements made in other particulate systems. They also indicated that the Coulter Counter results were not being biased toward the finer particles by breakdown of agglomerates in the aqueous dispersing medium used in the Coulter Counter, since the Coulter Counter gave a coarser distribution than the impactor.

Prior to the joint impactor sampling program with SoRI and APT, samples were taken by Exxon with a University of Washington (UoW) Mark III cascade impactor. The 7-stage impactor was used to obtain background experience with impactors at high temperature and high pressure. The impactor was used with the HTHP particulate sampling system. It was heated to 750°C and pressurized to 900 kPa. Several runs were made using the impactor which was designed for the dilute sampling conditions. These runs showed that the particulate was very adhesive and would stick to bare metal substrates. Also it was shown that the pre-cut cyclone used ahead of the impactor to prevent overloading of the first stages was not needed. It captured very little material and after most runs the first two stages of the impactor were empty.

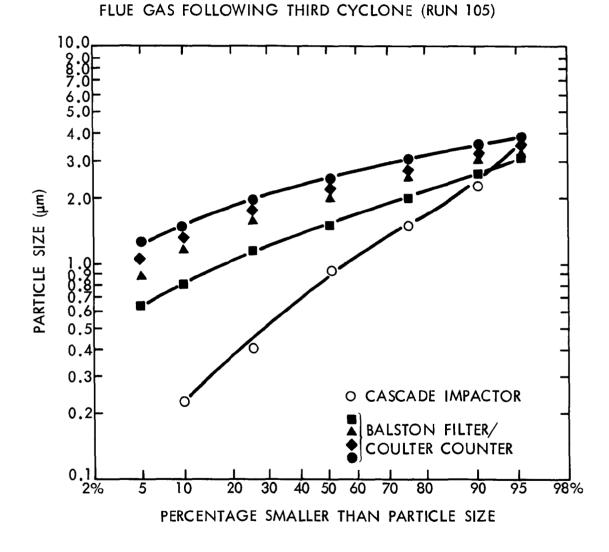
Particle size distributions obtained with the UoW impactor were indeterminate. The impactor was erroneously run at very high flow rates. This tended to increase jet velocities to the point where much of the material that should have remained on a given stage may have been "washed" off onto a following stage. This may be the reason that the Balston backup filter captured a substantial portion of the particulate. The stage cut diameters were calculated with classical formulas, and the size distribution obtained. The size distribution was much finer than that of a Balston filter (with Coulter Counter size analysis) taken at the same time. This is further evidence that the flow rates were much too high.

Mini Cyclone Train Sampling--

The SorI 5-cyclone train was used to try to obtain gram size quantities of particulates which pass through the tertiary cyclone. This sampling device was also intended to give an in-situ distribution of the particulates once it

FIGURE IV-3

BALSTON FILTER/COULTER COUNTER VS. APT IMPACTOR PARTICLE SIZE DISTRIBUTION



was calibrated. The cyclones were used with the HTHP sampling system. They were heated to between 720 and 870°C and pressurized to 900 kPa. The problems associated with the 5-cyclone train were mostly due to the very dilute nature and small size of the particulates in the flue gas. Long sample times were required and small amounts of particles had to be removed from the relatively large catch cups. The system was very prone to leaks and contamination by oxidized metal and the anti-seizing compound used on the nuts and bolts that hold it together. The total loading of particulates measured was typically one-half the loadings obtained with concurrent Balston filter sampling. Also. the size differentiation of the cyclone train was much too broad at the sampling conditions to describe the particle size distribution. The cyclone capture efficiencies probably due to re-entrainment of the dust from the catchpots on each cyclone. Therefore, with the cyclone stage cut diameters a function of sampling duration, calibration was not possible. For these reasons the SoRI 5-cyclone train was not deemed suitable for use in the downstream location to measure particulate loadings or size distributions. Modifications may have improved its performance under these conditions, but this was out of the scope of this program.

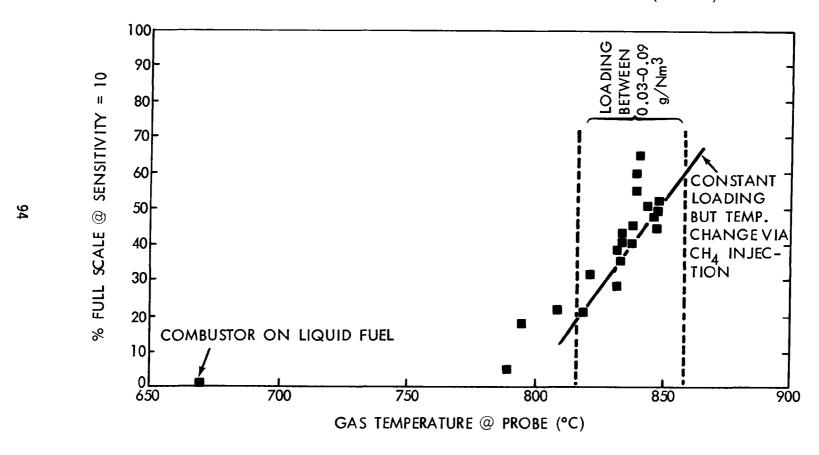
## Continuous Particulate Monitor

A continuous, on-line particulate monitoring system was evaluated during run 80. The monitoring system (manufactured by IKOR, Inc., Gloucester, Massachusetts) makes use of the surface charge that particulates accumulate during their flow through the combustion unit.

The IKOR probe measures the total electrical charge of impacting solids. The probe consisted of a 0.63 cm diameter solid rod of type 316 stainless steel that was 35.5 cm long. The last 8.9 cm of the probe was immersed in the 10 cm diameter exhaust duct of the miniplant at a location 2.1 meters downstream of the GE turbine test cascade. The probe was mounted on the duct's center line and normal to the gas flow direction. A constant purge of dry  $N_2$  was maintained around the probe to prevent condensation on the probe insulator.

The output of the probe was correlated with gas temperature measured near the probe location and with average particulate loadings measured by Balston filter samples. The data plotted in Figure IV-4 compares the IKOR signal strength with gas temperature during run 80 at relatively constant dust loading. The particulate loading data obtained using the Balston filter during run 80 are summarized in Appendix M-6. The particulate data show that the average mass mean diameter of the particles captured by the Balston technique following the third cyclone was 1.2  $\mu m$  and the average loading was 0.051 g/Nm³. The loading varied between 0.034 and 0.067 g/Nm³ during the IKOR testing period. The samples contained 6% of particles larger than 5  $\mu m$ .

The data in Figure IV-4 show that when the particulate loading downstream of the third cyclone averaged 0.051 g/Nm $^3$ , the IKOR probe signal strength varied almost linearly with gas temperature. Natural gas was injected into the combustor exhaust stream upstream of the third cyclone. The gas temperature at the probe location was varied by adjusting the amount of natural gas flow.



The unexpected sensitivity of the IKOR signal to probe temperature made comparison of signal amplitude with gravimetric sampling very difficult. Because of this and the unknown effect of thermionic emission from the probe or pressure vessel walls, further testing was stopped. IKOR agreed to investigate the problems and possibly develop a temperature compensator for the unit.

#### CYCLONE STUDIES

The miniplant was initially designed with only two stages of cyclone cleanup. The gas was sampled and expanded after the secondary cyclone. The particulate loading of the gas was much too high to pass through any gas turbine. A granular bed filter was then added to the flue gas stream. The problems of this device are contained in this and a previous report (1). A conventional cyclone was installed upstream of the granular filter to lower the dust loading and possibly improve efficiency. This cyclone performed very well and lead to a halt in granular bed filter testing. All subsequent hot gas cleanup was accomplished with three stages of cyclones. However, further tests of hot gas cleanup systems were conducted on a slipstream of flue gas withdrawn after the second stage cyclone. Low temperature systems were also tested. These tests are discussed in subsequent sections.

## Cyclone Efficiency Testing

Particulates in the flue gas leaving the miniplant recycle cyclone pass through the two additional cyclones which remove  $\sim 99\%$  of the particulate matter. Mass balance calculations based on particulate material captured in the second and third cyclones and in the flue gas leaving the third cyclone are used to determine the particulate size and concentration at the exit of all three miniplant cyclones.

These measurements and calculations show that material captured by the second cyclone has a mass median particle size of 20 to 25  $\mu m$ . The overall cyclone efficiency is about 95%. Material captured by the third cyclone has a mass median size of 3 to 5  $\mu m$ . The overall efficiency of this cyclone is about 90%. Particulate concentration in the flue gas leaving the third cyclone is generally 0.03 to 0.15 g/Nm³. The mass median size of the particulate ranges from 1 to 3 microns as determined by Coulter Counter. These results are summarized in Table IV-1. Detailed particulate loading and size data for each run are shown in Appendices M-3 through M-6.

TABLE IV-1. PARTICULATE CONCENTRATION AND SIZE MINIPLANT CYCLONE SYSTEM

	Part. Conc. (g/Nm <sup>3</sup> )	Mass Med (Mici	Cyclone		
Cyclone	Passing	Captured	<u>Passing</u>	Efficiency (%)	
Recycle	8-12		20-25	***	
Second	0.4-1.2	20-25	3-5	90-95	
Third	0.03-0.15	3-5	1-3	85-94	

The particulate emissions after the tertiary cyclone have been as low as 0.03 g/Nm³. This is slightly below the New Source Performance Standard (0.03 lbs/MBTU) which corresponds to  $\sim 0.035$  g/Nm³. The performance was not consistent however, and usually exceeded the emission standard. This performance is not good enough to use cyclones alone to meet the emission standards.

The third cyclone fractional efficiency is of greatest interest, since if a three stage cyclone system is used in a commercial PFBC system, the third stage must be very efficient to prevent damage to the gas turbine behind it. The collection efficiency of the tertiary cyclone has been measured over some 20 runs with run durations between 8 and 250 hours. Throughout this period the total collection efficiency averaged 90% with a standard deviation of only  $\pm$  3%. This summary is shown in Table IV-2. As can be seen from the table, these measurements were made with 3 different coals and 2 different sorbents with a variety of Ca/S ratios.

Both the total efficiency and the fractional (grade) collection efficiency was much higher than expected. This cyclone, which was designed with classical handbook formulas, averaged a cut diameter (50% efficiency), over this period, of 0.88  $\mu m$   $\pm$  0.02. Table IV-3 shows the grade efficiencies measured during runs 68 through 100. These efficiencies were obtained from outlet samples and captured samples sized with a sonic sifter and a Coulter Counter. The inlet concentrations were calculated by size differentiated mass balance under the assumption that no size change of particles occurred through the cyclone. During runs 99 and 100 this assumption was checked with filter samples taken upstream of the cyclone. Although cyclone performance may not be sufficient to meet emission standards, cyclones performing as well as those used on the miniplant may be sufficient to protect the gas turbine from serious damage by erosion.

The main purpose of the particle size distribution study (described earlier) by the Southern Research Institute and Air Pollution Technology was to measure cyclone fractional collection efficiency. Figure IV-5 shows the fractional efficiency of the third cyclone during run 105 as calculated from: (1) the SoRI/APT cascade impactor data, (2) the Exxon total filter/mass balance technique with Coulter Counter size analysis, (3) the Leith and Licht (10) cyclone fractional efficiency model. The cyclone operating conditions are shown in Table IV-4. The impactor and the total filter/Coulter Counter efficiencies agree fairly closely except in the small particulate size range. The cyclone cut diameter (50% efficiency) in both cases was approximately 0.7 microns. Therefore, the cyclone efficiencies calculated from cascade impactor data substantially confirms the efficiency obtained from total filter/Coulter Counter data. However, the predicted fractional efficiency curve is significantly lower than the measured results. The Leith and Licht model, shown in Figure IV-5, comes closer to predicting miniplant third cyclone performance than other available models tested. The reason for the lack of agreement between the measured and predicted results is not understood at the present time. It may be due to the fact that many of the models are semi-empirical and based on data obtained with particulate generated by other systems at lower temperatures and pressures.

TABLE IV-2. TERTIARY CYCLONE TOTAL EFFICIENCY SUMMARY

Run #	Total Eff. %	Coal Type	Sorbent Type	Ca/S Ratio
67.1	88.4	Illinois	Dolomite	1.25
67.2	83.4	Illinois	Dolomite	1.25
67.3	88.7	Illinois	Dolomite	1.25
68	97.0	Illinois	Dolomite	2.00
71	82.9	Illinois	Dolomite	1.52
72	91.3	Ohio	Dolomite	1.79
73	94.3	Ohio	Dolomite	2.14
74.1	94.6	Ohio	Limestone	7.60
74.2	94.5	Ohio	Limestone	4.80
75	89.1	Ohio	Limestone	3.65
78.2	89.2	Illinois	Dolomite	1.45
78.4	90.0	Illinois	Dolomite	1.45
78.10	90.1	Illinois	Dolomite	1.45
79	93.9	Illinois	Dolomite	1.45
80	84.6	Illinois	Dolomite	1.41
81	91.2	Illinois	Dolomite	1.56
83	92.4	Illinois	Dolomite	1.45
84	95.9	Illinois	Dolomite	1.45
85	94.7	Illinois	Dolomite	1.45
8 <b>6</b>	92.0	Illinois	Dolomite	1.45
87	94.2	Illinois	Dolomite	1.45
88	92.6	Illinois/Ohio	Dolomite	1.45
89	88.6	Ohio/Champion	<u>Dolomite</u>	1.40/1.87
90	90.9	Champion	Dolomi te	1.87
91	91.1	Champion	Dolomite	1.50
92	88.6	Champion	Dolomite	1.50
93	89.8	Champion	Dolomite	1.50
94	89.9	Champion	Dolomite	1.50
95	90.4	Champion	Dolomite	1.50
96	91. <u>1</u>	Champion	Dolomite	1.50
97	90.7	Champion	Dolomite	1.40
<b>9</b> 8	86.9	Champion	Dolomite	1.40
99	89.6	Champion	Dolomite	1.40
100	88.4	Champion	Dolomite	0.38

Average Of 90.6 ± 3.3
All Runs
± 1s

TABLE IV-3. TERTIARY CYCLONE FRACTIONAL EFFICIENCY SUMMARY

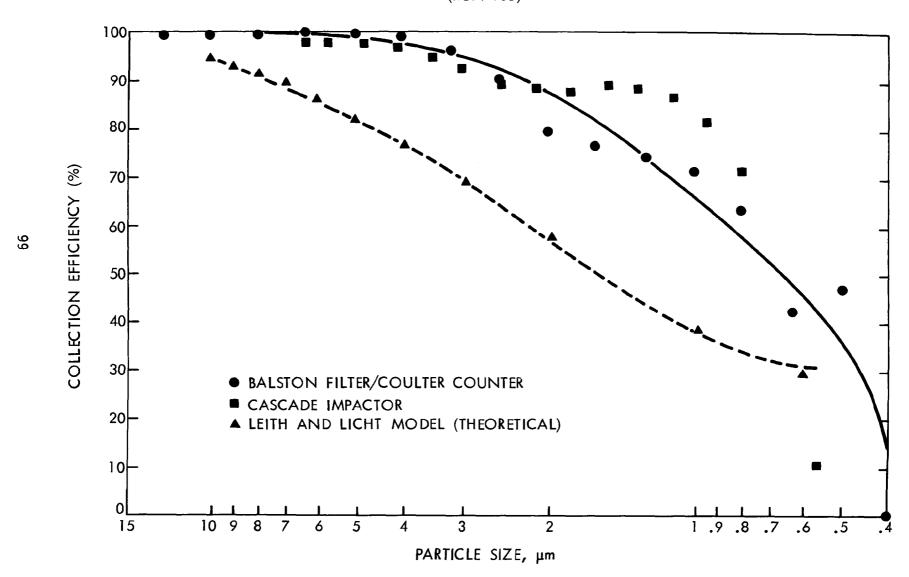
Particle Size Captured with Stated Efficiency (µm)

Run No.	50%	75%	85%	90%	95%
68	N/M	N/M	1.15	1.60	2.15
71	1.10	1.80	2.30	2.60	3.20
72.1	N/M	0.95	1.55	2.10	3.15
73.1	0.70	1.00	1.15	1.20	1.50
74.1	N/M	0.85	1.15	1.55	2.10
74.2	N/M	N/M	0.80	1.55	2.50
75	0.80	1.45	1.85	2.10	2.60
78.2	0.85	1.20	1.50	1.70	2.50
78.4	1.05	1.40	1.70	1.85	2.45
78.10	0.75	1.20	1.55	2.00	N/M
79	1.15	1.45	1.65	1.80	2.00
80	1.05	1.65	1.95	2.30	4.50
81	N/M	N/M	1.35	1.60	2.20
86	N/M	N/M	1.55	2.20	3.20
87	N/M	1.85	2.20	2.35	2.70
96	N/M	N/M	1.45	2.00	3.00
99.7	N/M	N/M	0.90	2.00	4.50
99.7*	N/M	1.00	2.50	4.00	6.35
100.3	0.70	1.25	1.60	2.00	2.30
100.3*	0.65	1.25	1.60	1.90	2.35
100.5	0.03	1.20	1.00	1.90	2.33
Average of	0.88 + 0.02	1.31 + 0.3	1.57 + 0.4	2.02 + 0.6	2.91 + 1.13
All Runs $\pm 1s$	<u> </u>	1.01 <u> </u>	1.3/ <u>·</u> 0.4	2.02 - 0.0	7.31 <u>1.13</u>

N/M = Beyond range of Coulter Counter.

<sup>\*</sup> Efficiency calculated based on upstream Balston total filter sample.

FIGURE IV-5
TERTIARY CYCLONE COLLECTION EFFICIENCY
(RUN 105)



# TABLE IV-4. THIRD CYCLONE OPERATING CONDITIONS (RUN 105)

Pressure 700 kPa
Temperature 635°C
Flow rate 14.6 Nm³/min
Inlet Velocity 36 m/sec
Pressure drop 4 kPa

Knowlton and Bachovin (11) found that pressures up to 5.6 M Pa had little effect on cyclone performance. Their work was based on much higher dust loadings than the current study. Perhaps other effects, peculiar to the PFBC system such as the nature of the particulates, or the operating temperature and pressure are responsible for the high efficiency. Additional work will be needed to explain the observed results.

The conclusions reached by this study were basically to confirm the high third cyclone efficiencies measured with the Balston filter/Coulter Counter technique. The Balston filter/Coulter Counter technique was not being biased toward finer size distributions by particle deagglomeration in the aqueous dispersing medium used in Coulter Counter measurements. In fact, the Coulter Counter gave a coarser size distribution than the impactor, which is as expected based on other measurements made in other particulate systems.

Further work is required to optimize cyclone performance under PFB conditions. The emissions of particulate with 3 stage cyclone cleanup have, at times, met the new source performance standard of 12.9 ng/J (0.03 lbs/MBTU), set by the EPA. However, the performance was not consistent enough to meet the standard over the long term. The improvement in performance required is small and may be met by cyclone optimization.

## Cyclone Optimization and Variables Study

A cyclone variables and optimization study was undertaken during the last 7 miniplant runs. During these runs (nos. 109 through 115) three different cyclones were exposed to various PFB operating conditions in the tertiary position. Each cyclone was located inside the vessel that once contained the granular bed filter elements. In this way, the plunger type valves that were used to provide blowback shutoff of the granular filter elements could be used to shut off the cyclones. All three cyclones could be tested, one at a time, during every run. Each cyclone was equipped with an orifice in the inlet line. In this way, valve leakage could be detected and flow to the cyclones verified. A  $\Delta P$  cell was used to measure the pressure drop between the pressure vessel inlet and outlet. This was the same location used in previous measurements with only one cyclone.

The dimensions of the cyclones are shown in Table IV-5 and Figure IV-6. The "A" cyclone was the original miniplant tertiary cyclone which had almost 2000 hours of combustor operating time at the start of the test program. The "B" and "C" cyclones were newly constructed, scaled down versions of cyclones currently being tested by American Electric Power and Stal-Laval in England at the National Coal Board facility in Leatherhead.

TABLE IV-5. TERTIARY CYCLONE DESIGN DIMENSIONS (cm)

Symbol	Docovintion	Cyclone	Cyclone	Cyclone
Syllbut	Description	A	<u>B</u>	<u>C</u>
	Inlet Type	Tangential	Scroll	Scroll
$D_{b}$	Barrel Diameter	15.2	17.8	17.8
W	Inlet Width	3.81	3.66	3.66
hi	Inlet Height	7.62	8.18	8.18
ho	Outlet Pipe Insertion	11.4	7.62	7.62
$h_{B}$	Barrel Height	40.6	22.9	22.9
hc	Cone Height	20.3	44.5	44.5
Do	Outlet Pipe Diameter	6.2	5.25	3.51
0/I	Outlet/Inlet Ratio	1.04	0.76	0.31
Dd	Diameter of Cone at Dust Pot.	None	1.94	1.94
Dp	Dust Pot Diameter	None	12.4	12.4
ħр	Dust Pot Height	None	25.4	25.4
Inlet V	Drop @ 30.5 m/sec elocity 840°C Pa (calculated)	6.6	11.0	28.6

FIGURE IV-6 BASIC CYCLONE DESIGN - D<sub>o</sub> (I.D.) ↑ ho hB D<sub>b</sub> (I.D.) h<sub>с</sub> – <sub>Dd</sub> – (I.D.) - D<sub>p</sub> 60°

The parameters that were changed in this study included: cyclone geometry, inlet velocity, inlet temperature, inlet particulate loading and combustor coal feed. Cyclone geometry was studied by using more than one cyclone at each condition. Inlet velocities were lowered during one test by lowering the combustor fluidization air by 26%. The lower combustor superficial velocity also lowered the cyclone inlet temperature. Cyclone inlet temperature was also varied by not injecting, or injecting very little natural gas. This in turn varied the inlet velocity. Inlet particulate loading was varied by deactivating the secondary cyclone for one series of tests. The effect of combustor coal feed on cyclone performance was determined by varying the combustor feed coal from the Champion coal used for most of the test to Illinois No. 6 coal. During this test the dolomite sorbent feed rate was approximately constant.

The cyclone performance tests were carried out in one day (6-10 hour) runs. The combustor was allowed to equilibrate for 1-1/2 to 2 hours before any testing was done. After steady state was attained, Balston total filter samples were taken simultaneously at the cyclone inlet and outlet. At the conclusion of the sampling a different cyclone was put on line. The next cyclone reached steady state during the turn around of the filter (one-half hour) and the sampling was started all over again. The cyclone particulate catch was also sampled before and after every gas particulate sample.

The total collection efficiencies are shown in Table IV-6. For the baseline case the total efficiency of the "A" cyclone was  $84\pm2\%$ . The B and C cyclones averaged  $83\pm4\%$  and  $72\pm6\%$  total collection efficiency respectively under baseline conditions. The standard deviations are such that most of the non-baseline data are within the data scatter of the baseline data. This obscured many of the effects intended to be studied.

The cyclone grade efficiencies were also calculated for each of the tests. These were obtained from the inlet and outlet filter sample concentration and size distributions. These grade efficiencies contained as much data scatter as the total efficiencies. For the baseline case, the cyclone cut diameters averaged 1.5, 1.1, and 1.7  $\mu m$  for the A, B and C cyclones respectively.

Further data concerning particle size distribution in the flue gas prior to, and after, the cyclones are presented in Appendices M-5 and M-6.

The pressure drop measured across all cyclone systems was fairly high (60-90 kPa). However, if the pressure drop due to the orifices and the rest of the flow distribution system are subtracted, the pressure drops of the cyclones were close to the calculated values. The pressure drop obtained for the "A", "B" and "C" cyclones was 6, 12 and 40 kPa respectively. These compare favorably with calculated pressure drops of 6, 12 and 29 kPa.

The conclusion reached from this study was that no significant effects of pressure, inlet velocity, temperature, and combustor coal feed were found. An increase in inlet particulate loading did increase overall efficiency significantly. The slight decrease in the cut diameter it caused was within the data scatter of the measurements. None of the cyclones worked as well as expected.

TABLE IV-6. SUMMARY OF CYCLONE TEST PROGRAM RESULTS

Run No.	Cyclone	Inlet Velocity (m/sec)	Inlet Temp. (°C)	Coal <sup>(2)</sup> Type	Inlet Loading (g/m³)	Inlet Loading (g/m³)	Total Collection Efficiency (%)	Co 1	ticle S lected d Effic (µ) 75%	with
					<u> </u>	331				
109	$A_{(1)}^{(1)}$	42	840	CH	1.87	0.268	85	NA	2.0	4.0
111	$A_{\Lambda}^{(1)}$	42	840	CH	1.61	0.143	85	NA	NA	NA
113	A(I)	42	840	CH	0.85	0.163	81	1.5	1.8	2.1
110	Α	39	760	CH	1.35	0.160	88	1.0	1.5	2.8
112	Α	25	700	CH	1.54	0.152	90	1.2	1.4	1.7
113	Α	42	840	IL	1.73	0.269	85	1.4	1.7	2.2
114	A(3)	42	840	CH	4.83	0.304	94	0.7	1.3	1.9
105	$A^{(3)}$	36	635	CH	1.62	0.176	89	1.2	1.6	2.1
109	B(1) B(1) B(1)	41	840	СН	1.23	0.255	79	1.0	1.6	2.9
iii	B(1)	41	840	CH	1.13	0.179	84	1.2	1.4	2.0
113	B(T)	41	840	CH	1.79	0.249	86	1.0	1.4	1.8
110	B	34	650	CH	1.36	0.240	82	1.4	1.8	2.4
110	В	38	760	СН	1.24	0.138	89	1.2	1.4	1.8
112	В	25	700	CH	1.25	0.302	76	1.3	1.6	2.2
114	В	41	840	СН	5.06	0.393	92	1.1	1.4	2.0
109	$C^{(1)}$	41	840	СН	1.19	0.387	68	2.1	2.8	3.8
111	G(1)	41	840	CH	0.91	0.191	79	1.3	1.8	2.7
115	$\tilde{c}^{(1)}$	41	840	CH	0.54	0.171	68	1.7	2.1	3.4
112	Č	25	700	CH	1.14	0.236	79	1.2	1.3	1.8

 <sup>(1)</sup> Baseline expected commercial/design operating conditions
 (2) CH = Champion Coal (Ca/S = 1.25)
 IL = Illinois No. 6 Coal (Ca/S = 0.76)
 (3) Pressure Reduced to 700 kPa, All others at 900 kPa

NA - Data Not Available

The "A" cyclone did not perform at the 90% collection efficiency measured over the previous 2000 hours. This was probably due to changes in geometry due to erosion, repair and thermal stress. The "B" and "C" cyclones also did not collect particulates with the expected efficiency. Ironically, this may be due to a lack of exposure. Any rough welds or edges had not had sufficient exposure to the erosive gas to smooth them to their steady operating conditions. As can be seen in Table IV-2 the total collection efficiency of the "A" cyclone was also lower and slightly more eratic during its first 100 hours of exposure (run 66-68) than later on.

Comparisons of "B" and "C" cyclone performance with the Van Tongeren Model 850 cyclones used by AEP and Stal-Laval in England are underway. The two cyclones used in England are geometrically similar to the "B" and "C" cyclones. The results from these tests will be followed to develop more reliable scale up procedures for cyclones in PFBC service.

#### CERAMIC FIBER FILTER EVALUATION

As mentioned previously, cyclones do not appear to be capable of consistently meeting the New Source Performance Standard for particulates. However, cyclones may be adequate to protect gas turbines, based on early results from extended materials tests. Two devices, with the potential for meeting the environmental emission standard as well as the turbine requirement, were tested on a small flue gas slipstream from the miniplant. The first of these was a ceramic fiber filter developed and supplied by Acurex Corporation under an EPA contract.

Previous development work on the ceramic fiber filter concept is reported elsewhere (28). The testing of the fiber filter on the miniplant will also be described in a separate report by Acurex.

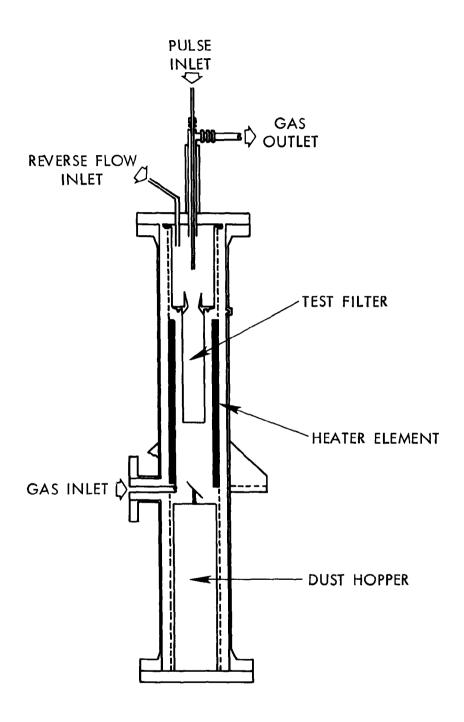
#### Test Description

The test device used in this study was provided by Acurex Corporation. It consisted of a single filter element contained in a heated pressure vessel with all necessary cycle controls. The filter consisted of a loosely packed mat of Saffil alumina generally about 1 cm thick sandwiched between an open weave fine gage 304 stainless steel support screen. The alumina mat, support screen sandwich was formed into a cylindrical filter element which was slipped over a heavy gage support cage and clamped at the top and bottom.

The objective of this study was to demonstrate the feasibility of ceramic filtration under actual PFBC conditions. Data were also obtained on the effects of cleaning cycle, face velocity, temperature and bag age on filtration efficiency.

A cross section of the filter housing is shown in Figure IV-7. Hot, dusty inlet gas enters the unit from the side, below the test filter. This gas impacts against a plate on the dust hopper. Heavy particles may remain in the hopper while others travel upwards to the filter element. The filter element was 10 cm in diameter by 45.7 cm long. A heater element surrounded

FIGURE IV-7
FILTER HOUSING PRESSURE VESSEL CROSS SECTION



the test filter and was used to maintain gas temperature in the test filter zone. After removal of particles by the test filter, hot gases exit the chamber through a pipe in the top of the vessel.

Figure IV-8 illustrates the installation of the test filter on a flue gas slipstream downstream of the miniplant second stage cyclone. The flue gas flow rate through the filter was 1.0 to 2.3  $\rm Nm^3/min$ .

The electronic time sequencer controlled the operation of the cleaning cycle. Cleaning cycle parameters were adjustable but the basic cleaning sequence was as follows:

- Start on a timed interval by closing a solenoid valve downstream, taking the filter off-line
- Start a gentle reverse flow of unheated gas
- Release one or more cleaning pulses, (amplitude, duration and pulse interval are all adjustable)
- Wait several seconds for dust removed during pulsing to fall into the dust hopper
- Stop reverse flow
- Open the downstream solenoid valve, returning the filter to service

Figure IV-9 is a photograph of the filter unit installed on the miniplant. The slipstream for the bag filter leaves the main flow duct through a l inch pipe. Two high-temperature valves, a l-inch Mohawk ceramic gate valve and a l-inch Kamyr ball valve, were used to isolate the filter from the PFBC. Just before the filter vessel, a gas bypass line allowed extra gas to be withdrawn from the PFBC to maintain temperature in the inlet line. This bypass line was also used to preheat the inlet line prior to the start of filtration.

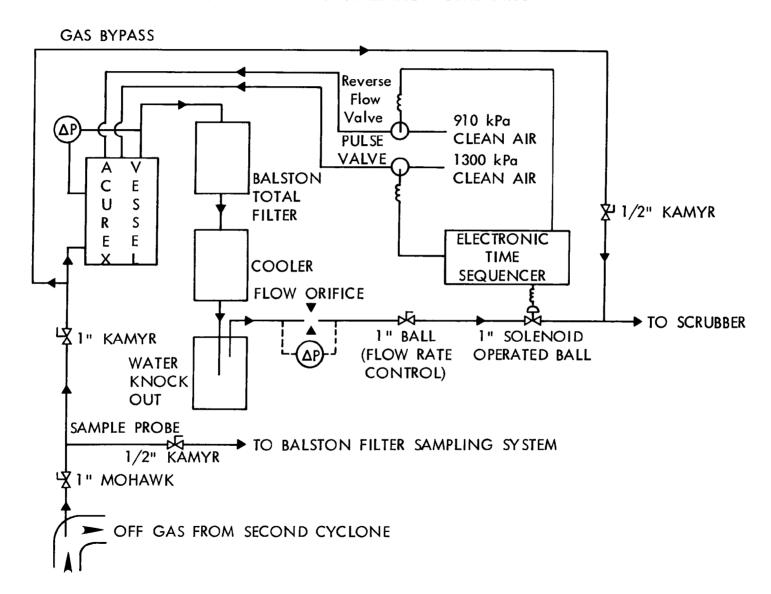
The filtered gas leaving the top of the filter pressure vessel cooled down to 440°C before it entered the Balston total filter shown in Figure IV-8. The weight gain of this filter was used to determine the outlet particulate concentration. The gas was further cooled and the water removed in a knockout vessel before it was measured through a flow orifice and expanded through a ball valve. Pressure drop across the ceramic filter was continuously measured and recorded. Inlet particulate concentration was measured by extracting a sample and passing it through a Balston total filter.

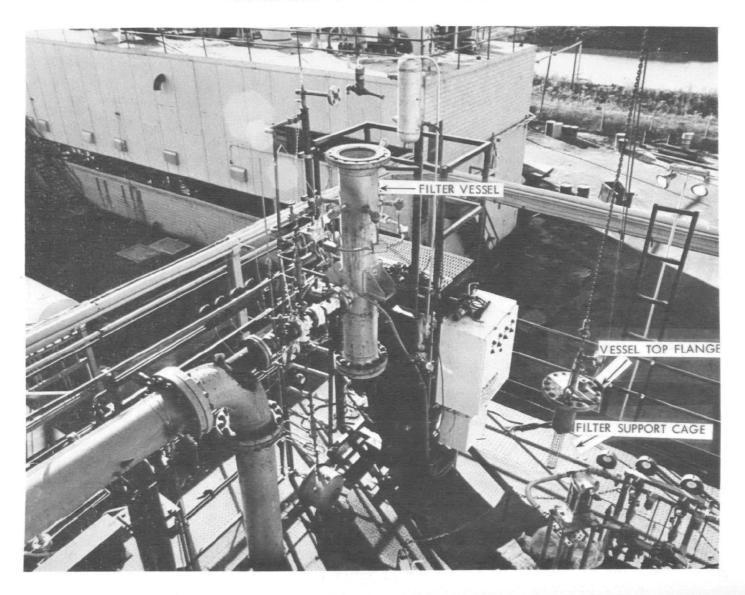
The filter was evaluated during runs 82 through 96. Runs 82 to 85 were devoted to system shakedown. Typically, a run lasted for one working day. At the end of that time the Acurex technician changed the test filter and a new test was begun the following day.

Several problems with valves occurred during the shakedown runs 82 through 85. The Kamyr valve failed during run 83. It was removed and not replaced. The Mohawk valve bonnet leaked during run 84. That gasket was replaced with a copper gasket and the valve performed satisfactorily until the alumina gate cracked during run 93. The solenoid valve that shuts off the filtered gas

FIGURE IV-8

ACUREX TEST FILTER INSTALLATION SCHEMATIC





flow during cleaning failed in run 85. The teflon seat of this valve had become damaged by hot gas. The valve was replaced with a solenoid operated ball valve which functioned well for the remainder of the test. Otherwise, shakedown ran smoothly and was mainly used to optimize the cleaning phase of the filtration cycle.

#### Test Results

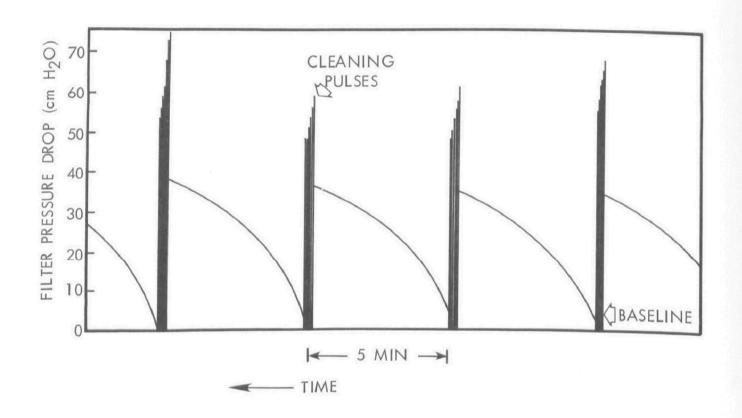
Pressure drop across the filter bags varied as a function of time in a manner typical of fabric filters. Figure IV-10 illustrates typical pressure drop and flow recordings. Baseline pressure drop was defined as the pressure drop of a bag at the start of the filtration cycle. Graphically, this point corresponds to the first point in each cycle in Figure IV-10. Many filters were operated for several hours at baseline pressure drops close to that of a new bag. The baseline pressure drop was always between 0.1 and 5.0 kPa, usually between 0.2 and 2.0 kPa. As shown in Table IV-7, the baseline pressure drop did increase with time in a number of runs, but this is typical of fabric filters during initial operation. Pressure drops before cleaning were never allowed to exceed 14 kPa to reduce the chance of bag failure. High pressure drops generally caused the inner filter support screen to bulge into the cage on which the filter was fastened. Baseline pressure drops were slightly higher when filtering Champion coal than when filtering Illinois No. 6 coal under similar conditions. Outlet particulate loadings were slightly lower with Champion coal than with Illinois No. 6 coal. The overall influence of coal type was small, and could not be quantified from the relatively few tests completed at the miniplant. Filter bags were used for from 4.5 to 19 hours. A summary of test conditions, pressure drops, and outlet particulate loadings is shown in Table IV-7.

Filtration efficiencies for the Acurex ceramic bag filter were generally over 90%, ranging from 96 to 99.5%. An exact filtration efficiency was difficult to determine because of problems in measuring the filter inlet particulate concentration. Filter inlet particulate concentration was measured or calculated by three methods: (1) Balston total filter catch on an extracted sample, (2) mass balance around the third miniplant cyclone, (3) mass balance around the ceramic bag filter. The results obtained by these three techniques were not consistent as shown in Table IV-8.

The test filter inlet line (Figure IV-11) was 1-inch schedule 80 pipe taking a sample from a 4-inch schedule 5 pipe. Isokinetic flow would have been 1.15 Nm³/min. This flow alone, would have cooled to 450°C before reaching the filter vessel. A bypass flow of 1.4-2.3 Nm³/min in addition to the filtered gas, was drawn through the line to help maintain temperature near 800°C. The flow into the filter inlet line was therefore 200 to 300 percent isokinetic. The Balston total filter inlet sample (1/4-inch probe) was incorrectly operated isokinetically with respect to the filtered gas only, neglecting the bypass gas which was present at that point. Therefore, the Balston total filter samples were taken at only 30 to 50% isokinetic rates. For this particle size range and loading, isokinetic flow appears to be important, and this inlet loading, measured with the Balston filter, may be treated as a lower limit of particle concentration.

FIGURE IV-10

ACUREX HTHP CERAMIC BAG FILTER PRESSURE DROP AND FLOW



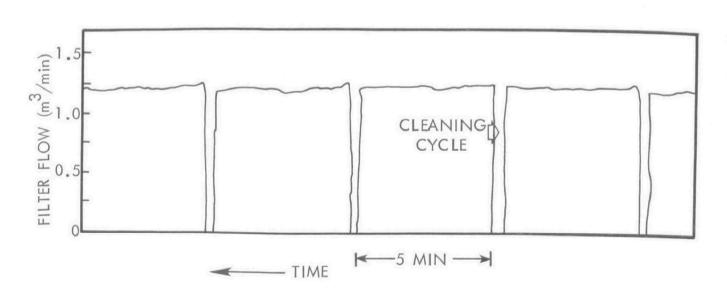


TABLE IV-7. ACUREX HTHP BAG FILTRATION SUMMARY

			Outlet	Flow	Face	Average		(kPa)	Filtration	Run
	0 - 41	Coal	Load	Rate	Velocity	Temperature	Baseline	Max Before	Cycle(6)	Ouration
Run No.	Bay No.	Type(4)	$(a/u_3)$	m3/min	m/min	(•c)	<u>min</u> max	Cleaning	Time (min)	hrs
83	i	1	NΛ	1.42	4.0	NA	0 0.2	3.7	NΛ	4
B <b>4</b>	1	I	NA	1.36	4.1	891	1.2 2.5	8.0	NA	3
85	2,,,	1	0.0684	1.36	3.9	847	0 2.0	4.0	10	3.5
86	$\frac{5}{2}(1)$	ĭ	0.0093	1.33	3.2	659	0 0.5	1.7	5	4.5
87.1	3	Ţ	0.0146	1.70	4.0	635	0 0.2	1.0	5	3
87.2	3	1	0.0087	1.73	4.4	701	0 0.2	1.0	7	2.3
87.3	3	٧	0.0027	1.81	4.5	679	0.2 0.2	1.7	15	3
88.1	3 <b>A</b>	٧	0.0205	2.01	5.4	762	0.4 0.5	2,5	20	2
88.2	3A, 2	C	0.0137	2.27	6.0	752	0.5 3.0	6.5	20	2
88.3	3A(2)	С	0.0114	1.70	4.9	832	1.2 2.0	7.5	20	1
89.1	4	С	0.0226	1.84	5.1	801	0.1 0.1	5.0	20	1
89.2	4	C	0.0226	1.84	5.2	812	0.5 0.5	6.7	20	1
89.3	4	С	0.0226	1.42	3.9	791	0.5 2.0	7.7	20	1
89.4	4	C	0.0226	1.42	4.0	812	1.9 2.5	7.5	10	3
90	5	C	0.0089	1.42	3.9	790	0.1 2.0	7.5	5	5
91.1	5	С	0.0066	1.42	3.8	<b>7</b> 50	1.9 2.5	8.7	4	2.5
91.2	5	С	0.0034	1.42	3.8	<b>7</b> 57	2.5 3.7	9.5	4	3
91.3	5	С	0.0036	1.42	3.8	<b>7</b> 58	3.7 5.0	<b>10.</b> 5	4	2.5
92.1	6	С	0.0059	1.13	3.1	<b>7</b> 62	0.2 0.5	5.2	10	3.3
92.2	6	C	0.0050	1.08	3.0	805	0.5 0.5	7.7	7	2.6
93.1	6	С	0.0071	0.99	3.1	787	0.2 0.5	3.5	5	3
93.2	6, ,	С	0.0046	0.99	2.8	828	0.5 1.5	5.0	4	5
94	7(3)	С	0.0661	2.05	5.8	823	0.7 1.7	3.5	4	6
95	8	C	0.0116	1.84	5.1	797	0.5 5.0	11.2(5)	4	4.5
96.1	9	C	0.0157	1.10	2.8	693	0 0.5	6.5	10	3
96.2	9	С	0.0066	1.10	2.7	665	0.5 1.0	10.0	10	2
96.3	9	С	0.0066	1.10	2.7	665	1.0 2.5	8.7	5	1
96.4	9	С	0.0155	1.10	2.9	754	1.9 2.5	13.7(5)	5	3
96.5	9	С	0.0296	1.10	2.9	727	0 0.5	3.7	5	7
96 <b>.6</b>	9	С	0.1870	1.10	2.6	648	0 0	0.5	5	2
96.7	9	С	0.2440	1.10	2.6	648	0 0	0.7	5	1

<sup>(1)</sup> Bag vacuumed prior to run.

(5) Chart recorder limit exceeded.

(6) Time for filtration between cleaning cycles. Cleaning cycle consisted of 6 pulses of about 0.75 s. each of 1300 kPa air with a 3 s interval between pulses. System pressure was 900 kPa.

<sup>(2)</sup> Pictured in Figures.

<sup>(3)</sup> Double thickness ceramic filter material.

<sup>(4)</sup> Coal type "I" is Illinois No. 6
"V" is Valley Camp (Ohio)
"C" is Champion (Pennsylvania)

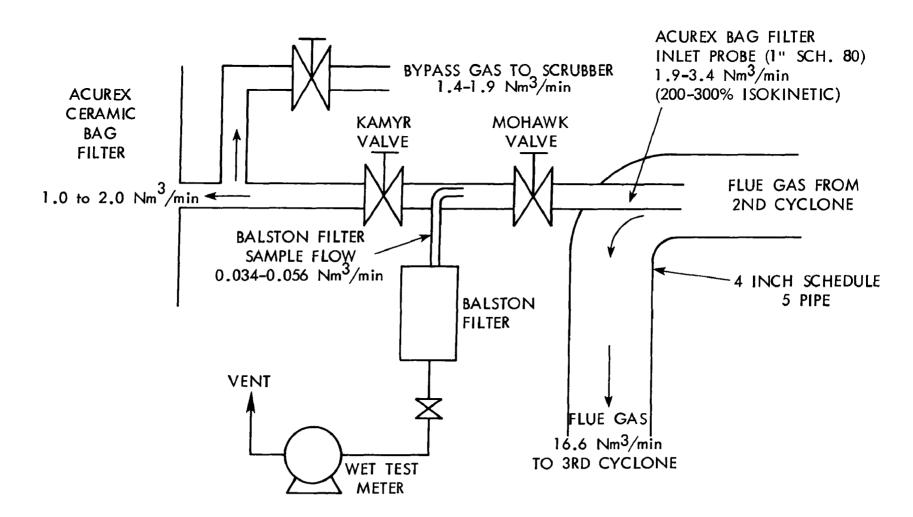
TABLE IV-8. ACUREX HTHP BAG FILTER INLET PARTICULATE LOADINGS

			2
Inle	et Particulate	Loading	(g/m³)
Commile by	Calculat	od from	Calo

	Inlet Particulate Loading (g/m²)					
	Sample by	Calculated from	Calculated from			
	Balston Total	Mass Balance	Mass Balance			
Run No.	Filter at Inlet	Around 3rd Cyclone	Around Filter			
86	0.40	0.79	N/A			
87.1	0.31	1.09	N/A			
87.2	0.30	1.09	N/A			
87.3	0.32	1.09	N/A			
88.1	0.30	0.77	N/A			
88.2	0.60	0.77	N/A			
88.3	0.60	0.77	N/A			
89.1	0.48	1.08	N/A			
89.2	0.48	1.08	N/A			
89.3	0.37	1.08	N/A			
89.4	0.37	1.08	N/A			
90	0.47	1.35	0.48			
91.1	0.40	1.06	0 <b>.4</b> 8			
91.2	0.40	1.06	0 <b>.4</b> 8			
91.3	0.57	1.06	0.48			
92.1	N/A	0.92	0.84			
92.2		0.92	0.84			
93.1	N/A 1.22	1.16	0.84			
93.2	1.22	1.16	0.84			
94	0.53	1.16	0.73			
95	N/A	0.96	0.73			
96.1	N/A	1.06	0.73			
96.2	N/A	0.94	0.73			
96.3	N/A	0.94	0.73			
96.4	N/A	1.65	0.73			
96.5	N/A	1.65	0.73			
96.6	N/A	1.65	0.73			
96.7	N/A	1.65	0.73			

\_\_\_\_\_

N/A = Not Available



A mass balance around the filter test vessel was attempted to resolve the inlet loading issue. Weight of the filter bags was not determined before exposure, so a tare of 0.2 kg was assumed by weighing other unexposed bags. These inlet loadings, intermediate to the other two results, can still be considered low because of the multitude of places where particulates could have been lost during cleaning and dismantling operations. However, from these three calculation methods a reasonable estimate can be made of the actual inlet particulate concentration.

Size distribution of the inlet particulate matter was not obtained since the samples were not taken under isokinetic conditions.

The bag filter outlet particulate concentration was determined by passing the entire filtered gas flow through a Balston total filter. The total particulate concentration was obtained by weighing the total filter before and after exposure. Overall ceramic bag filter efficiencies are shown in Table IV-9. These were calculated using the three methods of determining inlet concentration discussed previously. Despite some uncertainty in the inlet particulate concentrations, the collection efficiencies calculated by the three methods were generally in good agreement.

A size distribution of the outlet particulates could not be obtained. The amount of particulates on the Balston filter was so low that insufficient material was available for Coulter Counter analysis. The filters were washed off with Isoton II in an attempt to remove particulates without mechanical brushing. This method caused enough Balston filter material to be washed into solution to obscure completely the flyash particulates. A clean Balston filter, not exposed to any flyash but also washed with Isoton gave a sample which had a size distribution similar to that obtained from a used filter.

During the tests at the miniplant, eight single and one double thickness bags were exposed to PFBC conditions, as shown in Table IV-7. Most bags were exposed for 6 hours or more. Averaging the face velocity and exit particulate concentration over the first 6 hours of new bag exposure and plotting outlet loading as a function of face velocity provided the data shown in Figure IV-12. Bag number 4 results were not recorded on Figure IV-12 because of problems with the outlet filter. Bag number 7 was a double thickness bag (~2 cm). Normallv filter media thickness was about 1 centimeter. Bag 7 was physically less distorted and its pressure drop was less than bag 8 (a 1-cm thick bag which was run at similar conditions), and its filtration efficiency was much lower. The reason for the lower  $\Delta P$  and lower efficiency for the double-thickness bag is not certain (it probably had a leak). As seen in Figure IV-12, the particulate penetration of a bag seems to increase with face velocity. The trand lines shown on the curve were selected by drawing a line through the Illinois No. 6 results, which fell on a straight line, and then drawing a suitable parallel line for the Champion coal. However, the data may not be as precise as implied by this curve. For example, one objective of the off-line cleaning used in this study is to offset the increased penetration with velocity that is normally seen in filter tests and suggested by Figure IV-12. More testing is still required to determine better the effect of face velocity and off-line cleaning on penetration.

TABLE IV-9. ACUREX HTHP BAG FILTER COLLECTION EFFICIENCY

	Collection Efficiency (%) Based							
	Outlet							
	Particulate	Sampled by	Calculated from	Calculated from				
	Loading	Balston Total	Mass Balance	Mass Balance				
Run No.	(g/m3)	Filter at Inlet	Around 3rd Cyclone	Around Filter	Average			
86	0.0093	97.6	<b>98.</b> 8	N/A	98.2			
87.1	0.003	95.2	98.7	N/A	97.0			
87.2	0.009	97.1	99.1	N/A	98.2			
87.3	0.003	99.1	99.8	N/A	99.5			
88.1	0.021	93.0	97.3	N/A	95.2			
88.2	0.014	97.7	98.2	N/A	98.0			
88.3	0.011	98.1	98.5	N/A	98.3			
89.1	0.023	95.3	97.9	N/A	96.6			
89.2	0.023	95.3	97.9	N/A	96.6			
89.3	0.023	93.9	97.9	N/A	95.9			
89.4	0.023	93.9	97.9	N/A	95.9			
90	0.009	98.1	99.3	98.1	98.5			
91.1	0.003	98.3	99.4	98.6	98.8			
91.2	0.003	99.1	99.7	99.3	99.4			
91.3	0.003	99.4	99.7	99.2	99.4			
92.1	0.004	<b>33.4</b>	99.4	99.3	99.3			
92.1	0.005	<del></del>	99.5	99.4	99.4			
93.1		99.4	99.4	99.2	99.3			
93.1	0.007 0.005	99.6	99.6	99.5	99.6			
93.2		87 <b>.</b> 6	94.3	90.9	90.9			
9 <del>4</del> 95	0.066	N/A	98.8	98.4	98.6			
	0.012		98.6	97.8	98.2			
96.1	0.016	N/A	99.4	99.1	99.2			
96.2	0.007	N/A		99.1	99.2			
96.3	0.007	N/A	99.4	97.9				
96.4	0.016	N/A	98.6		98.3 96.7			
96.5	0.030	N/A	97.4	95.9				
96.6(1)	0.187	N/A	83.5	74.4	78.5			
96.7(1)	0.244	N/A	78.4	66.6	72.5			

(1) Bag Failed N/A = Not Available

ACUREX HTHP BAG FILTER OUTLET LOADING VS. FACE VELOCITY (AVERAGED OVER FIRST 6 HOURS OF EXPOSURE)

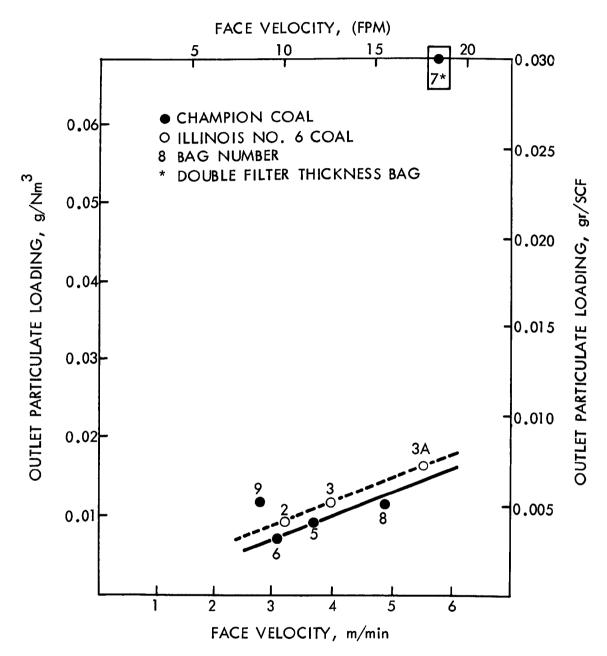
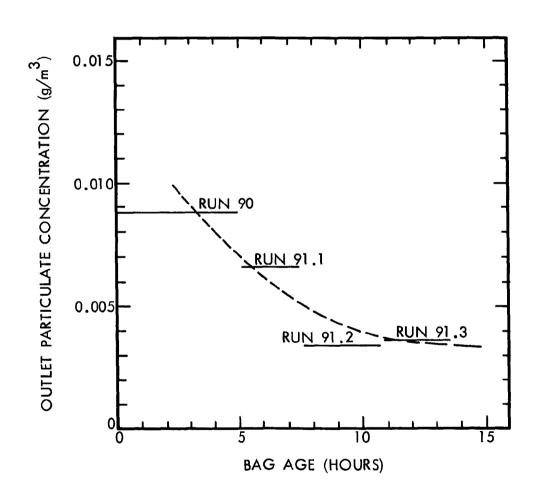


FIGURE IV-13

ACUREX CERAMIC BAG FILTER--BAG NO. 5
PARTICULATE PENETRATION HISTORY



Outlet loading plotted in Figure IV-12 was averaged over the first 6 hours of operation. Outlet loading actually decreased as a function of time in the same fashion that a conventional filter media test would show in similar tests under ambient conditions. This decrease as a function of time is shown as Figure IV-13. This bag was exposed to Champion coal at 775°C for a total of 13 hours. Along with the decrease in filter particle outlet loading there was an increase in baseline pressure drop from 0.1 to 3.0 kPa as expected as the filter cake formed and the efficiency improved.

The effect of bag cleaning conditions was not studied. After adjusting cleaning conditions during the first two runs, conditions were fixed for the rest of the runs at 6 pulses of 1300 kPa air for about 0.75 s each with a 3 s interval between pulses.

Examination of the filters after a test showed that the dust cake was deposited mostly on the surface of the filter media and the dust cake could be removed easily. Figure IV-14 shows bag number 3 immediately after removal from the filtration vessel. Figure IV-15 is a close up of the same bag after a strip was vacummed clean. This strip had the appearance of a virtually new bag indicating very little dust penetration into the media.

At the conclusion of the series of short runs, run 96, a long continuous test of the bag filter was attempted at conditions deemed optimum for extensive testing. Filtration commenced smoothly, however, the baseline pressure drop across the bag continued to increase during the first 6 hours of filtration. A possible reason for this increase became clear at the conclusion of the run when it was discovered that the pressure regulator used to set the pressure of the reverse flush air was set only slightly higher than filter vessel system pressure. It is possible that a slightly higher combustor pressure could have reduced reverse flush air flow to a level too small to clean the filter effectively. Since the reverse flush air flow rate was not measured, this hypothesis cannot be confirmed.

After 9 hours of filtration during run 96, a pressure drop of over 12.5 kPa across the filter was thought to be excessive for continued bag life. The vessel pressure was reduced to 300 kPa and a full high pressure (1300 kPa) pulse blow back was initiated into the lowered system pressure. This blow back reduced the pressure drop almost back to the clean baseline condition (0.5 kPa). However, outlet particulate concentration increased over the next 10 hours until it was almost identical to the inlet concentration. The run was terminated at that point. As expected, the bag had failed (see Figure IV-16). The bag failure probably began with the high pulse pressure blow back against the low system pressure, and was made worse by subsequent blow backs. The final cause of the failure appeared to be high temperature corrosion of the thin 304 stainless steel filter support screen. The blown out appearance was probably caused by the high pressure pulse into the lowered system pressure environment after the corrosion weakened support screen failed.

It is recognized that corrosion of a metal support screen is a potential problem in long term applications. The metal support screens used in these tests were used only for ease of construction. In addition, the early failure

FIGURE IV-14
CERAMIC FILTER BAG NO. 3



Vacuumed Strip

# FIGURE IV-15 CERAMIC FILTER BAG NO. 3 CLOSEUP OF VACUUMED STRIP

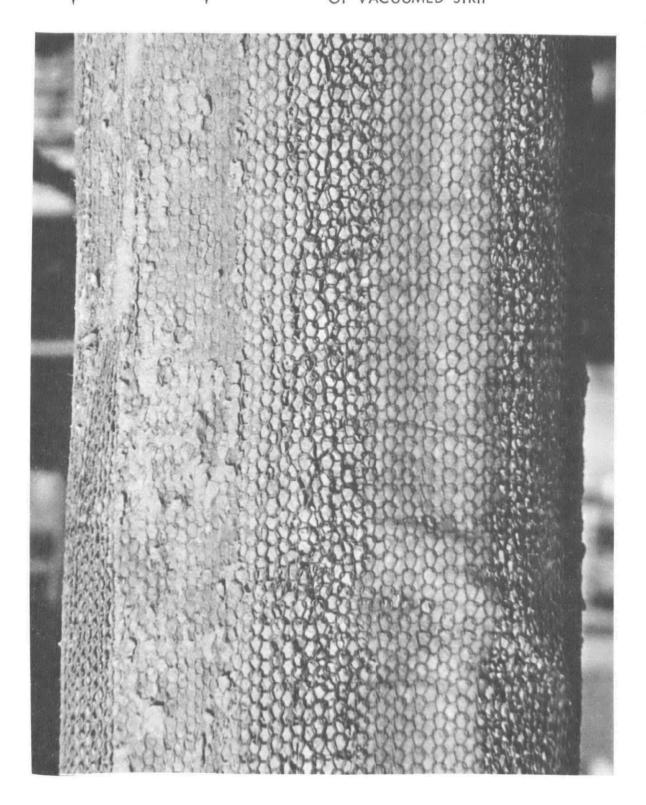
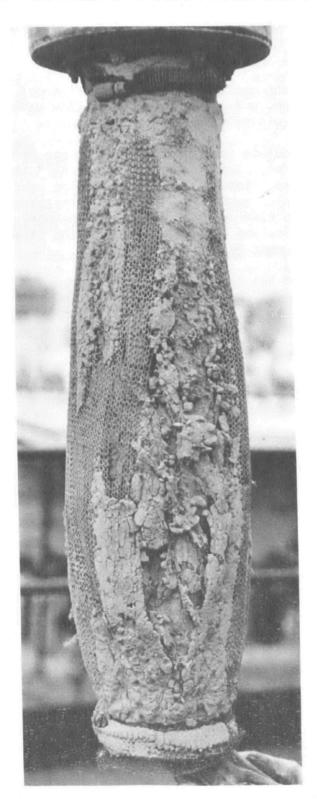


FIGURE IV-16

CERAMIC FILTER BAG NO. 9 AFTER RUN 96



of the support screen experienced in run 96 was probably caused by a malfunction in the temperature control system which caused the filter element to be overheated. Flexible woven ceramic fabric screens are available to perform this function for applications requiring greater corrosion resistance.

## Summary and Conclusions

During the miniplant tests, baseline pressure drops of under 2 kPa were maintained for over 6 hours average duration at face velocities of up to 6.0 m/min with removal efficiencies of 95 to 99%.

The average particulate concentration at the bag filter outlet was measured to be  $0.013 \pm 0.005$  g/Nm³. This is less than one-half the EPA New Source Emission Standard for particulates (0.03 lbs/MBTU). The particulate concentration was as low as one order of magnitude below the emission standard. In comparison to 3 stage cyclone cleanup, the average improvement was approximately a factor of 7. The particulate concentration with the ceramic filter easily meets both the EPA and most gas turbine standards.

These tests proved the ceramic filter was cleanable while subjected to flyash generated under PFBC conditions. High collection efficiency at high face velocity was also shown. In general, the test filter exhibited performance similar to that which would have been expected from a filter unit operating under more common conditions.

The failure of the filter element in the final test was a result of an unusual combination of events and is not an indication of an inherent filter problem. Further investigation of this potentially promising technique is felt to be warranted.

# GRANULAR BED FILTRATION STUDIES

Granular bed filters were also evaluated as high efficiency particulate control devices. Originally, the objectives of the test program were to measure particulate removal efficiency, operational stability and long term life of the filter hardware. However, operating difficulties, primarily caused by plugging of the filter inlet sections by the particulates, poor bed cleaning and loss of filter media during cleaning caused a change in the program direction. The program was then directed toward modifying the granular bed filter system to overcome the operating problems. The previous report describes the filter systems studied, the problems encountered and modifications made to solve the problems (1). These modifications allowed a 24 hour run to be completed. However, filtration efficiency decreased sharply during the test. It was found that the filter beds had not been adequately cleaned and gas was blowing through the filter beds in "rat holes." Additional modifications were subsequently made and the modified systems tested in an attempt to improve performance. These latter tests are described in this report.

#### Equipment

The filter configuration used at the start of the test program discussed in this report was described in detail in the previous report (1). This system

was installed on the miniplant following the second cyclone, and was capable of accepting the total flue gas flow. It consisted of two filter elements, each composed of five filter beds, as shown in Figure IV-17. A schematic of one of the filter beds is shown in Figure IV-18. The filter media contained in each bed consisted of granular alumina or quartz. This design had no screens across the dirty gas inlet opening at the top. This was done to prevent inlet screen plugging. The freeboard height was also increased to 18 cm to prevent loss of the filter media during the reverse (upflow) cleaning step with clean compressed air. In the cleaning step, the beds are fluidized by reverse-flow air, the fine particulates retained by the bed from the previous filtration step are entrained from the filter beds and blown out of the inlet opening at the top of the beds in a reverse direction. Figure IV-19 is a schematic which illustrates the operation of a filter element during the filtration and blow back (cleaning) steps. The fluidizing grids at the bottom of each bed were also redesigned to give better gas distribution during blow back. Each grid contained 56, 0.5 cm diameter holes.

Each filter element was installed in a shroud as shown in Figure IV-19. Flyash blown from an element during blow back is retained within the shroud, falls to the bottom and is collected in a lock hopper. Two filter elements were installed in parallel after the second stage cyclone in a single large refractory lined pressure vessel. The inside dimensions of the vessel are approximately 2.4 m diameter by 3.4 m inside height. The vessel is connected to the second stage cyclone by 12 inch pipe refractory lined and sleeved with stainless steel to an inside diameter of 10 cm.

Dirty gas entering the filter vessel is piped to each shroud, passing through orifices which measure flow rate to each filter element. Clean gas leaving each shroud fills the interior of the pressure vessel before leaving through a single outlet line. Blow back air enters each filter element through flanges at the top of the pressure vessel and flows in a reverse direction through each filter element. A blow back nozzle and seal plate are dropped down to engage the top of the filter element during blow back and direct the blow back gas in the proper direction. This is shown in Figure IV-19. The blow back gas leaving a filter element flows in a reverse direction through the clean gas inlet system into the other filter element which is in a filtration step. Each element is blown back separately.

A natural gas burner was installed to preheat the interior of the pressure vessel to a temperature above the dew point of the combustor flue gas before starting a filtration test. The burner fires into the vessel through a side port for an 8 to 12 hour period prior to the start of a run. The filter vessel is at atmospheric pressure during this period.

Natural gas injection into the flue gas piping between the second stage cyclone and the filter vessel was used to keep the flue gas temperature above  $840^{\circ}\text{C}$ . Gas was injected at four points, 2.3 m, 6.7 m, 9.8 m and 14.6 m downstream of the second stage cyclone. Approximately 0.03 Nm³/min of natural gas was injected through each of the four inlets.

All of the above systems are described in more detail in the previous report (1).

# FIGURE IV-17 MODIFIED GRANULAR BED FILTER ELEMENT (WITHOUT SHROUD)

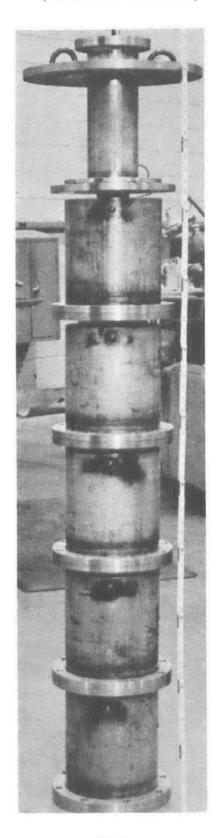


FIGURE IV-18

MODIFIED FILTER BED

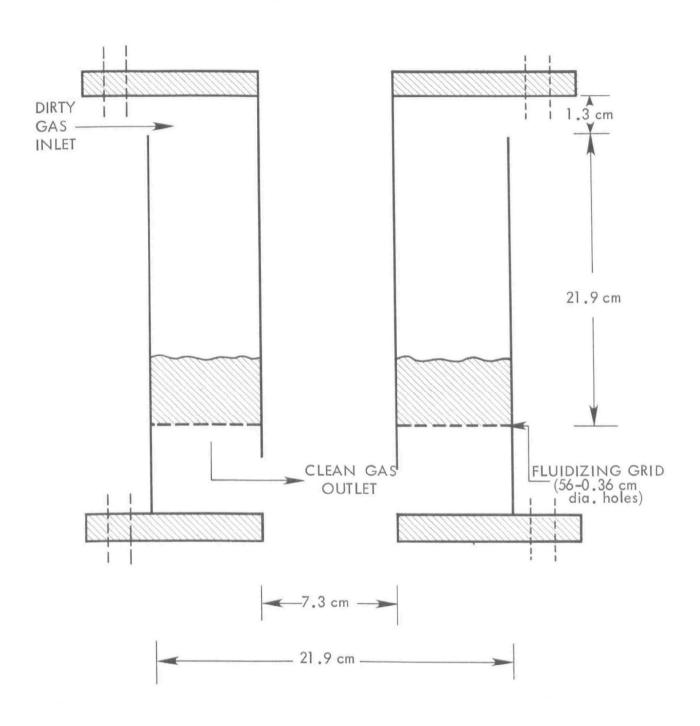
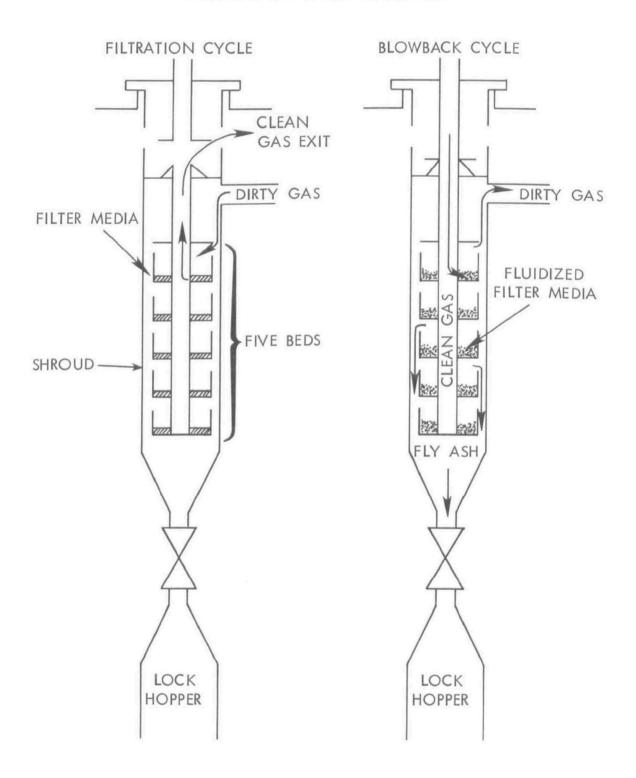


FIGURE IV-19

GRANULAR BED FILTER SCHEMATIC



System Modifications --

In order to improve the operation of the filter a few modifications were made to the filter system. The modifications were used at various times during the test program. None of them were permanent or needed for all tests. The section dealing with test results will specify which modifications were used for the various runs. This section will describe the modifications.

Blow back gas ejector—A blow back gas ejector was installed to replace the blow back gas inlet nozzle and seal plate assembly. It was described in the previous report (1). Briefly, the ejector used a small amount of motor air during blow back, but pulled in additional clean gas by the action of the ejector to supplement the blow back gas. There were no moving parts and no sealing surfaces. The ejector was tested during two runs and removed.

Inlet screens--In the above sections describing the design of the filter it was mentioned that the inlet screens were removed to prevent plugging and the bed freeboards increased to prevent loss of filter media during blow back. However, loss of filter media still occurred despite the increased bed freeboard and attempts to minimize blow back gas velocity. Therefore, for most runs, screens were strapped over the inlet slot. Three screen mesh sizes were used,  $10 \times 10$ ,  $20 \times 20$  and  $50 \times 50$ .

<u>Baffles</u>--Another modification was made to prevent loss of filter media, in this case, by the installation of baffles in the filter beds near the gas inlet. Figure IV-20 is a sketch of the baffle arrangement. The baffles were tested in one run.

Filter media--Initial tests were carried out using quartz granules with a particle size range of 250 to 600  $\mu m$ . In subsequent tests, 850 to 1400  $\mu m$  alumina particles were used. A very dense iron oxide (speculite) was also tested in two runs in an attempt to minimize filter media losses. The speculite particle size range was 400 to 2000  $\mu m$ .

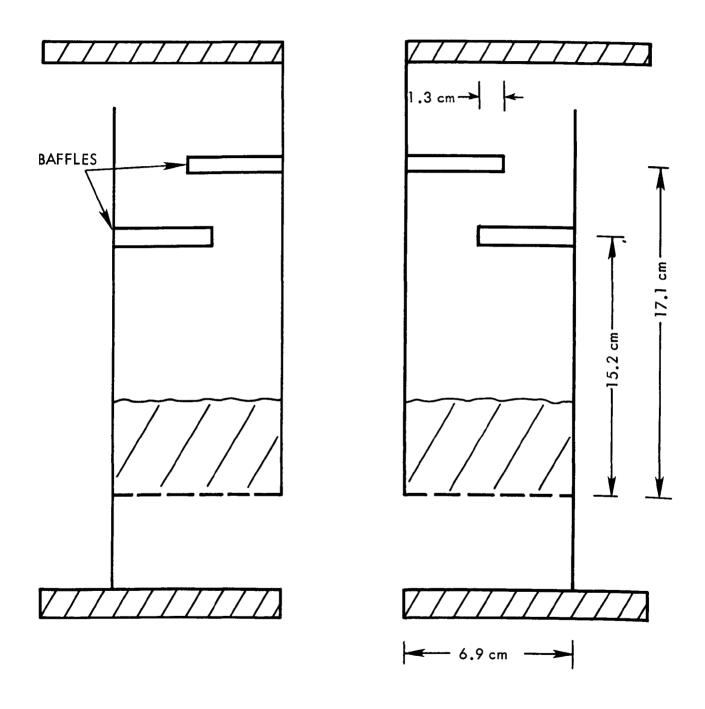
Outlet piping--Initially, the clean gas from the filter elements filled the interior of the pressure vessel and left through a single vessel outlet line. However, heat losses from the vessel were high and the temperature drop between the filter inlet and outlet lines was too great. The filter element outlets were then piped directly to the vessel outlet and the new piping and the filter shrouds were insulated to reduce heat loss.

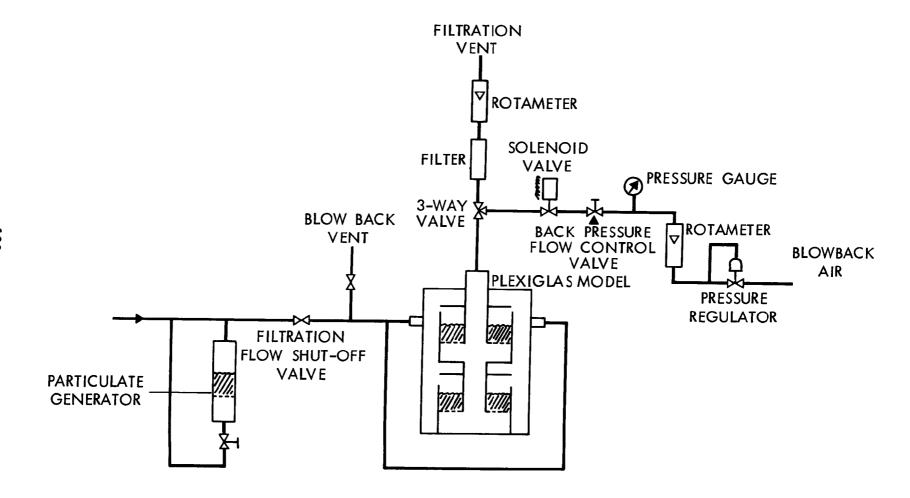
Ambient Temperature Models--

Because of various operating problems, especially loss of filter media and poor bed cleaning, transparent plastic models of the filter beds were built and tested. All the models operated at ambient temperature and close to atmospheric pressure. The first model was simply a plastic pipe, 2.4 cm in diameter. The pipe was filled with granular filter media, dust was added manually and the bed blown back with compressed air.

A two-dimensional plexiglas model of a granular bed filter was also built and tested. A schematic of the model is shown in Figure IV-21. The system was designed to operate in both the filtration mode and blow back mode although it was only used to study blow back operations. The model

FIGURE IV-20
FILTER BED WITH INTERNAL BAFFLE





consists of a two dimensional slice of two filter beds each with a filtration area of  $0.01~\text{m}^2$ . Each filter bed is 23 cm high with two 1.3 cm inlet slots. The filter media is supported by a 50 x 50 mesh screen supported by a fluidizing grid. Dirty gas enters the filter through two inlets, each feeding one side of the filter beds. Filtered gas exits the filter through a 5 cm central core and is passed through a backup filter to allow an estimation of collection efficiency to be made. Blow back air enters through the central core and is distributed to the filter beds. After passing through the filter beds, the blow back air is then vented to the atmosphere.

Another simple plastic model was built. This was 7.6 by 7.6 cm in cross section and 7.6 cm high. The chief feature of this unit is the design of the blow back system. Blow back air can be added through the bed support grid as in the other units, but it can also be added through a ring sparger, located at the top of the bed along the wall. The blow back air from the sparger is directed horizontally and was intended to shear the dust layer from the top of the bed without disturbing the filter bed itself to a large degree.

A second high temperature/high pressure filter was built and tested. This was a much smaller unit operated on a slipstream of flue gas withdrawn after the second cyclone. This unit and results of the tests are described in a later section.

#### Experimental Results

Six runs were made on the larger filter system during the reporting period. A summary of run conditions is given in Table IV-10. The changes in filter configuration described above were made during the test program. In addition, blow back conditions were varied to examine the effect of blow back gas velocity, duration and frequency on cleaning of the filter beds. At the conclusion of the six runs, an attempt was made to use the granular bed filter in a 100 hour test as part of the DOE sponsored program on hot corrosion and erosion of gas turbine materials.

The experimental program was not successful. Three major problem areas developed which were not satisfactorily resolved:

- low particulate outlet concentrations could not be maintained for more than a few hours,
- loss of filter media could not be prevented,
- serious operating problems persisted to the end of the program.

Outlet Particulate Concentration and Size Distribution--

Outlet particulate concentrations as low as 0.07 to 0.11  $g/Nm^3$  were measured in several runs. However, in all cases, the concentrations increased during the runs as much as a factor of three in 8 to 10 hours. Results from two typical runs are given in Figure IV-22. It is believed that this poor removal efficiency and the increase in particulate concentration with time were due to poor dust removal from the beds during blow back. This was supported by the observation that the filter beds, after completion of a run, were generally found to contain a very high concentration of dust intimately mixed

# TABLE IV-10. GRANULAR BED FILTER RUN SUMMARY

	Run No.	60	61
	Filter Description		
	Number of Parallel Elements Number of Beds/Element Retaining Screen Filter Media Bed Depth (cm) Filter Media Part. Size (µm) Other Information	2 5 50 Mesh Quartz 6.4 250-600 Ejector on One Element	2 5 50 Mesh Alumina 6.4 850-1400 Ejector on One Element
	Operating Conditions		
132	Preheat Temperature (°C) Filter Inlet Temperature (°C) Filter Outlet Temperature (°C) Filter Inlet Pressure (kPaa) Filter Face Velocity (m/s) Filter ΔP After Blow Back (kPa) Filter ΔP Before Blow Back (kPa) Run Length (hrs)	Run Unsuccessful Because of Poor Flow Distribution and High ΔP Measurement	760 840 690 900 0.26 and 0.47 <sup>(1)</sup> 9.6 and 30 <sup>(1)</sup> 24 and 45 <sup>(1)</sup> 5.5
	Blow Back Conditions		
	Superficial Velocity (m/s) Duration (s) Interval Between Blow Back (min)		0.61 8 10-15
	Particulate Emissions		
	Outlet Particulate Concentration (g/Nm <sup>3</sup> )		1.1

<sup>(1)</sup> Values for the two filter elements are different due to unequal flow split.

# TABLE IV-10 (CONT'D). GRANULAR BED FILTER RUN SUMMARY

Run No.	62.1	62.2	62.3	63
Filter Description				
Number of Parallel Elements Number of Beds/Element Retaining Screen Filter Media Bed Depth (cm) Filter Media Part. Size (µm) Other Information	2 5 10 Mesh Alumina 6.4 850-1400	2 5 10 Mesh Alumina 6.4 850-1400	2 5 10 Mesh Alumina 6.4 850-1400	2 5 None Speculite 1.9 425-2000
Operating Conditions				
Preheat Temperature (°C) Filter Inlet Temperature (°C) Filter Outlet Temperature (°C) Filter Inlet Pressure (kPaa) Filter Face Velocity (m/s) Filter \( \Delta \Pressure \) After Blow Back (kPa) Filter \( \Delta \Pressure \) Before Blow Back (kPa) Run Length (hrs)	760 830 700 900 0.41 14 to 20 35 2	830 700 900 0.41 20 to 24 35 2	 830 700 900 0.41 24 35 2	760 900 800 900 0.47 22 to 33 41 12
Blow Back Conditions				
Superficial Velocity (m/s) Duration (s) Interval Between Blow Back (min)	0.30 8 15	0.30 4 10	0.30 2 10	0.46 8 6
Particulate Emissions				
Outlet Particulate Concentration (g/Nm <sup>3</sup> )	0.069		0.48	0.11,0.16,0.27

# TABLE IV-10 (CONT'D). GRANULAR BED FILTER RUN SUMMARY

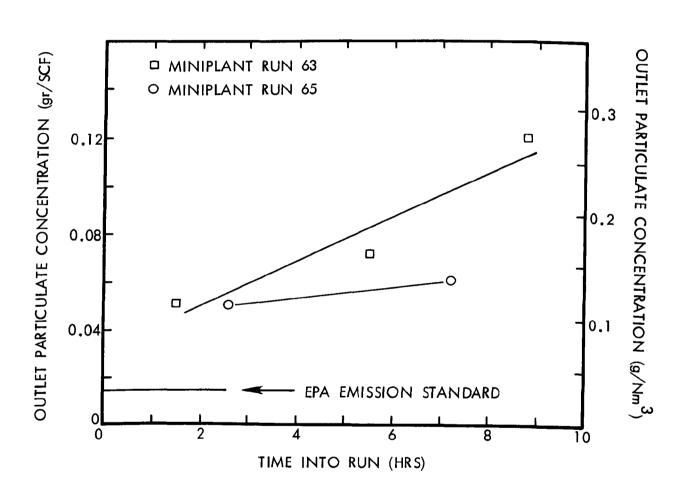
Run No.	64	65 <sup>(2)</sup>
Filter Description		
Number of Parallel Elements Number of Beds/Element Retaining Screen Filter Media Bed Depth (m) Filter Media Part. Size (µm) Other Information	2 5 None Speculite 6.4 425-2000	2 3 20 Mesh (One Element) Alumina 6.4 850-1400 Baffles On One Element (No Screen)
Operating Conditions		
Preheat Temperature (°C) Filter Inlet Temperature (°C) Filter Outlet Temperature (°C) Filter Inlet Pressure (kPaa) Filter Face Velocity (m/s) Filter ΔP After Blow Back (kPa) Filter ΔP Before Blow Back (kPa) Run Length (hrs)	760 950 840 900 0.36 10 and 14(1) 22 and 26(1)	760 850 800 860 0.51 7 to 34 34 to 62 8.5
Blow Back Conditions		
Superficial Velocity (m/s) Duration (s) Interval Between Blow Back (min)	0.82 4 to 5 3 to 5	0.61 4 4
Particulate Emissions		
Outlet Particulate Concentration (g/Nm <sup>3</sup> )	0.64,0.66,0.62,0.27	0.11,0.14

<sup>(1)</sup> Values for the two filter elements are different due to unequal flow split.

<sup>(2)</sup> A third cyclone was installed between the second cyclone and the filter vessel during this run, thus reducing the inlet particle concentration to the filter

FIGURE IV-22

INCREASE IN OUTLET PARTICULATE CONCENTRATION WITH TIME



with the filter media. Dust concentrations of 10 to 30% were typically measured. Tests made in the transparent models showed that the dust was only partly removed during blow back, some of it adhered to the filter media and was mixed into the bed during blow back by the motion of the fluidized filter media granules. The fine particles present in the PFBC flue gas are apparently very adhesive and are not removed easily during blow back. This mixing process probably explains the observed uniform distribution of the fine particulates throughout the filter beds after extended runs. Once the fine particulates were mixed into the filter media, they could be carried out through the bed support grid during the filtration step because of locally high gas velocities at the grid holes. As seen in Figure IV-22, at no time were outlet concentrations measured which meet the current emission standard of 0.03 lb/MBTU.

Attempts were made to improve bed cleaning by blowing back more frequently for longer periods and at higher velocities. The blow back velocity was limited by loss of filter media (with alumina or quartz media) to 0.3 to 0.6 m/s. The duration of the blow back was usually 4 to 6 s, although durations as short as 2 s and as long as 8 s were tested. The frequency of blow back was reduced from once every 15 min. to once every 4 min. As a result of these changes, the degree of filter cleaning appeared to improve, but the best run made, run 65, still showed an increase in outlet particulate concentration with time.

Another factor which could be responsible for the retention of particulates in the filter beds was the recycling of particulates from bed to bed during the blow back step. The shrouds which surround each element may impede the settling into the lock hoppers of the particulates blown from the elements during blow back. That is, if a new filtration step began before the particulates fell into the hoppers, the particulates could get carried back into the filter elements. This could result in high internal recirculation rates between elements, causing the particulates to build up in the beds and thereby causing an increase in the outlet particulate concentration.

In discussions with representatives of Ducon Company, the original supplier of the filters, Ducon claimed that the filtration velocities used in these tests were excessively high and could have promoted dust penetration (12). Ducon suggested a velocity of about 0.2 m/s.

Regardless of the reasons, the outlet concentrations measured in these tests were consistently higher than those measured when a third stage cyclone was used instead of the filter. Therefore, the filter showed no efficiency advantage over the cyclone. It should also be mentioned that the best run in the series of six shown in Table IV-10, run 65, was made with a cyclone installed between the second stage cyclone and the filter. The cyclone was not designed for the flow rate used in run 65 and was probably not too efficient, but it still probably contributed to the improved performance observed in that run. The cyclone was later removed and replaced with a more efficient cyclone which was used for the tests described in the earlier section on cyclone performance.

Filtration efficiencies were not measured during the test program since inlet particulate loadings could not be measured.

Typical particle size distributions of the dust entering and leaving the granular bed filter are given in Table IV-II. Additional particle size data after the filter for some of the filter runs are presented in Appendix M-6.

TABLE IV-11. PARTICLE SIZE DISTRIBUTIONS

		Percent	Less	Than Par	ticle Si	ze (µm)	
	5%			50%			95%
Filter Inlet	1.5	2.3	3.5	6.9	13	32	45
Filter Outlet	1.1	1.4	2.0	3.0	5.1	12	15

The outlet size distribution is relatively coarse when compared to typical distributions measured from the third stage cyclone. The cyclone consistently produced particulates with a median size of 1 to 2  $\mu m$  compared to 3  $\mu m$  shown in Table IV-11. The coarser particulate distribution in the filter outlet was also probably caused by dust penetration resulting from poor bed cleaning.

#### Loss of Filter Media --

The loss of filter media during blow back continued throughout the program and was never satisfactorily prevented. Screens were used in most runs to prevent loss, but the screens consistely plugged with flue gas particulate even though screen opening sizes as large as 10 mesh were used. The material plugging the screens was a loose agglomerate of dry, fine particles which could be removed by brushing. A photograph of a plugged screen was given in the previous report (1). Small openings above the screens and below the top flanges of each filter bed were used to allow entry of dirty gas and exit of blow back gas. This arrangement minimized but did not prevent loss of filter media. Alumina, 850 to 1400 µm, was used in all runs after 60 except 63 and 64 in which 425 to 2000 um speculite was used. The screens were removed in runs 63 and 64 since it was believed that the very dense and coarse speculite would not be lost from the beds. However, loss of the filter media still occurred. In addition, in run 64, a temporary upset caused an increase in the CO concentration in the flue gas. This apparently reduced the speculite (from oxide) to Fe° which was then oxidized after the CO concentration decreased. This is a highly exothermic reaction. As a result, the filter was severely damaged. Two filter beds could not be repaired. Fine iron oxide dust was also found in the filter outlet piping sections. The use of speculite was discontinued.

Testing of the two dimensional transparent model then began in an attempt to determine the cause and possible cure for the filter media loss problem. A series of tests were conducted in which various internal baffles and inlet gas slot sizes were used. The most significant finding from these tests was that most of the filter media loss occurred at the beginning of the blow back. The sudden opening of the blow back valve caused a surge in gas flow which blew filter media from the vessel. The baffles did not prevent loss caused by the

flow surge although some designs did decrease losses caused by high blow back velocities following the initial surge. Baffles were installed in one filter element and tested in run 65. Screens were removed from this filter element and retained on the other element. Both elements showed loss of filter media, the baffled element without screens showed the greater loss, 32% of the starting media bed compared to 18% from the second element. The use of unscreened baffled filters was then discontinued.

The plexiglas model tests would indicate that slower acting blow back valves or surge capacity between the valve and the filter would decrease the loss of filter media caused by the flow surge. Ducon representatives also claimed that filter bed freeboards 50% larger than those used in these tests are needed to prevent loss during blow back (12).

Other Operating Problems --

In addition to the problems described above, other operating problems occurred which persisted until the end of the program despite attempts to improve operations. One problem was an increase in the combustor pressure which occurred during filter blow back. At times, the increase was large enough to cause the combustor pressure to exceed the pressure in the coal injection vessel. When this happened, the hot solids in the combustor would back up into the coal injector vessel, causing a fire in the vessel. A nitrogen purge system on the coal injector was used to extinguish the fires, but these incidents always caused the termination of the run and removal of some charred coal from the injector vessel.

Temperature drops across the filter was another problem. Although this did not affect performance of the filter, it did cause problems for the gas turbine materials test. Flue gas temperatures were required to stay above 840°C at all times to prevent condensation of alkali sulfates, the material which could cause hot corrosion of the turbine parts. Injection of cold blow back air and heat losses from the filter elements to the interior of the pressure vessel resulted in temperature drops in excess of those required. Injection of natural gas into the filter outlets, insulation of the filter shrouds and piping of the clean flue gas from the filter outlet directly to the pressure vessel outlet decreased the temperature drop to an acceptable level.

An attempt was also made to replace the blow back nozzle and seal plate with an ejector. This would decrease the amount of cold blow back air needed and also replace the seal plate which occasionally did not work satisfactorily. An ejector was installed on one filter element and tested in runs 60 and 61. The second element was equipped with the nozzle and seal plate arrangement. In the tests with the ejector, it was found that the pressure drop through the ejector during the filtration step was high, giving rise to unequal flow rates between the two filter elements. The element with the ejector was not cleaned as well during blow back and the outlet loading from the test (run 61) was very high, 1.0 g/Nm³. The ejector was removed and not tested further.

Poor bed cleaning also led to high pressure drops across the filter. At the beginning of a run, the pressure drops were usually around 14 to 20 kPa after bed blow back, increasing to about 28 to 35 kPa before blow back. The

pressure drops usually increased as the run progressed, indicating poor bed cleaning. Pressure drops of 28 to 35 kPa after blow back and 40 to 60 kPa before blow back were measured after 8 to 15 hours of filter operation.

Another intrinsic problem with the filter was the fact that essentially all problems were non-corrective and as such, required termination of the run, a long wait to cool down the filter, followed by a long period to remove, clean and replace the filter elements.

The final run made with the filter was an attempt to run a 100 hour gas turbine exposure test (run 66). In this case, three filter elements were used in parallel, each with three filter beds. The filters were charged with alumina filter media (850 to 2000 um). The inlet slots were covered with 20 mesh screens except for a 3 mm split above the screens to allow gas flow if the screens plugged. Filter blow back occurred every 4 min. at 0.3 m/s for 6 s. These were judged to be the best conditions to assure a successful test. Over the first four hours of operation, a gradual increase in the filter  $\Delta P$  was noted which then increased sharply to 207 kPa. The blow back velocity and duration were increased to improve bed cleaning. The  $\Delta P$  dropped to 70 kPa. but a significant amount of filter media was lost. As a result of the unstable pressure drops and frequent high velocity blow backs, the combustor pressure control was upset and hot solids back flowed into the coal injector causing a fire. The run was terminated after 17 hours operation. After the run, it was found that the high pressure drops were caused by partly blocked filter support screens. At this point, the granular bed filter program involving the use of the multi-bed filter elements contained in the large pressure vessel was terminated.

During the test program, a series of tests were also made with the transparent models in an attempt to determine the cause of the poor bed cleaning. Some of the test results were described in a previous section of this report. They showed how a portion of the dust particles adhered to the filter media and were mixed into the filter bed by the motion of the fluidized filter media granules during blow back. In order to prevent this, a modified blow back scheme was tested. In this case, most of the blow back air entered the bed through a ring sparger located at the interface between the filter media and the dust layer. Blow back air was directed horizontally across the interface. This resulted in a shearing action which blew off the dust layer without disturbing the filter media. Blow back upwards through the filter media was minimized as was the mixing effect caused by it. This technique looked promising enough to be tested under PFBC conditions.

In order to test this as well as other concepts which could improve the filter performance, a program was planned to test a modified filter on a flue gas slip stream. The slip stream tests were to be made with a small, single bed filter incorporating a number of design modifications. The design of the new unit would build on the lessons learned in the previous test program and would attempt simply to show if the granular bed filter concept was feasible for pressurized fluidized bed combustion applications.

## Granular Bed Filtration Slip Stream Test

The single element granular bed filter device was tested on a miniplant slip stream during run 115. The filter element was a further modification of the "modified Exxon filter element" described in Figure IV-18. The location and instrumentation of the test vessel which contained the filter element, are almost identical to those of the Acurex high temperature ceramic filter vessel. This was done to allow a fair comparison of the two high efficiency hot gas cleanup devices.

The filter element tested was named Exxon Mark IV to distinguish it from earlier designs. The filter element body was the same one used in full scale tests. Three things were changed:

- 1. The fluidizing grid was replaced with a support grid with 1 cm diameter holes, in order to reduce  $\Delta P$  and reduce the velocity of the grid jets during below-bed blow back.
- 2. A fluidizing coil was installed just above the support grid.
- 3. An above-bed blow back coil was installed.

These three modifications were intended to lower the baseline pressure drop, reduce bed loss during blow back and improve the effectiveness of the blow back. Both coils have 0.84 mm holes every 1.6 cm along the coil. In the below-bed coil, the holes are oriented approximately 45° from the vertical to provide upward fluidizing jets. In the above-bed coil, the holes are horizontal to blow the dust cake off the surface of the filter media by a shearing action without disturbing the filter media granules to a great extent. In this way the superficial blow back velocity also increases just above the bed to blow more particulate and less filter media out of the bed. Air could still be added below the support grid to aid in fluidization of the bed. A schematic of the element is shown in Figure IV-23.

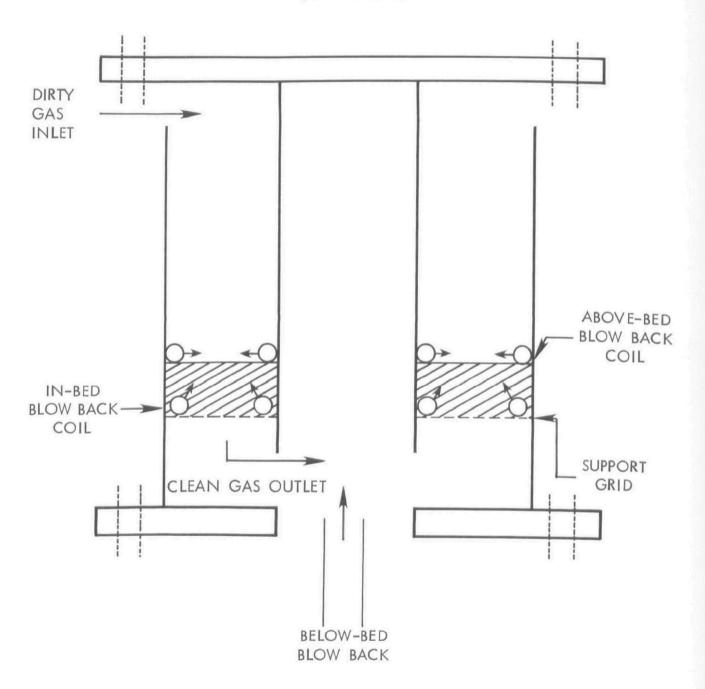
The test vessel which contained the Exxon Mark IV element, was installed on an isokinetic slip stream taken from a point between the second and third cyclones. A schematic of the installation is shown in Figure IV-24. The valves and electronic timers are the same ones used for the Acurex ceramic filter tests. The high pressure (1300 kPa) air supply is used on the above-bed coil for a pulsed blow back. The lower pressure air supply (1000 kPa) is used in the in-bed coil and for the below-bed blow back air. Particulate samples were taken before and after the ganular bed filter using the Balston filter method.

### Results and Discussions--

The Exxon Mark IV filter was run for 4 hours during run 115. Before filtration was begun, the inlet lines were preheated with flue gas to a temperature above  $400^{\circ}\text{C}$  to prevent condensation in the line. The filter could not be preheated. The filter element was charged to a 7 cm bed depth with  $\sim$ 7 kg of -8+14 U.S. mesh alumina. The blow back cycle was program 1 in Table IV-12. The filtration period was 5 min with a 20 sec cleaning cycle. After the first 45 min it became apparent that the pressure drop could not be controlled with this blow back program. At this time, the cleaning cycle was

FIGURE IV-23

EXXON MARK IV GRANULAR BED FILTER (SINGLE BED)



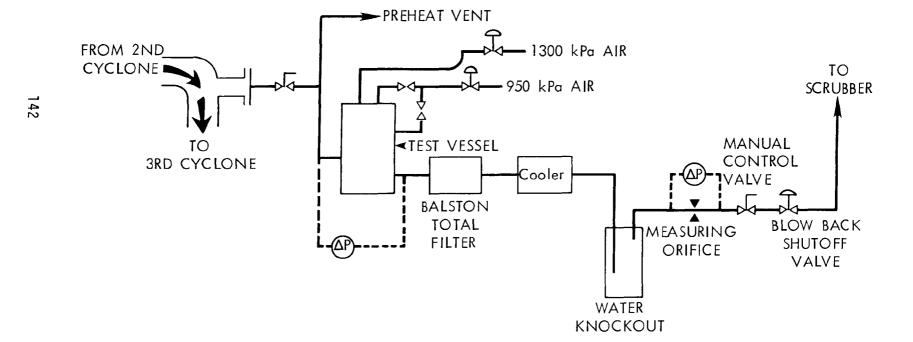


TABLE IV-12. GRANULAR BED FILTER CLEANING PROGRAM

# Program 1

Event		Time (m:s)
0	Forward Flow Off, Reverse Flow On	0:00
1	Above Bed Pulse (1.5 sec)	0:02
2	Above Bed Pulse (1.5 sec)	0:07
3	Above Bed Pulse (1.5 sec)	0:14
4	Reverse Flow Off, Forward Flow On	0:19
5	Reset To Start Cleaning Cycle	5:19

# Program 2

Event		Time (m:s)
0	Forward Flow Off, Reverse Flow On	0:00
1	Above Bed Pulse (1.75 sec)	0:01
2	Above Bed Pulse (1.75 sec)	0:05
3	Above Bed Pulse (1.75 sec)	0:10
4	Above Bed Pulse (1.75 sec)	0:14
5	Above Bed Pulse (1.75 sec)	0:17
6	Forward Flow On, Reverse Flow Off (Filtration)	0:19
7	Reset To Start Cleaning Cycle	5:19

TABLE IV-13. COMPARISON OF PARTICULATE SIZE DISTRIBUTION OF MATERIAL BEFORE AND AFTER FILTER TEST ELEMENT

	Particle Size (μm)						
				Finer T	han Size		
	5%	10%	25%	50%	75%	90%	95%
<u>Sample</u>							
Before Filter	1.4	1.7	2.4	3.1	4.2	5.6	6.4
After Filter							
Test 1	1.4	1.7	2.2	2.8	3.8	5.1	6.4
Test 2	1.4	1.7	2.4	3.0	4.0	5.4	6.2

changed to program 2 in Table IV-12. After 3 hours, the filter was isolated from the miniplant and the Balston total filter which follows the granular bed filter test vessel was replaced. This Balston sampling filter was completely destroyed by the high temperature of the gas exiting the filter vessel. A large amount of particulate matter was found in the off gas piping before and after the Balston total filter. This was evidence of a large amount of particulate penetration. A second Balston filter was also destroyed. After the removal of the second filter, the Balston filter vessel failed and the test was terminated. The size distribution of the particulates removed from the filter support screen is shown in Table IV-13. The average particle size of 3 microns is almost identical to the average size of material before the filter. Therefore it appears that very little filtration took place.

The filtration velocity that was used during these tests was higher than any tested before on the miniplant. This happened because the  $\Delta P$  cell which measures the pressure drop across the measuring orifice was miscalibrated. It was calibrated for 0-30 kPa rather than 0-30 in Wc. This error resulted in actual filtration face velocities much higher than expected.

During the second Balston total filter sampling test, the pressure drop across the filter was constant throughout the filtration cycle. This indicated a complete loss of filter media. This, along with the failure of the sampling filter vessel, caused the termination of the test.

After the run, the plenum of the test vessel was opened and approximately 7 kg of material was collected. This material was alumina bed material, which had been blown out of the filter bed, containing less than 10% flyash.

A summary of granular bed filter test conditions and results for Run 115 is shown in Table IV-14. Even with all of the things that went wrong during the test, some conclusions are possible. With a single element and controlled blow backs, the maintenance of a proper  $\Delta P$  was very difficult. The pressure drop continued to rise even during periods when some bed material was lost. The pattern of pressure buildup with time can be seen in Figure IV-25. Perhaps lower face velocities would have kept the pressure drop from rising as quickly. However, the fact remains there was a continued failure to clean the filter properly and check the increased pressure drop even with significant loss of filter media. The operating problems seen in this run, poor filter cleaning and loss of filter media, are the same problems that terminated the earlier program.

Unfortunately, the testing of the slip stream filter was terminated before all operability questions could be answered. An unresolved issue was whether the loss of filter media could have been prevented by modifying the blow back procedure, possibly by introducing blow back air more gradually instead of in short pulses. The effect of lowering the filter face velocity on filtration performance was also unresolved.

# TABLE IV-14. RUN 115 FILTER TEST SUMMARY

Run Length (total)	4 Hours
Number of Beds	1
Design	Exxon Mark IV
Filter Medium	Alumina
Bed Depth (cm)	7
Filter Media Particle Size (µm)	1410-2380
Vessel Preheat	Inlet Pipe Only

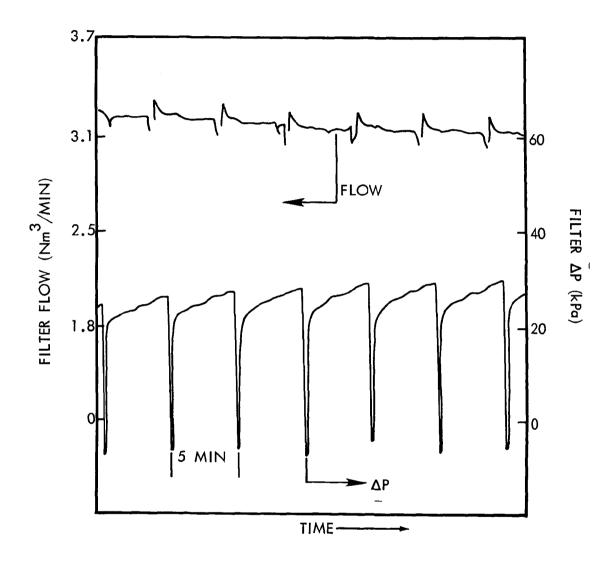
# Run Breakdown

Segment	_1	2	3
Run Length (hrs)	0.75	2.25	1
Operating Conditions			
Filter Inlet Temperature (°C) Filter Outlet Temperature (°C) Pressure (kPa) Baseline ΔP	400 350 900	NA 630 900	NA 630 900
Clean Bed (kPa)  AP Before Blow Back (kPa)  Superficial Velocity (m/min)  Duration: Filtration (min)	28 35 38 5	38 43 52 5	23 23 45 5
Blow Back Conditions			
Superficial Velocity Duration: Blow Back (s) Blow Back Program	NA 20 1	NA 20 2	NA 20 2

NA = Not Available

FIGURE IV-25

GRANULAR BED FILTER FLOW AND PRESSURE DROP VS. TIME



#### CONVENTIONAL PARTICULATE CONTROL

A series of long term (4 to 10 day) continuous tests were carried out in which a conventional low temperature, low pressure electrostatic precipitator (ESP) and bag house were tested on the flue gas from the miniplant. These tests were conducted during miniplant runs 103 through 108, with the three stages of high temperature/pressure cyclones in operation; the low temperature/pressure devices thus represented a sourth stage of particle cleanup. The purpose of the tests was to determine if an ESP or bag house could be used, after expansion of the flue gas through the gas turbine, to meet particulate emission standards. The possibility being considered was that cyclones may be sufficient to protect the gas turbine from excessive wear but may not be sufficient to meet environmental standards. The tests were performed using mobile, trailer mounted control devices operated by Acurex Corporation for the EPA. These units received a stream of expanded, diluted miniplant flue gas during 3 runs each for the ESP (runs 103, 104, 105) and the bag house (runs 106, 107, 108). Due to the system configuration, the flue gas was diluted by 50 to 70% with air used for pressure control. A sketch of the test configuration is shown in Figure III-10.

### Results

Preliminary results with the EPA mobile ESP indicate the applicability of conventional electrostatic precipitation for control of cyclone-cleaned PFBC particulate emissions. Overall results from 17 days of operation (runs 103, 104 and 105) reflect 87% efficiency, corresponding to an emission level of 8.6  $\,\mathrm{ng/J}$  (0.02  $\,\mathrm{g/Nm^3}$ ).

Preliminary results with the EPA mobile bag house also indicated the applicability of conventional fabric filtration for control of cyclone-cleaned PFBC particulate emissions. Overall results from 15 days of operation (runs 106, 107 and 108) reflect 99.3% efficiency, corresponding to an emission level of 0.46 ng/J (0.001 g/Nm $^3$ ).

A more detailed and comprehensive accounting of these tests will be contained in a report to be published in late 1979 by Acurex.

#### SECTION V

#### REGENERATION STUDIES

In 1976, the combined operation of the miniplant combustor and regenerator sections was demonstrated. That demonstration run, reported in the previous annual report, illustrated the operability of the system and demonstrated that sorbent regeneration does reduce the amount of makeup sorbent required. The next step in the regeneration study, and the primary objective of the combined combustor-regenerator test program conducted in 1979, was to quantify the effect of certain key variables on the performance of the combined combustor/regenerator system. The variables studied were makeup Ca/S ratio and sorbent recirculation rate.

EQUIPMENT AND PROCEDURES

### Equipment

The equipment and materials were described in detail in the previous two annual reports (1,2). No major changes have occurred since, and only brief summaries are included in this report.

Air System--

The two separate air systems are burner air and supplementary air. All air is supplied by the main air compressor. Automatic control systems, consisting of control valves, flow measuring orifices, and electronic controllers, are used to regulate air flow. Burner air is supplied to the burner, located beneath the fluidizing grid, in sufficient quantity to completely burn the fuel (natural gas). Supplementary air is added about halfway up the bed in order to create an oxidizing zone in the upper portion of the bed.

Fuel System--

The two fuel systems are burner fuel and supplementary fuel. Automatic control systems, similar to those used for air flows, are used to regulate the flow of natural gas. Burner fuel is supplied to the burner where it is burned with an approximately stoichiometric amount of air. Supplementary fuel is added directly to the regenerator column just above the fluidizing grid in order to produce reducing gases  $(CO, H_2)$ .

Off Gas Handling --

Hot pressurized gases leaving the regenerator are cooled in a single pass double pipe heat exchanger and expanded to nearly atmospheric pressure across a control valve. Dust is removed from the gas upstream of the cooler by a cyclone and upstream of the pressure control valve by a stainless steel knockout vessel.

Off gases from the regenerator are sent to a Research-Cottrell scrubber for cleanup before venting. Ammonia is injected to neutralize the scrubber water.

Gas Sampling System--

A slipstream of the off gas is taken downstream of the pressure reducing valve. The gas is filtered (Balston Model 33 filter) and dried (Perma-Pure Model PD-1000-24S self-regenerative membrane-type dryer) before entering the gas analyzers.

Fluidizing Grid --

The fluidizing grid has 88 holes of 3.6 mm (9/64 in) diameter for passage of the fluidizing gases (from the burner located beneath grid) and 14 water cooling channels of 4.8 mm (3/16 in) diameter. The cooling water flow is controlled independently through six separate cooling zones. The 14 channels were separated into groups of 3, 2, 2, 2, and 3, each group or zone having its own water supply.

#### Burner--

No changes were made to the regenerator burner since the last report. This unit is identical to that used in the miniplant combustor and is described in a previous annual report (2).

Sorbent Transfer System --

The solids transfer system used to circulate solids between the combustor and regenerator is shown schematically in Figure V-1. Pressure in the regenerator is maintained slightly higher than that in the combustor. Solids in the regenerator-to-combustor transfer line move into the combustor when a pulse of nitrogen is applied to the lower end of the transfer line. The flow rate of solids is controlled by adjusting the frequency, duration, and intensity of the pulse. Two slide valves are used in the combustor-to-regenerator transfer line in order to prevent back flow of gas from the regenerator up the line. These automatic valves trap solids in the piping between them. Solids are discharged into the regenerator when the bottom valve is opened. The two solids' take-off plugs shown in Figure V-1 are inserted into the ports during startup to prevent solids from entering the lines. Plugging can occur if the solids become wet due to water condensation during startup. The manual slide valve in the regenerator-to-combustor line is also closed during startup and during upsets.

The components of the sorbent transfer system (valves, expansion joints, etc.) are described in detail in a previous annual report (2). The transfer lines themselves were fabricated from 6 inch schedule 40 carbon steel pipe and refractory lined to an inside diameter of 7.6 cm. The sloping portions of the lines were sleeved with 2-1/2 inch Schedule 10 316 stainless steel pipe, which has an inside diameter of 6.7 cm.

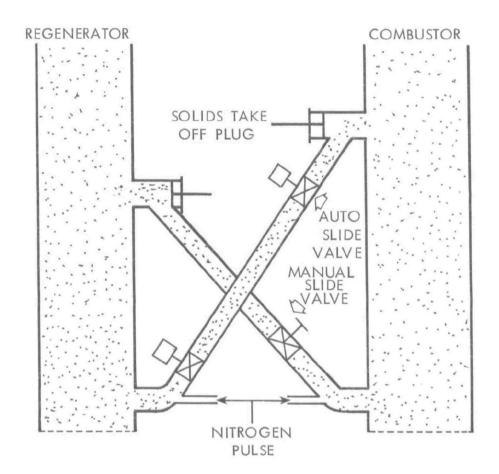
#### Operating Procedures

Startup of Transfer System--

The solids transfer system is not operated during startup to prevent moisture from entering the transfer lines. Plugs are inserted in the solids takeoff ports so that solids cannot spill into the lines from the combustor and regenerator vessels. Nitrogen is pulsed into the transfer line pulse pots

FIGURE V-1

MINIPLANT SOLIDS TRANSFER SYSTEM (SCHEMATIC)



in order to prevent solids from backing up into the lines. The slide valves are also cycled occasionally in order to dislodge any solids that may have passed by the takeoff plugs. Pressure in the regenerator is set slightly above that in the combustor (usually 2.5-10 kPa) and the beds are heated to near operating temperatures. Transfer of solids is started by pulling the plugs out of the takeoff ports. The transfer rate is controlled by setting the cycle time of the slide valves in the combustor-to-regenerator line.

Starting Reducing Conditions --

The regenerator is operated under net oxidizing conditions during heatup. Under these conditions, no regeneration of sulfated sorbent occurs. When the combustor and regenerator bed temperatures are uniform and close to the desired operating temperatures, the switchover to reducing conditions in the regenerator is made. This is accomplished by increasing flow of supplementary air to the required value and then increasing the flow of supplementary fuel. Supplementary air flow is always increased before supplementary fuel so that air is not added to a column already filled with a reducing gas. Temperature is continuously monitored and flow rates of burner air, burner fuel, supplementary air, and supplementary fuel are all adjusted to yield the desired bed temperature. Oxygen and CO concentrations in the off gas are also monitored and the supplementary air flow is corrected to produce low concentrations of CO (under 5000 ppm). The flow rate of supplementary air is the minimum value which will just produce CO in the oxidizing zone.

#### Shutdown--

It is important during shutdown, to empty the transfer lines of solids; otherwise plugging of the lines may occur when the unit is restarted. Hence, the first step is to shut the plugs in the solids takeoff ports, thereby preventing solids from entering the lines. Cycling of the slide valves in the combustor-to-regenerator line and pulsing of nitrogen in the regenerator-to-combustor line is continued until the miniplant is shut down. This assures that the lines are emptied.

Normal shutdown can be accomplished several ways. One method is to press the "emergency stop" button, which shuts all flows of air and fuel, fills the column with nitrogen and slowly depressurizes the column. This method is preferable if the final composition of solids is important, as nitrogen plus the rapid temperature drop "freezes" the composition of the bed.

Another method of shutdown is to turn off supplementary fuel and air flows, in that order. Burner fuel is shut next. Column pressure and burner air flow must be brought down together so as not to create high superficial velocities, which would blow solids out of the column.

EXPERIMENTAL RESULTS AND DISCUSSION

#### Shakedown

After some cold flow tests, a hot test, run 101, was made to insure that the solids transfer lines and control systems were operating smoothly and to determine the operating pressure for the test program (7 atm). Shakedown went

very smoothly with the combined units operating as well as they had during the first 100 hour demonstration run in 1976.

### Operating Performance

During the regenerator test series (runs 102, 103 and 105) over 400 hours of operation were logged with solids circulating between the regenerator and combustor, 370 of these hours were with sorbent regeneration. Including the 100 hour demonstration run in 1976, the combined combustor-regenerator system was operated for more than 500 hours. Overall, operations of the two units was smooth. Combustor problems were limited to infrequent interruptions in coal feed due to coal flow problems which were quickly remedied. Problems in regenerator operation, which were basically limited to two areas, required maintenance during and between runs but did not result in forced shutdown. The two problem areas were off gas handling and solids transfer.

Regenerator off gas passes through a cyclone before being cooled and pressure reduced. The cyclone was operational during run 102, but during the second day of run 103, the downcomer became plugged with fines, probably a result of wall buildup from the previous run coupled with moist particulates from the startup of 103. A large, semi-batch granular bed sand filter was put on stream to replace the cyclone rather than shutting down to clear the cyclone. The high dust loading in the regenerator off gas prior to the installation of the sand filter and during the filter blow back intervals caused appreciable erosion of the pressure control valve stem and housing.

The major problem with the solids transfer system was a blockage in the combustor to regenerator line that prevented solids movement at the start of run 103. It was found that a piece of refractory wall in the 45° angle section just above the pulse pot had fallen into the opening. The section was removed, the refractory recast, and the run resumed.

The solids transfer system also proved to be a limiting factor on the solids circulation rate. As solids are transferred from the combustor to regenerator, they are trapped and held between the two automatic knife valves. While the solids are trapped between cycles, they cool. If the transfer leg temperature drops too low, water vapor may condense making the solids mushy and preventing solids flow. The lowest solids circulation rate which seemed safe for the existing system was 10-12 kg/hr.

During the regeneration test series, additional testing was done by outside contractors. Acurex Company tested a mobile electrostatic precipitator using a slip stream of combustor flue gas during runs 103 and 105. GCA Technology Company performed Level I comprehensive analysis tests on both the combustor and regenerator during run 105. These test programs are discussed elsewhere in this report.

#### Run Conditions and Summaries

Run summaries giving operating conditions for both the combustor and regenerator are presented in Tables V-1 and V-2. During the 3 runs, six steady state conditions were reached. Temperatures, pressures, superficial

TABLE V-1. MINIPLANT REGENERATOR RUN SUMMARY
Regenerator Operating Conditions

Run No.	102	103.0	103.1	103.2	103.3	105
Run Length, (reducing) hrs	97.5	_54	48	20	56.5	80.7
Pressure, kPa	710	710	710	710	710	<b>7</b> 05
Air Flow Rate, m <sup>3</sup> /min	2.86	3.08	2,72	2.52	2,31	2.3
Fuel Flow Rate, m <sup>3</sup> /min	0.26	0.30	0,27	0.23	0.20	0.23
Average Bed Temp - Reducing Zone	1025	1004	1003	1008	974	1010
°C - Oxidizing Zone	1053	1024	1021	1025	985	1031
Superficial Gas Velocity, m/s						
- Reducing Zone	0.64	0.78	0.67	0.60	0.58	0.60
- Oxidizing Zone	0.91	1.08	0.98	0.87	0.78	0.77
Expanded Bed Height, m	1.65	1.37	1.38	1.38	1.37	1,85
Air to Fuel Ratio, φ						
- Reducing Zone	1.36	1.31	1.37	1,35	1.32	1.27
- Oxidizing Zone	0.92	0.95	0.94	0.91	0.96	1,02
Solids Recirculation Rate, kg/hr	88.5	22.7	22.7	12.2	12.2	15.4
Average SO <sub>2</sub> Emissions, %	0.32	0.40	0.46	0.25	0.16	0.51
Average CO Emissions, ppm	1610	934	484	231	996	1438
Average CO <sub>2</sub> Emissions, %	9.8	9.44	9.42	8.38	8.69	10.9
Average 0 <sub>2</sub> Emissions, %	0.17	0.21	0.31	0.42	0.18	0.26

TABLE V-2. MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Combustor Operating Conditions	102 <u>3/13<b>-</b>17/79</u>	103.0 3/29-4/1/79	103.1 <u>1/4-3/79</u>	103.2 4/3-4/79	103.3 4/4-6/79	105 4/30-5/5/79
Run Length, hrs.	107	81	49	22	59	99
Pressure, kPa	700	700	700	700	700	700
Air Flow Rate, m <sup>3</sup> /min	12.4	13.3	13.4	14.1	14.4	14.0
Temperature Gradient, °C/m	-15.2	-12,2	-14.0	-12.3	-17.7	-55.9
Average Bed Temperature, °C	908	913	901	901	902	894
Superficial Velocity, m/sec	1.43	1.52	1.52	1,59	1,62	1.46
Settled Bed Height, m						
Initial	2.06	2,11				2.23
Final	0.96				1.42	1.71
Expanded Bed Height, m	2.17	2.65	2.83	1.97	2.24	3.10
Coal Feed Rate, kg/hr	69.5	76.6	77 <b>.</b> 7	79.4	79.9	77
Ca/S Molar Feed Ratio-Set*	1.5	1.35	0.68	0.68	0.93	1.29
Ca/S Molar Ratio-Equivalent**	54	12	11	6,1	6.4	8.3
Excess Air, %	20.3	16.8	17.7	13.0	18.9	45.8
Sorbent	GL	GL	GL	GL	GL	GL
Coal	СН	CH	CH	СН	СН	CH
Flue Gas Emissions - Combuston	•					
SO <sub>2</sub> , ppm	27	28	41	22	77	70
NO <sub>X</sub> , ppm	126	77	56	65	55	59
CO, pom	90	71	62	64	56	77
co <sub>2</sub> , %	14.0	13.7	14.1	14.1	13.7	12.4
02, %	4.0	3.3	3.4	2.6	3.6	4.1
Results - Combustor						
SO <sub>2</sub> Retention, %	97.1	97.5	96.4	98.0	93.0	93.6
Ca Sulfation, %	5.1	18.2	23.7	22.2	28.0	23.4
Lb SO <sub>2</sub> /M BTU	0.06	0.06	0.09	0.05	0.17	0.15
Lb NOX/M BTU	0.19	0.12	0.09	0.10	0.09	0.09

GL = Grove Limestone

CH = Champion Coal

 <sup>\*</sup> Fresh sorbent feed only.
 \*\* Includes recirculating sorbent from regenerator, as well as fresh sorbent feed; assumes 100% regeneration of the recirculated sorbent.

gas velocities, bed heights, coal feed rates, sorbent type, coal type, and reducing atmosphere composition were held fairly constant throughout the runs. Solids recirculation rate and Ca/S ratio fed to the combustor were the variables studied for their effects on combustor and regenerator SO<sub>2</sub> emissions, sulfur retention, and makeup sorbent rate needed to meet EPA emission standards.

### Results

In all cases, sulfur retention exceeded 90% with makeup Ca/S ratios ranging between 0.68 and 1.5, compared to a Ca/S ratio of 3 to 4 which is required to achieve the same results for once through operation with limestone. The significant reduction in fresh (makeup) sorbent requirements is due to the regenerated sorbent which circulates from the regenerator to the combustor.

The Ca/s The

$$Ca/S_{EQ} = \frac{0.32 \times SCA \times (SFR \times SCR + 1.79 RSR \times (1 - SSL) \times (1 + SCR))}{CS \times SFR}$$

where SCA = % CaCO3 in sorbent, for limestone ~100%

SFR = coal + fresh sorbent feed rate, kg/hr

SCR = sorbent to coal ratio, kg/kg

RSR = recirculating sorbent rate, kg/hr

SSL = sorbent sulfation level of regenerated sorbent, wt. fraction

CS = coal sulfur content, %

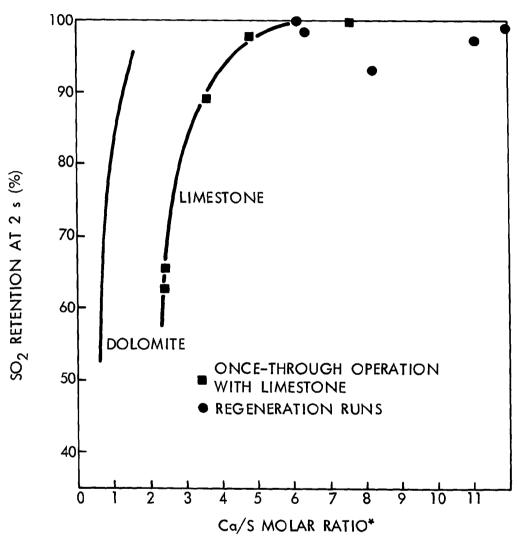
Comparison of the fresh sorbent Ca/S ratio with the Ca/S equivalent shows that the rate of circulating regenerated sorbent is much larger than the fresh sorbent rate and therefore, probably plays a larger role in controlling  $SO_2$  emissions.

Combustor SO2 Retention --

As mentioned previously,  $SO_2$  retention exceeded 90% for the runs made, covering a range of Ca/S ratios. An effective method to evaluate sorbent regeneration is to compare  $SO_2$  retention as a function of Ca/SEQ for the regeneration runs to the data obtained for once through operation. Figure V-2 shows such a comparison. All  $SO_2$  retentions were adjusted to a 2 second gas residence time using the first order rate expression.

Figure V-2 shows that the data points generated by the regeneration runs fall around the curve for once through operation with limestone. The values for Ca/SEQ are based on an assumption of 100% sorbent regeneration due to difficulty in obtaining regenerated bed samples. If the samples were not fully regenerated, i.e., the assumption was invalid, the data points would shift to the left toward a lower Ca/SEQ. For example, one regenerator bed sample was obtained during run 105 when a pressure upset caused some regenerator bed to be blown out of the column into the cyclone. The sample was analyzed and found to contain 14.6% CaSO4 by weight. The equivalent Ca/S molar ratio dropped from 8.3 to 6.0 when the sorbent sulfation level was taken into account in the Ca/SEQ expression.

FIGURE V-2 SO  $_2$  RETENTION ADJUSTED TO 2  $_{\rm S}$  RESIDENCE TIME VS Ca/S RATIO



\* FRESH SORBENT ONLY FOR ONCE-THROUGH RUNS FRESH PLUS RECIRCULATED SORBENT FOR REGENERATION RUNS

The scatter in the data is not appreciable and from this treatment of the data it can be concluded that the regenerated sorbent behaves as if it were fresh sorbent.

#### Sorbent Activity Loss--

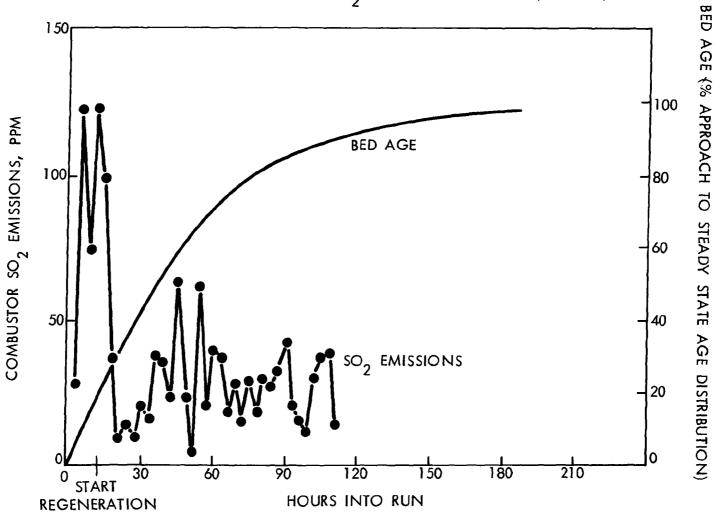
Previous work done in TGAs and batch units indicated a decline in sorbent activity as the sorbent was cycled between sulfations and regenerations (13). In a continuous unit such as the miniplant, the sorbent does cycle a number of times but the cycling is not comparable with batch cycling experiments. Sorbent lost by attrition must be replaced with makeup (fresh) sorbent. The fresh sorbent feed serves to maintain the age distribution of the sorbent in the system, and therefore the activity of the bed, at a steady state value. From the outset of a run, the average age of the sorbent bed changes exponentially and the steady state bed age distribution is approached asymptotically. The rate at which the bed steady state is reached is determined by the system time constant, t, where t = bed inventory/feed rate. (Based upon the range of values for t in these miniplant runs, the exponential expression would suggest that the combustor bed should approach within 90% of its steady state level in about 90 hours of operation.) Therefore, unless the sorbent suffers rapid and severe deactivation, sorbent deactivation would not be obvious in a continuous system once a steady state bed age distribution has been reached.

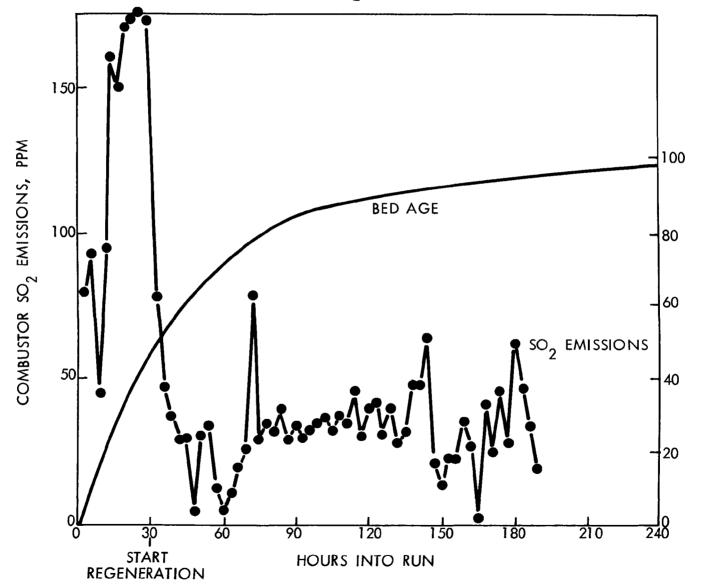
Figures V-3, V-4 and V-5 illustrate the change in average bed age with time as well as the combustor  $SO_2$  emissions for the regeneration test runs. When the regenerator was brought into reducing conditions and regenerated sorbent began to enter the combustor at high rates, combustor  $SO_2$  emissions dropped markedly. As seen in the figures, the combustor  $SO_2$  emissions leveled out at a steady state value a long time before the bed reached its steady state age distribution. If the sorbent experienced deactivation, this would have been detectable as an increase in combustor  $SO_2$  emissions during the initial line-out period.

Based on the results exhibited in Figures V-3 through V-5, it can be concluded that during the residence time of a sorbent particle, minimal or no sorbent deactivation occurs or if deactivation does occur, it does not greatly alter the overall activity of the bed.

The results of the regeneration test series which are reported here contradict the findings of the regenerator demonstration run reported previously (1). In the earlier run, after about 50 hours combustor SO<sub>2</sub> emissions began to increase rapidly for a period of 50 hours, then leveled out at 550 ppm. The increase in emissions was assumed to have been caused by a gradual decline in the activity of the regenerated sorbent. This hypothesis is not supported by the most recent miniplant regeneration studies where there was no sign of sorbent deactivation. It is also not consistent with the analysis of the recent runs described above. This analysis points out that if deactivation occurs, its effect would only be seen during the initial part of the run and not as far into the run as 50 hours. The increase in combustor SO<sub>2</sub> emissions during the previous run may have been a result of operational difficulties rather than sorbent deactivation. If sorbent regeneration or solids transfer was inhibited for any reason, such as a bed agglomerate, sulfur retention in the combustor would be adversely affected.

FIGURE V-3 COMBUSTOR BED AGE AND  $\mathrm{SO}_2$  EMISSIONS VS. TIME (RUN 102)

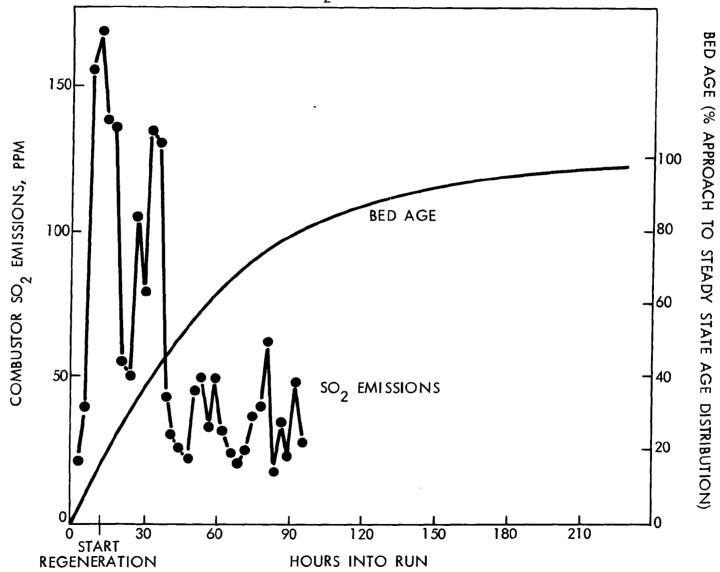




BED AGE (% APPROACH TO STEADY STATE AGE DISTRIBUTION)

FIGURE V-5

COMBUSTOR BED AGE AND SO<sub>2</sub> EMISSIONS VS. TIME (RUN 105)



Sorbent Elutriation Losses--

During the test series, the minimum Ca/S ratio fed to the combustor was set by sorbent attrition rather than by sorbent desulfurization activity. To maintain bed levels in the combustor and regenerator, bed lost through attrition was replaced by the incoming makeup sorbent. Sorbent losses represent elutriation of the fines formed by attrition.

Since sorbent elutriation was the factor limiting the Ca/S ratio, it is important to compare elutriation losses for the once through process and the regenerative process to see the effect regeneration has on attrition and subsequent elutriation. Sorbent elutriation losses for the once through process (miniplant combustor only) for both Grove limestone and Pfizer dolomite were presented in a previous report (2). Sorbent losses for the regeneration test series are presented in Table V-3. Losses for the once through process using both uncalcined and precalcined limestone are included for comparison. The sorbent loss calculations are based on the calcium contents of the materials entering and leaving the system and are essentially calcium losses.

Combustor sorbent losses expressed as the Ca/S ratio needed to offset the loss, and as percent of fresh sorbent, are extremely high. However, combustor elutriation losses expressed as a percent of bed inventory for the regeneration runs do not differ greatly from the values for once through operation. With the combined combustor-regenerator unit, the regenerator off gas provides an additional source of sorbent elutriation. Regenerator sorbent losses expressed as a percent of bed inventory tend to be higher than percent of bed inventory combustor losses. A possible explanation for higher regenerator elutriation losses is that the regenerated sorbent (CaO) is less attrition resistant than sulfated sorbent (CaSO4). The sorbent is also subject to thermal shock as it enters the regenerator. This may also contribute to higher attrition rates.

Combined losses from both the combustor and regenerator reflect the fact that the miniplant, under the conditions of the regeneration test series, was operating in the range of the minimum Ca/S ratio needed to maintain bed levels. In a number of the regenerative cases, elutriation losses exceeded the feed rate of fresh sorbent. Regeneration therefore can reduce the Ca/S ratio needed to meet EPA emission standards but not beyond the point were elutriation losses are larger than the makeup sorbent ratio. To specify the minimum Ca/S ratio necessary to provide 90% or less SO2 removal in the miniplant, a high sulfur coal would have to be burnt so that bed activity, rather than attrition, would be the limiting factor at those lower percentage SO2 removals.

SO<sub>2</sub> Content in Regenerator Off Gas--

Miniplant regenerator  $SO_2$  emissions are not limited by thermodynamics. For the operating conditions used in the test series, thermodynamics predicts an equilibrium  $SO_2$  concentration of  $\sim 3\%$ . In all runs,  $SO_2$  emissions were much lower than the equilibrium  $SO_2$  concentration. Average  $SO_2$  emissions ranged from 0.16 to 0.51%, equivalent to 6 to 16% of the equilibrium concentration (see Table V-4).

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TABLE V-3. SORBENT ELUTRIATION LOSSES

	Superficial		Com	Combustor Losses			Reg.	Reg. Combined Losses		
Sorbent Run No.	Gas Velocity m/s	Feed Rate Makeup Ca/S	Ca/S Loss Equivalent	% Fresh Feed	Vol. % Bed/Hr	% Total Feed	Losses Vol. % Bed/Hr	Ca/S Loss Equivalent	% Fresh Feed	Vol. % Bed/Hr
GL Uncalcined(	1) 1.4-2.2	1.5-2.8	0.2	12	1,1	12		0.2	12	1,1
GL Precalcined	(1) 1.6-2.5	2.5-4.0	0.2	8	8.0	8		0.2	8	0.8
GL 102	1.43	1,5	2.4	160	3.2	6	1.6	2,8	185	2.8
GL 103.0	1.52	1,35	0.80	58	1.3	11	6.2	1,5	113	2.1
GL 103.1	1.52	0.68	0.58	86	0.9	9	1.6	0.78	114	1.1
GL 103.2	1.59	0.68	0.85	126	2.1	23	4.3	1.4	200	2.6
GL 103.3	1,62	0.93	0.45	48	1.0	11	1.6	0.63	68	1.1
GL 105	1.46	1.29	0.55	43	0.9	11	0.7	0.67	51	0.8

GL = Grove Limestone

<sup>(1)</sup> Once-through operation.

TABLE V-4. COMPOSITION OF REGENERATOR OFF GAS

Run No.	Avg. SO2 Emissions (%)	Equilibrium SO <sub>2</sub> Conc. @ Conditions (%)	SO <sub>2</sub> Emissions (% of Equilibrium Conc.)
102	0.32	3.4	9
103.0	0.40	3.0	13
103.1	0.46	3.0	15
103.2	0.25	3.1	8
103.3	0.16	2.6	6
105	0.51	3.2	16
105 Peak	1.0	3,5	29

The low SO<sub>2</sub> concentrations are caused by limitations imposed by the size of the miniplant regenerator and combustor. SO<sub>2</sub> concentration in the regenerator off gas is determined by mass and energy balance constraints rather than chemical equilibria or reaction rates. Heat losses from the regenerator are high and require the addition of more hot gas than is needed to satisfy the requirements of the regeneration chemical reactions. Also, the amount of CaSO<sub>4</sub> (sulfated limestone) fed to the regenerator is set by the sulfur content of the coal and the size of the combustor. The regenerator is actually oversized for the CaSO<sub>4</sub> rates possible in the recent test series. Reducing the size of the regenerator or increasing the size of the combustor is not practical and coals with a higher sulfur content were not available for this program.

In addition to these constraints, the superficial gas velocity in the regenerator also had to be high enough to exceed the minimum fluidization velocity and promote vigorous solids mixing. Operating pressure had to be lowered from 900 kPa to 700 kPa in order to achieve the proper superficial velocity at the desired operating temperature without diluting the off gas even further.

Therefore, the SO<sub>2</sub> levels measured in this test program are set by the limitations imposed by the miniplant system and do not represent typical results from a larger facility which would not have the same limitations. The larger facility would be designed to allow the SO<sub>2</sub> levels to approach those predicted at chemical equilibrium.

The last portion of run 105 was a test to determine the maximum  $SO_2$  concentration attainable in the regenerator off gas and to compare this with thermodynamic predictions. The test was carried out by establishing oxidizing conditions in the regenerator to stop further sorbent regeneration and allow the sulfation level of the circulating sorbent to increase. It was intended to do this for a period of time until the combustor  $SO_2$  emissions increased to a fairly high level. This would indicate the sorbent was highly sulfated. When regeneration conditions were re-established, the initial  $SO_2$  concentration from the regenerator would approach an equilibrium concentration.

After 6 hours of oxidizing conditions,  $SO_2$  emissions from the combustor had not increased, indicating that before oxidizing conditions had been established, the sorbent had been regenerated to a high degree and was still very active. Due to time limitations, reducing conditions had to be established in the regenerator following the 6 hour oxidizing period but before combustor emissions had shown any increase. Regenerator and combustor SO<sub>2</sub> emissions following the start of reducing conditions are plotted as a function of time in Figure V-6. The cyclic nature of the regenerator SO2 emissions is due to sorbent transfer from the combustor which cools the regenerator bed and decreases the regeneration reaction. The initial increase in combustor SO2 emissions was probably caused by regenerator gas passing into the combustor since the regenerator operates at a slightly higher pressure to facilitate solids transfer. Regenerator SO2 emissions seemed to peak and level out at 1.0%. At the existing conditions, thermodynamics predicts an equilibrium concentration of 3.5%. Therefore the peak emission was only 29% of the equilibrium value (see Table V-4).

FIGURE V-6 REGENERATOR AND COMBUSTOR  $SO_2$  EMISSIONS RUN 105 1.0 0.8 180 REGENERATOR () REGENERATOR SO<sub>2</sub> EMISSIONS, % COMBUSTOR  $SO_2$  EMISSIONS, PPM 0.6 -60 165 0.4 40 COMBUSTOR 0.2 120 120 OXIDIZING 0 60 MINUTES INTO REDUCING CONDITIONS

..

Thermodynamics was obviously not the limiting factor in determining regenerator off gas  $SO_2$  concentrations. The Champion coal used in the test series had a low sulfur content (< 2%) which led to a low sulfur load on the unit, thereby limiting regenerator  $SO_2$  emissions due to mass and heat balance considerations as discussed previously.

#### Mass Balances --

Table V-5 summarizes steady state sulfur mass balances for the regenerator runs. Also included are total mass balances for each run. Total mass balance closure was  $99^+\%$  in all cases. Sulfur balances for three of the runs (102, 103.1, and 105) were very good, just slightly over 100%. Sulfur recovery for run 103.0 was 120%, indicating that the initial sulfur inventory (from the initial bed charge of sulfated sorbent) was being depleted by regeneration and was affecting the mass balance.

The last two segments of run 103 (103.2 and 103.3) had low sulfur recoveries, 74% and 48%, respectively. Since the total mass balances were 100%, the cause of the deficient sulfur balances is not obvious. A possible explanation is that the regeneration process was not keeping up with the sulfation process, increasing the sulfur inventory of the bed. Combustor bed probe samples exhibit a slight increase in sulfur level toward the end of the run, supporting this possibility. Another explanation would be a sudden change in sulfur load (coal sulfur content) which would not be accounted for in the sulfur balance. During run 103, an average coal sulfur content of 1.61% was used. In addition, two coal samples were taken during run 103.3 to determine the consistency of the coal sulfur content. The samples were taken 15 hours apart; the first had a sulfur content of 1.84%, the second dropped to 1.37%. If this drastic change in coal sulfur content was not just transient, the sulfur mass balance would be in error.

### Conclusions

Continuous operation of the miniplant provided a realistic way of measuring the potential benefits of a regenerative system compared to a once through system. Regeneration can reduce the makeup sorbent requirement needed to meet EPA emission standards by a factor of 3 to 4 but not below the makeup sorbent ratio needed to compensate for attrition losses and thus maintain bed inventory. Sorbent activity loss, if it occurs, was not apparent from the performance of the combined combustor-regenerator system on the basis of SO2 retention or combustor  $SO_2$  emissions. Results of this study may have been affected by the fact that all work fell in the high  $SO_2$  retention range.

If a higher sulfur coal (~4%) would have been available for the test series, perhaps more conclusive results would have been obtained. High sulfur coal would have allowed lower makeup Ca/S ratios to be tested without attrition being the limiting factor. The increased sulfur load would have also improved the  $SO_2$  concentration in the regenerator off gas, an important factor in downstream sulfur recovery.

TABLE V-5. MINIPLANT REGENERATOR MASS BALANCES

Run_No.	102	103.0	103.1	103.2	103.3	105
Sulfur Mass Balance		% of Sulfur Entering				
Sulfur Input Coal TOTAL	<u>100</u> 100	<u>100</u> 100	<u>100</u> 100	<u>100</u> 100	<u>100</u> 100	<u>100</u> 100
Sulfur Output						
Regenerator Off Gas	69.4	81.9	82,3	40.7	24.1	78.4
Combustor Off Gas	2.9	2.4	3.2	1.6	7	6.3
Combustor Overhead Solids						
2° Cyclone 3° Cyclone	22.8 2.9	19.5 3.2	13.6 2.4	19.6 3.1	10.9 2.3	13.5 1.6
Regenerator Overhead Solids	3.8	13.8	4.0	9.4	<u>3.9</u>	2.4
% Sulfur Recovery	101.7	120.2	105.9	74.3	47.7	101.4
Total Mass Balance	99.88%	99.86%	99.89%	99.79%	99.87%	100.6%

#### SECTION VI

### COMPREHENSIVE ANALYSIS OF EMISSIONS

#### LEVEL I AND II COMPREHENSIVE ANALYSIS TESTS

The U.S. Environmental Protection Agency has developed a phased approach to assess the environmental impact of solid, liquid and gaseous emissions from a process (26). This approach includes sampling and analysis for a wide range of organic and inorganic species. The first phase of the assessment (Level I) is intended to provide preliminary environmental assessment data and identify principal problem areas. The objective of the second phase (Level II) is to obtain more detailed and accurate data than is available from a Level I study. Level II studies are intended to identify and quantify specific compounds whose presence could be inferred from the results of Level I tests. Level II studies may also be initiated on the basis of results of the biotests employed as part of Level I (27). Exxon has participated in Level I and Level II testing programs using the miniplant facility. This work was done in cooperation with two other EPA Contracts, Battelle Columbus Laboratories and GCA/Technology Division.

#### Level I Emission Measurements

Level I comprehensive emission measurements from the PFBC miniplant unit without the regenerator unit operating were conducted in cooperation with Battelle Columbus Laboratories during run series 50 in early 1977 (14). These results will be reported in detail in a separate report prepared by Battelle and Exxon. In this test series, an eastern (Champion) coal was burned with Pfizer dolomite sorbent. In a subsequent test (run 69), made with Illinois coal and Pfizer dolomite, samples of solid materials were obtained and sent to Battelle for analysis. The samples were analyzed for inorganic elements using spark source mass spectroscopy (SSMS), the same method used on the solids samples from run series 50. The purpose of this supplementary test program was to provide a data base for Illinois coal use, to add to that already established for Champion coal. The data will be incorporated by Battelle in the interpretation of the Level I results. The results of the analyses are reported in Appendix I.

A second series of Level I tests was conducted during run 105 on the miniplant with the regenerator also in operation. Champion coal and Grove limestone were used in run 105 which was conducted in cooperation with GCA/ Technology Division. The principal effect of the GCA program was the collection of samples from the combustor flue gas and the regenerator flue gas using the Source Assessment Sampling System (SASS). Two SASS tests were conducted on both the combustor flue gas after expansion, diluted with air used for pressure control, and on the undiluted regenerator off gas. Two SASS train tests were also conducted on a filtered, undiluted slip stream of the combustor flue gas. Other gas samples were taken by GCA for analysis of SO3, HCl, nitrogen species and C1 to C7 hydrocarbons. Gas samples were also collected by Exxon personnel for analysis of the volatile sulfur compounds.

In support of the comprehensive analysis, solids samples and scrubber slurry samples were also collected by Exxon. The details of the sampling analysis effort were presented in a test plan prepared by GCA prior to sampling (15). GCA is responsible for analysis of the samples, and will report the results of the Level I tests from run 105.

### Level II Emission Measurements

An evaluation of the survey data of run 50 series by Battelle Columbus Laboratory (14) indicated the need for a Level II characterization of the emissions from the miniplant unit. A series of Level II tests was conducted in cooperation with GCA during run 107 on the miniplant without the regenerator unit operating. Operating conditions were similar to those used in the run 50 series. Champion coal was burned with Pfizer dolomite sorbent.

The gas and solid sampling techniques were the same as in run 105. A total of 5 SASS train tests were completed by GCA. Three SASS train tests were completed on the combustor flue gas after expansion, diluted with pressure control air. Particulates from the first SASS test on the diluted combustor flue gas were collected at about 100°C instead of 200°C, the normal sampling temperature, to check for organic deposition on the particulates at lower temperature. Two other SASS train tests were completed on a filtered undiluted slip stream of the combustor flue gas for analysis of gaseous emissions. Other gas samples were taken by GCA and Exxon personnel for analysis of SO3, C1 to C7 hydrocarbons and other sulfur compounds. Parallel solid samples were also collected by Exxon personnel. Details of the sampling analysis effort are included in a test plan prepared by GCA prior to sampling (16). The Level II analytical results of run 107 will be reported by GCA.

### PRESENCE OF Mg3 (CaSO4)4 IN 3RD CYCLONE FLYASH

As was reported by Argonne (17), the formation of Mg3Ca(SO4)4 or MgSO4. should be avoided when dolomite is used in PFBC if the sulfated solid waste is to be disposed of as landfill. In the presence of water,  $Mg_3Ca(SO_4)_4$  is unstable, decomposing to MgSO4 which is soluble in water, and CaSO4 which is relatively insoluble in water. The chemistry related to the formation of the binary salt Mg3Ca(SO4)4 is not known to date. However, it had been reported (17,18,19,20) that Mg3Ca(SO4)4 can form during the sulfation of dolomite under conditions similar to those in PFBC. Therefore, a short study was undertaken to determine if the binary salt is formed in the waste solids produced in the miniplant. Samples from run 67 of the bed overflow solids, particulate captured in the second and third stage cyclones and solids found in the turbine test section downstream of the third cyclone were analyzed by X-ray diffraction. In run 67, the flue gas contained about 700 ppm SO<sub>2</sub> and 3 to 5% O<sub>2</sub>. At the flue gas pressure, 9 atm, MgSO<sub>4</sub> was thermodynamically possible at temperatures below about 840°C. No evidence of the binary salt or MgSO4 was found in the second cyclone particulates even though the temperature was between 770 and 800°C. However, third cyclone material and turbine test section deposits did contain Mg3Ca(SO4)4 but no MgSO4. The temperature in the third cyclone was 700 to 820°C, the turbine test section 660 to 820°C. The absence of Mg3Ca(SO4)4 in the second cyclone material but its presence

in the third cyclone and turbine test section material could be due to a residence time effect. The residence time in the piping between the two cyclones is greater than the residence time between the combustor and the second cyclone. The temperature is also lower in the third cyclone and turbine section thus favoring the formation to a greater extent.

The extent of magnesium conversion in the third cyclone and turbine section solids was estimated by a sulfate balance to be from 30 to 100%. However, the 700 ppm  $SO_2$  level during run 67 was higher than the  $SO_2$  levels which will be experienced under the New Source Performance Standard (1.2 lb  $SO_2/10^6$  BTU or less, depending upon the percentage reduction required in a particular case). Furthermore, the residence time of the flyash in the piping from a PFBC combustor to an efficient third stage particulate removal device should be shorter than in the miniplant and the gas temperatures higher. Therefore, formation of soluble Mg3Ca( $SO_4$ )4 will be less under higher  $SO_2$  retention conditions, shorter residence times and higher temperatures between the combustor and the particulate removal devices. Additional tests should be conducted to determine the extent of the binary salt formation under more realistic conditions.

#### SECTION VII

#### BENCH UNIT STUDIES

Programs were carried out in both the bench combustor and regenerator sections. The combustor had been modified, as described in the previous report (1), to permit continuous solids feeding and removal. After an initial check out period, the combustor was used to evaluate three  $\mathrm{NO}_{\mathrm{X}}$  control methods, two stage combustion, NH3 injection and simulated flue gas recirculation. The regenerator was used in a series of tests using sulfated sorbent produced in the miniplant, studying the use of natural gas and coal to fuel the regeneration section. The following sections describe the results of the bench unit combustion and regeneration tests.

### COMBUSTION STUDIES

The bench combustion unit consists of a refractory lined combustor vessel which normally operates at temperatures of 840 to 950°C, pressures of five to eight atm, superficial velocities of 1 to 2 m/s and coal feed rates of 1 to  $12 \, \text{kg/hr}$ .

### Equipment

The unit was described in detail in previous reports (1,2,3). Additional modifications were made to permit the  $NO_X$  control studies to be carried out. A brief description of the combustor section and the recent modifications are described below.

### Combustor Vessel--

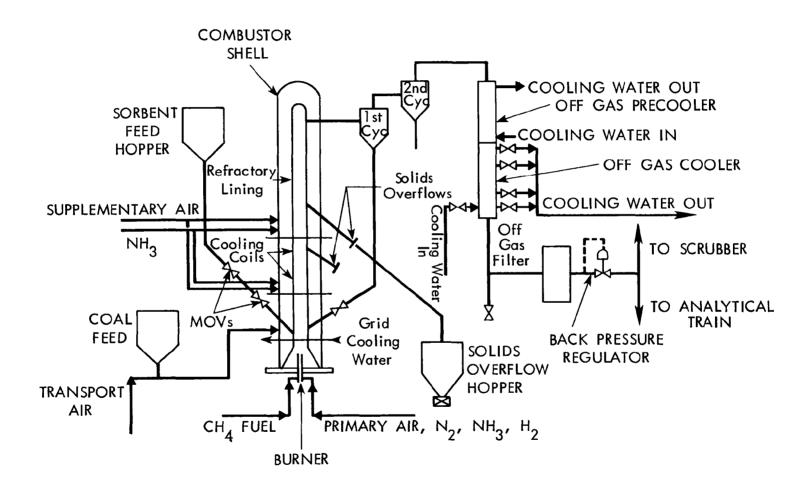
The combustor (shown in Figure VII-1) consists of four sections of 25 cm diameter standard wall carbon steel pipe, lined with Grefco 75-28 refractory, to an inside diameter of 11.4 cm. The height of the vessel above the fluidizing grid is about 4.9 m. Below the grid is a 61 cm burner section lined with Grefco Bubblite refractory. The preheat burner employs a mixture of natural gas and air to heat (and fluidize) the bed up to temperatures sufficiently high to ignite the coal.

Three sets of vertically mounted 316 SS water cooled coils are located inside the combustor to assist in temperature control. These coils remove 50-60% of the heat of combustion.

Two flanged inlet lines are welded into the final section above the grid at angles of 60° to the horizontal. These permit the charging of fresh sorbent and the return of the solids from the primary cyclone.

The second and third sections of the combustor have solids draw off lines inclined at  $60^\circ$  to the horizontal; these are used to control bed height and volume. Solids drawn off through them are discharged to lock hoppers. The lower draw off line is located 1.09 m above the grid and provides a bed volume of 0.0111 m³. It was used for all tests described in this report. The higher line is located 1.85 m above the grid and provides a bed volume of 0.0189 m³.

FIGURE VII-1
SCHEMATIC OF BATCH COMBUSTION UNIT



The use of these draw off facilities and variations in operating parameters permit operating over a wide range of gas residence times.

Ancillary Systems--

Coal is fed to the combustor by a means of a pneumatic transport coal injector capable of feeding up to 14 kg/hr for periods of up to 8 hours before refilling. Sorbent is charged through a separate system. The system consists of two cycling valves which trap a small amount of sorbent between them and discharge the contained sorbent into the combustor intermittently. Flue gas leaving the combustor passes through two cyclones, a cooler and filter before expansion across a control valve.

Combustor System Modifications --

The combustor was modified to permit the study of the effects of staged combustion, ammonia injection and simulated flue gas recirculation on  $NO_X$  emission levels (see Figure VII-1). Modifications were made to introduce supplementary combustion air (for two stage combustion) and ammonia directly into the fluidized bed, or above the bed, during combustor operation. Nitrogen (for simulated flue gas recirculation) and ammonia could also be injected into the inlet air, underneath the grid plate.

Probes used to inject supplementary air or ammonia into the combustor were constructed of 3/8 inch 316 SS tubing sealed off at one end. The probes extended horizontally across the diameter of the combustion zone to the far wall, and contain three or four holes drilled horizontally. The holes were sized and located so that high velocity gas streams could be obtained and directed to impinge upon the combustor walls. This provided adequate mixing of the incoming gases with the contents of the fluidized bed. Injection locations were varied, depending upon the test conditions; they are summarized in Table VII-1.

The reasons for the various probe locations are discussed in detail in those parts of the text relating to the specific program.

The NH $_3$  addition system consisted of a cylinder of NH $_3$  maintained at about 40°C in a heated water bath. NH $_3$  gas was transferred through heated lines to prevent condensation and metered through a rotameter. N $_2$  and H $_2$  can also be metered and mixed with NH $_3$ .

Other than for these modifications, the bench unit combustor remains essentially as described in the previous report (1).

### Two Stage Combustion

The program to study the effects of staged combustion upon  $NO_X$  emission was developed as a 2 X 3 X 2 factorial design and included the effects of overall excess air, primary air to fuel ratio (before injection of secondary air), and bed temperature (see Table VII-2).

Initially, it was intended to conduct the designed study at a combustor pressure of 8 atmospheres, but operational difficulties prevented this. At 8 atmospheres, superficial gas velocities in the primary combustion (reducing)

## TABLE VII-1. LOCATION OF GAS INJECTION POINTS BENCH UNIT $NO_X$ CONTROL STUDIES

		Injector	Injector Location				
	Program	No	Distance Above Grid		Top of Expanded Bed		
			(cm)		cm)		
	Staged Combustion	1	200	78	above		
74	_	2	15		below		
		3	33	89	below		
	Ammonia Injection	1	168	46	above		
	•	2	Ammonia mixed wi	th combustion air - no			
		3	290		above		
	Simulated Flue Gas Recirculation	-	N <sub>2</sub> added to	combustion air - no in	jectors used		

### TABLE VII-2. DESIGNED EXPERIMENT FOR STUDY OF STAGED COMBUSTION IN BENCH UNIT

			[	Bench Unit Comb	ustor Run	Number		
	Primary Air (% of Stoich) →	Unstaged Combustion		0	Staged Combustio			
	·			<del> </del>			75%	
	Average Bed Temp. (°C) →	<u>840</u>	930	840	<u>930</u>	840	930_	
175	Overall Percent Excess Air							
	15	21.1	20.1	21.2	20.2	21.3	20.3	
	30	17A1	22.1	17A2	22.2	17 <b>A</b> 3	22.3	

zone were too low for good fluidization. This resulted in repeated episodes of bed agglomeration. As a consequence, all of the runs in the designed experiment were made at 5 atmospheres. Champion coal (-16 mesh, 2.19% sulfur) was used in all runs, and the Ca/S ratio was set at 3.0.

Originally, it was planned to use the entire bed as the reducing zone and to inject the supplementary air above the bed to oxidize the CO formed. In the initial runs at 8 atm, the supplementary air probe was located 200 cm above the grid, or about 45 cm above the top of the bed. Pfizer dolomite was selected as the sorbent. This scheme was not practical. The dolomite attrited rapidly and was elutriated from the bed early in the run. This resulted in smaller (and shorter) beds, and greater distances between the top of the bed and the point of air injection. As a consequence, the off gas temperature at the point of air injection was on the order of 600-650°C rather than at the bed temperature of 870-930°C. This resulted in incomplete burnout of CO. The ignition point of CO in air at atmospheric pressure is about 610°C. Occasionally, there were brief temperature rises above the air injection point indicating that combustion of CO was taking place sporadically. In light of the above, it was decided to place the probe in the bed at 15 cm above the grid and to use Grove limestone (-8+25 mesh) as the sorbent to reduce attrition losses.

These changes provided adequate CO burnout, but resulted in a limited and unstable reducing zone. During several runs at 8 atm, fusion of the combustor bed occurred as a consequence of erratic coal feed rates and poor mixing in the bed, particularly at low primary air flow rates. Partial disassembly of the combustor revealed that the refractory lining immediately above the grid had been eroded. The diameter was 14 cm to 15 cm rather than the original diameter of 11.5 cm. This resulted in low gas velocities in this area and inadequate mixing in the bed. As a consequence, the low velocities and poor solids mixing were probably responsible for fusion of the bed. Another possibility was that CaS was formed and, upon occasion, was carried into the oxidizing zone. The oxidation of CaS to CaSO4 is highly exothermic, and would result in excessive bed temperatures and fusion of the bed material.

The refractory lining was repaired and the diameter of the combustor restored to the original 11.5 cm. At this point, the combustor pressure was reduced from 8 atmospheres to 5 atmospheres for subsequent runs. These changes resulted in higher gas velocities and improved mixing of the bed solids. The supplementary air probe was relocated to a height of 33 cm above the grid in order to provide a larger reducing zone than was obtainable when the probe was located 15 cm above the grid.

The results of the study were evaluated by Analysis of Variance (ANOVA) techniques for both main effects and interactions. If an effect could not be demonstrated at the 90% confidence level (or higher), it was assumed not to exist.

Emission levels were characterized as "Emission Indices" in terms of pounds of the components in the flue gas per MBTU of fuel supplied. This approach is preferable to using the component concentrations in the flue gas, as the latter is affected by excess air levels.

Detailed results of the test program are given in Appendix P-2.

Effect of Operating Parameters Upon NO<sub>X</sub> Emissions--

Data obtained at 8 atm pressure—The results of the present study were compared with those from earlier work to measure  $NO_X$  emissions during unstaged combustion. The earlier results were reported in a previous report (2). The results of the earlier work, at excess air levels of 0% to 40%, were combined with results of four new unstaged runs (runs 12.1, 13.1, 14.1, 17.1) made at 8 atm. A "t Test" with both paired and unpaired comparisons was used to determine the effect of staging. The staged tests (runs 10.1, 11.1, 11.2, 12.2, 13.2, 14.2, 17.2) were carried out at conditions similar to those in Table VII-2. The test indicated that staged combustion reduces  $NO_X$  emissions about 30% below those resulting from unstaged combustion (see Table VII-3). The "t Test" indicated that the confidence level was greater than 90% when the unpaired comparison was made and over 98% when the paired comparison was made.

Data obtained at 5 atm pressure—The data obtained at 5 atmospheres—through the completion of the test matrix in Table VII-2— are summarized in Table VII-4. Staged combustion reduced the emission index (lbs  $NO_2/MBTU$ ) from 0.272 (lbs/MBTU) (without staging) to 0.245 (lbs/MBTU) with the primary air at 90% of the stoichiometric amount required to burn the coal. This is a reduction of only 10%. A reduction of 20% to 0.220 (lbs/MBTU) was achieved by reducing the primary air to 75% of stoichiometric. The effect was observed at the 90% confidence level. These reductions are lower than those observed at 8 atm pressure.

The staged and unstaged data obtained in this series were also analyzed to determine the effects of variables other than staging on  $NO_X$  emissions. For all runs, staged and unstaged, increasing the overall excess air levels from 15% to 30% increased the  $NO_X$  emissions from 0.222 (lbs/MBTU) to 0.270 (lbs/MBTU), an increase of 22%. Increasing the combustor temperature from 843°C to 927°C increased emission indices from 0.218 (lb/MBTU) to 0.273 (lb/MBTU), a 25% increase with increasing temperature.

These results are generally consistent with previous findings from the bench and miniplant units (1,2,3).

No statistically significant interactions were found.

Effects of Operating Parameters on SO<sub>2</sub> Emissions--

The  $SO_2$  emissions observed during the staged combustion matrix at 5 atm, are shown in Table VII-5. Staged combustion appears to increase the  $SO_2$  emission levels. The emission index increased from 1.66 for all unstaged runs to 1.90 (lb/MBTU) for staged runs, an increase of 16%. Effects of temperature and excess air on  $SO_2$  emission levels were also found.

High combustion temperatures, regardless of staging or excess air levels, decreased SO<sub>2</sub> emission levels. Increasing the combustor's operating temperature from 840°C to 930°C reduced SO<sub>2</sub> emission by 21%.

TABLE VII-3. EFFECTS OF STAGED COMBUSTION ON NO, LEVELS AT 8 ATMOSPHERES

	Run No.	Staged	NO <sub>x</sub> (1b/M BTU) Unstaged(1)	Δ	Overall Excess Air (EA) (%)
	12,2	0.14	0.192	0.052	7.8
	13.2	0.19	0.235	0.045	15.7
	14.2	0.19	0.256	0.066	19.6
	17.2	0.21	0.317	0.107	30.6
$\overline{\mathbf{X}}$	~-	0.182	0.250	0.067	
$s^2$	40 00	0.030	0.052	0.028	

<sup>(1)</sup> Calculated from regression equation [NO<sub>X</sub> (1b/M BTU) = 0.00548 (% EA) + 0.149], for all unstaged combustor runs.

$$\frac{\overline{X}_{c}}{\overline{X}_{o}} = \frac{0.250}{0.182} = 1.374$$
 or 37% increase in NO<sub>X</sub> levels with unstaged combustion over the NO<sub>X</sub> levels with staged combustion.

$$1 - \frac{\overline{X}_0}{\overline{X}_C} = 1 - 0.728 = 0.272 \text{ or } 27\% \text{ reduction in NO}_X \text{ levels}$$
 when using staged combustion.

TABLE VII-4. EFFECTS OF STAGED COMBUSTION ON  $NO_{\mathbf{X}}$  EMISSIONS AT 5 ATMOSPHERES

			Em i	ssion Indi	ces for NO	(1b NO <sub>2</sub> /	M BTU)	
		Uns	Unstaged		Staged Co	ombustion		
	Primary Air (% of Stoich) →		ustion	9	0%		5%	Row
	Average Bed Temp. (°C) →	840	930	840	930	840	930	Means
	Overall Percent Excess Air							
	15	0.20	0.32	0.22	0.23	0.15	0.21	0.222
	30	0.26	0.31	0.23	0.30	0.25	0.27	0.270
179	Sub Column Means =	0.230	0.315	0.225	0.265	0.200	0.240	
	Column Means =	0.2	272	0.2	245	0.2	220	
	Group Means							
	Group 1 (840°C) = 0.218				Sign Effect	ificant Ef Co	fects onf, Limits	
	Group 2 (930°C) = 0.273				Temperatur	e	95%	
	0 14 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0 0				Staging		90%	
	Grand Mean = 0.246				Excess Air		95%	

# TABLE VII-5. EFFECT OF THE BENCH UNIT'S OPERATING PARAMETERS UPON SO<sub>2</sub> EMISSIONS AT 5 ATMOSPHERES

	Emission Indices for SO2 (1bs/M BTU)									
	Unstaged Staged Combustion									
Primary Air (% of Stoich) →	Comb	ustion	9	0%	7	5%	Row			
Average Bed Temp. (°C) →	840	930	840	930	840	930	<u>Means</u>			
Overall Percent Excess Air										
15	2.25	2.02	2.23	1.79	1.56	1.78	1.94			
30	1.48	0.890	2.16	1.63	2.53	1.56	1.71			
Sub Column Means =	1.86	1.45	2.20	1.71	2.04	1.67				
Column Means =	1.	.66	1.	95	1.	86				
Group Means					ignifican	t Effects				
Curry 1 (0409C) - 2 04				Eff	ect	***	Conf. Limits			
Group 1 (840°C) = 2.04			Temperat	ure			95			
Group 2 (930°C) = 1.61			Excess A	ir-Temper	ature Int	eraction	90			
Grand Mean = 1.82			Excess A	ir-Stagin	g Interac	tion	95			

Unstaged combustion at high excess air levels (30%) produced the lowest SO<sub>2</sub> emission levels. The mean emission level under these conditions was 1.18 (1b/MBTU) (the average of 1.48 and 0.890), while the mean emission level for all other twelve conditions was 1.97 (1b/MBTU), a 40% difference in emission levels. Combustion under the condition of high excess air levels (30%) and high temperature (930°C), regardless of staging, materially decreased SO<sub>2</sub> levels. The mean SO<sub>2</sub> emission level under these conditions was 1.36 (1b/MBTU), while the mean for all other eleven conditions was 1.82 (1b/MBTU) or a reduction in emission levels of 25%.

The effects of the combustor operating parameters upon  $SO_2$  emission levels may be interpreted in light of the following equations.

$$CaCO_3 \longrightarrow CaO + CO_2 \tag{1}$$

$$SO_2 + 1/2O_2 + CaO \longrightarrow CaSO_4$$
 (2)

The detrimental effect of staging is probably due to the need to have a sufficient oxygen level to promote equation 2. Since oxygen is depleted in the reducing zone, equation 2 is hindered there. Equation 2 will occur in the oxidizing zone, but because the gas residence time in the oxidizing zone is less in the staged configuration, equation 2 is further hindered.

The effects of the higher combustor operating temperatures upon  $S0_2$  emission levels stems from the fact that the sorbent is more completely calcined (Eq. 1) at  $930\,^\circ\text{C}$  than it is at  $840\,^\circ\text{C}$ . The higher the degree of calcination, the greater the degree of porosity and the more reactive the sorbent.

The apparent effect of excess air in the unstaged runs is not understood. Although the increase in excess air could promote equation 2, it is believed that at excess levels of 15% or so, oxygen is present in sufficient excess to have no further effect.

Overall, the SO<sub>2</sub> emissions measured in this test series were fairly high, compared to results obtained in the miniplant. The difference is due to a much lower gas phase residence time in the bench studies ( $\sim$ 0.5 s) compared to normal miniplant operations (2 to 3 s).

Effects of Operating Parameters Upon CO Emissions--

Staged combustion increases CO emission moderately, and then only at low levels of primary air (see Table VII-6). The mean value of the CO emission index of 0.292 (lbs/MBTU) for a primary air rate of 90% of stoichiometric is not significantly different from the mean value of 0.277 (lbs/MBTU) for unstaged combustion. However, the mean value of the CO emission indices of 0.335 (lbs/MBTU) for a primary air rate of 75% of stoichiometric is 20% higher, which is significantly different from the value of unstaged combustion.

CO emission indices are markedly increased by low combustion temperatures an effect seen in previous studies (2). Decreasing the combustor's operating temperature from  $930\,^{\circ}\text{C}$  to  $840\,^{\circ}\text{C}$  increased the mean CO emission indices from

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### TABLE VII-6. CO EMISSIONS FOR STAGED AND UNSTAGED COMBUSTION AT 5 ATMOSPHERES

			ission Indi	ices for CC	) (1bs/M B	<b>Γ</b> U)	
Primary Air (% of Stoich) →	Unstaged Combustion		90%		7	5%	Row
Average Bed Temp. (°C) →	843	927	843	927	843	927	Means
Percent Excess Air							
15	0.35	0.25	0.29	0.22	0.34	0.29	0.290
30	0.33	0.18	0.38	0.28	0.44	0.27	0.313
Sub Column Means =	0.340	0.215	0.335	0.250	0.390	0.280	
Column Means =	0.	277	0.	292	0.	335	
Group Means				S	ignificant	Effect_	
Group 1 (843°C) = 0.355				Ef	fect		Conf. Limi
Group 2 (927°C) = 0.248			Tempera	ture			99%
Group 2 (927°C) = 0.248			Staging				95%
Grand Mean = 0.302			Tempera	ture, Exce	ss Air Int	eraction	95%
			Staging	. Excess A	ir Interac	tion	95%

0.248 (lbs/MBTU) to 0.355 (lbs/MBTU), an increase of 43%. Over the range of 15 to 30% excess air, the level of excess air has no significant effect upon the CO emission indices. This finding was unexpected.

High excess air levels (30%) and low combustion temperatures ( $840^{\circ}$ C) increase CO emission levels. At these operating conditions the mean CO emission index was 0.38 (lb/MBTU) while the mean value for other operation conditions was 0.30 (lb/MBTU), or an increase of 27%. This may be the result of the combined effect of higher levels of CO formation at the lower temperature and the introduction of large amounts of cold secondary air into the bed, which has the effect of causing local cooling, thereby preventing CO burnout.

The combination of high excess air levels (30%) and low primary air rates (75%) also increases CO emission levels. This effect may be due to the higher levels of CO formed in the bed at low primary air rates and the introduction of large volumes of cold secondary air, which would retard CO burnout.

### Conclusions --

Two stage combustion was shown to reduce  $NO_X$  emissions by about 20%. However,  $SO_2$  and CO emissions were both increased about 20% by staging. Increasing the gas phase residence time in the oxidizing section of the combustor slightly should offset the increased  $SO_2$  and CO emissions. Two stage combustion could also create a boiler tube materials problem if cooling coils were subjected to alternate low and high oxygen concentrations. This was not addressed in this study but must be at some point, if two stage combustion is to be considered further.

### NH<sub>3</sub> Injection

Exxon Research and Engineering Company has developed a process based on the selective, homogeneous, gas phase reduction of NO by NH3. The amount of NH3 needed is comparable to the amount of NO reduced. Temperature has an effect on the effectiveness of the reaction. When the temperature is too low, NH3 and NO tend to remain unreacted, when the temperature is too high, NH3 tends to form additional NO. Thus, it is possible to achieve an efficient reduction of NO with little NH3 remaining, but only within a narrow temperature range. The NO $_{\rm X}$  destruction and production reactions are:

$$4N0 + 4NH_3 + O_2 \rightarrow 4N_2 + 6H_2O \tag{1}$$

$$4NH_3 + 50_2 \rightarrow 4NO + 6H_2O$$
 (2)

Reaction (1) dominates at temperatures around 950°C whereas reaction (2) dominates above 1100°C.

R. K. Lyon (21) proposed a free radical chain mechanism for the reaction of  $\rm NH_3$ ,  $\rm O_2$  and  $\rm NO$  to reduce  $\rm NO_x$ :

$$NH_2 + NO \rightarrow N_2 + H + OH$$
 (1)

$$NH_2 + NO \rightarrow N_2 + H_2O$$
 (2)

$$H + O_2 \rightarrow OH + O$$
 (3)

$$0 + NH_3 \rightarrow OH + NH_2 \tag{4}$$

$$OH + NH_3 \rightarrow H_2O + NH_2$$
 (5)

$$H + NH_3 \rightarrow H_2 + NH_2 \tag{6}$$

The addition of a third component such as hydrogen, carbon monoxide or various hydrocarbons which form reactive radical intermediates, reduces the optimal temperature for the reaction (2). Hydrogen is preferred since it is itself not an air pollutant. Although hydrogen reduces the optimal temperature for the reaction, it has the disadvantage of decreasing the selectivity with which NH<sub>3</sub> reduces the NO. Hence, if too much hydrogen is added, NH<sub>3</sub> may react to form NO rather than to reduce NO. The preferred levels of the gases based on tests at low pressure are:

$$NH_3$$
  $NH_3/NO = 0.5-3.0$   $H_2$   $H_2/NH_3 < 3$   $0.1-20.0$  volume %

At  $\rm H_2/NH_3$  ratios around 2/1, the  $\rm NO_X$  reduction reaction can proceed at 700°C. By selecting the proper  $\rm H_2/NH_3$  ratio, the reaction can be accomplished at any temperature between 700°C and 950°C (22).

Laboratory data has shown that reductions in  $NO_X$  emissions up to 70% are possible. The process has been commercially demonstrated in gas and oil-fired steam boilers and process furnaces. Until this work, no tests have been made on a pressurized fluidized bed coal combustion system.

Experimental Conditions--

A series of eight runs covering 27 conditions were made to test the effect of NH3 injection on NO $_{\rm X}$  emissions. The NH3/NO $_{\rm X}$  and H $_{\rm 2}$ /NH $_{\rm 3}$  ratios were varied and 3 injection levels were tested. Table VII-7 lists the runs which were made. Figure VII-2 shows the location of the 3 injection levels and Table VII-8 indicates the port (and height above the grid) which was used at each level.

The injection level, or probe location, was set prior to the start of a run. In each experimental day, a series of steady state periods was obtained. Each series began with a steady state period at baseline conditions (no NH3 injection) and usually ended at baseline conditions to see if the emissions levels returned to the same base level.

Constant operating conditions were as follows:

Champion Coal (-16 mesh, 2.19% S) Grove Limestone (-8 + 25 mesh) Ca/S mole ratio: 3.0

TABLE VII-7. AMMONIA INJECTION RUN CONDITIONS

		NH3 Injecti	lon
Run Number	Location(1)	Approx. Temp. (°C)	Conditions NH3/NO <sub>X</sub> H <sub>2</sub> /NH3
1.1 1.2 1.4	L1	816	No Injection 0.88 0 0.88 2.00
3.1 3.2	Ll	816	No Injection 2.27 1.48
4.1 4.2 4.3	L1	816	No Injection 2.00 1.00 No Injection
5.1 5.2 5.3 5.4 5.5 5.6 5.7	L2	954	No Injection 2.18 2.94 1.27 2.50 No Injection 3.41 0 8.05 0 No Injection
6.1 6.2 6.3	L3	704	No Injection 0.82 0 No Injection
7.1 7.2 7.3 7.4	L3	704	No Injection 1.36 0 2.25 0 No Injection
8.1 8.2 8.3 8.4 8.5	L1	816	No Injection 1.24 0 2.29 0 1.39 1.46 No Injection

<sup>(1)</sup> See Figure VII-2 and Table VII-8 for description of each location.

FIGURE VII-2

BENCH COMBUSTOR SHOWING LOCATIONS
FOR AMMONIA INJECTION

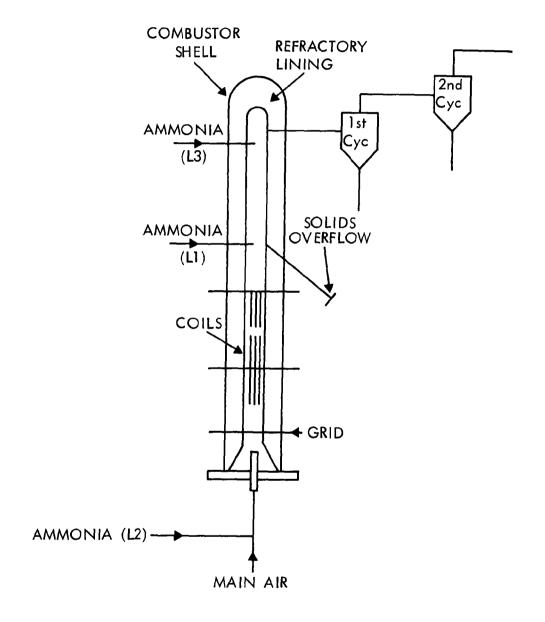


TABLE VII-8. AMMONIA INJECTION LOCATION

Probe Location	Description	Height Above Grid (cm)
L3	Inject ammonia near top of column	290
L1	Inject ammonia above bed (at overflow port)	168
L2	Mix ammonia with main air stream	Below Grid

Pressure: 657 to 758 kPaa Bed Temperature: 900°C

Excess Air: 15%

Results and Discussions --

The operating conditions for each of the runs is summarized in Appendix Table P-3. In all, 27 steady state periods were run which included 13 periods at baseline conditions (with no ammonia injection). Figure VII-3 shows the emission indices for NO $_{\rm X}$ , SO $_{\rm 2}$  and CO, respectively. These bar graphs show the 95% confidence interval for each steady state level. Standard deviations for NO $_{\rm X}$ , SO $_{\rm 2}$  and CO emission index, used to draw the confidence interval (or 2 standard deviation bars), were calculated from the 13 base level steady states.

 $NO_X$  Emissions—In the 5 and 7 run series a statistically significant change in the  $NO_X$  emissions index occurred. Within each of the run series 1, 3, 4, 6, and 8 the confidence intervals overlap, suggesting that a real change in the level may not have occurred. Significant changes in the  $NO_X$  emissions from the baseline level occurred in runs 5.5, 5.6, 7.2, and 7.3.

Table VII-9 summarizes the results. The NO $_{\rm X}$  emission index is a strong function of the NH $_3$  injection location and NH $_3$ /NO $_{\rm X}$  ratio. With injection into the main air stream (run 5) the NO $_{\rm X}$  level increased 50%. With injection above the bed (runs 1, 3, 4, and 8) the NO $_{\rm X}$  level remained unchanged. With the injection near the top (runs 6 and 7), the NO $_{\rm X}$  level decreased 30 to 50%. Table VII-10 shows that with injection into the main air (run 5) the NO $_{\rm X}$  level increased in proportion to the amount of NH $_3$  injected (NH $_3$ /NO $_{\rm X}$  ratio) and with injection near the top (run 7) the NO $_{\rm X}$  level decreased in proportion to the amount of NH $_3$  injected.

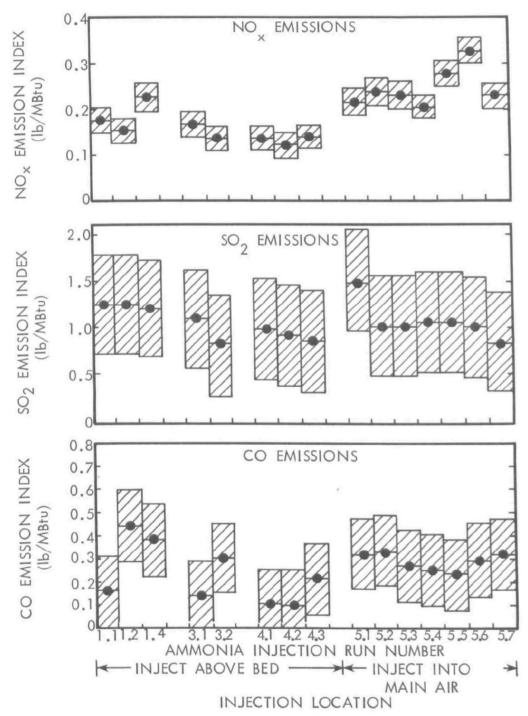
Because changing the injection location changes the injection temperature, the effect of injection location on the NO $_{\rm X}$  emissions may be simply a temperature effect. If this is true, then it would appear that in this combustion system, the NH $_3$ -NO reaction is faster at 700°C, the NH $_3$ -O $_2$  reaction is faster at 950°C, and at 820°C the rates of the two reactions are equal. However, oxygen partial pressures and mixing may be different, as well as temperature, at each injection location. High oxygen partial pressures, which probably exist immediately above the grid, favor NO $_{\rm X}$  production. This may explain NO $_{\rm X}$  production at injection location L2. Slugging may cause a high oxygen level above the bed (L1) also.

Table VII-ll compares the results of this work to the laboratory data obtained in another study (21). The optimal temperature for the  ${\rm NO_X}$  reduction reaction without hydrogen addition appears to have shifted down from 950°C in the earlier laboratory study to 700°C in this work with the bench combustor. This shift is possibly due, in part, to the presence of CO or unburned hydrocarbons in the flue gas. CO and hydrocarbons may act in place of hydrogen as a source of radicals for the  ${\rm NH_3-NO}$  reaction.

Effect on SO<sub>2</sub> and CO Emissions--Examination of the bar graphs for SO<sub>2</sub> and CO emission indices (Figure VII-3) reveals that no significant change occurred due to ammonia injection location or  $\rm NH_3/NO_\chi$  ratio.

FIGURE VII-3

EMISSION INDICES FOR AMMONIA INJECTION PROGRAM SHOWING 95% CONFIDENCE INTERVALS



### FIGURE VII-3 (CONT'D)

### EMISSION INDICES FOR AMMONIA INJECTION PROGRAM SHOWING 95% CONFIDENCE INTERVALS

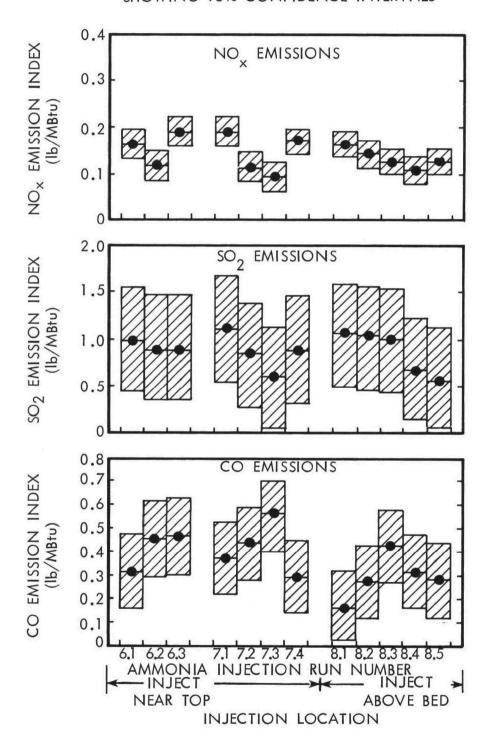


TABLE VII-9. RESULTS OF PROGRAM TO REDUCE NO BY AMMONIA INJECTION

Run Series No.	Injection Level	Temperature at Injection Point (°C)	Significant Change in NO <sub>X</sub> Emission Index?	Increased/Decreased NO <sub>X</sub> Levels	Maximum Change (%)
1	Ll	816	No		
3	L1	816	No		
4	L1	816	No		
5	L2	954	Yes	Increased	50
6	L3	704	Yes	Decreased	30
7	L3	704	Yes	Decreased	50
8	L1	816	No		

TABLE VII-10. RESULTS OF AMMONIA INJECTION RUNS 5 AND 7

			NO <sub>X</sub> Emissions					
Run No.	NH3 Inj Condi NH3/NO <sub>X</sub>	ection tions H2/NH3	Base Level	b NO2/MBTU) Level with NH3 Injection	Change From Base Level (%)	Significant Change ?		
	Base ( Injec	No NH <sub>3</sub>	0.21					
5.2	2.18	2.94		0.24	+14	No		
5.3	1.27	2.50		0.23	+10	No		
5.5	3.41	0.0		0.27	+29	Yes		
5.6	8.05	0.0		0.32	+52	Yes		
	Base ( Injec	No NH <sub>3</sub> tion)	0.18					
7.2	1.36	0.0		0.12	-33	Yes		
7.3	2.25	0.0		0.09	-50	Yes		

# TABLE VII-11. COMPARISON OF THE RESULTS OF THIS WORK AND AN EARLIER WORK (21) ON AMMONIA INJECTION TO REDUCE NO

			perature (°C)	
Add NH3	ed H2	Earlier Work (21) (Lab Data)	This Work (PFBCC - Bench Combustor)	Change in NO <sub>X</sub> (Increase/Decrease)
Yes	No	1093		Increase †
Yes	No		954	Increase ↑
Yes	No		816	No Change
Yes	No	949	704	Decrease ↓
Yes	Yes	871		Decrease ↓
Yes	Yes	704		Decrease ↓

Effect of Excess Air Level on  $NO_X$ --Figure VII-4 shows that the excess air level varied only slightly from a mean of 17.2%. The two steady states for which the  $NO_X$  emissions increased significantly (run 5) and the two steady states for which the  $NO_X$  emissions decreased significantly (run 7) are shown in addition to the 13 base level steady state periods. Figure VII-4 shows that the change in  $NO_X$  emissions due to NH3 injection is not due to a change in the excess air levels. The uncontrolled  $NO_X$  emissions in this study are generally lower than in earlier bench combustor studies (2). This is presently unexplained.

Response Time--Figure VII-5 shows graphically how response times were determined for run 5. The response time is the time required for the system to reach a new steady state  $NO_X$  level once a change in the ammonia injection conditions (NH3/NO<sub>X</sub> and H<sub>2</sub>/NH<sub>3</sub>) is made. Table VII-12 shows the response times for run 5. The system response is very rapid, averaging 10 minutes.

### Ammonia Material Balance--

NH3 material balances were attempted by analyzing all solid and gaseous product streams. The balances were very low and variable, ranging from 14 to 69%. Analysis of solids collected by the cyclones and by the final filter indicated that ammonium salts were forming and precipitating on the collected solids. The compounds were most likely NH4HSO3 and/or NH4HSO4. The mass of ammonia which would have to have been lost to account for the low mass balances was very little, ranging from 4 to 130 gm. Therefore, the poor balances could have been caused by the formation and deposition of ammonium salts, possibly in the flue gas ducting. Other sampling and analytical problems may have also occurred which contributed to the low balances.

#### Conclusions --

NH<sub>3</sub> injection at temperatures around 700°C in PFBC flue gas can reduce NO<sub>X</sub> emissions 30 to 50%. The temperature level is lower than expected and further work may be needed to determine if it is the true optimum. If such a temperature is required, NH<sub>3</sub> could be injected ahead of the gas turbine. The desired temperature would occur within the turbine itself, where the NO<sub>X</sub> removal reactions would take place.

### Simulated Flue Gas Recirculation

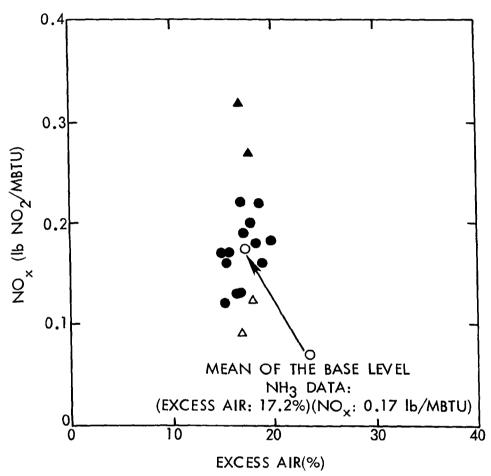
In the simulated flue gas recirculation (SFGR) program, nitrogen was mixed with the main air stream to simulate flue gas recirculation. The variables studied were:

Recirculation Ratio, R = N<sub>2</sub>/Air = 0.10, 0.20 Average bed temperature = 840, 900°C Excess Air = 15, 30%

### Constant operating conditions were:

Pressure = 808 kPaa Ca/S mole ratio = 3.0 Champion coal (-16 mesh, 2.19 wt. % S) Grove limestone (-8 + 25 mesh)

FIGURE VII-4 AMMONIA INJECTION PROGRAM: EFFECT OF EXCESS AIR LEVEL ON NO  $_{\mathbf{x}}$  EMISSIONS



AIMONIA

INJECTION • BASE LEVEL (No NH3 INJECTION)

△ WITH NH3 INJECTION (RUN 7)

▲ WITH NH3 INJECTION (RUN 5)

FIGURE VII-5
RESPONSE TIME

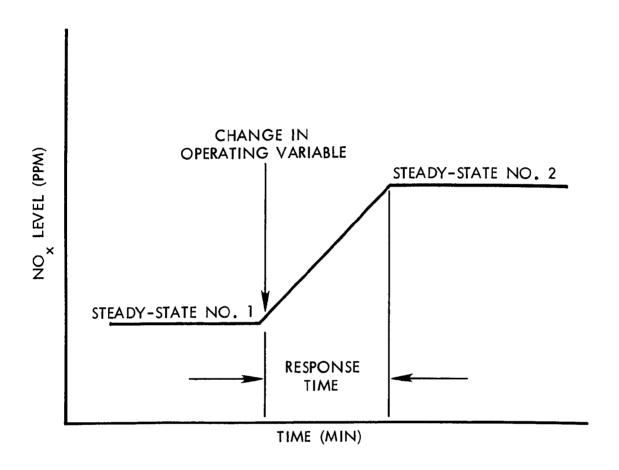


TABLE VII-12. RESPONSE TIME FOR AMMONIA INJECTION RUN 5

Run No.	Operating Vari Subject to Ch NH3/NO <sub>X</sub>		Response Time (min)	NO <sub>X</sub> Level (ppm)
5.1	Base Level (No Ir	njection)		145
5.2	2.18	2.94	10	210
5.3	1.27	2.50	10	200
5.4	Base Level (No	Injection)	20	160
5.5	3.41	0.0	7.5	210
5.6	8.05	0.0	5	250
5.7	Base Level (No	•	7.5	170
		A	vg. = 10	

The N<sub>2</sub> flow system used to cool down the combustor during an emergency shutdown was used to inject N<sub>2</sub> to simulate flue gas recirculation. The N<sub>2</sub> is mixed with the main air stream far upstream of the injection point and therefore, perfect mixing may be assumed.

### Results and Discussion --

The run conditions for the nine steady state periods are summarized in Appendix Table P-4. In each run, a steady state period was maintained without nitrogen addition and then in a second period, nitrogen was added without adding additional air. However, additional coal had to be burned to heat the nitrogen which was injected cold. Therefore, the excess air levels reported for the second period are all lower than for the first. Figure VII-6 shows the bar graphs for each emission index (E.I.) for NO2, SO2, and CO, and the 95% confidence interval for the emission levels. The error bars shown on the graphs were drawn using the standard deviations derived from a statistical analysis of the ammonia injection program data.

Table VII-13 summarizes the results of the SFGR program, Table VII-14 indicates the significance of changes in the emissions for each run. Significant reductions in the  $NO_X$  emissions, observed in runs 2 and 4, are correlated with changes in the excess air level which accompanied  $N_2$  addition during these tests. Increases in both the  $SO_2$  emission index and CO emission index are correlated to changes in the excess air level and the gas residence time. These changes in the  $NO_X$ ,  $SO_2$  and CO emissions are discussed below.

 $NO_X$  Emissions--Figure VII-7 compares the  $NO_X$  emissions vs. excess air results for the SFGR program with the baseline  $NO_X$  emissions from the ammonia injection program (no NH3 injection). Table VII-15 shows that the mean of the SFGR data (all data) nearly coincides with the mean of the ammonia injection data (base levels only). Also, because  $NO_X$  emissions decrease with decreasing excess air levels, it is believed that the lower  $NO_X$  emissions which resulted from  $N_2$  addition in these tests are really the result of lowering the excess air level. If the test had been conducted in a way which would have prevented an accompanying decrease in excess air with  $N_2$  addition (e.g., by increasing the air rate), it is expected that no change in  $NO_X$  emissions would have been observed. Therefore, flue gas recirculation should not have an effect on reducing  $NO_X$  emissions.

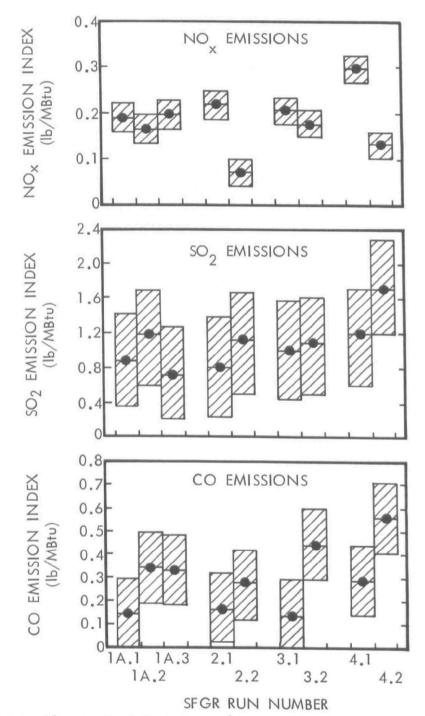
 $SO_2$  and CO Emissions--As seen in Table VII-13, both  $SO_2$  and CO emissions increase as a result of  $N_2$  addition. Although the increase in  $SO_2$  emissions could not be shown unequivocally to be statistically significant (Table VII-14), it was consistent.

Adding nitrogen to the main air has two effects:

- 1. The partial pressure of oxygen is lowered.
- 2. The gas velocity is increased, which lowers the residence time of the combined gas stream.

### FIGURE VII-6

### EMISSION INDICES FOR SIMULATED FLUE GAS RECIRCULATION (SFGR) PROGRAM SHOWING 95% CONFIDENCE INTERVALS



Note: The standard deviation values used to draw the error bars are from a statistical analysis of the Ammonia Injection Program data.

TABLE VII-13. SIMULATED FLUE GAS RECIRCULATION PROGRAM - SUMMARY OF RESULTS

SFGR	Independent Variables			Dependent Variables		
Run	R	Pad Tomp (9C)	Excess	Emission	n Index (1	
No.	<u>(%)</u>	Bed Temp. (°C)	<u>Air (%)</u>	NOx	SO <sub>2</sub>	CO
1A.1		874	18.6	0.19	0.89	0.14
1A.2	11.2	88 <b>6</b>	12.9	0.16	1.17	0.34
1A.3		882	17.2	0.19	0.72	0.33
		Average = 881				
2.1		874	18.4	0.22	0.81	0.17
2.2	23.4	887	7.4	0.07	1.09	0.27
		Average = 881				
3.1		906	26.5	0.20	1.01	0.14
3.2	11.2	897	25.0	0.17	1.07	0.45
		Average = 901				
4.1		819	16.7	0.29	1.18	0.29
4.2	11.3	817	13.0	0.13	1.72	0.56
		Average = 818		Avg = 0.18	Avg = 1.07	Avg = 0.30

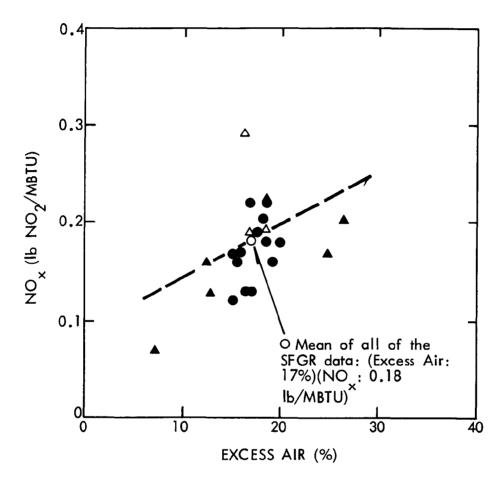
# TABLE VII-14. SIGNIFICANCE OF CHANGES IN EMISSIONS - SIMULATED FLUE GAS RECIRCULATION PROGRAM

### **Emission Index**

	(1b/MBTU)						
Run No.	Base Level	Level With N <sub>2</sub> Addition	Change From Base Level (%)	Significant Change?			
74.7	NO <sub>X</sub> Emissions						
1A.1 1A.3	0.190						
1A.2		0.164	-14	Maybe			
2.1	0.216						
2.2		0.068	-67	Yes			
3.1	0.203						
3.2		0.173	-15	Maybe			
4.1	0.293						
4.2		0.129	-56	Yes			
SO <sub>2</sub> Emissions							
1A.1 1A.3	0.804	2					
1A.2		1.168	+45	Maybe			
2.1	0.813						
2.2		1.094	+35	Maybe			
3.1	1.014						
3.2		1.066	+5	No			
4.1	1.177						
4.2		1.722	+46	Maybe			
		CO Emiss	ions				
1A.1	0.237						
1A.3	0,20						
1A.2		0.341	+44	No			
2.1	0.170						
2.2		0.274	+61	No			
3.1	0.135						
3.2		0.446	+230	Yes			
4.1	0.292						
4.2		0.562	+93	Yes			

FIGURE VII-7

COMPARISON OF THE SIMULATED FLUE GAS RECIRCULATION (SFGR)
PROGRAM WITH THE AMMONIA INJECTION PROGRAM



SFGR PROGRAM A (BASE LEVEL)

SFGR PROGRAM  $\blacktriangle$  (WITH N<sub>2</sub> ADDITION)

AMMONIA • (BASE LEVEL)

INJECTION PROGRAM

## TABLE VII-15. COMPARISON OF AMMONIA INJECTION AND SIMULATED FLUE GAS RECIRCULATION RESULTS

	Mean Value		
	Excess Air (%)	NO <sub>X</sub> (1b NO <sub>2</sub> /MBTU)	
Ammonia Injection (Base Levels Only)	17.2	0.17	
Simulated Flue Gas Recirculation (All Data)	17.3	0.18	

The first effect, the lowering of the oxygen partial pressure, may reduce both the rate of reaction of  $SO_2$  to form  $CaSO_4$  and the rate of reaction of CO to form  $CO_2$ . As was mentioned earlier, some excess oxygen is required for the reaction of  $SO_2$  with CaO according to the reaction:

$$Ca0 + SO_2 + 1/2O_2 \rightarrow CaSO_4$$

Therefore, reducing the oxygen partial pressure to low levels, may decrease the  $SO_2$  retention and increase the  $SO_2$  emissions. CO reacts with  $O_2$  in the gas phase according to the reaction:

$$c0 + 1/20_2 + c0_2$$

Similarly, decreasing the oxygen partial pressure substantially will decrease the rate of CO combustion and increase CO emissions.

The second effect, the lowering of the gas residence time, means that there is less time for both of the above reactions to occur. Hence, adding  $N_2$  to the gas stream means that the rate of each reaction and the time available for each reaction to occur are less. Therefore, both the  $\rm SO_2$  and  $\rm CO$  emissions increase with  $\rm N_2$  addition as shown by these runs.

For each steady state period, the sorbent feed rate was adjusted to maintain the Ca/S mole ratio at a constant value of 3.0. However, Table VII-16 shows that the Ca/S ratio varied about the average of 3.0, ranging from 2.65 to 3.36. In two runs, Ca/S increased when  $N_2$  was injected and in two runs Ca/S decreased when  $N_2$  was injected. This variation was not systematic; Ca/S did not change in proportion to the amount of  $N_2$  injected into the main air. Therefore, any systematic changes in  $SO_2$  emissions would not be expected to be caused by changes in the Ca/S ratio.

 $\frac{Conclusions-N_2}{NO_X} \ addition to the main air stream has no direct effect on NO_X emissions. The observed decrease in the NO_X emissions in this program can be explained by changes in the excess air level; N_2 addition lowered the percent excess air which in turn lowered the NO_X emissions. If the excess air level had been held constant, no change in NO_X emissions would have been expected.$ 

 $N_2$  addition results in increased  $SO_2$  and CO emissions probably because of lowered oxygen partial pressure and the gas residence time.

### Combined Techniques

A series of tests was conducted to determine if a combination of  $NO_X$  control techniques could result in further reductions in  $NO_X$  emissions. Since only staged combustion and ammonia injection caused a reduction in  $NO_X$  emissions, only the combination of these two methods was studied.

Two runs were completed and are discussed below. In run 1, two steady states were run at base level conditions, two with  $\rm NH_3$  injection, and then one with staged combustion. No sulfur retention data were obtained because the  $\rm SO_2$  analyzer was inoperable during this run. In run 2 one steady state

TABLE VII-16. VARIATION IN Ca/S MOLE RATIO

	Ca/S			
Run No.	Without N <sub>2</sub> Addition (Base Level)	With N <sub>2</sub> Addition	Change in Ca/S (%)	
1A.1	2.97	2 10	+2.1	
1A.2 1A.3	3.10	3,10	TZ , I	
2.1	3.36	2,85	-15.2	
3.1 3.2	3.05	2.87	-5.9	
4.1	2.65	2.87	+8.3	

Average = 2.98

was run at base level conditions, one with staged combustion, one with both NH3 injection and staged combustion and then one more with staged combustion only. The results are summarized in Appendix Table P-5. The constant operating conditions for both runs were:

Pressure 505 kPa 900°C Temperature Ca/S 3.0 25% Excess Air Coal Champion, -16 mesh, 2.19% S Grove, -8 + 25 mesh Limestone 290 cm above the grid Ammonia injection probe location Supplementary air probe location 33 cm above the grid 75% (2-stage combustion) Primary Air as % of Stoich 1.5 (ammonia injection) NH<sub>3</sub>/NO,

The values of the staged combustion and the NH3 injection variables are those found to be promising in the previous testing.

Table VII-17 shows the changes in NO $_{\rm X}$  levels in each run as changes were made from base level to various NO $_{\rm X}$  control and combination of NO $_{\rm X}$  control techniques.

Table VII-17 shows that in this work, either ammonia injection or staged combustion or a combination of the two techniques tend to lower NO $_{\rm X}$  emissions by 15 to 50% below the base level. The reduction in NO $_{\rm X}$  emissions by staged combustion is reasonably consistent with the earlier study at 505 kPa in which reductions of 10 to 20% were observed. The reduction in NO $_{\rm X}$  emissions by ammonia injection at 505 kPa with an NH $_{\rm 3}/{\rm NO}_{\rm X}$  ratio of 0.89 to 1.43 is consistent with the earlier ammonia injection program at 808 kPa in which a reduction of 33% was observed with an NH $_{\rm 3}/{\rm NO}_{\rm X}$  ratio of 1.36. Figure VII-8 shows that the reduction in NO $_{\rm X}$  emissions from the base level when both techniques were used simultaneously was not greater than the reduction when either technique was used alone. However, since only one data point was obtained when using the two techniques simultaneously, it cannot be stated with any certainty whether an interaction does or does not exist. Perhaps, if a larger NH $_{\rm 3}/{\rm NO}_{\rm X}$  ratio had been used, a greater reduction in NO $_{\rm X}$  may have been achieved.

 $SO_2$  and CO emission data were obtained for run 2. Table VII-18 shows that the  $SO_2$  emissions increased from 0.87 to 1.03 lb/MBTU going from base level conditions (run 2.1) to staged combustion (run 2.2) and the CO emissions were unchanged at 0.26 lb/MBTU. However, over the entire run both the  $SO_2$  and CO emissions increased; runs 2.2 and 2.4 were both with staged combustion only, but the  $SO_2$  emissions increased from 1.03 to 1.22 lb/MBTU and the CO emissions increased from 0.26 to 0.35 lb/MBTU. These upward drifts in the emission levels are unexplained.

#### BENCH REGENERATION STUDIES

The bench unit regenerator, because of its relatively small size, provides a means to experiment with operating conditions and methods quickly

0.7

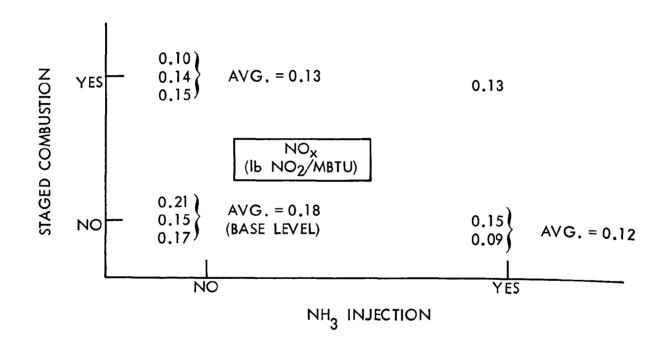
TABLE VII-17. COMBINED NO<sub>X</sub>-CONTROL TECHNIQUES PROGRAM. CHANGES IN NO<sub>X</sub> LEVELS.

				Control Tec	hnique		Change in NO <sub>x</sub>
Run <u>No .</u>	Excess Air (%)	Average Bed Temp. (°C)		jection (NH <sub>3</sub> /NO <sub>X</sub> )	Staged Combustion (Yes/No)	NO <sub>X</sub> (1b/MBTU)	from Base Level (1) (%)
1.1	25.1	891	No		No	0.21	-
1.2	22.6	896	Yes	0.89	No	0.15	-15
1.3	25,2	894	No		No	0.15	-
1.4	23.3	900	Yes	1.43	No	0.09	<b>-49</b>
1.5	22.0	901	No		Yes	0.10	-43
		Avg. = 896					
2.1	31.3	876	No		No	0.17	-
2.2	24.5	883	No		Yes	0.14	-21
2.3	25.3	887	Yes	1.61	Yes	0.13	<b>-</b> 26
2.4	24.2	883	No		Yes	0.15	-15
		Avg. = 882					

<sup>(1)</sup> Base level  $NO_X$  emission = 0.18 lb/MBTU (Average of Runs 1.1, 1.3, 2.1)

FIGURE VII-8

REDUCTION IN NO<sub>x</sub> EMISSIONS BY USING TWO CONTROL TECHNIQUES SIMULTANEOUSLY



# TABLE VII-18. $SO_2$ AND CO EMISSIONS FOR COMBINED $NO_X$ -CONTROL TECHNIQUES RUN 2

Run No.	Control Technique	Emission Ind	ex (1b/MBTU) CO
2.1	Base level	0.87	0.26
2.2	Staged-combustion	1.03	0.26
2,3	Staged-combustion with ammonia injection	1.15	0.32
2.4	Staged-combustion	1.22	0.35

and inexpensively. Promising results obtained in the bench regenerator can then be scaled up to miniplant regenerator operation.

The test program in the bench regenerator was divided into two segments, natural gas fueled regeneration and coal fueled regeneration. Most of the work concentrated on developing satisfactory operating techniques. The work done with natural gas-fueled regeneration included a study of the most effective way to introduce fuel to the regenerator and a limited study of operating variables and their effect on sorbent regeneration. Studies made with coal-fueled regeneration were preliminary in nature and were intended to determine the feasibility of operating a regeneration unit with coal. The incentive to use coal instead of natural gas is to reduce operating costs and to reduce reliance upon gaseous (or liquid) fuels.

The results of the bench unit regenerator test program will be discussed in the following sections.

#### Equipment

The batch regenerator unit was modified to permit semi-continuous operation and renamed the bench regenerator unit. The modified facilities were discussed in the previous annual reports (1,2) and will not be described in detail.

The regenerator vessel is 4.57 m high and is constructed of 12 inch carbon steel pipe and lined with Grefco 75-28 refractory to an inside diameter of 9.52 cm. The burner plenum chamber below the grid is 0.69 m high and is lined with Grefco Bubblelite refractory.

Sulfated sorbent is intermittently charged to the regenerator from a lock hopper via a line equipped with two cycling valves to meter in the sorbent (see Figure VII-9). The sorbent is introduced near the bottom of the fluid bed (9.5 cm above the grid) to insure complete mixing. Sulfated sorbent from miniplant operations is used. Regenerated sorbent exits the bench regenerator through a bed overflow line which leads to an overflow lock hopper. The below-bed burner is used to heat up the bed and provides some of the energy required by the regeneration reactions. A reducing zone is established immediately above the grid by adding supplementary fuel directly to the bed. This creates the CO and H<sub>2</sub> rich zone needed to carry out the regeneration reactions. Supplementary air is then added further up the bed. This creates a mildly oxidizing zone at the top of the bed which oxidizes any undesirable CaS formed in the reducing zone. All remaining fuel is also burned in the oxidizing zone, to maximize fuel use efficiency.

#### Natural Gas-Fueled Regeneration

Operating Procedures--

The initial charge of bed material is heated to the desired temperature under oxidizing conditions by operation of the below-bed burner. Sometimes, small amounts of supplementary fuel are added directly to the bed to increase bed temperature, but not enough is added to produce reducing conditions in the bed. Supplementary fuel can only be added when bed temperature is above 650°C, the temperature at which natural gas will ignite easily.

When the bed temperature is uniform and close to the desired run temperature, the switchover to reducing conditions is made. This is accomplished by increasing flow of supplementary air to the required value and then increasing the flow of supplementary fuel. Supplementary air flow is always increased before supplementary fuel to avoid adding air to a column already filled with a reducing gas. Temperature is continuously monitored and flow rates of burner air, burner fuel, supplementary air, and supplementary fuel are all adjusted to yield the desired bed temperature. Oxygen and CO concentrations in the off gas are also monitored and the supplementary air flow is corrected to produce low concentrations of CO (under 5000 ppm). The flow rate of supplementary air is the minimum value which will just produce CO in the oxidizing zone.

#### Shakedown --

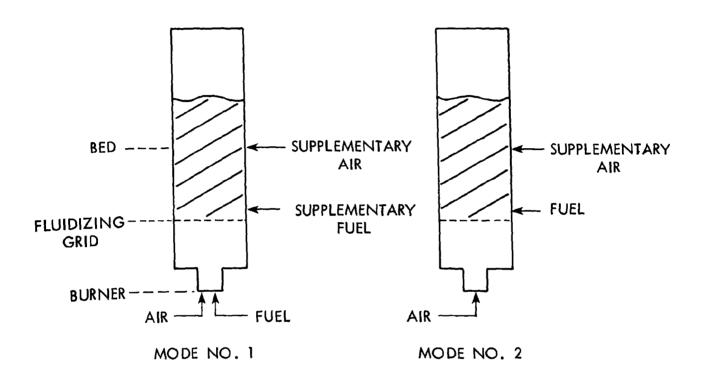
Shakedown of the bench regenerator was completed in two runs. During the first shakedown run, all systems except the sorbent feeder operated successfully. The opening of the feed line in the regenerator was found to be partially blocked with fused bed and refractory, which had been left from an earlier batch run. The next run proved to be more successful. The sorbent feed system functioned properly and the high temperature necessary for regeneration was achieved using the air preheater.

The main operational difficulty during shakedown runs was maintaining a satisfactory bed height. Although the solids overflow port is 1.0 m above the grid, the highest expanded bed height obtained was under 0.5 m. It is believed that the bed slugged badly and the solids were moving, piston-like, up the regenerator and out the sorbent overflow due to high superficial gas velocities. This was a continuing problem during the test program. The effect was partly offset by minimizing the gas velocity.

#### Effect of Fuel Injection Mode--

One objective of the work done with natural gas-fueled regeneration was to determine the best way of introducing the natural gas. The energy required to maintain the temperature of the unit can be supplied in two ways. The burner below the fluidizing grid can be operated at oxidizing conditions and supplementary fuel added to the bed to create the reducing zone (Mode 1). or all fuel can be added directly to the bed where it would partially burn in the fluidizing air (Mode 2) (see Figure VII-10). In both modes of operations, the reducing gas is produced by the incomplete combustion of methane which is added directly to the bed through the supplementary fuel probe. Adding all the fuel to the bed eliminates the heat losses associated with operation of the burner, thereby decreasing the fuel requirements and possibly increasing the SO2 content in the off gas. One drawback to adding all the fuel directly to the bed is that the entering cold fuel must heat up to ignition temperature before it burns. The heat up requires a certain residence time in the bed, causing the methane to move further up the bed before it ignites. As the level of combustion varies, the location of the reducing zone also varies which might have a detrimental effect on temperature control and on regeneration.

FIGURE VII-10
FUEL INJECTION MODE



Burner section heat losses—In determining the best mode of introducing fuel into the regenerator, it was observed that a significantly smaller amount of fuel was required to maintain bed temperature when all the fuel was added directly to the bed as opposed to operation with the burner (e.g., 0.079 compared to 0.045 Nm³/min). To account for this discrepancy in energy requirements, the heat losses associated with burner operation were determined. Losses include radiation and convection of heat from the burner plenum, and heat loss to the cooling water in both the fluidizing grid and the burner head. These heat losses were calculated and are summarized below:

Radiation and convection from plenum:

$$Q_1 = Ak\Delta T = 0.63 \text{ m}^2 \times 21.8 \text{ JM}^{-2} \text{s}^{-1} \text{K}^{-1} (533-300) \text{K}$$
  
= 3200 Js<sup>-1</sup>

Fluidization grid cooling water:

$$Q_2 = mCp\Delta T = 4 \times 3.17 \times 10^{-2} \text{kg sec}^{-1} (4.187 \times 10^3 \text{Jkg}^{-1} \text{K}^{-1}) (306-294) \text{K}$$
  
= 6364 Js<sup>-1</sup>

Burner cooling water:

$$Q_3 = mCp\Delta T = 3.17 \times 10^{-2} kgs^{-1} (4.187 \times 10^3 Jkg^{-1} K^{-1})(328-294) K$$
  
= 4508 Js<sup>-1</sup>

Total heat loss =  $Q_1 + Q_2 + Q_3 = 14072 \text{ Js}^{-1}$ 

where Q = heat loss

A = area

k = combined heat transfer coefficient for radiation and convection

 $\Delta T$  = temperature differential

m = mass flow rate
Cp = heat capacity

The heating value of methane is  $3.73 \times 10^7 \, \mathrm{Jm}^{-3}$ . Therefore, the heat loss associated with burner operation is equivalent to the heating value of a flow rate of  $3.78 \times 10^{-4} \mathrm{m}^3 \mathrm{s}^{-1}$  of methane. Thus, the difference in fuel requirements between operation with and without the burner can be largely explained by the heat losses associated with burner operation. Since about  $0.079 \, \mathrm{Nm}^3/\mathrm{min}$  of methane are required to operate the regenerator with the burner, a significant portion (about 30 percent) of the fuel requirement is used to overcome heat losses.

Results and Discussion --

Mode No. 2 operations—Six runs were made in the bench regenerator to evaluate operation in which all the fuel (methane) is added directly into the bed. In five of the runs, supplementary fuel was injected at the wall of the reactor. In the sixth run (R-8), a new supplementary fuel probe designed to give better fuel distribution across the bed was used. Operation with all of the fuel added to the bed generally resulted in an uniform temperature pro-

file throughout the bed but controlling the temperature to the desired level proved difficult. During the series of six runs, only one steady state regeneration condition was reached. The steady state was a 15 minute segment of run R-6. Operating conditions for run R-6 are presented in Table VII-19.

Many of the regenerator runs in which all the fuel was added directly into the bed were ended by high temperature shutdowns. Controlling the temperature proved difficult when mode No. 2 was used. The factors which might cause a temperature runaway are:

- 1. While in oxidizing conditions during heat up:
  - a. an increase in fuel flow, thereby adding additional energy input
  - b. a decrease in air flow thereby increasing the temperature of the preheated incoming gas
- 2. While in reducing conditions:
  - a. a decrease in fuel flow causing oxidizing conditions and oxidation of CaS (an exothermic reaction)
  - b. an increase in air flow causing oxidizing conditions
- 3. In either reducing or oxidizing conditions:
  - a. a decrease or stoppage in solids feeding
  - b. poor mixing, including fuel bypassing, air bypassing, and poor solids circulation, causing hot spots and agglomeration

Possible reasons for the problems of in-bed fuel injection into the regenerator were examined and it is believed that the cause is related to mixing. In order for fuel addition into the bed to work, the fuel must be heated above its ignition temperature shortly after injection into the bed. If the cold fuel entering the regenerator through the probe does not mix with the hot bed solids and is not brought up to ignition temperature, the "flame" may be blown out. It may be that the large, sudden, changes in temperature were caused by ignition or extinction of the methane flame. These changes could have been brought about by shifts in the pattern of solids mixing within the fluidized bed.

The hypothesis that poor solids and gas mixing results in "flame blow outs" is supported by experimental evidence. During many of the runs, it was observed that the  $0_2$  concentration in the off gas increased suddenly, although no change in operating conditions (fuel or air flow rates) had occurred. The sudden increase in the  $0_2$  concentration in the off gas would be characteristic of the effects of a flame blow out. The increase of  $0_2$  concentration, since the air was no longer used in combustion, led to oxidation of the CaS present, an exothermic reaction. The exothermic reaction resulted in high bed temperatures and the subsequent high temperature shutdowns.

It is possible that the problem of a flame out could have been lessened, or even eliminated, by heating to higher temperatures the air entering the regenerator through the fluidizing grid. This would have reduced the heat transfer requirement produced by solids backmixing. Unfortunately, with the equipment at hand, it was possible to heat the incoming air only to 150-200°C.

TABLE VII-19. BENCH REGENERATOR RUN SUMMARIES

Run Number	Avg. Bed Temp. K	P <u>atm</u>	Expanded Bed Height m	Sup. Gas Vel. m/s	Gas Res. Time sec.	φ <sup>(2)</sup> Equiv <u>Ratio</u>	Solid Res. Time min.	SO2; Mole % Average (Range)	Peak % SO2 Equil. % SO2	Extent of Solids Regeneration
R-6(1)	1370	6.9	0.9	0.7	1.34	1.43	29	1.17 (0.24-1.74)	0.25	NA
R-9	1340	7.5	0.6	1.1	0.56	1.42	48	0.62 (0.18-0.99)	0.25	NA
R-10	1350	7.6	0.7	1.0	0.70	1.64	35	0.58	0.25	44
R-11.1	1350	7.6	0.6	0.9	0.66	1.85	43	0.59 (0.09-1.32)	0.29	71
R-11.2	1350	7.6	0.6	0.9	0.70	1.83	29	0.24 (0.06-0.92)	0.20	71
R-12	1345	7.6	8.0	0.9	0.89	1.50	51	0.27 (0.03-0.66)	0.15	48
R-13	1395	7.6	0.6	1,2	0.50	1.57	22	0.53 (0.1-1.35)	0.15	83
R-14	1360	7.5	0.6	1.1	0.55	1.45	37	0.43 (0.15-0.84)	0.15	79

<sup>(1)</sup> Mode No. 2 Operation (all fuel added to bed, burner not used).

NA = Data not available.

<sup>(2)</sup>  $\phi$  = Stoichiometric air to fuel ratio x  $\frac{\text{fuel flow rate}}{\text{air flow rate}}$ 

Also, in a larger bed with improved solids mixing, the difficulty in maintaining the flame should be lessened and in-bed injection of the fuel should be feasible.

Mode No. 1 operations—The alternative to in-bed fuel injection, as described previously, is operation of the burner supplemented by in-bed fuel injection. The disadvantage of this method of operation is the increased heat losses which accompany burner operation; increased heat losses require additional air and fuel inputs thereby diluting the off gas SO2 concentration if thermodynamics is not limiting. An advantage of this method is that the burner head serves as a flame holder, an element that was evidently lacking in the previous method described. The combustion gases rising from the burner also provide the heat necessary to ignite the incoming supplementary fuel, eliminating the problem of flame blow outs.

Six successful runs were made with the burner in operation while supplementary fuel is added above the fluidizing grid to produce a reducing zone. The results of these six runs, which represent the variable study, are presented in the next section.

Results of process variable study--Operation with both burner and supplementary fuel resulted in a steadier and more easily controlled bed temperature. The important operating conditions for runs R-9 through R-14 are summarized in Table VII-19. It was intended to determine the effect of operating variables on SO2 concentration in the off gas and extent of sorbent regeneration. However, because of the data scatter and the limited amount of data obtained, it was not possible to draw conclusions about the relationships between operating variables and sorbent regeneration.

SO2 concentration in the regenerator off gas can be limited by thermodynamics, kinetics or the heat and mass balance. The SO2 concentration from the bench unit, as in the case of the miniplant, is limited by the heat and mass balance. The operating variables in Table VII-19 (temperature, pressure, gas residence time, solids residence time, and equivalence ratios) are either thermodynamic or kinetic variables used to define the system. Since the system is heat and mass balance limited, no conclusive relationships between the variables and the SO2 concentration in the off gas were expected. Indeed. from the data in Table VII-19, no such relationships were apparent, confirming that the SO2 levels were not kinetically or thermodynamically controlled. A further consequence of the heat and mass balance limitation is very low SO2 concentrations in the regenerator off gas. As seen, the concentrations ranged from 0.2 to 0.6%. The approach to thermodynamic equilibrium of the system can he expressed as the ratio of peak SO2 concentration to the SO2 concentration at equilibrium for the prevailing temperature and pressure. From Table VII-19, it is seen that this ratio was below 0.3 (or 30% of equilibrium) for all runs.

# Coal Fueled Regeneration

The objective of the test series using coal in the bench regenerator was to evaluate the feasibility of coal-fueled regeneration in a PFB. Factors which needed to be evaluated were: uniformity of temperature in the fluidized

bed, temperature control, tendency for bed agglomeration,  $SO_2$  levels produced, and degree of reduction of CaSO<sub>4</sub> to CaO. As can be seen, these are primarily operability questions.

Operating Procedures --

Coal is fed to the regenerator by using the same coal feeding and control system used for the bench combustor. The system was described in the previous reports (1,2). A coal probe, similar to the design used for the bench combustor, is directed downward at about a 45° angle and delivers coal at the inner regenerator wall about 8 cm above the fluidizing grid. Coal is fed to the regenerator after the bed temperature is heated above 650°C with the natural gas burner.

Experimental Results and Discussions--

Nine experimental runs (Runs R-15 through R-23) were made with coal-fueled regeneration. Four runs, R-19 to R-22, were shut down soon after coal injection was started because of high and erratic bed temperatures which could have resulted in serious bed agglomeration. The other five runs were more successful; about ten hours of operation on coal were accumulated. Operating conditions for the steady state periods are reported in Table VII-20. The major operational problems during the runs were plugging of the off gas filter, maintaining proper superficial gas velocities, and bed temperature control.

It was necessary to end three of the runs, R-15, 16 and 18, because the sintered metal off gas filter became plugged, resulting in loss of column pressure control. Filter plugging might have been caused by an increased solids loading in the regenerator off gas due to the carryover of unburnt coal and ash particles. Another factor which might have contributed to the plugging of the filter was increased bed solids carryover due to excessive superficial gas velocities.

There is a problem in maintaining proper superficial gas velocities when fueling the regenerator with coal. When operating the regenerator with coal, combustion air is introduced to the regenerator from two sources: primary combustion air enters through the grid, and coal transport air enters through the coal probe, which is positioned about 8 cm above the fluidizing grid and aimed downward. Transport air flow is approximately 0.26 m³/min, which is almost half of the total air flow. Exactly how the air entering via the coal probe contributes to bed fluidization is unknown. Therefore, to insure good bed fluidization, a minimum superficial gas velocity of 0.61 m/s was maintained just above the fluidizing grid by adjusting the primary air flow. Operating pressure was varied during the coal run series to adjust the superficial gas velocity to a level which would insure good bed fluidization and avoid slugging of the bed.

The ability to control bed temperature is crucial to the operation of a coal-fueled regenerator. Temperature runaways to 1100°C can result in extensive bed agglomeration because of the presence of coal and ash in the bed. During the coal runs, a few temperature runaways occurred, resulting in a high temperature shutdown of the unit and a limited amount of bed fusion.

TABLE VII-20. COAL FUELED BENCH REGENERATOR RUN SUMMARIES

Run Number	Avg. Bed Temp. K	P atm	Expanded Bed Height m	Sup. Gas Vel. m/s	Gas Res. Time sec.	Equiv	Solid Res. Time min.	SO <sub>2</sub> ; Mole % Average (Range)	Peak % SO <sub>2</sub>	Extent of Solids Regeneration
R-17	1422	4.6	2.1	1,6	1.32	1.52	62	1.32 (0-3.5)	0.16	40
R-18	1422	4.7	0.7	1.5	0.44	1.54	16	2,1 (0,1-3,7)	0.17	38
R-23	1338	6.1	0.8	1.5	0.51	1.56	31	0.9		40

# (1) $\phi = \frac{Air \ Required \ for \ Complete \ Combustion}{Air \ Supplied}$

Assumes 100% Combustion Efficiency

Sorbent: Sulfated Dolomite

Coal: Champion

When the unit was operated in oxidizing conditions, temperature control was not a problem. However, temperature control was difficult under reducing conditions. To maintain reducing conditions in the unit, the coal feed rate must be such that the air-to-fuel ratio is substoichiometric. With the large amount of transport and primary air required, the coal feed rate needed to produce reducing conditions exceeded that needed to satisfy the energy requirements of the unit. That is, operating temperatures were too high and resulted in high temperature shutdowns. To reduce the coal feed rate, combustion air flow rates also had to be reduced.

To achieve this reduction in air rate, the coal feeding system was modified to use nitrogen, instead of air, as the transport medium, making the primary air the only source of combustion air. Only one run, R-23, was made with the modified coal transport system. During the run bed temperatures were more easily controlled than in previous runs. However, after 25 minutes of steady state regeneration, bed temperatures again began to rise. At this point, the run was ended to avoid agglomeration of the bed. The tendency for the bed temperature to rise after a given period of steady state regeneration has been noted not only in the bench regenerator studies but also in the batch studies carried out in the batch and miniplant regenerator units. The most probable reason for the temperature increase is that the bench unit operates as a "batch regenerator" during startup because the unit was charged with sulfated sorbent prior to the run. Initially, the energy released by the combustion of the coal is absorbed in the endothermic regeneration of this large mass of startup bed material. When the entire bed inventory is regenerated, the endothermic regeneration reaction takes place at a lower rate, since only the incoming sorbent is being regenerated. Unless the coal feed rate is reduced to compensate for the lower heat requirement. a temperature increase could result after the initial charge is regenerated. Changes in the off gas composition which occurred concurrently with the temperature rise support this explanation. The SO<sub>2</sub> level dropped even though the regenerator remained in reducing conditions.

The results of the few runs in the bech regenerator are promising. problems were encountered which would rule out the use of coal. Maintaining good bed fluidization while still maintaining a constant bed height was a problem as it was with natural gas fueled regeneration. The partial bed agglomerations that did occur were probably a result of poor fluidization. The small diameter (9.5 cm) of the regenerator makes good gas and solids mixing and fluidization difficult. Temperature control was a problem at times but again, the cause could have been poor gas and solids mixing in the small regenerator vessel. Regeneration levels for the coal runs ( $\sim 40\%$ ) were not as high as the natural gas runs. A possible explanation is that the concentration of CO and Ho in the reducing zone was lower than in the previous runs. Obviously, more work must be done in the area of coal-fueled PFB sorbent regeneration on both small and larger scales to make a complete evaluation. Such questions as where and how to add supplementary air and coal to maintain proper fluidization, mixing and temperature control must be answered. Modifications to the regenerator vessel, such as a tapered cross section in the lower part of the vessel, should also be considered.

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## SECTION X

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#### APPENDIX A

# EFFECT OF PARTICLE SIZE ON CONVERSION RATE OF LIMESTONE AND DOLOMITE: THERMAL GRAVIMETRIC EXPERIMENTS

The sulfation of limestone and dolomite has been described by models which are based on the idea that the conversion rate is governed by diffusion. Several models, called "grain" models, assume that individual limestone particles are composed of many sub-particles, or grains (23,24). These grains are imagined to be non-porous. When limestone reacts with  $SO_2$ ,  $SO_2$  diffuses through the pores separating the grains (intergranular pores) and reacts on the grain surfaces. The speed of the reaction is determined by the local  $SO_2$  concentration inside the pores and by the chemical reaction rate at the surface of the grain. Alternatively, and this is probably the case, when  $SO_2$  reacts with limestone or dolomite, the speed of the reaction is determined by the local  $SO_2$  concentration inside the pore and by the thickness of the solid reaction product layer which forms on the surface of each grain.

The grain model implies the primary importance of physical properties such as porosity, pore size distribution, surface area, and grain size, in determining the conversion rate of a limestone or dolomite particle. Experimental studies have confirmed that these properties are important.

The effect of particle size on the conversion rate of limestone depends on which step in the reaction scheme is rate controlling. If diffusion through intergranular pores is controlling, then a concentration gradient of  $SO_2$  could exist from the surface of the particle to its center. Grains near the surface would sulfate faster because of the higher  $SO_2$  concentration and a shell of reaction product ( $CaSO_4$ ) would form on the surface of the particle. In this situation, particle size has the greatest effect on conversion rate and the time required to achieve a given fractional conversion of the particle is proportional to  $1/R^2$ , where R is the particle radius. At the other extreme is the situation where  $SO_2$  is spread uniformly through the pores of the particle and all of the particle's interior reacts at the same rate. In this case, particle size will have no effect on conversion rate.

In order to explore the effect of particle size on conversion rate, limestone and dolomite particles of different sizes were sulfated in a TGA at conditions of 900°C, 0.25% SO<sub>2</sub>, 5% O<sub>2</sub>, and balance N<sub>2</sub>. Four particle size ranges were investigated: -8 + 25 mesh, -16 + 40 mesh, -100 + 200 mesh, and -325 mesh. These size ranges correspond approximately to average particle diameters of about 1500, 700, 100 and 40  $\mu m$ , respectively. Table A-1 gives data on initial conversion rates, the time to reach 15 percent conversion, and the percentage conversion after 75 and 150 minutes for each of the four average particle sizes.

A number of significant observations can be made from the data of Table A-1.

TABLE A-1. EFFECT OF PARTICLE SIZE ON THE SULFATION OF DOLOMITE AND LIMESTONE

# Dolomite (Pfizer)

Average Particle	Initial Rate,	Time for 15%	% Con	version
Diameter, µm	mg SO3/(mg CaO)(min.)	<u>Cónversion, min.</u>	75 min.	150 min.
1500 <sup>(1)</sup>	0.24	32	28	32
700 <sup>(1)</sup>	0.044	4.0	40	44
100	0.100	1.5	39	45
40 <sup>(2)</sup>	0.087	2.0	75	90

# Limestone (Grove)

Average Particle Diameter, µm	Initial Rate, mg SO <sub>3</sub> /(mg CaO)(min.)	Time for 15% Conversion, min.	% Conv 75 min.	version 150 min.
1500	0.010	150	6.4	7.1
700	0.017	150	5.9	6.7
100 <sup>(2)</sup>	0.056	3.5	59	70
40 <sup>(2)</sup>	0.055	3,3	79	89

<sup>(1)</sup> Average of 3 runs.

<sup>(2)</sup> Average of 2 runs.

- Particle size has a large effect on the percentage conversion or utilization of both limestone and dolomite. For example, the percentage conversion of limestone increases by about a factor of ten as particle size is reduced from 1500 to 100 μm.
- 2. Coarse dolomite achieves much higher utilizations than coarse limestone. However, utilizations for fine (100 and 40  $\mu$ m) limestone and dolomite are comparable.
- 3. The initial rates for dolomite are higher than for limestone for each of the particle sizes studied.
- 4. Initial rates, time for 15% conversion, and percentage conversion after 75 and 150 minutes are all more sensitive to changes in particle size for the larger size particles.

Each of the above observations can be interpreted in terms of the grain model. Observation (1), the effect of particle size on utilization, implies that a concentration gradient of  $SO_2$  through the particle does exist and that diffusion of  $SO_2$  through a sulfate shell surrounding the particle does influence the conversion rate. Observation (2) suggests that dolomite has larger intergranular pores than limestone. Indeed, pore diameters for a limestone and dolomite, different from the stones used in this study, were measured by Borgwardt and Harvey (25). Their limestone had pores about 1  $\mu$ m in diameter whereas their dolomite had 0.5-20  $\mu$ m pores. Because of the wider intergranular pores, large dolomite particles achieve higher utilizations than large limestone particles. However, as particle size is reduced, so is the influence of intergranular diffusion. Thus, for small particles, limestone and dolomite give comparable utilization.

Observation (3), that the initial rate for dolomite is higher than for limestone is consistent with the hypothesis that dolomite has larger pores. Also, dolomite may have smaller grains than limestone. Observation (4), that the effects of particle size become less as size is reduced, again implies that intergranular diffusion becomes less important as the particle size becomes smaller.

There are practical implications for fluidized bed coal combustors from the above results. Using smaller sorbent particles, particularly when the sorbent used is limestone, can result in a significant reduction in the quantity of sorbent required. Although, the use of smaller particles will result in higher entrainment rates, oversized cyclones can be used to return sorbent fines to the bed. Lower superficial gas velocities may also be used to reduce entrainment. Whether or not the use of fine particles is practical would depend on the trade-off of reduced sorbent and sorbent disposal costs vs. increased equipment costs (i.e., the cyclone recycle) and potentially lower equipment capacity.

#### APPENDIX B

#### TESTING OF VARIOUS STONES FOR ABILITY TO ABSORB SO<sub>2</sub>

Several limestones and dolomites were screened in the thermogravimetric analyzer (TGA) for their ability to absorb  $SO_2$ . Two gas compositions were used, one to promote calcination and the other to suppress it. Table B-1 gives the reaction conditions and the fraction of each stone sulfated after 60 minutes. The "standard" sorbents, Grove limestone and Pfizer dolomite are shown for comparison.

The data of Table B-l shows that there is considerable variability in reactivity among the stones, even when testing was done under calcining conditions (condition "A" in Table B-l). The activity of Plum Run dolomite is fairly high, even under conditions which suppress calcination of CaCO<sub>3</sub> (condition "B" in Table B-l) because the MgCO<sub>3</sub> in the dolomite always calcines, introducing porosity into the stone. The substantial MgO content of the low grade limestone is probably responsible for its good activity under condition B. The high grade Mulzer limestone, however, shows fair activity even under "non-calcining" conditions, even though it contains little MgO. This result was probably caused by partial calcination of the CaCO<sub>3</sub> in the stone. The other sorbents showed relatively low activity even under "calcining" conditions. The reasons are not known but could be due to other structural differences in the sorbents.

It should be noted that all stones of Table B-I were tested as powders. The fractional sulfations observed would have been quite different, probably much lower, had the stone bed tested in the form of 2-3 mm granules, commonly used in fluidized bed combustors.

TABLE B-1. REACTIVITY OF VARIOUS STONES UNDERGOING SULFATION

	Fraction Sulfated in 60 Minutes						
Sample (Powder)	Test Conditions "A"	Test Conditions "B"					
	("Calcining")	("Non-Calcining")					
Dolomite, Plum Run <sup>(1)</sup>	0.51	0.29, 0.58 <sup>(2)</sup>					
Limestone, Mulzer, High Grade <sup>(1)</sup>	0.36	0.11, 0.13 <sup>(2)</sup>					
Limestone, Consolidation Coal, Low Grade(1)	0.45	0.28, 0.37 <sup>(2)</sup>					
Limestone, Ste. Genevieve, High Purity (3)	0.17						
Limestone, Dolomitic <sup>(3)</sup>	0.062	•=					
Limestone, Grove, BCR No. 1359	0.30	••					
Dolomite, Pfizer, BCR No. 1337	0.39						

#### Test Conditions

- A 870-900°C; 0.25% SO<sub>2</sub>, 5% O<sub>2</sub>, <u>bal N2</u> Sample heated to 870 C <u>in N2</u> prior to sulfation
- B  $870^{\circ}$ C; 0.25% S0<sub>2</sub>, 5% O<sub>2</sub>, <u>bal CO<sub>2</sub></u> Sample heated to 870 C <u>in CO<sub>2</sub></u> prior to sulfation to suppress calcination
- (1) Samples supplied by American Electric Power Service Corp.
- (2) Replicate run
- (3) Samples supplied by Western Materials Company

#### APPENDIX C

#### DATA MANAGEMENT SYSTEM

The data generation and management system for the miniplant combustor was thoroughly described in the previous annual report (1). The entire system has been augmented to include the collection, computation, and summarization of miniplant regenerator data. Description of the major additions follow.

#### HOKE SYSTEM

Figures C-1 and C-2 are examples of the tabular form of regenerator data printout received from a run. This printout is then used in conjunction with the combustor printout and the continuous recorder charts to determine the steady state period.

#### INPUT/OUTPUT PROGRAMS

Regenerator segments of the computer printout generated by the I/O program are shown in Figures C-3 and C-4. The first figure is an example of the experimental data inputs used in the program and Figure C-4 is an example of the calculated outputs. Addition regenerator data, such as off gas emissions and solids chemical analyses and particle size distributions, are inputed in the appropriate sections. The regenerator effluent streams are included in the overall mass accountability section.

FIGURE C-1

MINIPLANT ANALYSIS PROGRAM

4/30/79

#### REGENERATOR SECTION

	TEMPE	RATURES	(DEG	F)											
	1	TIME													
	2	PORT !	NO.	2	(5IN)										
	3	PORT !		3	(11IN)										
	4	PORT !		4	(17IN)										
	5	PORT I		5	(23IN)										
	6		NO.	7	(35IN)										
233	7	PORT /		8	(41IN)										
ຜ	8	PORT !		9	(48IN)										
	9	PORT 1		10	(56IN)										
	10		NO.		(64IN)										
	11	PORT 1	NO.	12	(74IN)										
		RUN		1		2	3	4	5	6	7	8	9	10	11
		5.12	18	29		1820.	1837.	1852.	1859.	1874.	1889.	1885.	1887.	1890.	1893.
		5.13	19			1819.	1833.	1849.	1856.	1871.	1886.	1881.	1884.	1887.	1889.
		5.13	20	29		1818.	1832.	1848.	1855.	1869.	1883.	1878.	1880.	1883.	1886.
		5.13	21	29		1815.	1833.	1851.	1857.	1871.	1884.	1879.	1880.	1883.	1888.
		5.12	22	29		1814.	1834.	1852.	1858.	1871.	1885.	1880.	1880.	1884.	1887.

5.13

5.12

21 29

22 29

FIGURE C-2

#### MINIPLANT ANALYSIS PROGRAM

2.15 1839.76

2.15 1840.09

4/30/79

1.02

1.02

7.03 1424.60

7,56 1385.56 0.54

0.52

REGENERATOR SECTION

12	TIME										
REDUC	TION ZO	INE									
13 14 15	AVERA	FICIAL VELOG GE BED TEMPE UEL RATIO(PE	ERATURE (DEG								
OXIDI	ZING ZO	NE									
16 17 18	AVERA	FICIAL VELOO GE BED TEMPE UEL RATIO(PH	RATURE (DEG								
19 20 21	OFFGA	DED BED HEIG S CO LEVEL(F S SO2 LEVEL(	PM)								
	RUN	12	13	14	15	16	17	18	19	20	21
	5.12 5.13 5.13	18 <b>29</b> 19 29 20 29	2.14 1 2.14 1 2.14 1	840.33	1.27 1.26 1.27	2.75	1886.46 1883.37 1880.16	1.01 1.01 1.01	5.26	1274.00 1347.48 1249.00	0.55 0.45 0.52

2.74 1881.18

2.75 1881.35

1.27

1.27

# FIGURE C-3

1.37

3.74 2.13

\*\*\*\*

TEMPERATURE (CELSIUS)		SETTLED BED HEIGHT (M)
PORT NO. 2 ( 5 IN) FORT NO. 3 ( 11 IN) PORT NO. 4 ( 17 IN) PORT NO. 5 ( 23 IN)	994. 1004. 1012. 1016. 1025.	INITIAL FINAL
PORT NO. 7 ( 35 IN) PORT NO. 9 ( 41 IN) PORT NO. 9 ( 43 IN) FURT NO. 10 ( 56 IN) FORT NO. 11 ( 54 IN)	1025. 1032. 1029. 1030. 1031.	HEIGHT AVEC GRID(M)
FORT NO. 12 ( 74 IN) FORT NO. 13 ( 83 IN)	1032. 1026.	BED BULK DENSITY (GR/CM3
PORT NO. 14 ( 92 IN) PORT NO. 16 (110 IN) PORT NO. 18 (128 IN) PORT NO. 21 (158 IN) FORT NO. 24 (194 IN) FORT NO. 26 (218 IN) CYCLONE DISCHARGE COOLER OFFGAS	1013. 871. 775. 936. 886. 881. 1443.	INITIAL FINAL
PRESSURES (KPA)		
NOMINAL OPERATING R-C FRESSURE DROP GRID PRESSURE DROP BED PRESSURE DROPS	775.961 5.961 2.194	
PORTS 29 TO 37 (5 IN TO 4 FORTS 29 TO 34 (5 IN TO 2		
FLOW RATES (M3/MIN)		
BURNER AIR PURNER FUEL SUPPLEMENTARY AIR SUPPLEMENTARY FUEL	1.85 3.18 2.46 2.26	
C-R TRANSFER RATE(KG/HR)	15.4	
(NIM/EM)WCJE SN YRAIJJIKU	ð.15∂	

# FIGURE C-4

### REGENERATOR SECTION

	SUPERFICIAL VELOCITY (M/SEC)		SULFATION LEVEL OF SORPENT(APCT)		
	SEDUCING ZONE	<b>7.6</b> 0.79	TO REGENERATOR FROM REGENERATOR	23.4 <i>€</i> ******	
AVERAGE BET TEMPERATURES (CHLSIUS)					
	REDUCING ZONE - PORTS 2 TO 5 OXIDIZING ZONE - PORTS 7 TO 12	1006.79 1029.77	REGENERATION LEVEL(PCI) COMBUSTOR FEED SULFUR IN RA	LJEN OFFJAS (4PCT)	39.76 73.06
236	SETTLED BED HEIGHT (M) EXFANDED BED HEIGHT (M) GAS RESIDENCE TIME (SEC) SOLIDS RESIDENCE TIME (HRS)	******* 1.85 2.34 3.23			
	ENERGY INPUT(MBTU/HR)	<b>0.</b> 51			
	AIR TO FUEL RATIO(PHI)				
	REPUCING ZONE OXIDIZING ZONE	1.28 1. <b>3</b> 3			

236

#### APPENDIX D

# PARTICLE SIZE DISTRIBUTION AND CONCENTRATION MEASUREMENT PROCEDURES

The measurement of PFB generated particulates is in its infancy. Sophisticated high temperature high pressure in-situ size distribution measuring devices are being developed and tested to understand better the particulate emissions. While these systems are being developed, simpler systems are being used to generate data which will give some indication of the particulate emissions and particle size distribution. In the miniplant this is done by sampling for flue gas particulates with Balston total filters. Some stream concentrations which cannot be sampled directly are calculated based on mass balances. Size analysis of all fine particulates (-45 µm) is done with the Coulter Counter Model TAll. The procedure developed for the miniplant will be described in this section. This section assumes a working knowledge of particulate sampling and Coulter Counter operations. It is meant to supplement, rather than replace good sampling practice and/or the Coulter Counter operations manual.

# Balston Filter Sampling

They use a 2.5 cm diameter by 18 cm long Grade BH filter tube. This filter is 99.95% efficient for 0.6 µm particles. The filter cartridge is stored in its plastic wrapping in a dry box to make sure it is dry. Shortly before sampling, the cartridge is unwrapped and weighed on an analytical balance. The weight is recorded, the filter support screen is inserted, and the filter assembled. When everything else is ready, the filter is installed for outside/in flow. The sampling is started soon after filter installation to prevent unknown quantitites of gas and particulate from leaking through the valve onto the filter. The combustor side of the valve is continually purged with air or nitrogen to prevent plugging of the probe. If sampling must be delayed after the filter is installed, the filter is pressurized and purged to minimize leakage further.

To start the sampling, the purge flows are shut off, and the hot isolation valve opened. The flow is set on the flow control system to the isokinetic sampling rate. Temperatures, flow, and flow rate are recorded every 15-20 minutes. Most sampling after the tertiary cyclone requires 2 hours to build up sufficient filter cake. Samples taken before the cyclone only require 1 hour. After sampling, the high temperature isolation valve is shut and both purges are again turned on. The filter is allowed to cool and is removed from the system and taken into the lab.

Note: Coulter Counter is a registered trademark of Coulter Electronics Inc.
Isoton and Accuvette are registered trademarks of Coulter
Diagnostics Inc.

In the lab, a one square foot piece of aluminum foil is tared and the filter is opened over the foil. The filter cartridge is carefully removed and any loose particles are dumped onto the foil. The inner support screen is removed from the filter cartridge and the cartridge is wrapped with the foil. The cartridge in foil is weighed on the analytical balance and the tare weight subtracted to obtain a particulate weight. The concentration is obtained by dividing this by the total gas flow and multiplying by a conversion factor. The cartridge with foil wrapping is then stored in a dry box. Unless the filter appeared wet, the filter is reweighed on a random quality control check basis only.

#### Fine Particle Sizing by Coulter Counter

Most fine particle sizing in the miniplant is done with the Coulter Counter Model TAll with either 30  $\mu m$  and/or 100  $\mu m$  aperture probes. Material with a fair fraction of particles larger than 45  $\mu m$  is prescreened with a sonic sifter through the appropriate screens. The two distributions are then combined.

#### Equipment --

The Coulter Counter used during these studies is a Model TAll. Modifications have been made to decrease outside disturbances and increase precision. The instrument rests on a large well grounded metal plate. The sample stand, next to the instrument is completely enclosed in a Faraday cage constructed of a lucite box covered with wire mesh. The sample stand and the Faraday cage also rest on the grounded plate. The sample vacuum pump is outside the cage, separately isolated and grounded. The electric stirrer motor supplied with the sample stand was replaced with an pneumatic stirrer. This equipment configuration has given very repeatable performance and has correlated well with other units in other labs.

The electrolyte use in this study is Isoton II. The Isoton (1% NaCL in distilled water) is continuously filtered through 0.45 and 0.2  $\mu m$  millipore filters so that it is ultra clean. This has achieved 12 second background counts (30  $\mu m$  aperture) as low as 10.

The sifter used is an ATM sonic sifter with an assortment of sieves from 5600 to  $45~\mu m$  (3-1/2 to 325 U.S. Mesh). Various screens are used to bracket the expected size distribution.

#### Procedure --

A small piece of the Balston filter cake is carefully removed to avoid contamination by the filter substrate. The filter cake or the minus 45  $\mu m$  (-325 U.S. Mesh) material from the sonic sifter is used as the sample. The sample (~ 0.1 g) is placed in a small plastic vial of known volume called an Accuvette. A cationic (Type 3A) dispersant is added (~10 drops) until the solid is wetted. Ultra clean Isoton II is added to fill the Accuvette half way. The mixture is placed in an ultrasonic bath for 5-15 minutes. While the Accuvette is in the ultrasonic bath, the Coulter aperture and sample beaker (500 ml) are rinsed several times with ultra clean Isoton II. Several rinses are required to reduce the background (no sample) count to less than 100 in 12 second accumulation with the 30  $\mu m$  aperture (less than 30 with the

100  $\mu$ m aperture). After a low background count is achieved, the Isoton II is retained in the sample beaker and several drops of sample are placed in the beaker. Sample is added while stirring until the proper sample concentration is reached (~2.5 to 5%). Coulter accumulation and operation from this point are the same as in the operations manual.

Magnetic Particulates --

The above procedure works well for most ordinary PFB particulates. Occasionally particulate samples are found that are fairly magnetic. The test for magnetism is carried out with a simple pocket magnet. When magnetism is evident, further standardization of technique and care must be exercised to obtain a true distribution. Failure to dilute the sample properly may lead to magnetic particle agglomeration and settling. The idea is simply to deagglomerate and dilute the sample enough that agglomeration due to magnetism is negligible.

For this procedure, a working sample of 0.100 g is used. This sample is wetted with 25 drops of dispersant (Type 3A) in a 500 ml beaker. This sample is diluted with 300 ml of ultra clean Isoton II. The beaker with its contents is placed in the ultrasonic bath for 5-15 minutes. Once the sample is well dispersed, a 25 ml aliquot is taken with vigorous mixing of the sample. This aliquot is added to the 440 ml of ultra clean Isoton II in the 500 ml beaker on the sample stand. This is sufficient for a good accumulation with the Coulter Counter. The approximate concentration of the sample is 18 micrograms sample/ml.

This procedure has been found to give repeatable size distribution. It is not as sensitive to slight delays in sampling as the other less rigid procedure. Successive Coulter Counter accumulations do not tend to finer size distribution as happened with the other procedure.

# Mass Balance Calculations

Some flue gas streams such as the second cyclone inlet cannot be sampled for particulates directly with a filter. A calculation and sampling procedure has been developed to obtain particle size and concentration information by mass balances. These calculations have been used to determine second cyclone inlet and outlet concentration as well as second and third cyclone efficiencies.

Usually in the operation of the miniplant, Balston filter samples of the flue gas after the third cyclone are taken at planned intervals. Lock hopper dumps of the cyclones during or immediately after the filter sampling period are also sampled. The Balston filter catch, second and third cyclone dump samples are analyzed for particle size distributions. The amount of material in the lock hopper is averaged over 3-5 of the bihourly dumps. In this way, lock hopper hang up and small inconsistencies are removed. The various steps of the differential size distribution which is obtained from the size analysis are multiplied by the average dump weight and divided by the gas flow rate. The various steps of the Balston filter differential size distribution are

multiplied by the particulate loading measured. All size distribution steps are on the same weight/flow basis and a size differentiated mass balance may be completed starting with the third cyclone. Once concentrations in all streams are known, cyclone efficiencies may be calculated directly. The only assumptions in this calculation procedure are that loadings are fairly constant and that there is no change in particle size through the flue gas system.

#### APPENDIX E

#### ANALYTICAL TECHNIQUES

#### Analysis of Solids

Solids from combustion and regeneration runs were analyzed for  $S0_4^{-2}$ ,  $C0_3^{-2}$ ,  $Ca^{+2}$ ,  $Mg^{+2}$ ,  $Na^+$ , carbon and total sulfur. The analytical techniques that were used are described below.

s0 <sub>4</sub>	-2

 The sample was treated with acidic BaCl<sub>2</sub> solution. The BaSO<sub>4</sub> precipitate was weighed.

co<sub>3</sub>-2

- HCl was added to an acidified sample. The solution was stripped with  $N_2$  and the gas passed through drierite,  $CuSO_4$  and ascarite.  $CO_3^{-2}$  was determined from the weight gain of the ascarite.

Ca+2 - before Dec. 1978 - Mg+ Na+

 The sample was digested by heating vigorously in a medium of perchloric acid/nitric acid. The determination of Ca, Mg and Na was made by atomic absorption.

Ca+2 - after Dec. 1978 Mg

- The sample was fused with  $Na_2CO_3$  at 950°C, then dissolved with HCl. The determination of Ca and Mg was made by atomic absorption.

Total - before Aug. 1978 Sulfur - (Dietert Sulfur Method) - The sample was combusted in an oxygen atmosphere at 1250°C. The SO<sub>2</sub>-SO<sub>3</sub> products in the effluent gas were analyzed by an automatic Leco titrator.

Total - after Aug. 1978 Sulfur The sample was added to a  $V_2O_5$  catalyst and combusted in an oxygen atmosphere in an induction furnace at  $1650^{\circ}$ C. The  $SO_2$  formed is selectively measured by an infra-red detector. A Leco IR-23 sulfur analyzer was used (ASTM D-1552).

Total - before Aug. 1978 - Carbon

(Carbon on Catalyst Method) - The sample was combusted in an oxygen atmosphere at 1200°C. The CO<sub>2</sub> evolved was determined from the weight gain of ascarite.

Total - after Aug. 1978 Carbon The sample was combusted in an industion furnace at  $1650^{\circ}\text{C}$  followed by removal of sulfur, conversion of any CO to  $\text{CO}_2$ , trapping of  $\text{H}_2\text{O}$  and trapping of the  $\text{CO}_2$  on molecular sieves. In the next cycle, the molecular sieves are heated to  $300^{\circ}\text{C}$  where the  $\text{CO}_2$  is expelled and measured by thermal conductivity detection. A Leco WR-12 carbon analyzer was used.

## Analysis of Flue Gas by Wet Chemical Analysis

s0<sub>2</sub>

SO<sub>3</sub> - before July 1978 - The amount absorbed by an 80% isopropanol solution was determined titrimetrically using 0.01N barium perchlorate as the

titrant and thorin as the indicator.

SO<sub>2</sub> - after July 1978

- SO<sub>3</sub> was collected as H<sub>2</sub>SO<sub>4</sub> by using a Goksoyr-Ross controlled condensation coil (maintained at 60°C above the water dew point). The amount of SO<sub>3</sub> is determined by SO<sub>4</sub> titration with 0.01N barium perchlorate as the titrant and thorin as the indicator.

the indicat

- The amount absorbed by a 3% hydrogen peroxide solution was determined titri-metrically using 0.01N barium perchlorate as the titrant and thorin as the indicator.

#### APPENDIX F

#### COMPARISON OF SO<sub>2</sub> MEASUREMENTS BY UV. GC. WET CHEMISTRY

SO2 concentration in the miniplant flue gas was continuously analyzed by a UV instrument. Periodic measurements of SO2 concentration were also made by analysis of grab samples by the standard wet chemistry method (barium perchlorate to thorin end point) and by GC. The results from the UV analysis were consistently lower than the wet chemistry and GC results. The UV results averaged 14% lower than wet chemistry and 29% lower than GC results. Assuming the UV measurements were truly low by 14%, the impact on the measured SO2 retention results would only be two percentage points at the 85% SO2 retention level and one percentage point at the 95% SO2 retention level. This degree of bias, if it did occur, would have a negligible effect on the estimate of dolomite requirements needed to retain 85 to 95% of the SO2.

The difference between UV and GC results was not considered important, since the wet chemistry method is generally regarded as the standard method for  $SO_2$  measurement. Therefore, it would be concluded that the GC results are 15% higher than the wet chemistry results. A comparison of UV, wet chemistry and GC results is given in Table F-1.

TABLE F-1. COMPARISON OF SO<sub>2</sub> MEASUREMENTS BY UV, GC, WET CHEMISTRY

			•	_ % Diff	erence
Run <u>No.</u>	<u>UV</u>	02 (ppm <u>WC</u>	GC GC	UV-WC	UV-GC UV
69	72	57		+21	
•	62	79	-	-27	
	89	-	118		-33
70	131 23	136 24	-	-4	
	23 106	- -	127	-4 	-20
	90	-	137		-52
71	85	104	•	-22	
	269 279	251 315	-	+7 -13	
	49	-	307	-13	-527
	<b>6</b> 02	-	973		-62
	201	-	257		-28
72	140	191	100	-36	
	140 82	-	188 103		-34 -26
	54	-	55		-2
73	3	14	-	<b>-</b> 367	
	5 7	-	3 7		+40
78	360	437	<b>,</b>	-21	0
70	150	195	-	-21 -30	
	108	180		-67	
79	319	323	-	-1	
	220	256	-	-16	
80	310	335	-	-8 -00	
0.1	59	116	-	-100	
81	322	324	-	-1	
99	228	208	-	+9	
100	320	305	-	+5	
102	10 14	8 3	-	+20	
102			-	+78	
103	132 28	88 146	-	+33 -421	
	412	406	-	+1	
	23	64 227	•	-178	
	98 210	327 210	-	-234 0	
	_ , <del>_</del>	-· <del>-</del>		•	

TABLE F-1 (CONT'D). COMPARISON OF SO<sub>2</sub> MEASUREMENTS BY UV, GC, WET CHEMISTRY

Run	9	02 (ppm)	)	% Dif	ference UV-GC
No.	UV	WC	GC	UV	UV
104	134 23	158 36	-	-18 -56	
105	44	17	-	+61	**
Averages				14+49(1)	29+20(2)

<sup>(1)</sup> Three Points Excluded = -367, -421, -234%

<sup>(2)</sup> One Point Excluded = -527%

 $\label{eq:appendix} \mbox{ APPENDIX } \mbox{ G} \\ \mbox{RESULTS OF FLUE GAS $SO_3$ ANALYSES} \\$ 

Run No .	SO <sub>3</sub> Analytical Method	Concentration (ppm)	Run No.	S0 <sub>3</sub> Analytical Method	Concentration (ppm)
62	Method 8	1.8 0.9	78	Controlled Cond.	0.6 9.9
65	Method 8	3 <b>4.</b> 2			12.8 0.4
67	Method 8	19.6 26.5	79	Controlled Cond.	1.1 11.1
69	Method 8	3.6 3.6	80	Controlled Cond.	3.2 0.3
70	Method 8	24.4 10.6	81 99	Controlled Cond. Controlled Cond.	•
71	Method 8	10.0 30.0 213	1 00 1 02	Controlled Cond. Controlled Cond.	
72 73 74 75 76	Method 8 Method 8 Method 8 Method 8 Controlled Co	2.5 2.5 0 23	103	Controlled Cond.	0 0 21.3 1.7 6.7 29.8
			104	Controlled Cond	. 0.5 O
			105	Controlled Cond	

# Averages (1)

All Points - 8.6  $\pm$  11 ppm Method 8 Results - 12  $\pm$  12 ppm Controlled Cond. - 6  $\pm$  9 ppm

<sup>(1)</sup> Two Samples Excluded - Run 71 (213 ppm), Run 100 (73 ppm)

APPENDIX H
REGENERATOR GC ANALYSES

Run/		Dr	y Off Gas Sam	p1 <u>e</u>	
Sample No.	H <sub>2</sub> S (ppm)	CS <sub>2</sub> (ppm)	S0 <sub>2</sub> (%)	COS (ppm)	RSH (ppm)
102 #1 #2 #3 #4	15 9 < 1 75	< 1 < 1 < 1 < 1	0.32 0.36 0.22 0.16	6 < 1 < 1 8	<1 <1 <1 <1
103 #1 #2 #3 #4 #5 #6 #7 #8	< 0.1 230 12 2 2 < 1 < 0.1 < 0.1 < 0.1	< 1 < 0.1 < 1 < 1 < 1 < 0.1 < 1	0.07 0.43 0.34 0.37 0.42 0.44 385 ppm 0.20 125 ppm	< 0.1 2 3 < 0.1 2 < 0.1 < 0.1 < 0.1	< 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1 < 0.1
105 #1 #2 #3 #4 #5 #6 #7	< 0.1 115* 77 180  100 93	< 0.5 110* 100   < 0.5	35 ppm 0.30* 0.35 0.16 770 ppm 0.25 0.32	< 0.1 30* 26   28	< 0.1 < 0.1* < 0.1   < 0.1

<sup>\*</sup> Questionable Results

APPENDIX I

MASS SPECTROGRAPHIC ANALYSES OF SOLID SAMPLES OF RUN 69

	Illinois Coal	Filter Part	Pfizer Dolomite	Initial Bed	Final Bed	Bed Overflow	2° Cyclone	Bed Probe
Element	Conc. µg/g	Conc. µg/g	Conc. µg/g	Conc. µg/g	Conc. µg/g	Conc. µg/g	Conc. μ <b>g/g</b>	Conc. µg/g
Li	50	30	20	10	30	10	10	10
Be	3	3	0.01	0.5	0.5	0.8	2	0.8
В	400	125	5	100	100	100	100	100
F	< 2	50	34	17	30	17	17	17
Na	50ื้00	3500	350	1000	3500	1000	1000	350
Mg	~1%	~12%	~12%	~12%	~12%	~12%	~12%	~16%
AĨ	~10%	~2%	500	~1%	~1%	500C	~2%	~1%
Si	~8%	~3%	2000	~2%	~2%	~2%	~2%	~1%
P	300	120	0.2	120	120	120	120	60
S	~7%	~2%	200	~2%	~2%	~2%	~2%	~2%
Č1	2000	40	120	40	40	80	160	80
ĸ	~1%	~1%	300	1000	2000	500	3000	2000
Ca	3000	~12%	~12%	~12%	~12%	~20%	~12%	~20%
Sc	< 20	< 20	< 2	< 2	< 2	< 2	< 2	< 2
Ti	210	3000	15	300	400	300	1000	1000
Ÿ.	130	200	2	30	30	40	100	40
Cr	130	80	12	40	40	120	40	20
Mn	200	40	40	40	40	120	40	40
Fe	~7%	6000	180	6000	6000	6000	6000	6000
Co	30 ~	5	3	2	1	2	3	2
NÍ	200	400	3	100	100	100	100	100
Ĉu	70	35	4	20	20	35	35	35
Zn	30	10	10	20	10	4	10	10
Ga	30	8	0.2	2	2	3	3	3
Ge	30	15	0.1	15	15	40	10	<b>6</b> 0
As	10	3	8	8	2	8	8	24
Se	< 5	< 1	< 0.1	< Î	< ]	< 0.03	< 0.03	< 0.05
Br	0.3	0.5	5	2	2	2	2	2
Rb	35	70	2	10	10	10	34	7
Sr	35 70	2000	150	150	150	300	300	300

APPENDIX I (CONT'D)

MASS SPECTROGRAPHIC ANALYSES OF SOLID SAMPLES OF RUN 69

Element	Illinois Coal Conc. µg/g	Filter Part Conc. µg/g	Pfizer Dolomite Conc. µg/g	Initial Bed Conc. µg/g	Final Bed Conc. µg/g	Bed Overflow Conc. µg/g	2° Cyclone Conc. μg/g	Bed Probe Conc. µg/g
Sr	70	2000	150	150	150	300	300	300
Ÿ.	14	30	0.4	4	4	5	5	5
Żr	20	140	0.1	8	40	14	40	14
Nb	< 7	14	0.1	ì	4	4	4	4
Мо	12	100	2	8	8	8	8	16
Ru	< 1	< 0.2	< 0.2	< 0.2	< 0.3	< 0.5	< 0.5	< 0.5
Rh	< 0.4	< 0.1	< 0.04	< 0.1	< 1	< 1	< 0.4	< 0.6
Pd	< 1	< 0.4	< 0.4	< 0.4	< 0.4	< 1	< 1	< 1
Ag	< 0.3	< 0.2	< 0.1	< 0.04	< 0.04	< 0.1	< 0.2	< 0.1
Cď	2	< 0.5	< 0.2	< 0.2	< 0.2	< 0.3	< 0.7	< 0.3
In	< 0.2	< 0.2	< 0.2	< 0.3	< 0.2	< 0.5	< 1	< 0.3
Sn	1	0.8	0.8	0.8	2	2	2	8
Sb	4	0.4	0.2	0.4	0.4	1	1	1
Te	< 0.5	< 0.1	< 0.05	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
I	2	< 0.07	< 0.07	< 0.07	< 0.07	< 0.07	< 0.2	< 0.07
Cs	2	10	0.1	2	3	3	10	1
Ba	100	360	4	60	60	60	140	140
La	12	20	0.4	6	6	12	12	7
Се	20	60	1	8	8	28	28	14
Pr	4	4	0.05	0.3	1	1	3	1
Nd	12	25	0.3	0.1	1	10	15	10
Sm	1	< 2	< 0.2	< 1	< 1	< 4	< 3	< 3
Eu	0.5	1	< 0.03	< 0.2	0.3	< 0.3	< 0.5	< 0.5
Gd	< 2	3	< 0.2	< 2	< 2	< 3	< 2	< 2
Tb	0.4	0.5	< 0.05	< 0.5	0.5	< 0.05	< 0.6	< 0.2
Dy	3	5	< 0.2	< 0.5	1	< 2	< 3	< 1
Ho	0.3	1	< 0.05	< 0.1	0.1	< 0.3	< 0.3	< 0.2
Er	1	7	< 0.2	< 0.3	0.4	< 0.4	< 0.3	< 0.4
Tm	7	< 0.2	< 0.06	< 0.06	0.1	< 0.1	< 0.1	< 0.1
Yb	1	< 1	< 0.05	< 0.2	0.2	< 0.3	< 0.3	< 0.3

APPENDIX I (CONT'D)

MASS SPECTROGRAPHIC ANALYSES OF SOLID SAMPLES OF RUN 69

	Illinois	Filter	Pfizer	Initial	Final	Bed		
	Coal	Part	Dolomite	Bed	Bed	Overflow	2° Cyclone	<b>Bed Probe</b>
	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.	Conc.
<u>Element</u>	<u>hā\a</u>	_րց/ց_	<u>μ<b>g/g</b></u>	<u> </u>	μ <b>g/g</b>	<b>րց/ց</b>	µ <b>g/g</b>	<u> </u>
Lu	0.4	< 0.2	< 0.03	< 0.06	0.1	< 0,2	< 0.2	< 0.2
Hf	< 5	< 3	< 0.6	< 0.6	< 0.6	< 0,6	< 0.6	< 0.6
Ta	< 2	< 0.2	< 0.2	< 0.2	< 0.6	< 0.2	< 0.6	< 0.6
W	< 2	0.3	< 0.1	< 0.2	< 0.2	< 0,2	< 0.2	< 0.2
Re	< 1	< 0.2	< 0.06	< 0.2	< 0.06	< 0.2	< 0.2	< 0.2
0s	< 2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Ir	< 1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1	< 0.1
Pt	< 2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2	< 0.2
Au	< 1	< 0.07	< 0.07	< 0.07	< 0.07	< 0.07	< 0.07	< 0.07
Hg	< 10	< 2	< 1	< 1	< 1	< 1	< 1	< 3
ΤĬ	< 2	14	< 0.05	< 0.05	< 0.05	< 0.3	1	< 0.3
Pb	< 2	32	0.1	0.1	0.1	0.1	14	0.1
Bf	< 1	0.2	< 0.03	< 0.03	< 0.03	< 0.03	< 0.2	< 0.1
Th	< 2	20	0.2	1	4	4	5	4
U	< 1	12	0.2	1	4	4	4	4

APPENDIX J
MINIPLANT COMPONENT MASS BALANCES

## Weight Percent

Run No.	Total Mass	<u> </u>	<u>C Total</u>	Ca	Reactive 02	Mg.	Solids <u>Inorganics</u>
61	100.34	77.07	87.89	313.29	92,12	191,19	127.87
62.1	100.97	33.83	88.90	173.99	91.93	84 .62	205.09
62.3	102.05	293.19	90.96	173,33	93.39	04,02	264.21
63	101.15	227.28	98.13		97 <b>.</b> 77		189.20
67.1	99.52	82.94	109,82	96.63	91.64	88.03	101.50
67.2	99.62	102.83	110.60	95.83	96.05	90.05	101.98
67.3	99.80	94.56	86.39	115.42	81.02	113.82	-
67.4	99.71	90.37	98.27		91,19		106.55
68	100.65			104.81		100.42	103.27
69		155.56	105.17	123.27	102.83	131.83	146.42
70	99.33	111.35	104.94	89.09	100.27	81.45	98.93
70 71	99.39	102.50	126.16	88.03	111,31	83.65	100.43
	99.83	123.92	117.37	122.35	102.69	104.39	110.83
72.1	99.80	78.03	157.59	67.59	125.74	59.95	69.56
73.1	99.38	125.89	122.13	88,50	105.14	75.93	94.95
74.1(1)	97.68	66,39	136.44	68,13	118.07		70.61
74.2(1)	98.89	68,20	136.45	125,22	117.83		90.59
76	99.59	97.54	119.23	133.60	100.49	81.24	83,08
77	99.91	128.51	115.26	126.76	102.17	99.75	111.77
99.2	91.75	83.26	88.55	75.73	84.43	84.92	
99.3	93.17	67.00	92.57	63,61	80.10	75.12	
99.4	94.80	62.82	110.97	65.93	87.93	60.65	
99.5	96.08	87.18	93.77	93.43	86.29	86.48	

#### APPENDIX J (CONT'D)

#### MINIPLANT COMPONENT MASS BALANCES

#### Weight Percent

Run No.	Total Mass	<u> </u>	C Total	Ca	Reactive 02	Mg	Solids <u>Inorganics</u>
100.2	93.88	61.44	81,14	110,71	82,69	93.35	
100.4	96.58	62.25	108.30	44.75	82.41	35.59	
102(2)	99.88	101.70	93.92	174.92	80.44		130.00
103.0(2)	99.86	120,16	97.96	118.27	75.91		90.83
103.1(2)	99.89	105.91	100.51	118.94	79.88		76,62
103.2(2)	99.79	74.32	102.34	198.85	77.99		104.35
103,3(2)	99.87	47.65	101.32	70.44	81.43		81,55
105.2(2)	100.60	101.16	92.30	65.22	79.99		78.41
Average + 1s	98.79 <u>+</u> 2.48	101.16 <u>+</u> 51.32	105.85 <u>+</u> 17.33	111.90 <u>+</u> 53.65	93.37 <u>+</u> 13.18	91 .11 <u>+</u> 31 .1	114.11 <u>+</u> 46.15

Pfizer Dolomite Used for All Runs Except as Noted.

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(2) Grove Limestone - Combined Combustor-Regenerator Operation

<sup>(1)</sup> Grove Limestone

#### APPENDIX K

## MINIPLANT RUN OBJECTIVES

Run No	Major Objective
60	GBF - ejector evaluation (ejector replaced plunger type blow back nozzle)
61	GBF - ejector evaluation
62 vary filter media and	GBF - coarse alumina filter media/large inlet retaining screens
63 blow back conditions	GBF - speculite filter media
64	GBF - speculite/natural gas injection used
65	GBF - internal baffles vs. screens in filter bed media retention
66	DOE Fireside Corrosion/Erosion Test - 1st attempt
67	DOE Fireside Corrosion/Erosion Tests - 100 Hr Shakedown
68-75	High SO <sub>2</sub> retention
76	High SO <sub>2</sub> retention using fine particle dolomite
77	Testing of unit modifications for DOE materials corrosion test program - bed solids overflow system
78-81	DOE Fireside Corrosion/Erosion Tests, 78 (250 hours), 79 (100 hours), 80 (215 hours), 81 (170 hours)
82-96	Test Acurex ceramic bag filter
97-98	Shakedown of auxiliary coal feed system
99	SO <sub>2</sub> response to step change in coal sulfur content
100	SO <sub>2</sub> response to step change in Ca/S ratio
101	Shakedown of regenerator
102-103	Sorbent regeneration
104	Acurex - electrostatic precipitator
105	Sorbent regeneration; comprehensive analysis
106	DOE Fireside Corrosion/Erosion Tests (265 hours)
107-108	Acurex mobile bag house
109-114	3° cyclone efficiency tests
115	Slipstream GBF - Exxon Mark IV

APPENDIX L. MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

	61	62.1	62.3	63	64
Operating Conditions	8/24/77	9/1/77	9/1/77	9/15/77	9/29/77
Run Length, hrs.	5.0	2.67	5.0	10.5	10.5
Pressure kPa	795	795	795	790	850
Air Flow Rate, m <sup>3</sup> /min	17.6	17.6	17.6	17.6	15.6
Temperature Gradient, °C/m	4.1	8.1	6.5	8.2	5.5
Avg. Bed Temperature, °C	940	929	932	931	939
Superficial Velocity, m/sec	1,77	1.77	1.77	1.80	1.48
Settled Bed Height, m		1 0		1.0	1 7
Initial	1.4	1.8 	1.8	1.8	1.7
Final	1.5	3.7	4.2	1.7 4.1	2.0 3.6
Expanded Bed Height, m	3.6 103	109	103	102	95 <b>.</b> 6
Coal Feed Rate, kg/hr	0.75	1.15	0.00	0.00	0.00
Ca/S Molar Feed Ratio-Set (1) Ca/S Molar Feed Ratio-Calc. (2)					
Excess Air, %	36,05	37.7	38.3	28.9	14.6
Sorbent	PD	PD	PD	PD	PD
Coal	CH	СН	СН	СН	ĊĤ
Flue Gas Emissions					
SO <sub>2</sub> , ppm			126	632	593
SO <sub>2</sub> , ppm NO <sub>X</sub> , ppm CO, ppm	115	160	89	93	87
CO, ppm	156	*	*	162	*
CO <sub>2</sub> , %	11.6	11.9	11.7	12.2	14.7
02, %	5.6	5.8	5.3	6.3	2.7
Results					
SO <sub>2</sub> Retention, %			87	37	43
Ca Sulfation, %	52	29	35	38	33
Lb SO <sub>2</sub> /M BTU			0.28	1.41	1.25
Lb NO <sub>X</sub> /M BTU	0.18	0.24	0.14	0.15	0.13
X'					

PD = Pfizer Dolomite

CH = Champion Coal

GL = Grove Limestone

<sup>\* =</sup> No analyzer

<sup>(1)</sup> Ca/S determined based upon settings on coal and sorbent feed system.

<sup>(2)</sup> Ca/S calculated based upon level of sulfation of spent bed material.

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	65	66	67.1	67.2	67.3
	10/13/77	11/29/77	12/12-13/77	12/14/77	12/15/77
Run Length, hrs.	7	17	28	25	10
Pressure, kPa	910-960	930	930	930	930
Air Flow Rate, m <sup>3</sup> /min	17.4-20.2	18.6	19.8	19.8	18.7-19.3
Temperature Gradient, °C/m	6.52	36.4	40.1	30.1	31.5
Avg. Bed Temperature, °C	937	918	915	915	875-915
Superficial Velocity, m/sec Settled Bed Height, m Initial Final	1.45-1.66  2.0	1.6  2.0	1.7	1.7	1.6
Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	3.4	3.2	3.3	3.4	3.2
	95	130	118	130	125
	0.00	1.25	1.25	1.25	1.25
			1.8	1.2	1.5
	41.7	10	19	14	14
	PD	PD	PD	PD	PD
	CH	ILL	ILL	ILL	ILL
Flue Gas Emissions					
SO <sub>2</sub> , ppm	80-380	370	736	725	718
NO <sub>X</sub> , ppm	13-98	20	100	65	65
CO, ppm	20-50	205	300	160	312
CO <sub>2</sub> , %	1-13	12	14	15	12
O <sub>2</sub> , %	4.6	1.9	3.0	2.5	2.5
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>X</sub> /M BTU	41 	89  0.68 0.03	74 41 1.60 0.16	77 66 1.43 0.09	77 50 1.41 0.09

PD = Pfizer Dolomite

ILL = Illinois Coal No. 6 CH = Champion Coal

#### APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

O Die Oudibles	67.4	68	69 273677	70	71 446479
Operating Conditions	12/16/77	3/8/78	3/16/77	3/22/78	4/6/78
Run Length, hrs.	37	12	11	11.75	13
Pressure kPa	930	930	930	930	930
Air Flow Rate, m <sup>3</sup> /min	19.3	18.4	19.0	20.6	19.0
Temperature Gradient, °C/m	38	16	17.1	28	35
Avg. Bed Temperature, °C	875	946	947	936	933
Superficial Velocity, m/sec	1.6	1.7	1.8	1.9	1.72
Settled Bed Height, m					
Initial			7 0		
Final	1.8	1.8	1.8	1.8	2.3
Expanded Bed Height, m	3.0	2.8	3.0	3.0	3.8
Coal Feed Rate, kg/hr	115	127	132	127	122
Ca/S Molar Feed Ratio-Set	1.25	2.0	1.5	1.5	1.52
Ca/S Molar Feed Ratio-Calc.	1.9	1.6	1.5	1.4	1.7
Excess Air, %	31	17 pp	14 PD	23 PD	12.6 PD
Sorbent	PD	PD	ILL	ILL	ILL
Coal	ILL	ILL	166	166	166
Flue Gas Emissions					
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO <sub>x</sub> , ppm	739	50	197	94	61
NO2, ppm	105	79 *	56	81	38 *
CO, ppm	200	*	*	*	
co <sub>2</sub> , %	12	16	16	17	16.9
02, %	~5	3.2	2.6	4.0	2.4
Results					
SO <sub>2</sub> Retention, %	74	98	94	96	98
Ca Sulfation, %	38	60	64	70	56
Lb SO <sub>2</sub> /M BTU	1.60	0.09	0.37	0.20	0.12
Lb NOX/M BTU	0.61	0.11	0.07	0.12	0.05
- X.					

ILL = Illinois Coal No. 6
PD = Pfizer Dolomite
\* CO Analyzer Malfunctioned

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	72.1	72.2	73.1	73.2	74.1
	4/13/78	<u>4/13/78</u>	<u>4/19/78</u>	4/19/78	4/28/78
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min Temperature Gradient, °C/m Avg. Bed Temperature, °C	9.3	2.66	8.5	3	4.5
	915	694	930	930	930
	20.4	17.0	19.6	19.6	19.2
	11	12	20	13	40
	943	940	939	946	933
Superficial Velocity, m/sec Settled Bed Height, m Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	1.84  3.7 116 1.79 1.8 9.37 PD OH	2.02 2.2 3.9 112 1.79 1.3 1.02 PD 0H	1.78  3.8 117 2.14 1.5 9.12 PD OH	1.79 1.7 2.7 119 2.14 1.8 5.43 PD OH	1.68 2.2  3.7 113 7.6 6.8 16.5 GL OH
Flue Gas Emissions	•	<b>U</b>	<b></b>	VIII	Oil
SO <sub>2</sub> , ppm	60	23	4.0	6.0	1.0
NO <sub>x</sub> , ppm	28	50	51.0	32.0	79.6
CO <sub>x</sub> , ppm	*	*	*	*	*
CO <sub>2</sub> , %	22.4	18.8	17.9	17.5	19.5
0 <sub>2</sub> , % <u>Results</u>	2.1	0.2	2.0	1.2	3.3
SO <sub>2</sub> Retention, %	96	99	99	99	99
Ca Sulfation, %	55	76	67	55	14
Lb SO <sub>2</sub> /M BTU	0.13	0.04	0.01	0.01	0.002
Lb NO <sub>x</sub> /M BTU	0.04	0.01	0.07	0.05	0.13

OH = Ohio, Valley Camp PD = Pfizer Dolomite

GL = Grove Limestone

<sup>\*</sup> CO Analyzer Malfunctioned

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	74.2	7 <b>5</b>	76	77	78.1
	4/28/78	5/12/78	6/1/78	6/8/78	6/19-20/78
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec	5.75 930 19.5 27 937 1.71	9.0 922 19.5 30 936 1.71	12.3 922 15 936 1.32	7.5 912 17.5 41 943	34 925 21.2 60 895 1.81
Settled Bed Height, m Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	2.2 112 4.8 8.2 18.9 GL OH	2.2 2.5 3.8 115 3.65 4.1 17.0 GL OH	1.5 2.3 2.0 87.3 1.0 1.2 13.3 PD OH	2.2 2.0 2.3 92.0 1.25 1.2 27.3 PD OH	2.0 3.8 101 1.25 34.1 PD ILL
Flue Gas Emissions  SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm CO <sub>2</sub> , %	31.4	173	422	446	286
	62.7	50	43	47	45
	*	*	*	*	*
	19.0	15.2	16.0	14.3	12.5
	3.7	3.4	2.8	5.1	5.6
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>x</sub> /M BTU	98	90	74	70	88
	11	22	60	60	
	0.07	0.34	0.91	1.05	0.81
	0.10	0.07	0.07	0.08	0.03

<sup>\*</sup> CO Analyzer Malfunctioned

GL = Grove Limestone

PD = Pfizer Dolomite

OH = Ohio, Valley Camp ILL = Illinois Coal No. 6

<sup>\*</sup> CO Analyzer Malfunctioned

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	78.2	78.3	78.4	78.5	78.6
	6/21/78	6/22/78	6/23/78	6/24/78	6/25/78
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec	24	24	24	24	24
	925	925	925	925	925
	21.2	21.2	21.2	21.2	21.2
	53	53	49	46	35
	905	912	916	921	931
	1.83	1.83	1.84	1.85	1.87
Settled Bed Height, m Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	3.5	2.9	3.4	3.1	3.3
	121	118	122	111	148
	1.45	1.45	1.45	1.45	1.45
					1.4
	24.0	23.1	21.1	14.8	24.9
	PD	PD	PD	PD	PD
	ILL	ILL	ILL	ILL	ILL
Flue Gas Emissions  SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm CO <sub>2</sub> , % O <sub>2</sub> , %	231	169	157	460	270
	38	41	42	37	32
	*	*	*	*	*
	13.9	13.7	14.3	14.3	15.3
	4.1	4.0	3.7	2.8	4.3
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>x</sub> /M BTU	90  0.60 0.05	93  0.45 0.04	93  0.40 0.07	79 1.30 0.06	93 69 0.41 0.04

<sup>\*</sup> CO Analyzer Malfunctioned

PD = Pfizer Dolomite

ILL = Illinois Coal No. 6

	Onematine Conditions	78.7 6/26/78	78.8 6/27/78	78.9 6/28/78	78.10 6/29/78	79 7/31 <b>-</b> 8/4/78
	Operating Conditions Run Length, hrs.	24	24	24	24	100
	Pressure, kPa	925	925	925	925	925
	Air Flow Rate, m <sup>3</sup> /min	21.2	21.2	21,2	21.2	21.4
	Temperature Gradient, °C/m	35	33	33	26	
	Avg. Bed Temperature, °C	936	937	938	895	936
	Superficial Velocity, m/sec Settled Bed Height, m	1.77	1.88	1.77	1.81	1.88
	Initial	-				2.1
	Final				2.0	1.8
	Expanded Bed Height, m	3.3	2.9	2.9	3.8	3.1
	Coal Feed Rate, kg/hr	141	146	145	145	132
	Ca/S Molar Feed Ratio-Set	1.45	1,45	1,45	1.45	1.45
	Ca/S Molar Feed Ratio-Calc.	1.7		15	24 21	24.2
	Excess Air, %	15	11.6	PD	34,21 PD	PD
260	Sorbent	PD ILL	PD ILL	ILL	ILL	ILL
Ö	Coa 1	166	166	166	166	166
	Flue Gas Emissions					
	SO <sub>2</sub> , ppm	113	363	260	286	293
	SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm	28	41	36	15	56
	CO, ppm	*	*	*	*	*
	CO <sub>2</sub> , %	14	15.6	16	12.5	14.6
	02 %	3.2	3.2	2.8	5.5	4.2
	Results					
	SO <sub>2</sub> Retention, %	95	88	90	87	88
	Ca Sulfation, %	55				••
	Lb SO <sub>2</sub> /M BTU	0.27	0.72	0.62	0.81	0.70
	Lb NO <sub>X</sub> /M BTU	0.05	0.06	0.06	0.03	0.10
	••					

<sup>\*</sup> CO Analyzer Malfunctioned

ILL = Illinois Coal No. 6

PD = Pfizer Dolomite

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	80.1 9/13-16/78	80.2 9/16-22/78	81 10/9-16/78	83 11/28/78	84 11 <u>/</u> 29/78
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min	72.3 922 21.4	142.6 922 21.5	171 912 21.4	9.9 900 20.6	6.8 900 20.5
Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	929 1.77	910 1.76	933 1.80	920 1.83	921 1.7
Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	2.1  3.9 126 1.41  27.2 PD ILL	1.3 2.1 130 1.41 1.6 17.8 PD ILL	2.1 1.1 1.9 113 1.56 1.3 28.5 PD ILL	1.8 3.0 119 1.45  37 PD ILL	2.8 4.6 128 1.45  23 PD ILL
Flue Gas Emissions					
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO <sub>x</sub> , ppm CO <sub>2</sub> , % O <sub>2</sub> , %	217 78 * 13.0 5.0	152 121 * 12.4 3,5	296 86 * 13.2 5.1	120  11,5 6	115 92  14.1 4.2
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>X</sub> /M BTU	93  0.40 0.10	95 59 0.27 0.16	90 69 0.61 0.13	  0.22	95  0.28 0.16

<sup>\*</sup> CO Analyzer Malfunctioned

PD = Pfizer Dolomite

ILL = Illinois Coal No. 6

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	85	86	87	88	89
	11/30/78	12/1/78	12/4/78	12/5/78	12/6/78
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min	6	9	11.3	9.5	10.7
	900	900	900	900	900
	21.0	20.8	20.8	20.6	19.2
Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	918 1.7	921 1.7	918 1.7	918 1.7	917 1.7
Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr	2.8	2.9	2.9	2.0	2.2
	4.6	4.7	4.7	3.3	3.6
	132	125	122	115	101
	1.45	1.45	1.45	1.45	1.4
Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	21	24	18	28	23
	PD	PD	PD	PD	PD
	ILL	ILL	ILL	ILL/V	V/CH
Flue Gas Emissions SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm	146	50	150	145	155
	100	100	105	105	150
CO; ppm CO <sub>2</sub> , % O <sub>2</sub> , % Results	 4.1	13 4.3	16 3.7	11.4 5.1	14 4.8
SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU	94	99 <sup>+</sup>	93	93	85
	0.35	0.12	0.38	0.39	0.37
Lb NOX/M BTU	0.17	0.18	0.19	0.20	0.26

PD = Pfizer Dolomite
ILL = Illinois Coal No. 6

ILL/V = Illinois/Valley Camp
V/CH = Valley Camp/Champion

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	90	91	92	93	94
	12/7/78	12/8/78	12/11/78	12/12/78	12/13/78
Run Length, hrs.	6.5	10.5	7.2	10.3	7.0
Pressure, kPa	900	900	900	900	900
Air Flow Rate, m <sup>3</sup> /min	19.5	19.5	19.1	19.3	18.1
Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	918 1.6	916 1.6	918 1.7	910 1.7	914 1.6
Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set	1.8	1.9	1.7	1.8	1.8
	2.9	3.1	2.8	2.9	3.0
	101	104	104	107	96
	1.87	1.5	1.5	1.5	1.5
Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	28 PD CH	23 PD CH	21 PD CH	24 PD CH	23 PD CH
Flue Gas Emissions SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm	20	65	50	85	115
	40	100	115	125	108
CO <sub>2</sub> , % O <sub>2</sub> , %	65 12 5	75 11.2 4.7	14 4.4	90 11 4.5	100 12.2 4.4
Results SO <sub>2</sub> Retention, % Ca Sulfation, %	98 	94 ·	95 	92 	89
Lb SO <sub>2</sub> /M BTU	0.05	0.15	0.12	0.19	0.27
Lb NO <sub>X</sub> /M BTU	0.07	0.17	0.19	0.21	0.19

PD = Pfizer Bolomite CH = Champion Coal

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	95 12/15/78	96 12/18/78	97 1/23/79	98 1/24/79	99.1 1/25/79
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min	5.5 900 18.1	21.0 900 17.9	5.5 912 17.7	6.25 922 18.1	4.0 912 17.4
Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	914 1.6	927 1.6	64 902 1.53	64 904 1.55	74 895 1.41
Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr	1.8 3.0 98	2.3 3.8 103	2.1 1.8 3.0	  3.1 109	 3.0 90.3
Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, %	1.5  21	1.5  36	116 *  38.4	*  37.3 PD	1.40  34.9 PD
Sorbent Coal Flue Gas Emissions	PD CH	PD CH	PD *CH/ILL	*CH/ILL	CH
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm CO <sub>2</sub> , % O <sub>2</sub> , % Results	290 102 90 8.9 4.2	250 100 40 14 6.2	** 124	** 111 ** 7.8 6.2	203 120 435 9.7 6.0
SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>x</sub> /M BTU	73 0.69 0.17	78 0.55 0.16	  0.18	  0.17	83 0.51 0.21

PD = Pfizer Dolomite

\*CH/ILL = Champion and Illinois Coals used - Champion Analyses used when necessary.

\*\* = SO<sub>2</sub> Response Test - No Steady Emissions

CH = Champion Coal \* = Switching between Champion (Ca/S = 1.4) and Illinois (Ca/S = 0.76) coals

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	99.2	99,3	99.4	99.5	99.6
	1/25/79	1/25/79	1/25/79	1/25-26/79	1/26/79
Run Length, hrs. Pressure, kPa Air Flow Rate, m³/min Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	3.0	3.67	4.0	7.0	6.0
	912	912	912	912	912
	16.9	16.9	16.9	16.8	16.5
	62	56.2	58	63	62
	911	910	911	913	913
	1.38	1.37	1.37	1.37	1.35
Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	3.0	3.3	3.7	3.9	3.9
	93.0	99.4	99.4	82.8	81.8
	1.40	0.76	0.76	1.40	1.40
	1.2	0.95	0.93	1.1	
	33.9	30.1	30.0	33.1	32.2
	PD	PD	PD	PD	PD
	CH	ILL	ILL	CH	CH
Flue Gas Emissions  SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO, ppm CO <sub>2</sub> , % Results	194	740	721	171	210
	125	110	113	121	118
	61	34	15	11	6
	10.0	9.39	11.3	10.5	11.2
	5.9	5.6	5.4	5.8	5.7
SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>X</sub> /M BTU	85	70	70	85	82
	70	73	75	75	
	0.46	1.87	1.82	0.45	0.55
	0.21	0.20	0.21	0.23	0.22

PD = Pfizer Dolomite

CH = Champion Coal ILL = Illinois Coal No. 6

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	99.7 1/26/79	100.1 1/30/79	100.2 	100.3 1/30-31/79	100.4 
Run Length, hrs. Pressure, kPa	6.83 912	8.0 912 17.6	4.0 912	2.0 912	7.0 912 17.6
Air Flow Rate, m <sup>3</sup> /min Temperature Gradient, °C/m	16.5 61	61	17.6 61	17.6 65	57
Avg. Bed Temperature, °C Superficial Velocity, m/sec Settled Bed Height, m	913 1.34	914 1.45	914 1.45	916 1.41	913 1,43
Initial Final	2.4	2.1			
Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set	4.0 81.0 1.40	85.2 1.43	3.5 80.9 0.38	3.5 85.8 0.38	3.6 83.9 1.43
Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent	1.0 30.6 PD	0.90 41.9 PD	0,68 38,2 PD	35.3 PD	0.89 16.1 PD
Coal Flue Gas Emissions	СН	СН	СН	СН	СН
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO <sub>x</sub> , ppm CO <sub>2</sub> , % O <sub>2</sub> , %	183 120 11 12.24 5.42	360 135 83 14 6.3	508 100 40 10 5.4	488 129 61 15.4 6.1	364 135 76 12.2 3.3
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>x</sub> /M BTU	84 84 0.48 0.23	67 75 0.93 0.25	51 76 1.39 0.20	56  1.25 0.24	66 75 0.96 0.25

PD = Pfizer Dolomite CH = Champion Coal

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	100.5 1/31/79	100.6 1/31/79	102* 3/13-17/79	103.0* 3/29-4/1/79	103.1* 4/1-3/79
Run Length, hrs.	6.0	4.5	107	81	49
Pressure, kPa	912	912	700	700	700
Air Flow Rate, m <sup>3</sup> /min	17.6	17.5	12.5	13.4	13.5
Temperature Gradient, °C/m	53	54	-15 908	-12 913	-14
Avg. Bed Temperature, °C	912	913	1.43	1,52	901
Superficial Velocity, m/sec Settled Bed Height, m	1.43	1.43	1,43	1,52	1.52
Initial			2.1	2.1	
Final		2.3	1.0		
Expanded Bed Height, m	3.8	3.8	2.2	2.7	2.8
Coal Feed Rate, kg/hr	83.6	83.7	69.5	76.6	77.7
Ca/S Molar Feed Ratio-Set	1.43	1.43	1.5	1.35	0.68
Ca/S Molar Feed Ratio-Calc.		0.96	19.	5.4	4.1
Excess Air, %	37.5	36.9	20.3	16.9	17.7
Sorbent	PD	PD	GL	GL	GL
Coal	СН	CH	CH	CH	СН
Flue Gas Emissions					
SO <sub>2</sub> , ppm	274	311	27	28	41
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO <sub>x</sub> , ppm	129	135	126	77	56
CO; ppm	86	71	90	71	62
CO <sub>2</sub> , %	11.5	10.8	14.1	13.8	14.2
CO <sub>2</sub> , % O <sub>2</sub> , %	5.4	6.2	4.03	3.32	3.5
<u>Results</u>					
SO <sub>2</sub> Retention, %	74	71	97	97	96
Ca <sup>-</sup> Sulfation, %		7.4 74	5	18	23
Lb SO <sub>2</sub> /M BTU	0.72	0.81	0.06	0.06	0.09
Lb NO <sub>X</sub> /M BTU	0.24	0.25	0.19	0.12	0.09
^		<b>→ →</b> -		-	- •

PD = Pfizer Dolomite

GL = Grove Limestone CH = Champion Coal

<sup>\*</sup> Combined Combustor-Regenerator Runs; Combustor Data Only

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

	103.2*	103.3*	104	105*
Operating Conditions	4/3-4/79	4/4-6/79	4/17-20/79	<u>4/30-5/5/79</u>
Run Lenght, hrs.	22	59	83.5	99
Pressure, kPa	700	700	850	700
Air Flow Rate, m <sup>3</sup> /min	14.2	14.4	11.5	14.0
Temperature Gradient, °C/min	-12	-18	-42	-56
Avg. Bed Temperature, °C	901	902	899	894
Superficial Velocity, m/sec	1.59	1.62	1.62	1.46
Settled Bed Height, m				
Initial			2,1	2.2
Final		1.4	1.8	1.7
Expanded Bed Height, m	2.0	2.2	3,0	3.1
Coal Feed Rate, kg/hr	79.4	79.9	93	77
Ca/S Molar Feed Ratio-Set	0.68	0.93	1.49	1.29
Ca/S Molar Feed Ratio-Calc.	4.4	3.3	1.4	4.0
Excess Air, %	13.1	19.0	42.1	45.8
Sorbent	GL	GL	PD	GL
Coal	СН	СН	CH	CH
Flue Gas Emissions				
SO <sub>2</sub> , ppm	22	77	71 (5-160)	70
NO ppm	65	55	81	59
NO ppm	64	56	104	77
CO2. %	14.2	13.8	13.7	12.5
CO <sub>2</sub> , %	2.7	3.7	3,8	4.1
Results				
SO <sub>2</sub> Retention, %	98	93	93(99-84)	93
Ca Sulfation, %	22	28	66	23
Lb SO <sub>2</sub> /M BTU	0.05	0.17	0.16(0.01-0.37)	0.15
Lb NO <sub>X</sub> /M BTU	0.10	0.09	0.13	0.09
n				

GL = Grove Limestone

PD = Pfizer Dolomite

CH = Champion Coal

<sup>\*</sup> Combined Combustor-Regenerator Runs; Combustor Data Only

APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

Operating Conditions	106.1	106.2	106.3	107	108
	5/29-6/10/79	5/29-6/10/79	5/29-6/10/79	<u>6/18-23/7</u> 9	6/26 <b>-</b> 29/79
Run Length, hrs. Pressure, kPa Air Flow Rate, m <sup>3</sup> /min Temperature Gradient, °C/m Avg. Bed Temperature, °C Superficial Velocity, m/sec	43	5	217	108	71
	902	902	912	912	912
	20.6	19.7	20.2	16.5	16.4
	-64	-69	-71	-71	-100
	914	904	908	872	877
	1.71	1.62	1.64	1.31	1.31
Settled Bed Height, m Initial Final Expanded Bed Height, m Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set Ca/S Molar Feed Ratio-Calc. Excess Air, % Sorbent Coal	2.0  2.4 139 1.35  15.6 PD ILL	3.3 145 1.19  5.8 PD ILL	1.9 3.1 149 1.25  9.0 PD ILL	2.1 1.1 1.7 84.4 1.25 1.4 31.4 PD CH	2.0 0.96 1.6 86.3 1.25 1.2 23.0 PD CH
Flue Gas Emissions					
SO <sub>2</sub> , ppm NO <sub>x</sub> , ppm CO <sub>x</sub> , ppm CO <sub>2</sub> , %	21.4 47.5 332 14.8 3.1	37.8 42.4 507 16.4 1.1	91.4 64.1 540 13.4 2.1	41.5 26.4 64.7 12.8 5.1	27.8 44.6 130.8 14.2 4.0
Results SO <sub>2</sub> Retention, % Ca Sulfation, % Lb SO <sub>2</sub> /M BTU Lb NO <sub>x</sub> /M BTU	98	96	99	99	98
				72	79
	0.02	0.10	0.11	0.04	0.0
	0.06	0.06	0.08	0.13	0.08

PD = Pfizer Dolomite
CH = Champion Coal
ILL = Illinois Coal No. 6

#### APPENDIX L (CONT'D). MINIPLANT FLUIDIZED BED COAL COMBUSTION RUN SUMMARY

	109	110	111	112	113	114	115
Operating Conditions	7/25/79	<u>7/26/79</u>	7/27/79	7/30/79	7/31/79	8/1/79	8/7/79
Run Length, hrs. Pressure, kPa	9.5 915	6.5 890	8.0 900	6.5 900	8.2 915	4.6 915	6.7 91 <b>5</b>
Air Flow Rate, m <sup>3</sup> /min	17.6	17.6	17.6	13.1	17.6	17.6	17.6
Temperature Gradient, °C/m							
Average Bed Temperature, °C	870	870	870	870	870	870	870
Superficial Velocity, m/sec Settled Bed Height, m	1.65	1,65	1,65	1.22	1,65	1.65	1.65
Initial							
Final							
Expanded Bed Height, m	2.5	2.8	2.9	3.4	3.4	3.4	3,5
Coal Feed Rate, kg/hr Ca/S Molar Feed Ratio-Set	87 1.25	98 1.25	98 1.25	79 1.25*	93 1.25(0.76)**	95 1.25	99 1.25
Ca/S Molar Feed Ratio-Calc.						~-	
Excess Air, %	19.5	39	16	19.5	23	21 .5	25
Sorbent	PD	PD	PD	PD	PD	PD	PD
Coal	СН	СН	СН	СН	CH/(ILL)**	СН	СН
Flue Gas Emissions							
SO <sub>2</sub> , ppm	70	50	160	60(500)*	120(600)**	110	50
NO <sup>2</sup> , ppm	140	110	90-175	175	145	90	N.A.
CO^ ppm	175	175	200	200	200	N.A.	200
CO <sub>2</sub> , %	15	15.5	17.5	17.5	16.5	16	15
02, %	3.5	6.0	3.0	3.5	4.0	3.8	4.0

<sup>\*</sup> Near end of run Ca/S = 0, sorbent hopper empty \*\* Auxiliary feed system used to switch coal feed.

N.A. = Data not available.

PD = Pfizer Dolomite
CH = Champion Coal
ILL = Illinois Coal No. 6

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# APPENDIX M-1. PARTICLE SIZE DISTRIBUTION SPENT PFIZER 1337 DOLOMITE SORBENT (EXCEPT AS NOTED)

		Particle Size (μm)						
		5%	10%	25%	50%	75%	90%	95%
Run No.	Material	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than
61	Initial Bed			175	1004	1520	2000	2200
62	Final Bed	230	290	510	880	1230	1700	1900
63	Initial Bed	800	920	1080	1360	1800	2040	2200
64	Final Bed	550	650	820	1075	1400	1850	2050
65	Initial Bed	500	740	900	1200	1500	1900	2100
65	Final Bed	420	560	770	1075	1450	1900	2150
66	Final Bed	240	330	700	1325	1875	2200	2300
67	Final Bed		445	740	1755	2150	2330	2500
68	Final Bed	300	460	650	870	1150	1560	1800
69	Final Bed	300	450	850	1200	1630	2000	3000
70	Final Bed	175	210	300	480	980	1380	1700
71	Final Bed	96	150	290	640	1150	1600	1850
72	Final Bed	100	125	185	360	980	1400	1700
73	Final Bed	115	160	280	620	1100	1400	1600
74(1)	Final Bed	600	700	810	1020	1350	1700	1900
75(1)	Final Bed	420	520	780	1000	1250	1600	1800
76	Final Bed	150	200	3 <b>50</b>	690	1170	1500	1780
78	Bed Overflow	105	135	210	430	1000	2000	5600
	Dump No. 72							
80	Bed Overflow	130	150	205	530	1450	2000	2500
81	Initial Bed	760	940	1100	1350	1600	1900	2500
81	Final Bed	620	760	940	1300	1700	1950	2350
81	Bed Overflow	125	425	740	1075	1450	1850	2250
99	Bed Probe No. 1	820	935	1200	1550	1900		
	No. 3	600	890	970	1250	1600	1950	
	No. 5	510	600	830	1100	1400	1750	1950
	No. 11		910	1200	1550	1850		
	No. 12	640	750	1025	1325	1775	2000	
	No. 14	700	820	1100	1400	1750		
	No. 32			1100	1400	1700	2000	

(1) Grove Limestone Sorbent

APPENDIX M-2. PARTICLE SIZE DISTRIBUTION PRIMARY CYCLONE DIPLEG

	Particle Size (µm)								
Run <u>No.</u>	5% Less Th <b>a</b> n	10% Less Th <b>a</b> n	25% Less Than	50% Less Than	75% Less Than	90% Less Than	95% Less Than		
61				140	185	890	1250		
62		180	900	1300	1850	2100	2300		
65	as 40 40		110	272	880	1400	1750		
67	350	680	1375	1950	2175	2435	2600		
81		33	54	105	170	345			
85		60	80	110	180	500			
88		77	97	140	190	290	610		

			Pa	rticle Size	(µm)		
	5%	10%	25%	50%	75%	90%	95%
Run/Sample No.	Less Than	Less Than	Less Than	Less Than	Less Than	<u>Less Than</u>	Less Than
61	4.4	6.4	11.4	24	42	60	90
61	5.0	6.8	13.0	26	48	70	90
61	3.4	4.5	6.9	11	18	25	31
62 No. 6	4.0	5.6	10.5	20	50	70	105
No. 7	3.7	5.0	9.4	17	36	73	113
63 No. 3	3.5	5.4	10.0	19	38	62	105
No. 9	4.0	6.0	11.0	23	49	120	230
64 No. 2	3.5	5.0	9.8	18	30	50	60
No. 3	4.0	5.0	9.8	16	30	58	80
No. 6	4.0	5.6	10.0	18	35	56	86
65 No. 2	5.0	6.5	11.5	20	35	54	70
No. 6	4.4	5.8	10.0	17	31	50	67
67 No. 7	5.0	7.1	12.5	22	39	57	100
No. 12	4.9	7.3	13.0	23	38	59	80
No. 20	5.0	7.0	12.0	20	35	49	60
No. 25	4.7	6.9	11.5	20	34	50	60
No. 30	4.5	6.4	10.5	19	31	47	55
No. 33	4.0	5.8	10.5	18	34	51	69
No. 44	4.0	6.1	11.3	19	33	47	63
No. 49	4.8	7.2	13.0	23	39	53	70
No. 50	2.1	2.6	4.3	9.2	16	24	32
68.1 No. 6	8.0	13.0	26.5	48	84		
68.2 No. 8.1	10.0	13.9	35	64	100	112	120
No. 8.2	10.0	14.0	32	60	100	110	115
69 No. 7	2.0	3.0	6.5	11	17	24	30
69.1 No. 5	8.6	10.8	19	50	69	119	230
69.2 No. 10	7.0	10.0	16	28	50	90	200
70.1 No. 6	8.2	11.3	30	53	59	8 <b>6</b>	130
70.2 No. 10	7.6	10.0	17.5	29	50	160	215
71 No. 4	5.0	6.6	10.5	16	25	35	50
72.1 No. 8.1	3.8	5.0	7.6	13	25	40	54
73.1 No. 6	4.8	6.4	12.5	25	43	64	100

## APPENDIX M-3 (CONT'D). PARTICLE SIZE DISTRIBUTION SECONDARY CYCLONE CAPTURE

				Par	rticle Size	(um)		
		5%	10%	25%	50%	75%	90%	95%
Run	<u>Sample No.</u>	<u>Less Than</u>	Less Than	Less Than	Less Than	<u>Less Than</u>	<u>Less Than</u>	Less Than
74.1	No. 4	14	20	72				
	No. 10	8.0	14.0	38	430	850		
75	No. 6	4.3	5.8	10	18.5	35	150	
76	No. 6	7.0	9.6	15	27	70	127	
78	No. 52	4.4	6.0	9.6	16	32	63	90
	No. 85	6.0	8.2	13	23	43	74	100
	No. 130	4.4	6.0	9.6	15.5	32	68	94
79	No. 18	7.4	10.0	15	28	55	92	120
80	No. 64	<b>5.</b> 9	8.1	13	24	47	92	120
81	No. 15	5.8	8.0	14.5	33	92		
	No. 74	5.4	8.0	15	36	100		
	No. 86	6.4	8.4	15.5	35	86	155	
87	No. 5	52	70	115	165	240	370	460
96	No. 4	4.4	6.4	11.5	21	47	100	150
99	No. 13	10.0	15.0	29	61	135	270	385
	No. 14	13.5	20	38	88	170	330	450
	No. 15	11.0	15.0	27	56	135	275	390
	No. 21	9.0	12.4	23.5	51	130	275	410
	No. 22	10.5	14.0	26.5	55	125	300	370
	No. 23	14.0	19	35	68	150	300	410
	No. 36	12.5	18	38	98	215	370	470
100	No. 4	10.5	14.0	28	58	115	212	300
	No. 5	10.5	15.0	28.5	68	175	360	480
	No. 6	9.0	13.0	26	60	155	310	425
	No. 12	9.0	12.5	24.5	56	150	310	425
	No. 13	7.4	10.5	19.5	40	92	190	300
	No. 14	9.0	13.0	27	64	155	300	420
	No. 31	8.2	11.5	22.5	50	125	270	385
102	No. 30	5.2	7.2	10.3	32	96	220	320
103	No. 33	4.3	5.6	9.4	15	26	50	94
	No. 99	4.2	5.8	9.8	18	54	160	260
104	No. 6	4.4	5.6	9.8	16	33	72	135
	No. 30	3.8	5.2	9.0	16	32	66	105

#### APPENDIX M-3 (CONT'D). PARTICLE SIZE DISTRIBUTION SECONDARY CYCLONE CAPTURE

		Particle Size (µm)						
Run/	Sample No.	5% <u>Less Than</u>	10% <u>Less Than</u>	25% <u>Less Than</u>	50% <u>Less Than</u>	75% <u>Less Than</u>	90% <u>Less Than</u>	95% <u>Less Than</u>
105	No. 28	3.7	5.0	8.5	14	27	64	125
	No. 44	4.2	5.6	10.0	17	32	82	150
106	No. 2	5.0	6.6	10.5	18	38	83	140
	No. 11	7.0	14.0	34	84	165	310	430
	No. 23	4.2	6.0	11.5	23	52	105	160
	No. 57	4.8	6.4	11.5	23	52	105	160
107	No. 15	4 6	6.0	10.8	21	48	100	150

	No.
	No.
	No.
	No.
68.1	No.
68.2	No.
71	No.
72.1	No.
73.1	No.
74.1	No.
74.2	No.
5	No.
	No.
76	No.
78	No.
	No.
	No.
	No.
79	No.

				Pai	rticle Size	(um)		
		5%	10%	25%	50%	75%	90%	95%
Run/	Sample No.	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than
67	No. 7	1.30	1.60	2.3	4.0	7.0	12.0	18
	No. 12	1.30	1.75	2.6	4.7	8.4	12.5	16
	No. 20	1.80	2.30	4.3	7.4	12.3	17.5	25
	No. 25	2.30	1.66	2.4	4.2	7.3	12.0	20
	No. 30	1.55	2.00	3.4	6.4	11.0	18	25
	No. 33		1.15	1.6	2.5	4.6	8.0	11.0
	No. 43	1.60	2.00	3.5	6.6	10.5	13.5	16
	No. 48	1.60	2.10	3.8	7.2	11.5	16	19
68.1	No. 6	1.50	1.90	3.0	5.2	8.8	17	
68.2	No. 8	1.50	2.00	3.4	6.1	10.5	16	30
71	No. 4	1.35	1.75	2.8	4.9	8.2	15.0	44
72.1	No. 8	1.15	1.40	2.1	3.5	6.0	9.5	16
73.1	No. 6	1.30	1.60	2.3	4.0	7.6	30	100
74.1	No. 4	1.20	1.50	2.1	3.4	5.8	8.5	10.5
74.2	No. 10	1.50	1.90	3.0	5.0	8.8	23	100
5	No. 6.1	1.25	1.60	2.3	4.2	8.4	32	
	No. 6.2	1.85	2.10	2.8	5.3			
76	No. 6	1.60	2.00	3.0	5.0	10.0	44	
78	No. 31	1.40	1.75	2.5	3.5	5.2	8.4	11.0
	No. 52	1.80	2.30	3.5	5.1	7.6	10.0	12.5
	No. 85	2.10	2.50	3.4	4.8	6.6	8.8	10.0
	No. 120	1.65	2.20	3.1	4.5	6.6	10.0	13.0
79	No. 1	1.75	2.15	2.7	3.4	4.5	8.0	11.8
	No. 18	1.60	2.10	2.9	4.4	6.4	9.6	10.2
	No. 42	2.40	2.90	4.2	5.9	8.9	12.5	16
	No. 50	2.20	2.90	4.3	6.1	8.8	11.5	12.0
80	No. 64	1.35	1.70	2.5	3.9	6.0	8.6	9.8
81	No. 15	1.20	1.40	1.9	3.3	6.2	10.0	12.7
	No. 74	1.80	2.20	3.1	4.8	7.8	12.0	14.3
86	No. 5	1.60	2.10	3.1	4.7	7.1	10.0	12.7
		1.80	2.30	3.2	4.7	7.2	11.0	14.0

#### APPENDIX M-4 (CONT'D). PARTICLE SIZE DISTRIBUTION TERTIARY CYCLONE CAPTURE

		Particle Size (μm)								
		5%	10%	25%	50%	75%	90%	95%		
Run	/Sample No.	<u>Less Than</u>	<u>Less Than</u>	<u>Less Than</u>	<u>Less Than</u>	<u>Less Than</u>	<u>Less Than</u>	<u>Less Than</u>		
87	No. 5	1.75	2.30	3.4	5.1	7.0	11.0	13.0		
		2.10	2.70	3.7	5.3	8.0	12.0	15.0		
96	No. 4	1.45	1.80	2.6	3.8	6.3	11.0	15.0		
	No. 10	1.40	1.80	2.6	3.9	6.3	10.0	12.0		
99	No. 13	1.42	1.75	2.4	3.3	5.2	8.4	10.0		
	No. 15	1.30	1.59	2.2	3.1	5.4	8.8	10.0		
		1.42	1.70	2.4	3.4	5.8	9.8	12.0		
	No. 21	1.30	1.59	2.2	3.1	4.9	7.6	9.6		
	No. 23	1.45	1.70	2.2	3.3	5.3	8.8	11.0		
	No. 34	1.13	1.35	1.8	2.6	3.8	5.8	7.6		
	No. 36	1.42	1.65	2.2	2.9	4.2	6.3	8.4		
100	No. 2	1.10	1.40	1.9	2.7	3.9	6.0	7.8		
	No. 4	1.13	1.35	1.8	2.6	3.8	6.3	9.2		
		1.30	1.70	2.2	3.1	5.0	8.0	10.0		
	No. 6	1.26	1.45	2.0	2.8	4.0	6.8	9.4		
	No. 10	1.20	1.45	1.9	2.7	3.9	6.0	7.8		
	No. 12	1.40	1.70	2.2	3.0	4.4	7.0	9.2		
	No. 14	1.30	1.50	2.1	2.8	3.9	6.3	8.0		
	No. 31	1.15	1.40	1.9	2.6	3.8	5.9	7.4		
102	No. 30	1.05	1.36	2.1	3.4	5.5	8.5	10.0		
103	No. 33	1.10	1.41	2.1	3.3	5.0	8.0	10.9		
	No. 52	1.10	1.35	1.9	2.8	4.5	6.9	9.2		
	No. 75	1.35	1.70	2.5	3.6	5.4	8.0	10.0		
	No. 99	1.20	1.40	1.9	2.8	4.4	6.9	9.1		
104	No. 6	1.43	1.70	2.3	3.3	5.0	7.3	8.9		
	No. 18	1.60	1.95	2.6	3.9	6.3	9.8	12.0		
	No. 30	1.45	1.78	2.5	3.7	5.8	8.8	10.0		
	No. 42	1.50	1.80	2.6	3.9	6.6	9.8	12.0		
105	No. 28	1.59	1.90	2.6	3.4	4.8	7.1	8.8		
	No. 44	1.45	1.80	2.5	3.4	4.9	7.0	8.8		
108	No. 5	1.59	1.90	2.5	3.3	4.6	7.1	9.4		

		Particle Size (µm)							
	(2) Grain Loading	5%	10%	25%	50%	75%	90%	95%	
Run/Sample No.	(3) Grain Loading gr/SCF	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	
96 BF-2	0.463	1.26	1.59	2.2	3.1	4.5	6.35	8.4	
99 BF-4	0.335	1.16	1.40	1.9	2.9	4.8	9.0	11.0	
99 BF-6	0.364	1.15	1.45	2.1	3.0	5.2	8.6	11.0	
100 BF-1	0.492	1.35	1.75	2.6	3.6	5.5	8.0	10.0	
100 BF-4	0.476	1.00	1.26	1.7	2.4	3.9	7.6	10.0	
100 BF-6	0.635	1.00	1.30	1.8	2.4	3.5	5.7	8.0	
100 BF-8	0.346	1.20	1.45	2.0	2.8	4.0	6.0	8.0	
106 BF-2	3.451	1.50	1.90	3.0	5.0	8.6	14.0	18	
106 BF-4	1.771	1.65	2.1	3.4	5.6	8.4	12.5	14.8	
106 BF-14	0.563	1.70	2.2	3.5	6.0	10.0	14.0	17.3	
106 BF-18	0.718	1.75	2.2	3.4	5.6	9.1	12.7	15.0	
106 BF-21	0.623	1.70	2.2	3.3	5.6	9.2	13.5	16	
106 BF-25	0.739	1.80	2.4	3.6	6.0	9.4	13.0	16	
106 BF-35	0.212	1.50	1.80	2.6	4.2	7.0	10.5	14.0	
106 Avg. (1)	1.09	1.70	2.1	3.3	5.7	9.1	13.1	16.2	
106 Avg. (2)	0.583	1.79	2.2	3.4	5.8	9.3	13.1	16.2	
107 BF-5	0.402	1.40	1.50	1.9	2.8	5.8	12.0	16	
107 BF-7	0.597	1.35	1.45	1.8	2.7	4.6	8.4	11.8	
107 BF-11	0.49	1.33	1.45	1.8	2.8	6.0	11.0	14.3	
108 BF-2	0.336	1.45	1.70	2.5	3.8	7.0	11.4	14.0	
108 BF-6	0.728	1.50	1.79	2.6	3.5	5.5	9.0	11.6	
109 BF-2	0.819	0.84	1.08	1.5	2.2	3.5	5.8	7.2	
109 BF-4	0.540	1.15	1.42	1.9	2.6	3.7	5.0	6.0	
109 BF-6	0.522	1.35	1.70	2.3	3.0	4.2	5.8	7.0	
110 BF-2	0.598	1.35	1.60	2.3	3.2	4.4	6.0	7.1	
110 BF-5	0.546	1.60	1.90	2.6	3.3	4.4	5.6	6.4	
110 BF-8	0.593	1.50	1.78	2.7	3.3	4.6	6.0	7.1	

<sup>(1)</sup> All

<sup>(2)</sup> Excluding 2, 4(3) BF indicates Balston filter samples taken upstream of the tertiary cyclone.

		Particle Size (µm)								
Pun/Sample No. (2)	Grain Loading	5%	10%	25%	50%	75%	90%	95%		
Run/Sample No. (2)	gr/SCF	Less Inan	Less Inan	Less Ihan	Less Ihan	Less Than	Less Than	Less Than		
111 BF-2	0.399	1.1	1.37	2.0	3.0	4.5	6.3	7.6		
111 BF-5	0.494	1.2	1.5	2.1	2.9	4.2	5.8	6.8		
112 BF-2	0.675	1.6	1.9	2.6	3.3	4.3	5.8	6.9		
112 BF-5	0.545	1.4	1.7	2.4	3.0	4.1	5.6	6.6		
112 BF-8	0.500	1.2	1.4	2.0	2.8	3.9	5.8	6.9		
113 BF-4	0.784	1.1	1.4	1.9	2.7	3.7	5.5	7.0		
113 BF-7	0.373	1.4	1.7	2.2	3.2	4.8	6.0	7.1		
113 BF-9 <sub>/1</sub>	0.760	1.4	1.7	2.4	3.0	4.2	6.0	7.6		
114 BF-2)¦{	2.12	1.3	1.7	2.7	4.4	6.0	8.0	9.1		
114 BF-4 <sup>(1)</sup>	2.22	1.4	1.8	2.7	4.0	5.8	7.4	8.2		
115 BF-2	0.237	1.4	1.75	2.4	3.0	4.2	5.6	6.5		

<sup>(1)</sup> Secondary Cyclone Disabled

<sup>(2)</sup> BF indicates Balston filter samples taken upstream of the tertiary cyclone

	(3)Grai	n Loading	5%	10%	P <b>ar</b> 1 25%	ticle Size 50%	(μm) 75%	90%	95%
Run/Sample	<u>No .</u>	gr/SCF	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than
61 (1)		0.456	2.3	3.2	7.5	16	40	~-	
62.1 (1) 62.3 (1)		0.026 0.206	2.2 1.2	2.6 1.4	5.3 2.1	16 3,1	30 7.0	45 1 <i>7</i>	23
64 No. 1 No. 2 No. 3	(1) (1) (1)	0.280 0.290 0.271	1.0 1.1 1.05	1.2 1.3 1.3	1.8 1.9 1.8	3.1 2.7 2.6	5.5 3.9 3.7	10.5 6.3 6.0	17 11 9.7
65 No. 1 No. 2	(3)	0.048 0.061	1.2 1.0	1.4 1.2	2.2 1.9	3.3 2.8	6.3 4.6	16 10.0	24 16
67.1 No. 1 No. 2		0.057 0.053			1.0	1.3 2.5	3.5 9.4	12.5 16.7	17 20
67.2 No. 1 No. 2 67.3 No. 2		0.081 0.058 0.066			1,1 1,0	1.7 1.8	2.9 8.0	10.5 14.0 10.7	17 20 15
68.2 No. 1 No. 2		0.033			1.1 1.6	1.4 1.7 2.1	4.7 2.9 4.8	11.0 20	20 40
69.1 No. 1 No. 2	\ — <i>/</i>	0.416 0.350	1.7 1.4	2.2 1.8	3.6 2.9	6.5 5.0	9.5 8.5	13.0 13.0	16 18
70.1 70.2 No. 1 70.2 No. 1	(2)		1.1 1.2 1.0	1.4 1.7 1.1	2.2 2.8 1.6	3.8 5.6 2.7	7.8 9.6 5.3	11.0 12.6 8.6	13 14 11
71 No. 1		0.054			1.3	2.1	4.5	12.5	19
72.1 No. 1		0.040			1.1	1.7	2.6	4.5	7
73		0.023				1.2	3.2	13.0	18

<sup>(1)</sup> Runs prior to installation of 3rd cyclone. (2) Third cyclone bypassed.

<sup>(3)</sup> Runs prior to run 80, the sample no. refers to the order in which Balston filter samples were taken with the old Balston filter sampling system.

		Particle Size (µm)						
(1)	Grain Loading	5%	10%	25%	50%	75%	90%	95%
Run/Sample No. (1)	gr/SCF	Less Than	Less Than	Less Than	Less Than	Less Than	Less Than	<u>Less Than</u>
74.1	0.024			1.15	1.70	2.5	8.0	13.5
74.2	0.024	1.1	1.35	2.0	3.8	8.0	15.0	20.0
75 No. 1	0.024			1.10	1.95	5.5	12.5	16.0
No. 2	0.031			1.15	1.70	9.3	14.5	18.5
78 No. 2	0.033	0.48	0.56	0.74	0.96	1.4	5.0	8.7
No. 3	0.034	0.6	0.75	1.10	1.95	4.8	8.5	9.8
No. 4	0.028	0.62	0.78	1.10	3.1	6.8	10.8	12.5
No. 5	0.039	0.46	0.54	0.74	1.10	2.8	6.1	8.4
No. 6		0.48	0.55	0.79	2.1	5.9	12.0	14.0
No. 7	C.042	0.48	0.58	0.74	1.00	1.5	3.1	5.04
No. 8	0.056	0.79	1.00	1.49	2.5	5.5	9.2	11.5
No. 9	0.045	0.74	0.97	1.47	2.5	5.7	9.6	11.5
No. 10	0.036	0.6	0.76	1.30	3.0	5.7	8.8	11.0
No. 11	0.054	0.79	1.10	1.75	2.5	3.7	5.7	8.0
Avg of Test 2-11	0.041	0.58	0.66	0.98	1.90	4.6	8.5	11.0
79 No. 1	0.012	0.54	0.68	1.00	1.75	3.7	6.3	8.8
No. 2	0.0086	0.47	0.53	0.69	0.98	2.4	8.8	13.5
No. 3	0.034	0.47	0.56	0.75	1.10	1.8	2.2	8.0
80 BB-1 <sup>(2)</sup>	0.0143	0.52	0.60	0.69	0.92	1.2	1.8	2.2
80 BA-2	0.0227	0.55	0.61	0.76	1.06	1.9	6.0	9.0
80 BA-3	0.0257	0.54	0.61	0.78	1.11	2.0	4.9	9.0
80 BB-4	0.021	0.53	0.59	0.70	0.93	1.3	1.8	2.5
80 BA-5	0.023	0.56	0.64	0.91	1.30	2.0	3.4	4.9
80 BB-6	0.021	0.46	0.53	0.70	0.98	1.41	2.2	5.0
80 BA-7	0.028	0.58	0.71	1.05	1.50	2.4	4.5	6.3

(1) Runs prior to run 80 the sample number refers to the order in which the Balston filters were taken with the old Balston filter sampling system.

(2) Runs after run 80 BA refers to Balston filter samples taken with the HTHP sampling system; BB refers to samples taken with the old Balston filter sampling system.

	Particle Size (µm)								
(1)	Grain Loading	5%	10%	25%	50%	75%	90%	95%	
Run/Sample No. (1)	gr/SCF	Less Than	Less Than	<u>Less Than</u>	Less Than	Less Than	<u>Less Than</u>	<u>Less Than</u>	
80 BB-8	0.0145	0.47	0.54	0.73	1.10	1.6	2.5	4.0	
80 BA-9	0.024	0.96	1.18	1.45	1.95	2.8	4.7	6.0	
80 BB-10	0.023	0.47	0.56	0.76	1.13	1.6	2.4	5.0	
81 BA-4	0.063	0.56	0.60	0.79	1.2	2.8	7.5	11.5	
81 BA-10	0.031	0.56	0.60	0.76	1.1	1.7	6.3	13.0	
81 BB-11	0.037	0.63	0.80	1.20	1.7	3.4	6.0	8.0	
81 BA-13	0.037	0.56	0.62	0.77	1.2	3.2	9.0	14.0	
81 BA-16	0.039	0.58	0.70	1.00	1.4	3.1	9.4	16.0	
81 BB-18	0.021	0.52	0.59	0.72	1.6	5.8	11.5	16.0 8.0	
81 BB-20	0.018	0.58	0.63	0.80	1.2	2.9	5.8		
83 BB-1	0.027	1.26	1.70	2.20	2.8	3.5	4.3	5.0	
84 BB-2	0.050	1.40	1.75	2.20	2.8	3.5	4.0	4.9	
86 BA-1	0.028	0.88	1.10	1.50	2.0	2.9	6.3	10.08	
87 BA-1	0.028	0.94	1.15	1.50	2.2	4.6	10.0	13.0	
	(2)	1.15	1.30	1.70	3.2	8.6	19.0	28.5	
89 BB-6	0.050	0.60	0.68	0.92	1.2	2.0	7.1	11.5	
	(2)	1.10	1.20	1.46	2.7	6.8	12.7	20.2	
91 BB-3	0.0459	1.20	1.38	1.78	2.3	3.1	7.4	12.7	
96 BB-3	0.049	1.00	1.26	1.65	2.2	3.7	8.0	11.0	
99 BB-1	0.036	1.00	1.25	1.63	2.1	2.5	3.0	3.5	
99 BB-2		0.74	0.98	1.40	1.9	2.5	3.5	5.0	
99 BB-3	0.094	0.63	0.83	1.20	1.6	2.4	3.9	5.4	
99 BB-5	0.055	0.96	1.20	1.60	2.1	2.8	4.0	7.0	

<sup>(1)</sup> Runs prior to run 80 the sample number refers to the order in which the Balston filters were taken with the old Balston filter sampling system.

<sup>(2) 100</sup> μm aperture used on Coulter Counter: all others used 30 μm aperture.

	(1)	Grain Loading	5%	10%	25%	ticle Size 50%	75%	90%	95%
Rur	/Sample No. (1)	gr/SCF	<u>Less Than</u>	Less Than	Less Than	Less Than	Less Than	Less Than	<u>Less Than</u>
	) BB-2	0.059	1.10	1.35	1.75	2.2	2.7	3.1	5.0
100	) BB-3	0.060	0.63	0.79	1.15	1.5	2.1	3.0	6.35
	) BB-5	0.048	1.15	1.40	1.85	2.4	3.1	3.9	4.6
100	BB-7	0.049	0.84	1.10	1.43	1.8	2.3	2.7	3.0
102	BB-5	0.025	0.63	0.75	1.05	1.4	2.7	5.0	9.2
103	BB-4	0.044	0.75	0.96	1.3	1.7	2.4	3.5	5.5
103	BB-7	0.075	1.00	1.26	1.6	2.1	2.6	3.0	3.5
	BB-11	0.029	0.60	0.72	1.0	1.6	3.2	5.6	8.6
103	BB-15	0.099	1.00	1.26	1.6	2.1	2.65	3.1	3.8
	BB-1	0.057	1.20	1.40	1.8	2.2	2.9	3.5	3.9
	BB-2	0.099	0.79	1.10	1.4	1.9	2.5	3.0	3.3
	BB-3	0.117	1.15	1.35	1.8	2.4	2.9	3.5	3.9
	BB-4	0.084	1.00	1.17	1.4	1.9	2.4	2.8	3.1
104	BB-5	0.140	1.00	1.26	1.7	2.2	2.8	3.1	3.7
	BB-1	0.077	0.64	0.80	1.1	1.5	2.0	2.5	3.1
	BB-2	0.074	1.05	1.30	1.7	2.2	2.6	3.1	3.6
	BB-3	0.072	0.88	1.17	1.5	2.0	2.5	3.0	3.2
105	BB-5	0.106	1.26	1.49	1.9	2.4	3.0	3.5	3.8
106	BA-1	0.083	0.79	1.05	1.4	2.1	3.3	5.6	7.4
	BA-3	0.029	0.74	0.93	1.2	1.7	2.4	4.6	7.0
	BA-5	0.049	0.64	0.84	1.1	1.7	2.8	5.0	8.0
106		0.037	0.79	1.05	1.4	2.0	2.8	4.0	5.0
106		0.460	1.10	1.40	2.2	3.5	5.6	7.8	9.8
106		0.048	1.70	2.20	2.1	4.9	6.9	9.2	10.0
106		0.094	0.79	1.15	1.6	3.1	4.5	6.3	8.0
106	BA-13	0.80	0.79	1.10	1.7	2.8	4.3	6.3	9.2

<sup>(1)</sup> Runs prior to run 80 the sample number refers to the order in which the Balston filters were taken with the old Balston filter sampling systems.

		Particle Size (μm)							
Dun (Sample No. (1)	Grain Loading	5%	10%	25%	50%	75%	90%	95%	
Run/Sample No. (1)	gr/SCF	Less Than	Less Than	Less Than	Less Than	Less Than	<u>Less Than</u>	<u>Less Than</u>	
106 BA-17	0.055	0.56	0.62	0.8	1.2	2.4	4.9	0.8	
106 BA-20	0.063	0.64	0.79	1.2	1.7	2.7	4.9	7.2	
106 BA-24	0.054	0.56	0.65	0.9	1.3	2.7	5 <b>.5</b>	8.0	
106 BA-28	0.047	0.58	0.65	0.8	1.2	1.9	4.6	8.0	
106 BA-32	0.043	0.59	0.68	0.9	1.3	2.1	4.6	7.0	
106 BA-34	0.043	0.60	0.70	1.0	1.4	2.1	3.8	6.3	
Avg. (2)	0.056	0.75	0.95	1.3	2.0	3.1	5.3	7.6	
Avg. (3)	0.050	0.65	0.80	1.1	1.5	2.5	4.7	7.2	
107 BA-4	0.099	0.74	0.98	1.4	1.9	2.5	3.1	3.6	
107 BA-6	0.117	1.05	1.35	1.9	2.5	3.2	4.8	8.0	
107 BA-10	0.121	1.00	1.30	1.7	2.2	2.9	3.8	5.0	
108 BA-1	0.118	0.79	1.05	1.4	1.9	2.5	3.4	4.0	
108 BA-5	0.129	1.05	1.30	1.7	2.2	2.6	3.0	3.4	
109 BA-1	0.118	0.93	1.26	1.7	2.3	2.9	3.9	5.3	
109 BA-3	0.112	0.80	1.21	1.7	2.1	2.6	3.2	3.7	
109 BA-5	0.170	1.00	1.30	1.7	2.1	2.6	3.2	3.85	
110 BA-1	0.105	0.64	0.77	1.0	1.4	2.0	3.2	4.3	
110 BA-4	0.061	0.56	0.60	0.8	1.3	2.8	5.7	7.6	
110 BA-7	0.070	0.82	1.20	1.7	2.4	3.2	4.0	5.1	
TTO DA-7	0.070	0.02	1.20	1.7	4.4	3.2	4.0	J. I	
111 BA-1	0.084	0.69	0.84	1.2	1.6	2.4	3.6	4.8	
111 BA-4	0.079	0.58	0.68	0.8	1.3	2.0	3.3	4.7	

<sup>(1)</sup> Runs prior to run 80 the sample number refers to the order in which the Balston filters were taken with the old Balston filter sampling system.

<sup>(2)</sup> Excluding 7

<sup>(3)</sup> Excluding 7, 8, 9, 13

Run/	/Sample No.(1)	Grain Loading gr/SCF		10% Less Than	25%	ticle Size 50% Less Than		90% Less Than	95% Less Than
	BA-1	0.067	0.60	0.68	0.9	1.4	2.4	4.9	6.4
	BA-4 BA-7	0.132 0.203	0.72 0.56	0.90 0.64	1.3 0.8	1.7 1.3	2.3 2.4	3.4 4.2	4.3 5.4
	BA-3	0.109	0.62	0.73	1.0	1.4	2.6	5.2	6.6
	BA-6 BA-8	0.072 0.118	0.68 0.79	0.78 0.98	1.1 1.3	1.4 1.7	2.0 2.2	3.3 3.9	4.8 5.2
114		0.133	0.68	0.82	1.2	1.7	2.6	4.6	6.5
115	BA-3	0.172 0.075	0.78 0.74	0.98 0.92	1.4	1.9 1.8	2.9 2.6	<b>4.</b> 9 <b>4.</b> 3	6.4 5.6

(1) Runs prior to run 80 the sample number refers to the order in which the Balston filters were taken with the old Balston filter sampling system.

Weight Percent

Run No.	Source	Ca	S	_S04	C03	Total C	Mg
61	Initial Bed	29.3	7.21	22.52	24.06	5.01	1.83
•	Final Bed	30.4	10.50	33.28	0,62	0.28	4.92
	Second Cyclone No. 1	19.2	4.24	13.48	11.27	5.62	7.19
	No. 2	13.8	3.34	10.84	4.05	3.05	5.91
	GBF Filter 2 Dump No. 3	10.6	4.44	13.20	3.73	1.59	5.44
	Filter 3 Dump No. 3	11.9	3.83	10.39	3.73	2.14	5.53
	Filter 2 Dump No. 6	11.2	4.65	13.56	1.42	7.44	5.39
	Filter 3 Dump No. 6	9.57	4.51	12.47	0.84	1.70	4.73
	GBF Flange Fines	8.63	4.26	10.42	4.14	1.63	4.28
	Particulates - GBF Outlet	7.95	2.41	15.85	1.17	1.19	3.27
	Bed Probe Sample No. 1	13.9	7.04	21.83	27.60	5.96	5.27
	No. 2	16.1	7.23	24.93	0.54	0.35	2.36
	No. 3	30.3	8.68	28.70	0.71	0.37	4.10
	Dipleg - 1st Cyclone	14.6	5,45	20.38	2,52	1.51	7,37
62	Initial Bed	32.0	10.0	33,37	19.91	5.46	0.43
	Final Bed	38.0	10.51	32.15	0.53	0.42	0.88
	Second Cyclone No. 1	16.4	3.24	9.03	4.78	3.50	4,33
	No. 2	13.7	3.06	9,20	2.17	1.87	4.18
	No. 6	8.42	2.83	8.08	0.32	2,21	2,26
	No. 7	8.26	3.09	7.17	0.28	3.16	1.93
	GBF Filter 2 Dump No. 1	10.4	5.67	15.57	2.89	0.98	4.62
	Filter 3 Dump No. 1	24.4	5.20	14.24	4.20	1.49	11.6
	Filter 2 Dump No. 2	12.8	4.70	12.49	4.33	1.72	5.06
	Filter 3 Dump No. 2	14.5	4,20	12.38	3,93	1.86	5.05
	Filter 2 Dump No. 7	7.70	4.51	12.57	0.68	1.52	1,62
	Filter 3 Dump No. 7	6,82	5.33	11.54	0.50	0.85	1,32
	GBF Flange Fines	12.1	5,60	17.02	1,53	1.75	4,54
	Particulates - GBF Outlets		5.98	18.20	0.01	2.78	•
	Bed Probe Sample No. 2	40.2	9,10	27.96	1,71	0.37	1.56
	No. 5	37.3	11.0	34.66	0.65	0.58	1.08
	No. 6	37.6	11.0	32.39	1.78	0.32	1.02
	Dipleg - 1st Cyclone	12.7	5,67	16.62	1.44	0.61	4.91

					Weight Per	cent	
Run No.	Source	Ca	<u> </u>	<u> </u>	<u>CO3</u>	Total C	Mg
63	Initial Bed	37.7	8.55	27,36	11.88	2.52	0.76
	Final Bed	36.4	10.32	33.96	0.50	0,23	0.74
	Second Cyclone No. 3	7.62	2.31	6.13	0.51	4.51	0.73
	<b>No.</b> 6	7.15	2.64	4.25	0.63	4.38	0.80
	<b>No.</b> 9	4.93		4.18	0.16	5.00	0.43
	GBF Filter 2 Dump No. 3	3.34	1.49	6.47	0.36	0.81	0.48
	Filter 3 Dump No. 3	2.81	1.55	4.14	0.30	1,15	0.39
	Filter 2 Dump No. 7	2.15	2.14	3.98	0.06	0.80	0.18
	Filter 3 Dump No. 7	2.01	1.15	2.82	0.05	0.49	0.14
	Filter 2 Dump No. 10	2.32	1.21	3,30	0.03	0.24	0.18
	Filter 3 Dump No. 10	1.62	1.08	2.87	0.24	0.59	0.11
	GBF Flange Fines Filter 2	4.47	4.93	11.12	0.29	1.76	0.74
	Filter 3	4.55	3.97	7.66	0.53	1.22	0.40
	Particulates No. 1	4.74	-	-	-	-	0.29
	No. 2	4.14	-	-	-	-	0.21
	No. 3	5.02	-	-	-	0.12	0.49
	Bed Probe Sample No. 2	78.0	8.00	29.07	6.37	1.34	1,66
	No. 3	35.9	9.83	30.97	0.43	0.12	0.64
	No. 4	39.8	10.10	32.54	0.42	0.30	0.69
	No. 5	36.1	9.39	33,63	0.46	0.50	
	No. 6	35.6	10.41	34.23	0,79	0.33	0.73
	Dipleg - 1st Cyclone	9.42	3.13	8.02	1.10	0.57	1,56
64	Initial Bed	32.6	9.36	28.90	3.35	2.14	0.60
	Final Bed	30.0	11.6	34.16	0.78	0.35	0.54
	Second Cyclone No. 2	4.93	1.55	5.27	0.18	7.30	0.14
	No. 3	6.13	0.80	4.77	0.25	15.51	0.23
	No. 6	3.46	1.00	3.24	0.11	4.92	0.17
	GBF Filter 2 Dump No. 3	0.72	0.34	0.61	0.05	<0.05	0.06
	Filter 3 Dump No. 3	2,16	1.52	4.28	0.12	1.14	0.34
	Filter 2 Dump No. 6	0.89	0.59	1.00	0.27	0.18	0.25
	Filter 3 Dump No. 6	0.92	0.45	5.03	0.02	<0.05	0.16
	Filter 2 Dump No. 8	1.96	0.48	4.71	0.22	0.17	0.34
	Filter 3 Dump No. 8	1.80	1.17	1.39	0.81	0.42	0.47

					Weight Per	rcent	
Run No.	Source	<u>Ca</u>	<u></u>	<u> S04</u>	C03	Total C	Mg
64	GBF Flange Fines No. 2	4.56	2.17	3.23	0.03	0.46	0.40
(Cont'd	No. 3	5.52	1.85	8.19	0.02	1.09	0.28
	Particulates No. 1	5.60	3.82	9.28	-	•	0.23
	No. 2	4.92	4.52	15.32	-	1,61	0.23
	No. 3	4.50	5.81	24.88		1,63	0.21
	Bed Probe Sample No. 4	43.4	8.85	28.56	0.45	0.44	0.56
	No. 7	41.1	8.78	31.24	0.27	0.34	0.65
	No. 9	32.8	10.09	33.56	0.52	<0.05	0.68
	Dipleg - 1st Cyclone	5.53	1.59	4.83	0.08	0.23	0.36
65	Initial Bed	37.8	10.6	28.32	7.95	2,21	0.75
	Final Bed	37.4	12.72	35.95	0.11	0,29	0.59
	Second Cyclone No. 2	5.10	2.62	6.26	0.52	6,21	0.24
	No. 6	2.80	1.31	3,27	0.10	5.05	0.16
	GBF Filter 2 Dump No. 3	1.52	0.74	1.64	0.22	0.12	0.19
	Filter 3 Dymp No. 3	8.25	0.44	1.15	0.10	0.21	0.35
	Filter 2 Dump No. 7	1.06	0.78	1.78	0.01	0.17	0.05
	Filter 3 Dump No. 7	1.78	1.55	3.01	0.07	0.62	0.12
	Bed Probe Sample No. 3	39.1	11.37	33.42	0.53	0.25	0,62
	' No. 7	30.5	12.78	35.51	0.62	0.12	0.70
	Dipleg - 1st Cyclone	6.40	2.99	6.38	0.01	0.29	0.27
67	Initial Bed	22.3	6,87	14.54	-	4.43	11.7
07	Final Bed	18.9	8,17	24,82	12.36	2.15	10.7
	Dipleg - 1st Cyclone	15.1	7.07	17.95	9.38	1.94	8.0
67.1	Second Cyclone Nos. 5-9	7.3	5,39	14.01	0.45	4,69	3.1
07.1	Nos. 10-14	7.1	5.03	20.84	0.51	4.20	3.4
	Bed Overflow Nos. 5-9	20.8	7.16	21.08	17.19	2,63	11.2
	Nos. 10-14	21.6	6.62	14,13	15,77	2.67	12.0
	Third Cyclone Nos. 5-9	6.7	6.05	19.85	0.03	1.26	3.9
	Nos. 10-14	6.8	6.90	20,16	0.01	1.23	4.1
	Bed Probe Sample	19.6	10.53	15.72	17.80	3.18	10.9
	Bed Probe Sample	22.1	7.01	24,71	20.27	4.50	12.2
	BCG 110BC 0dp10	•		• • •	•		

					Weight Per	cent	
Run No.	Source	<u> </u>	<u> </u>	<u>S04</u>	CO3	Total C	Mg
67.1 (Cont'd.)	Turbine Section No. 1 (Pressure Reduc. Section)	4.3	4.04	11.46	0.58	0.33	2.0
•	Turbine Section No. 4 (Turbine Housing After Distribution Plate)	6.4	9.18	29.55	0.39	0.18	3.2
67.2	Second Cyclone Nos. 18-22	8.4	5.26	16.37	0.84	3,43	4.3
	Nos. 23-27	8.4	5.44	17,68	0.49	3,66	4.2
	Bed Overflow Nos. 18-22	20.6	10.19	34.36	12.80	1,78	12.1
	Nos. 23-27	20.1	10.19	34.61	10.00	2.47	10.9
	Third Cyclone Nos. 18-22	6.9	8,02	16,20	0.13	2,26	3.5
	Nos. 23-26	6.8	6.19	18,64	0.08	1.16	3,4
	Bed Probe Sample	16.3	8.07	29,55	15.74	4.39	11.9
	Bed Probe Sample	20.9	8.73	23.20	20.62	3.73	12.1
67.3	Second Cyclone Nos. 28-32	7.3	4.99	15,34	0.51	4.01	3.6
	Bed Overflow Nos. 28-32	23.1	8.16	28,83	17.86	4.44	13.7
	Third Cyclone Nos. 28-32	6.1	8.02	21,53	0.21	0.40	3.6
	Bed Probe Sample	23.3	8.74	29.96	16.44	3.50	13.5
67.4	Second Cyclone Nos. 42-46	4.3	5,17	15,66	0.49	4.78	2.1
	Nos. 47-51	6.9	5.78	16,19	0.48	4.53	3.4
	Bed Overflow Nos. 42-46	22.1	7.45	19,20	24.12	4.53	12.8
	Nos. 47-51	22.1	6.63	23,81	21.95	5.07	12.4
	Third Cyclone Nos. 41-45	6.7	9.30	28.78	0.09	1.06	5.0
	Nos. 46-50	6.9	8.78	28.29	0.04	1,15	4.9
	Bed Probe Sample	23.5	6.75	24.80	21.53	4.91	13.4
	Bed Probe Sample	20.4	11.84	21.58	22.43	2.46	11.9
	Bed Probe Sample	22.1	7,99	22.59	21.38	4.31	12.7
	Bed Probe Sample	23.1	6.38	23.22	18.87	5,26	13.4

					Weight Per	cent	
Run No.	Source	<u>Ca</u>	<u> </u>	<u> </u>	C03	Total C	Mg
68	Initial Bed	22.6	-	30.50	17.87	3.44	13,8
••	Final Bed	21.6	-	37.12	0.60	0.18	14.0
68.1	Second Cyclone No. 6	15.8	-	23.19	4.49	2.29	10.0
	No. 7	14.9	-	21.70	5.32	2.78	9.3
	Third Cyclone No. 6	11.0	-	24.01	0.72	4.14	5.9
	No. 7	11,1	-	25.90	1,10	4.63	6.0
	Bed Probe Sample No. 6	23.8	-	38.80	0.93	0.12	14.6
	No. 7	24.2	-	33,26	0.71	0.31	15.2
	No. 8	23.7	-	32.98	1.40	1.04	15.6
68.2	Second Cyclone No. 8.1	17.4	-	25.68	4.93	2.01	11.4
68.2	No. 8.2	18.7	-	24.96	5.21	2.40	11.7
	No. 9	18.5	-	24.92	5.04	1.90	12.1
	No. 10	17.6	-	25.54	5.13	2.53	11.1
	Third Cyclone No. 8.1	10.8	-	24.42	1.28	4.62	5.79
	No. 8.2	11.1	-	24.95	1.57	5.78	5.85
	No. 9	12.2	-	25.00	0.97	3.40	5.68
	No. 10	13.1	-	21.20	1.32	3.16	7.3
	Bed Probe Sample No. 9	25.5	-	35.36	0.88	0.21	16.6
	No. 10	22,6	-	33.84	0.69	0.32	15,2
	No. 11	21.3	-	36.05	0.58	0.62	15.1
	No. 12	23.7	-	32.73	0.72	0.44	16.1
69	Initial Bed	27.2	-	29.53	6.42	1.34	12.9
<del>0</del> ,5	Final Bed	24.1	-	38.16	0.71	0.13	11.9

	•	, , , , , , , , , , , , , , , , , , , ,		- //	Weight Per	cent	
Run No.	Source	<u>Ca</u>		<u> </u>	C03	Total C	Mg
69.1	Second Cyclone No. 5	15.6	-	27.02	2.87	1,41	9.0
	No. 6	16.5	-	26.40	2.20	1.12	9.36
	No. 7	15.0	-	28.78	2.14	0.81	9.14
	Bed Overflow No. 3	18.0	~	35.76	3.56	0.71	11.4
	Bed Probe Sample No. 1	22.9	-	26.29	2.97	0.87	12.2
	No. 5	23.5	-	40.38	0.71	0.19	12.0
	No. 6	22.4	-	40.44	0.74	0.17	11.5
	No. 7	15.1	-	38.36	1.41	0.14	10.4
	No. 8	19.3	-	31.77	0.94	0.62	11.7
	Balston Filter No. 1	14.1	-	44.74	-	0.52	6.40
69,2	Second Cyclone No. 9	13.4	-	27.61	1.94	3,37	8.9
	No. 10.1	14.0	9.43	27.94	1.72	1.42	8.82
	No. 10.2	13.7	9.70	26.79	1.76	1.30	9.22
	No. 11	10.5	-	24.92	1.42	3.59	7.34
	Bed Overflow No. 5	23.2	-	34.29	4.93	1.06	11.0
	No. 6.1	23.0	12.19	36.88	4.14	0.82	12.2
	No. 6.2	22.1	10.94	33.05	5.79	1.35	12,4
	No. 7	20.0	-	32.42	5.73	1.24	12,2
	Bed Probe Sample No. 10	18.7	-	40.20	0.44	0.15	9.9
	No. 11	21.0	12.92	40.28	0.73	0.18	10.9
	No. 12	23.4	-	39.02	0.65	0.15	12.5
	Balston Filter No. 2	1.99	-	24.48	-	9.47	0.91
70	Initial Bed	24.7	_	31.12	1.65	0.41	13,5
	Final Bed	24.6	-	33.68	1.14	0.25	13.4
70.1	Second Cyclone No. 6	16.2	_	27.72	2.89	1.46	9.0
	No. 7	17.7	_	29.78	2.78	0.98	9.81
	Bed Overflow No. 1	21.9	_	36.19	3.92	0.86	12.6
	No. 2	26.5	-	29.04	6.40	1.29	14.6
	Balston Filter No. 1	13.8	-	30.56	•	0.51	5.90
	Bed Probe Sample No. 1	25.5	-	37.45	1.59	0.40	15.9
	No. 8	32.8	_	35.25	2.33	0.58	19.6
	No. 9	24.4	_	37.93	1.02	0.25	14.3
	No. 10	26.2	-	31.39	1.50	0.32	15.7

					Weight Pe	rcent	
Run No.	Source	<u>Ca</u>	<u> </u>	<u> </u>	C03	Total C	Mg
70.2	Second Cyclone No. 8	15.3	-	28.28	2.99	1.13	9.92
	No. 9	11.7	_	28.10	2.59	1.14	7.85
	No. 10.1	15.1	-	28.22	2.40	1.00	10.5
	No. 10.2	16.2	-	28.63	2.63	0.98	9.59
	No. 11	16.8	-	27.57	2.70	1.04	9.40
	Bed Overflow No. 3	16.1	-	32.06	6.11	1.40	9.09
	No. 4	19.9	-	31.71	5.74	1.25	10.8
	No. 5.1	18.0	-	35.75	4.82	0.98	12.3
	No. 5.2	21.7	-	35.84	6.12	1.28	12.7
	No. 6	24.6	•	33.43	4.86	0.97	13.5
	Balston Filter No. 2	11.9	-	27.52	-	0.42	6.43
	Bed Probe Sample No. 11	24.4	-	33.44	1.05	0.31	13.7
	No. 12	22.7	-	34.69	1.12	0.27	13,4
	No. 13	24.0	-	29.39	1.30	0.30	13.3
	No. 14	23.9	•	35.30	1.41	0.33	13.2
	No. 15	20.5	•	39.57	0.56	0.13	12.2
71	Initial Bed	25.3	-	27.70	2,21	0,53	14,5
	Final Bed	22.4	-	31.95	3.22	0.69	13.0
	Second Cyclone No. 4	14.9	-	27.60	0.91	0.96	7.74
	Bed Overflow No. 9	22.0	-	32.94	9.35	2.00	12.1
	Third Cyclone No. 4	10.6	-	23.58	1.32	1.09	4.71
	Bed Probe Sample No. 1	24.5	•	33.74	5.93	1.36	13.0
	No. 5	25.1	-	31.85	3.86	0.88	13.6
	No. 6	23.0	•	33.44	2.22	0.57	12.1
	No. 18	22.3	-	34.51	3,16	0.81	12.9
72	Initial Bed	21.1	-	39.55	3.56	0.95	13.8
	Final Bed	18.9	-	33.96	0.46	0.15	12.8

					Weight Per	rcent	
Run No.	Source	_Ca	<u> </u>	<u> 504</u>	C03	Total C	<u>Mg</u>
72.1	Second Cyclone No. 8.1	15.8	-	27.07	1.34	1.84	8.82
·	No. 8.2	16.2	-	27,60	1.33	1.58	9,42
	No. 9	19.1	-	27.17	0.83	1.45	10.6
	Bed Overflow No. 3	20.9	-	27.83	11.49	0.99	12.8
	Third Cyclone No. 8.1	11.4	_	22.64	0.15	1.18	4.19
	No. 8.2	11.3	-	23.30	0.27	1.14	4.00
	<b>No.</b> 9	11.1	-	20.93	0.99	1,07	4.48
	Bed Probe Sample No. 1	25.7	_	26.90	6.43	1.37	14.9
	No. 8	21.0	-	34.34	0.85	0.33	12.7
	No. 9	20.4	-	37.38	0.88	0.34	12.9
	No. 10	19.2	-	29,65	0.74	0.40	12.7
72.2	Second Cyclone No. 12	17.4	-	23,23	1.33	8,56	10.3
	Third Cyclone No. 12	14.5	-	15.98	1.71	4.91	5.93
	Bed Probe Sample No. 12	22.0	-	36.65	1.08	0.93	13.6
	No. 13	18.8	-	38,65	0.72	0.23	13.0
	No. 14	23.4	-	32.94	0.53	0.17	14.1
73	Initial Bed	27.3	-	30.29	5.52	1,21	16.2
	Final Bed	21.8	-	34.75	1.03	0.24	12.9
73.1	Second Cyclone No. 6	16,7	-	26,55	3,33	2,32	9.75
	No. 7	16.5	-	26.33	3.04	2.28	8.80
	Bed Overflow No. 3	21.9	-	35,41	3.98	0.87	11.6
	Third Cyclone No. 6	16.2	-	24.97	1.60	1.13	7.44
	No. 7	16.3	-	24.78	1.42	1.09	7.02
	Bed Probe Sample No. 1	28.1	_	34.70	3.41	0.76	15.7
	No. 5	25.7	-	24,81	2.18	0.59	14.3
	No. 6	23.7	-	30.73	2.27	0.61	13.4
	No. 7	26.4	-	30.23	1.93	0.55	15.3
	No. 8	24.7	-	35.20	1.01	0.33	14.1

					Weight Per	cent	
Run No.	Source	<u>Ca</u>	<u> </u>	<u> </u>	C03	Total C	Mg
73.2	Second Cyclone No. 11	19.1	_	16.88	2.48	2.44	10,2
	Third Cyclone No. 11	16.6	_	19.86	1.63	1.06	6.50
	Bed Probe Sample No. 11	24.6	_	33,63	0.86	0.47	13.9
	No. 12	25.2	_	33.04	1.04	0.46	14.5
	No. 13	24.5	-	31.87	0.48	0.20	14.3
74	Initial Bed	38.6	_	19.62	28.60	6,34	•
	Final Bed	47.7	-	19.7	1.54	0.34	-
74.1	Second Cyclone No. 3	24.0	=	14.7	2.62	5.44	_
	No. 4	27.2	-	14.3	2.46	5.21	_
	Third Cyclone No. 3	19.3	-	17.2	3.54	3.99	_
	No. 4	13.7	_	16.6	4.56	3.04	_
	Bed Probe Sample No. 1	44.8	_	15.7	5.99	1.37	_
	No. 4	47.5	-	18.0	2.59	0.65	•
	No. 5	47.8	-	15.8	2.08	0.47	-
74.2	Second Cyclone No. 9	22.8	_	13.4	1.93	5.85	•
	No. 10	20.3	-	13.6	1.51	6.03	-
	Third Cyclone No. 9	19.4	-	19.8	3.93	2.84	-
	No. 10	15.6	-	18.9	3.96	3.06	-
	Bed Probe Sample No. 10	52.9	•	12.8	2.32	0.43	•
	No. 11	52.8	-	14.6	1.84	0.53	-
	No. 12	51.9	-	17.85	2.11	0.43	-
75	Initial Bed	52.9	-	17.43	1.61	0.36	-
	Second Cyclone No. 5	13.73	-	13.11	0.67	6.87	-
	No. 6	14.2	-	14.12	0.78	5.48	-

				ļ	Weight Per	cent	
Run No.	Source	<u>Ca</u>	<u> </u>	S04	C03	Total C	Mg
75	Bed Overflow No. 5	46.15	-	24.76	1,41	0.37	•
(Cont'd.)	Third Cyclone No. 5	13.3	-	19.19	-	3,05	-
	No. 6	13.8	-	18.69	8.72	3.26	-
	Bed Probe Sample No. 1	46.5	-	16.98	2.21	0.53	-
	No. 6	51.7	-	19.23	1.89	0.43	-
	No. 7	50.8	-	20.99	1.70	0.44	•
	No. 8	50.7	-	19.04	0.61	0.45	-
	Final Bed	45.6	-	24.72	1.36	0.29	-
76	Initial Bed	25.3	-	39.63	0.61	0.22	13.6
	Final Bed	29.7	-	40.83	0.33	0.11	11.9
	Second Cyclone No. 6	15.0	-	16,53	0.30	4.38	5.33
	No. 7	16.4	-	16.28	0.32	4.12	5,84
	Third Cyclone No. 6	12.6	-	14.70	1.57	2.01	2.02
	No. 7	12.0	-	14.07	1.34	2.39	2.36
	Bed Probe Sample No. 1	27.0	-	38.44	3.61	0.69	13.1
	No. 7	27.7	-	40.13	0.99	0.22	10.8
	No. 8	29.2	-	39.14	0.93	0.25	11.6
	No. 13	24.0	-	38.16	0.40	0.13	11.7
77	Second Cyclone No. 6	13.0	-	20.87	0.52	3.54	6.83
	Bed Overflow No. 4	24.8	-	36.00	0.49	0.15	12.3
	Third Cyclone No. 6	9.16	-	17.87	0.90	1.86	2.83
78	Second Cyclone No. 72	12.9	-	9.14	0.27	0.56	7.43
	Bed Overflow No. 72	23.5	-	39.10	0.68	0.27	12.9
	Third Cyclone No. 72	8.41	_	15.16	0.82	0.50	3.82
	Bed Probe Sample No. 8	24.8	-	39.41	1.27	0.43	13.5
	No. 9	22.13	-	29.53	2.65	0.62	14.35
80	Second Cyclone No. 100	12.30	6.48	20.19	0.90	0.62	7,48
	Bed Overflow No. 100	13.67	11.07	31.93	3.03	0.65	7.67
	Third Cyclone No. 100	24.69	5.06	15.12	0.57	0.39	14.44

_					Weight Pe	rcent	
Run No.	Source	<u>Ca</u>	<u> </u>	<u> </u>	_C03	Total C	Mg
81	Initial Bed	25.34	11.43	37.24	1.22	0.31	15.06
	Final Bed	24.47	13.15	40.87	0.10	0.07	14.23
	Second Cyclone No. 15	-	-	-	4,48	2.22	-
	No. 51	17.18	7.65	32.10	4.93	1.64	10.52
	No. 62	16.74	7.92	24.16	5.43	2.93	11.01
	No. 74	-	-	-	5.07	1.55	-
	No. 86	-	-	-	3.89	1.77	-
	Third Cyclone No. 15	-	-	-	0.44	1,14	-
	No. 51	10.51	8.40	26.01	0.49	0.54	6.52
	No. 62	10.15	8.60	26.44	0.22	1.57	7.25
	No. 74	-	-	-	0.11	0.44	-
	No. 86	-	-	-	0.08	0.56	-
	Bed Overflow No. 15	25 20	-	-	0.95	0.26	-
	No. 51	25.38	12.65	37.78	0.40	0.08	15,18
	No. 62	23.11	13.93	42.50	0.39	0.16	14.41
	No. 74 No. 86	-	-	-	0.28	0.11	-
	110.00	-	-	-	0.33	0.25	-
99	Dolomite Blend "A"	21.67	-	-	64.70	-	13.19
99.2	Second Cyclone No. 13	14,76	7.17	20,20	4.91	4.29	8.59
	Third Cyclone No. 13	6.63	4.93	18.81	0.24	2.35	4.19
	Bed Probe Sample No. 1	25.24	14.95	42.25	0.31	0.09	12.91
	No. 2	24.73	14.33	42.43	0.37	0.13	13.55
99.3	Second Cyclone No. 14	12.94	6.30	19.54	3.73	2.39	7.96
	No. 15	9.87	5.52	16,63	1.77	1.57	6.05
	Third Cyclone No. 15	5.98	5.93	14.98	0.03	3.11	3.59
	Bed Probe Sample No. 3	24.40	15.04	43.76	0.37	0.09	12.94
	No. 5	25.28	13,50	44.34	0.25	0.12	13.76

					Weight Per	rcent	
Run No.	Source	<u>Ca</u>	<u> </u>	<u> </u>	_C03	Total C	Mg
99.4	Second Cyclone No. 2	13,46	6.02	18.94	1.54	1.46	6.36
	Third Cyclone No. 21	7.06	7.21	23.14	0.01	0.75	5,14
	Bed Probe Sample No. 11	24.47	14.09	44.46	0.54	0.21	13.23
99.5	Second Cyclone No. 22	17.21	7.96	21.84	2,62	3,76	8.11
	No. 23	19.49	7.11	23.07	3,69	5.59	9.21
	Third Cyclone No. 23	6.91	7.78	23.28	0.10	1.62	5.43
	Bed Probe Sample No. 12	24.49	13,35	44.5	0.70	0.14	12.95
	No. 14	23.53	14.95	42.38	0.32	0.08	13.14
99.7	Second Cyclone No. 36	18,40	6,81	20.46	4,71	2,60	8,71
	Third Cyclone No. 36	7.90	5,58	18.15	0.91	1,46	4.06
	Bed Probe Sample No. 32	22.32	14.16	45.03	0.39	0.09	13.02
100	Dolomite Blend "A"	21.85	0.05	-	64.97	-	13.07
100.1	Second Cyclone No. 4	14,16	4.81	15.10	2,19	5.48	6.19
	No. 5	14.47	4,62	14.69	2.16	4.38	6.21
	Third Cyclone No. 4	7,83	4,65	15.45	1.21	1.95	3.95
	Bed Probe Sample No. 6	25,16	14.58	46.44	0.15	0.04	11.99
	. No. 7	25.03	13,60	44.01	0.28	0.06	12.00
100.2	Second Cyclone No. 6	10,36	3,59	11.93	1.40	4.09	4.52
	Third Cyclone No. 6	6.36	4.04	11.67	0.25	1.66	2.95
	Bed Probe Sample No. 8	24.19	13.73	44.60	0.14	0.05	11.60
100.4	Second Cyclone No. 12	10.00	3.45	9.20	1.01	3.97	3.78
	No. 13	9,31	2.92	9.85	1.08	5.56	3.89
	No. 14	12.91	4.79	13.55	2.19	4.39	5.61
	Third Cyclone No. 12	6.43	3,37	10.75	0.35	1,65	2.54
	No. 14	5,03	3,16	10,61	0.00	1.86	2.41
	Bed Probe Sample No. 13	24,51	13.89	44.71	0.32	0.08	11,96
	No. 15	24,87	14.12	45,33	0.17	0.06	11.71
	No. 17	24.74	13.76	44.04	0.13	0.07	11.96

					Weight Pe	rcent	
Run No.	Source	Ca	<u> </u>	<u> </u>	C03	Total C	Mg
100.6	Second Cyclone No. 29	15,61	5.52	17.00	3.26	3.43	7,24
	No. 31	14,25	5.33	15,31	2,42	4.09	6.33
	Third Cyclone No. 31	6.59	4.47	13.16	0.12	1.26	3.11
	Bed Probe Sample No. 32	22.99	13.49	42.43	0.06	0.03	10.86
	No. 33	24.33	13.32	42.09	0.10	0.02	11.60
102	COMBUSTOR						
	Second Cyclone No. 30	29.2	2.63	7.01	1.70	4.63	_
	Third Cylcone No. 30	18.6	2.87	8.02	3.13	3,11	-
	Bed Probe Sample No. 4	55.3	2.05	6,87	0.83	0.25	-
	Final Bed	47.8	3.43	11.67	0.69	0.22	-
	REGENERATOR			•			
	First Cyclone No. 29	40.0	3.74	9.67	3.91	-	-
	Final Bed	49.9	1.65	4.24	0.18	0.13	-
103	COMBUSTOR						
	Second Cyclone No. 33	23.3	4.32	12.5	1.23	2.60	-
	No. 40	15.8	4.29	11.0	0.52	6.43	-
	No. 52	12.9	3.00	9.19	0.29	8.80	-
	No. 60	18.0	3.58	10.89	0.71	4.69	-
	No. 75	19.0	4.00	11.97	0.93	3.28	-
	No. 87	14.1	2.95	8.83	0.65	5.64	•
	No. 99	6.04	1.58	4.61	0.05	9.60	-
	Third Cyclone No. 33	16.3	3.15	9.34	1.24	1.49	-
	No. 40	10.5	3.70	11.08	1.25	3.23	-
	No. 52	8.93	3.14	9.07	0.20	3.52	-
	No. 60	13.5	2.98	9.48	0.55	1.19	-
	No. 75	13.9	2.96	8.99	0.49	0.93	-
	No. 87	12.1	2.34	7.49	0.26	1.57	-
	No. 99	7.46	2.14	6.67	0.05	2.45	-

					Weight Per	rcent	
Run No.	Source	Ca	<u>s</u>	<u>\$04</u>	C03	Total C	Mg
103	Bed Probe Sample No. 28	47.8	8.76	24.21	0.53	0.14	_
(Cont'd.)	No. 40	48,3	6.10	17.88	4,33	0.85	_
	No. 52	46.1	8.60	22.72	0.54	0.17	-
	No. 60	45.1	10.15	29,21	1.83	0.44	-
	No. 15	44.1	7.67	23.48	0.91	0.20	-
	No. 87	45.2	8.05	22.90	1.34	0.32	_
	No. 99	38.8	11.64	33.51	1.45	0.33	-
	Final Bed	39.4	13.60	40.5	0.88	0.21	-
	REGENERATOR						
	Sand Filters No. 40	35.4	5,16	13.51	1.03	1.46	_
	No. 50	32.8	4.95	13,73	0.76	1.34	_
	No. 97	30.1	5.61	14.29	3.78	1.67	-
	Reg. to Comb. Pulse Pot	39.1	11.07	31.87	1.53	0.42	_
	Final Bed	40.1	9.26	27.58	0.69	0.21	-
104	Second Cyclone	12.6	5,92	19.43	0.04	1.06	7,96
	Third Cyclone	8.36	3,24	10.69	0.00	0.43	3.22
	Bed Overflow	20.8	11.50	33.34	0.64	0.19	12.4
	Bed Probe	21.7	11,93	39.64	0.39	0.15	14.0
	Final Bed	20.7	10.82	36.09	1.07	0.33	12.5
105	COMBUSTOR						
103	Second Cyclone No. 28	14.7	2.70	19.56	0.42	6.10	_
	No. 44	13.4	3.56	9.84	0.24	6.14	_
	Third Cyclone No. 28	15.9	2.08	7,54	0.18	1.69	_
	No. 44	13.2	2.21	7.50	0.10	1,95	_
	Bed Probe Sample	44.2	7.60	25.31	0.33	0.26	-
	bed frome sample	43.1	7.16	23.80	0.33	0.22	-
	Initial Bed	37.8	9.42	16.84	9.09	7.99	-
	Final Bed	37.3	14.77	31.66	0.70	0.18	_
	i iligi Deu	57.5	17,77	000	0	0,	

			<u>Weight Percent</u>							
Run No.	Source	Ca	<u> </u>	<u> </u>	C03	Total C	Mg			
105	REGENERATOR									
(Cont'd.)	Cyclone No. 25	32.8	2.46	7.26	3.66	2.45	-			
•	No. 41	35.0	1.78	7.44	3.57	1.91	-			
	Bed Sample	37.7	3.84	10.28	1.58	3.80	-			
	Initial Bed	35.7	7.01	24.59	8.19	1.27	_			
	Final Bed	44.7	3.11	10.47	0.59	0.27	-			
107	Second Cyclone No. 27	8.35	4.07	12,14	0.45	4,62	4.41			
	No. 50	9.16	3,42	10.42	0.22	5.78	4.88			
	Third Cyclone No. 27	5.70	2.97	9.57	0.20	1.66	2.41			
	No. 50	6.10	3,21	10.06	0	1.33	2.48			
	Bed Overflow No. 24	20.4	11,07	34.42	7.27	1.54	12.5			
	No. 44	22.2	12.84	39.72	4.81	1.06	13.6			
108	Second Cyclone - Final	11.5	-	16.93	0.36	3,33	6.49			
	Third Cyclone - Final	7,62	-	10.68	0.03	1.24	3.32			
	Bed Overflow - Final	21.9	-	41.90	3.39	0.93	13.8			

	Reguestor	Sample Description	Sorbent	Sorbent Run No. Amount		Date	
	Acurex-Aerotherm Mountain View, CA	3rd Cyclone Flyash 2nd Cyclone Flyash 2nd Cyclone Flyash	Dolomite Dolomite	67 67 Combination of Many Runs	15 Gallons 2 Drums 18 Drums	1/27/78 4/4/78 4/6/78	
	Atlantic Richfield Corp. Alexandria, VA	3rd Cyclone Flyash	Dolomite	67	2 Grams	4/4/78	
	Battelle Columbus, OH	3rd Cyclone Flyash	Dolomite	80	20 Grams	12/4/78	
	Colorado State University Fort Collins, CO	Scrubber Water 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite	81 81 81	l Drum l Quart l Quart	11/7/78 11/7/78 11/7/78	
301	Cornell University Ithaca, NY	2nd Cyclone Flyash Bed Overflow 2nd Cyclone Flyash Bed Overflow 3rd Cyclone Flyash	Limestone Limestone Dolomite Dolomite Dolomite	74 74 78 78 78	10 Gallons 10 Gallons 10 Gallons 10 Gallons 5 Gallons	8/15/78 8/15/78 8/15/78 8/15/78 9/28/78	
	EFB, Inc. Woburn, MA	Bed Overflow 3rd Cyclone Flyash Fresh Dolomite	Dolomite Dolomite	78 78 	4 Drums 1/2 Drum 1-1/2 Tons	11/14/78 11/28/78 12/21/78	
	ERE						
	Baton Rouge, LA	3rd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite	67 78	10-1/2 lbs. 1 Drum	6/16/78 7/19/78	
	Florham Park, NJ	2nd Cyclone Flyash 2nd Cyclone Flyash	Dolomite Dolomite	80 93	1 Gallon 1 Drum	1/8/79 3/9/79	
	Linden, NJ	2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite	67 67	1 1b. 1 1b.	9/28/78 9/28/78	
	EPA						
	Research Triangle Park, NC	2nd Cyclone Flyash 3rd Cyclone Flyash 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite Dolomite Dolomite	50.1B 80 81 81	1 Drum 20 Grams 5 Gallons 5 Gallons	8/30/77 12/4/78 3/28/79 3/28/79	

Requestor	Sample Description	Sorbent	Run No.	Amount	Date
EPA (Cont'd)		· · · · · · · · · · · · · · · · · · ·		<u> </u>	
Rivesville, WV	2nd Cyclone Flyash 3rd Cyclone Flyash Bed Overflow Bed Overflow Bed Overflow 2nd Cyclone Flyash	Dolomite Dolomite Dolomite Dolomite Dolomite Dolomite	108 108 108 66 67 67	<ul><li>1 Quart</li><li>1 Quart</li><li>1 Quart</li><li>1 Drum</li><li>12 Drums</li><li>5 Drums</li></ul>	8/13/79 8/13/79 8/13/79 2/2/78 2/2/78 2/2/78
GCA Corporation Bedford, MA	Final Bed (Combustor) Final Bed (Regenerator)	Limestone Limestone	105 105	10 Gallons 10 Gallons	5/14/79 5/14/79
General Electric King of Prussia, PA Schenectady, NY  Valley Forge, PA	3rd Cyclone Flyash 3rd Cyclone Flyash 3rd Cyclone Flyash Illinois Coal No. 6 Pfizer Dolomite 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite Dolomite Dolomite Dolomite	68 68 81  50.4 80	5 Gallons 5 Gallons 1 Small Jar 1 Quart 1 Quart 1 Drum 5 Gallons	4/17/79 1/18/79 11/13/78 3/14/78 3/14/78 9/30/77 9/22/78
MIT Cambridge, MA	Bed Overflow Bed Overflow 2nd Cyclone Flyash	Dolomite Dolomite Dolomite	78 78 78	5 Gallons 25 Gallons 25 Gallons	8/18/78 10/17/78 10/17/78
N.J. Institute of Technology Newark, NJ	Illinois Coal No. 6			25 lbs.	10/20/78
N.Y. University Westbury, NY	Champion Coal			1 Drum	3/20/79
Pratt & Whitney Aircraft Co. Middletown, CT	2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite	68 67	10 lbs. 10 lbs.	5/2/78 5/2/78
Radian Corporation Austin, TX	Bed Overflow 2nd Cyclone Flyash Bed Overflow 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite Dolomite Dolomite	79 79 80 80 80	2 Drums 3 Drums 21 Drums 15 Drums 1 Drum	1/24/79 1/24/79 1/24/79 1/24/79 1/24/79

### APPENDIX 0 (CONT'D). MINIPLANT SAMPLE SHIPMENTS

	Requestor	Sample Description	Sorbent	Run No.	Amount	Date
	Radian Corporation (Cont'd)	3rd Cyclone Flyash Bed Overflow	Dolomite Dolomite	81 79	1/2 Drum 1 Small Bottle	1/24/79 1/19/79
		2nd Cyclone Flyash Bed Overflow	Dolomite Dolomite	79 80	1 Small Bottle 1 Small Bottle	1/19/79
		2nd Cyclone Flyash 3rd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite Dolomite	80 80 81	1 Small Bottle 1 Small Bottle 1 Small Bottle 1 Small Bottle	1/19/79 1/19/79
	Sandia Laboratory Livermore, CA	2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite	78 78	10 lbs. 10 lbs.	2/6/79 2/6/79
	TVA Muscle Shoals, AL	Bed Overflow	Dolomite	78	6 Drums	5/8/79
303	University of Cincinnati Cincinnati, OH	3rd Cyclone Flyash 3rd Cyclone Flyash 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Dolomite Dolomite Dolomite	80 81 78 106	5 Gallons 5 Gallons 5 Gallons 10 Gallons	2/6/79 2/27/79 2/27/79 8/9/79
	University of Denver Denver, CO	2nd Cyclone Flyash Granular Bed Filter Particulates	Dolomite Dolomite	59 59	1 1b. 1 1b.	9/7/77 9/7/77
	University of S. California Los Angeles, CA	Illinois Coal No. 6 Pfizer Dolomite 2nd Cyclone Flyash	Dolomite	 78	1 1b. 1 1b. 1 1b.	8/15/78 8/15/78 8/15/78
		3rd Cyclone Flyash Bed Overflow Champion Coal Ohio Coal	Dolomite Dolomite	78 78 	1 1b. 1 1b. 1 1b. 1 1b.	8/15/78 8/15/78 10/2/78 10/2/78
		Pfizer Dolomite Grove Limestone 2nd Cyclone Flyash	  Dolomite	  50	1 1b. 1 1b. 1 1b.	10/2/78 10/2/78 10/2/78 10/2/78
		Bed Overflow 2nd Cyclone Flyash 3rd Cyclone Flyash	Dolomite Limestone Limestone	50 75 75	1 1b. 1 1b. 1 1b.	10/2/78 10/2/78 10/2/78 10/2/78

### APPENDIX O (CONT'D). MINIPLANT SAMPLE SHIPMENTS

Requestor	Sample Description	Sorbent	Run No.	Amount	Date
University of S. California	Bed Overflow	Limestone	75	1 1b.	10/2/78
(Cont'd)	2nd Cyclone Flyash	Dolomite	77	1 1b.	10/2/78
	3rd Cyclone Flyash	Dolomite	77	1 16.	10/2/78
	Bed Overflow	Dolomite	77	1 1b.	10/2/78
Valley Forge Laboratories	Bed Overflow	Dolomite	67	25 lbs.	2/2/78
Devon, PA	2nd Cyclone Flyash	Dolomite	67	25 lbs.	2/2/78
Vanderbilt University	Bed Overflow	Limestone	26	5 1bs.	4/26/78
Nashville, TN	Bed Overflow	Dolomite	45	5 lbs.	4/26/78
	Bed Overflow	Dolomite	52	5 lbs.	4/26/78
Westinghouse Research Labs	3rd Cyclone Flyash	Dolomite	67	500 Grams	2/16/78
Pittsburgh, PA	3rd Cyclone Flyash	Dolomite	79	1 Drum	10/27/78
	3rd Cyclone Flyash	Dolomite	<b>5</b> 9	10 lbs.	10/27/78
	2nd Cyclone Flyash	Dolomite	79	10 lbs.	10/27/78
	2nd Cyclone Flyash	Dolomite	68	l Small Jar	1/8/79
	3rd Cyclone Flyash	Dolomite	<b>68</b>	l Small Jar	1/8/79
	2nd Cyclone Flyash	Dolomite	69	l Small Jar	1/3/79
	Bed Overflow	Dolomite	69	1 Small Jar	1/3/79
	2nd Cyclone Flyash	Dolomite	70	l Small Jar	1/3/79
	Bed Overflow	Dolomite	70	l Small Jar	1/3/79
	2nd Cyclone Flyash	Dolomite	71	1 Small Jar	1/3/79
	3rd Cyclone Flyash	Dolomite	71	l Small Jar	1/3/79
	Bed Overflow	Dolomite	71	l Small Jar	1/3/79
	2nd Cyclone Flyash	Dolomite	72	l Small Jar	1/3/79
	3rd Cyclone Flyash	Dolomite	72	l Small Jar	1/3/79
	Bed Overflow	Dolomite	72	l Small Jar	1/3/79
	2nd Cyclone Flyash	Dolomite	73	l Small Jar	1/3/79
	3rd Cyclone Flyash	Dolomite	73	l Small Jar	1/3/79
	Bed Overflow	Dolomite	73	1 Small Jar	1/3/79
	2nd Cyclone Flyash	Limestone	74	1 Small Jar	1/3/79
	3rd Cyclone Flyash	Limestone	74	l Small Jar	1/3/79
	Bed Overflow	Limestone	74	1 Small Jar	1/3/79
	3rd Cyclone Flyash	Dolomite	81	1 Gallon	2/26/79

#### APPENDIX 0 (CONT'D). MINIPLANT SAMPLE SHIPMENTS

Requestor	Sample Description	Sorbent	Run No.	Amount	<u>Date</u>	
Westinghouse Research Labs (Cont'd)	lst Cyclone (Dipleg) Material	Dolomite	79	2-1/2 Gallons	4/28/79	
	3rd Cyclone Flyash	Dolomite	81	2 Gallons	5/11/79	
	2nd Cyclone Flyash	Limestone	105	1 Small Jar	8/17/79	
	3rd Cyclone Flyash	Limestone	105	1 Small Jar	8/17/79	
	Bed Probe	Limestone	105	1 Small Jar	8/17/79	
	Regenerator Cyclone Ash	Limestone	105	1 Small Jar	8/17/79	
	Regenerator Final Bed	Limestone	105	l Small Jar	8/17/79	

APPENDIX P-1. BENCH COMBUSTOR RUN SUMMARY

	Program		Initial Checkout						
	Run No.	1.1(1)	1.2(1)	2.1(1)	3.1(1)	3.2(1)	4.1(1,2)	4.2(1,2)	
	Operating Conditions								
	Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State	770 880 2.9 146 10.3 0.5 1.9 0.28 2.43	770 890 2.9 153 10.5 0.5 1.9 0.27 2.71	760 910 3.0 111 10.1 0.5 1.9 0.26 3.54 1.9	760 880 1.6 37 8.0 0.9 1.1 0.83 2.78 2.2	760 780 1.7 41 7.5 0.9 1.1 0.88 2.87	770 865 2.2 16 12.1 1.1 0.77 2.87	770 870 2.2 13 12.9 1.2 1.4 0.84 2.68 0.5	
•	Flue Gas Emissions	(111 ) 3.7	0.0	1.5	۷,٤	1,5	1.4	0.3	
	SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sub>x</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	550 200 175 12.0 13	450 180 125 12.0 13	570 270 500 11.4 11	790 190 325 13.6 5.8	900 200 350 11.4 6.2	1000 120 200 16.8 2.9	800 140 225 17.8 2.5	
	Results								
	SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	62 2.0 0.50	69 1.6 0.46	60 2.2 0.74	57 2.0 0.35	47 2.6 0.42	54 2.4 0.20	65 1.8 0.22	

<sup>(1)</sup> Grove Limestone; Arkwright Coal

<sup>(2) 1</sup>st Cyclone Solids Recycled

#### APPENDIX P-1 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

	Program	Init	ial Checkout	•						
	Run No.	5.1 <sup>(1,2)</sup>	6.1(1)	6.2 <sup>(1)</sup>	7.1 <sup>(1)</sup>	8.1(1,2)	9.1 <sup>(1)</sup>			
	Operating Conditions									
307	Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr)	760 880 1.8 16 10.5 0.9 1.2 0.76 0.96 2.2	760 915 1.9 41 10.2 0.8 1.3 0.61 1.09	750 900 2.0 13 11.3 1.1 1.7 0.84 1.58 0.4	750 950 1.3 44 4.7 0.5 1.0 0.54 3.77	750 930 1.6 37 7.1 0.6 1.0 0.61 1.30 1.2	770 960 1.4 11 11.0 0.9 1.3 0.66 1.97			
	Flue Gas Emissions	•	·		-					
	SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sub>x</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	630 95 175 15.4 3.0	430 150 125 14.2 6.2	100 120 150 15.0 2.5	100 180 75 12.5 6.5	160 160 250 14.8 5.8	218 160 125 16.4 2.1			
	Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU	72 1.4 0.15	81 1.0 0.26	95 0.23 0.20	92 0.36 0.46	91 0.47 0.34	89 0.35 0.18			

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<sup>(1)</sup> Pfizer Dolomite; Arkwright Coal

<sup>(2) 1</sup>st Cyclone Solids Recycled

APPENDIX P-2. BENCH COMBUSTOR RUN SUMMARY

Program	Two Stage	Combustic	n-NO <sub>X</sub> Contr	lor			
Run No.		10.1(1)	11.1(1)	11.2(1)	12.1 <sup>(2)</sup>	12.2 <sup>(2)</sup>	13.1 <sup>(2)</sup>
Operating Conditions							
Pressure (kPaa)		770	765 070	765	770	770	770
Bed Temperature (°C) Air Flow Rate (m <sup>3</sup> /min)		920 0.9	870 1 <b>.</b> 9	870 1 <b>.</b> 8	960 1.5	950 1.4	950 1.7
Excess Air (%)		39	23	18	25	8	17
Coal Feed Rate (kg/hr) Expanded Bed Height (m)		8.3 0.9	18.2 1.0	18.2 1.2	10.9 1.1	10.9	13.5
Superficial Velocity (m/s)		0.6	1.2	1.1	1.3	1,3 1,3	1.1 1.5
Gas Residence Time (s) Ca/S Molar Ratio		1.4 6.2	0.8 2.9	1.1 2.9	0.79 3.5	1.03 3.5	0.70 3.1
Run Length at Steady State (hr)		8.0	0.3	0.2	0.7	0.4	0.6
Primary Air (% Stoic.) Secondary/Primary Air		70 0.37	66 0.32	61 0.34	105 0	75 0.34	97 0
Secondary Air Injection (cm Abo	ve Grid)	30.7	30.7	30.7		30.7	
Flue Gas Emissions							
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sup>x</sup> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)		900 120 200 13.0 6.0	1455 60 >5000 15.4 4.0	1170 140 650 18,6 3,2	588 250 125 15.4 4.3	960 120 150 19.0 1.6	400 240 140 17.6 3.1
Results							
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU		42 1.27 0.12	17 1.86 0.06	37 1.47 0.12	65 0.98 0.30	53 1.54 0.14	79 0.62 0.27
LDS HOX/HB IO		••••	•••	••••	• • • •	- • •	• • -

<sup>(1)</sup> Pfizer Dolomite; Champion Coal

<sup>(2)</sup> Grove Limestone; Champion Coal

### APPENDIX P-2 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program Two Stage	Combustio	n-NO <sub>x</sub> Cont	rol			
Run No.	13.2 <sup>(1)</sup>	14.1 <sup>(1)</sup>	14.2 <sup>(1)</sup>	17.1(1)	17.2 <sup>(1)</sup>	17A.1 <sup>(2)</sup>
Operating Conditions						
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr) Primary Air (% Stoich.) Secondary/Primary Air	770 950 1.7 16 11.4 1.0 1.5 0.70 3.1 1.0 91	760 880 1.8 19 12.5 1.6 1.5 1.1 2.8 1.0	760 930 1.5 20 11.0 1.2 1.4 0.87 2.9 2.0 79 0.38	770 850 1.3 21 10.5 1.2 1.0 1.2 2.7 0.5 92	770 830 1.3 31 9.1 1.2 1.1 1.1 3.2 0.5 86 0.33	480 870 1.5 41 10.0 2.0 0.5 2.9 0.6 116 0
Secondary Air Injector (cm Above Grid)	5.9	0	5.9		5.9	
Flue Gas Emissions						
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sup>x</sup> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	600 145 125 16.4 2.9	840 180 150 15.2 3.4	453 150 150 15.4 3.5	769 140 275 14.6 3.8	829 160 250 14.0 5.0	795 190 400 14.4 6.2
Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	68 1.01 0.19	60 1.48 0.23	75 0.79 0.19	57 1.15 0.15	50 1,52 0,21	48 1.48 0.26

<sup>(1)</sup> Grove Limestone; Champion Coal

<sup>(2)</sup> Grove Limestone; Arkwright Coal

#### APPENDIX P-2 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program Two St	age Combustion		1 (2)	(0)		4-3
Run No.	17A.2 <sup>(1)</sup>	17A.3 <sup>(1)</sup>	20.1 (2)	20.2(2)	20.3(2)	21.1 <sup>(2)</sup>
Operating Conditions						
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr)	480	480	490	490	490	480
	810	820	900	890	900	840
	1.3	1.4	1.6	1.5	1.5	1.2
	18	18	15	18	19	18
	9.4	9.5	10.8	11.2	11.3	10.0
	1.4	1.2	1.1	1.2	1.2	1.2
	1.7	1.8	2.2	2.0	2.1	1.6
	0.76	0.67	0.52	0.59	0.57	0.77
	3.1	3.1	3.0	2.9	2.8	2.9
	1.9	2.0	1.3	1.5	2.5	1.3
Primary Air (% Stoich.) Secondary/Primary Air Secondary Air Injector (cm Above Grid	89	72	115	85	74	93
	0.24	0.57	0	0.22	0.42	0
	1) 15	15		30	30	
Flue Gas Emissions  SO <sub>2</sub> (ppm) NO <sup>2</sup> (ppm) CO <sup>3</sup> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	1230	1390	1080	1080	1060	1490
	185	190	240	190	190	170
	500	550	300	300	300	540
	15.0	15.0	16.4	15.8	15.8	14.4
	3.3	3.2	2.8	3.2	3.4	3.3
Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU	33	25	43	42	42	19
	2.16	2.53	2.02	1.79	1.78	2.25
	0.23	0.25	0.32	0.23	0.20	0.20

<sup>(1)</sup> Grove Limestone; Arkwright Coal

<sup>(2)</sup> Grove Limestone; Champion Coal

### APPENDIX P-2 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program Two	o Stage	${\tt Combustion-NO_X}$			4- 1	•-•
Run No.		21.2 <sup>(1)</sup>	21.3 <sup>(1)</sup>	22.1 <sup>(2)</sup>	22.2(1)	22.3(1)
Operating Conditions						
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio		480 860 1.3 16 10.6 1.3 1.7 0.74 2.7	505 840 1.2 15 9.6 1.4 1.5 0.91 2.93	515 950 2.0 35 14.2 1.3 2.7 0.48 3.20	505 880 1.7 37 11.5 1.1 2.2 0.52 3.13	505 890 1.6 31 11.4 1.2 2.1 0.56 3.15
Run Length at Steady State (hr) Primary Air (% Stoich.) Secondary/Primary Air Secondary Air Injector (cm Above	e Grid)	1.3 80 0.23 30	1.4 73 0.33 30	1.5 111 0 	2.0 91.2 0.24	1.7 80 0.39
Flue Gas Emissions  SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sup>x</sup> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)		1410 190 425 15.0 3.0	1020 140 500 15.6 2.8	500 245 225 14.2 5.5	900 230 350 15.0 5.7	880 210 350 15.0 5.0
Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU		25 2.23 0.22	46 1.57 0.15	67 0.89 0.31	43 1.63 0.30	47 1.56 0.27

<sup>------</sup>

<sup>(1)</sup> Grove Limestone; Champion Coal

<sup>(2)</sup> Pfizer Dolomite; Champion Coal

APPENDIX P-3. BENCH COMBUSTOR RUN SUMMARY

Program	NH <sub>3</sub> Injection-NO <sub>X</sub> Control						
Run No.	1.1	1.2	1.4	3,1	3.2	4.1	4.2
Operating Conditions							
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr) NH3/NO <sub>X</sub> Molar Ratio	760 885 1.5 19 8.4 1.3 1.3 0.98 3.05 1.5	760 890 1.5 16 8.4 1.3 0.95 3.07 2.2 0.88	760 885 1.5 16 7.9 1.2 1.3 0.93 3.22 0.9 0.88	760 890 1.5 15 9.4 1.5 1.3 1.14 2.94 2.8	760 890 1.5 13 10.7 1.4 1.3 1.09 2.57 1.3 2.27	760 895 1.4 16 11.5 1.4 1.2 2.08 1.8	760 900 1.4 14 11.9 1.4 1.2 1.14 2.04 1.4
H <sub>2</sub> /NH <sub>3</sub> Molar Ratio NH <sub>3</sub> Injection Location		0.0 L1	2.00 L1		1.48 L1		1.00 L1
Flue Gas Emissions							
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sub>x</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	700 140 200 16.0 3.8	675 117 550 16.0 3.1	600 160 450 16.0 3.0	675 145 200 16.0 2.9	588 133 500 16.0 2.7	825 155 200 15.2 3.3	800 143 200 15.4 2.9
Results							
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	61 1.25 0.18	63 1.24 0.16	65 1.17 0.23	68 1.08 0.17	75 0.81 0.13	70 0.98 0.13	72 0.90 0.12

### APPENDIX P-3 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program	NH <sub>3</sub> Inje	ction-NO <sub>x</sub>	Control				
Run No.	4.3	5.1	5.2	5.3	5.4	5.5	5,6
Operating Conditions							
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr) NH3/NO <sub>X</sub> Molar Ratio H <sub>2</sub> /NH <sub>3</sub> Molar Ratio NH <sub>3</sub> Injection Location	760 900 1.4 17 11.9 1.4 1.2 1.13 1.99 1.0	760 895 1.5 19 7.9 1.3 1.01 3.07 1.5	760 905 1.5 19 10.0 1.1 1.3 0.89 2.46 0.8 2.18 2.94 L2	760 915 1.5 18 10.0 1.4 1.3 1.06 2.46 1.7 1.27 2.50 L2	760 905 1.5 18 9.0 1.4 1.3 1.03 2.67 1.3	760 900 1.5 18 8.8 1.3 1.3 0.99 2.78 1 3.41 0.0 L2	760 890 1.5 17 8.8 1.4 1.3 1.06 2.70 1.1 8.05 0.0 L2
Flue Gas Emissions							
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sub>x</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	725 170 450 15.4 3.4	725 145 350 14.8 3.3	625 210 475 14.8 3.4	625 200 375 14.8 3.3	600 160 325 15.2 3.3	575 210 300 15.2 3.2	550 250 375 15.2 3.1
Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	75 0.79 0.13	58 1.49 0.22	71 0.99 0.24	71 0.90 0.23	69 1.04 0.20	70 1.03 0.27	71 0.99 0.32

## APPENDIX P-3 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program	NH <sub>3</sub> Inj	ection-NO <sub>X</sub>	Control				
Run No.	5.7	6.1	6.2	6.3	7.1	7.2	7.3
Operating Conditions							
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr) NH3/NO <sub>X</sub> Molar Ratio	760 890 1.5 17 8.8 1.3 1.3 1.0 2.70 0.9	660 890 1.4 16 9.2 1.3 1.4 0.93 2.77 1.2	660 880 1.4 18 8.4 1.3 1.4 0.92 3.03 1.0 0.82	660 890 1.4 17 8.5 1.4 1.4 0.96 2.97 1.0	660 890 1.4 19 9.2 1.3 1.5 0.90 2.80 1.2	660 890 1.4 18 9.2 1.3 1.4 0.92 2.80 1.1 1.36	660 890 1.4 17 9.2 1.3 1.4 0.90 2.80 1.3 2.25
H <sub>2</sub> /NH <sub>3</sub> Molar Ratio NH <sub>3</sub> Injection Location			0.0 L3			0.0 L3	0.0 <b>L</b> 3
Flue Gas Emissions							
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sup>x</sup> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	475 170 400 15.4 3.1	650 150 475 16.0 3.0	563 103 650 16.8 3.5	575 170 675 16.8 3.5	675 155 525 14.8 3.3	525 100 625 15.2 3.3	375 83 825 16.0 3.3
Results							
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU	75 0.85 0.22	70 0.99 0.16	71 0.89 0.12	71 0.88 0.19	68 1.11 0.18	75 0.84 0.12	82 0.58 0.09

#### APPENDIX P-3 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program	NH <sub>3</sub> Injection-NO <sub>x</sub> Control					
Run No.	7.4	8.1	8.2	8.3	8.4	8.5
Operating Conditions						
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (hr) NH3/NO <sub>X</sub> Molar Ratio H2/NH3 Molar Ratio	660 890 1.4 16 9.2 1.2 1.4 0.84 2.8 0.8	660 890 1.3 19 7.9 1.3 1.3 0.95 3.12 0.9	660 890 1.3 18 7.9 1.3 1.3 0.98 3.12 1.1 1.24 0.0	660 890 1.3 19 7.9 1.3 1.3 0.97 3.12 0.7 2.29 0.0	660 885 1.3 17 10.1 1.3 0.96 2.44 0.8 1.39 1.46	660 880 1.3 15 10.1 1.3 1.3 0.93 2.48 1.1
NH <sub>3</sub> Injection Location			LÌ	L1	Ll	
Flue Gas Emissions  SO <sub>2</sub> (ppm)  NO <sub>x</sub> (ppm)  CO <sub>x</sub> (ppm)  CO <sub>2</sub> (%)  O <sub>2</sub> (%)	550 150 425 15.8 3.0	625 133 225 15.8 3.6	600 115 363 15.4 3.3	563 100 550 14.8 3.4	513 115 550 15.2 3.2	450 132 500 15.8 3.0
Results SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	74 0.87 0.17	68 1.04 0.16	70 1.02 0.14	71 1.00 0,13	79 0.68 0.11	82 0.58 0.12

#### APPENDIX P-4. BENCH COMBUSTOR RUN SUMMARY

Program Simulated	d Flue Gas R	ecirculation-N(	Control		
Run No.	1A.1	1A.2	1A.3	2.1	2.2
Operating Conditions					
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min) Excess Air (%) Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Velocity (m/s) Gas Residence Time (s) Ca/S Molar Ratio Run Length at Steady State (h		810 890 1.4 13 9.0 1.3 1.3 1.02 3.10 2	810 880 1.4 17 9.0 1.3 1.2 1.15 3.10	810 875 1.4 18 7.9 1.3 1.2 1.13 3.36 0.5	810 890 1.5 7 10.4 1.5 0.92 2.85 1.0
N <sub>2</sub> Flow Rate (m <sup>3</sup> /min) Recirculation Ratio (%)		0.16 11.2			0.35 23.4
Flue Gas Emissions					
SO <sub>2</sub> (ppm) NO <sub>X</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	550 160 200 15.4 3.5	600 117 400 15.4 2.1	450 170 475 16.0 3.3	419 155 200 14.8 3.2	525 70 300 14.5
Results					
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	73 0.89 0.19	67 1.17 0.16	78 0.72 0.19	77 0.81 0.22	72 1.09 0.07

## APPENDIX P-4 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program Sir	nulated Flue Gas	Recirculati	ion-NO <sub>x</sub> Control		
Run No.		3.1	3.2	4.1	4.2
Operating Condit	tions				
Pressure (kPaa) Bed Temperature Air Flow Rate (n Excess Air (%) Coal Feed Rate Expanded Bed He Superficial Velo Gas Residence T Ca/S Molar Ratio Run Length at S N2 Flow Rate (m Recirculation Ra	n3/min) (kg/hr) ight (m) ocity (m/s) ime (s) teady State (hr)	810 910 1.8 27 9.6 1.3 1.5 0.86 3.05 1.5	810 900 1.8 25 10.9 1.2 1.7 0.73 2.87 2.0 0.20 11.2	810 820 1.3 17 8.4 1.3 1.0 1.39 2.65 1.0	810 820 1.3 13 8.7 1.4 1.1 1.29 2.87 2.0 0.14 5.0
Flue Gas Emission	ons				
SO <sub>2</sub> (ppm) NO <sub>x</sub> (ppm) CO <sub>x</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)		575 160 175 16.0 5.0	575 130 550 15.4 4.0	750 260 425 14.8 3.0	938 150 700 14.2 2.0
<u>Results</u>					
SO <sub>2</sub> Retention (9 Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU	<b>%)</b>	67 1.01 0.20	67 1.07 0.17	66 1.18 0.29	54 1.72 0.13

#### APPENDIX P-5. BENCH COMBUSTOR RUN SUMMARY

	Combined NO <sub>x</sub> C	ontrol Method	ls		
Run No. <sup>(1)</sup>	1.1	1.2	1.3	1.4	1.5
Operating Conditions					
Pressure (kPaa)	500	500	500	500	500
Bed Temperature (°C) Air Flow Rate (m³/min)	890 1.5	900 1.5	890 1.5	900 1.5	900 1.5
Excess Air (%)	25 9.0	23 8.7	25 9.0	23	22 9.0
Coal Feed Rate (kg/hr) Expanded Bed Height (m) Superficial Volcation (m/s)	1.2 1.9	1.3	1.2 2.0	10.4 1.2	1.3 2.0
Superficial Velocity (m/s) Gas Residence Time (s) Co./S. Molon Potio	0.63 3.08	1.9 0.65 3.17	0.63	2.0 0.60	0.64 3.08
Ca/S Molar Ratio Run Length at Steady State (F		0.6	3.08 0.4	2.64 0.6	0.7 72
Primary Air (% Stoich.) Secondary/Primary Air (2) NH <sub>3</sub> /NO <sub>x</sub> Molar Ratio (3)	0	109 0 0.89	106 0	91 0 1.43	0.53
Flue Gas Emissions		0.03		1,40	
SO <sub>2</sub> (ppm) NO <sub>X</sub> (ppm) CO <sub>X</sub> (ppm) CO <sub>2</sub> (%) O <sub>2</sub> (%)	180 325 14.8 4.5	120 550 14.8 4.0	125 650 14.8 4.5	95 740 14.8 4.3	82 525 15.2 4.0
Results					
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>x</sub> /MBTU	0.21	 0.15	 0.15	 0.09	0.10

Grove Limestone; Champion Coal
 Supplementary air injected 33 cm above grid.
 NH<sub>3</sub> injected 290 cm above grid.

#### APPENDIX P-5 (CONT'D). BENCH COMBUSTOR RUN SUMMARY

Program	Combined NO <sub>x</sub> Control	Methods		
Run No. (1)	2.1	2.2	2.3	2.4
Operating Conditions				
Pressure (kPaa) Bed Temperature (°C) Air Flow Rate (m³/min Excess Air (%) Coal Feed Rate (kg/min Expanded Bed Height Superficial Velocity Gas Residence Time (some Ca/S Molar Ratio Run Length at Steady Primary Air (% Stoick Secondary/Primary Air	31 r) 9.7 (m) 1.1 (m/s) 2.0 s) 0.56 3.02 State (hr) 0.4 h.) 101	500 880 1.6 25 9.7 1.2 2.1 0.58 3.02 0.4 72 0.50	510 890 1.6 25 9.3 1.3 2.1 0.61 3.15 0.5 75	510 880 1.6 24 9.0 1.2 2.1 0.60 3.28 0.3 78 0.50
NH <sub>3</sub> /NO <sub>x</sub> Molar Ratio	(3)		1.61	
Flue Gas Emissions  SO <sub>2</sub> (ppm)  NO <sub>x</sub> (ppm)  CO <sub>x</sub> (ppm)  CO <sub>2</sub> (%)  O <sub>2</sub> (%)	575 153 400 14.2 5.5	650 120 375 15.2 4.5	675 105 425 14.8 5.0	675 113 440 14.8 4.3
Results				
SO <sub>2</sub> Retention (%) Lbs SO <sub>2</sub> /MBTU Lbs NO <sub>X</sub> /MBTU	73 0.87 0.17	68 1.03 0.14	65 1.15 0.13	63 1.22 0.15

<sup>(1)</sup> Grove Limestone; Champion Coal
(2) Supplementary air injected 33 cm above grid.
(3) NH<sub>3</sub> injected 290 cm above grid.

TECHNICAL REPORT DATA (Please read Instructions on the reverse before comp	leting)
1. REPORT NO. 2. E PA-600/7-80-013	3. RECIPIENT'S ACCESSION NO.
4. TITLE AND SUBTITLE Miniplant and Bench Studies of Pressurized Fluidized- bed Coal Combustion: Final Report	6. PERFORMING ORGANIZATION CODE EXXON/GRU.18GFGS.79
Goodwin, A. R. Garabrant, I. B. Radovsky, A. S. Lescar- ret, R. R. Bertrand, L. A. Ruth, V. J. Siminski (see blk 15)	8, PERFORMING ORGANIZATION REPORT NO.
Exxon Research and Engineering Co.	INE825
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919/541-2825. (From blk 7: M.S. Nutkis, M.D. Loughnane, H.R. Silakowski, M.W. Gregory, and A. Ichel.) EPA-600/7-78-069, -77-107, and -76-011 are related reports. 16. ABSTRACT The report gives further results of studies on the environmental aspects of the pressurized fluidized-bed coal combustion process, using the 218 kg coal/hr continuous combustion/sorbent regeneration Miniplant (0.63 MW equivalent), and a 13 kg coal/hr bench-scale system. Tests on the Miniplant combustor confirmed its ability to achieve over 90% SO2 removal with either limestone or dolomite sorbent. Studies of dynamic response indicated that the combustor responds much more quickly to changes in coal sulfur content than to changes in sorbent feed rate. High temperature/pressure particle control device testing on the Miniplant addressed: conventional and alternative-design cyclones, a ceramic fiber filter, and a granular bed filter. Three stages of cyclones may reduce dust loading sufficiently to protect a gas turbine. A conventional low-pressure electrostatic precipitator and fabric filter were also tested. Further tests on the Miniplant regenerator confirmed that regeneration can reduce fresh sorbent feed requirements by a factor of 3 to 4. Additional sampling was completed on the Miniplant combustor and regenerator for comprehensive analysis of emissions. NOx control studies in the bench combustor suggested that NOx emissions might be reduced by 20 to 50% through two-stage combustion or ammonia injection; flue gas recirculation had little effect.

17.	KEY WORDS AND D	OCUMENT ANALYSIS		
a. C	ESCRIPTORS	b.IDENTIFIERS/OPEN ENDED TERMS	c. COSATI F	eld/Group
Pollution	Calcium Carbonates	Pollution Control	13B	07B
Coal	Dolomite	Stationary Sources	21D	08G
Combustion	Sulfur Dioxide	Particulate	21B	
Fluidized Bed	Dust	Fabric Filters		11G
Processors	Cyclone Separators		13I,07A	
Flue Gases	Ceramics Fabrics		,	llB llE
18. DISTRIBUTION STATEM		19. SECURITY CLASS (This Report)	21. NO. OF PA	
		Unclassified	332	
Release to Publ	ic	20. SECURITY CLASS (This page) Unclassified	22. PRICE	