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TECHNICAL REPORT
December 1, 1991-February 29, 1992

Project Title:	Stabilization and/or Regeneration of Spent Sorbents From Coal Gasification
Principal Investigator:	Javad Abbasian, Institute of Gas Technology
Other Investigators:	Andy H. Hill, Institute of Gas Technology James R. Wangerow, Institute of Gas Technology
Project Monitor:	Daniel D. Banerjee, CRSC

ABSTRACT

The objective of this investigation is to determine the effects of SO_2 partial pressure and reaction temperature on the conversion of sulfide containing solid wastes from coal gasifiers to stable and environmentally acceptable calcium-sulfate, while preventing the release of sulfur dioxide through undesirable side reactions during the stabilization step. An additional objective of this program is to investigate the use of the Spent Sorbent Regeneration Process (SSRP) to regenerate spent limestone, from a fluidized-bed gasifier with in-bed sulfur capture, for recycling to the gasifier.

To achieve these objectives, selected samples of partially sulfided sorbents will be reacted with oxygen at a variety of operating conditions under sufficient SO_2 partial pressure to prevent release of sulfur from the solids during stabilization that reduces the overall sorbent utilization. Partially sulfided limestone will also be regenerated with water to produce calcium hydroxide and release sulfur as H_2S. The regenerated sorbent will be dewatered, dried and pelletized. The reactivity of the regenerated sorbent toward H_2S will also be determined.

During this quarter, the partially sulfided samples of limestone and dolomite in two particle sizes were sulfated in the Thermogravimetric Analyzer (TGA) reactor using a reactant gas containing 1% oxygen and 20% SO_2. The tests were conducted at temperatures of 1500°F and 1800°F. The results of these tests indicate the presence of SO_2 (in excess of equilibrium) prevents SO_2 release during oxidation of calcium sulfides. The unreacted calcium oxide also reacts with SO_2 and oxygen to produce calcium sulfate increasing sorbent utilization.

The partially sulfided samples were also sulfated in the 2-inch fluidized-bed reactor at the baseline sulfation condition. The results of these tests are currently being analyzed.

This project is funded by the U. S. Department of Energy (METC) and by the Illinois Department of Energy and Natural Resources as part of their cost-shared program.

EXECUTIVE SUMMARY

The sulfur compounds present in coal are converted to hydrogen sulfide when the coal is gasified. To comply with the New Source Performance Standards (NSPS), a high fraction of the sulfur must be removed from the gas stream. Calcium-based sorbents such as limestone and dolomite are among the prime candidates for in-bed capture of sulfur from the fuel gas.

High efficiency advanced electric power generation systems using Integrated Coal Gasification Combined Cycle (IGCC) or advanced/staged pressurized fluidized-bed combustion, require very low levels of H_2S in the fuel gases. More than 90% of the H_2S in the fuel gas can be removed in the gasifier by using calcium-based sorbents such as limestone or dolomite. An external hot gas cleanup system using regenerable mixed metal oxides such as zinc ferrite is then used to further reduce the H_2S content of the fuel gas to acceptable levels for IGCC application.

The removal of sulfur takes place through the reaction of hydrogen sulfide with calcium oxide, calcium carbonate, or calcium hydroxide and produces calcium sulfide. This compound is not stable, and therefore, is not suitable for direct disposal. However, calcium sulfide can be either reacted with oxygen (or air) to produce calcium sulfate, which is a stable and environmentally acceptable compound for disposal, or reacted with water to regenerate the sorbent and release sulfur as hydrogen sulfide.

The extent of the conversion of calcium sulfide to stable calcium sulfate has been shown to depend on the sorbent type (i.e., limestone or dolomite), the extent of sulfidation reaction (i.e., Ca/S ratio in the gasifier), sorbent particle size, and magnesium content of the sorbent. The reaction temperature has been shown to have a significant effect on the overall sorbent utilization. Stabilization is generally accompanied by release of sulfur in the form of SO_2 through a side reaction. The fraction of sulfur released as SO_2 increases with increasing temperatures. The release of sulfur during stabilization should be prevented in order to maintain the overall sorbent utilization. This is essential to the development of an economical process scheme for successful stabilization of sulfide containing solid waste products from gasification or staged combustion of high sulfur Illinois coal.

The spent sorbents may also be regenerated in the Spent Sorbent Recovery Process (SSRP). In the proposed process, the ash discharge from a fluidized-bed gasifier, consisting essentially of spent limestone containing CaS and coal ash, is sent to a reactor containing water at temperatures of 150-300°F and pressures of 25 to 350 psia. Process derived CO_2 is bubbled through the ash-water slurry. Under these conditions, the water and CO_2 will react with the CaS to liberate H_2S and produce $Ca(OH)_2$ and/or $CaCO_3$, depending upon the amount of CO_2 used. The subsequent stages of dewatering, drying, pelletizing and recycling of the regenerated materials need to be investigated to determine the viability of the SSRP.

The objective of this investigation is to determine the effects of SO_2 partial pressure, reaction temperature and sorbent type for conversion of sulfide containing solid wastes from coal gasification (that utilize calcium-based sorbents for in-bed sulfur capture) to stable and environmentally acceptable calcium sulfate while preventing the release of sulfur dioxide during the stabilization step. An additional objective of the program is to investigate the viability of the spent sorbent regeneration using the SSRP.

Two calcium-based sorbents were tested in this study which included one limestone and one dolomite. These sorbents are the same as those used in previous projects sponsored by CRSC (Subgrant 89-4 and 90-8). The sorbents were crushed and screened into narrow particle size ranges. Two cuts from each sorbent were selected for this investigation which include fine particles (-60+80 mesh) with an average particle diameter of 0.02 cm and coarse particles (-20+30 mesh) with an average particle diameter of 0.07 cm.

Sulfation tests were conducted with the partially sulfided sorbents in the Thermogravimetric Analyzer (TGA) reactor at 1500°F and 1800°F using both fine and coarse sorbent particles. The reactant gas in these tests contained 1% oxygen, 20% SO_2, and the balance was nitrogen. The reacted sorbents were analyzed to determine the extent of CaS and CaO conversion to $CaSO_4$. Sulfation tests were also conducted in the 2-inch quartz fluidized-bed reactor with partially sulfided sorbents at temperatures of 1500°, 1650°, and 1850°F using a reactant gas containing 5% oxygen, 20% SO_2, and 75% nitrogen. The reacted sorbents are currently being analyzed.

OBJECTIVES

The goal of this program is to develop processes for stabilization and/or regeneration of spent calcium-based sorbent that have been used to capture sulfur in fluidized-bed gasification of high sulfur coals.

The overall objective of this program is to determine the effect of sorbent type, SO_2 partial pressure, and reactor temperature, on the conversion of calcium sulfide to calcium sulfate while preventing the release of sulfur dioxide during the stabilization process. An additional objective of this program is to investigate regeneration of spent Ca-based sorbent using Spent Sorbent Regeneration Process (SSRP) in which spent limestone containing calcium sulfide is reacted with water and carbon dioxide to release sulfur as H_2S and product calcium hydroxide and/or calcium carbonate that can be recycled back to the gasifier.

The proposed program goal is specifically directed towards waste management, Research Priority Section 5.4A: Studies related to disposal and utilization of waste materials generated in gasifiers, or fluidized-bed combustors, or flue gas scrubbers, including in-duct injection. The specific objectives of this program are to:

1. Prepare partially sulfided samples of a limestone and a dolomite (that have been used in previous CRSC funded projects)
2. Obtain experimental data on oxidation of the sulfided sorbents in a quartz batch fluidized-bed reactor. The test parameters will include reaction temperature, SO_2 concentration, oxygen concentration and solid residence time.
3. Analyze oxidized sorbents in terms of their physical and chemical properties to determine the reaction path during oxidation.
4. Conduct sulfur leaching tests with the stabilized sorbent to determine their suitability as the end product.
5. Determine the effects of regeneration on recycling limestone.
6. Analyze and characterize both original limestone and regenerated pellets.
7. Evaluate the reactivity of regenerated sorbent toward H_2S.

INTRODUCTION AND BACKGROUND

The market-share of Illinois high-sulfur coal can be expanded to industrial users as well as power plants if the coal can be used in an environmentally acceptable manner with the minimum gas cleanup costs. For example, an integrated coal gasification/combined-cycle power plant offers the potential for both lower cost power and lower emissions than coal-fired plants with flue gas desulfurization for power generation.

The sulfur compounds present in coal are converted to hydrogen sulfide when the coal is gasified. To comply with the New Source Performance Standards (NSPS), a high fraction of the sulfur must be removed from the gas stream. Calcium-based sorbents such as limestone and dolomite are among the prime candidates to capture the sulfur compounds from the fuel gas.

The solid wastes produced from the reactions of calcium-based sorbents with hydrogen sulfide in the fuel gas, contain calcium sulfide which is unstable and has the tendency to decompose when it contacts moisture in the air, releasing hydrogen sulfide to the atmosphere. To dispose the solid waste products in an environmentally acceptable manner, calcium sulfide can be converted to stable calcium sulfate through reaction with oxygen (air) --

$$CaS + 2O_2 = CaSO_4 \qquad (A)$$

However, the calcium sulfate produced by reaction (A), at temperatures of interest to the overall process (1500°-1800°F), can also react with the calcium sulfide in an apparent solid-solid reaction to release SO_2 -

$$CaS + 3CaSO_4 = 4CaO + 4SO2 \qquad (B)$$

Reaction (A) is a highly exothermic reaction (ΔH_{rxn} > 350 Kcal/g mole) resulting in a significant increase in the particle temperature. Reaction (B) becomes more favorable as the particle temperature increases, resulting in release of SO_2, and reducing overall sorbent utilization. The highly exothermic nature of reaction (A) and its effect on the reaction temperature significantly affects the final distribution of the product.

The spent sorbent containing calcium sulfide can also be regenerated to release sulfur as H_2S and produce calcium hydroxide and/or calcium carbonate that can be recycled back to the gasifier.

$$CaS + 2H_2O = Ca(OH)_2 + H_2S \qquad (C)$$

$$CaS + CO_2 + H_2O = CaCO_3 + H_2S \qquad (D)$$

Reactions (C) and (D) can be carried out at temperatures of 250-300°F at the gasifier pressures of 25 to 350 psia.

The results of investigations on the stabilization of calcium sulfide in previous CRSC funded projects(1-5) indicate that limestone can only be partially sulfated while dolomite can be essentially completely sulfated at 1500°F. The extent of sulfation depends not only on the sorbent type but also on the extent of sulfidation in the gasifier. The results of previous studies also indicate that the fraction of sulfur in calcium sulfide converted to SO_2 significantly increases with increasing temperature.(6-14)

To prevent SO_2 evolution during stabilization, Reaction (B) must be suppressed. One approach for suppression of SO_2 release during stabilization is to carry out the stabilization at a lower temperature (T<1500°F) while effectively controlling the reactor temperature by using a low concentration of O_2 in the reactant gas. Lower reaction temperature coupled with low oxygen concentration will result in a slow rate of sulfation reaction requiring large reactors to carry out the stabilization step.

A more attractive approach is to carry out the stabilization step in the presence of a significant concentration of SO_2 in the reactant gas. In order to carry out the stabilization of calcium sulfide according to Reaction (A) and to prevent Reaction (B) from occurring, the partial pressure SO_2 in the reactant gas must exceed the equilibrium value at the selected reaction temperature. The overall reaction for release of SO_2 can be obtained by a combination of Reactions (A) and (B) [i.e. 3(A) + (B) = 4(E)] -

$$CaS + 3/2O_2 = CaO + SO_2 \qquad (E)$$

The familiar reaction (F) for sulfation of calcium oxide --

$$CaO + SO_2 + 1/2O_2 = CaSO_4 \qquad (F)$$

is also a dependent reaction with regard to independent reactions (A) and (B)

$$(A) - (B) = 4(F)$$

Based on the Gibbs free energies of these reactions, the thermodynamic equilibrium of reaction (B) dictates the reaction path, as long as all four substances (i.e., CaO, CaS, $CaSO_4$, SO_2) are present. By maintaining the desired concentration of SO_2 during oxidation, partially sulfided calcium-based sorbents can be stabilized through Reaction (A) while the release of SO_2 through Reaction (B) is prevented.

A number of researchers have investigated different methods of CaS regeneration using steam(15-23) with regeneration efficiencies up to 96%. Regeneration of spent sorbents in the SSRP process has also been investigated in a previous CRSC funded project (under Subgrant 90-8). The results of this work indicate that up to 99% of the calcium sulfide can be regenerated in a reactor containing water at temperatures of 150-300°F and pressure of 25 to 350 psia. The regenerated sorbent can be dewatered, dried, pelletized and recycled back to the gasifier. To prevent buildup of coal ash, a slipstream of the treated slurry is removed from the system and dried to be used as fertilizer to adjust soil Ph or fully carbonated to be used aggregate for concrete.

This investigation is targeted toward determination of the sulfur capturing capabilities of both limestone and dolomite during stabilization of partially sulfided sorbents in the presence of excess SO_2. The investigation on regeneration of spent sorbent will expand the current process to include the subsequent stages of dewatering, drying, pelletizing, and recycling of the regenerated material. The result of this work will provide the necessary information in the development of economically attractive processes for the stabilization and/or regeneration of calcium sulfide containing spent sorbents from gasification of Illinois high sulfur coal.

EXPERIMENTAL PROCEDURES

The experiments performed during this quarter were conducted in an ambient pressure quartz Thermogravimetric Analyzer (TGA) reactor as well as a 2-inch-diameter quartz fluidized-bed reactor, wherein the partially sulfided samples of both limestone and dolomite (prepared last quarter) were sulfated using a reactant gas containing oxygen and sulfur dioxide.

TGA Sulfation Tests

Eight sulfation tests were conducted in the ambient-pressure quartz TGA reactor. Partially sulfided samples of limestone and dolomite from both cuts were used in these tests. The tests were conducted at 1500°F and 1800°F using a reactant gas containing 1% oxygen and 20% SO_2. The schematic diagram of the TGA unit used in these tests is shown in Figure 1.

During a test, a simultaneous recording is made of the sample weight, the rate of weight change, and the temperature of the furnace in close proximity of the sorbent being tested. The sample is contained in a platinum basket while a metered gas flow is introduced at the bottom of the furnace chamber. The composition in the reactant gas is predetermined by mixing (if necessary) several streams of gases at a specified ratio. The platinum basket is suspended from an arm of a recording balance by a platinum wire chain. Any movement of the balance arm, because of a change in sample weight, is sensed by a linear differential transformer on the left arm of the balance. A restoring force that is proportional to the change in weight is supplied to the right arm with an electromagnet. The recording balance control unit senses the force required to maintain a null and converts this into a signal proportional to the weight of the sample. The sample weight signal and the temperature signals are recorded by a computer as a function of time.

The procedure for these tests included heating the sorbent in nitrogen atmosphere to the predetermined temperature. At this point the gas mixture containing 1% oxygen and 20% SO_2 is allowed to flow past the sorbent while the change in the sample weight is continuously monitored. The sample is exposed to the reactant gas containing oxygen until the sulfided sorbent is converted to its peak value.

The calcium sulfide can react with oxygen to produce calcium sulfate --

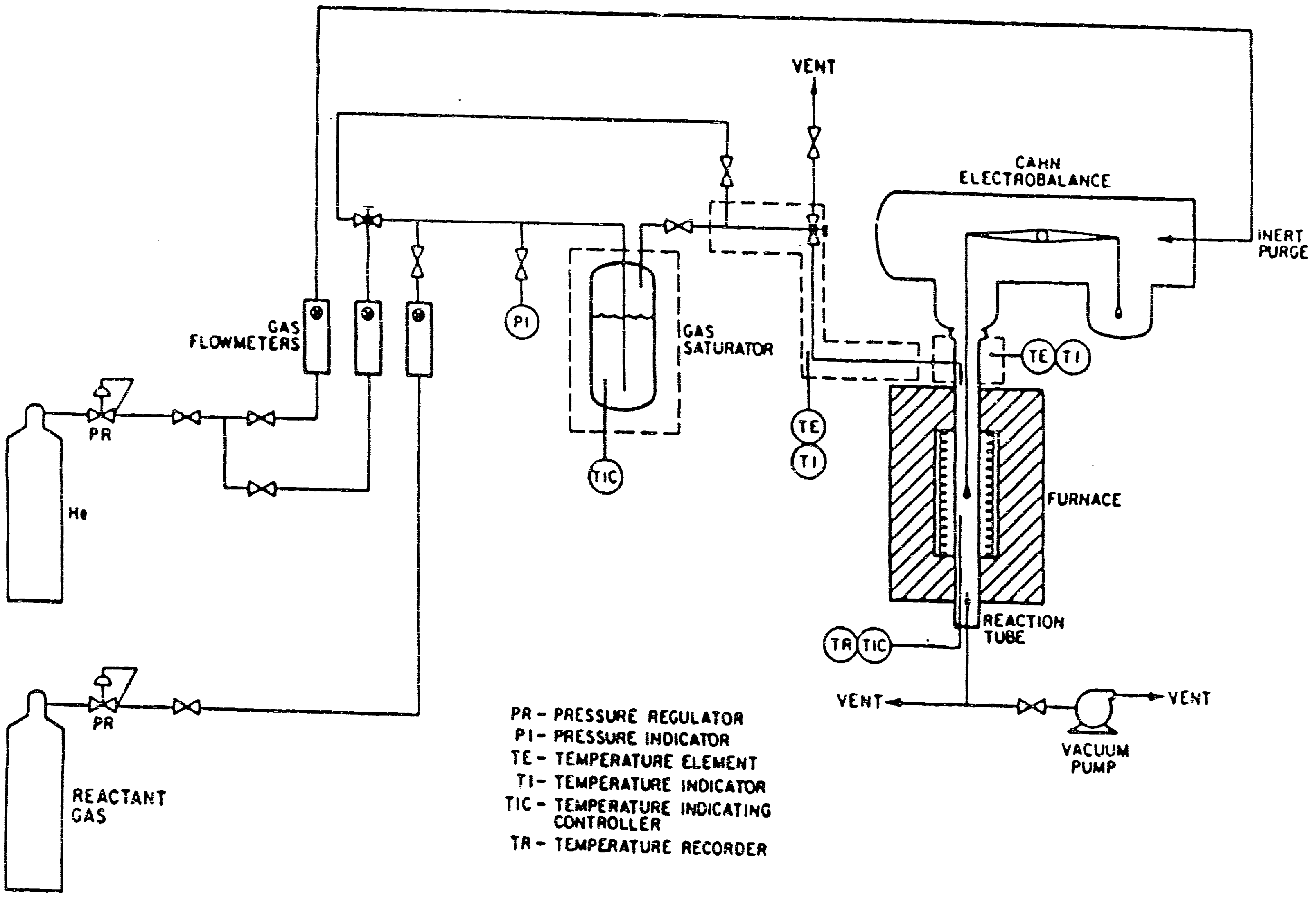

Figure 1. PROCESS FLOW DIAGRAM OF THE AMBIENT-PRESSURE THERMOGRAVIMETRIC REACTOR (TGR) UNIT

$$CaS + 2O_2 = CaSO_4 \qquad \text{(A)}$$

However, calcium sulfate produced by reaction (A) at temperatures of interest (1500°-1800°F) can also react with calcium sulfide in an apparent solid-solid reaction to release SO_2 --

$$CaS + 3CaSO_4 = 4CaO + 4SO_2 \qquad \text{(B)}$$

In the TGA experiments, the SO_2 partial pressure (0.2 atm) exceeded the equilibrium partial pressure for reaction (B) at all temperatures tested, resulting in no sulfur loss. Additionally, because of the high partial pressure of SO_2, reaction (B) proceeded toward the left. The overall sulfation reaction involving SO_2 and CaO --

$$SO_2 + CaO + 1/2O_2 = CaSO_4 \qquad \text{(E)}$$

can be obtained by combining reactions (A) and (B).

$$\text{(A)} - \text{(B)} = 4\text{(E)}$$

The reacted samples are analyzed to determine the sample weight gain in each reaction (A) and (E). The weight gains are converted to conversion of CaS and CaO in reactions (A) and (E), respectively.

Baseline Sulfation Tests

Six sulfation tests were conducted in the 2-inch quartz fluidized-bed reactor. Partially sulfided fine limestone and dolomite (dp = 0.02 cm) were sulfated at temperatures of 1500°, 1650°, and 1800°F using a reactant gas containing 5% oxygen, 20% SO_2, and 75% nitrogen. The schematic diagram of the unit is shown in Figure 2. The unit essentially consists of a quartz fluidized-bed reactor which is externally heated by two electric furnaces, equipment for feeding and measuring the flow rates of the gases, measuring and controlling the bed temperature, and monitoring the fluidized-bed pressure. The fluidized-bed distributor is made of a quartz fritted disk.

In a typical test, about 50 grams of the partially sulfided sorbents were loaded into the fluidized-bed reactor. The bed of solids is heated by the external furnace, while a stream of nitrogen flows through the reactor until the reactor reaches the desired temperature. The gas flow rate is then increased to fluidize the bed. At this point, the gas is

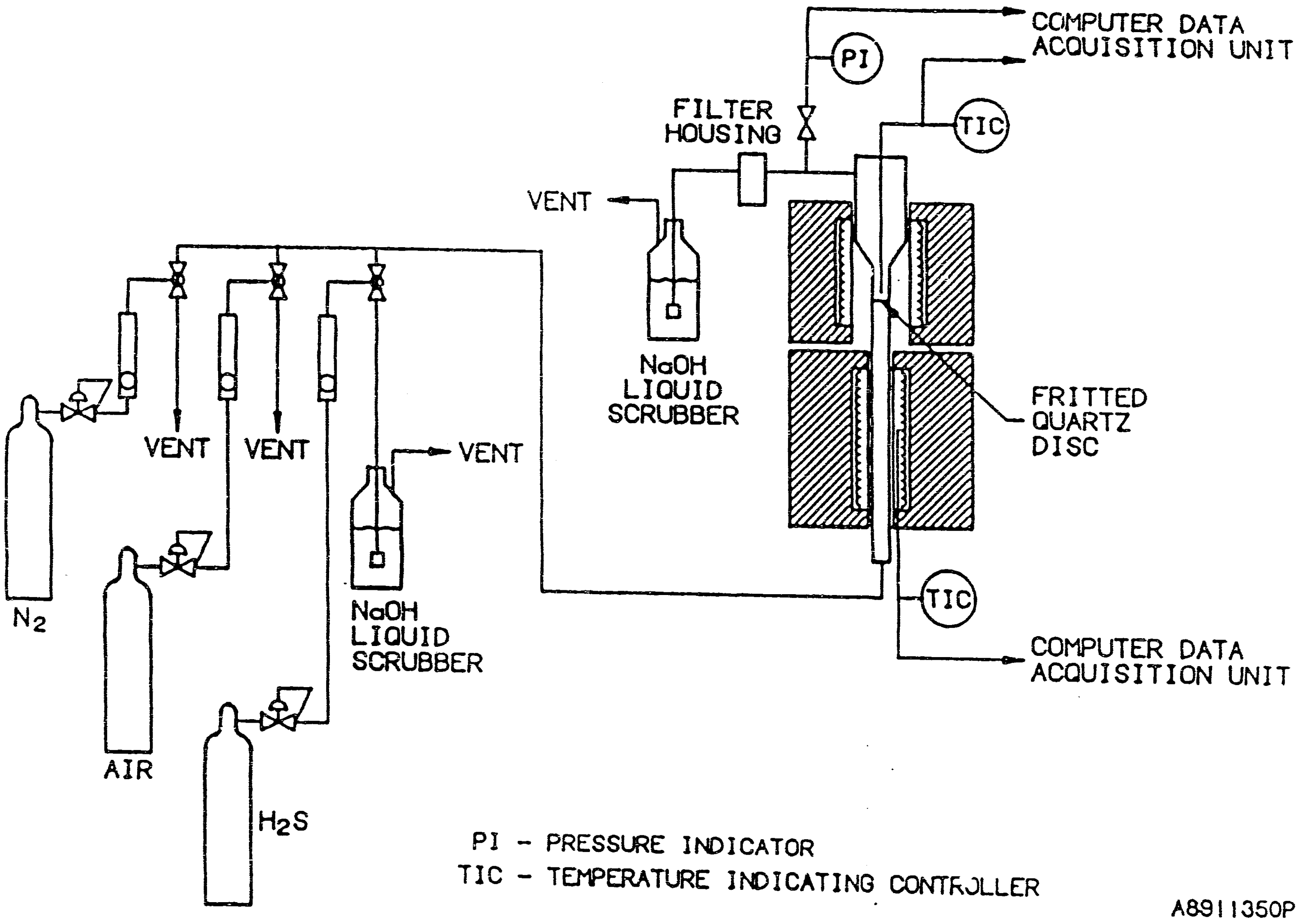

Figure 2. SCHEMATIC FLOW DIAGRAM OF THE QUARTZ FLUIDIZED-BED UNIT

switched to a reactant gas containing the desired concentration of oxygen and sulfur dioxide. The desired composition of the reactant gas is achieved by mixing streams of air, sulfur dioxide, and nitrogen at predetermined ratios. The reaction continues until the desired solids residence time is reached. During the test, the temperature is controlled by the temperature controller. The reactor temperature and pressure are monitored by a computer data acquisition system. The reactor off-gas passes through a filter for collection of elutriated fines and then through a liquid scrubber before being vented. After the test, the reactor solids are analyzed to determine the extent of sulfation of calcium sulfide and calcium oxide during the test.

RESULTS AND DISCUSSION

TGA Sulfation Tests

Samples of both cuts [-60+80 mesh ($\bar{d}p$ = 0.02 cm) and -20+30 mesh ($\bar{d}p$ = 0.07 cm] from both sorbents were sulfated in the ambient pressure quartz TGA reactor at temperatures of 1500° and 1800°F using a reactant gas containing 1% oxygen and 20% sulfur dioxide. The reacted sorbents were analyzed to determine the extent of CaS and CaO conversion to $CaSO_4$. The final conversions in all eight tests are shown in Table 1. The results indicate that in the case of limestone, less than 30% of the sulfide can be converted to sulfate. The conversion of CaS in coarse sulfided limestone sample is essentially zero. The conversion of calcium oxide in the sulfided limestone was in the range of 57 to 81%. Higher conversion of calcium oxide in the coarse sulfided limestone sample, coupled by lower conversion of calcium sulfide (compared to the fine limestone sample), indicates that calcium sulfate formed during sulfation of calcium sulfide may have plugged the pores, preventing calcium oxide reaction with SO_2. In the case of dolomite, conversion of sulfide to sulfate was in the range of 58 to 85%, while the amount of sulfate corresponded to more than 100% CaO conversion. This is probably due to formation of magnesium sulfate during sulfation. Figures 3 through 6 show conversions of calcium sulfide and calcium oxide to calcium sulfide.

Baseline Sulfation Tests

The reacted solids from these tests are currently being analyzed to determine the extent of calcium sulfide as well as calcium oxide conversion to calcium sulfate.

Table 1. EXTENT OF CaS AND CaO CONVERSION TO $CaSO_4$

Sorbent	Particle Diameter, cm	Sulfation Temperature, °F	Extent of Sulfidation, %	Extent of CaS Sulfation, %	Extent of CaO Sulfation, %
Limestone	0.02	1500	40.49	22.92	57.82
Limestone	0.07	1500	47.98	0	66.83
Dolomite	0.02	1500	44.46	58.721	131.39*
Dolomite	0.07	1500	47.12	62.97	111.98*
Limestone	0.02	1800	40.49	16.63	62.89
Limestone	0.07	1800	47.98	0	81.19
Dolomite	0.02	1800	44.46	85.64	108.6*
Dolomite	0.07	1800	47.12	77.36	102.86*

* The excess sulfate is due to formation of magnesium sulfate.

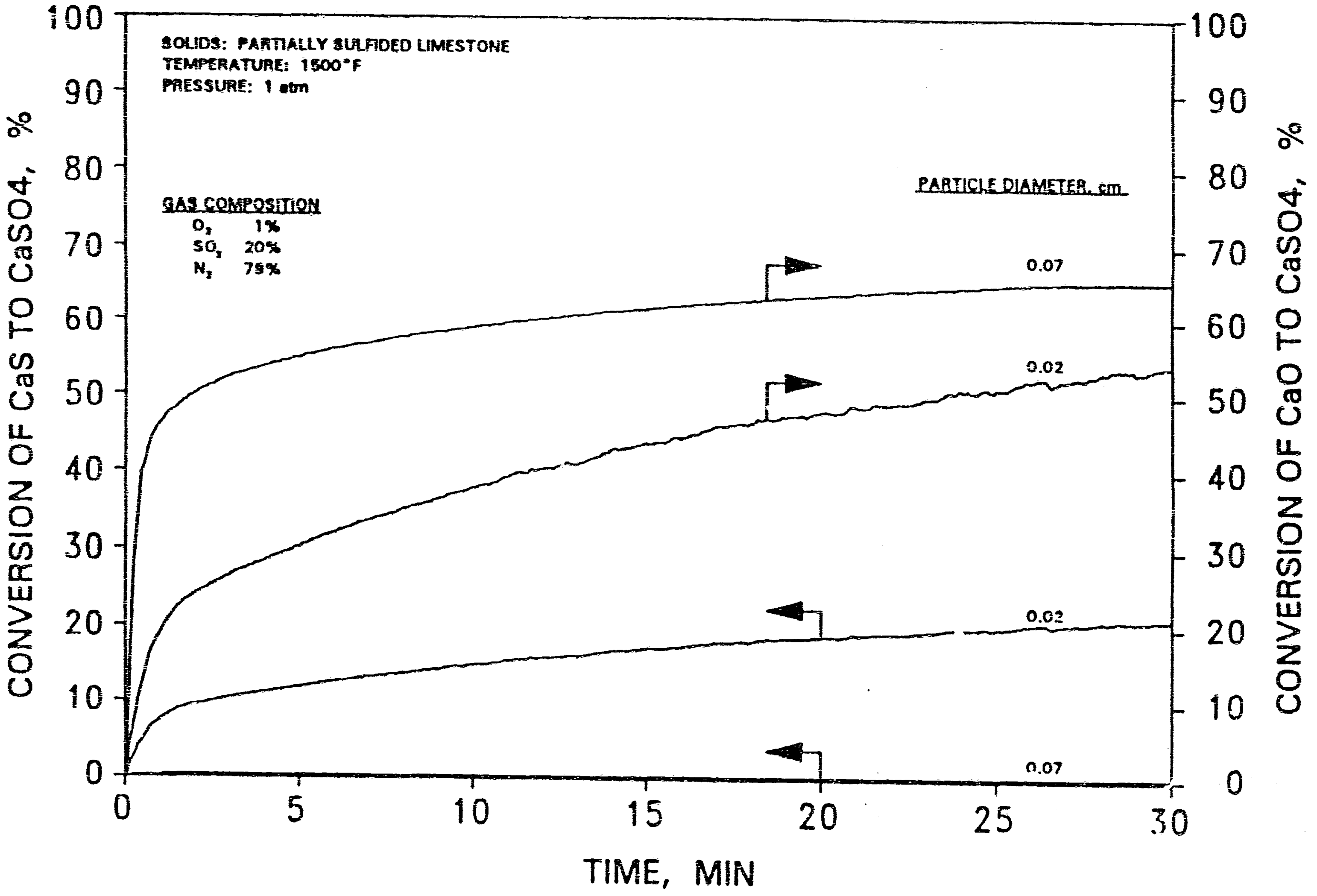

Figure 3. CONVERSION OF CaO AND CaS IN PARTIALLY SULFIDED LIMESTONE TO $CaSO_4$ (1500°F)

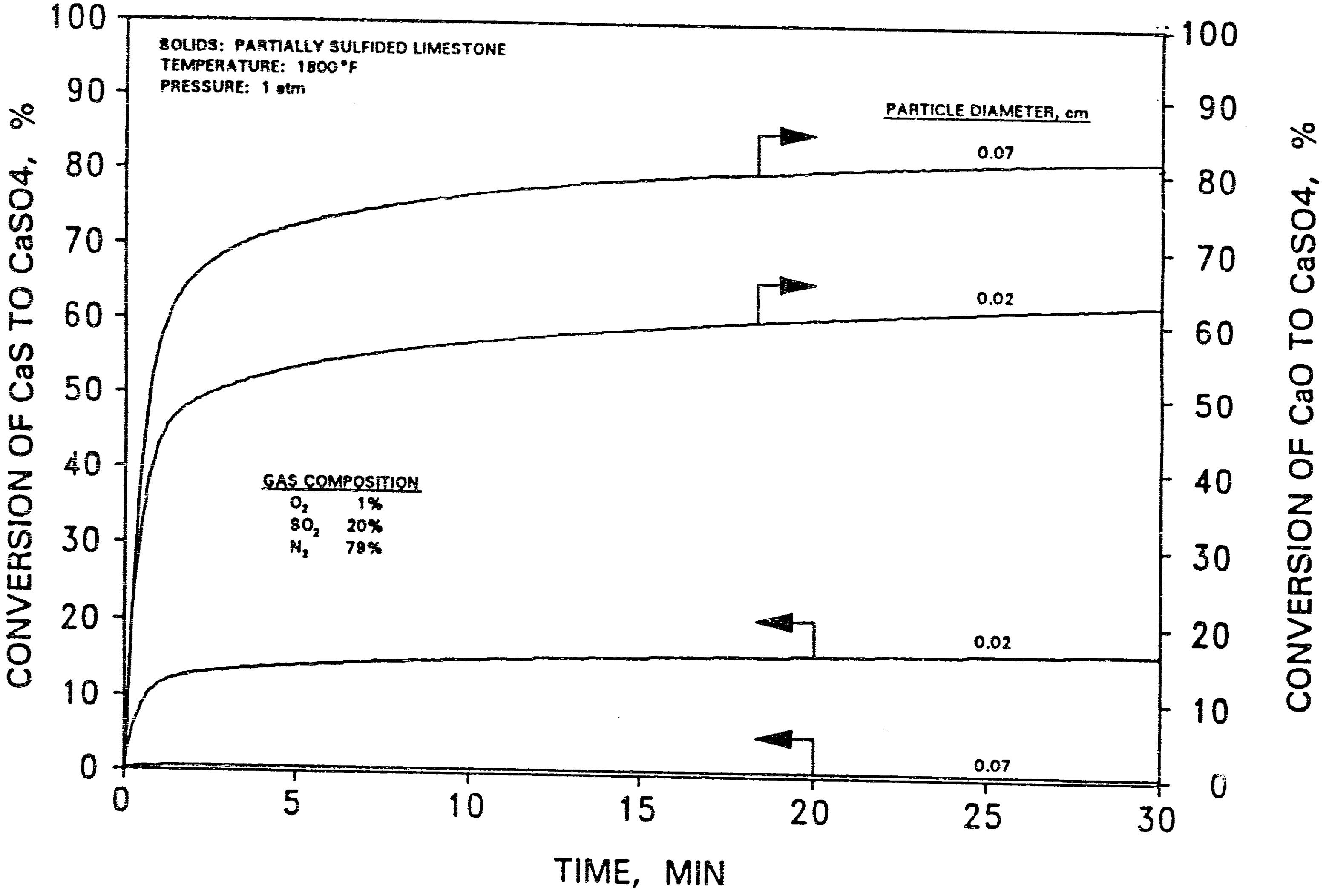

Figure 4. CONVERSION OF CaO and CaS IN PARTIALLY SULFIDED LIMESTONE TO $CaSO_4$ (1800°F)

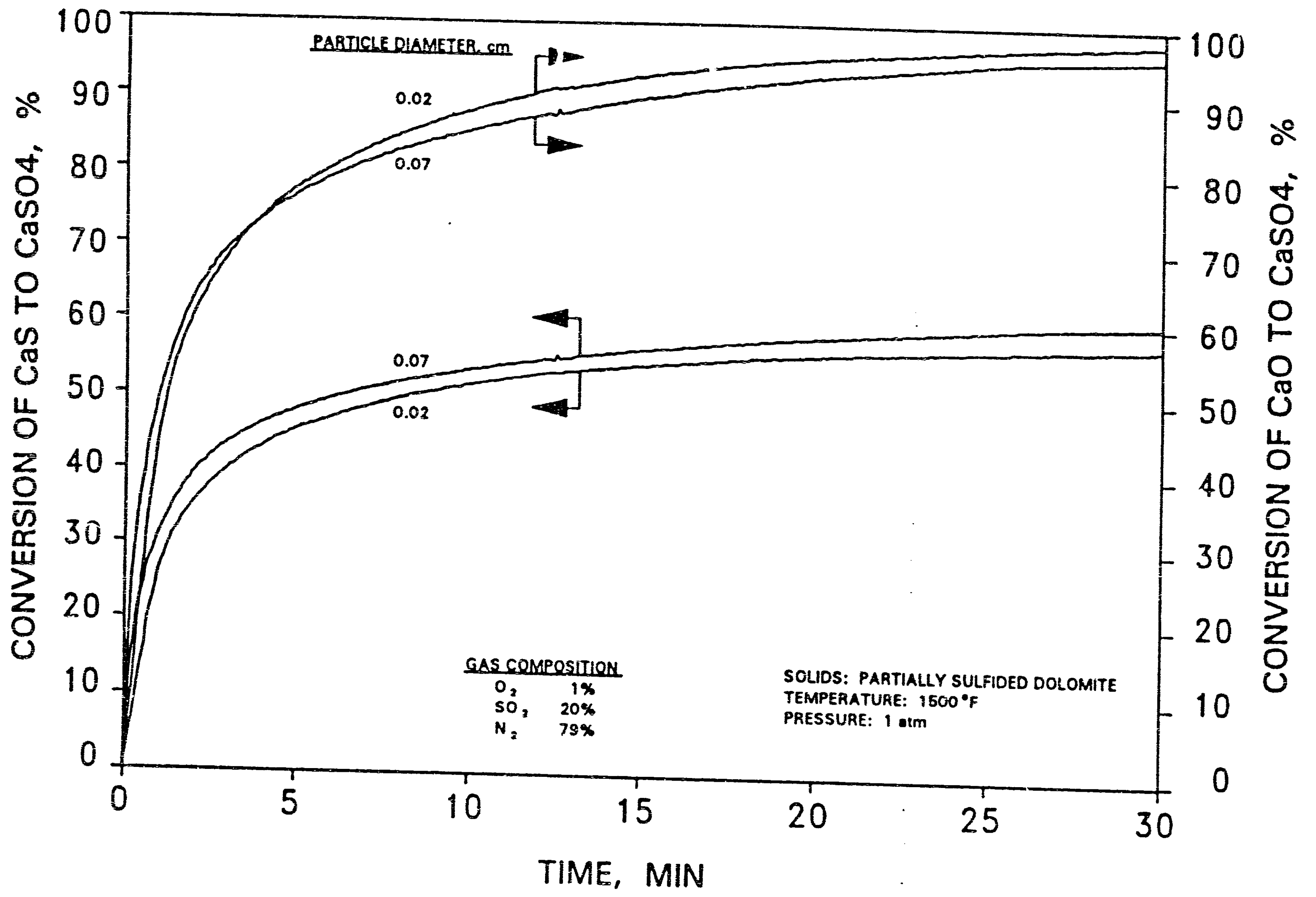

Figure 5. CONVERSION OF CaO and CaS IN PARTIALLY SULFIDED DOLOMITE TO $CaSO_4$ (1500°F)

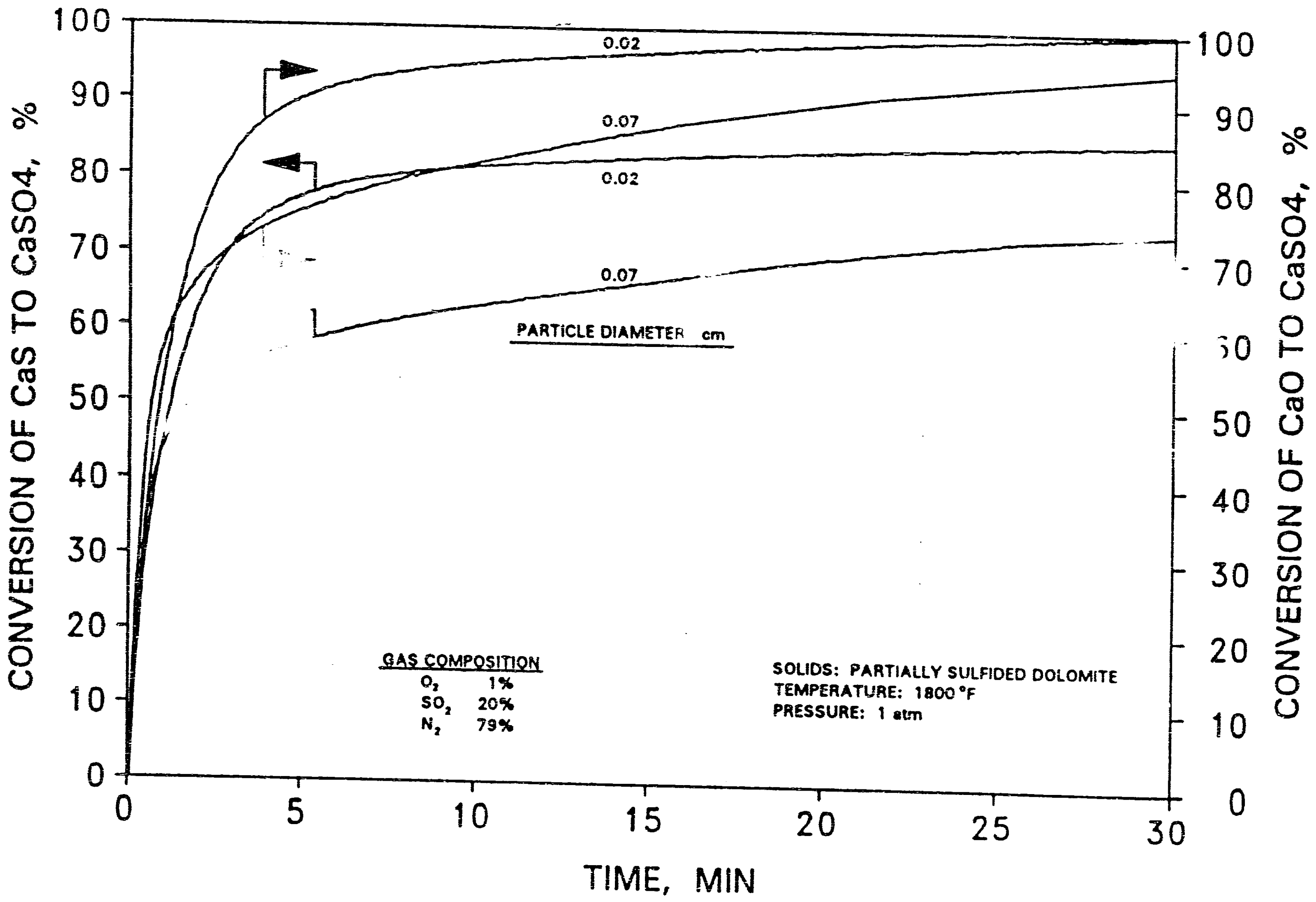

Figure 6. CONVERSION OF CaO and CaS IN PARTIALLY SULFIDED DOLOMITE TO $CaSO_4$ (1800°F)

CONCLUSIONS AND RECOMMENDATIONS

The following conclusions can be made from the work performed during this quarter:

- Reaction of calcium sulfide and oxygen can be carried out in the temperature range of 1500° to 1800°F without any sulfur loss.
- The unreacted calcium oxide in the partially sulfided limestone and dolomite reacts with SO_2 increasing sorbent utilization.
- The magnesium oxide also reacts with SO_2 to produce magnesium sulfate.

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PROJECT MANAGEMENT REPORT
December 1, 1991 through February 29, 1992

Project Title:	Stabilization and/or Regeneration of Spent Sorbents From Coal Gasification
Principal Investigator:	Javad Abbasian, Institute of Gas Technology
Other Investigators:	Andy H. Hill, Institute of Gas Technology James R. Wangerow, Institute of Gas Technology
Project Monitor:	Daniel D. Banerjee, CRSC

COMMENTS

Regeneration of spent sorbent was not initiated during this quarter. These tests will be conducted in the next quarter and the project will be completed on schedule.

This project is funded by the U. S. Department of Energy (METC) and by the Illinois Department of Energy and Natural Resources as part of their cost-shared program.

STABILIZATION AND/OR REGENERATION OF SPENT SORBENTS FROM COAL GASIFICATION

EXPENDITURES - EXHIBIT B

Projected and Estimated Actual Expenditures by Quarter

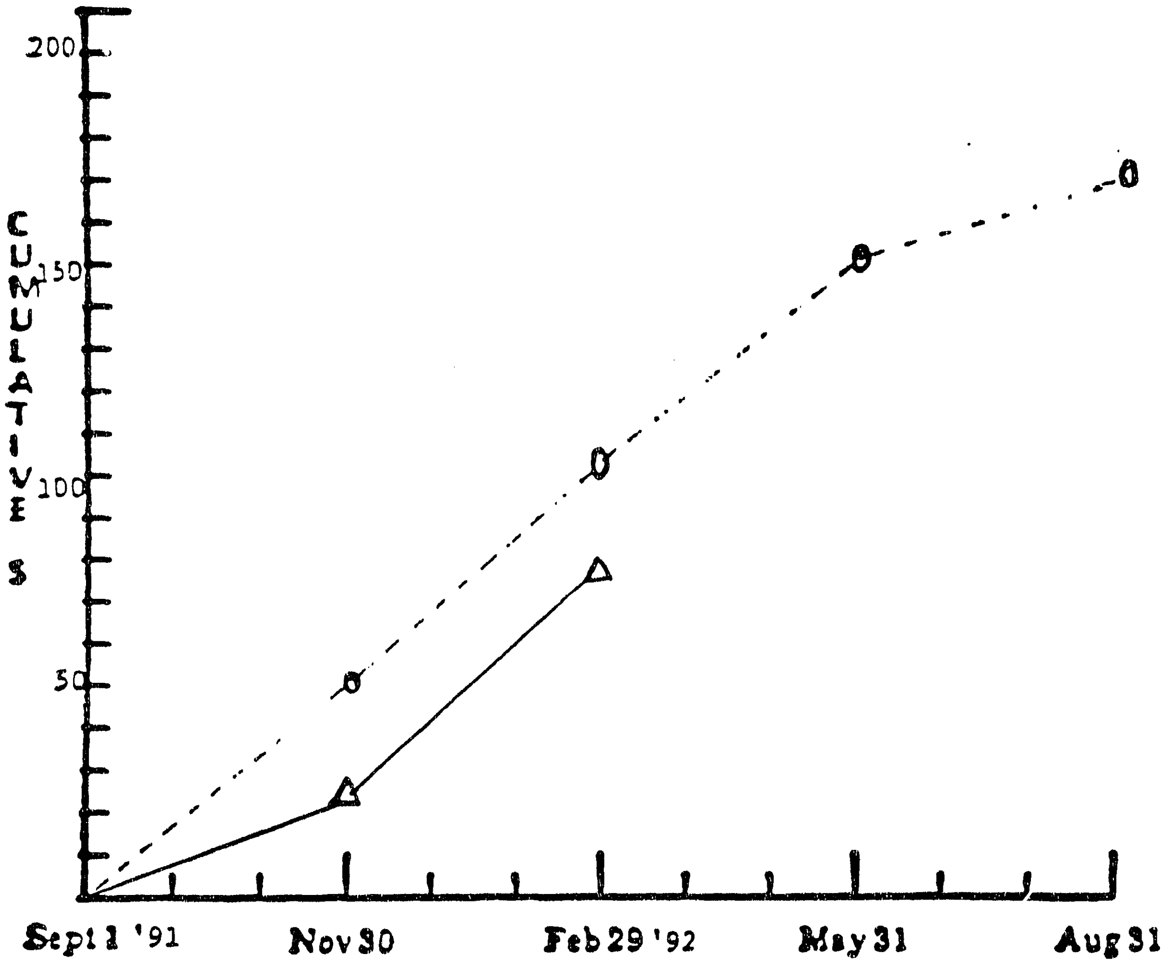
Quarter*	Types of Cost	Direct Labor	Materials and Supplies	Travel	Major Equipment	Other Direct Costs	Indirect Costs	Total
Sept. 1, 1991 to Nov. 30, 1991	Projected	16,500	2,400	1,890			30,720	51,510
	Estimated Actual	6,300	2,000	1,800			12,183	22,283
Sept. 1, 1991 to Feb. 29, 1992	Projected	33,000	6,000	1,890			61,295	102,185
	Estimated Actual	24,372	2,346	1,715			44,900	73,333
Sept. 1, 1991 to May 31, 1992	Projected	49,300	8,800	1,890			91,340	151,330
	Estimated Actual							
Sept. 1, 1991 to Aug. 31, 1992	Projected	54,831	9,750	3,512			101,875	169,968
	Estimated Actual							

* Cumulative by quarter

COSTS BY QUARTER - EXHIBIT C

(In Thousands)

Stabilization and/or Regeneration of Spent Sorbents
from Coal Gasification



Months and Quarters

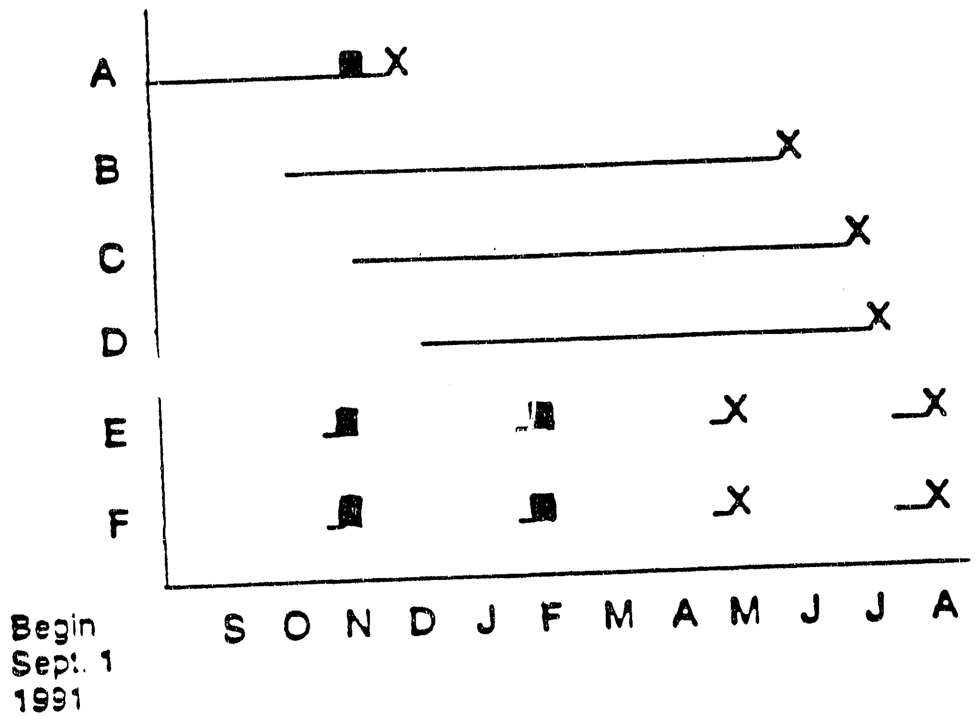
0 = Projected Expenditures 102,185

Δ = Actual Expenditures 73,333

Total CRSC Award $ 169,968

STABILIZATION AND/OR REGENERATION OF SPENT SORBENTS FROM COAL GASIFICATION

SCHEDULE OF PROJECT MILESTONES



A. Preparation of Sulfided Sorbents

B. Stabilization of Partially Sulfided Sorbents

C. Physical and Chemical Analyses of the Stabilized Sorbents

D. Regeneration of Spent Sorbent

E. Preparation of Technical Reports

F. Preparation of Project Management Reports

END

DATE FILMED

9/10/92