**DIRECT SULFUR RECOVERY DURING SORBENT
REGENERATION**

FINAL REPORT

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EXECUTIVE SUMMARY

The objective of this research project was to improve the direct elemental sulfur yields that occur during the regeneration of SO_2 -saturated MgO -vermiculite sorbents (MagSorbents) by examining three approaches or strategies. The three approaches were regeneration-gas recycle, high-pressure regeneration, and catalytic reduction of the SO_2 gas using a new catalyst developed by Research Triangle Institute (RTI).

Prior to the project, Sorbent Technologies Corporation (Sorbtech) had developed a sorbent-regeneration process that yielded directly a pure elemental sulfur product. In the process, typically about 25 to 35 percent of the liberated SO_2 was converted directly to elemental sulfur. The goal of this project was to achieve a conversion rate of over 90 percent.

Good success was attained in the project. About 90 percent or more conversion was achieved with two of the approaches that were examined, regeneration-gas recycle and use of the RTI catalyst. Of these approaches, regeneration-gas recycle gave the best results (essentially 100 percent conversion in some cases).

In the regeneration-gas recycle approach, saturated sorbent is simply heated to about 750°C in a reducing gas (methane) atmosphere. During heating, a gas containing elemental sulfur, water vapor, H_2S , SO_2 , and CO_2 is evolved. The elemental sulfur and water vapor in the gas stream are condensed and removed, and the remaining gas is recycled back through the sorbent bed. After several recycles, the SO_2 and H_2S completely disappear from the gas stream, and the stream contains only elemental sulfur, water vapor and CO_2 .

In addition to the favorable results described above, several interesting observations were made during the project. For example, it was discovered that up to 77 percent elemental sulfur yield could be obtained with a single pass of reducing gas through hot saturated-sorbent beds at atmospheric pressure by improving the design of the regenerator and improving the methods of sulfur capture and removal. Also discovered was that a bed of fresh MgO -vermiculite sorbent, or of MgO alone, can act as a catalyst in converting other SO_2 -rich gas streams to elemental sulfur with yields of up to 74 percent. Conversions in all cases were also carried out at atmospheric pressure. This latter discovery is believed important because it could lead to a low-cost alternative to a costly Claus plant, which is probably the best commercial technology available today for treating sulfur-rich gas streams to recover elemental sulfur.

The project results provide a sound foundation for further development, demonstration, and commercialization of the new direct sulfur recovery technology. Required now is a demonstration that high elemental sulfur yields can be maintained in equipment scaled-up in size and operated on a continuous basis (Sorbtech processed only small batch quantities of sorbent in the project). Also needed are further improvements in sulfur capture methods and the development of methods to treat small tail-gas streams that may be produced. Goals of any future programs should be 100 percent SO_2 conversion and zero wastes. These are goals that are believed attainable. The new technology, of course, is directly applicable to all Ohio coals. Sorbtech plans to carry the new technology forward. It is a key element in Sorbtech's MagSorbent Development and Commercialization Plan.

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INTRODUCTION

Existing flue-gas desulfurization (FGD) systems produce millions of tons of wastes annually in scrubbing the SO_2 from coal-fired boilers. The disposal of these wastes has become a problem. In some cases, utilities have been forced to switch to lower-sulfur coals because they lack the space to treat or dispose of the wastes.

In recent years there has been increased interest and activity in developing regenerable FGD systems. Such systems produce little or no wastes. Examples of such systems are the NOXSO Process, the Activated Coke Process, the Copper Oxide Process, and Sorbent Technologies Corporation's MagSorbent Process.

Most regenerable FGD processes produce an off-gas that is rich in sulfur dioxide (SO_2). The processing of this SO_2 to elemental sulfur is one of the most attractive options for the disposal of the SO_2 . Elemental sulfur can be stored and transported easily, sells for over \$100 per ton, and is one of the largest industrial commodities traded.

Project Objectives and Approach

The overall objective of this project was to optimize a sorbent-regeneration process developed by Sorbent Technologies Corporation (Sorbtech) as part of its MagSorbent system to directly yield a pure elemental sulfur product. The goal of the project was to develop a laboratory system that would demonstrate elemental sulfur yields of over 90 percent.

Prior to this project, the off-gases from Sorbtech's regeneration process was observed to contain SO_2 , H_2S and approximately 35 percent elemental sulfur. To achieve near 100 percent conversion of the sulfur in the off-gases to elemental sulfur today, a modified Claus plant would generally be the preferred route. Claus plants, however, are very expensive. Test results at Sorbtech and by others suggested that simpler, much less expensive approaches may be possible. The first technical objective of the project was to explore several of these approaches for converting the sulfur in Sorbtech's regenerator gas directly to elemental sulfur, with a conversion rate goal of over 90 percent.

More specifically, three approaches or strategies for improving sulfur conversion from regeneration off-gases were examined in the project. These approaches or strategies were:

1. Regeneration Off-Gas Recycling
2. High-Pressure Regeneration-Gas Processing
3. High-Pressure RTI Catalyst Use

In regenerator-gas recycling, the regenerator exhaust gas, after its elemental sulfur and water are condensed out, is returned to the regenerator. The theory is that returning SO_2 will shift the generation of sulfur species toward elemental sulfur. In examining the high-pressure regeneration approach, regeneration was performed at various pressures of up to about 20 atm. In the catalytic reduction approach, a new catalyst developed by Research Triangle Institute (RTI) was studied in combination with the Sorbtech regenerator gas. The three approaches or strategies were examined in three separate project phases.

If technical success were achieved in the project, a second technical objective of the research was to obtain operating data important for the design of a pilot-plant system, to identify potential problems in scaling up the process and to examine the economics and commercialization of the technology.

Technology Discussions

Many industrial exhaust gases, like Sorbtech's regenerator gas, contain significant levels of SO_2 . For environmental and economic reasons, it is desirable to recover this SO_2 and to convert it to a useful industrial form, with elemental sulfur being one of the most attractive.

The Claus Process. The most commonly employed method today to convert SO_2 to elemental sulfur is the modified Claus Process. The Claus Process, developed in Germany more than 60 years ago, involves multiple steps and produces an undesirable tail gas. In the Claus Process, concentrated H_2S can be converted to elemental sulfur in a conventional, vapor-phase Claus unit. The overall reaction occurring in the Claus unit, in this case, is:

The Claus unit generally includes three or more converter stages to achieve a 95 percent conversion of H_2S to elemental sulfur. Most Claus plants in the U.S. today produce, not elemental sulfur, but sulfuric acid as their principal product.

To utilize the Claus reaction for converting SO_2 into elemental sulfur, two-thirds of the SO_2 must first be converted to H_2S , since the Claus reaction requires a feed gas with approximately a 2:1 molar ratio of H_2S to SO_2 . To accomplish this, a portion of the SO_2 in a concentrated SO_2 gas is commonly reacted with carbon monoxide or hydrogen as follows:

After conversion of the required portion of the SO_2 to H_2S , the gases are cooled and are sent to a liquid phase Claus reactor.

The conversion of H_2S and SO_2 to elemental sulfur in the liquid phase Claus reactor occurs according to the following reaction:

The above method is commonly referred to as a modified Claus Process. Reaction (4) can be performed thermally at temperatures between 950° and 1350°C , or catalytically at temperatures from 130° to 340°C . Common Claus catalysts are activated aluminum oxide (alumina) and activated carbon.

Because the Claus-unit reactions generally do not go to completion, residual tail gases are produced. These Claus-unit tail gases have been an environmental problem in the past. Five types of control systems, ARCO, Beavon, IFP-1, SCOT, and Wellman-Lord, are presently being used in the U.S. to control emissions from Claus plants.

An approach similar to the modified Claus Process described above is used with the Wellman-Lord Process, when elemental sulfur is desired. In this case, a portion of the SO_2 produced in the Wellman-Lord Process is reacted with methane or propane with the results being a mixture of elemental sulfur, H_2S , CO_2 , H_2O and some unreacted SO_2 . In the Claus section of the process, the H_2S and remaining SO_2 react to produce additional elemental sulfur and H_2O , according to Reaction (4). A tail gas is produced that is incinerated and recycled to the absorber.⁽¹⁾

Sorbtech's Work. Sorbtech's regenerable sorbent is made from MgO coated onto individual vermiculite exfoliates.⁽²⁾ The sorbents are then given a special heat treatment. The resultant product is highly reactive toward SO_2 and NO_x in the dry state. Sulfur oxides are removed from a flue gas by two mechanisms: chemical reactions and capillary micropore condensation. SO_2 reaction products have been identified as principally magnesium sulfite (MgSO_3) and a small amount of magnesium sulfate (MgSO_4). During micropore condensation, SO_2 forms a complex with water that is drawn into the sorbent's micropores. (This same SO_2 -removal mechanism is observed in the Activated Coke process under development in Germany and Japan). These same two sorption mechanisms also appear to play parts in NO_x removal. Nitrates have been identified as a major reaction product in saturated Sorbtech sorbents by Kent State University researchers.⁽³⁾

Because Sorbtech's sorbents are relatively inexpensive, only a few regeneration cycles are required to make their use cost-effective. The sorbents are granular in size, and are sorbed in a variety of ways. One way is in a radial panel-beds; a second is as a duct injectant. Both approaches can be retrofitted into power plants before the smokestack. Sorbtech recently completed the demonstration of the panel-bed approach at a 2-MWe level (5000 scfm) at Ohio Edison's Edgewater power station in Lorain, Ohio. It is currently carrying out a project that will demonstrate the duct-injection approach at Ohio Edison's Burger power station in Dilles Bottom, Ohio. In duct-injection, small granules of sorbent are injected into the power plant duct after the electrostatic precipitator (ESP) and are collected downstream in a cyclone/small baghouse combination. The Department of Development/Ohio Coal Development Office has co-funded both projects.

The regeneration of the new sorbents, returning them to a usable condition after they have become saturated with SO_2 and NO_x , was found to be easily accomplished. The saturated sorbents can be regenerated by simply heating them in air.⁽⁴⁾ At approximately 300°C , NO_x begins to be liberated from the saturated sorbent. All NO_x is removed by about 450°C . Heavy SO_2 is evolved over the temperature range 500°C and 700°C . To obtain complete removal of SO_2 in air, it is necessary to heat the sorbent above 900°C to decompose the sulfate phase. A sorbent conditioned in air at 550°C is 90 to 95 percent regenerated.

In later work, partially funded by the U.S. EPA, Sorbtech investigators began altering the atmosphere under which regeneration was carried out to determine if these changes would affect performance.⁽⁵⁾ Surprisingly, they found that in a reducing atmosphere, several beneficial events occurred. When as little as 15 percent natural gas was included in the atmosphere, the following were observed:

- 1) The NO_x that evolved in the presence of vermiculite broke down to N_2 .
- 2) The sulfate decomposition temperature was lowered approximately 200°C degrees.
- 3) A significant portion of the SO_2 was converted directly to elemental sulfur and H_2S .

A typical regeneration-gas composition after regeneration that was observed in early test runs is provided in Table 1.

TABLE 1. REGENERATION-GAS COMPOSITION
FROM SORBTECH SORBENTS

N ₂	60 %
H ₂ O	15 %
CH ₄	5 %
CO ₂	2.5 %
SO ₂	8 %
H ₂ S	4 %
S _n	5 %
CO	4,000 ppm
NO	300 ppm

The improved performance could be achieved with natural gas (methane), CO, or hydrogen. For cost and safety reasons, natural gas was selected for use in the improved regeneration process. Sorbent regenerated with 15 percent or more natural gas present in the atmosphere performed as well as, and sometimes better than, fresh sorbent.

Several reactions occur during regeneration. Among the important reactions are the reforming or decomposition of methane (when methane is employed as the reducing gas), the decomposition or reduction of MgSO₃ and MgSO₄, and other reduction reactions involving the reducing gases. Reactions involving methane that have been observed include:

Regeneration reactions involving magnesium sulfite and sulfate that have been observed include:

Additional reactions that are believed to occur include:

For the direct recovery of elemental sulfur, reactions (10), (11) and (12) above are most important. It is desirable that these reactions go to completion and occur in preference to reactions (13) and (14).

Research Triangle Institute's Work. Under contract from the U.S. DOE-Morgantown Energy Technology Center, researchers at the Research Triangle Institute (RTI) have been developing a proprietary catalyst process for converting the concentrated SO_2 off-gases from integrated gasification/combined cycle desulfurization circuits into elemental sulfur. They have made some interesting discoveries, which were believed to be possibly translatable to Sorbtech's regeneration process.

RTI researchers found that, contrary to standard thermodynamic predictions, raising the pressure of their catalytic reduction process increased their elemental sulfur yields dramatically.⁽⁶⁾ (See the associated graph in Figure 1.) They called their scheme the Direct Sulfur Recovery

From: S. Gangwal et al,
"The Direct Sulfur Recovery Process,"
1990 Summer National Meeting,
American Instit. of Chemical Engineers,
August 19-22, 1990.

Figure 1. Elemental Sulfur Conversion as a Function of Pressure

Process (DSRP). While the nature of their catalytic material remains proprietary, much of their experimental data is public. Their process is said to be capable of greater than 90 percent direct-sulfur conversion in a once-through mode at relatively high space velocities and, otherwise, seems parametrically robust.

Before this project, Sorbtech never tried regenerating its sorbents under pressure and there were indications that the chemistry could proceed similarly to that observed by RTI. First, it was known that vermiculite, which supports and distributes the reactive species in the sorbents, is a low-grade catalyst itself. Second, the regenerative behavior of Sorbtech's sorbents mimicked that of RTI's materials at atmospheric pressure. With a reducing gas and a 1 to 5 vol% SO_2 level,

both reacted to provide about 20 to 25 percent conversions directly to elemental sulfur. Moreover, the two processes worked over the same temperature ranges. And finally, they both performed with significant levels of steam present – from 5 to 30 vol%. (The Claus equilibrium is limited by the water content of the gas, which must be removed in an intercooling step between stages.)

Research by Others. An indirect approach for converting SO_2 to elemental sulfur has been suggested by Deschamps.⁽⁷⁾ In Deschamps' approach, the SO_2 -containing gas is contacted and reacted with an aqueous solution containing ammonium sulfite or Thiosulfate and the resulting solution is then contacted with H_2S to produce elemental sulfur.

Additional processes involving the direct conversion of SO_2 in a SO_2 -rich gas to elemental sulfur include that of Eisenlohr, in which hydrogen is added to the gas and the gas is passed through a bed of cobalt oxide, nickel oxide, molybdenum oxide, or tungsten oxide at 200° to 450°C , and that of Moss, in which the sulfur oxide-rich gas is passed through a fluidized bed of solid carbon char particles at 600° to 1200°C .^(8,9) Disadvantages of the Eisenlohr process are the high cost of the catalysts, the possibility of heavy metal pollution, and the added requirement of a Claus plant to process the gas exiting the catalyst after the elemental sulfur is removed. The disadvantages of the Moss process include the relatively complicated nature of the equipment that is required, the ignition dangers in employing hot, reactive char, and the reported low sulfur yields that occur.

REGENERATION OFF-GAS RECYCLE STUDIES

Background

A single saturated sorbent material was employed for all runs performed in this phase of the work and for nearly all runs in other phases, as well. The sorbent material was saturated with SO_2 , and to a lesser degree, NO_x in pilot plant tests at Ohio Edison's Edgewater power plant in Lorain, Ohio during the summer of 1992.⁽¹⁰⁾ Approximately a barrel of sorbent was removed from the pilot plant sorber and was brought to Sorbtech's Twinsburg laboratories where it was thoroughly mixed and sampled. Samples of exposed sorbent and regenerated sorbent were then sent to Premier Chemical Company's research laboratories where they were analyzed. Premier is one of the U.S.'s leading MgO producers and has relatively elaborate analyses facilities for MgO materials. The composition of the exposed material is given in Table 2.

Experimental Procedures

Work in Off-Gas Recycle Studies was actually performed in two parts or tasks. During the first part, the test apparatuses were designed, constructed, and installed, and a number of single-pass experiments were performed to test out the equipment and to obtain baseline data. Two different test apparatuses were employed. One system, constructed of clear quartz glass, was similar to that employed in earlier studies at Sorbtech. Figure 2 shows this system.

Advantages of this system were that the reactions that occur could be observed visually, that samples could be heated relatively rapidly, and that almost complete capture and recovery of emitted elemental sulfur were possible. Bright yellow sulfur was deposited on the exit walls of the reactor and inside the condenser. This material could be recovered and weighed. Very little yellow sulfur appeared after the condenser. The disadvantages of the quartz glass system were that elevated pressures were not possible and recycling was somewhat difficult to carry out.

The second test-apparatus system, shown in Figures 3 and 4, was designed to be used in all studies in the project. It was a robust system. For Recycle Studies, the principal components consisted of a gas-delivery system; heat-traced, stainless steel tubing; mass-flow controllers to control the flow of purge, carrier and reaction gases in the system; a gas preheater; an externally-heated regenerator vessel to hold the saturated sorbent samples; a sulfur condenser to trap and collect sulfur produced during regeneration; a water condenser to remove water vapor (later the two condensers were combined); a back-pressure valve to control the system pressure; and various flowmeters, filters, thermocouples, sampling ports, gages, and valves to control the flow of gases. The system also contained a recycle loop consisting of a gas-holding vessel, a gas pump, and stainless steel tubing. Essentially all components in the system were constructed of stainless steel. Gases during recycling were removed from the primary line after the water condenser and were reintroduced into the primary line before the gas preheater, as indicated in Figure 3. Design drawings for individual system components are provided in the Appendix of this report.

The step-by-step procedures generally followed in single-pass experiments consisted of:

1. Introducing a saturated sorbent sample into the regenerator vessel and sealing the system.
2. Initiating a flow of a purge gas (nitrogen) through the system and checking for leaks (and taking corrective action, when necessary).

3. Turning on the gas preheater, regenerator heater, heating tapes, and other heaters in the system, while initiating the flows of the desired carrier/reaction gases.
4. Recording as a function of time, the temperatures at various locations in the system, the flow rates of the gases entering and exiting the system, and the system pressure.
5. Collecting syringe samples of gases exiting the system at regular intervals and analyzing these samples with a gas chromatograph.
6. After a pre-selected time (about 2 hours) or when SO_2 was no longer detected in the exit gas, the system was again purged with nitrogen and allowed to cool to room temperature.
7. When at room temperature, the regenerator and sulfur condenser were dismantled, and the regenerated sorbent sample and sulfur that was collected in the condensers were weighed.

TABLE 2. COMPOSITION OF EXPOSED SORBENT

36.3%	MgO
13.7%	H ₂ O
13.4%	MgSO ₄
8.1%	MgSO ₃
0.0%	NO _x
28.5%	Vermiculite

Figure 2. Quartz Reactor Test Set-Up

Figure 3. Off-Gas Recycle Studies Arrangement

Figure 4. Direct-Sulfur Test Apparatus Used
in Recycling Experiments

In this apparatus, saturated sorbent samples were placed inside a special regeneration reactor enclosed in the vertical furnace shown in the center of this photograph. A drawing of the regeneration reactor is given in the Appendix. Methane, supplied from the cylinder on the left, was heated in a preheater (shown below the reactor furnace) before being supplied to the bottom of the regenerator. Regeneration gas from the reactor was then passed through heated and insulated piping to a sulfur and water condenser. The insulation is not shown in the photograph. From the condenser, the gas was pumped to a storage vessel and then recycled back to the preheater. The pumping system is shown on the right, and major items in the recycle system are located behind the panel. Other important system components, such as valves, controls and gages, appear in the photograph.

The gas chromatograph was routinely checked and calibrated against standard gases provided by Matheson Gas Products. Other gas analysis methods were also employed during many runs. These methods included chemical cells for SO₂, NO_x, CO and oxygen levels, and length-of-stain indicator tubes for SO₂, NO_x, CO₂ and H₂S. There was good agreement between all the methods employed.

All experimental runs in the second part of the work involved recycling the regenerator gases. Eight recycling runs were performed.

The procedures followed in recycling runs were similar to those described above, except:

1. After the system was purged with N₂ and checked for leaks, a known volume of CH₄ was introduced. The system was then isolated by closing gas inlet and exit valves.
2. With the recycle pump on, gas was continually recycled through the system. As before, gas temperatures, pressures, and flow rates at various locations in the system were continually monitored as a function of time, and gas samples were removed and analyzed.
3. The recycling runs were terminated after a preselected time or when no SO₂ was detected in the regenerator gas.

Results

It was clear from sorbent analyses before and after runs and from gas analyses in earlier regeneration tests in a reducing environment, that most of the sulfur in saturated sorbent materials was emitted to the regenerator exhaust gas as SO₂, H₂S, and elemental sulfur. Although the SO₂ and H₂S levels in these earlier tests could be accurately measured, the levels of elemental sulfur could not. This was due to difficulties in capturing and collecting the elemental sulfur. In this project, however, these difficulties appeared to have been overcome with the improved design of the quartz system. It was observed that most elemental sulfur was collectible and recoverable in the quartz system, and, as a consequence, reliable data on elemental sulfur yields could be obtained.

Quartz System Runs. Six regenerator runs were performed using the quartz system. They were performed with 100 percent methane (50 to 100 sccm) at essentially atmospheric pressure. In two runs conducted on saturated sorbent, the average sulfur yield was 73.7 percent. This is significantly higher than was expected on the basis of earlier test results. SO₂ in the regenerator off-gas accounted for 12.4 percent of the sulfur present in the sorbent; H₂S accounted for 8.5 percent of the sulfur; 5.4 percent of the sulfur remained in the sorbent. The maximum regeneration temperature in these runs was about 720°C.

In several quartz system runs, particular attention was given to temperature at which significant elemental sulfur production begins to occur. In these cases, the sorbent sample was heated slowly and sulfur in the gas was noted as it deposited on the exit tubing walls and in a special condenser. With saturated sorbents, it appeared that heavy generation of elemental sulfur began at 690°C.

A quartz-system run was then performed using 100 percent commercial-grade magnesium sulfate, 7-hydrate ($\text{MgSO}_4 \cdot 7 \text{H}_2\text{O}$), supplied by J. T. Baker Chemical Co. Results similar to those observed with saturated MagSorbent were seen with this material. The regeneration temperature at which elemental sulfur was first produced, however, was significantly higher. The MgSO_4 decomposed at 1124°C . No sulfur was observed until the material reached 760°C in a 100 percent methane environment. The commercial magnesium sulfate was then coated onto vermiculite particles, and the combination was examined in a methane environment in another run. Similar results were seen, except the first appearance of elemental sulfur occurred at 688°C .

Stainless Steel System, Single-Pass Runs. Nine separate single-pass runs were performed. These runs are summarized in Table 3, and additional data on many of these runs are given in the Appendix. The runs were carried out mainly to uncover potential problems in the stainless steel system and to correct them, when possible, and to attempt to duplicate the results observed in the quartz system.

Two major problems were observed with the stainless steel system. One related to the capture and recovery of the elemental sulfur; the second involved the cracking of methane. The use of a special sulfur condenser, patterned after RTI's sulfur condenser design, proved to be impractical in the project owing to the batch nature of the apparatus. Because only relatively small quantities of sorbent were employed in each run, relatively small quantities of elemental sulfur were produced. The sulfur condenser was designed to operate at a constant temperature, a temperature below the vaporization (or boiling) point of sulfur, but above its melting point. The concept was to collect the sulfur as a liquid and then tap it off after each run.

Because the sulfur quantities produced were so small and because most sulfur condensed as tiny liquid particles, not within the sulfur condenser itself, but on the walls of tubing and the reactor and within the gas stream, full collection and recovery of sulfur in the condenser was not possible. Also, because the condenser required immersion in a flammable, odorous Isotherm liquid, great difficulties and safety problems were encountered in opening and recovering sulfur that deposited there. The original sulfur condenser was replaced with one of a simpler design. The new design was water-cooled and contained a collection media. The initial media examined was virgin expanded vermiculite. This material performed satisfactorily, but glass wool in later tests proved to be better. The use of the new sulfur condenser, however, did not solve the problem of sulfur condensation on piping and other components. Heat tracing and heavy insulation improved the situation, but did not solve the problem. In nearly all runs, more sulfur came out on the inside surfaces of process components than inside the sulfur condenser itself. Sulfur in the piping could be clearly seen and could be removed by heating up the entire system and passing air through it. When this was done, heavy concentrations of SO_2 were produced.

It has been reported in the literature that methane will begin cracking at temperatures above 785°C , particularly when in the presence of nickel or iron oxide.^(11,12) The stainless steel preheater employed in the apparatus unfortunately contained both nickel and iron oxide (from air tests performed early in the project). The result was that methane often cracked when it was heated. The products of cracking included hydrogen and carbon. The hydrogen served as a reducing agent for the sorbent; much of the carbon remained inside the gas preheater. Because carbon generally occurs on regenerated sorbent particles during normal regeneration in methane or CO, cracking probably occurs regardless of whether or not a gas preheater is employed.

TABLE 3. SUMMARY OF SINGLE-PASS RUNS
IN THE STAINLESS STEEL SYSTEM

Run No.	Purpose of Run	Atmosphere	Total Flow Rate (ccm)	Max. Reg'n Temp. (°C)	System Pressure (psig)	Results	S Removal from Sorbent %	Wt. Loss of Sorbent %
1	Check out system	N ₂	500	979	15	Found and corrected leaks.	61.6	22.30
2	First examination of CH ₄	CH ₄	500	865	15	Some S collected; open bulb collection is not effective collector.	97.7	28.90
3	Another examination of CH ₄	CH ₄	500	871	15	Some S collected; vermiculite bed is better collector.	93.1	28.50
4	Lower CH ₄ flow rate	CH ₄	50	895	15	Less S collected, but glass wool trap is better still; S deposited in tubing.	92.8	28.20
5	Examine an air atmosphere	Air	100	908	15	Black and green deposits, changed to yellow on drying. High SO ₂ concentration; no H ₂ S.	71.9	25.23
6	Examine a N ₂ atmosphere	N ₂	100	949	15	Green liquid changed to yellow on drying; High SO ₂ concentration; no H ₂ S.	68.8	26.56
7	Examine CH ₄ /N ₂ mixtures	CH ₄ :N ₂ (1:50)	250	682	6	Green liquid; Low SO ₂ concentration; No H ₂ S.	9.0	18.24
8	Examine CH ₄ /N ₂ mixtures	CH ₄ :N ₂ (1:4)	250	678	8	Green liquid; Low SO ₂ concentration; No H ₂ S.	9.6	16.45
9	Repeat of the runs conducted in the quartz system	CH ₄	100	734	15	Yellow sulfur deposits and green liquid; Low SO ₂ and low H ₂ S concentrations.	93.4	27.06

Stainless Steel System, Recycling Runs. Nine recycling runs were performed. The initial runs were made to shake down the system. Six runs were made with saturated sorbent materials (See Appendix A). One of the initial sorbent runs was conducted at relatively low temperatures (the average and maximum temperatures were 650 and 692°C). This run, which employed N_2 and CH_4 mixture having a volume ratio of 8 to 1 and a 0.5 to 1 methane:sulfur ratio, showed relatively poor regeneration performance. Although yellow sulfur was collected during the run, the percentage weight loss of the sample during the run was low, the total amount of sulfur collected was small, and 100 percent sulfur recovery was not achieved. All later recycling runs with sorbent were performed at higher average regeneration temperatures and with higher levels of CH_4 . All resulted in essentially 100 percent conversion of the off-gassed sulfur to elemental sulfur.

Three recycling runs were performed with no saturated sorbent in the system. In one case, a gas stream containing 0.012 mole SO_2 , 0.029 mole CH_4 and 0.342 mole N_2 was recycled through the apparatus. There appeared to be some reaction of SO_2 with the CH_4 , but no elemental sulfur was observed. In a second case, a gas stream containing 0.017 mole SO_2 , 0.368 mole N_2 and no CH_4 was recycled through the apparatus. In this case, the level of SO_2 slowly decreased and CO_2 began appearing in the gas, but again no elemental sulfur was seen. It was suspected that the SO_2 was reacting with carbon deposits in the system. To investigate this possibility, a third recycling run was performed in which only air was recycled through the system. This run resulted in heavy concentrations of SO_2 and CO_2 in the gas stream. These results clearly indicated that significant quantities of carbon and sulfur were present in the system beforehand from prior runs. Passing air through the system at high temperatures was found to be an effective approach to cleanse the system of carbon and sulfur and became a standard procedure in performing recycling runs.

Recycling of the regeneration gas with saturated sorbent in the system was easy to carry out and to control. It was found possible to convert essentially all SO_2 and H_2S present in a regeneration gas by recycling the gas three or more times. Representative data from three runs are presented in Table 4.

Regenerating at temperatures above 750°C appeared to result in essentially complete removal of sulfur from saturated sorbents in single-pass runs. Regenerating at temperatures above 850°C, however, appeared to degrade physically the vermiculite phase of the sorbents.

Elemental sulfur quantities collected during quartz system runs correlated well with totals of collected elemental sulfur plus sulfur lost in the system during cycling runs. If it is assumed that most of the sulfur lost in the system during recycling runs was coated inside the tubing and other system components, which from visual observations appeared to be the case, then some of the data appearing in Table 4 can be restructured to indicate the percentage of released sulfur that occurred as elemental sulfur during each cycle in regenerating sorbent at about 710° to 760°C. These percentages are shown graphically in Figure 5.

TABLE 4. SULFUR CONVERSIONS BY
RECYCLING THE REGENERATOR GAS

<u>Average/Maximum Regeneration Temperature (°C)</u>	<u>Initial S in Sorbent (g-mol)</u>	<u>CH₄:S Ratio</u>	<u>Number of Recycles</u>	<u>% Reduction of SO₂ Remaining in the Gas After the Initial Pass</u>
621/648	0.088	2.1:1	1	13
			2	37
			3	42
			4	100
704/757	0.088	1.7:1	1	33
			2	64
			3	100
			4	100
717/762	0.088	2.2:1	1	62
			2	78
			3	95
			4	100

Figure 5. Total Cumulated Elemental Sulfur
Recovered as a Percentage of Sulfur
Released During Regeneration
for Progressive Cycles

Discussion of Results

Recycling was observed to be a relatively straight-forward approach to regenerate sorbent samples and to achieve a 100 percent reduction in the SO_2 released from the sorbent during regeneration. Capturing and measuring elemental sulfur that was produced during successive recycling cycles with the closed stainless steel laboratory system, however, was not possible owing to the nature of the system, to the manner in which the sulfur condensed, and to the continuous manner of recycling. The elemental sulfur that was produced began condensing out immediately upon leaving the reactor vessel. In doing so, it coated the inside surfaces of stainless steel tubing and other components in the system leading to the condenser. There was no way of measuring elemental sulfur collected with each successive cycle. For the batch system, on the other hand, it was found possible to collect the elemental sulfur on glass wool and to make weight measurements. The quantity of sulfur delivered to the condenser, and not deposited in the lines, was observed to be a function of the velocity of the gas passing through the system, as the data in Table 5 below indicate.

TABLE 5. SULFUR COLLECTED WITH TWO
DIFFERENT GAS-FLOW RATES

Gas Flow Rate	500 sccm CH_4	50 sccm CH_4
Maximum Reactor Temperature	871°C	895°C
Weight Loss of Sorbent	28.5%	28.2%
Sulfur Removed from Sorbent	93.1%	92.8%
Sulfur in Sulfur Condenser	1.54g	0.25g

It was also observed during recycling that a small amount of the sulfur in the recycle gas appeared to be resorbed by the sorbent. The result was that, although sulfur disappeared from the recycle stream and additional elemental sulfur was collected with each successive cycle, the overall regeneration performance (as indicated by the amount of sulfur remaining in the sorbent after regeneration) decreased with recycling.

The sulfur that was collected in most runs was bright yellow in color. It was collected using a fresh bed of glass wool for each run. Although the glass-wool collection approach appeared to be adequate for laboratory runs, for a continuous process, glass wool does not appear to be a practical collection media; water sprays or some other approach would appear better. It is recommended that some attention be given to capturing and collecting sulfur on a large scale in future work.

HIGH-PRESSURE REGENERATION STUDIES

Background

As mentioned earlier, researchers at Research Triangle Institute (RTI), working with U.S. DOE support, examined ways of improving the performance of a new catalyst that they had developed. They found that raising the pressure of their catalytic reduction system increased their elemental sulfur yields dramatically. RTI recommended to Sorbtech that it consider increasing the pressure of its system to increase elemental sulfur yield. It should be noted, however, that RTI observed essentially zero percent elemental sulfur yield at atmospheric pressure.

The objectives of this project phase then were to determine if improvements in sulfur yield can be achieved by increasing the pressure of the system during the regeneration of saturated sorbents and, if so, to determine what improvements could be expected.

Experimental Procedures

The test system that was designed and installed for this phase of the project is shown schematically in Figure 6. The system was a modification of the one employed in Recycling Studies. Key components of this system included gas supply units, mass-flow controllers, a gas pre-heater, a heated reactor vessel (where regeneration of the sorbent took place), a sulfur and water condenser, a Balston filter, a back-pressure regulator and miscellaneous meters and instruments. Gas was supplied from individual gas cylinders that were connected together with a manifold.

Only one problem of significance was encountered in this phase of the research. During the first run, 100 percent methane was introduced into the system initially. During heat-up of the saturated sorbent sample under these conditions, a strong exothermic reaction occurred in the sorption-regeneration vessel that increased the temperature of the system uncontrollably. The result was a softening and ballooning of the stainless steel vessel and little elemental sulfur being recovered. The problem was eliminated by pressurizing the system initially with nitrogen and then introducing methane as a methane-nitrogen mixture.

For each run, a 50-gram sample of saturated sorbent was placed into the reactor, and the system was flushed with nitrogen. The system was then pressurized with nitrogen to the desired pressure level. Afterwards a flow of 100 sccm of methane and 100 sccm of nitrogen was begun. Concurrently the gas preheater, heating tapes, and the reactor heater were energized, and the system was allowed to come up to temperature. The time of each run was 120 minutes.

Throughout each run, temperature, pressure and flow rate data were collected. Gas samples were taken periodically and analyzed with a gas chromatograph. A computer program was used to determine the amount of sulfur dioxide (SO_2) and hydrogen sulfide (H_2S) which was generated during the run. Elemental sulfur was captured in the sulfur trap. Samples of the saturated and regenerated sorbents were sent to an outside laboratory for analysis of the sulfur content of the sorbent before and after regeneration. From these data, material balances of the sulfur-bearing species were calculated.

Figure 6. Arrangement for High-Pressure Runs

Results

Table 6 shows the results from six runs performed at four different pressures. The run at 0 psig represents the baseline conditions. Regenerating at elevated pressures would appear justified only if the yield of elemental sulfur was significantly better than that obtained at baseline conditions.

TABLE 6. DATA FOR RUNS AT DIFFERENT PRESSURES

<u>Run No.</u>	<u>30**</u>	<u>38***</u>	<u>14</u>	<u>42***</u>	<u>29</u>	<u>45***</u>
Pressure, psig	0	0	15	115	290	290
Maximum Temperature °C	719	734	895	745	772	760
Sorbent Wt. Loss, %	26.72	27.06	28.22	23.64	28.34	29.03
Sulfur as SO ₂ , %	13	23	8	0	0	0
Sulfur as H ₂ S, %	6	2	7	43	7	28
Captured Sulfur, %	77	55	9	25	9	11
Sulfur Remaining in Sorbent, %	4	5	7	31	12	12
Missing Sulfur*, %	0	15	69	1	72	49
Captured S Plus Missing S, %	77	70	78	26	81	60
<hr/>						
Initial Mass, g	21.86	50.37	50.07	50.46	50.45	50.56
Initial S, wt%	5.62	5.62	5.62	5.62	5.62	5.62
Final Mass, g	16.02	36.74	35.94	38.53	36.15	35.88
Final S, wt%	0.31	0.37	0.56	2.26	0.94	0.95

Note: The sulfur values above represent sulfur as a percentage of the total sulfur in the system.

* Much of the missing sulfur coated the inside of tubing and vessels before the sulfur trap.

** Quartz system employed.

*** The sulfur trap was moved closer to the regenerator for these runs.

Examples of chemical analyses data collected during the runs are shown in Figures 7, 8, and 9. Figure 7 shows the compositions of gases leaving the regenerator and the regenerator temperature as a function of time for Run 38 (conducted at near atmospheric pressure). Here, it can be seen that some SO_2 was generated during heat-up, but after the regenerator temperature reached 700°C , the SO_2 decreased and eventually reached zero percent.

Very small levels of H_2S and CO were also present in the gas at 700°C . After about 100 minutes into the test, the methane (CH_4) level abruptly increased and the CO_2 level abruptly decreased, indicating that the sorbent was probably fully regenerated at that time. Figures 8 and 9 show similar data for Runs 42 (115 psig) and 45 (290 psig). For these cases at elevated pressures, different evolution patterns occurred. Increasing levels of H_2S and essentially no SO_2 were observed in the residual gases. At 290 psi, the regeneration of the saturated sorbents appeared to be incomplete, even after 120 minutes. An analyses of sorbent samples after the runs appeared to show higher residual sulfur levels for samples run at higher pressures.

Discussion of Results

On the basis of the test results, it was concluded that operating at elevated pressures does not present any significant advantages relating to elemental sulfur yields. The yields at 290 psig (20 atm) pressure were essentially the same as those at normal pressures. One might justify the use of higher pressures if he or she wants no SO_2 present in the exiting gas stream but can tolerate some H_2S . However, even then, the added cost of high-pressure operation makes the use of high pressures not particularly attractive.

Atmospheric Pressure Run

0 psig

Figure 7. Regenerator Off-Gas Composition and Temperature vs. Time for an Atmospheric Pressure Run

Medium Pressure Run

115 psig

Figure 8. Regenerator Off-Gas Composition and Temperature vs. Time for a Run at 115 psig.

Pressurized Run

290 psig

Figure 9. Regenerator Off-Gas Composition and Temperature vs. Time for a Run at 290 psig.

HIGH-PRESSURE RTI CATALYST STUDIES

Background

Since 1988, Research Triangle Institute (RTI) has been investigating the conversion of SO_2 directly to elemental sulfur. It has screened a number of potential catalysts that will result in the conversion of SO_2 to elemental sulfur when a near stoichiometric mixture of SO_2 -containing gas and a reducing gas is reacted at high temperatures. RTI has discovered one catalyst that will result in over 90 percent conversion at 450 to 650°C when the off-gas is processed at 20 atm. At 1.7 atm, however, the catalyst will give a typical conversion to sulfur of only about 18 percent and at 1.0 atm, essentially zero percent. RTI will not disclose the composition of its catalyst, but did provide Sorbtech researchers with small quantities of the catalyst for this study.

The objective of this phase of the work then is to determine what improvements in sulfur yield can be expected by employing the special catalyst recently developed by RTI.

Experimental Procedures

Sorbtech initially modified the test system employed in the earlier phases to include two heated reactor vessels, as shown in Figure 10. The first reactor was designed to regenerate batches of saturated sorbent and the second reactor was designed to hold a bed of RTI catalyst to process further the regenerator off-gases to elemental sulfur. However, after two test runs it was determined that the RTI catalyst could not be used in a batch-type reactor setup, as designed, because during the regeneration of the sorbent, the concentration of SO_2 in the off-gas varied and the proper ratio of reducing gas (CH_4) to SO_2 could not be maintained. According to RTI, if the ratio of reducing gas to SO_2 exceeds 2:1, the catalyst will be degraded. Severe degradation of the catalyst was observed during these two runs.

Because the RTI catalyst could not be used to process the off-gases from the regeneration of a batch of saturated sorbent, it was decided to further modify the test system to process a continuous, simulated regeneration off-gas stream of a composition normally observed during regeneration. The modified system is shown in Figure 11.

A material balance of the off-gases from the regeneration of a batch of sorbent at atmospheric pressure in a 50 percent methane/50 percent nitrogen environment was used to determine the approximate concentration of a gas stream which would be evolved during the continuous regeneration of sorbent. It was assumed that all of the elemental sulfur and water produced during the regeneration were removed. The composition of the estimated gas stream is given in Table 7.

TABLE 7. COMPOSITION OF OFF-GASES FROM THE
CONTINUOUS REGENERATION OF THE MgO /VERMICULITE SORBENT

5%	SO_2
2%	H_2S
1%	CO
7%	CH_4
35%	CO_2
50%	N_2 and other species

Figure 10. High-Pressure Catalyst-Test System with Two Heated Reactor Vessels

Figure 11. Modified High-Pressure Catalyst-Test System

A cylinder of gas containing 7 percent SO₂ and balance N₂ was prepared and supplied by Matheson Gas Products. When mixed with the reducing gas (CH₄ was used to simulate the three reducing gases present in the regeneration off-gas: H₂S, CO, and CH₄), the composition of the resultant inlet gas stream was 6 percent SO₂, 12 percent CH₄ and balance N₂. For runs where a 1:1 ratio of reducing gas to SO₂ was required, the inlet gas stream's composition was 6.4 percent SO₂, 6.4 percent CH₄ and balance N₂.

An EG & G Chandler Engineering series 100 AGC gas chromatograph with a thermal conductivity detector was used to determine the concentration of SO₂, CO₂, H₂S, O₂, N₂, CH₄, COS and CO in the reactor's outlet stream. A Horiba model PIR 2000 Process Gas Analyzer with a non-dispersive infrared detection cell was used when the SO₂ concentration in the outlet stream fell below the GC's lower detection limit of 2000 ppm SO₂.

Results

Seven runs were performed to determine what effects system pressure, catalyst temperature, and CH₄-to-SO₂ molar ratio have on the conversion of SO₂ to elemental sulfur. After the desired operating conditions were obtained, the system was maintained at these conditions for 30 minutes and then the exit gas stream was sampled. Electronic mass flow controllers were used to accurately meter the inlet gases. Since the flowrate of N₂ in the inlet stream was known and assuming that N₂ did not react, the total outlet gas flowrate could be calculated from the concentration of N₂ in the outlet gases. From the total flowrate and the GC results, the flowrate of the other exhaust gases could then be determined. It was assumed that sulfur which did not leave the reactor as SO₂, H₂S or COS had been converted to elemental sulfur. Table 8 presents the percent conversion of SO₂ to elemental sulfur and H₂S at the various operating conditions. The data in Table 8 are presented in Figures 12 and 13.

TABLE 8. COMPILATION OF RTI CATALYST TEST DATA

Run #	Catalyst Temp, °C	System Pressure, psia	CH ₄ -to-SO ₂ Molar Ratio	Conversion To Elemental Sulfur, %	Conversion To H ₂ S, %	Conversion To COS, %	Remaining as SO ₂ , %
46	350	300	2:1	1.4	0	0	98.6
47	500	300	2:1	29.0	0	0	71.0
48	665	300	2:1	12.8	70.4	14.8	2.0
49	665	300	1:1	89.8	9.0	0.9	0.4
50	665	217	2:1	22.6	74.0	2.0	1.3
51	665	217	1:1	53.9	46.0	0	0.1
52	665	115	2:1	5.2	93.0	0	1.8

Figure 12 shows the effect of catalyst temperature on the conversion of SO_2 to elemental sulfur. The CH_4 -to- SO_2 ratio was 2:1, and the system pressure was 300 psia. At 350°C, only 1.4 percent of the SO_2 was converted to sulfur and no H_2S or COS was produced. At 500°C, the yield of sulfur increased to 29.0 percent with still no production of H_2S or COS. Upon increasing the temperature to 665°C, the amount of SO_2 converted to elemental sulfur was 12.8 percent. However, at 665°C, 70.4 percent of the SO_2 was converted to H_2S , 14.8 percent was converted to COS, and 2.0 percent remained as SO_2 .

Figure 12 also shows the effect of pressure on the conversion of SO_2 to elemental sulfur. The CH_4 -to- SO_2 ratio was 2:1, and the catalyst temperature was 665°C. At these conditions, the yield of elemental sulfur was highest when the pressure was 217 psia (14.5 atm). Increasing the pressure to 300 psia (20 atm) favored the formation of COS.

Figure 13 shows the effect of varying the CH_4 -to- SO_2 ratio at system pressures of 217 psia and 300 psia, respectively. At both pressures, the yield of elemental sulfur decreased, and the production of H_2S and COS increased when the CH_4 -to- SO_2 ratio was increased from 1:1 to 2:1. At a CH_4 -to- SO_2 ratio of 1:1, a catalyst temperature of 665°C, and a pressure of 300 psia (20 atm), nearly 90 percent of the SO_2 was converted to elemental sulfur.

Discussion of Results

RTI engineers were able to achieve over a 90 percent conversion of SO_2 to elemental sulfur when they operated their catalyst at 450 to 650°C, 20 atm pressure and a reducing gas-to- SO_2 ratio of 2 to 1. RTI's reducing gas was a simulated, dry KRW-type coal gas containing CO, H_2 and some H_2S .

In RTI's tests, a 2-to-1 ratio was required to provide a near-stoichiometric mixture of reducing gas and SO_2 . In this project, CH_4 was used as the reducing gas because CH_4 is commonly employed to regenerate saturated MagSorbents and would be present in the regeneration off-gas. We obtained a nearly 90 percent conversion of SO_2 to elemental sulfur at 665°C, 20 atm pressure, and a 1:1 ratio of CH_4 to SO_2 . When the ratio was 2:1, the CH_4 was in excess and the reaction was not driven to completion, resulting in an increase in H_2S and COS.

The tests conducted in this project demonstrated that the catalyst developed by RTI can be used to convert the SO_2 in the regeneration off-gas to elemental sulfur. A nearly 90 percent yield was obtained at a catalyst temperature of 665°C, a system pressure of 300 psia (20 atm), and a CH_4 -to- SO_2 ratio of 1:1. It was observed that the amount of methane and SO_2 must be maintained at a CH_4 -to- SO_2 ratio of 1:1 to achieve suitable results.

Effect of Varying Temperature

CH₄ to SO₂ Ratio, 2:1 Pressure, 300psia

Effect of Varying Pressure

CH₄ to SO₂ Ratio, 2:1 Temperature 665C

Figure 12. Effects of Varying Temperature and Pressure on SO₂ Conversions with a RTI Catalyst

Effect of Varying CH₄ to SO₂ Ratio

Pressure, 217 psia Temperature 665C

Pressure, 300 psia Temperature 665C

Figure 13. Effects of Varying CH₄-to-SO₂ Ratio on SO₂ Conversions with a RTI Catalyst

TECHNICAL DISCUSSIONS

The project's technical objectives were attained. Figure 14 summarizes the best results that were achieved with different approaches that were examined. Two approaches, the use of recycling and the use of a RTI catalyst at relatively high pressure, met the project's goal of a 90 percent or better conversion of SO_2 to elemental sulfur, although recycling appeared to decrease overall regeneration efficiency somewhat.

Also, other noteworthy discoveries were made on the project. They included:

1. By improving the desorption equipment design, including the addition of improved sulfur collection facilities, it was possible to attain a 77 percent conversion of sulfur present in saturated sorbent to elemental sulfur during a single pass of gas.
2. By employing magnesium oxide, by itself or in combination with vermiculite or perlite, it was found possible to convert SO_2 in other gas streams to elemental sulfur. Magnesium oxide, by itself, proved to be a good catalyst for converting SO_2 to elemental sulfur at temperatures above 650°C and at atmospheric pressure.

Figure 14. Comparison of Approaches to Maximize Elemental Sulfur Production

In the project, multiple recycling of the regeneration gas (methane) through the initially-saturated sorbent bed resulted in essentially complete conversion of the SO_2 and H_2S in the gas to elemental sulfur. How multiple recycling can be applied commercially poses some interesting questions. For example, the continuous addition of new saturated sorbent to a desorption apparatus will result in additional desorbed gas volumes. In any continuous system, some product gas therefore must be continually removed from the system. But any gas removed before it is recycled 3-4 times will not be fully converted to elemental sulfur unless the CO_2 can be separated from the other components in the gas and this CO_2 can be removed from the system. Is it possible to do this? If not, 100-percent conversions may not be possible by recycling in a continuous process. On the other hand, employing a single cycle of gas through multiple beds could allow one to carry out a continuous process, and perhaps achieve 100-percent conversions. Such an approach is recommended for further study. Also, if only a small quantity of H_2S remains in the product gas stream, it should be possible to convert (burn) this H_2S to SO_2 simply and relatively inexpensively. Commercial burners are available to accomplish this. The resulting dilute SO_2 -containing gas can then be returned to the original gas stream to be captured by the sorbent.

The RTI-developed catalyst demonstrated the ability to convert a SO_2 -containing gas stream of a composition similar to that of a desorbed sorbent gas stream to elemental sulfur. The resulting gas stream contained, in addition to elemental sulfur, H_2S , H_2O , N_2 , CO_2 , CH_4 , and sometimes CO , COS and SO_2 . The percentages of each component varied with temperature. High conversions to elemental sulfur were possible only at high pressures and were dependent on good control of temperatures, pressures and flow rates. It would appear that any residual H_2S will require some additional processing. Two possible approaches are (1) converting the H_2S to SO_2 and reinjecting the gas product back into the feed-gas stream in a manner as described earlier, and (2) directly recycling the H_2S -containing gas to the gas stream entering the RTI-catalyst bed.

MARKETING/COMMERCIALIZATION DISCUSSIONS

Sulfur dioxide has always been considered a troublesome gas with respect to air pollution. It is created through the combustion of any sulfur-bearing material. Common sulfur-bearing materials include ores of lead, zinc and copper, and fuels, such as coal and fuel oil. Sulfur dioxide is of particular concern when burning high-sulfur coals, and most Ohio coals are considered high-sulfur coals.

In recent years, there have been extensive efforts to capture and remove the sulfur species in gases produced in burning or gasifying coal. In cases where regenerative sorbents are involved (and these processes generally are most attractive from an environmental standpoint), the results are often gas streams rich in SO_2 . Studies performed by the U.S. Department of Energy have shown that elemental sulfur is the most desirable sulfur by-product one might obtain from these gas streams. Up until now, the only widely accepted approach to obtain elemental sulfur from SO_2 -rich streams is one of several expensive and multiple-step processes involving the Claus Process. What is needed today is a simpler, less expensive process than those that currently employ the Claus Process. The results of this project indicate that such a process may be possible.

A number of systems incorporating Claus technology are currently in operation. As mentioned, these processes are expensive, but they are competitive today. It is obvious that a new process achieving comparable results, but with lower capital and operating costs, would be indeed attractive market-wise. This is believed to be the case with the new technology. The new technology offers the following expected advantages over Claus or modified-Claus plants:

1. Lower capital costs because fewer catalyst or sorbent beds are required.
2. Lower capital costs because no pretreatment or special compositional-control equipment is necessary to achieve and maintain a constant $\text{SO}_2\text{:H}_2\text{S}$ ratio.
3. Lower operating costs because no catalysts or very inexpensive catalysts are used with the new technology.
4. Lower maintenance costs because fewer equipment items are involved and operations are carried out at atmospheric pressure.

Energy is an important operating cost both for Claus plants and for the new technology. To reduce energy costs, heat recovery and reuse should be considered in the design of facilities for both processes.

The natural gas cost for regeneration should be relatively small in comparison with total SO_2 removal costs. Calculations indicate that about 11,000 cu ft of natural gas would be required to regenerate sorbent that removes a ton of SO_2 from flue gases. This equates to a natural gas cost of about \$30 per ton of SO_2 removed.

The speed at which the new technology can be commercialized will depend on: (1) how quickly the small problems relating to new technology can be solved (These problems include identifying a suitable system to extract and consolidate on a very large scale the small yellow sulfur particles that are produced in the process) and (2) how fast the new technology can be scaled-up and demonstrated. Once these steps are completed, the new technology should be ready for the market.

CONCLUSIONS & RECOMMENDATIONS

Conclusions

On the basis of the results of this project, the following conclusions can be made:

1. During the regeneration of Sorbtech's SO_2 -saturated sorbents, it is possible to obtain as much as 77 percent direct conversion of SO_2 to elemental sulfur if temperatures are controlled well and a suitable sulfur condensing and collecting device is installed in the system.
2. The direct conversion rate of SO_2 to elemental sulfur can be increased significantly by recycling back through the sorbent bed the product gas after elemental sulfur removal. Three to four cycles are required to achieve essentially 100 percent conversion of the SO_2 to elemental sulfur. Regeneration efficiency, however, may decrease with recycling.
3. The direct conversion rate of SO_2 to elemental sulfur can be increased to 90 percent by increasing the system pressure to 20 atm and passing the product gas through a bed of special catalyst developed by Research Triangle Institute. Increasing the system pressure alone will improve conversion yields, but only marginally.
4. Magnesium oxide, alone or in combination with vermiculite or perlite, will act as a catalyst in converting SO_2 in a gas stream containing a reducing gas to elemental sulfur. Conversions occur only at temperatures above 650°F at atmospheric pressure. Elemental sulfur yields as high as 74 percent are possible with a single pass of gas through a catalyst bed.

Recommendations

Recycling of the product gas through the sorbent-regeneration system after elemental-sulfur removal, or perhaps, cycling the product gas through one or more additional beds of sorbent or MgO , appears to be the most suitable approach to achieve complete conversion of SO_2 to elemental sulfur. Most runs in the experimental program were performed on batch samples of saturated sorbent. It is important now that the process be scaled up in size to be more meaningful and that saturated sorbent be processed on a continuous basis. For this reason, it is recommended, as a next step:

1. That the recycling approach be scaled-up and demonstrated on a small pilot-plant scale, processing sorbent continuously, and
2. That the approach of passing the product gas through additional beds of sorbent or MgO to eliminate residual H_2S and SO_2 be examined, as an alternate to recycling.

In the recycling approach, methods by which CO_2 and other gases may be separated from H_2S and SO_2 in the tail gas and then discarded, should be investigated.

Also, if a single-pass system becomes practical and some H_2S remains in the tail gas, approaches to eliminate this H_2S should be examined. In particular, the oxidation of the tail gas and the return of the oxidized gas to the original gas stream (as is done in the Wellman-Lord process) should be considered. This approach might also be considered for a recycling strategy, should traces of H_2S in the tail gas be a problem.

*Budget Removed
at*

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APPENDICES

APPENDIX A

SUMMARY OF DATA AND RESULTS FOR SELECTED TEST RUNS

Quartz System Runs

Run No.	Atmosphere	<u>Gas</u>		Pressure (psig)	Sorbent Wt.Loss (%)	Results
		Flow Rate (sscm)	Max.Temp (°C)			
27	CH ₄ (100%)	75	716	atm.	26.0	Heavy yellow sulfur collected. Collected 77% S, 13% SO ₂ , 6% H ₂ S, 4% lost.
30	CH ₄ (100%)	100	719	atm.	26.7	
32	CH ₄ (100%)	50	754	atm.	26.5	Sulfur 1st seen at 690°C.
33	CH ₄ (100%)	50	825	atm.	N.M.	Pure MgSO ₄ yielded S at 760°C.
36	CH ₄ (100%)	100	726	atm.	N.M.	MgSO ₄ on Vermiculite yielded S at 690°C.
39	CH ₄ (100%)	100	743	atm.	N.M.	Pure MgSO ₃ yielded S at 490°C.

Stainless Steel System Runs - Single Pass

7	N ₂ (100%)	500	979	15	22.3	No yellow sulfur seen.
8	CH ₄ (100%)	500	865	15	28.9	Sulfur observed.
9	CH ₄ (100%)	500	868	300	28.2	Heavier S observed.
12	CH ₄ (100%)	500	871	15	28.5	Sulfur observed.
14	CH ₄ (100%)	50	895	15	28.2	Bright yellow sulfur observed.
15	Air (100%)	100	908	15	25.2	Black & green deposits.
16	N ₂ (100%)	100	949	15	26.6	Green liquid turned yellow on drying.
17	CH ₄ (100%)	100	> 1093	300	32.1	Temperature runaway; black liquid product; only trace of sulfur.
19	2% CH ₄ in N ₂	250	682	6	18.2	Green liquid turned yellow on drying.
20	20% CH ₄ in N ₂	250	678	8	16.4	Green liquid turned yellow on on drying.
29	CH ₄ ; then N ₂ (100%)	100	772	290	28.3	Yellow sulfur and black carbon.
42	50% CH ₄ in N ₂	200	745	115	23.6	Yellow sulfur observed.
45	50% CH ₄ in N ₂	200	758	290	29.0	Black turned yellow on drying.
38	CH ₄ (100%)	100	734	atm.	27.1	Sulfur observed.

APPENDIX A

SUMMARY OF DATA AND RESULTS FOR SELECTED TEST RUNS

Stainless Steel System Runs - Recycling

<u>Run No.</u>	<u>Atmosphere</u>	<u>Gas</u>		<u>Pressure</u> <u>(psig)</u>	<u>Sorbent</u> <u>Wt.Loss</u> <u>(%)</u>	<u>Results</u>
		<u>Flow Rate</u> <u>(sscm)</u>	<u>Max.Temp</u> <u>(°C)</u>			
21	CH ₄ :N ₂ (0.8:1)	500	827	18	25.6	Sulfur observed.
22	CH ₄ :N ₂ (0.8:1)	500	668	18	17.6	Sulfur observed, but sm amount.
23	CH ₄ :N ₂ (0.6:1)	500	770	18	24.4	Heavy sulfur observed.
24	CH ₄ :SO ₂ :N ₂ (0.07:0.17:2)	500	718	18	N.M.	No sorbent - CH ₄ & SO ₂ wi react to form CO ₂ , but no elemental S.
25	SO ₂ :N ₂ (0.09:2)	500	758	18	N.M.	No sorbent - SO ₂ level decreased and CO ₂ was formed.
26	Air	500	671	11	N.M.	No sorbent - Large amount of S in system burned to SO ₂ .
28	11% CH ₄ in N ₂	500	692	20	19.3	Two low temp; only small S amount.
31	75% CH ₄ in N ₂	500	724	18	44.8	New sorbent sample fr Edgewater. Sulfur observed but much C.
37	CH ₄ (100%)	500	728	25	25.6	Sulfur observed.
35	12% CH ₄ in N ₂	500	735	18	21.1	Sulfur observed.

N.M. - Not Measured

APPENDIX A

SUMMARY OF DATA AND RESULTS FOR SELECTED TEST RUNS

RTI Catalyst Runs - Using Two Beds in Series

<u>Run No.</u>	<u>Atmosphere</u>	<u>Gas</u>		<u>Pressure</u> <u>(psig)</u>	<u>CH₄-to-</u> <u>SO₂</u> <u>Ratio</u>	<u>Results</u>
		<u>Flow Rate</u> <u>(sscm)</u>	<u>Catalyst</u> <u>Temp(°C)</u>			
10	100% CH ₄	500	678	300	—	RTI Catalyst broke down due to excess CH ₄ . No sulfur produced.
13	100% N ₂	500	817	300	—	

RTI Catalyst Runs - Using Bottled Gas and One Reactor Bed

43	3.8% SO ₂ 7.4% CH ₄ Balance N ₂	3240	657	269	2:1	50.6% conversion to elemental S, 47.9% conversion to H ₂ S, 1.4% COS, 0% remaining as SO ₂ .
46	6% SO ₂ 12% CH ₄ Balance N ₂	1710	350	300	2:1	1.4% conversion to elemental S, 0% conversion to H ₂ S, 0% COS, 98.6% remaining as SO ₂ .
47	6% SO ₂ 12% CH ₄ Balance N ₂	1710	500	300	2:1	29.0% conversion to elemental S, 0% conversion to H ₂ S, 0% COS, 71.0% remaining as SO ₂ .
48	6% SO ₂ 11% CH ₄ Balance N ₂	770	665	300	2:1	12.8% conversion to elemental S, 70.4% conversion to H ₂ S, 14.8% COS, 2.0% remaining as SO ₂ .
49	6.4% SO ₂ 6.4% CH ₄ Balance N ₂	1710	665	300	1:1	89.8% conversion to elemental S, 9.0% conversion to H ₂ S, 0.9% COS, 0.4% remaining as SO ₂ .
50	6% SO ₂ 12% CH ₄ Balance N ₂	1710	665	217	2:1	22.6% conversion to elemental S, 74.0% conversion to H ₂ S, 2.0% COS, 1.3% remaining as SO ₂ .
51	6.4% SO ₂ 6.4% CH ₄ Balance N ₂	1400	665	217	1:1	53.9% conversion to elemental S, 46.0% conversion to H ₂ S, 0% COS, 0.1% remaining as SO ₂ .
52	6% SO ₂ 12% CH ₄ Balance N ₂	1705	665	115	2:1	5.2% conversion to elemental S, 93.1% conversion to H ₂ S, 0% COS, 1.8% remaining as SO ₂ .

APPENDIX B

EXAMPLES OF GAS-COMPOSITION HISTORIES FOR FIVE TEST RUNS

During each test run, the reactor temperature, system pressure, and inlet and outlet gas flow rates were recorded. Gas samples were taken with a syringe through a septum port and the composition of CO_2 , H_2S , O_2 , N_2 , CH_4 , CO and SO_2 in the system was determined with a gas chromatograph. A computer spreadsheet was used to analyze these data. A summation of the milligram moles of each species leaving the reactor plus the milligram moles of each species in the system at the end of the test was used to perform an overall material balance for each test run.

A test run using the stainless steel system lasted from 90 to 120 minutes because the reactor required 45 to 60 minutes to reach the desired regeneration temperature. Gas samples were taken approximately every five minutes throughout each run.

Figure B-1 shows the composition of the gas exiting the stainless steel reactor system during a test run in which 100 sccm of CH_4 was passed through a 50 gram bed of saturated MagSorbent, as it was heated. From the analysis of the MagSorbent before and after regeneration and from the overall material balance, 23.4 percent of the sulfur in the saturated sorbent came off as SO_2 , 1.5 percent as H_2S , 55.4 percent as elemental sulfur condensed in the sulfur trap, 6.5 percent remained with the sorbent and 13.2 percent was unaccounted for. If it is assumed that the 13.2 percent was elemental sulfur that condensed in the tubing leading to the sulfur trap, then the overall elemental sulfur yield was 68.6 percent.

From Figure B-1, it can be seen that much of the SO_2 was evolved before the reactor reached 690°C , the temperature at which elemental sulfur begins to be produced. The stainless steel reactor could not be heated rapidly. Rapid heating, however, could be achieved with a quartz reactor. The quartz reactor could be inserted into an oven at the proper temperature. Also, all parts of the quartz reactor could be dried in a desiccator and weighed to determine the mass of elemental sulfur which condensed in the reactor and tubing.

Figure B-2 shows the composition of the gas exiting the quartz reactor during a test run where 100 sccm of CH_4 was passed through a 21.86g bed of saturated sorbent, after the quartz reactor was inserted into an oven at 716°C . In this run, a gas sample was taken every minute for 21 minutes. From the analysis of the MagSorbent before and after regeneration and from the overall material balance, 12.8 percent of the sulfur in the saturated sorbent came off as SO_2 , 6.0 percent as H_2S , 77.3 percent as elemental sulfur, 3.4 percent remained with the sorbents, and 0.5 percent was unaccounted for. A good accounting of all reaction products was possible with the quartz reactor apparatus.

Figure B-3 shows the composition of the gas in the stainless steel reactor during a recycling run. Initially, the system contained enough CH_4 so that there was four times more CH_4 in the system than sulfur in the MagSorbent. The gas was recycled at a flow rate of 500 sccm and the system was heated to 827°C . At this flow rate, 10 minutes was required to recycle all of the gas

in the system once through the reactor. After four recycles at a reactor temperature above 720°C, none of the sulfur initially in the sorbent came off as SO₂, 4.7 percent came off as H₂S, 10.5 percent was collected in the condenser, and the remainder coated the insides of tubing and other system components and was unaccounted for. If all the unaccounted-for sulfur was elemental sulfur that condensed in the tubing, then the elemental sulfur yield was 74.2 percent.

Figure B-4 shows the composition of the gas exiting the stainless steel reactor during a test run where 100 sccm of CH₄ and 100 sccm of N₂ were passed through 50.46g of saturated sorbent at 115 psig (8.8 atm). Of the initial sulfur present in the saturated sorbent, none came off as SO₂, 43.3 percent as H₂S, 24.6 percent as elemental sulfur, 29 percent remained with the sorbent and 3.1 percent was unaccounted for. If the unaccounted-for sulfur was elemental sulfur that condensed in the tubing, then the elemental sulfur yield for this run was 27.7 percent.

Figure B-5 shows the effect that different operating temperatures have on the exit gas composition when a gas stream containing 6 percent SO₂, 12 percent CH₄ and 82 percent N₂ is passed through a 50cm³ bed of the RTI catalyst at a system pressure of 300 psia. The exit gas was analyzed several times while that catalyst temperature was held steady at 350°, 500° and 665°C. The exit gas was not analyzed during the transitions to a higher temperature. The concentration of SO₂ and H₂S in the exit gas was used to calculate the percent conversion of SO₂ to elemental sulfur. Figure B-6 compares the percent conversion of SO₂ to H₂S and elemental sulfur at 350°, 500° and 665°C.

Stainless Steel Reactor

100 sccm CH₄, 0 psig

Figure B-1. Data-Stainless Steel Reactor Run

Quartz Reactor

50 sccm CH₄

Figure B-2. Data-Quartz Reactor Run

Recycle Run

2:1 CH₄ to Sulfur Ratio

Figure B-3. Data-Recycling Run

Pressurized Run

115 psig, 100 sccm CH₄

Figure B-4. Data-Pressurized Reactor Run

Figure B-5. Exit Gas Compositions During Exposures to an RTI Catalyst at Different Temperatures

Figure B-6. The Effects of Catalyst Temperature on RTI Catalyst Performance

APPENDIX C

EQUIPMENT LAYOUT AND REACTOR DRAWINGS

Figure C-1 shows the overall layout of equipment in the stainless-steel system employed in the project. It should be noted that this system was modified several times during the project. Figure C-2 is a drawing of the principal reactors that were used to hold sorbent and catalyst beds. Early in the project, the internal components of the sulfur condenser were replaced with glass wool, which proved to be more effective in capturing sulfur that reached the condenser.

Figure C-1. Equipment Layout Design

CHECKED:
 REVIEWED:
 REVIEWED:
 REVIEWED:

SANITECH. INC.

DWG. NO.: EPA03-B4
 DWG. DATE: 24 JUL 92
 PROJECT NO.: EPA-03
 DRAWN BY: B W NELSON
 SCALE: NONE

REVISION NO.: 1
 27 JUL 92
 DRAWING OF

NOTE: THREE (3) REACTOR
UNITS TO BE MADE.

Figure C-2. Regenerator and Catalyst Reactors

SORBTech

CHECKED:
REVIEWED:
REVIEWED:
REVIEWED:

DWG. NO.: EPA03-B6

REVISION NO.:

DWG. DATE: 28 JUL 92

PROJECT NO.: EPA-03

DRAWN BY: B V NELSON

SCALE:
DRAWING OF