

FINAL REPORT

Test of TDA's Direct Oxidation Process for Sulfur Recovery

by

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Contract No. DE-AC26-01BC15333

Period of Performance: March 13, 2001 – February 29, 2004

January 2005

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Abstract

This project was a Phase III pilot plant test of TDA's gas sweetening process done under realistic conditions. TDA Research Inc successfully completed the test at Whiting Petroleum's Sable San Andreas Gas Plant. The feed was approximately 228,000 standard cubic feet per day (SCFD) of gas that contained approximately 60 vol% CO₂, 20 vol% CH₄ and 10 vol% C₃+ and higher hydrocarbons. The feed was associated gas from CO₂ flooding operations carried out on Whiting's oil wells. The gas is collected and piped to the Sable gas plant where it is normally flared. We sited our pilot plant in line with the flare so that we could remove the hydrogen sulfide (H₂S) prior to flaring. The average H₂S concentration in the gas during the field test was 7341 ppm. The selectivity of our process for converting H₂S into elemental sulfur was essentially 100% and the catalyst converted 90% of the H₂S into sulfur and water (the remaining 10% of the H₂S passed through unconverted). Importantly, no catalyst deactivation was observed for over the course of the 1000+ hour test. Minimal (ca. 10-15 ppm) of SO₂ was formed during the test. Approximately 3.6 tons of elemental sulfur was recovered from a total inlet of 3.9 tons of sulfur (as H₂S). The total amount of SO₂ released from the plant (taking into account flaring of the unconverted 10% H₂S) was 0.86 tons. This amount of SO₂ is much lower than the normal 8 tons that would have been emitted if all of the H₂S were flared over the time of the pilot plant test. The pilot plant was simple to operate and required much less operator intervention than is typical for a new unit being commissioned. Our operator (Mr. Eugene Peeples) has more than 30 years of experience operating commercial scale liquid redox sulfur recovery processes and in his opinion, TDA's Direct Oxidation pilot plant is easier to operate than liquid systems. The ease of use and low capital and operating costs of TDA's Direct Oxidation process makes it an attractive technology to be used where traditional sulfur recovery technologies are too expensive (e.g. small to medium sized plants). Currently, TDA's direct oxidation process has been exclusively licensed to SulfaTreat, and is being offered commercially under the trade name SulfaTreat-DO[®]. We anticipate that the first plant will be installed in 2005.

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1. Executive Summary

This project was a Phase III pilot plant test of TDA's gas sweetening process done under realistic conditions. TDA Research Inc successfully completed the test at Whiting Petroleum's Sable San Andreas Gas Plant. The feed was approximately 228,000 standard cubic feet per day (SCFD) of gas that contained approximately 60 vol% CO₂, 20 vol% CH₄ and 10 vol% C₃₊ and higher hydrocarbons. The feed was associated gas from CO₂ flooding operations carried out on Whiting's oil wells. The gas is collected and piped to the Sable gas plant where it is normally flared. We sited our pilot plant in line with the flare so that we could remove the hydrogen sulfide (H₂S) prior to flaring. The average H₂S concentration in the gas during the field test was 7341 ppm.

The selectivity of our process for converting H₂S into elemental sulfur was essentially 100% and the catalyst converted 90% of the H₂S into sulfur and water (the remaining 10% of the H₂S passed through unconverted). Importantly, no catalyst deactivation was observed for over the course of the 1000+ hour test. Minimal (ca. 10-15 ppm) of SO₂ was formed during the test.

Approximately 3.6 tons of elemental sulfur was recovered from a total inlet of 3.9 tons of sulfur (as H₂S). The total amount of SO₂ released from the plant (taking into account flaring of the unconverted 10% H₂S) was 0.86 tons. This amount of SO₂ is much lower than the normal 8 tons that would have been emitted if all of the H₂S were flared over the time of the pilot plant test.

The pilot plant was simple to operate and required much less operator intervention than is typical for a new unit being commissioned. Our operator (Mr. Eugene Peebles) has more than 30 years of experience operating commercial scale liquid redox sulfur recovery processes and in his opinion, TDA's Direct Oxidation pilot plant is easier to operate than liquid systems. The ease of use and low capital and operating costs of TDA's Direct Oxidation process makes it an attractive technology to be used where traditional sulfur recovery technologies are too expensive (e.g. small to medium sized plants). Currently, TDA's direct oxidation process has been exclusively licensed to SulfaTreat, and is being offered commercially under the trade name SulfaTreat-DO[®]. We anticipate that the first plant will be installed in 2005.

2. Objectives

The objective of the project was to perform a field test of TDA's gas sweetening process under realistic conditions. In TDA's process, hydrogen sulfide gas (H₂S) is catalytically oxidized (using air) to form elemental sulfur and water. The elemental sulfur is formed in the vapor phase and is subsequently condensed downstream of the catalytic reactor. The small amount of water vapor produced by the process remains in the sweet gas exiting the sulfur condenser. Prior to the field test, all testing had been done at the laboratory scale where synthetic mixtures of feed gas were tested. While laboratory scale testing is sufficient for catalyst development and initial studies, field-testing at the pilot plant scale is necessary to determine how the catalyst will perform when exposed to the wide variety of hydrocarbons, mercaptans and other impurities commonly found in natural gas and associated gas streams. Also, by testing at the pilot scale, temperature gradients within the catalyst bed, non-uniformities in gas flow, and engineering scale issues are addressed that cannot be investigated at the much smaller laboratory scale. This report describes the results from a successful field test of TDA's process to remove H₂S from an associated gas stream that contained approximately 8000 ppm of H₂S, 60% CO₂, 20% CH₄, and 20% ethane, propane, butane and traces of higher hydrocarbons. We also report an

economic analysis based on the field test data that shows that TDA's process (which we refer to as Direct Oxidation or DO) is much less complex and lower in cost than competing commercial technologies.

3. Project Description and Background

Direct Oxidation (DO) uses a proprietary patented catalyst that was developed by TDA. The catalyst oxidizes hydrogen sulfide (H_2S) to elemental sulfur and water, with minimal if any SO_2 formation. DO catalyst development and demonstration at the bench scale was done with funding from the U.S. Department of Energy under Phase I and Phase II SBIR grants. After the laboratory scale demonstration of DO, the Gas Technology Institute (GTI) and Unocal funded the design and fabrication of a \$1,300,000 pilot plant to test the process at close to commercial scale. The pilot plant was originally scheduled to be field tested on a Unocal offshore



Figure 1. Location of Plains, TX.

platform in Mobile Bay, located off the coasts of Mississippi and Alabama. Unocal anticipated that the offshore platform would be subjected to more stringent SO_2 emissions regulations and saw the Direct Oxidation process as a way to achieve increased gas production while reducing SO_2 emissions. The environmental regulations on the platform however, were not increased and Unocal decided not to spend the additional money needed to install and operate the plant.

After extensive negotiations and exploring possible sites for the field test, TDA completed construction of the pilot plant and installed it at a new site in Texas (Figure 1) with funding from the Department of Energy (this project). The plant was installed at Whiting Petroleum Company's Sable San Andreas gas plant outside Plains, TX and the field test was completed in 2003. The Whiting gas plant is located about 5 miles northwest of Plains, TX, which is about equidistant between Midland, TX, Lubbock, TX and Hobbs, NM (Figure 1). The gas that was used as the feed for the pilot plant contained about 8000 ppm of H_2S , 20% methane and about 60% CO_2 with the balance being ethane, propane and butanes. This gas is associated gas obtained after CO_2 flooding of Whiting oil wells.

The pilot plant test was completely successful and the data have enabled us to evaluate the economics of the process and compare our process to currently available small-sulfur recovery technologies such as various liquid redox processes. Having successfully demonstrated the TDA DO process, potential customers will be much more confident about the reliability of the technology. As a result of the field test and favorable economics (discussed later), TDA has licensed the process to SulfaTreat, a business unit of M-I. The process is currently being offered commercially under the trade name SulfaTreat-DO[®]. Westfield Engineering is the primary contractor for detailed engineering design and for subcontracting the construction of commercial SulfaTreat[®]-DO units. Westfield is a well-established company with a long and successful history in designing and building sulfur recovery units, including Claus and SulFerox units. The first commercial SulfaTreat-DO[®] plant is expected to be installed some time in 2005.

4. Description of the Pilot Plant

4.1. Overview

The pilot plant tests were conducted at the Whiting Petroleum Sable San Andreas Gas Plant located near Plains, TX. Startup and shakedown of the plant was done during the first week of October in 2002. This included installation of the main electric gas heater, tuning controllers and establishing the flow of process gas. Figure 2 is a photograph of the site where the pilot plant was eventually located, and Figure 3 is a photograph of the plant during its initial installation (before being heat traced and insulated).

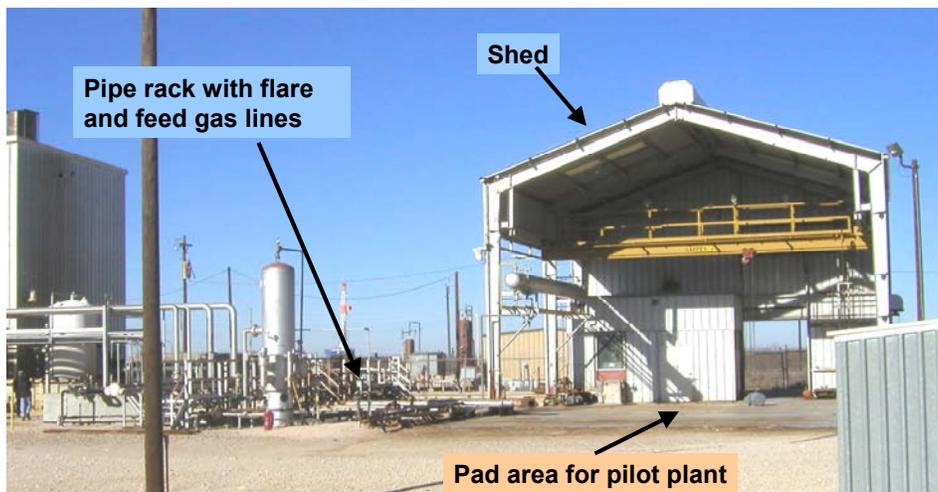


Figure 2. Photograph of Whiting Sable San Andreas gas plant before installation of the TDA sulfur pilot plant.

The pilot plant has five main “subsystems:” 1) the feed gas system which includes the gas manifold, a liquid knockout (KO) drum and a reactor bypass; 2) the catalytic reactor where H_2S oxidation into sulfur vapor and water takes place; 3) a sulfur condenser; 4) a sulfur u-trap and heat traced liquid sulfur handling piping; and 5) sulfur storage bins.

The liquid knockout (KO) drum is located upstream of the catalytic reactors and its function is to remove and hydrocarbon liquids that may be present in the gas stream. This minimizes the amount of hydrocarbon vapors to which the catalyst can be exposed. The KO drum is equipped with level controls, automatic drainage, a flare bypass etc. The P&ID for the inlet piping and KO are shown in Figure 4.

Figure 5 is the process and instrumentation diagram for the reactor and condenser sections of the plant. Radian International did the detailed engineering (and made the drawings) early in the history of the project under subcontract to TDA. Two fixed bed reactor vessels are shown in Figure 5 because in the original plan was to place the plant on a Unocal offshore platform in Mobile Bay. In this application, Unocal wanted the option of drying the gas over a desiccant prior to desulfurization by catalytic oxidation. The second "reactor" was to be used to contain the desiccant bed. In the field test at Plains, this vessel was empty and was valved off.

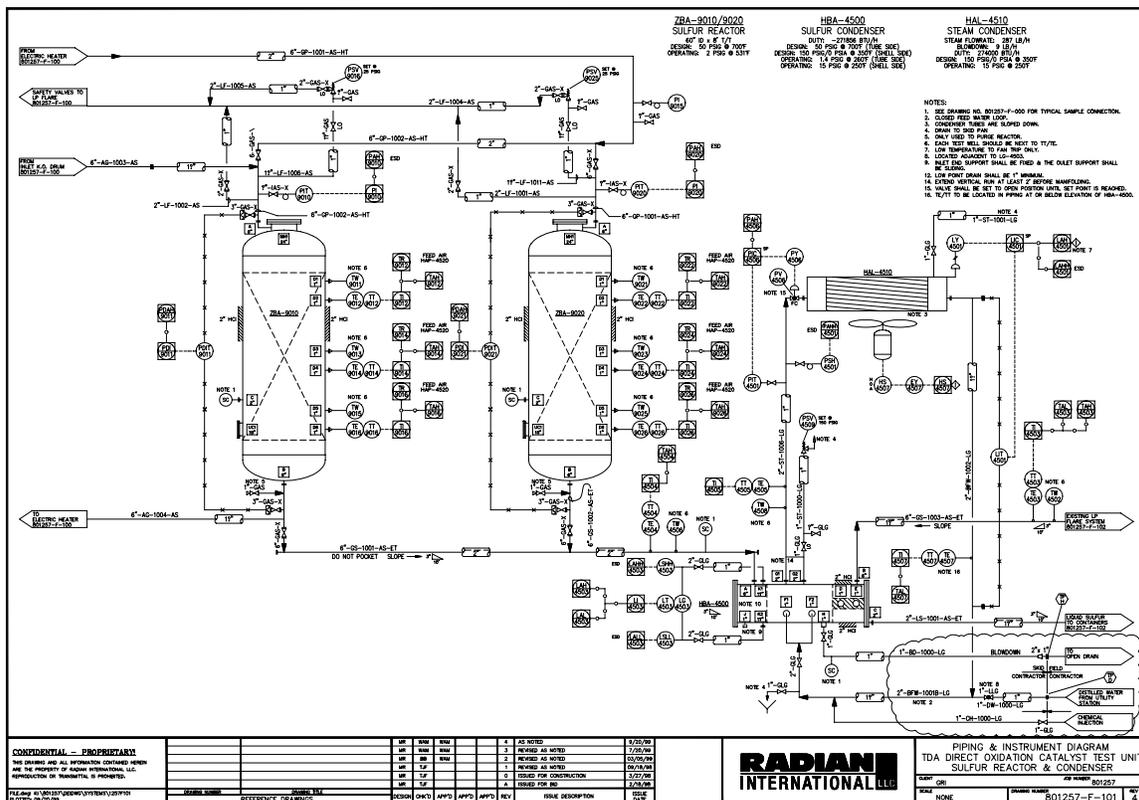


Figure 5. P&ID for reactor and sulfur condenser section of the pilot plant.

One of the reactor was used and was filled with catalyst. The reactor is made of carbon steel and is lined with refractory (described in detail in Section 4.2). The sulfur condenser (described bed in Section 4.3) is a heat exchanger with a half-height tube sheet that is half filled with boiling water. The process gas exiting the catalytic reactor (that contains the elemental sulfur vapor) flows tubeside. The temperature of the sulfur condenser tubes is maintained by controlling the steam pressure on the shell side (boiling water) of the condenser. Because the saturation pressure of the shellside steam determines the saturation steam temperature, controlling the steam pressure controls the temperature of the sulfur condenser. The condenser is mounted slightly off horizontal so that liquid sulfur exiting the condenser will flow downhill and into a U-tube type trap and into sulfur bins (Figure 6).

The gas exiting the sulfur condenser was at the sulfur condensation temperature, which was 250-260°F in the field test. Liquid sulfur exits the bottom of the bonnet of the condenser and sweet gas exits at the top. Because this gas is at 250-260°F and is in equilibrium with the liquid sulfur leaving the condenser, the sweet gas contains about 75 ppm of sulfur vapor. Unless this vapor is removed, it will plug downstream piping. To prevent plugging of Whiting's flare header,

we installed about 75 ft of 6 inch SCH40 carbon steel pipe between the pilot plant and Whiting's flare header. This pipe was unheated so the 75 ppm sulfur vapor would condense in it leaving only clean sulfur free gas to pass into the flare header. This is discussed in detail in Section 4.5. Commercially available technologies exist for condensing sulfur vapor, and because the focus of the pilot plant test was proving the catalytic oxidation of H₂S to sulfur with a real gas stream, we installed the long pipe because this option was quick and inexpensive. In a commercial application, we will use a heated coalescing filter (as recommended by Westfield Engineering).

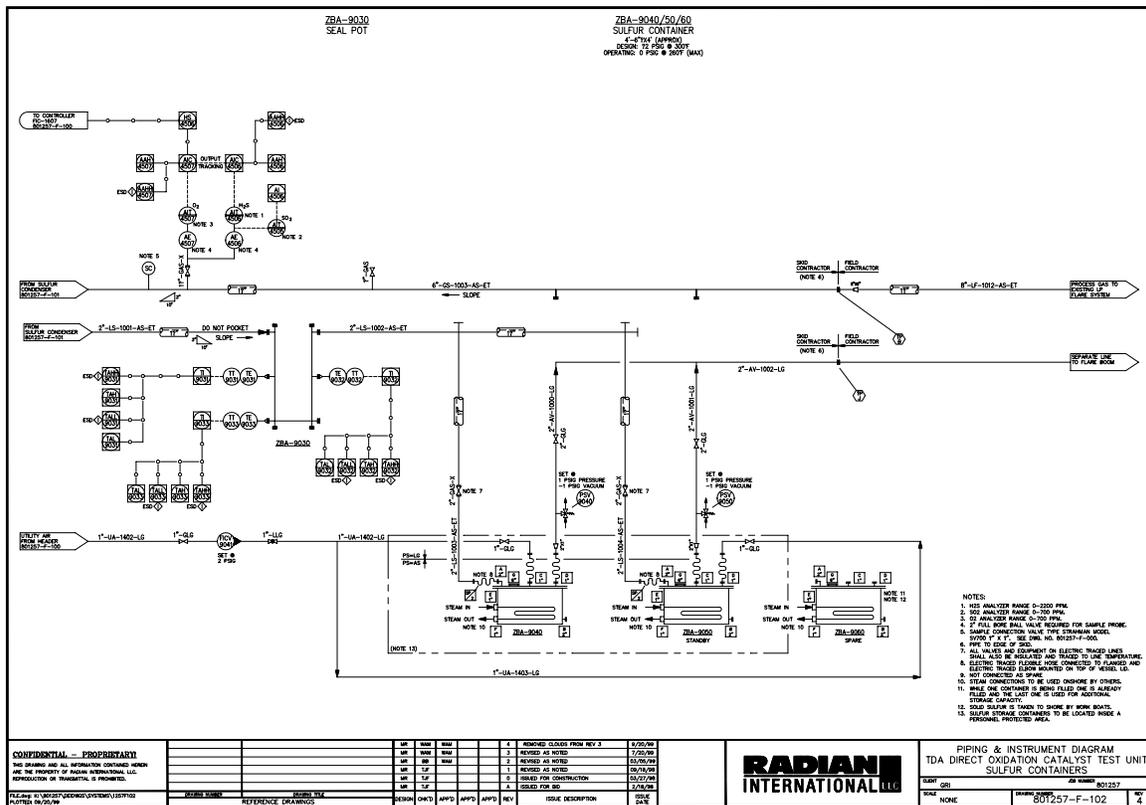


Figure 6. P&ID for sulfur collection "pots."

The sulfur condenser and sulfur storage vessels are located on the bottom level of the unit (the unit has two levels to save space as per the original off-shore requirement). Liquid sulfur exiting the sulfur condenser passed through a U-trap that acts as a sulfur seal and into collection bins. The bins are purged with air to degas the sulfur (Figure 6). Unfortunately, during the field test, we found that the bins badly leaked gas and were therefore not used. In addition, the heat tracing on the P-trap was not sufficient to maintain the proper liquid level. Therefore, we removed sulfur directly from the heat exchanger bonnet, storing the sulfur in 55 gal drums.

Several contractors were involved in the installation and start up of the unit. The primary contractors were Huzyk Energy Management, Denver, CO; Wilbanks Engineering, Houston, TX; and Ref-Chem, Odessa, TX. Sandra Huzyk of Huzyk Energy Management was the project manager for installing the plant and found the test site.

4.3. Sulfur Condenser

The sulfur condenser is essentially a thermosiphon “reboiler” type of heat exchanger that operates at about 265°F. At this temperature the saturation pressure of steam is about 38 psia (26 psig at the altitude of Plains, TX). By controlling the steam pressure (back pressure on the steam line from the condenser to the air fin cooler (see P&ID in Figure 5), the temperature of the boiling water inside the condenser can be controlled. Figure 9 is a schematic of how the condenser functions and Figure 10 is a close up photograph of the sulfur condenser (the gas outlet is difficult to see in the photograph – it is located on top of the bonnet at the end of the condenser just left of the water level gauge).

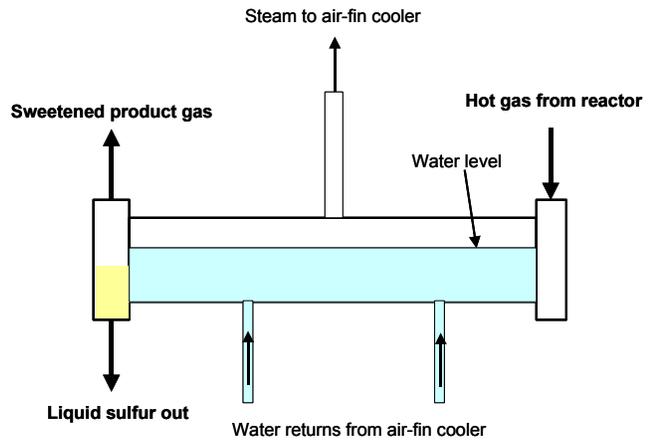


Figure 9. Schematic of sulfur condenser.

(the gas outlet is difficult to see in the photograph – it is located on top of the bonnet at the end of the condenser just left of the water level gauge).

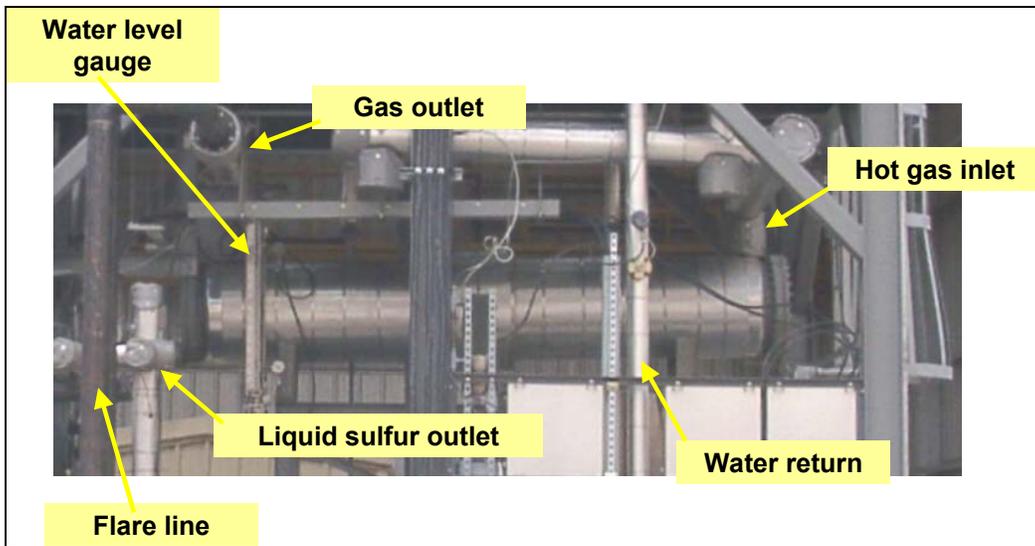


Figure 10. Photograph of sulfur condenser.

It is important to keep the sulfur condenser temperature between about 240°F (the melting point of sulfur) and 320°F (where the viscosity rises dramatically) because of the peculiar viscosity versus temperature behavior of liquid sulfur. Our typical operating temperature range is 250°F to 280°F. At these temperatures, liquid sulfur has its minimum viscosity (Figure 11). The viscosity data shown in Figure 11 below 240°F (left side) are for the supercooled liquid, which we never have in our application. Above 320°F, the viscosity of sulfur rapidly increases, eventually reaching enormous values (right side of Figure 11) when the so-called plastic or μ -sulfur exists (Tuller 1954). For example, Figure 11 shows that the viscosity of sulfur at 375°F is about 60 lb/ft-sec, which is equal to 89,000 centipoise. For comparison, the viscosity of water at room temperature is about 1 centipoise. The huge increase in viscosity of liquid sulfur with

increasing temperature above 320°F is due to the rupture of the normal eight-atom sulfur rings (Figure 12) followed by polymerization of the now linear S₈ chains. The resulting material, (called μ-sulfur) is extremely viscous. The phase change is irreversible, and therefore once μ-sulfur is formed, the equipment will plug and will require shutdown for cleaning.

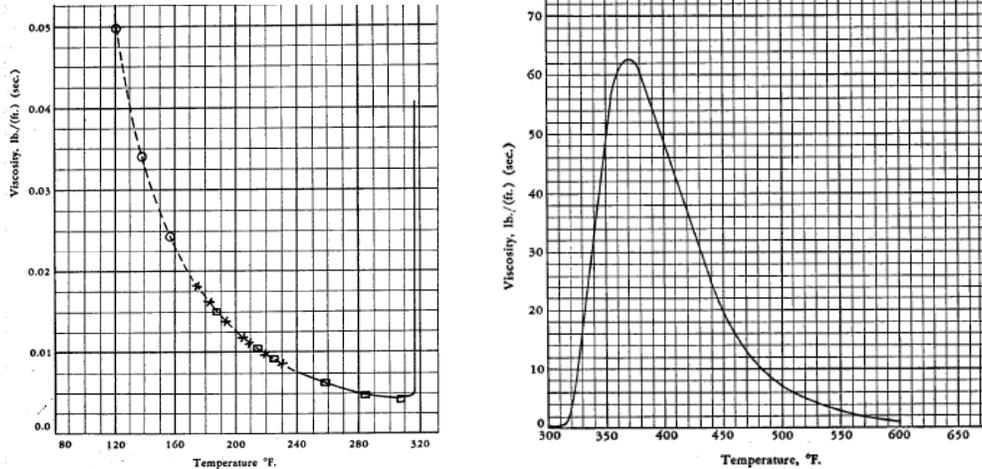


Figure 11. Sulfur viscosity at *low* temperatures (left) and *high* temperatures (right); data from the Sulfur Data Book (Tuller 1954).

The narrow temperature window (ca. 60°F) for operating the sulfur condenser requires that there be a minimal temperature gradient along the length of the condenser tubes (which are about 15 ft long). In a conventional cold-fluid-in, hot-fluid-out, counter-current type of heat exchanger, it is difficult to guarantee enough a uniform enough temperature along the length of the tubes so that overheating (making μ-sulfur) or too much cooling (sulfur solidification) can be prevented. Either situation would plug the condenser. To avoid this, the constant temperature/boiling water design was used. Figure 10 is a close-up of the condenser with the various lines labeled.

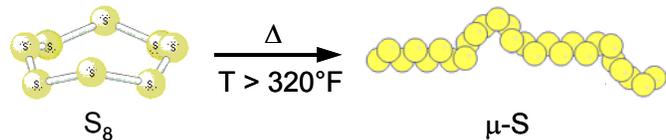


Figure 12. S₈ ring breaking and polymerization.

The sulfur condenser is a standard design and it functions essentially as a boiler with a thermosiphon. Steam generated on the shell side of the condenser (by heat transfer from the hot product gas in the tubes) flows up to an air-fin cooler where it is condensed and returned through a standpipe (see Figure 10). The height of the standpipe determines the hydraulic head that drives the water back into the shellside of the condenser. In our case this was about 15 ft of water (approx 2 psi). One problem we observed with this design during operation was that the volume of water in the standpipe was too small. This was because the return pipe was 2 inch SCH40 and there was no water reservoir. As a result, establishing natural circulation was very sensitive to the operation of the steam control valve. Too much steam pressure would blow the standpipe dry and with too little steam pressure, the steam in the condenser would drive water backwards up the standpipe into the air-fin cooler. We were able to control the loop and operate with continuous circulation, but in commercial models we will use a larger diameter standpipe so that the standpipe volume is greater.

4.4. Sulfur Bins (“Pots”)

The sulfur bins were not used in the field test because of an operational difficulty in the lines that transport liquid sulfur from the outlet of the sulfur condenser to the bins. During the test, we found several cold spots between the exit of the sulfur condenser and the bins that would plug with solid sulfur. Worse, the bins were not gas tight, which would have made using them dangerous.

Rather than shut down the plant and re-heat-trace the entire downstream liquid piping, we modified the system so that we could withdraw the liquid sulfur directly from the sulfur condenser. While not a permanent solution, this enabled us to continue the field test and was acceptable because the test was focused on the performance of the Direct Oxidation catalyst, and not how to handle liquid sulfur (which is existing technology that can be easily engineered by Westfield Engineering for commercial units).

4.5. Modification of Flare Tie-In to Prevent Sulfur Vapor Plugging

The sulfur condenser was operated at about 260°F. While the vapor pressure of sulfur at this temperature is relatively low ($P_{\text{vapor}} = 0.063 \text{ Torr} = 1.22 \times 10^{-3} \text{ psia}$; see Figure 13) the volume of gas passing over the liquid sulfur is large enough that considerable sulfur will exit the condenser as vapor. The average sour gas flow rate was about 200 SCFM. Assuming that this gas is in equilibrium with the liquid sulfur in the condenser when it exits the condenser, then the sulfur vapor concentration in the gas would be approximately 75 ppm. At this concentration and 200 SCFM, about 100 lb of sulfur would deposit in

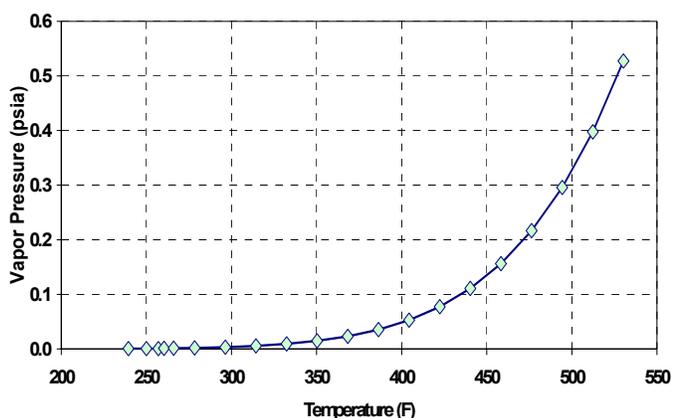


Figure 13. Sulfur vapor pressure (Daubert et al. 1997).

the piping downstream of the condenser during the test. If not removed, this sulfur would plug Whiting’s flare lines, which was unacceptable. The goal of the pilot plant test was to demonstrate that the *catalytic* technology; methods and equipment for handling liquid and vapor phase sulfur is commercially available. Thus, we needed a quick and importantly, *inexpensive* way to prevent sulfur vapor from condensing and depositing in Whiting’s flare line while permitting us to finish the field test on budget.

We investigated several options to remove the sulfur vapor before the gas reached Whiting’s flare header. Among the options considered were: 1) treating the gas with a cold water spray; 2) scrubbing the gas with a solvent; 3) plumbing the gas through a packed tower scrubber (using water) – there was an unused tower located on-site, and 4) adding a long section of unheated, large diameter, pipe section upstream of the flare heater to allow the sulfur to deposit in that line before reaching the flare header.

We chose to protect Whiting's flare line by installing 75 ft of 6 inch SCH40 pipe between the vapor outlet of the sulfur condenser and Whiting's flare header. With this length of pipe, the gas exiting the sulfur condenser was cooled to below 200°F before it entered the flare header. In addition, the pipe was not a straight run; several tees were installed (instead of elbows) to permit both cleaning (if necessary) and to create turbulence where sulfur would preferentially deposit. Several thermocouples were installed along the length of pipe so that we could check the gas temperature. In a commercial application, we would install a commercially available heated sulfur coalescing filter, but time and cost constraints prevented us from using such a device during the field test.

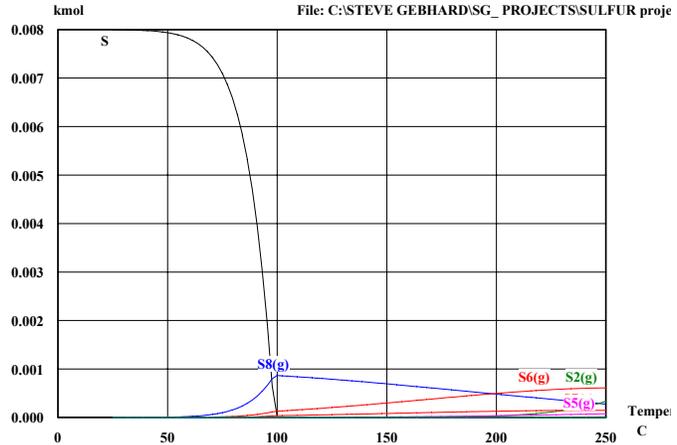


Figure 14. Composition and dew point of for 8000 ppm of sulfur vapor.

The sulfur vapor recovery line was sized by assuming that free convection around the pipe was the only mechanism that would cool the gas. Most sections of the pipe would be cooled by radiation (especially at night when the air was cool); however during the day, solar radiation would slightly heat the pipe. Ignoring radiation heat transfer simplified the analysis and more importantly, gave a pipe length that was longer than actually needed because natural convection is less efficient at cooling in this situation; "too much cooling" is not a problem.

To make the calculations even more conservative, the temperature of the air was assumed to be rather warm (80°F). The properties of air at this temperature were used in the free convection calculations. The gas inlet temperature was assumed to be 266°F and the outlet was assumed to be 230°F (10°F below the sulfur melting point). The temperature of the surface of pipe was assumed to be the average of these temperatures (248°F). For free convection from a horizontal pipe in air, the Rayleigh number (Ra) is given by Equation 1. This dimensionless number expresses the balance between thermal expansion, temperature, thermal diffusivity, viscosity, and the thickness of the convection layer of the gas in which the body being cooled is immersed (in this case air). Natural convection begins near $Ra = 2000$ and becomes turbulent for $Ra \geq 10^6$.

$$Ra = \frac{g\beta(T_{\text{surface}} - T_{\text{air}})d_{\text{pipe}}^3}{\nu_{\text{air}}\alpha_{\text{air}}}$$

Equation 1. Rayleigh number for free convection (Incropera and Dewitt 1996).

The definitions of the terms in Equation 1 are as follows: α_{air} is the thermal diffusivity of air [$k/(\rho \cdot C_p)$], β is the coefficient of thermal expansion of air ($2.725 \times 10^{-3} \text{ K}^{-1}$), g is the gravity constant (32.2 ft/s^2), and ν_{air} is the kinematic viscosity of air (taken at the average pipe wall temperature, $\nu_{\text{air}} = 1.74 \times 10^{-4} \text{ ft}^2/\text{sec}$). The value of Ra calculated from Equation 1 was found to be $Ra = 5.8 \times 10^7$ which is in the turbulent flow regime.

The Nusselt number is used to calculate a heat transfer coefficient from which the required heat transfer area can be calculated. The Nusselt number is given by Equation 2 when $10^{-5} \leq Ra \leq 10^{12}$ and in this case was $Nu = 47.9$ (using a Prandtl number for air of $Pr = 0.707$). The heat transfer coefficient under these conditions was calculated from Nu using Equation 3 and was $h_{fc} = 0.97 \text{ Btu}/(\text{ft}^2 \text{ hr } ^\circ\text{F})$. This low value of heat transfer coefficient is typical for free convection situations where values are typically 1-2 $\text{Btu}/(\text{ft}^2 \text{ hr } ^\circ\text{F})$.

$$Nu = \left[0.6 + \frac{0.387Ra^{\frac{1}{6}}}{\left[1 + \left(\frac{0.559}{Pr} \right)^{\frac{9}{16}} \right]^{\frac{8}{27}}} \right]^2$$

Equation 2. Nusselt number for horizontal pipe in free convection (Incropera and Dewitt 1996).

$$h_{fc} = \frac{k_{air}}{d_{pipe}} Nu$$

Equation 3. Heat transfer coefficient

Because the inlet and outlet temperatures are different, a log-mean temperature difference (LMTD) was used to calculate the heat transfer area required for cooling. The LMTD is given by Equation 4 and the calculated value was $LMTD = 168^\circ\text{F}$.

$$LMTD = \frac{(T_{hotIN} - T_{cold}) - (T_{hotOUT} - T_{cold})}{\ln \left[\frac{(T_{hotIN} - T_{cold})}{(T_{hotOUT} - T_{cold})} \right]}$$

Equation 4. LMTD calculation

The amount of heat to be removed is the heat needed to cool the gas from 266°F to 230°F and was $q = m \cdot C_{p,gas} \cdot (T_{hotIN} - T_{hotOUT}) = 12,190 \text{ Btu}/\text{hr}$. There is so little sulfur in the vapor (75 ppm) that its contribution to the total heat duty (via the heat of fusion) is miniscule and essentially all of cooling duty is used in removing sensible heat from the gas.

The heat transfer surface area required to remove 12,190 Btu/hr of heat from the gas given a $LMTD = 168^\circ\text{F}$ and $h_{fc} = 0.97 \text{ Btu}/(\text{ft}^2 \text{ hr } ^\circ\text{F})$ is $A = q/(h \cdot LMTD) = 75 \text{ ft}^2$. This corresponds to 43 feet of 6 inch SCH40 pipe (the schedule number is relevant because it determines the outside diameter of the pipe for a standard pipe size). To be conservative we installed 75 ft of pipe, an overdesign factor of 1.7.

The flow of gas inside the pipe is turbulent (Reynolds number = $Re = 1.1 \times 10^5$) which improves the collection efficiency of sulfur depositing on the walls of the pipe rather than being carried along as tiny solid particles in the streamlines of cool gas (as would occur if the flow were

laminar). The calculated pressure drop (assuming incompressible at these low pressures) was less than 1 psi and in practice, we observed a pressure drop of about 0.6 psi.

Figure 15 is a photograph of the pilot unit with an overlay of how the new line was attached (the actual configuration was somewhat different when installed, but the basic concept was unchanged). In the original configuration, the pilot plant was attached to Whiting's flare header (on the left side of the photograph) using 6 inch SCH40 uninsulated pipe that served as both the inlet gas bypass as well as the exit from the sulfur condenser (red line in Figure 15). If this line were to plug with condensed sulfur vapor, then there would be no way to bypass the sour gas feed at the pilot plant. In addition, this line was much shorter than 75 ft cooling line making it more likely that sulfur would reach Whiting's flare system. More alarming, sulfur powder was observed in the original line at a small sampling port located about 15 ft upstream of the header entrance, so sulfur was making it to the flare piping in the original layout.

In the new configuration, the original tie-in to the flare header remained connected but was only used for bypassing and flaring the feed gas. The tee that connected the original flare line to the exit of the sulfur condenser was disconnected and blinded, and the independent, 75 ft x 6 inch SCH40 line was attached between the exit of the sulfur condenser and the flare header (yellow line in Figure 15). Blocking valves were placed at both ends so that the line so that it could be taken out of service and cleaned if necessary. Flanged tees were used rather than elbows to facilitate cleaning and to increase turbulence in the piping to help with sulfur collection.

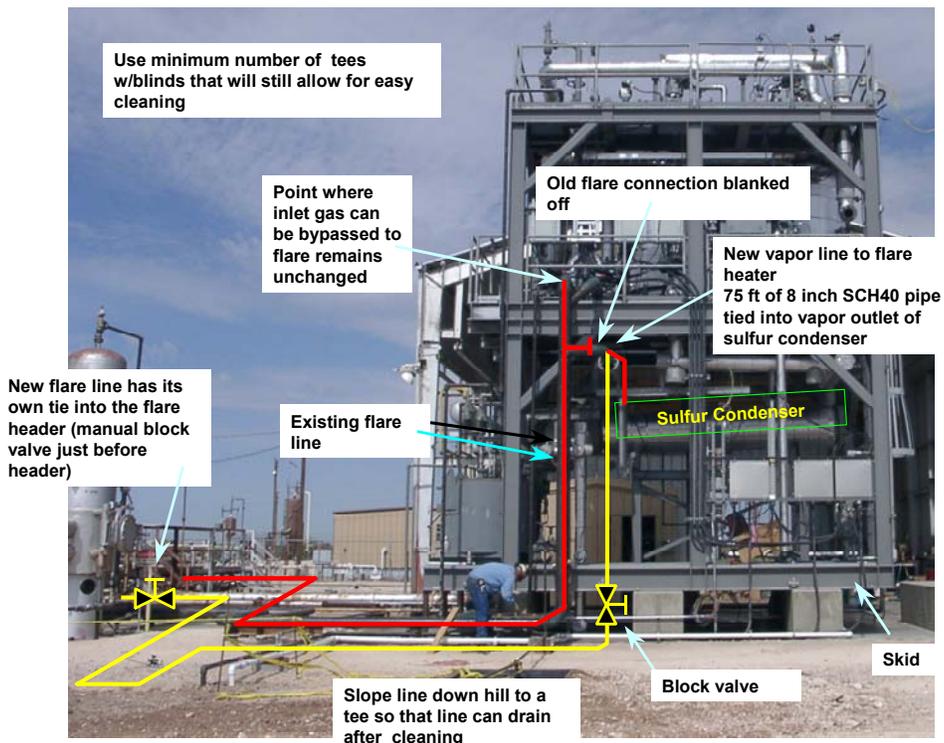


Figure 15. Photograph of pilot plant showing original flare line (highlighted in red) and planned location of new flare line (yellow).

We found that the long cooling pipe was necessary and functioned as designed. At the end of the run after the pilot plant had been shut down, about 100 lb of sulfur was removed from the flare extension when it was opened for inspection (we predicted that about 102 lb should have been deposited). Most of the sulfur was deposited close to the first tee where the concentration of sulfur vapor was highest and where the flow first impinges on a pipe tee at a right angle. Importantly, smaller amounts of sulfur were found farther downstream, as close as 20 ft upstream of the flare header; but fortunately, no sulfur made it to the flare header. Therefore, our solution for removing entrained and vapor phase sulfur from the product gas exiting the sulfur condenser worked quite well and was inexpensive to implement. For a commercial application of course, a more permanent solution such as a heated sulfur coalescing filter would be used.

5. Field Test Results

As mentioned previously, the field test was very successful. In this section we describe the results in detail in Section 5.1 we discuss the composition of the feed stream; in Section 5.2 we discuss the catalytic oxidation results and finally in Section 5.3 we compare the results of laboratory testing the catalyst used in the field test with fresh catalyst.

Whiting's Sable San Andreas Gas Plant is used to collect and flare associated gas from CO₂ oil well flooding operations. The total gas flow varied somewhat but averaged about 300,000 ft³/day (ca. 200 SCFM) and contained about 6000 to 8000 ppm of H₂S. The H₂S concentration varied from day to day, as did the total feed flow rate when different oil wells went on and off line. The main components of the gas were methane (ca. 20%), CO₂ (ca. 60%), ethane (9-10%), propane (6-7%) and butanes (ca. 3%). The gas also contained about 3000 ppm of hydrocarbon liquids as vapor in equilibrium with the liquid in the knockout drum located upstream of the feed preheater.

5.1. Feed Gas

A detailed analysis of the feed and product gases was performed on grab samples by an outside laboratory. The results of those analyses are given in Table 1.

Most of the C₅+ hydrocarbons in the gas are removed when they enter the Whiting plant using a knockout drum. In addition there is a second liquid knockout located on the pilot plant skid (Figure 4). Still the inlet gas analysis in Table 1 shows that the gas contained almost 2.8 vol% of C₅+ hydrocarbons. Under these conditions, the catalyst was continuously exposed to these hydrocarbons at the reaction temperature of 360°F (182°C) for the entire 1000+ hours of operation. After 1000 hours at sour gas flow rate of 200 SCFM, more than 13 million standard cubic feet of gas were processed and assuming that the total concentration of hydrocarbons with molecular weights higher than butane (i.e. C₅ and up) was a constant 2.8 vol%, the catalyst (1700 lb) was exposed to over 35 short tons of C₅+ hydrocarbons over the course of the test. This represents over 40 times the weight of the catalyst in higher molecular weight hydrocarbons. If these hydrocarbons had been deleterious to catalyst performance, we would have observed severe catalyst deactivation and we did not; no catalyst deactivation was observed over the 1000+ hour course of the test.

Table 1. Gas analysis from Laboratory Services, Hobbs, New Mexico (only compounds that were detected are listed).

Component	Mol% (inlet)	Mol% (outlet)	Component	Mol% (inlet)	Mol% (outlet)
H ₂ S	0.8000	0.095	cyclohexane	0.1698	0.1387
N ₂	1.5972	2.8794	n-heptane	0.0976	0.0703
CH ₄	17.6750	17.7853	methylcyclohexane	0.0517	0.0378
CO ₂	58.6264	58.6625	toluene	0.0218	0.0136
Ethane	8.7006	8.5607	n-octane	0.0053	0.0032
Propane	6.4640	6.2643	ethyl benzene	0.0029	0.0024
isobutane	0.9798	0.9228	p and m xylene	0.0029	0.0027
n-butane	2.3841	2.3154	o-xylene	0.0007	0.0004
isopentane	0.7385	0.7115	n-nonane		0.0022
n-pentane	0.7715	0.7453			
cyclopentane	0.0105	0.012	Mercaptans (ppm)	101.00	20
2-methylpentane	0.1512	0.1354			
3-methylpentane	0.1345	0.1203	Specific gravity (air =1)	0.45	
n-hexane	0.2658	0.2353	Gross BTU/CF dry	731.00	
methylcyclopentane	0.1375	0.1144	Gross BTU/CF wet	718.00	
benzene	0.2107	0.1692			

5.2. Catalytic H₂S Oxidation Test Results

Figure 16 shows the sour gas flow rate and the inlet H₂S concentration during the 1000+ hour test. During the first 150 hours of the test (6 days), the flow rate was about 280 SCFM but the H₂S concentration was low (6000 ppm). There was a significant change in the sour gas flow rate during the pilot plant test as different wells were brought on and off line. The H₂S concentration gradually increased over the first 300 hrs (12.5 days), but after that the concentration was essentially constant at about 8000 ppm. The inlet and outlet H₂S, SO₂ and O₂ concentrations were measured using GasTech stain tubes. The stain tubes provided a simple and reliable method to determine the H₂S, SO₂ and O₂ concentrations without the problems we encountered with gas chromatography (GC). The hydrocarbon vapors present in the gas (few thousand ppm) contaminated the GC column making the GC gas analysis results erratic. We also analyzed for O₂ to determine if there was any oxygen slipping through the catalyst bed; none was detected. The oxygen concentration was also measured using an electrochemical analyzer. Because H₂S will damage the electrochemical cell, this instrument was protected from H₂S and hydrocarbons by a series of molecular sieve, and Purafil (KMnO₄ impregnated alumina) beds. The molecular sieve traps hydrocarbons and the potassium permanganate (KMnO₄) oxidized H₂S to sulfate (which remains adsorbed on the Purafil beads).

Figure 17 shows the H₂S conversion as a function of time on stream during the pilot plant direct oxidation test. The H₂S conversion was approximately 90% for the duration of the test and did not decrease with time. Because we kept the catalyst temperature constant during this time, the constant H₂S conversion indicates that no catalyst deactivation had occurred in 1000+ hours of the test (this was confirmed by testing the spent catalyst from the pilot reactor in the laboratory with fresh catalyst as discussed in Section 5.3). The reason that the conversion remains constant even through the flowrate of feed gas is changing, is because the reaction is mass transfer limited. The lack of deactivation is especially significant considering that the catalyst was exposed to over 40 tons of C₅+ hydrocarbon vapors and even larger quantities of methane,

ethane and butanes during the test without any adverse affects. The resistance of the catalyst to deactivation when exposed to hydrocarbon vapors is consistent with our earlier laboratory investigations where we contaminated our synthetic gas with toluene, xylene and hexane. No deactivation was observed in the laboratory studies and the hydrocarbons simply passed over the catalyst bed unreacted without coking or fouling the catalyst.

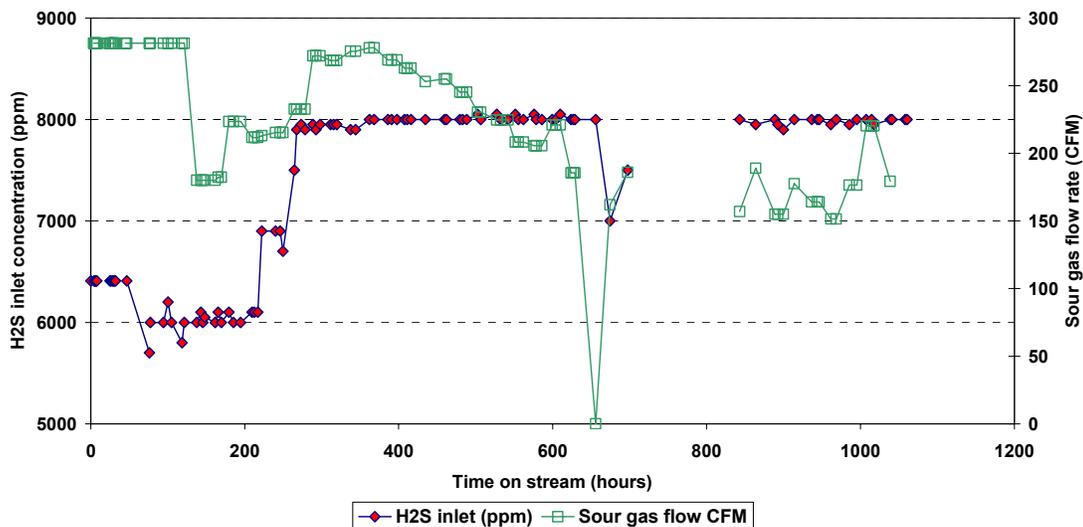


Figure 16. Sour gas flow rate and inlet H₂S concentration during field test.

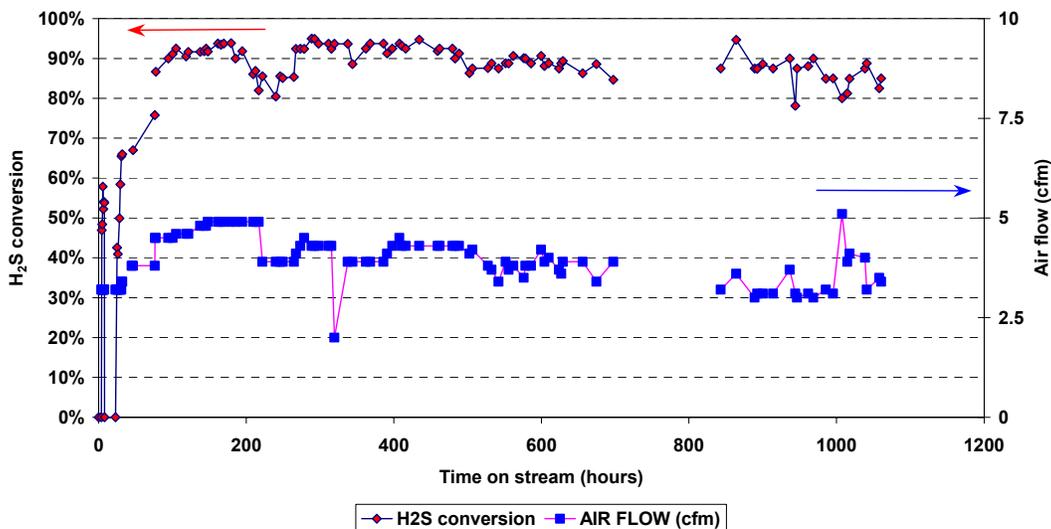


Figure 17. H₂S conversion and air flow rate during field test

Figure 17 also shows the airflow as a function of time during the pilot plant test. The airflow is included in Figure 17 to show how the fluctuations observed in the H₂S conversion (although small) were due to our intentionally varying the airflow (and consequently the O₂/H₂S ratio). This was done to determine the affect of changing O₂/H₂S ratio on H₂S conversion and the catalyst's selectivity to sulfur and SO₂. When the airflow was held constant, the H₂S conversion was constant (see for example the data between 400 and 500 hours in Figure 17).

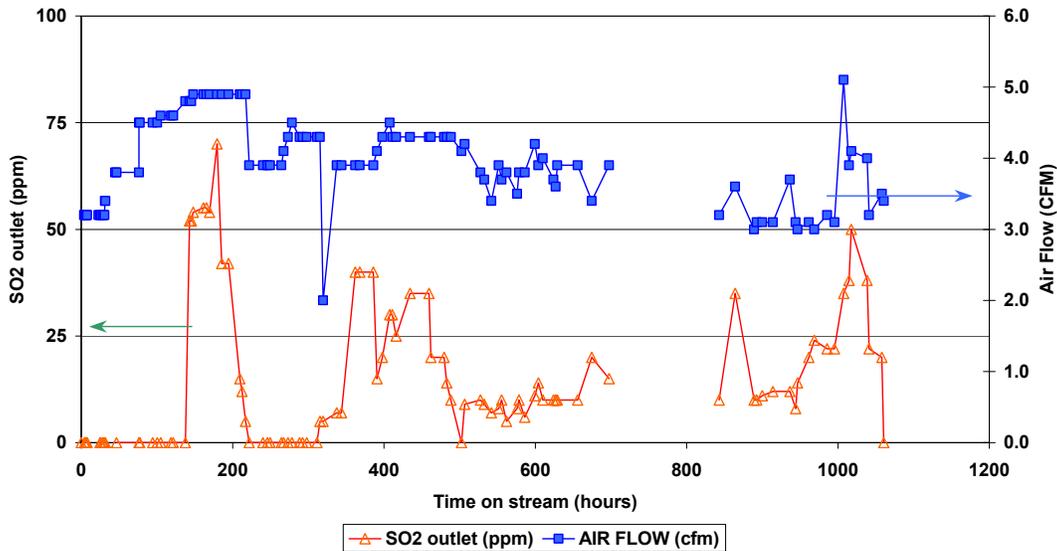


Figure 18. SO₂ outlet concentration and air flow during field test.

Figure 18 shows the concentration of SO₂ in the outlet (product gas) during the test. The airflow is shown again to illustrate that by varying the airflow, we can control the concentration of SO₂ in the outlet gas. Note that the SO₂ concentration tracks with the airflow quite well. In any case, the SO₂ concentration never exceeded 75 ppm and was typically about 12 ppm when operating under conditions that produced a high selectivity for elemental sulfur (e.g. the time between 200 and 700 hours in Figure 18).

Table 2 compares the sour gas flow rates, sulfur production and SO₂ emissions when the pilot plant is removing an average of 89% of the sulfur in the gas. Because the pilot plant processes

Table 2. Production summary for field test at time = 1000 hours.

	Field Test Case	No Sulfur Recovery Case
Average sour gas flow rate (SCFM)	228	228
Average air inlet flow rate (SCFM)	3.5	0
Average H ₂ S inlet (ppm)	7341	7341
Average H ₂ S outlet (ppm)	790	0
Selectivity to elemental sulfur	100%	N/A
Average sulfur yield	89%	0
Total sulfur inlet (lbs)	7953	7953
Total sulfur inlet (tons)	3.9	3.9
Total sulfur recovered (lbs)	7097	0
Total sulfur recovered (tons)	3.6	0
Total sulfur flared as SO ₂ (tons)	0.86	7.95

all of the gas that would normally be flared by Whiting, and the selectivity to elemental sulfur is practically 100%, the reduction on SO₂ emissions from the Whiting flare is equal to the mass of sulfur (as H₂S) converted. For example, operation for over 1000 hours with an average H₂S concentration of 7341 ppm, a total of 3.9 tons of sulfur enters the plant. The DO process converts 89% of that into elemental sulfur. The unconverted (11%) H₂S is burned in the flare in the usual manner producing SO₂. Over the course of 1000 hours, our process discharged about 0.8 tons of SO₂ including the amount of unconverted H₂S that is flared. For comparison, if

the feed gas were directly flared, then almost 8 tons of SO₂ would have been emitted. This comparison illustrates one advantage of TDA's direct oxidation process, that even at modest low sulfur recovery efficiencies (ca 90%), very large reductions in SO₂ emissions are possible.

The Direct Oxidation process also can be used if higher sulfur recovery efficiencies are required. In that case, a tail gas treatment unit (TGTU) such as liquid redox, amine with recycle or scavengers (e.g. SulfaTreat[®]) is placed downstream of the DO unit to remove the unconverted H₂S.

5.3. Laboratory Testing of Fresh and Used Catalyst

One concern we had was that because the H₂S conversion remained constant at about 90%, but that over time the flow rate of feed gas slowly decreased (Figure 16), was that this effect might have masked catalyst deactivation. To test this possibility, we tested used catalyst taken from the pilot plant reactor after the end of the pilot run, with fresh catalyst. The tests were done in the laboratory under carefully controlled conditions of temperature, gas composition and gas flow rate.

Figure 19 shows the results from the laboratory testing of fresh (blue bars) and used (red bars) catalyst. The used catalyst was removed from the pilot plant reactor after the 1000+ hour test. The samples were taken from three radial locations at three depths in the catalyst bed. The used catalyst tested in the laboratory was an average sample made by grinding and sieving a portion of each sample from the reactor and then mixing equal amounts of these samples together.

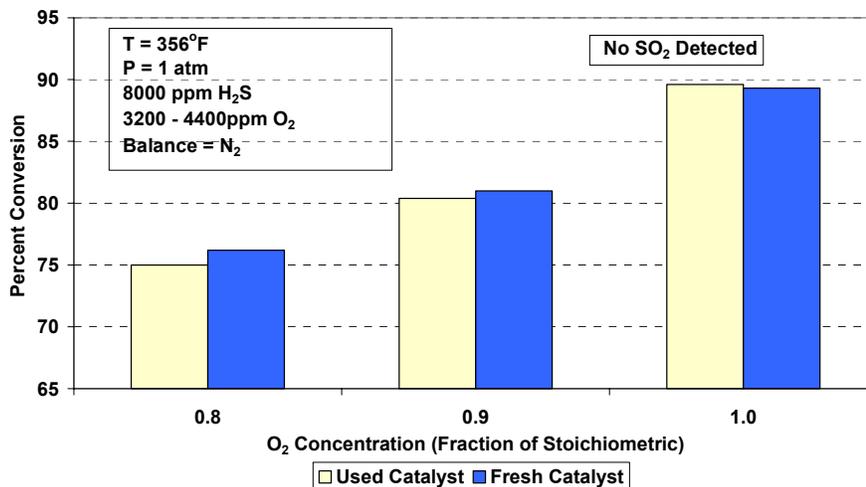


Figure 19. Laboratory testing of fresh (dark bars) and used (light bars) catalyst.

The reaction conditions for the laboratory test were essentially identical to those used during the field test. The temperature was 356°F (180°C), the space velocity was approximately 600 ft³_{gas}/ft³_{catalyst}/hr, and the gas contained 8000 ppm of H₂S. The oxygen concentration was varied between 3200 ppm and 4400 ppm and data were obtained at O₂/H₂S ratios that corresponded to 80%, 90% and 100 % of the stoichiometric amount of O₂ need for the reaction $H_2S + \frac{1}{2} O_2 \rightarrow S + H_2O$ (i.e. O₂/H₂S = 0.40, 0.45 and 0.5 respectively). Fresh catalyst (from the same batch that was used when filling the reactor on the pilot plant) was tested under identical conditions. Both the fresh and the used catalysts exhibited essentially identical performance indicating that

there was no deactivation of the catalyst while it was in service in the pilot plant test. The reason that the conversion remained at 90% while the gas flow dropped was that, at least at the lower flow rates, the H₂S oxidation reaction was mass transfer limited.

6. Technology Transfer

6.1. Technology/Information Transfer

TDA has successfully completed the Phase III pilot plant testing project and has shown that the TDA catalyst developed for the direct oxidation of H₂S into sulfur and water is active, selective and long-lived. The test lasted for more than 1000 hours (42 days) desulfurizing 100,000–300,000 SCFD of CO₂-rich, associated gas that contained 8000 ppm of H₂S. The overall H₂S conversion was approximately 90% during the course of the test, and minimal SO₂ was formed. Currently, TDA's direct oxidation process has been exclusively licensed to SulfaTreat, a business unit of MI and is being offered commercially under the brand name SulfaTreat[®]-DO. The original pilot plant (now in Plains, TX) will be disassembled and shipped back to GTI in Chicago, IL by June 2005.

6.2. Public Relations and Marketing

TDA's H₂S oxidation process has been licensed to SulfaTreat as the SulfaTreat-DO[®] process (SulfaTreat[®]-“Direct Oxidation”). SulfaTreat is a business unit of M-I, LLC. M-I is a Smith-International/Schlumberger Company. SulfaTreat is actively marketing the technology, and the first commercial unit will be installed in 2005. SulfaTreat is in final negotiations with the customer. That unit will process approximately 1 million SCFD of natural gas containing about 1% H₂S.

The SulfaTreat-DO[®] process is being advertised in appropriate trade magazines and has generated many inquiries. Figure 20 is a copy of the advertisement published in *Oil, Gas and*

Direct Oxidation Technology & Process Unveiled For Large-Scale 1 to 50 Tons Per Day Sulfur Removal

SULFATREAT RECENTLY SIGNED an exclusive commercialization agreement with TDA Research Inc. (Wheaton Ridge, Colorado) for its new **Direct Oxidation technology**.

This new technology is targeted at applications with sulfur removal requirements between 1 and 50 tons per day of sulfur. Target markets include natural gas production, landfill gas, and synthesis gas. SulfaTreat is seeking end user customers, as well as sublicense opportunities for this new technology.

Developed and patented by TDA Research Inc., the new technology oxidizes H₂S into elemental sulfur and water at temperatures between 300° and 500° F. and at pressures from ambient to 1,000 psi. The Direct Oxidation process includes a new catalyst that is selective to sulfur production and stable in the presence of hydrocarbons. The process removes sulfur from natural gas, associated gas, amine off-gas, and synthesis gas feedstocks.

TDA completed a pilot scale demonstration test using an associated gas stream at Whiting Oil and Gas Corporation's Sable San Andreas Plant near Plains, Texas.

In TDA's Direct Oxidation process, H₂S is oxidized to sulfur and water in the presence of a solid catalyst:

$$\text{H}_2\text{S} + 0.5\text{O}_2 \rightarrow \text{S} + \text{H}_2\text{O}$$

More than 90% of the H₂S is converted into elemental sulfur in a single stage. The sulfur is recovered in a condenser located downstream of the Direct Oxidation reactor. The gases exiting the sulfur condenser contain some unreacted H₂S and may contain small amounts of SO₂. A polishing process removes these gases to meet the required specification. The TDA catalyst operates in the presence of such hydrocarbons as methane, ethane, propane, BTEX (benzene, toluene, ethylbenzene, xylene) or in the presence of synthesis gas (CO/H₂) using either air or oxygen as the oxidant. The hydrocarbons, CO and H₂ are not oxidized in the process.

Demonstration test: The pilot scale demonstration test was conducted at Whiting's Sable San Andreas Gas Plant, and is shown here during installation. The sulfur condenser and sulfur storage vessels are located on the bottom level. The adiabatic fixed bed oxidation reactor is located on the upper level, above the sulfur condenser. The catalyst was in the form of 1/8-in. x 1/4-in. pellets.

Whiting's plant collects and flares associated gas from oil production. The total gas flow rate varied from 300 to 800-Mscfd and contained between 6,000 and 8,000-ppm of H₂S. The gas consisted of about 20% methane and 60% CO₂ along with moderate amounts of ethane (9-10%), propane (6-7%), and butanes (ca. 3%), as well as about 3,000-ppm of high hydrocarbon liquids (C₅+) and about 100-ppm of mercaptans.

The field test demonstrated the process could remove more than 90% of the H₂S as sulfur along with about 80% of the mercaptans. More than 1,200 hours of testing was conducted at the site. The demonstration test and subsequent laboratory tests were conducted on the used catalyst. The used catalyst had the same activity and selectivity as fresh catalyst indicating that exposure to the hydrocarbons in the feed had no detrimental effect on catalyst performance.

Economic analyses carried out by independent parties indicate TDA's patented process offers significant cost savings over liquid redox or amine/Claus/tail gas treatment sulfur recovery processes.

The Direct Oxidation process by itself recovers greater than 90% of the sulfur from the H₂S in the feed. In order to get >99% sulfur recovery, a polishing process is placed downstream of the Direct Oxidation process for tail gas treatment. This polishing step can be a scavenger like SulfaTreat[®], or a sorbent, liquid redox, biological, or amine-based process. This combination of Direct Oxidation followed by a tail gas treatment will prove to be the low cost option in the 1 to 50 tons per day sulfur recovery scale.

This approach is attractive because it combines the low cost bulk sulfur removal of the Direct Oxidation technology with the high efficiency scavenger, sorbent or liquid redox polishing step to provide an overall recovery efficiency of greater than 99%. Such a process provides an affordable alternative to conventional liquid redox and amine/Claus/tail gas treatment processes for the same tonnages of sulfur recovered.

Westfield Engineering of Houston will carry out the detailed engineering and fabrication of the Direct Oxidation plants.

SulfaTreat, A Business Unit of M-I L.L.C., A Smith / Schlumberger Company, 17998 Chesterfield Airport Road, Suite 215, Chesterfield, MO 63005.

Circle 1 On Reader Service Card Or Go To www.oilgas.com For Free Direct Oxidation Information

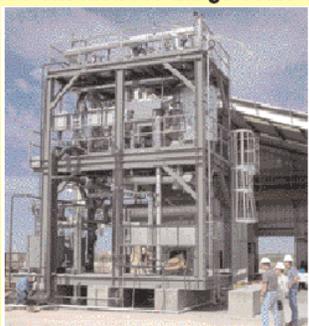


Figure 20. Advertisement in *Oil, Gas and Petrochemical Equipment* for the SulfaTreat-DO[®] process.

Petrochemical Equipment and Figure 21 is a copy of the advertisement for the process that appeared in the March 2004 issue of *Hydrocarbon Processing* (Gulf Publishing Co).

**Are you caught between
the SULFATREAT process
and liquid redox?**

If so, we'd like to hear from you. We have a new process that may be just what you need to end the dilemma.

This process, which we are simply calling "Direct Oxidation," is not for every application. It is meant for gas streams with one-half to 25 tons of sulfur per day, and up to 20,000 ppm of hydrogen sulfide (30,000 ppm if the gas is used for fuel gas or sent to flare.)

What's so special about Direct Oxidation?

Well, to start with, it has the simplicity of the SULFATREAT process. It uses no liquid chemicals of any kind, and the H₂S is eliminated as the gas passes downward through a fixed bed of a proprietary, dry catalyst. But unlike SULFATREAT technology, Direct Oxidation can economically remove large amounts of sulfur — up to 25 tons per day.

On the other end of the spectrum, Direct Oxidation eliminates the long-tolerated operating problems of liquid redox plants; problems such as sulfur plugging and pump failures are no longer encountered because there are no liquids or moving parts.

The by-product of Direct Oxidation is marketable, bright yellow sulfur, rather than the "dirty," brown sulfur caused by contamination from the liquid redox chemical.

So, if you want the simplicity of the SULFATREAT process and the capacity of liquid redox, call us now toll-free at 1-800-726-7669 and we'll send you an article about the process and give you an immediate cost estimate of a Direct Oxidation system for you.

SULFATREAT is a business unit of M-I LLC
27108 Chestfield Airport Road, Suite 225 - Chestfield, MO 63006
Toll-free: 1-800-726-7669 - Tel: 636-532-2232 - Fax: 636-532-2704
Select SE at www.hydrocarbonprocessing.com/HS

Figure 21. Ad for direct oxidation in March 2004 issue of *Hydrocarbon Processing* (scanning and slight rotation of the image caused blurring of the text – the original is crisp and clear).

7. Conclusions

The objective of the project was to perform a field test of TDA's gas sweetening process under realistic conditions. TDA Research Inc has successfully completed this Phase III pilot plant test at Whiting Petroleum's Sable San Andreas Gas Plant. The average gas flow during the test was 228,000 SCFD. The gas contained significant quantities of CO₂ (ca 60%), CH₄ (ca 20%) and C₃+ hydrocarbons (ca 10%). The average H₂S concentration was 7341 ppm. No catalyst

deactivation was observed for over the course of the 1000+ hour test. Minimal (ca 10-15 ppm) of SO₂ was formed during the test.

The selectivity to sulfur was essentially 100% and the catalyst converted 90% of the H₂S into sulfur and water. Approximately 3.6 tons of sulfur (in the elemental form) were recovered from a total inlet of 3.9 tons of sulfur (as H₂S). The total SO₂ emissions from the plant (taking into account flaring of the unconverted H₂S) were 0.86 tons. This much lower than the normal 8 tons of SO₂ that would have been generated with straight flaring of the feed gas over the same period of time. The pilot plant was simple to operate and required much less operator intervention than is typical for a new unit coming on line. It was also considerably easier to operate than a typical liquid redox system. The chief plant operator during the test was Mr. Eugene Peebles. Mr. Peebles has more than 30 years of experience operating commercial scale liquid redox processes, and thus he is highly qualified to judge the ease of operation of the Direct Oxidation process compared to existing commercial liquid redox sulfur recovery processes. Mr. Peebles indicated that the Direct Oxidation process was considerably easier to run from an operator's standpoint. The ease of use and low capital and operating costs of the TDA Direct Oxidation process makes this an attractive technology to be used where traditional sulfur recovery technologies are too expensive. As a result, the Direct Oxidation process is well suited for sulfur recovery in small to medium sized plants. Currently, TDA's Direct Oxidation process has been exclusively licensed to SulfaTreat, and is being offered commercially under the trade name SulfaTreat-DO[®].

SulfaTreat is a division of M-I, which is a joint venture between Schlumberger and Smith International. SulfaTreat's flagship H₂S scavenging technology is the market leader and is currently used to treat nearly 3 trillion cubic feet of gas annually from more than 1,000 applications in more than 20 countries worldwide. The SulfaTreat Company has become a widely recognized and trusted leader in the industry serving international markets of Energy, Environmental and Water Treatment. A new SulfaTreat system is installed somewhere in the world nearly every business day.

With SulfaTreat's background and expertise, they are ideally suited for marketing the Direct Oxidation process. In SulfaTreat-DO[®], the Direct Oxidation process can be operated as a stand alone unit, or used upstream of a gas polishing step such as the SulfaTreat[®] scavenger, a liquid redox process or other sulfur recovery technologies. In addition to stand-alone units, the SulfaTreat-DO[®] process can be used to "unload" existing sulfur recovery systems, thus providing customers with a low cost simple alternative to increasing the sulfur handling capacity of their equipment.

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January 2005