

**SCREENING PROCESSES FOR REMOVAL OF H₂S FROM
ENHANCED OIL RECOVERY CO₂ STREAMS**

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ABSTRACT

This paper discusses work done for Denbury Resources (Denbury) to build a spreadsheet tool to rapidly provide screening level capital and operating costs for hydrogen sulfide (H₂S) removal and (as applicable) sulfur recovery from naturally occurring, H₂S-contaminated carbon dioxide (CO₂) sources used as an enhanced oil recovery (EOR) agent.

The H₂S-contaminated source CO₂ streams have pressures in the 800-1600 psig range. Three H₂S removal technology size niches are considered in this work: 1) the large-scale niche -- selective amine followed by conventional Claus-based sulfur recovery was chosen as the representative technology, 2) the medium-scale niche -- iron-based liquid redox processes were chosen as the representative technology, and 3) the small-scale niche -- iron-based solid and triazine-based liquid H₂S scavengers were chosen as the representative technologies. Operating pressure limits were placed on the chosen representative technologies based upon given known commercial installations and available data. The capital and operating costs of compression were calculated and added to the total capital and operating costs for those options requiring depressurization of the inlet CO₂ stream prior to H₂S removal. General capital and operating cost correlations were developed for each process option and integrated into a predictive spreadsheet tool.

The paper discusses the representative technologies chosen and the reasons for selection. The paper also summarizes some of the various sources of capital and operating cost data that were collected from process licensors and from literature sources and how that data are used to create a predictive (within bounds) capital and operating cost screening spreadsheet tool. The paper gives experimental capacity data for liquid and solid H₂S scavengers applied to ~1500 psi CO₂ streams. The paper also reviews the predicted boundaries of the small-scale niche, the medium-scale niche, and the large-scale niche for H₂S removal from CO₂ in terms of LTPD of elemental sulfur. Although created for a CO₂ producer, the data and methodologies could be useful for others who need to achieve a screening level understanding of H₂S removal and sulfur recovery cost from streams containing very high CO₂ partial pressures.

I. Introduction

Denbury Resources (Denbury) is a growing independent oil and natural gas company. The company is the largest oil and natural gas operator in Mississippi, owns the largest reserves of CO₂ used for tertiary oil recovery east of the Mississippi River, and holds significant operating acreage in Louisiana, Alabama, and Southeast Texas. The

company's goal is to increase the value of acquired properties through tertiary recovery operations, combined with a combination of exploitation, drilling and proven engineering extraction practices.

Denbury's CO₂ source field, Jackson Dome, located near Jackson, Mississippi was discovered during the 1970s while being explored for hydrocarbons. This significant source of CO₂ is the only known one of its kind in the United States east of the Mississippi River. Mississippi's first enhanced oil recovery project began in the mid 1980s in Little Creek Field following the installation of Shell Oil Company's Choctaw CO₂ pipeline. The 183-mile Choctaw Pipeline (now referred to as the NEJD Pipeline) transported CO₂ produced from Jackson Dome to Little Creek Field. While the CO₂ flood proved successful in recovering significant amounts of oil, commodity prices at that time made the project unattractive and Shell later sold their oil fields in this area, as well as the CO₂ source wells and pipelines.

Currently ~800 MMscfd of CO₂ is produced from Jackson Dome. Source well operating pressures range from 800 to 1600 psig with relatively low concentrations of H₂S. Denbury desires to maintain low concentrations of H₂S (<10 ppmv) for industrial contract obligations and EOR operations. For continued operations when H₂S concentrations in excess of 10 ppmv are encountered, Denbury has been investigating the use of H₂S removal technologies.

Denbury contracted Trimeric Corporation to provide a screening-level guidance spreadsheet tool for evaluating the costs of H₂S removal for CO₂ source and recycle streams. H₂S in CO₂ pipelines and in gathering redistribution piping at EOR sites is a health and safety concern for Denbury. The purpose of the spreadsheet tool was to help Denbury rank projects relative to one another but not necessarily provide budget-quality data for specific projects. The spreadsheet tool allows Denbury to determine the answers to project specific questions such as: 1) Is the capital cost for a specific project likely to be \$10 million or closer to \$100 million?; 2) Do the total treating costs for H₂S removal add around \$0.05/Mscf of cost to the CO₂, or is it more like \$0.50/Mscf?; 3) Is the technology that would be used likely in the small-scale niche, the medium-scale niche, or in the large-scale niche? The answers to these types of questions allow Denbury to rapidly screen specific projects for economic viability. If a project is found to be attractive, Denbury can then examine the H₂S removal technology choice more carefully to see if a project is economically viable, including expanding beyond the representative technologies considered in the spreadsheet tool.

Denbury is interested in technologies that can treat high-pressure CO₂ streams with CO₂ contents ranging from 70 to 100 mole%, H₂S contents ranging from trace ppmv to 30 mole%, and CO₂ rates ranging from ~50 to 200 MMSCFD. These CO₂ conditions limit the available technology choices and/or the conditions at which H₂S removal occurs, as discussed later in the paper.

The H₂S removal screening spreadsheet tool estimates costs for removing H₂S from the CO₂ gas to low ppmv levels (less than ~10 ppmv). In all scenarios considered,

the CO₂ product stream is recompressed back to original pressure, if necessary. The CO₂ property correlations that are used in the spreadsheet tool are suitable for a range of pressures and temperatures of 250 psig to 1400 psig and 110 to 150°F, respectively.

Size Ranges of H₂S Removal and Sulfur Recovery

The work considered three size ranges of H₂S removal and sulfur recovery technology:

- Large-scale niche – selective amine/Claus was chosen as the technology representative of this size niche. Amine/Claus combinations are normally best suited for greater than ~15 LTPD of elemental sulfur when used in typical natural gas applications.
- Medium-scale niche -- iron-based liquid redox processes were chosen as representative technology of this size niche. Redox processes are normally considered best suited for ~0.1 to ~15 LTPD of elemental sulfur production when used in typical natural gas applications.
- Small-scale niche -- iron-based solid and triazine-based liquid H₂S scavengers were chosen as representative technologies. In natural gas applications, H₂S scavengers are normally considered economical for less than ~0.1 LTPD of elemental sulfur.

The treating ranges (up to 0.1 LTPD, 0.1 LTPD to ~15 LTPD, and greater than ~15 LTPD of elemental sulfur) are approximate ranges for typical natural gas applications and are primarily based on economics; each technology type can be used well outside the range. For example, technologies that are typically in the large-scale niche have been used for much lower sulfur tonnages; the authors know of a few amine/Claus applications in natural gas or oil refining service that operate as low as 3 LTPD. Further, as will be described later in this paper, the sulfur capacity ranges may be quite different when considering H₂S removal from streams that are predominantly CO₂, particularly when the need to recompress the CO₂ exists.

Generally speaking, the reasons technologies fall into these niches has to do with the total treating costs. A substantial portion of the total treating cost for technologies in the large-scale niche (e.g., amine/Claus, physical solvent/Claus, bulk distillation approaches, etc.) is usually due to high capital costs. Operating costs per unit of sulfur removed tend to be low. However, even if the operating costs were zero, the high capital costs of these types of technologies may not be justified for small-scale gas streams and/or for minor amounts of sulfur. Thus, these technologies tend to be best suited to sizable sulfur tonnages.

Conversely, the total treating cost for technologies in the small-scale niche (e.g., H₂S scavengers) is dominated by high operating costs (i.e., chemical costs) per unit of sulfur removed, while capital costs are very low. This combination of high operating

costs but low capital tends to be attractive when an application only has a few pounds of sulfur per day that need to be removed.

With the technologies in the medium-scale niche (e.g., liquid redox), total treating costs tend to be more evenly split between capital and operating (e.g., chemical) costs. Thus, this class of technologies tends to fit in a range between the technologies in the small-scale niche and the large-scale niche.

While a number of technology variations exist within each niche, screening costs were only developed for a basic flow scheme for the specific technology chosen as the representative of each niche.

Screening Spreadsheet Tool Background

The costs used in the screening spreadsheet tool were obtained from reliable literature sources and vendor data. Capital and operating cost correlations were developed to make the spreadsheet tool “predictive” for gas compositions greater than 70% CO₂, operating pressures from 250 psig to 1400 psig, operating temperatures from 110 to 150°F, and CO₂ flow ranges from 50 MMscfd to 200 MMscfd.

The gas conditions (i.e., temperature, pressure, composition and flow rate) are entered into the spreadsheet tool. Warnings within the spreadsheet tool indicate whether the information entered is out of acceptable range or is known to pose concerns related to the economic outcome for a particular case. Default values for utilities and chemicals are built into the spreadsheet tool but may be adjusted by the user if they are significantly different for a particular application. The Chemical Engineering Plant Cost Index is used to adjust capital costs to a particular date. See Table 1 for an example input page from the spreadsheet tool; shaded items in the “Value” column can be entered by the user.

The spreadsheet tool can be used for screening purposes only. Detailed information should be obtained from process licensors and/or a qualified engineering company prior to finalizing technology selection and field implementation.

The remainder of the paper discusses the H₂S removal technologies that are considered in the spreadsheet tool and how the capital and operating costs were developed. The spreadsheet tool output and limitations are discussed, followed by several case studies to show how the spreadsheet tool can be used to screen technology options.

Table 1. Example Screening Spreadsheet Tool Input Page

Gas Data			
Gas Data	Units	Value	Warnings on Input Data
Gas flow	MMscfd	50	
Gas temperature	F	120	
Gas pressure	psig	1400	
Outlet gas H ₂ S content	ppmv	10	
Basic Feed Gas Composition:			
Hydrogen sulfide	mol%	0.2	
Methane	mol%	2.2	
Nitrogen	mol%	0.6	
Carbon dioxide (by difference)	mol%	97	

Utility Data			
Utility Data	Units	Value	Warnings on Input Data
Operating days per year	day/yr	365	
Electrical cost	\$/kW-hr	0.06	
Steam/fuel gas cost	\$/Mmbtu	7.5	
Selective amine cost	\$/lb	2	
Catalyst cost	\$/lb	1	
Loaded cost of operator	\$/yr	80,000	
Installed compressor cost	\$/hp	1,000	

Process Specific Data			
ChE Plant Cost Index	d-less	532	Default of 437 is for March of 2005.
# of base solid scavenger vessels	d-less	1	
Solid scavenger vessel redundancy for lead/lag:			0% Redundancy <input type="text"/>

II. H₂S Removal Technologies and Costs

This section discusses the reasoning for the choice of representative technologies in each H₂S removal and sulfur recovery niches. Each of the representative technology choices are described, including desired operating conditions. Process descriptions are then followed by information on the capital and operating cost estimates.

Choice of Representative Technologies

Choice of selective amine/Claus as representative of large-scale niche: Many different types and technology combinations exist in the large-scale niche for H₂S removal and sulfur recovery including: amine/Claus approaches, physical solvent/Claus approaches, hot potassium carbonate/Claus, distillative separations, and others. Although the principles of operation vary widely, all of these would generally be used in large-scale applications with sizable sulfur tonnages. Further, with the exception of distillative

approaches, most of these were developed for natural gas and similar streams, not for streams consisting primarily of CO₂. For the purposes of the screening spreadsheet tool, it was necessary to pick one specific representative technology for the entire large-scale niche. Selective amine/Claus was chosen as the representative technology for several reasons, including the following two. An analogous application at a CO₂ flood EOR site was known. Data on amine selectivity, amine unit costs, Claus unit costs, and adjustments to those costs for various CO₂ to H₂S content ratios were available in the literature and could be built into a spreadsheet tool (whereas, commercial applications and/or cost data for other approaches [e.g., physical solvent/Claus, hot potassium carbonate/Claus, distillative methods, and others] were not as readily available).

The maximum operating pressure of the selective amine system was artificially limited to 300 psig maximum. Limiting the maximum pressure for the selective amine system was a concern. If a selective amine truly required dropping the pressure of the CO₂ stream down to 300 psig before it could be treated, and if some other technology could operate acceptably at pressures well above 300 psig, then the cost of recompression might swing the choice of the technology from selective amine/Claus to some other technology that could operate at a higher pressure, perhaps selective physical solvent/Claus. If a less expensive technology within the large-scale niche was chosen, then the break point between the medium-scale niche and the large-scale niche might be significantly different than predicted by the spreadsheet tool. (Later in this paper is an example showing how much the break points between the small-scale niche, the medium-scale niche, and the large-scale niche change if the pressure limit is relaxed.) However, selective amine/Claus was deemed a reasonable choice for the limited purposes of the screening spreadsheet tool, even with the artificial maximum pressure limitation.

Choice of iron-based liquid redox as representative of the medium-scale niche: The medium-scale niche also has a number of different technologies including several liquid redox processes (e.g., LO-CAT, SulFerox, Sulfint HP, Stretford, etc.), biological processes (e.g., Paques), and non-aqueous processes (e.g., CrystaSulf). Combinations that use a front end consisting of an amine or physical solvent unit with a back end consisting of one of the above technologies also exist within this medium-scale niche. For the purposes of the screening spreadsheet tool, direct-treat, iron-based liquid redox technology (e.g., LO-CAT, SulFerox, etc.) was chosen as the representative of the medium-scale niche. Iron-based redox technologies have been used more on CO₂ streams than the other technology variations, and are a likely commercial choice. The technology uses liquid-filled absorbers, which are reasonably easy to estimate and scale in a spreadsheet tool. Published data exist for a type of redox system that is reasonably analogous to the regeneration and sulfur separation sections of the redox plant. There are also published data for actual user-experienced chemical costs and other costs.

Choice of iron-based solid and triazine-based liquid H₂S scavengers as representative of small-scale niche: The small-scale niche for H₂S removal is dominated by H₂S scavenging approaches. With scavengers, a chemical is used to react with the H₂S, and the spent scavenger is then usually discarded. A wide variety of solid and liquid

scavengers exists. However, iron-oxide on a mineral substrate (e.g., SulfaTreat), and triazine-based liquid H₂S scavengers were chosen as the representative technologies in this small-scale niche because: 1) capacity data from tests on high-pressure CO₂ at a Denbury site were available, 2) chemical cost data suitable for use in the spreadsheet tool were readily available, and 3) capital costs could be estimated and scaled reasonably in a spreadsheet tool. Based on the spreadsheet tool, liquid scavengers are generally more economic at the lower end of the small-scale niche and solid scavengers at the higher end.

A. *Amine/Claus Technology*

The amine/Claus combination is an industry standard technology for removing H₂S and converting it into molten elemental sulfur. The spreadsheet tool assumes use of a selective amine in order to minimize CO₂ pickup; severely sterically hindered amines such as Flexsorb, specialized designs with limited contact times or with chilled MDEA, and other selective amine approaches are examples of selective amines. The authors are aware of one chilled MDEA system that was used in commercial operation for a number of years on a ~300 psig, 80+% CO₂ stream -- the Shell SAGA design that was used at the Denver Unit CO₂ Recovery Plant in Denver City, Texas. (The Shell SAGA unit at Denver City was replaced by the current SulFerox liquid redox unit in the early 1990s.)

There are amine units in use on high-pressure natural gas streams to selectively remove H₂S while slipping most of the CO₂. Theoretically, an amine system could also be used to selectively remove H₂S from a primarily CO₂ stream at high pressure. However, to the authors' knowledge, there are no amine systems in use to selectively remove H₂S from CO₂ streams (i.e., ≥70% CO₂) at pressures above 300 psig. Using a selective amine at higher pressures may involve risks or be economically unattractive for reasons discussed in the next two paragraphs.

Some of Denbury's streams are almost pure CO₂. The critical point of CO₂ is at about 88°F and 1070 psia, well below the ~1600 psig maximum operating pressures present in streams from some of Denbury's CO₂ wells. Treatment with operating pressures near or above the critical point of a stream may be risky. The stream will be very dense in comparison with normal gas treating at similar conditions, and the design of an amine contactor for such conditions may require an unusual degree of care. Further, supercritical CO₂ can be a very good solvent that dissolves and carries away other substances¹; high losses of amine into the treated gas might occur.

Treatment in CO₂ streams with operating pressures below the critical point of the mixture involves less risk. The highest pressure commercial amine system known to the authors that selectively removed H₂S from a CO₂ stream is the ~300 psig example given above. Since the highest pressure in known commercial operation was 300 psig, it was decided to use 300 psig as the upper limit for the spreadsheet tool. This means that if the pressure of the inlet gas is higher than 300 psig, then the spreadsheet tool assumes that the pressure is reduced to 300 psig before H₂S removal occurs. Costs of recompressing the treated gas stream back to the original pressure (only) are included in the screening costs and represent a significant economic penalty in many cases.

(Aside: There is a known instance of the use of the physical solvent, Selexol, to selectively remove H₂S from a high-pressure stream with relatively high CO₂ content. The application is at the LaBarge facility in Wyoming. The high pressure stream contains approximately 66% CO₂, 21% methane, 7% nitrogen, 5% H₂S, and 0.6% He². The operating pressure is 1000-1100 psig. Two Selexol units are used in series. The first unit selectively removes the H₂S using techniques to minimize CO₂ pickup (possibly including using solution that is already presaturated with CO₂ from the 2nd Selexol unit). The acid gas from this first Selexol unit contains 65% H₂S³, which is suitable as feed for a Claus unit. The second Selexol unit then removes the CO₂ from the remainder of the stream. A portion of the CO₂ is then used for enhanced oil recovery purposes. If all of the H₂S is removed by the first Selexol unit, then the first Selexol unit selectively removes H₂S while allowing about 96% of the CO₂ to pass on in the treated stream. While the CO₂ content of the LaBarge application is below the stated lower level of 70% CO₂ to which this spreadsheet tool applies, and while the bulk of the CO₂ streams of concern to Denbury are more in the range of 95+% CO₂, physical solvents and other non-amine approaches for selective H₂S removal at high pressure should probably be given serious consideration during technology selection efforts, after pre-screening.)

Process Description - In the amine/Claus process (see Figure 1) the gas to be treated passes through an amine absorber containing trays or packing where H₂S and some CO₂ are removed. Sweet gas (i.e., less than 10 ppmv H₂S for Denbury's cases) exits the top of the absorber and rich amine passes to a regenerator where heat is used to strip acid gases from the amine solution. The "acid gas" stream flows directly to the Claus unit if the H₂S content is sufficiently high (i.e., greater than 10% minimum, but preferably greater than roughly 20-30% although the ranges may vary depending on the sulfur plant configuration). If the H₂S content is below this level, an acid gas enrichment (AGE) unit might be required to concentrate the H₂S in the acid gas stream to levels that the Claus unit can handle.

The Claus unit processes acid gas from the amine unit, producing molten elemental sulfur as a product. Some of the amine acid gas is burned in a reaction furnace with enough air to combust any hydrocarbons to CO₂ and 1/3 of the H₂S to SO₂. The SO₂ produced then reacts with the remaining H₂S to produce elemental sulfur vapor. The "Claus" reaction occurs in the reaction furnace and in the downstream catalyst beds. The sulfur vapor is condensed and removed after the reaction furnace and then again after each catalyst bed. Tail gas from the last condenser is then incinerated to convert residual H₂S, sulfur vapor, and any other sulfur species to SO₂ prior to venting. The Claus unit costs used in the screening spreadsheet tool are within plus or minus 50% for a new Claus unit with a SuperClaus (direct-oxidation-type tail gas treater) integrated into the unit as the third catalytic bed. The spreadsheet tool only considers the situation of three catalytic beds total (e.g., two Claus catalyst stages plus one direct oxidation stage). This SRU configuration (two Claus stages plus one direct oxidation stage) can achieve ~ 99% sulfur recovery, but recoveries drop as the H₂S/CO₂ ratio in the SRU feed gas drops. Additional tail gas treatment might be required by regulations, especially when sulfur loads are greater than ~20 LTPD.

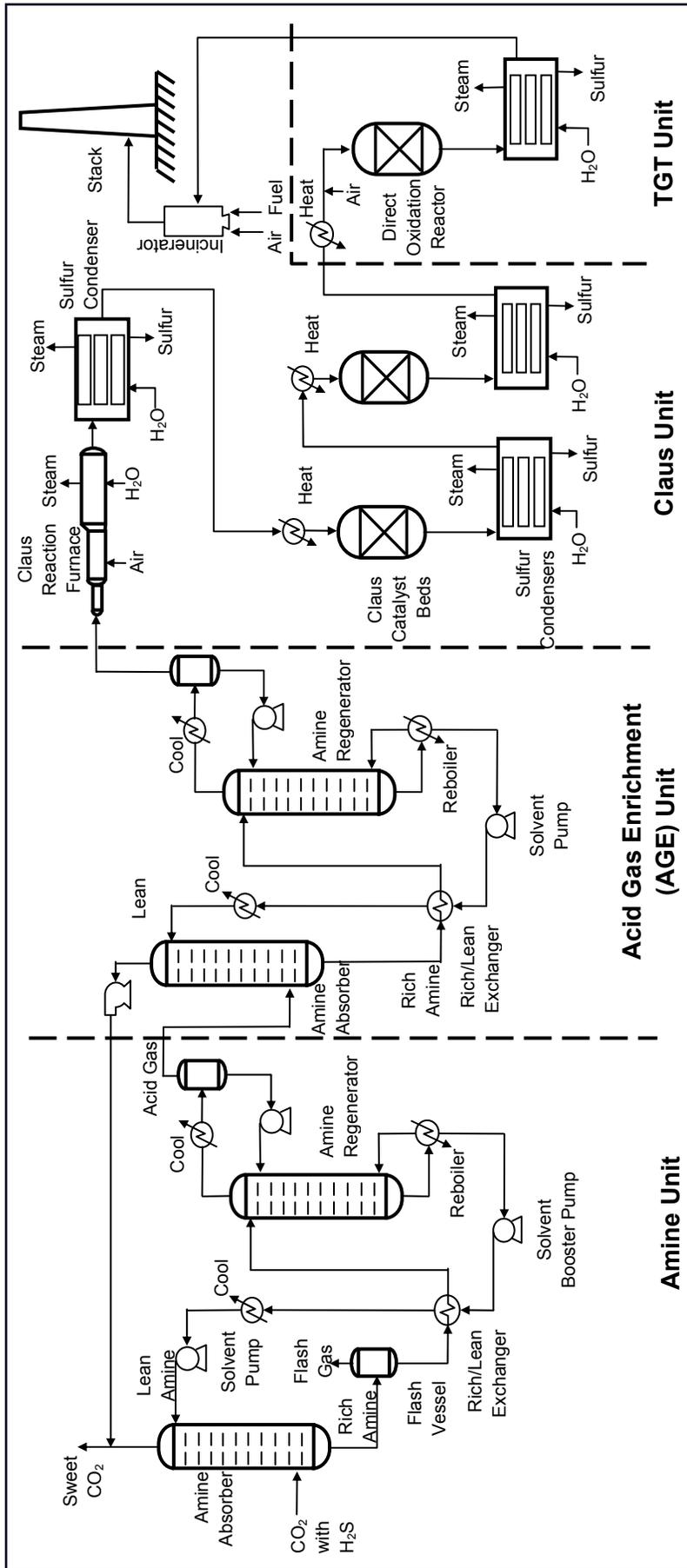


Figure 1. Amine/Claus/TGT for treating CO₂ Streams

Cost Estimating Basis - The amount of H₂S and CO₂ picked up in the selective amine unit is based on assuming essentially all of the H₂S is removed and then using a selectivity ratio of 12.5 [(H₂S/CO₂) in liquid / (H₂S/CO₂) in inlet gas]. The selectivity ratio chosen for the spreadsheet tool results in 92% to 95% slip of the CO₂, which was near the highest selectivity found in the literature⁴ and is believed to represent a selectivity on the higher end of what is possible for selective amines as a group.

The amine circulation rate is then calculated from the amount of H₂S and CO₂ in the liquid and the specific circulation rates used in the literature cost references. One literature cost reference used a circulation rates of 0.3 gal/scf of acid gas⁵, and the other a circulation rate equivalent to 0.16 gal/scf of acid gas (obtained via personal communication with the author of the other literature source)⁶. Both sources correlate the circulation rate to capital costs for an amine unit, and the capital costs were calculated according to each source's cost curve and circulation assumptions, and the average value from these two sources is reported in the spreadsheet tool. Although both of these sources involve secondary amines and not selective amines, the circulation rates and capital costs per unit of circulation rate of the nonselective amines vs. the selective amines should be close enough for the purposes of the rough costs desired for this screening work. The cost of the amine absorber was not calculated separately, because the cost data used were for complete amine systems including absorbers.

The spreadsheet tool is also set up to calculate costs for an AGE unit, which is a second amine unit that takes the acid gas from the main (first) selective amine unit and further concentrates the H₂S. Capital costs for the AGE unit are calculated in the same manner as the main selective amine unit.

Operating costs for the main selective amine unit and AGE unit (if needed) are based on a correlation found in the literature⁵ for amine units. These costs include electricity consumption for amine pumps, heat for the reboiler assuming 60 psig steam, makeup amine, and requirements to operate the unit. Cooling water costs are not included because it is assumed that air coolers are used for the lean amine and still reflux condenser.

The capital costs for a Claus unit with three catalytic beds are also based on literature data^{5,6} that are correlated to the tonnage of sulfur removed by the process. The Claus capital costs used as a basis are for a unit designed for H₂S feed in the 80+% range⁶, 90 to 97% recovery with three catalytic beds, waste heat boiler, a sulfur storage/receiving tank, and incinerator. With the high-CO₂ streams like those considered in this work, the acid gas stream from the main selective amine unit or AGE unit frequently contains less than 80+% H₂S. Since a Claus unit processing low concentration (e.g., 25%) H₂S acid gas costs more than an equivalent capacity Claus unit processing high concentration (e.g., 80% H₂S) acid gas, the capital costs obtained from the two sources are adjusted to account for the effect of the H₂S concentration fed to the Claus unit. This is done by using the following relationship⁷: $\text{Cost 2} = \text{Cost 1} * ([\text{H}_2\text{S concentration 1}] / [\text{H}_2\text{S concentration 2}])^{0.4}$. "H₂S concentration 1" (i.e., the basis for the published cost correlations) was taken as 85% for the purposes of this adjustment calculation. The average of the costs obtained from the two sources is used in the spreadsheet tool. Operating costs are based on literature data⁵ and include such items as electricity, net steam production, initial catalyst charge, and staff requirements to operate the unit.

The horsepower requirement for recompressing the CO₂ from the absorber of the main amine unit back to its original pressure, if needed, is estimated using a general equation documented in the GPSA Engineering Data Book⁸. The installed compressor cost is based on user-provided cost data in \$/hp. The operating cost for electricity is estimated based on the user supplied electricity cost. In general, when the inlet stream is well above 300 psig, the CO₂ recompression cost adds significantly to amine/Claus costs.

It should be noted that the treating cost reported in the spreadsheet tool for the amine/Claus technology type is based on the process flow scheme with the minimum estimated cost (i.e., either main selective amine/Claus or main selective amine/AGE/Claus). The spreadsheet tool calculates the cost of a system with a single main selective amine unit followed by a Claus unit and compares that cost with the costs for a system with a main selective amine unit, an AGE unit, then a Claus unit. The system with the minimum cost is then chosen automatically by the spreadsheet tool.

B. Liquid Redox Technology

Liquid redox processes use aqueous solutions containing metal ions, e.g. iron, which are capable of transferring electrons in reduction-oxidation (redox) reactions. The iron is held in solution by chelating agents. This chelated iron catalyst accelerates the overall oxidation reaction of H₂S to form elemental sulfur.

Process Description - Figure 2 gives the main flow scheme for liquid redox technology. When applied to a pressurized high-CO₂ stream, the contactor is assumed to be a liquid-filled contactor. The gas stream sparges into the liquid so that H₂S from the gas absorbs into the aqueous, chelated iron solution where it reacts with iron to form elemental sulfur particles. The spent iron solution is regenerated with air in the oxidizer. A portion of the sulfur is then settled from the slurry. The thin slurry from the top of the settler, which contains a lower weight percentage of sulfur particles, is then returned to the absorber. The thick slurry from the bottom of the settler goes to a filter system where a sulfur cake is removed.

Due to the presence of several phases of matter throughout the process (gas, aqueous, surfactant/hydrocarbon, solid sulfur), liquid redox processes involve slurries and materials which can plug equipment. As a result, liquid redox processes have poorer reliability than processes that handle only gases and liquids^{9,10,11}. Redox processes operating at higher pressures have historically been associated with more operating problems than those at lower operating pressures. Further, high CO₂ partial pressure exacerbates the operating problems¹¹. A number of publications have reviewed some of the problems observed when operating liquid redox units at high pressures^{9,10,12}. However, to the authors' knowledge, at least one iron-based liquid redox process of the configuration discussed above (including liquid-filled, sparged absorbers) is in satisfactory commercial operation on 80+% CO₂ gas at ~300 psig. It is the iron-based liquid redox unit at the Denver Unit CO₂ Recovery Plant. (Other liquid redox units may process very high concentration CO₂ gas, but generally at lower pressures. And, liquid redox units which operate at high pressures generally process gases that are not composed primarily of CO₂.) For these reasons, the spreadsheet tool assumes that a conventional liquid redox process can treat CO₂ streams up to a limit of ~300 psig.

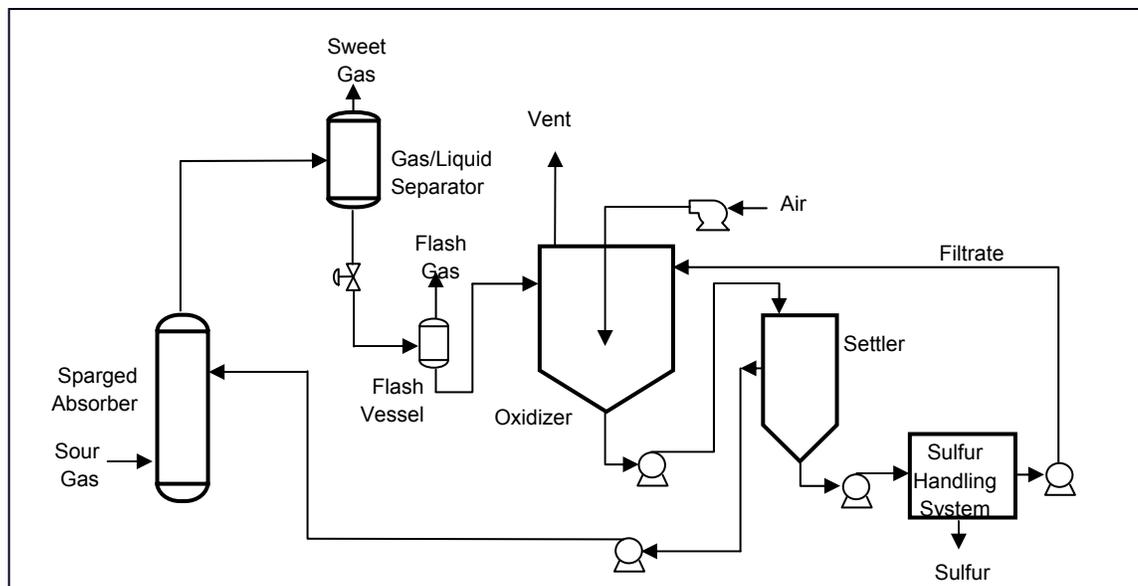


Figure 2. Aqueous-iron Liquid Redox System

Cost Estimating Basis - The liquid redox cost estimate in the spreadsheet tool assumes that the inlet pressure of the gas is first reduced to 300 psig (assumed to be the highest pressure for reliable operation) and then recompressed to the original pressure after the sulfur has been removed from the CO₂ stream. Liquid redox technology has been around for many years and much data exist in the literature that summarizes the capital and operating expenses for the process. As a result of the reliability problems mentioned previously, the capital cost estimate in the spreadsheet tool is based on two, 100% capacity trains (this is examined further near the end of the paper).

The best available capital cost data are for an auto-circulation style system. Auto-circulation style systems are reasonably analogous to the back end of a conventional liquid redox system, but, unlike a conventional system, do not contain a stand-alone absorber or main circulation pump, and do contain other special features (e.g., an absorber is integrated into the oxidizer). For the purposes of the spreadsheet tool, the costs of the main circulation pump and other special features (which offset to some extent) were neglected, but the cost of a stand-alone absorber was estimated separately. The high-pressure absorbers are sized based on the approximate superficial gas velocity from a liquid redox unit located at a west Texas plant. A height to diameter ratio of 3 was used. The basis for the absorber vessel cost in the spreadsheet tool is a vendor quote for a single, stainless steel vessel sized appropriately for a particular Denbury application and scaled for all other cases. Stainless steel construction is assumed for the liquid redox process. The cost of the absorber vessel is then adjusted based on the approximate weight of the vessel as it changes with operating pressure, height, and diameter. An exponent of 0.6 is used in the spreadsheet tool to scale purchased cost with weight for this vessel, then a factor of 3.5 is used to estimate the equivalent installed capital for this vessel. The installed capital cost for the back-end equipment in the processes (i.e., oxidizer and sulfur recovery equipment) is reasonably similar to an auto-

circulation unit. Three sources of installed capital cost data for auto-circulation units, two of which also contained cost data for conventional units were analyzed and compared^{6,13,14}. Based on the analysis, the back end equipment cost was approximated by escalating to current year the cost data for the autocirculation flow scheme presented in a regional GPA paper¹³.

Operating costs for the liquid redox process are based in part upon data presented in an AIChE paper¹⁴. The operating costs include chemicals, labor, electricity, steam, cooling water, maintenance, and overhead expenses. These operating costs are not expected to have changed much based on input from user panel discussions at the GRI Sulfur Recovery Conference^{15,16}. In those users' panel discussions, chemical costs were documented as varying from \$250/LT to \$520/LT and generally make up 10 to 36% of the total operating costs. While the chemical portion of the operating costs may have varied over time, possibly even coming down from what it was in the 1990s, after comparing the data sources, it was the authors' choice to use the data source mentioned above. Using lower operating (and chemical) cost values could significantly affect the results of the screening, thus for cases where the economics are close, vendors should be consulted for current chemical consumption and costs.

The cost for recompressing the CO₂ back to its original pressure is calculated in the same manner as with the amine/Claus technology type. In general, the requirement of 100% redundancy and the cost of having to recompress the CO₂ make liquid redox technology more expensive than it might be without recompression and without the redundancy. .

C. H₂S Scavenger Technology

Two types of H₂S scavengers were considered, direct injection of liquid scavengers and use of solid scavengers.

Liquid Scavengers

H₂S can be removed from high-pressure CO₂ streams using direct injection H₂S scavengers. Although batch processes where gas passes through a vessel filled with the liquid scavenger are known to be used commercially, many studies have shown that the direct injection method is a lower cost alternative when a suitable pipeline length is available for contact. Generally speaking, suitable lengths of pipeline are expected to be available at the Denbury sites.

Process Description - As shown in Figure 3, the direct injection scavenging process is fairly simple and consists of only a chemical injection pump, a means of introducing the scavenger into the sour gas pipeline, a length of pipe to allow for gas/liquid contact, and a downstream device for separating spent or excess scavenging agent from the treated gas. The amount of gas/liquid contact time is dependent on the type of contacting device, the gas velocity, and residence time. Because of this, the degree of mixing and efficiency is sensitive to changes in gas flow. Good separation of the scavenger and gas is required since some scavengers can cause problems with downstream equipment (e.g., foaming in dehydrators). The Denbury cases all involved wet CO₂ streams, so materials were either stainless or coated carbon steel, which is also suitable for liquid scavenger.

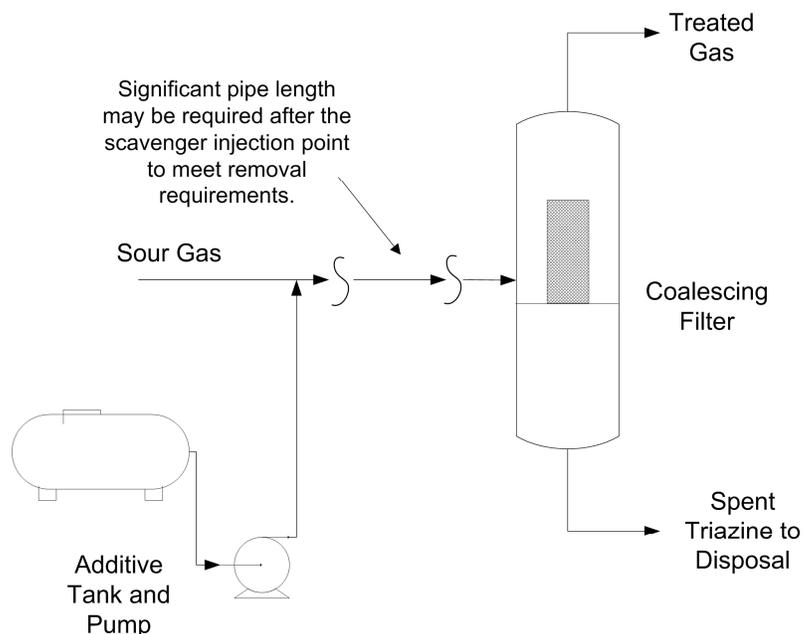


Figure 3. Direct Injection Liquid Scavenger

Cost Estimating Basis - The capital costs for direct injection scavenging are quite low (conservatively fixed at a constant \$150,000 in the spreadsheet tool), but the cost of the liquid scavenger chemical limits this technology to low sulfur tonnage applications. Costs for two different triazine-based scavengers were obtained directly from vendors. These scavengers were tested at a Denbury facility. Results are shown in Table 2. The results indicate that direct injection scavenging is effective for high-pressure CO₂ streams, but that injection rates of 0.2-0.3 gallons of scavenger per MMscf per ppmv of H₂S that is removed (gal/[MMscf*ppmv]) or higher may be required. This is higher than a typical natural gas application, which may require about 0.12 gal/(MMscf*ppmv H₂S)¹⁷.

Note - potential users of triazine scavenger for removal of H₂S from supercritical CO₂ should consider the existence of patent literature having to do with this topic¹⁸.

The screening spreadsheet tool uses a triazine scavenger usage rate of 0.22 gal/(MMscf*ppmv H₂S) and the average cost from the two liquid scavengers are used to estimate annual operating costs. Although the 0.22 gal/(MMscf*ppmv H₂S) usage rate resulted in outlet H₂S concentrations around 20-40 ppmv, it is anticipated that with sufficient pipe length and time to react, outlet H₂S concentrations below 10 ppmv could potentially be expected from the 0.22 gal/(MMscf*ppmv H₂S) usage rate. Costs to dispose of spent scavenger are not included; given the E&P waste exemption from hazardous classification, disposal costs are generally minor relative to the cost of purchasing fresh scavenger agent.

Table 2. Triazine Scavenger Test Results on High-Pressure CO₂

Test Description:		Triazine Scavenging Agent 2									
Date:		27-Oct-04									
Test	Time	Gas Flow MMscfd	Pressure psia	Temperature F	Scavenger gph	Inlet H2S ppmv	Sample 1 ppmv	Sample 2 ppmv	Sample 3 ppmv	Separator ppmv	Usage Rate gal/(MMscf*ppmv)
C1	9:36	8.3	1718	118	1.8	72					
	10:20	8.2	1748	123			43	45		43	
	11:00					70	38	43	40	40	
	Average	8.3	1733	121	1.8	71	41	44	40	42	0.18
C2	11:32										
	12:30					70		21	20	20	
	13:30	8.1	1768	128	3.8		22	20	23	19	
	14:05	8.1	1770	128		69	20	23	20	19	
	Average	8.1	1769	128	3.8	70	21	21	20	19	0.22
C3	14:29										
	15:09				4.8	71	12	11	11	10	
	15:30	8.2	1776	129		70	14	11	13	9	
	16:05	8.1	1784	129		72	12	11	10		
	Average	8.1	1780	129	4.8	71	13	11	11	10	0.23
Test Description:		Triazine Scavenging Agent 1									
Date:		28-Oct-04									
Test	Time	Gas Flow MMscfd	Pressure psia	Temperature F	Scavenger gph	Inlet H2S ppmv	Sample 1 ppmv	Sample 2 ppmv	Sample 3 ppmv	Separator ppmv	Usage Rate gal/(MMscf*ppmv)
B1	11:00	7.8	1752	123	1.8	66	46	40	43	36	
	11:40	8.5	1818	128	1.8	64	49	49	42	49	
	12:05	8.2	1789	127	1.9	70	49	42	46	42	
	Average	8.2	1786	126	1.8	67	48	44	44	42	0.22
B2	13:00				3.8						
	13:55	8.2	1810	130	3.9	70	27	27	22	22	
	14:20	8.3	1821	130	3.9	68	29	28	26	25	
	14:55	8.2	1811	130	3.8	78	28	26	23	23	
	Average	8.2	1814	130	3.9	72	28	27	24	23	0.23
B3	15:45	8.3	1827	131	4.8	62	21	20	13	17	
	16:20	8.3	1830	132	4.6	72	20	13	19	10	
	16:50	8.4	1842	132	4.7	65	21	21	12	17	
	17:20	8.3	1837	133	4.6	70	23	9	9		
	Average	8.3	1836	132	4.7	67	21	16	13	15	0.26

Solid Scavengers

Solid scavengers can also be used for H₂S removal from high-pressure CO₂ streams using vessels containing packed beds in down flow service. The solid scavengers considered in this work are generally iron-based materials (e.g., SulfaTreat) that, unlike iron sponge, form mostly innocuous iron pyrite (FeS₂) as a reaction product, although there are traces of iron sulfide (FeS).

Process Description - To achieve maximum efficiency, the gas stream must be saturated with water and a “lead-lag” processing scheme employed. In this processing scheme (see Figure 4), two or more vessels are employed in series. When the H₂S concentration in the outlet of the first vessel in the series rises until it approaches the inlet concentration, the first vessel is isolated while the gas is diverted directly to the second vessel. The media in the first vessel is changed, and the first vessel is put back into service as the backup vessel in the series.

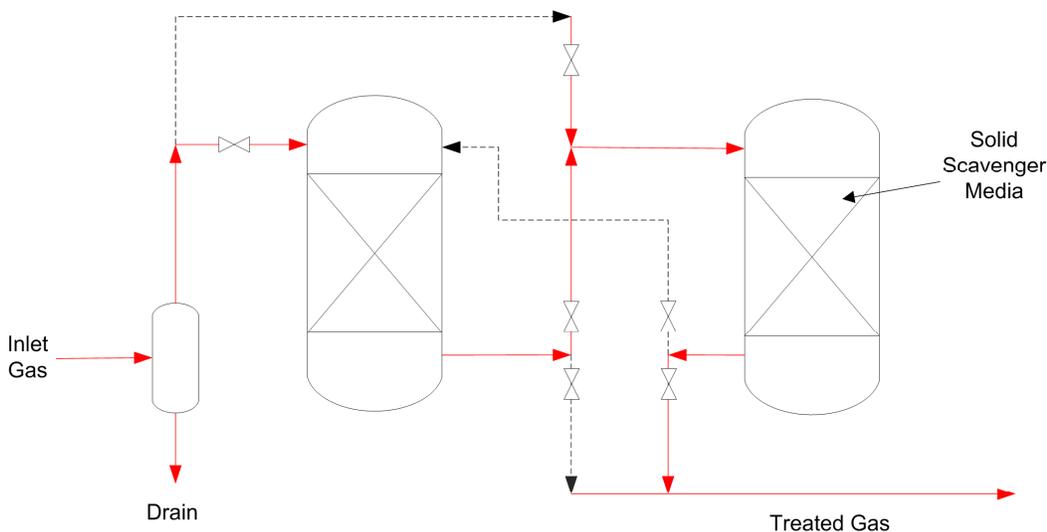


Figure 4. Solid Scavenger Lead-Lag Flow Diagram

Unlike direct injection of liquid scavengers, the effectiveness of solid scavengers is mostly independent of gas flow rate. Therefore, there are typically few problems with turndown. Because of the cost of replacing the solid scavenger media, solid scavengers are limited to low tonnage sulfur removal applications.

Cost Estimating Basis - The capital cost calculations in the spreadsheet tool are based on contactor sizing calculations that assume a constant height of about 20 feet and a superficial velocity of 2.7 feet per second (per field tests at a Denbury site). The cost for the solid scavenger vessel in the spreadsheet tool is estimated in a similar manner to the liquid redox absorber except it is assumed that the solid scavenger vessel is carbon steel material (approximately 50% of the stainless steel value) and lined with a polymeric coating (20% more expensive than carbon steel alone) known to protect against corrosion by wet CO₂ per Denbury experience. The spreadsheet tool has a warning if the diameter of the solid scavenger beds exceeds 10 ft, prompting the user to consider additional parallel vessels. In some cases, if the CO₂ stream is being blended with other sweet CO₂ streams, some H₂S slip may be tolerated for a short period of time. Because of this, the user can select the level of redundancy for the solid scavenger vessels. The user can select 100% redundancy (i.e., a typical lead/lag arrangement), which would be necessary to guarantee low level H₂S removal

on a continuous basis, or 0% redundancy (which could be a single vessel) when infrequent spikes in H₂S content can be tolerated in downstream processing.

Operating costs are dominated by the cost of the solid scavenger since it must be replaced when breakthrough occurs. The cost of the solid scavenger was obtained from the vendor of the media. The utilization rate was measured in tests with a lead/lag configured test apparatus at a Denbury facility. The results, shown in Table 3, were used in the spreadsheet tool. Given that the measured capacity of 0.06 lb S per pound media is significantly lower than the usual expected maximum capacity of 0.11 lb S per pound media (about 12 % H₂S by weight) when operating on natural gas, it appears that the very high CO₂ partial pressures result in a loss of capacity. Given the E&P waste exemption from hazardous classification, media disposal costs tend to be minor relative to the cost of purchasing new media and were neglected.

Table 3. Iron-based Solid H₂S Scavenger Test Results on High-Pressure CO₂

Iron-based Solid H₂S Scavenger Test Equipment	
I.D. of pipe spool, inches	4
Bed Depth, inches	66
Volume of solid scavenger media, ft ³	0.48
Bulk density of solid scavenger media, lb/ft ³	70
Mass of solid scavenger media in test equip, lb	33.6
Test Conditions	
Flow, Mscfd	108
Pressure, psia	1530
Temperature, F	109
CO ₂ , %	98+
H ₂ O	Saturated
Inlet H ₂ S, ppmv	63
Time until first H ₂ S detected in outlet, hours	22
Time until first H ₂ S above 3 ppmv detected in outlet, hours	76
Total length of capacity test, hours	123
Outlet H ₂ S at end of capacity test, ppmv	58
Measured Capacity	
Measured capacity supercritical CO ₂ stream , lb sulfur per pound of media	0.06
Normal maximum capacity, lb sulfur per pound of media	0.11

III. Case Studies

Several case studies are provided below where the spreadsheet tool was used to show the approximate break points between the small-scale niche, the medium-scale niche, and the large-scale niche for H₂S removal and sulfur recovery. In some cases, this can differ greatly from the usual ranges for natural gas streams that have been previously published.

The basis for this work is a gas stream at 120°F containing 2.2 mol% C₁, 0.6 mol% N₂, with the balance H₂S and CO₂. Effects of gas flow, pressure, equipment redundancy for the liquid redox process, and the artificial pressure limitation for the selective amine/Claus process are explored in the case studies discussed below. It should be noted that the case studies illustrate the general trends in treating tonnage anticipated when using the H₂S removal technologies on high pressure CO₂ streams; however, the ranges may vary some in actuality given the accuracy of the screening cost estimates.

A. Effect of Gas Flow Rate

Table 4 shows the typical sulfur tonnage range for operation of each of the H₂S removal technologies on natural gas streams compared to that obtained with the H₂S screening spreadsheet tool at different gas flow rates and an inlet pressure of 1400 psig. As shown in Table 4, the small-scale technologies appear economical to as high as 0.6 LTPD to 2 LTPD for these high CO₂ streams. This is because of the cost of having to recompress the gas from 300 to 1400 psig for the representative technology for the medium-scale niche, liquid redox. Scavengers do not require that the stream pressure be lowered and, thus, recompression costs are avoided.

Table 4. Effect of Gas Flow Rate on Niche Break Points for H₂S Removal and Sulfur Recovery from High-Pressure CO₂ Streams (100% redundancy for liquid redox, 300 psig max limit for liquid redox and selective amine/Claus)

Technology Size Niche	Units	Typical Natural Gas	50 MMscfd CO ₂ 1400 psig	100 MMscfd CO ₂ 1400 psig	200 MMscfd CO ₂ 1400 psig
Small Scale (e.g., H ₂ S Scavengers)	LTPD	0 to 0.1	0 to 0.6	0 to 1.1	0 to 2.0
Medium Scale (e.g., Liquid Redox)	LTPD	0.1 to 15	0.6 to 11	1.1 to 22	2 to 39
Large Scale (e.g., selective amine/Claus)	LTPD	15+	11+	22+	39+

The high end of the sulfur treating range for the liquid redox technology also increases with gas flow. The reason for this is that gas flow rate and CO₂-to-H₂S ratio has only a minor effect on liquid redox technology, whereas the CO₂-to-H₂S ratio has a greater effect on selective amine/Claus costs due to the poorer Claus plant feed.

In general, the treating tonnage range for high-pressure CO₂ streams for the technologies considered in this paper differ significantly compared to typical natural gas

applications, partly because of the imposed limit of 300 psig maximum for the selective amine technology and the resulting need for recompression to pipeline pressure.

B. Effect of Pressure

Table 5 shows the effect of pressure on the treating tonnage ranges for high-pressure CO₂ streams. As shown in the table, for a given gas flow rate, changes in pressure greatly impact the high end of the small-scale niche. The representative technology for the medium-scale niche (liquid redox) can operate at up to 300 psig and, therefore, does not require recompression at 300 psig. At the 300 psig condition shown in the table, the niche break points are similar to the typical natural gas ranges. However, as the pressure increases, the cost of recompression for the liquid redox becomes greater and scavengers are more economic over a wider range of sulfur tonnage (up to 0.6 LTPD).

Table 5. Effect of Pressure on Niche Break Points for H₂S Removal and Sulfur Recovery from High-Pressure CO₂ Streams (100% redundancy for liquid redox, 300 psig max limit for liquid redox and selective amine/Claus)

Technology Size Niche	Units	Typical Natural Gas	50 MMscfd CO2 300 psig	50 MMscfd CO2 800 psig	50 MMscfd CO2 1400 psig
Small Scale (e.g., H ₂ S Scavengers)	LTPD	0 to 0.1	0 to 0.1	0 to 0.45	0 to 0.6
Medium Scale (e.g., Liquid Redox)	LTPD	0.1 to 15	0.1 to 11.5	0.45 to 10.9	0.6 to 10.7
Large Scale (e.g., selective amine/Claus)	LTPD	15+	11.5+	10.9+	10.7+

On the other hand, pressure has little effect on the break point between the medium-scale niche and the large-scale niche. This is because the representative technologies chosen for both niches (liquid redox and selective amine) were restricted to 300 psig maximum pressure, thus both require recompression, for all pressures above 300 psig. (The 300 psig limit on the selective amine is artificially imposed, and a later section explores the effect of this limit.)

C. Effect of Liquid Redox Equipment Redundancy

Because of reliability problems with liquid redox technology, the authors decided that the spreadsheet tool should estimate costs for the medium-scale niche based on 2, 100%

capacity liquid redox trains. Table 6 shows how the break points between niches would change if this redundancy requirement was eliminated and only one train were required. The results can be compared directly to those in Table 4. By comparing the two tables, one can see that the high-end tonnage range for liquid redox technology ranges from 11 to 39 LTPD with two 100% redundant liquid redox trains and from 15 to 45 LTPD with no redundancy.

Table 6. Effect of Liquid Redox Equipment Redundancy on Niche Break Points for H₂S Removal and Sulfur Recovery from High-Pressure CO₂ Streams (0% redundancy for liquid redox, 300 psig max limit for liquid redox and selective amine/Claus)

Technology Size Niche	Units	Typical Natural Gas	50 MMscfd CO ₂ 1400 psig	100 MMscfd CO ₂ 1400 psig	200 MMscfd CO ₂ 1400 psig
Small Scale (e.g., H ₂ S Scavengers)	LTPD	0 to 0.1	0 to 0.5	0 to 0.9	0 to 1.8
Medium Scale (e.g., Liquid Redox)	LTPD	0.1 to 15	0.5 to 15	0.9 to 26	1.8 to 45
Large Scale (e.g., selective amine/Claus)	LTPD	15+	15+	26+	45+

D. Effect of Relaxing Artificial 300 psig Limit for Selective Amine

Table 7 shows the break points for the small-scale niche, the medium-scale niche, and the large-scale niche for H₂S removal from CO₂ streams if the artificially imposed limit of 300 psig maximum for selective amine/Claus is relaxed to 800 psig. At 800 psig, the CO₂ partial pressure of the stream to be treated is similar to the CO₂ partial pressure of the La Barge case described in Section II, A. For the purposes of Table 7, it was assumed that selective amine could operate at 800 psig but the liquid redox technology was still limited to 300 psig maximum. Therefore, the selective amine/Claus technology would not require recompression while the liquid redox would.

Comparing Table 5 with the “100% Liquid Redox Redundancy” column in Table 7, the break point between the medium-scale niche and the large-scale niche appears to shift from roughly 11 LTPD in Table 5 down to about 9 LTPD if the pressure limit for selective amine is relaxed.

Table 7. Effect of Relaxing Artificial Pressure Limit for Selective Amine on Niche Break Points for H₂S Removal and Sulfur Recovery from High-Pressure CO₂ Streams (100% redundancy for liquid redox, 300 psig max limit for liquid redox only)

Technology Size Niche	Units	50 MMscfd 800 psig 100% Liquid Redox Redundancy	50 MMscfd 800 psig 0% Liquid Redox Redundancy	Comments
Small Scale (e.g., H ₂ S Scavengers)	LTPD	0 to 0.45	0 to 0.38	
Medium Scale (e.g., Liquid Redox)	LTPD	0.45 to 8.9	0.38 to 12.8	Includes recompression from 300 psig
Large Scale (e.g., selective amine/Claus)	LTPD	8.9+	12.8+	Assumes amine/Claus unit can operate at 800 psig; no recompression

E. General Results from H₂S Removal Screening Spreadsheet Tool

Figure 5 shows the type of information that is reported in the screening spreadsheet tool, namely the operating, capital, and overall treating costs for the sulfur removal. The base case gas conditions used to generate Figure 5 were 50 MMscfd, 120°F, 1400 psig, 0.2 mol% H₂S, 2.2 mol% C₁, 0.6 mol% N₂, and 97% CO₂ (3.8 LTPD). Relative costs are shown for the purposes of this paper, while estimated actual costs are given in the spreadsheet tool.

As shown in the figure, liquid redox is calculated to have the lowest operating costs for this particular case, followed by selective amine/Claus. The liquid and solid scavenging treating costs are considerably higher than the other technologies for this 3.8 LTPD sulfur case due to the need to replace the spent scavenging agent. As expected, the capital costs for the liquid and solid scavenging technologies are the lowest since the equipment in the process is relative simple (i.e., mainly vessels for the scavenging agent and pumps). The overall treating costs show that the liquid redox technology, even with 100% redundancy, is most applicable for this 3.8 LTPD application.

(For reference, compression accounted for 23 and 33% of the capital costs and 36 and 54% of the annual operating costs for the amine/Claus and liquid redox systems, respectively.)

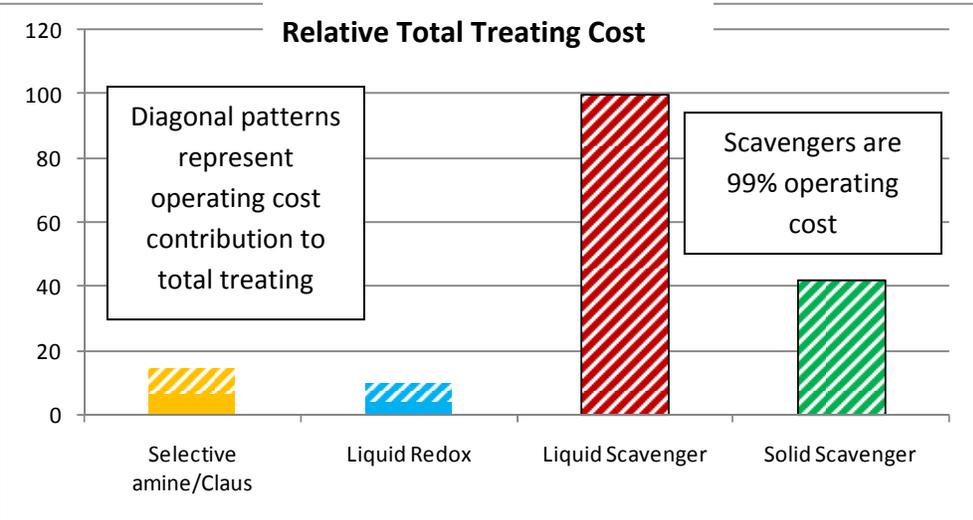
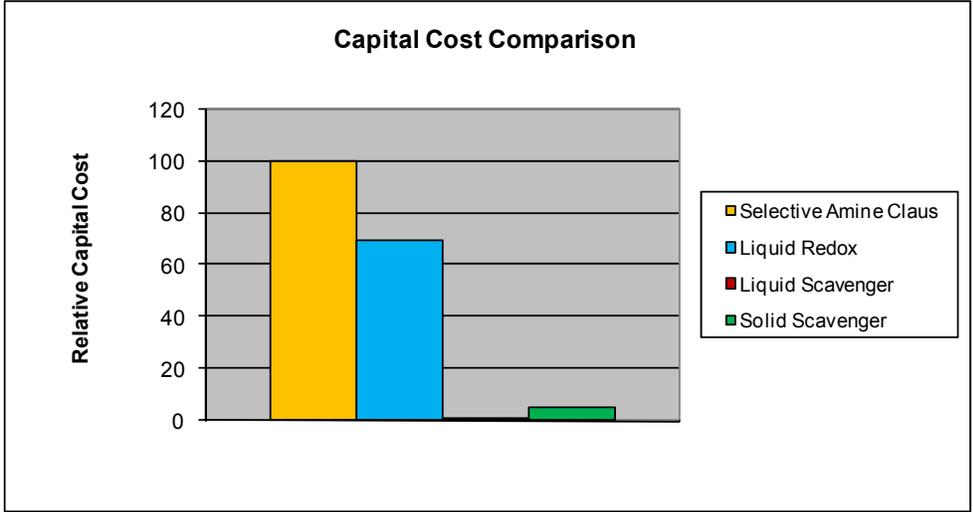
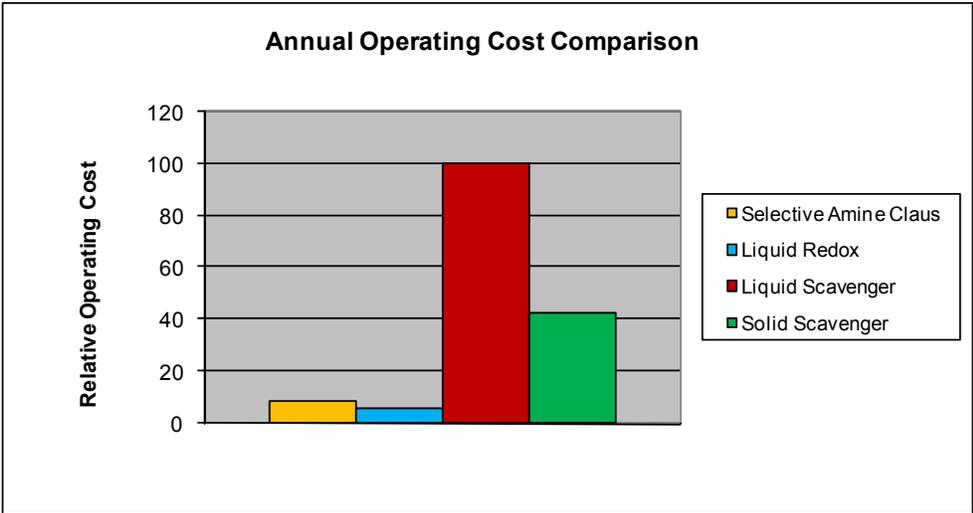


Figure 5. Case Study – H₂S Removal Cost (50 MMscfd, 1400 psig, 120F, 97% CO₂, 2.2% C₁, 0.2% H₂S, 0.6% N₂)

IV. Screening Spreadsheet Tool Summary

Although the cost estimates for the technology types in this paper were checked with literature data and vendor quotes, the calculations are considered screening-level at best. The H₂S removal technologies have been used in the industry for many years and there is a considerable database of cost and equipment sizing data to draw from.

The spreadsheet tool can be used for cost screening purposes (+/- 40% to +/-50%), but more detailed information should be obtained from a licensor or vendor if a particular technology appears applicable to the CO₂ stream of interest. Even though the spreadsheet tool was created for a CO₂ producer, the data and methodologies should be useful for others who need to achieve a screening level understanding of the costs of H₂S removal and sulfur recovery from streams containing very high CO₂ partial pressures. As shown in the case studies, the typical operating range for the H₂S removal technologies can vary greatly from those used with natural gas streams. The spreadsheet tool can help users understand the way total treating costs vary with such differences in CO₂ to H₂S ratio, pressure, equipment redundancy, and operating costs.

It should also be noted that this version of the spreadsheet tool does not directly consider using combinations of available technologies (hybrid processes) that could potentially be better performing and more economic. Evaluation of hybrid processes would typically occur for a specific project once technology screening indicated that the project could be economical.

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