

Use of Caustic in a Short Contact Time Approach to Selectively Scrub H₂S from CO₂-Contaminated Gas Streams

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Abstract

The use of caustic to scrub H₂S from gases is well established technology that is often applied in situations where the H₂S volumes are low, the required serviced is intermittent, and/or the NaSH solution product from the scrubber is valuable. Since caustic solution is a strong base it will absorb both CO₂ and H₂S. Therefore, gases containing significant amount of CO₂ in addition to H₂S are usually not treated with caustic due to the concurrent absorption of CO₂ with the H₂S; the absorption of CO₂ is usually unwanted since it represents waste of caustic, contamination of the NaSH product with carbonate salts, and the potential of plugging process equipment with carbonate salt solids.

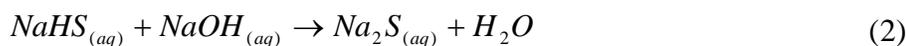
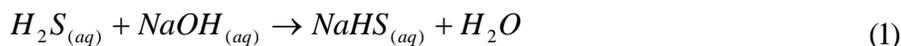
However, the absorption of CO₂ in caustic has significantly slower kinetics than that of H₂S. This difference can be exploited to obtain selective absorption of H₂S over CO₂ by limiting the contact time of the caustic solution and the gas. The use of down-flow static mixers have been shown to be effective in the contacting of caustic and gas for a very short time (approximately 1/100 to 2/10 of a second), resulting in good selectivity of H₂S absorption over CO₂. Data from an operating plant using short contact time (SCT) technology showed selectivities ranging from 26 to 34, which agreed well with predictive models that were created based on information from the literature. (Selectivity is defined here as the ratio of the NTU's for H₂S and CO₂ absorption.)

This paper discusses the background of this technology, the rational for application, and the design and control of SCT units. Additionally, kinetic and chemical/phase equilibrium issues are critical to the successful implementation of this technology; the impact of these issues on the design and operation of the SCT unit is discussed in some detail. Finally, some data from an operating plant is presented which demonstrates the efficacy of the technology.

Background

The scrubbing of hydrogen sulfide (H₂S) using sodium hydroxide (NaOH, caustic) solution is an established technology that has been applied in a variety of industries. Since this process uses a solvent which cannot be easily regenerated, caustic scrubbers are most often applied in situations where only small quantities of H₂S need to be removed. However, if an aqueous sodium bisulfide (NaSH) product of high quality can be made, it can often be sold. Depending on the product value, caustic scrubbing may be cost effective in the United States at sulfur throughputs of higher than 10 ton/day.

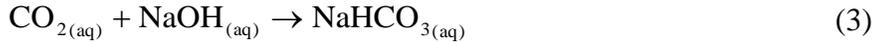
In the scrubbing process, NaOH reacts with H₂S dissolved in aqueous solution to form NaSH and sodium sulfide (Na₂S). The reactive absorption of H₂S into caustic solution can be represented as:



The extent of reactions 1 and 2 are dependent upon the strength of caustic, with higher pH favoring reaction 2. In order to maintain NaSH product quality, it may be necessary to control

the pH of the spent caustic solution so that NaSH is the predominant sulfide species rather than Na₂S.

Carbon dioxide may also be present in gas streams. CO₂ complicates the use of caustic for H₂S scrubbing because CO₂ is readily scrubbed into the caustic as well. The reactive absorption of CO₂ into caustic solution can be represented as:



Two main categories of factors control the absorption of H₂S and CO₂ into caustic solution: equilibrium considerations and rate (kinetic) considerations. The species equilibrium of the above reactions (often described in terms of pK_a) defines the lowest level to which the acid gases can be reduced in the treated gas. For situations where there is more than one ionization in solution (as is the case for both CO₂ and H₂S), the pK_a of the first and second reactions are usually referred to as pK_{a1} and pK_{a2}, respectively. The precipitation of the salt species is also generally controlled by equilibrium considerations. The rate processes that are important include various mass transfer rates of reacting species and products, and reaction rates.

At equilibrium with a high pH solution, the vast majority of both CO₂ and H₂S would be removed from the gas phase. The pK_{a1} values and equilibrium partial pressures of H₂S and CO₂ are not dissimilar enough to be able to achieve a high degree of selectivity for the absorption of one species over the other merely by manipulating the pH of the solution. However, the rate of absorption of CO₂ and H₂S into caustic are known to be different, with CO₂ being significantly slower.

Technology for the exploitation of the kinetic differences between the absorption of CO₂ and H₂S into caustic solution for the selective scrubbing of H₂S from gases was developed by the Dow Chemical Company (1, 2, 3), and was patented in 1956. More recently, attempts were made to mathematically model this system (4, 5). Although some industrial applications of SCT caustic scrubber systems are known, it does not appear that this technology has been widely adapted.

The economic efficacy for the use of caustic scrubbing for H₂S is dependent upon the size of the application, demand for the NaSH product, the cost of caustic, the relative amounts of CO₂ and H₂S in the gas, and the H₂S treatment specification. NaSH solution is used in the paper, mining, tanning, and other industries; the value of this product can vary locally. As the application size becomes larger (with respect to the H₂S throughput rate), the value of the NaSH product becomes more important to economic feasibility.

If conventional caustic scrubbing equipment (e.g., packed or tray towers) is used for gases containing a significant amount of CO₂, it is typical for the CO₂ absorption efficiency to be similar to that of the H₂S; therefore the NaSH product solution would be contaminated by carbonate salts. Although various grades of NaSH exist, the highest value grades typically have less than 3 wt. % sodium carbonate. Therefore, conventional scrubbing technologies are usually not appropriate for caustic scrubbing of H₂S if the gas has a large amount of CO₂ relative to H₂S.

Typical Short Contact Time (SCT) Process Flow and Contacting Devices

A schematic of a typical single-stage SCT application is shown in Figure 1. The sour gas is mixed with the caustic operating solution for a short amount of time, and then the phases are quickly separated in order to halt mass transfer. A portion of the operating solution is continuously bled off as the NaSH product, with the remaining solution returned to the contactor. Fresh caustic solution (typically 20+ wt %) is continuously added to maintain the solution strength and pH.

Figure 1 shows the use of in-line mixers as the contacting device. (Other contacting devices, such as venturis, could also be used provided that the gas-liquid contact is adequately controlled.) The gas and caustic solution flow through the contactor in the vertical, downward arrangement to better promote mixing of the gas and liquid phases, minimize pressure drop, and minimize unwanted extra piping that would provide undesired additional contact time after the contactor.

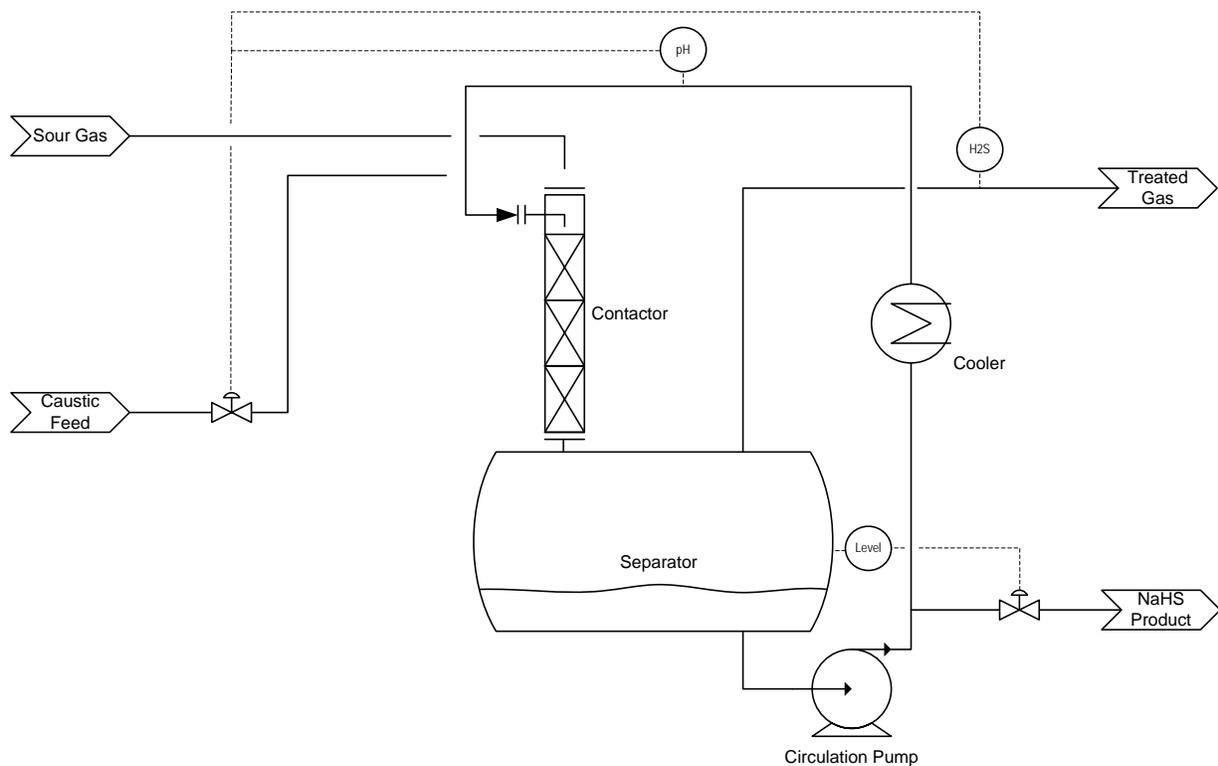


Figure 1. Typical Implementation of Short Contact Time H₂S Scrubbing (Single-Stage)

For some applications, one contacting stage may not be sufficient to achieve the desired H₂S removal while maintaining a high quality NaSH product. For these cases, a two-stage system may be required; this is discussed in a later section.

Potential Applications of SCT Technology

The application of SCT caustic scrubbing should be considered for the following situations:

- Relatively small amount of H₂S to scrub (e.g., less than about 10 - 20 ton/day). In comparison, liquid and solid H₂S scavengers are typically used with low-tonnage applications (<0.6 ton/day), liquid redox and similar biological and chemical processes for combined H₂S removal and sulfur recovery are most frequently used in the 0.2-15 ton/day range, and large-scale amine/Claus/tail gas treating units are usually designed for capacities of at least ~15 ton/day. (Note: the ranges are generally accurate, but with each category of process there are a few examples of applications well outside of the stated ranges.)
- CO₂ is present and it does not need to be scrubbed to meet a CO₂ specification;
- CO₂:H₂S ratios are significant, but not too high (e.g., less than roughly 5:1);
- Product NaSH solution has a potential market, and volume of NaSH to be generated is high enough to merit the added effort of dealing with this product stream;
- The total sulfur to be removed is on the lower end of the aforementioned range and a NaSH solution disposal route has been identified (e.g., down hole, dilution into a wastewater treatment system).

As implied in the last bullet item, even if the product NaSH solution cannot be sold SCT might be merited if one desires to use caustic scrubbing, but the amount of CO₂ relative to the H₂S is high enough to cause carbonate precipitate plugging and/or result in excessive caustic usage. A preliminary economic evaluation can readily be made based on expected H₂S and CO₂ throughputs, caustic cost, and NaSH solution value or disposal cost.

Effect of Chemical Equilibrium on Scrubbing Efficiency and Product Quality

As stated earlier, the key concept in short contact time absorbers is in taking advantage of the faster kinetics of H₂S absorption compared to that of CO₂. However, kinetics of absorption is not the only important issue in the design of these systems; chemical equilibrium, especially with respect to H₂S, must be considered since it can impact the H₂S removal efficiency and the quality of the NaSH solution product.

At high pH values (roughly above pH 12), the equilibrium of Equation 2 moves significantly to the right hand side, resulting in the formation of Na₂S from NaSH. The aqueous equilibria of H₂S and CO₂ in solution are shown in Figure 2; the aqueous species are shown as a function of solution pH. The data in these graphs were produced based on pK_a (25°C) values found in Reference 7. The second pK_a value of H₂S is somewhat uncertain, since a rather wide variety of values were found in the literature; this topic is discussed in more detail in Reference 6.

NaSH solution products are usually required to have low Na₂S content, with 2 to 4 % maximum being common. For this reason, the pH of the caustic solution must be controlled such that the majority of the H₂S will be in solution as the HS⁻ form. If pH is too high, a large fraction of the

HS^- converts to S^{2-} . Solubility of the reaction products is also a concern, since the salts of the different aqueous species of H_2S and CO_2 have different solubilities.

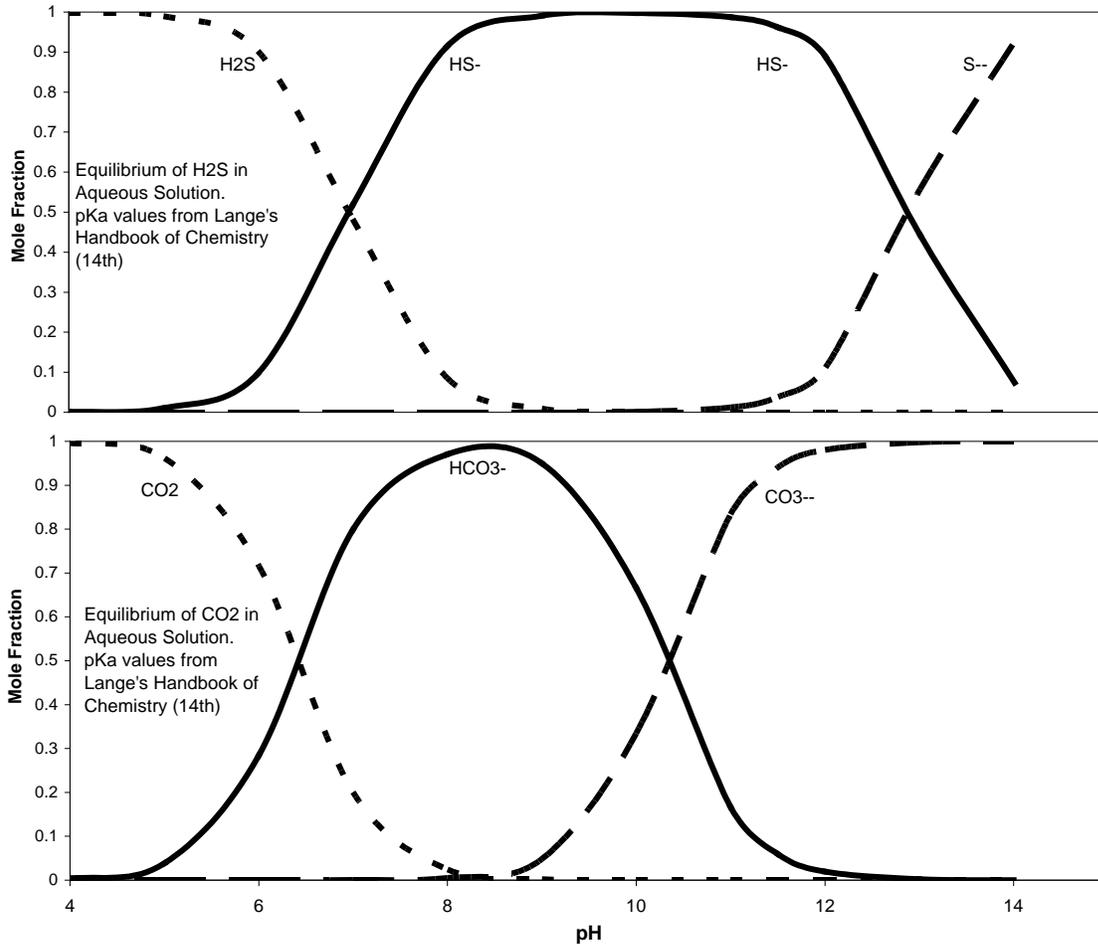


Figure 2. CO₂ and H₂S Speciation in Aqueous Solution (based on pKa values from Ref. 7)

The solubility limits of the product salts are a concern in the design and operation of a short contact time selective scrubber. The primary issues of concern are the precipitation of sodium salts of carbonate and sulfide. Figure 3 shows a schematic of the general trends of salt precipitation in the scrubbing solution as a function of a few variables: solution pH, temperature, CO₂ absorption amount (relative to the H₂S absorption), and the caustic feed strength (i.e., the concentration of NaOH in the fresh caustic feed to the process).

A few conclusions which can be surmised from this graph include:

- *Control of solution properties to prevent carbonate precipitation.* The pH and caustic strength strongly influence the solubility of the salts in the product solution. Assuming a significant concentration of carbonate in the solution, it is important to maintain the solution in the basic region in order to avoid the precipitation of sodium bicarbonate

salts; these salts are particularly troublesome since they tend to stick to metal surfaces and are not readily dissolved by raising the solution temperature.

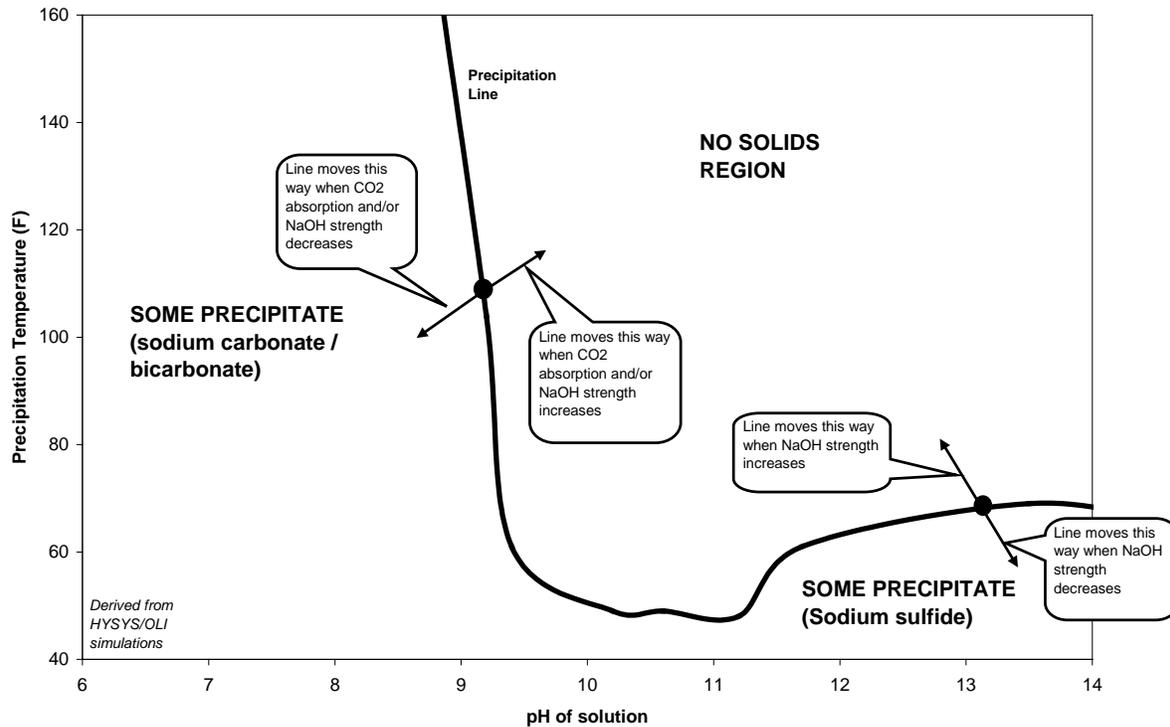


Figure 3. General Trends of Solubility Limits in Scrubbing Solution. (Shows the change in the precipitation line as a function of process variables.)

- *Control of solution properties to prevent sulfide precipitation.* Higher pH values decrease the solubility of sodium sulfide in solution. Although the effect is not as dramatic as on the solubility of carbonate, it is still clear that very high pH values are better avoided since it raises the precipitation temperature. Higher caustic feed strength also decreases sulfide solubility. However, unlike with bicarbonate precipitation, higher solution temperature can be useful for preventing sulfide precipitation.
- *The absorption of CO₂ has a large impact on potential precipitation.* As the absorption of CO₂ decreases, the potential for carbonate salt precipitation is reduced. Achievement of a high selectivity of H₂S absorption over CO₂ absorption is therefore important for a variety of reasons: maintenance of NaSH product quality, minimization of wasted caustic, and minimization of the potential for plugging of the system with carbonate salt solids.

The need to achieve a specific requirement of H₂S content in the treated gas is often at odds with the need to produce a high quality NaSH product, especially at higher operating temperatures.

The equilibrium partial pressure of H₂S in the gas phase with sulfides in the liquid phase increases with temperature, so at higher temperatures the removal of H₂S from the gas phase may be equilibrium limited. Additionally, if the pH range is kept low enough to produce NaSH solution with low Na₂S content, the equilibrium partial pressure of H₂S may limit the gas treat efficiency.

For cases in which the gas must be treated to a moderately low H₂S level (i.e., <160 ppm, as is required for refinery fuel gases in some cases) in the presence of substantial amounts of CO₂, while still maintaining good NaSH quality, it may be necessary to design a multi-stage system (Figure 4). Low H₂S concentration in the treated gas *can* be achieved in a single-stage system, but the operating solution pH required to achieve this may be rather high; the high solution pH results in elevated levels of Na₂S in the NaSH solution and can also result in higher levels of carbonates.

A two-stage system would be designed with the fresh caustic fed to the second stage (relative to gas flow path) and the second-stage solution fed by overflow to the first stage. The second stage can thus be operated at a higher pH (e.g., 13 - 14) than the first stage (e.g., 10-12), resulting in the capability to achieve deeper H₂S removal while maintaining NaSH product quality. pH control in the first stage is critical since too high a pH can result in higher than desired Na₂S concentrations and possible precipitation of this solid from solution; too low a pH can result in the precipitation of sodium bicarbonate.

In summary, a few general statements can be made regarding equilibrium, product quality, and scrubbing efficiency:

- Maintenance of proper solution characteristics is critical in order to ensure the desired H₂S removal efficiency and to avoid precipitation of solids. Carbonate salts can precipitate at lower pH range regardless of temperature, while sulfide salts can precipitate at higher pH levels and lower temperatures;
- The H₂S content of the treated gas may be limited by temperature and/or NaSH product quality;
- For cases in which the content of CO₂ in the feed gas is rather high relative to the H₂S it is important to design for, and maintain by proper operation, a high selectivity for H₂S removal. Achievement of a high selectivity limits waste of caustic, allows for a higher quality NaSH product, and limits potential carbonate precipitation problems;
- Two-stage systems may be required for cases in which deep H₂S removal levels are required in the presence of high CO₂. For some cases the liquid product from the second stage can be totally reused as liquid feed to the first stage. For other cases in which it is required to achieve very deep H₂S removal in the presence of a large amount of CO₂, the second stage liquid product may absorb too much CO₂ and would be totally or partially purged as a waste stream.

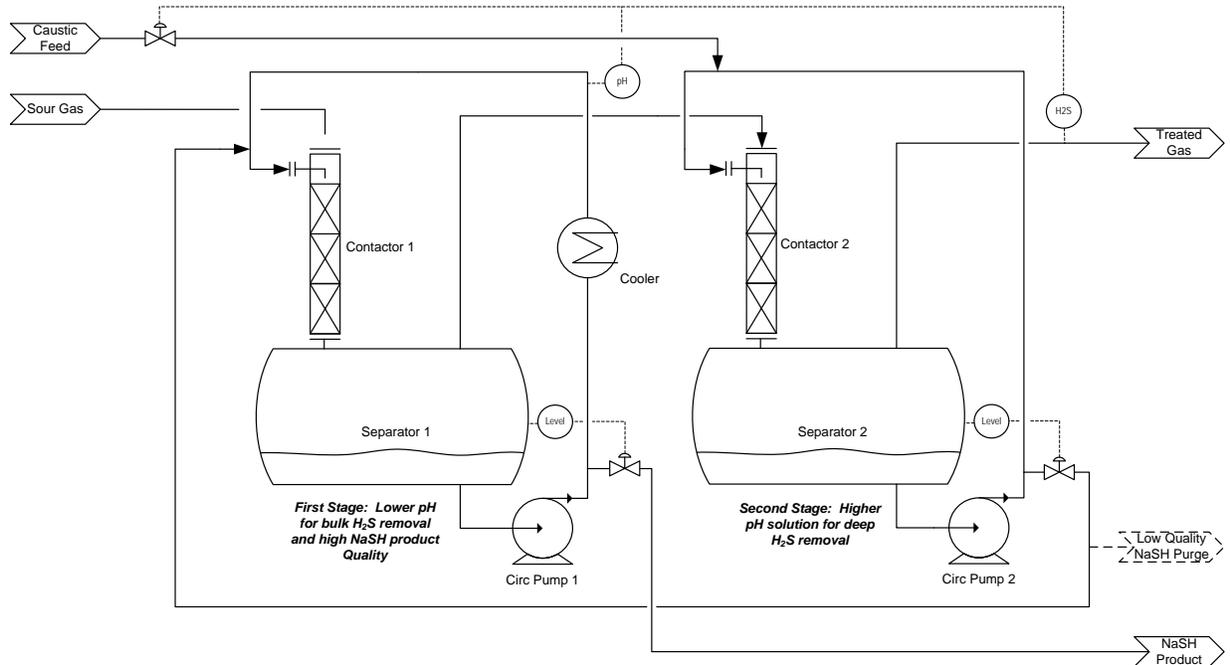


Figure 4. Two-Stage Short Contact Time System

Key Design and Control Aspects of SCT Systems

The essential issue in the design of SCT systems is to limit the gas-liquid contact to create a system where absorption is controlled by kinetics and not by thermodynamic or equilibrium considerations. Static mixers (and some other devices) can achieve intimate contact of the liquid and gas phases with large surface area for mass transfer for a very short amount of time. The phases then must be disengaged quickly in order to avoid too much CO₂ absorption. Some key design and control features for SCT systems are discussed below.

Gas-Liquid Contact – Venturi's, concurrent spray contactors, and in-line mixers are among the technologies that can be applied to the SCT system. Superficial contact times in a range of 0.01 to 0.2 seconds are typical. Trimeric has designed SCT systems using vertical, down flow static mixer contactors with SMV elements from Sulzer.

Flexibility in Contactor Design – Since limiting the contact of the gas and liquid phases is a critical issue, if the gas flow rate and/or H₂S and CO₂ content of the gas are not well defined or are known to vary greatly, it is important that the contactor device be designed with some flexibility. For the use of static mixer contactors, the units can be designed with removable sections of contactor elements (see Figure 5). In the figure, the contactor contains two pipe spools. One pipe spool contains the caustic injection nozzle with several elements, while the other spool contains the remaining elements. By rearrangement of the pipe spools and addition or removal of contacting elements, the system can be tuned to the typical gas conditions after startup of the process.

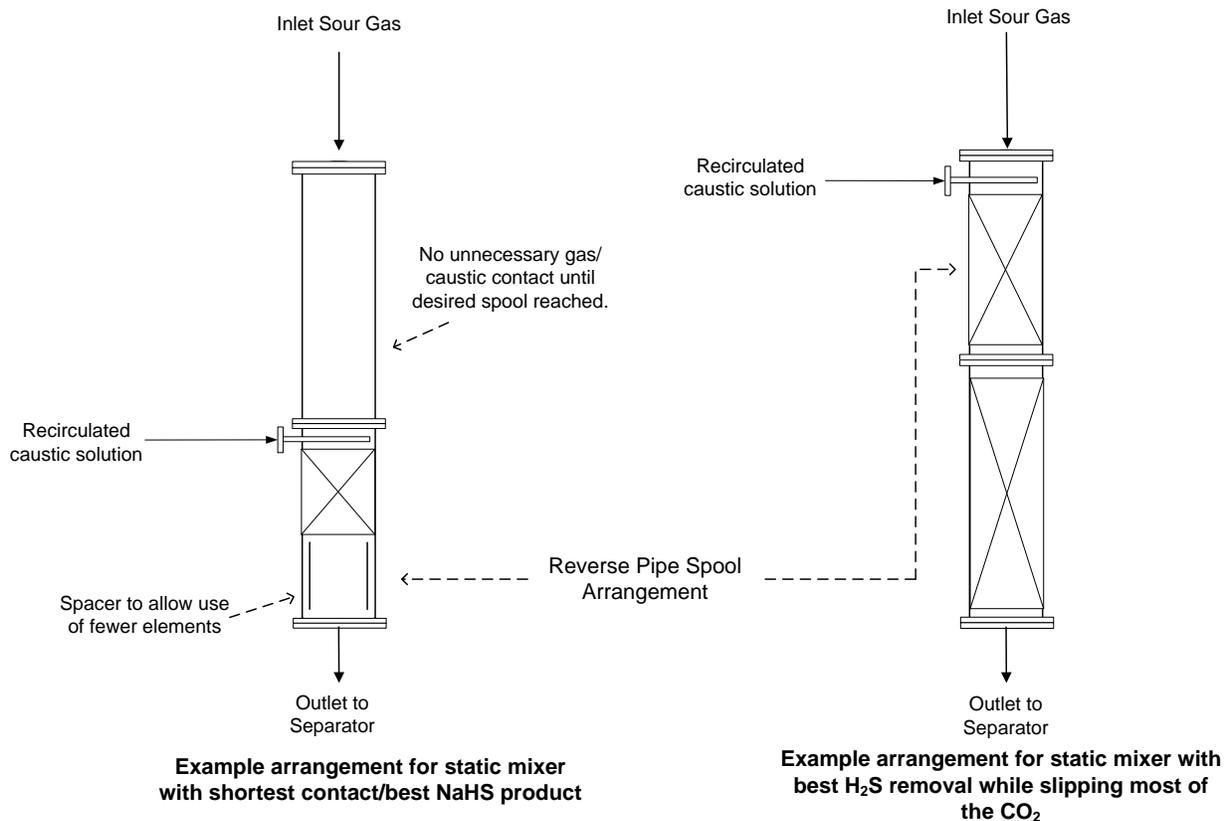


Figure 5. Flexible In-line Mixer Contactor Arrangement.

Vapor / Liquid Disengagement – Another important design criterion is that the gas and liquid leaving the contactor must be disengaged quickly to stop mass transfer. The need for quick disengagement entails that the contactor must be mounted very closely to, or directly on, the vessel. Any piping/fittings necessary between the contactor and the disengagement vessel should be as short as possible. Excessive length of path from the end of the contactor to the disengagement vessel will result in loss of scrubbing selectivity.

Control Strategies – Adequate control of the SCT system is important. The solution quality must be maintained in order to ensure gas treat and avoid precipitation of salts. Addition of caustic to the system can be done via pH control, H₂S control on the outlet gas, or both. If the feed gas is highly variable in nature, then the use of a pH probe may become important. Outlet gas analyzers are also important to aid in controlling the caustic feed and also to ensure that the H₂S abatement requirements are met. If the outlet H₂S content is not continuously monitored, gas samples should be routinely taken and analyzed. It is usually not desired to greatly overshoot H₂S removal requirements from the feed gas; if more H₂S is removed from the feed gas than required, additional CO₂ will also be absorbed, and caustic consumption will be higher than necessary.

Other measurable parameters, such as solution density, can also be considered to aid in troubleshooting and can also be used to control dilution of the caustic feed if desired. Coriolis meters are useful for these systems, because they can be configured to output fluid density and temperature in addition to the mass flow rate.

Control of the temperature of the circulating solution may also be required. For the treatment of cool gases with low concentrations of CO₂ and H₂S, cooling of the circulating solution may not be needed since the treated gas will carry away the reaction heat. For more concentrated acid gases, cooling of the circulating solution may be required in order to maintain low enough solution temperatures to avoid material of construction issues, and to improve the H₂S treat. Implementation and control of the cooling exchanger must be done carefully to avoid precipitation of solid material (carbonates) on heat transfer services. A startup heater or heater to maintain solution temperature when the unit is not processing gas may also be advisable.

Precipitation of carbonate or sulfide salts can occur well above the normal freezing point of water. Therefore, even in warmer climates, heat tracing is essential for liquid-filled dead legs, and all small diameter liquid lines should be (as a minimum) well insulated. Complete insulation and thermostatically controlled heat tracing to maintain desired process temperatures in lines and vessels may be advisable.

Hard piped flushing connections may also be a very good idea for SCT systems, especially if multiple parallel contactors of different sizes are used to handle fluctuations in gas flow rate. When flow is switched from one size contactor to another, the newly-idled contactor can be immediately flushed with warm water to prevent salt crystallization.

Modeling and Performance Prediction

Trimeric uses a few calculation tools for design and performance prediction of SCT units. Apart from routine material balance calculations, two more complex issues are critical: equilibrium and kinetics.

Equilibrium Prediction

Equilibrium calculations for SCT contactors define the limiting case for H₂S and CO₂ removal from the feed gas. Additionally, equilibrium calculations are also used to determine solubility of salts, and the distribution of the various ionic species in solution. The primary software tools used for equilibrium calculations by Trimeric are AspenTech HYSYS with the OLI properties package add-in, and an in-house chemical equilibrium model.

There is a wide range of variability in the literature for the second dissociation constant (pK_{a2}) of H₂S: literature values range from 11.97 to 19. The value of pK_{a2} has a significant impact on the equilibrium modeling of this system with respect to the H₂S that can be achieved, the amount of NaOH required to absorb a given amount of H₂S, the speciation (i.e., relative amounts of sulfide and bisulfide) of the product liquid, and the formation of solid sulfide species. Based on comparison with plant data and some equilibrium data from the literature, a pK_{a2} value of about

14.9 agreed best with the data available to Trimeric. More details on this topic have been published previously (6).

Kinetics Prediction

As discussed previously, the relative kinetics of the absorption of CO₂ and H₂S is the primary reason for the potential selective absorption of H₂S. Selectivity is defined here as:

$$\text{Selectivity} = \text{NTU}_{\text{H}_2\text{S}} / \text{NTU}_{\text{CO}_2}$$

A few different approaches can be used to determine the absorption rates of H₂S and CO₂, and define the selectivity of an SCT unit: predictive calculations based on absorption theory, equipment vendor calculations, and comparison with operating plant data.

It is typically desired to base the design of new equipment on models that have been proven by operating plant data. Given the scarcity of available data from operating SCT units and proven design techniques, Trimeric sought other methods for designing these contactor devices. One equipment vendor (Sulzer) has a proprietary method for designing SCT contactors and predicting CO₂ and H₂S selectivities. These vendor calculations were then compared by Trimeric to a theoretically-based method from the literature; the theoretically-based method is described below.

The calculation of the kinetics of the absorption of CO₂ and H₂S were estimated loosely based on the methods described by Bontozoglou and Karabelas (4, 5). The method involves numerically solving the differential equations that govern the diffusion and reaction kinetics and reaction equilibrium of the various chemical species, as they diffuse between the bulk liquid and the liquid/gas interface subject to appropriate boundary conditions. The resulting concentration profiles of H₂S and CO₂ across the diffusion film are then used to determine the mass transfer enhancement factors for the two gas species.

Trimeric used a slightly different numerical method to solve the equations, using a relaxation method rather than the finite-difference method from the Bontozoglou paper. The relaxation method is computationally simpler, but requires more computer time. Enhancement factors calculated from the kinetic model were used to estimate the HTU of the contactor (with respect to both H₂S and CO₂ separately) and thereby predict the NTU.

It was found that the theoretical model gave selectivities similar to that predicted by the equipment vendor, although the equipment vendor estimates tended to be more conservative (i.e., predicted lower selectivity).

Data from an Operating SCT Unit

An SCT system (similar to Figure 1) was applied to a fuel gas stream at a refinery. The design basis called for the sour gas stream to be highly variable with respect to both flow rate and H₂S content, with H₂S content in the 0.7 – 3.9 % range, and CO₂ around 2.4 – 3.5 %; inlet gas flow

rate was designed to vary from about 370 – 840 Mscfd, at a pressure of about 50 psig. It was desired to remove the H₂S down to rather low levels (<160 ppm).

Initial design calculations indicated that a two-stage unit (similar to Figure 4) was preferred, and would allow for the achievement of the required H₂S abatement with good NaSH product quality for the majority of the feed gas conditions. However, it was decided (at least initially) to build a single-stage unit in order to minimize capital cost and operating complexity. In order to achieve the desired H₂S removal in a single stage, it was required to operate the solution at a relatively high pH; this results in a significant fraction of the absorbed H₂S going to the Na₂S form.

Given the great variability in feed gas conditions, this unit was designed for flexibility: two in-line mixer contactors of different size (2” and 3” diameter) were built; automation allowed for the switching between the two different contactor sizes as a function of gas flow rate. Additionally, the in-line mixers were provided in spool piece sections (similar to that shown in Figure 5) so that an optimal contactor configuration could be easily set up after startup, once the actual operating condition ranges were better defined.

Using the kinetic modeling techniques discussed above, the performance of the SCT system was predicted for a few of cases. A selectivity of 31 was predicted for the average design conditions.

After startup of the plant, data was taken to determine how well the model agreed with the actual operating data. A summary of operating data from the plant is shown in Table 1. As shown in the table, the operating data agreed well with the mathematical model with respect to the selectivity of H₂S absorption over CO₂ absorption.

As shown in Table 1, the H₂S is removed to below desired specifications, while little of the CO₂ is removed, which is what is desired in a selective scrubbing unit. The unit from which this data came has been operating continuously for a few years with no significant downtime due to plugging with carbonates or sulfides.

The strength of the NaSH solution is another factor in the product value. This particular plant was intentionally operating with a rather dilute solution (20% source NaOH typically diluted to 10 - 15%) in order to minimize problems due to the rather extreme fluctuations in feed gas properties and flow rate. Given large concentration changes occurring over a time span of less than one hour, it is difficult to adequately control circulating liquid quality such that the potential for solids precipitation is reduced or eliminated. Operating at a low NaOH feed concentration proved to be an effective technique for minimizing the problems introduced by the rapid feed gas changes.

Table 1. Operating Data from an SCT Unit				
<i>Variable</i>	<i>Units</i>	<i>Data Point 1</i>	<i>Data Point 2</i>	<i>Date Point 3</i>
<i>Inlet Gas Properties</i>				
Flow Rate	MSCFD	490	457	494
Pressure	psig	55.3	51.9	54.9
Temperature	F	89	93	94
H ₂ S	Vol %	2.73	1.74	1.63
CO ₂	Vol %	3.36	3.4	3.43
<i>Circulating Solution</i>				
Temperature	F	129	135	138
<i>Outlet Gas Properties</i>				
H ₂ S	ppm	66	149	50
CO ₂	Vol %	2.80	2.97	2.79
<i>Gas Treatment Calculated Values</i>				
H ₂ S removal	NTU	5.65	4.58	5.32
CO ₂ removal	NTU	0.18	0.14	0.21
Selectivity		31	34	26
<i>Circulating Solution Properties</i>				
NaSH	Wt %	5	7.8	8.2
Na ₂ S	Wt %	7.3	6.8	5.9
Na ₂ CO ₃	Wt %	2.4	2.7	3.8
pH		12.8	12.4	12.7

Conclusions

Short contact time technology can selectively remove H₂S from gas streams contaminated with CO₂ using caustic. Recognition of the importance of various chemistry issues and process limitations is essential for designing and operating SCT plants effectively.

The contactor design must provide for limited contact of the gas and liquid over the full range of operating conditions.

The proper pH range and temperature of the operating solution must be maintained in order to avoid problems with salt precipitation, to ensure adequate H₂S treat, and to achieve the highest quality NaSH product. Good control of the process and frequent solution quality monitoring is essential.

Data from an operating plant was used to show good selective removal of H₂S in the presence of CO₂; the plant data agreed well with model predictions.

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