

Eliminating guess work

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Process simulation has become a commonplace tool in plant design and plant operations analysis. In most commercial hydrocarbon fractionation operations, it provides a fairly close approximation to what is observed in the field. One of the important factors contributing to its success is the engineer's ability to estimate reasonably well the tray efficiencies and the HETPs of the column internals being used. This is because hydrocarbon systems are close to thermodynamically ideal, and efficiencies are well documented for such systems. Chemicals fractionation is less ideal, sometimes to the extent that efficiencies are hard to estimate without field experiences. The complexity and non ideality of amine based gas treating applications is not evident from the apparent simplicity of typical process configurations. However, sensible estimates for the CO₂ and H₂S mass transfer rates, tray efficiencies and HETPs very often cannot be made at all. In fact, these parameters

change with such variables as acid gas loading and temperature, so much so that efficiency and HETP usually show enormous variation across the height of a column.

On the timescale of chemical engineering innovation, gas treating aimed at both the selective removal of H₂S, with either maximum (or targeted) rejection of CO₂, and the use of promoted solvents for bulk CO₂ removal, are recent developments. While total acid gas removal processes are simpler to analyse, build, and operate, more often than not their economics are poorer. This frequently makes selective H₂S and CO₂ removal the preferred approach. The goal in selective H₂S removal is to meet (usually) a ppmv specification on H₂S and either produce a gas containing a maximum specified residual percentage of CO₂ (normally 2 - 3%, which avoids the additional capital and operating costs for the removal of the extra CO₂), or leave as much CO₂ in the gas as possible, exemplified by tail gas treating. In either situation, column

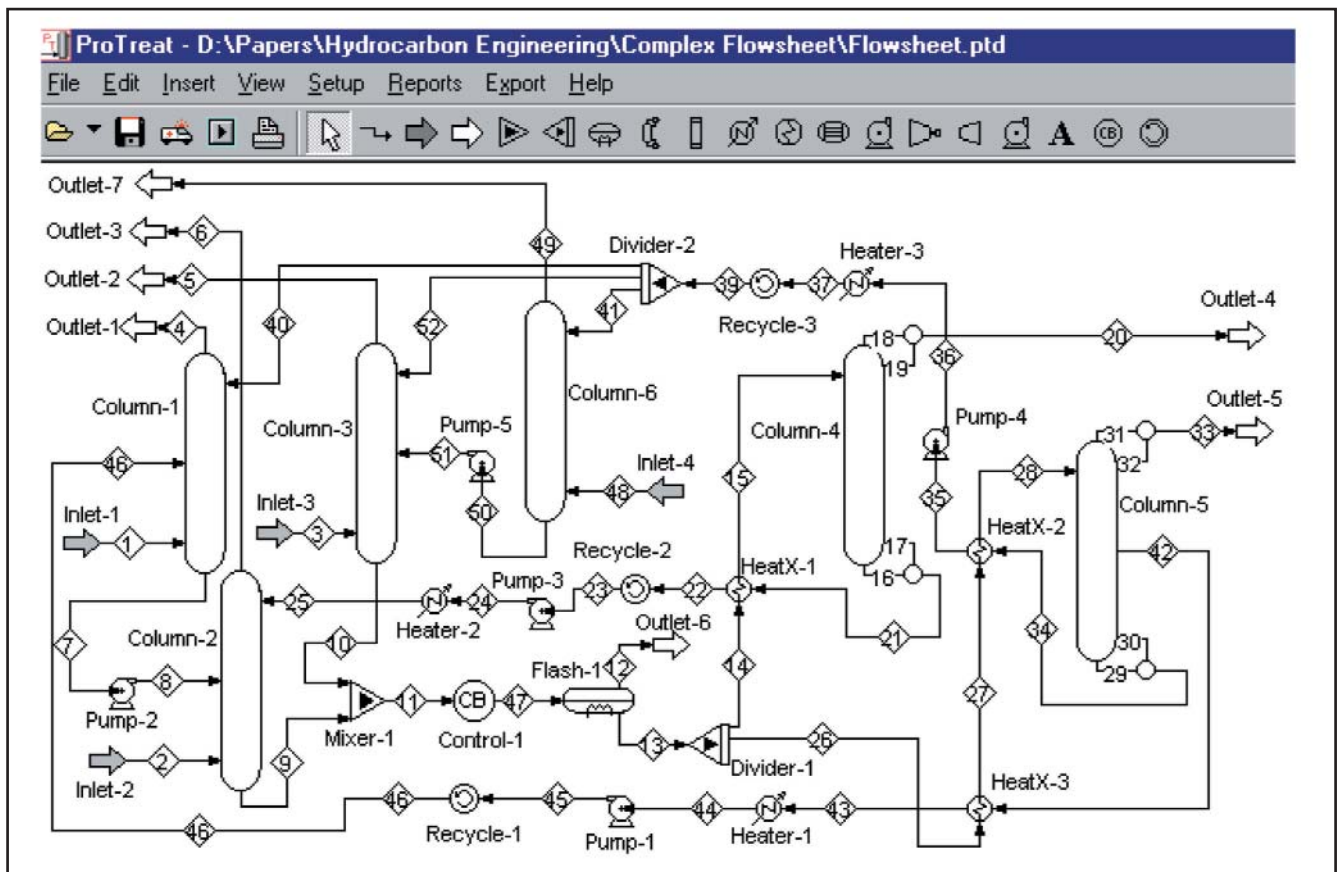


Figure 1. Complex flowsheet.

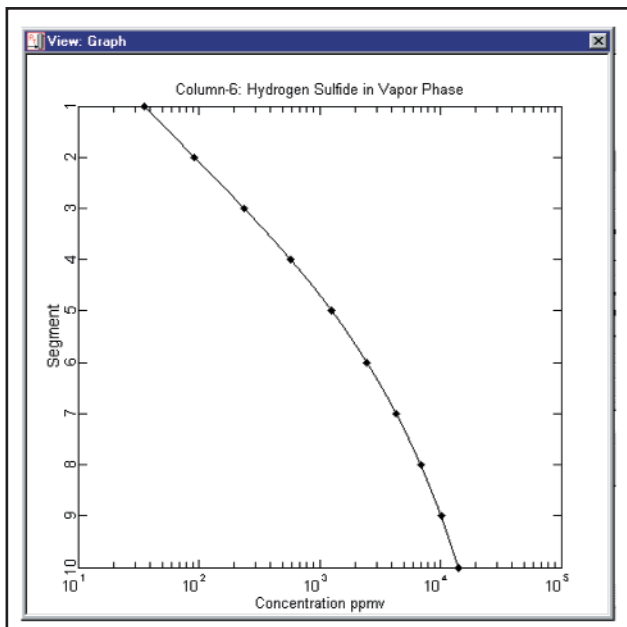


Figure 2. Tail gas unit H₂S profile.

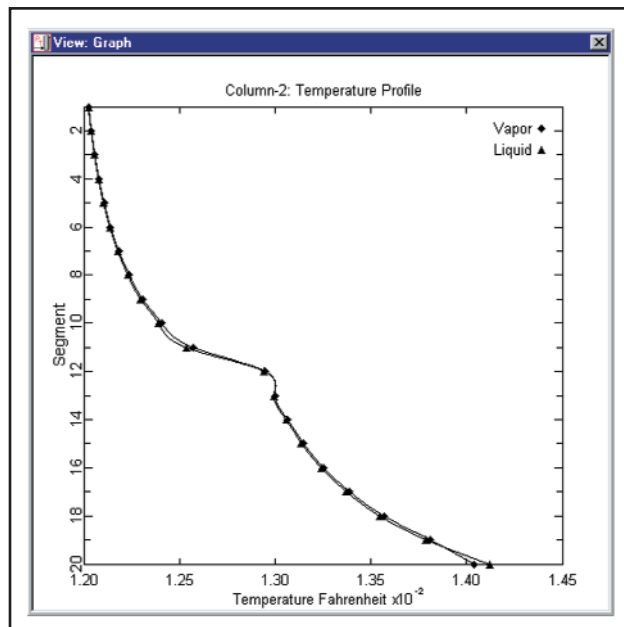


Figure 3. High pressure absorber temperature profile.

over design is unacceptable, because firstly, a column with too many trays or too much packing will remove too much CO₂, and secondly, because the additional CO₂ absorption can result in failure to meet the H₂S specification.

Equilibrium stages vs. mass transfer rates

The rigorous, mass transfer rate approach used for all column calculations by *ProTreat* modelling entirely eliminates the need for empirical adjustments to simulate new applications correctly. The *ProTreat* mass transfer rate model is built on five key elements:

- Mass and energy balances around individual phases on a tray or in a packed segment.
- Conventional thermodynamic phase equilibrium.
- Equilibrium across interfaces.
- Chemical kinetics effects on mass transfer rates, particularly in the liquid phase.
- Mass and heat transfer rate models for transport across interfaces.

In contrast, even the most sophisticated equilibrium stage model uses only two of these elements, namely mass and energy balances around an entire ideal stage, plus thermodynamic phase equilibrium. Often, an attempt is made to include reaction kinetics by empirical modelling via an adjustable parameter (H₂S and CO₂ tray efficiencies and/or liquid residence times), that forces the simulation to reproduce a conventionally operated column's treated gas composition. Some might call this a 'rate' model but it is not a mass transfer rate model, it is an attempt to force a reaction parameter into a model that is inherently unrelated to rate processes in order to match some measure of plant performance. The fact is, regardless of any embellishments one might add, equilibrium stage models are non predictive. This makes them unable to deal with applications dependent on differences between mass transfer rates of CO₂ and H₂S, for which operating experience is lacking. Furthermore, all equilibrium stage models deal exclusively with ideal stages, with no accurate way to translate into the real world of actual tray counts and packed bed depths, let alone to deal with such questions as the effect of packing type, size and material, or tray type and configuration.

In a mass transfer rate model, there are intricate inter-related effects between the five key elements. They respond by a variety of chemical and physical, phase and component transport properties such as:

- Chemical kinetics.
- Diffusion coefficients, solvent viscosity, and other transport properties.
- Salting out effects on solubilities.
- The mass transfer characteristics of the actual hardware being used.

When a *ProTreat* column simulation is run, the tower is being modelled in full detail as a piece of real equipment, not as an idealisation. From a separations standpoint, two-pass and single-pass trays perform differently. Metal packing gives different results from plastics and ceramics. In addition, all solvent properties, including the changes in these properties caused by acid gas loading, affect mass transfer coefficients and thereby influence the actual separation that a given column under a given set of operating conditions will achieve. A mass transfer rate model is rich in detail and is a faithful mirror of the real world. In the language of process control, a true mass transfer rate simulation uses a distributed parameter model that consequently is mechanistic, detailed, and fully predictive. This is in contrast to the equilibrium stage approach which is a lumped parameter model (it assigns all the physical and chemical complexities to one or two parameters such as efficiency, or liquid residence time), and is accordingly non predictive.

Understanding what affects selectivity

In processes where H₂S and CO₂ are present together and selectivity towards H₂S removal is an issue, the extent to which each gas is removed in the column is related directly to the rate at which that species is absorbed by the solvent. So, what affects mass transfer rates? If we rely on an equilibrium stage model for the answer, we will be disappointed because rate is not part of the equilibrium vocabulary, no matter how much we embellish the model. In an equilibrium stage model, phase equilibrium and the stage-to-stage L/V ratio are logically the only factors affecting the separation. However, in reality, these parameters are not

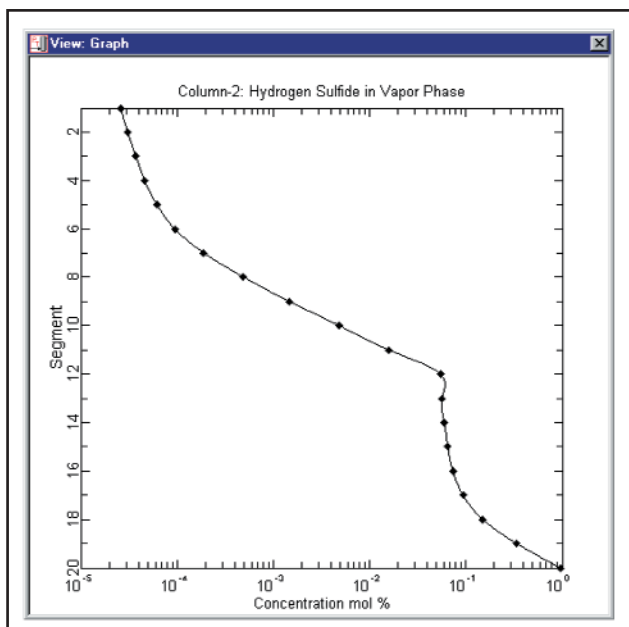


Figure 4. High pressure absorber gas-phase H₂S profile.

more important than reaction kinetics, diffusion, reaction equilibrium, and especially the mass transfer characteristics of the tower internals. Why are these other factors so important in reactive absorption?

The key to understanding the selectivity of a particular process is appreciating the fact that all alkaline solvents are thermodynamically selective towards CO₂ but kinetically selective towards H₂S. If process performance depended solely on phase equilibrium, CO₂ would always be absorbed preferentially over H₂S, and selectivity wouldn't be possible at all. However, CO₂ and H₂S react quite differently in solution, and their absorption rates are controlled by resistances in entirely different phases.

CO₂ forms reaction products with the amine in the solvent by binding chemically to the amine at finite rates of reaction. Consequently, after a long enough time, very little CO₂ remains free (unreacted) in solution and the CO₂ back pressure is very low. (It should be noted that the CO₂ back pressure is determined by the concentration of free, molecular, unreacted CO₂ in solution, not by the total CO₂ content of the solution. Furthermore, the reacting species are free physically dissolved CO₂ and unreacted molecular amine and free CO₂ is always at a very low concentration because it is a sparingly soluble gas.) The slowness of the CO₂ reaction can be used to advantage in achieving selectivity because when short contact times are used, only part of the CO₂ has a chance to react, the balance passes through the contactor in the treated gas. CO₂ absorption is itself controlled by the reaction-diffusion process in the liquid phase; therefore, changing the reaction kinetics profoundly affects the CO₂ absorption rate.

When H₂S absorbs into an amine, it converts immediately to sulfide and bisulfide ions via simple instantaneous protonation reactions without directly involving the amine at all. The important factor for H₂S absorption is solvent alkalinity, not reaction kinetics. Alkalinity is affected by the amount of CO₂ already absorbed and the amount of molecular (unreacted) amine still available in the solution. Indeed, if CO₂ continues to be absorbed, it will cause the alkalinity to decrease with the result that already absorbed H₂S will be expelled from the solution. In addition, H₂S absorption is controlled by the diffusional resistance in the

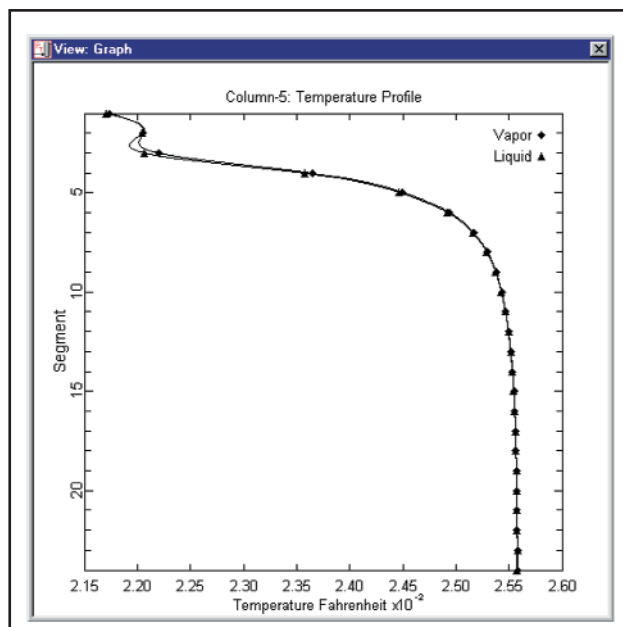


Figure 5. Regenerator (column 5) temperature profile.

gas phase. This gives us another variable to manipulate in achieving a desired degree of selectivity because different types of column internal components have different vapour, and liquid, side mass transfer characteristics. For example, flows over packing typically show much lower liquid-side mass transfer coefficients than trays, which makes them better at rejecting CO₂ because CO₂ absorption is liquid-phase controlled.

The kinetics of CO₂-amine reactions and the fact that CO₂ absorption is liquid-phase controlled are crucial to achieving and controlling CO₂ slip. The careful selection of the amine or amine blend formulation (for kinetic selectivity), as well as the column internals (for equipment selectivity) are important in any optimisation for a desired level of treating. The secret lies in choosing an amine with the right alkalinity and the right reactivity towards CO₂, allowing gas-liquid contact for the right length of time, and using equipment with the right internals. Equilibrium stage models simply cannot capture these effects.

Anatomy of a mass transfer rate model

In principle, operating parameters like temperature and tray hydraulics, chemical kinetics, reaction equilibria, and mass transfer equipment characteristics can be accounted for via their effect on stage efficiencies in the case of trays, or via HETP and HTU values for packing. In actual practice, however, the relationships are so extraordinarily complex in amine-based gas treating that they are beyond our ability to generalise because of the very limited amount of basic data available. In other words, there is no realistic possibility of translating amine-treating experience into valid correlations for efficiency or HETPs. The equilibrium stage approach will therefore remain mired in numbers of ideal stages. For example, tray efficiency depends markedly on the species being transferred (CO₂ vs. H₂S), on the operating conditions (temperature, pressure, acid gas loading of the solvent and phase flow rates) and on the physical characteristics of the tray (geometric factors such as tray type, weir heights, approach to jet flood, not to mention trays versus packing, and so on). The more direct, albeit more difficult, basic approach used by mass transfer rate modelling is not subject to the limitations associated with equilibrium stages.

Table 1. Comparison between equations for mass and heat transfer rates

	Species transfer	Heat transfer
Phase V/inside tube	$R_v = k_g a (y_i^b - y_i^*)$	$Q = h_i A_i (T_i^b - T^w)$
Phase L/outside tube	$R_L = E k_l a (x_i^* - x_i^b)$	$Q = h_o A_o (T^w - T_o^b)$
Equality of fluxes	$R = k_g a (y_i^b - y_i^*) = E k_l a (x_i^* - x_i^b)$	$Q = h_i A_i (T_i^b - T^w) = h_o A_o (T^w - T_o^b)$
Interfacial equilibrium	$y_i^* = H x_i^*$	$T^w = T_o^w$

Notes:

a Vapour-liquid interfacial area (mass transfer)	k_l Liquid side mass transfer film coefficient	i Inside of tube (heat transfer); component identity (mass transfer)
A Surface area of tubes (heat transfer)	Q Heat transfer rate in heat exchanger	L Liquid side of the interface
E Enhancement factor, accounting for effect of reaction kinetics on mass transfer rates	R Mass transfer rate on a tray or segment of packing	o Outside of tube (heat transfer)
h Tube side or shell side heat transfer film coefficient	T Temperature	V Vapour side of the interface
k_g Vapour side mass transfer film coefficient	x Mole fraction in liquid	w At the tube wall (heat transfer)
	y Mole fraction in vapour	* In physical equilibrium at the interface (mass transfer)
	b In the bulk phase (far from the interface)	

The most direct way to develop a feel for the basics of mass transfer rate modelling of columns is by drawing a comparison with heat exchanger design and analysis. Shell-and-tube heat exchangers, for example, are designed using computer software (or sometimes still by hand) that contains:

- A database of correlations for heat transfer film coefficients for the tube and shell sides of the exchanger as functions of hydraulics (as measured for example by Reynolds numbers), tube bundle layout, tube and shell passes, and physical arrangements of shell-side baffles.
- A database of relevant fluid properties such as heat capacity, viscosity, thermal conductivity and density.
- The heat transfer properties of the tubes.

In the simplest case of analysing an existing exchanger without phase change, the general approach is to determine the overall heat transfer rate, or perhaps rates at different positions along the flow paths if properties vary with temperature in some unusual way. What is important to recognise is that the calculation hinges on determining the heat transfer rate. Mass transfer is strongly analogous, but for a number of reasons it is enormously more complex. This is particularly important when chemical reactions play a key role, as they do in amine treating. It scarcely needs to be pointed out that never is there talk of equilibrium heat exchangers, either with or without efficiency. All heat exchanger calculations are strictly rate based, and have been for nearly a century. With the advent of heavy duty computing, mass transfer equipment has become as amenable to completely rational design as is heat exchange equipment.

The equations that describe the transfer of a component 'i' between phases are compared with their heat transfer counterparts in Table 1. The similarities in the equations are quite striking, but of course, there are also differences. One of the important differences is that the bulk phase composition in the liquid is determined by a complex set of reaction equilibrium relations. Another is the appearance of the factor E_i , called the enhancement factor. This is a dimensionless number that accounts for the effect of reaction on diffusional mass transfer, found by solving the differential equations that describe the particulars of that sub process. There are literally dozens of different sets of kinetics for which solutions are available, with each in the context of several theories of mass transfer such as the two film theory, Danckwerts surface renewal theory, and so on. A third difference is that although temperatures in the two phases at the tube surface are the same (in the absence of significant tube wall resistance and scale), the concentrations of an acid gas on the two sides of an interface are related by physical solubility. Yet another difference, and this is the one responsible for the almost one century lag between heat and mass transfer, is

the large number of chemical species versus only one temperature variable. Thus, mass transfer involves an order of magnitude more equations than heat transfer, and to make matters worse, the majority of the equations (e.g., reaction equilibrium) are non linear. But the mass transfer rate equations can be solved for complete columns and entire flowsheets containing multiple columns quite routinely and in computing times that have shrunk to the point where time is no longer an issue.

While modelling of absorber operations has traditionally received the most attention, it is equally important to be able to model solvent regeneration accurately. There are two major

concerns involved in the regeneration of the rich amine solvents. The first is the need for an accurate prediction of the H₂S and CO₂ loadings of the lean solvent produced in the regeneration step. This is critical to an absorber's ability to meet treated gas specifications. The second concern is the ability to predict accurately the amount of reboiler heat duty (energy requirement) needed to obtain the desired level of solution regeneration. Simulating regenerators is inherently more difficult for all models due to 'pinch' conditions at the bottom. Equilibrium stage models don't work very well here either because the chemical reactions affect mass transfer in just as important ways as in absorption. None of the trays in a stripper comes even close to an equilibrium stage. The desorption rate of each acid gas and its concentration in the vapour and liquid phases influences the stripping of the other. From a technical standpoint, the *ProTreat* stripper model treats amine regenerators every bit as rigorously as absorbers. It produces the best possible predictions of stripper performance, without the need for empirical adjustment parameters. When the absorber and stripper models are tied together in a recycle flowsheet, one obtains the best possible prediction of total treating plant performance, again without the use of empirical corrections of any kind whatever. This complete freedom from gross empiricism permits the reliable design of new facilities for which absolutely no operating data or prior experience exists.

The *ProTreat* package does the mass transfer equivalent of heat exchanger design calculations in the setting of acid gas treating with amines. In this model, the mass transfer coefficients for the two phases are calculated from film coefficient correlations for specific trays and packings. The correlations depend on tray type (bubble, sieve, valve), tray geometry (weir height and length, tray active area), on packing type (Intalox saddles, Pall rings, Cascade Mini-Rings, and so on), packing size and material (metals, plastic, ceramic), and on the flow rates and physical properties of the phases. Even the effect of acid gas loadings on physical and transport properties is included. The *ProTreat* mass transfer rate model is detailed and mechanistic and does its calculations in the context of a fully flexible flow sheeting environment.

Case studies

Three sets of examples are presented. These are:

- A somewhat complex, integrated plant containing four contactors, two regenerators and a flash unit.
- A packed column treating a high pressure gas with a hypothetical three amine mixture.
- A commercial absorber removing CO₂ from high pressure natural gas.

Case study 1: Complex flowsheet

For economy of presentation, a number of cases of absorption and regeneration have been combined into the single flowsheet shown in Figure 1 and several of these columns are examined here in detail. The purposes of this example are to show the importance of flexibility in a flowsheeting package and to indicate how a number of different operations can be very effectively integrated. With the exceptions of Control and Recycle, the unit operations blocks in the Figure have names that make block functionality self explanatory. Control is a block (usually one per flowsheet) that allows the solvent composition and flow rate to be fixed, monitored, and controlled somewhere in the plant. Solvent makeup requirements are also reported here. The *ProTreat* simulator solves recycled flowsheets iteratively and sequentially. The blocks labelled Recycle permit input of an estimate for stream composition to initiate the solution process and act as stations where progress to a solution is monitored. The sequence in which blocks are to be solved is internally generated.

In this example, the entire processing scheme uses a nominal 45 wt% generic MDEA solution for treating gas streams ranging from tail gas (Column 6) to 900 psig natural gas (Column 2) at H₂S to CO₂ ratios from 2.5 (35 mol% total acid gas) to 0.25 (10 mol% total acid gas). Three of the absorbers (Columns 1, 2 and 3) operate in split flow mode and the rich amine from two of them (1 and 6) is used as mid column semi lean feed for two others (2 and 3, respectively). One of the regenerators is used in split flow mode with the semi lean stream (about 40% of the rich feed to the regenerator) being used as intermediate feed to a low pressure absorber (Column 1); the fully stripped solvent from this regenerator is fed to the top of the tail gas unit and the low and intermediate pressure contactors.

The tail gas unit is packed with 30 ft of FLEXIMAX 400 packing and is modelled as 10 segments, each containing 3 ft of packing. The column is automatically sized for 80% flood. It operates at a bottom pressure of 3 psig and treats 2 MMSCFD of Claus-plant tail gas at 100 °F containing 8% CO₂ and 2%

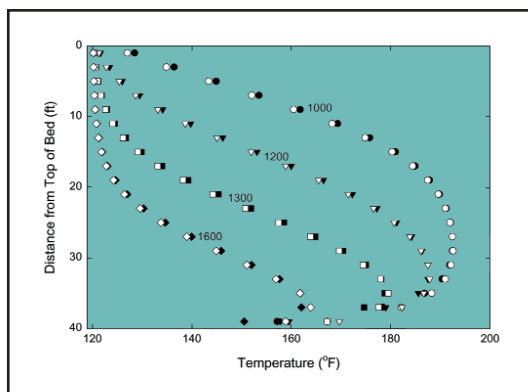


Figure 6. Effect of solvent flow on packed bed absorber temperature profiles.

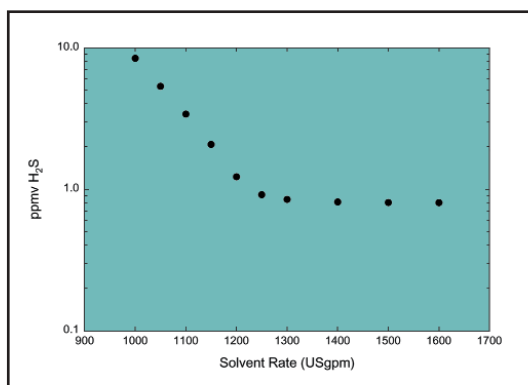


Figure 7. Effect of solvent flow rate on H₂S content of treated gas.

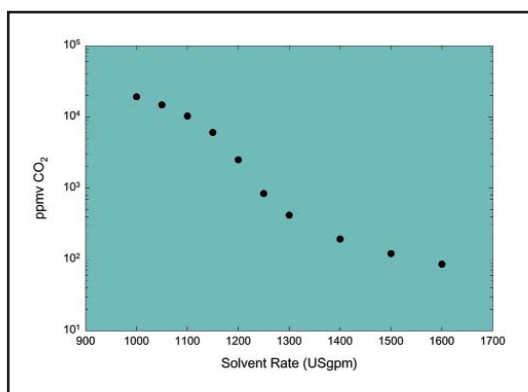


Figure 8. Effect of solvent flow rate on CO₂ content of treated gas.

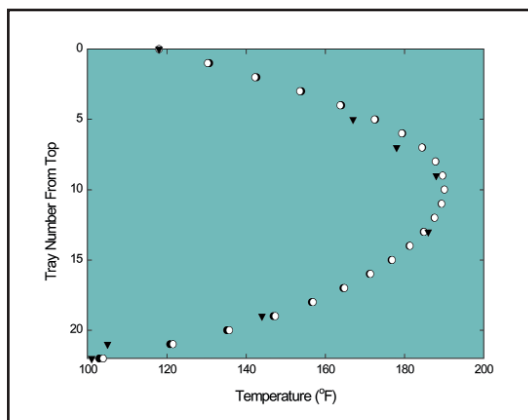


Figure 9. ProTreat modelled temperature profile vs. plant performance measurements.

H₂S in nitrogen. Solvent is the fully stripped amine from the reboiler of Column 5 at 80 USgpm, 120 °F, containing about 49 wt% MDEA loaded to 0.00024 and 0.0019 mol/mol H₂S and CO₂, respectively. The column picks up very little acid gas (total loading increase of only 0.0275) but, as shown in the plot of Figure 2, it manages to produce a 35 ppmv H₂S treated gas. It also slips fully 97% of the CO₂. The rich solvent leaving this contactor is actually still quite lean and is perfectly suitable as a solvent for injection into the middle of Column 3. The vapour phase H₂S profile is typical of H₂S removal using amines, the H₂S concentration falls off almost logarithmically with position in the column. Note that the concentration shown for any segment is the concentration of the gas leaving that segment. Thus, the gas leaving segment 7 is about 4000 ppmv which is the concentration at a position in the column 18 ft from the top of the packing (the bottom of segment 7 is 21 ft below the top of the bed).

Column 3 presents an interesting case of the effect of using a semi lean amine to a mid column tray to do bulk removal in the bottom of a column and a fully regenerated lean amine stream to the top of the absorber to do the final cleanup. This column uses 20 generic valve trays to process 5 MMSCFD of 900 psig natural gas containing 10% CO₂ and 3% H₂S. Treated gas is 0.26 ppmv H₂S and 0.125% CO₂. The tray-to-tray temperature and gas-phase H₂S profiles are shown in Figures 3 and 4, respectively. The semi lean stream to tray 12 is somewhat hotter than the liquid from tray 11 and this causes the temperature on tray 12 to rise several degrees more than it otherwise would. The temperature and H₂S profiles track each other because, of course, the temperature rise of the solvent is directly related to the amount of acid gas absorbed. Note that these profiles show conditions on the actual trays in the column. The mass transfer rate model even distinguishes between the temperatures of the vapour and liquid on the same tray. Although in this particular instance, the differences are not great, in some cases they are.

Sour gas		Lean solvent	
Temperature (°F)	100	Temperature (°F)	120
Pressure (psig)	1000	Pressure (psig)	1050
Flow (MMSCFD)	100	Flow (USgpm)	Various
H ₂ S (mole %)	0.25	H ₂ S (mol/mol loading)	0.0005
CO ₂ (mole %)	10.00	CO ₂ (mol/mol loading)	0.05
CH ₄ (mole %)	80.00	MDEA (wt%)	17
C ₂ H ₆ (mole %)	5.00	DEA (wt%)	15
C ₃ H ₈ (mole %)	3.00	Piperazine (wt%)	8
C ₄ H ₁₀ (mole %)	1.75	Hydrocarbons	Nil
Water (% saturation)	100		

	Sour gas	Lean solvent
Temperature (°F)	80	118
Pressure (psig)	910	910
Flow (MMSCFD or USgpm)	48.76	175
Carbon dioxide (mol% or loading)	1.99	0.037
Methane (mol%)	85.44	0
Ethane (mol%)	6.66	0
Propane (mol%)	2.36	0
n-Butane (mol%)	0.77	0
i-Butane (mol%)	0.89	0
n-Pentane (mol%)	1.70	0
Nitrogen (mol%)	0.19	0
DEA (wt%)	0	15
MDEA (wt%)	0	28

Because gas plant performance is markedly affected by the quality of the lean amine being fed to the top of the absorbers, it is just as important to be able to simulate regenerators accurately, as it is absorbers. Column 5 is a 24 tray regenerator with rich amine to tray 3 and a 40% semi lean amine draw from tray 14. Its temperature profile is shown in Figure 5, where it can be seen that the top 2 trays are used to heat reflux, but it takes the top 10 trays for the solvent to reach anything approaching a constant temperature. Using the semi-lean amine drawn from Column 5 for bulk acid gas removal from the very sour low pressure gas being fed to Column 1, then feeding the top of Column 1 with a portion of the very well stripped lean amine from the same regenerator, permits extremely good H₂S removal. Split flow solvent regeneration produces an unusually high effective stripping ratio in the bottom half of the regenerator.

Case study 2: Mixed solvent contactor

The second example is a hypothetical case using a three component solvent (17 wt% MDEA, 15 wt% DEA plus 8 wt% piperazine), to remove H₂S and CO₂ from a high pressure natural gas described in Table 2. The column was simulated for solvent flows ranging from 1000 - 1600 USgpm in 100 USgpm increments. It contained 40 ft of FLEXIMAX 400 steel packing and the column was sized for 80% flood.

The results of this set of simulations reveal several interesting aspects of treating with a mixed amine solvent. First, the temperature profiles shown in Figure 6 at four solvent rates show that the temperature profile is extremely sensitive to solvent flow. At 1000 USgpm, the column produced a treated gas containing 8 ppmv H₂S and a residual CO₂ content of just under 2%; at solvent rates above 1100 USgpm, the H₂S content was less than 4 ppmv (usually less than 1 ppmv); the residual CO₂ in the treated gas

ranged from 100 ppmv (at 1600 USgpm) to 1.0% (at 1100 USgpm). As the solvent flow is reduced, the temperature bulge shifts up the column and becomes increasingly wider, so that at 1000 USgpm, most of the column is experiencing high temperatures. The large broad temperature profile is a result of both the low L/V ratio and the high acid gas loadings through most of the column. The higher solvent flows carry an increasingly higher proportion of the heat of absorption down the column and this moves the position of the temperature bulge downward; however, the CO₂ absorbed at low solvent flow rates causes higher loadings, and therefore it produces a hotter solvent so the temperature at the bulge itself is higher.

As shown in Figures 7 and 8, solvent flow rate has a profound impact on both the H₂S and CO₂ contents of the treated gas. Variability in solvent flow rate has little or no effect on the treated gas when the solvent flow is high. However, as it is reduced, a value is reached (approximately 1300 USgpm), below which the H₂S in the treated gas rises exponentially with further reductions in solvent rate. At about this same solvent flow, the residual CO₂ in the gas also begins to rise exponentially. Of course, normal column operation is usually at the lowest possible solvent flow rate, in this example at about 1100 USgpm, where the H₂S can be kept below 4 ppmv. This is exactly where column performance is most sensitive to the amine flow.

Again, it should be emphasised that these temperature profiles and performance predictions are based on the way 40 ft of FLEXIMAX 400 packing would be expected to behave in this particular service. Theoretical stages and HETPs have not been used.

Case study 3: Commercial absorber

The final example compares a *ProTreat* mass transfer rate based simulation with actual field performance data for an operating column. The owners asked that the facility not be identified. Having already seen how sensitive absorber temperature profiles can be to operating conditions, a good test of any treating model is its ability to predict both observed temperature profiles and treated gas quality.

The gas and lean solvent are as shown in Table 3. The solvent is a blend of 15 wt% DEA and 28 wt% MDEA. The 5 ft diameter contactor contains 22 single pass sieve trays having 8% open area and treats the gas to about 1600 ppmv CO₂. The simulated temperature profile is shown in Figure 9 where it is compared with the temperatures measured in several downcomers.

The agreement between the mass transfer rate model and the measured data is excellent. This is especially the case in light of the sensitivity of temperature profiles to operating conditions, as discussed in the preceding example.

Conclusion

Mass transfer rate based modelling of amine treating operations produces the most accurate facsimile possible of the actual physical behaviour of the columns and the effect of the details of their internals on performance. In the fully flexible flowsheeting environment of the *ProTreat* package, detailed performance predictions can be easily obtained in the familiar Windows environment.

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