Sulfur Recovery Process Fundamentals

TECHNICAL PAPER

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1.0 INTRODUCTION

Sulphur recovery by the modified-Claus process is universally practised. Although the process remains the same, the equipment and plant configuration varies with each application. This paper is a review of the process fundamentals and the equipment and configurations used in the recovery of sulphur from acid gas streams consisting mainly of $\rm H_2S$ and $\rm CO_2$. The process is discussed in detail by Paskall (1) and much of this paper is taken from that publication.

The process reaction is carried out in two steps:

The first step is a free-flame total oxidation of one-third of the ${\rm H_2S}$ to ${\rm SO_2}$ and the second step is reaction over a catalyst, of the ${\rm SO_2}$ thus formed, with the remaining two-thirds of the ${\rm H_2S}$.

$$3H_2S + \frac{3}{2}O_2 \xrightarrow{950-1200 \text{ °C}} 5O_2 + 2H_2S + H_2O + 520kJ$$
.
$$\begin{cases} \text{catalyst} \\ 170-370 \text{ °C} \end{cases}$$

Heat is recovered from the process at the intermediate stage in order to allow operation at lower temperatures in the catalytic stages.

2.0 PROCESS CONFIGURATIONS

The configuration of the plant is dependent on the acid gas feed to be processed. Amine acid gas feed streams generally have $\hat{\rm H}_2{\rm S}$ and ${\rm CO}_2$ as the two principal constituents with lesser amounts of ${\rm H}_2{\rm O}$ and hydrocarbons.

Other streams are processed in the sulphur plant as well, and these might include a sour water stripper off-gas containing NH $_3$, H $_2$ S and H $_2$ O, and sour fuel gas containing principally hydrocarbons with small amounts of H $_2$ S.

The selection of an appropriate configuration is based on the $\rm H_2S$ content of the amine acid gas and will generally include a thermal stage consisting of reaction furnace and wasteheat exchanger/condenser followed by two or three catalytic stages consisting of reheaters, catalytic converters and condensers.

The following table is a general guide covering the total range of applications.

Type of Acid Gas (Percent H ₂ S)	Configuration
50 - 100 (Rich Feed)	Straight through
40 - 50 (Med. Feed)	Straight through with acid gas/air preheat
25 - 40 (Lean Feed)	Split-flow
15 - 25 (Lean Feed)	Split-flow with acid gas and air prehea
<15 (Very Lean Feed)	Recycle Selectox Sulphur Recycle

In all cases for processing gas less than 50 percent $\rm H_2S$, the use of oxygen enrichment of the air stream has recently become more popular (2) and can allow straight through operation in cases where split-flow was previously required. The ranges noted above are by no means rigid and other factors can influence the configuration selection. Injection of fuel gas to boost reaction furnace temperatures for very lean acid gases is also used, however this practice is generally not recommended.

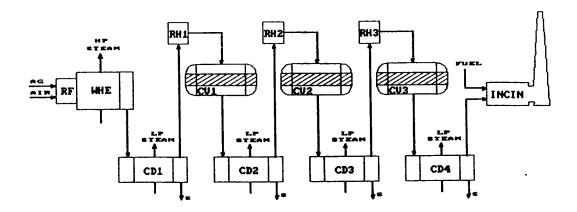
Processing of an additional stream such as sour water stripper off-gas containing NH₃, requires a specially designed reaction furnace and can really only be considered for rich feed plants.

The straight through configuration (Figure 1) is by far the simplest with all the acid gas passing through the flame zone of the reaction

furnace. Significant sulphur production occurs in the reaction furnace and is recovered prior to the catalytic stages.

For medium feed acid gases inclusion of a preheater on the acid gas and/or air enables use of the straight through process.

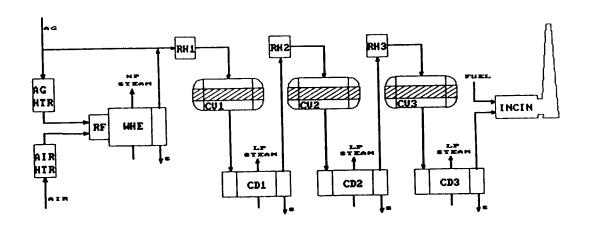
Figure 1



RICH FEED PLANT - STRAIGHT THROUGH CONFIGURATION

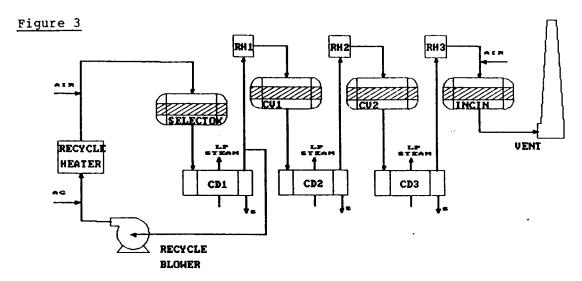
The split-flow configuration (Figure 2) bypasses up to two-thirds of the acid gas around the reaction furnace and wasteheat exchanger where it is mixed with the effluent gases. Depending on the quantity of bypass the production of sulphur in the reaction furnace ranges from 70 percent down to zero. Addition of acid gas and air preheat to this operation provides needed stability and allows processing of leaner gases.

Figure 2



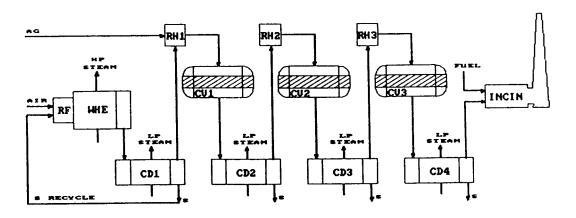
LEAN FEED PLANT - SPLIT-FLOW CONFIGURATION

At levels of $\rm H_2S$ in the acid gas that will not sustain a flame the use of the Recycle Selectox process (3) (Figure 3), which relies on direct oxidation of the $\rm H_2S$ over catalyst, or the sulphur recycle process (Figure 4), which produces $\rm SO_2$ by burning product sulphur, is necessary.



VERY LEAN FEED PLANT - RECYCLE SELECTOX PROCESS

Figure 4



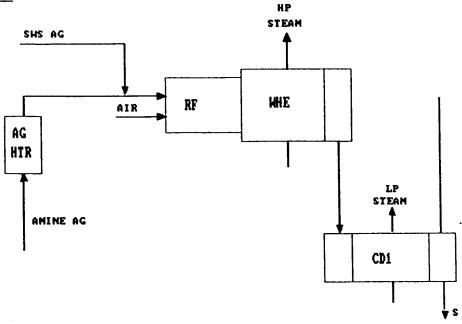
VERY LEAN FEED PLANT - SULPHUR RECYCLE CONFIGURATION

It is a general conclusion that processing rich gas with the straight through configuration is preferred both because of equipment size per tonne of sulphur and because operating problems are reduced considerably.

Two principal methods of burning NH3 are in current use, both requiring high reaction furnace temperatures. The Comprime

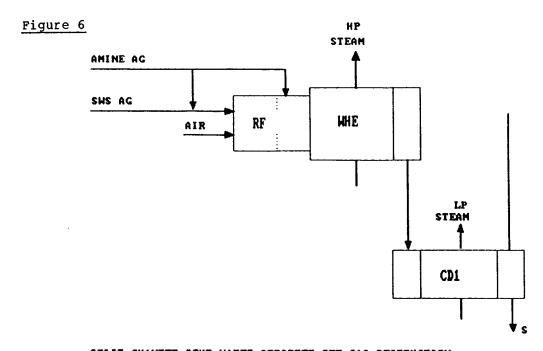
Ammonia Destruction System (Figure 5), relies on a revolutionary burner which achieves very efficient mixing of the air, the preheated acid gas and the sour water stripper off-gas streams.

Figure 5



COMPRIMO AMMONIA DESTRUCTION SYSTEM

The other process (Figure 6) originally patented by Parsons, splits the acid gas to two chambers to raise the flame temperature and as a result has some of the same operating problems as the conventional split-flow configuration. Total destruction of NH3 is required to avoid plugging problems downstream, caused by NH3-salt formation.



SPLIT-CHAMBER SOUR MATER STRIPPER OFF-GAS DESTRUCTION

3.0 THE THERMAL STAGE

In conventional sulphur plants the thermal stage consists of a reaction furnace and wasteheat exchanger. It may also include, depending on configuration, feed preheaters and an effluent condenser.

3.1 Reaction Furnace

The reaction furnace is designed to operate in the 1000-1500°C range with preferred operation in the mid-range. Its key functions are:

- * to burn one-third of the H₂S to SO₂ for subsequent reaction with the remaining H₂S;
- * to produce elemental sulphur directly by the partial oxidation of H₂S; and
- * to destroy any contaminants in the H2S feed streams.

The use of the split-flow configuration for NH₃ combustion or lean feed processing violates the last of these functions as it allows unburned contaminants to pass through to the catalytic converters. The contaminants, such as heavier hydrocarbons, can poison the catalyst, resulting in lower than expected plant efficiencies.

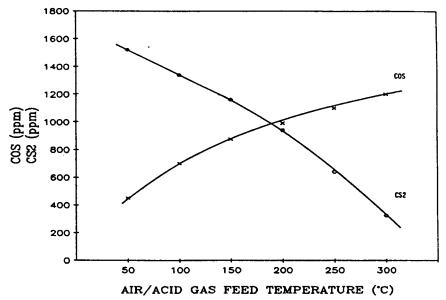
The chemical reactions that can occur in the reaction furnace are numerous and many of the most likely ones have been listed in the literature (1). The fact that the reaction furnace does not generally achieve equilibrium increases the number of possible products in the effluent and these have a great effect on the sulphur plant operation. In the kinetically limited reaction furnace, the principal side products of interest are $\rm H_2$, $\rm CO$, $\rm COS$ and $\rm CS_2$.

The $\rm H_2$ and CO clearly originate from the $\rm H_2S$ and $\rm CO_2$ and as a result affect the air demand for the process stoichiometry. The COS and $\rm CS_2$ are tied to the CO formation and to the hydrocarbon content of the acid gas.

The formation and existence of COS and CS_2 in the furnace effluent is a symptom of kinetic limitation because thermodynamic equilibrium does not predict their presence in any quantity. Since as much as 8 percent of the total inlet sulphur has been measured as CS_2 , and 4.5 percent as COS_1 , in the wasteheat exchanger outlet, the presence of COS_2 and CS_2 is a concern (4). The dependence of these species on operating conditions has been documented through extensive field studies (5) and it has been found that the presence of CS_2 is strongly dependent on the reaction furnace operating temperature. Thus when processing sour water stripper off-gas in the reaction furnace, adding significant preheat to the air and acid gas (Figure 7), or, in the case of the split-flow configuration, increasing the temperature by bypassing significant quantities of acid gas, the quantity of CS_2 produced decreases dramatically.

Interestingly enough, the increased temperature has the opposite effect on COS.

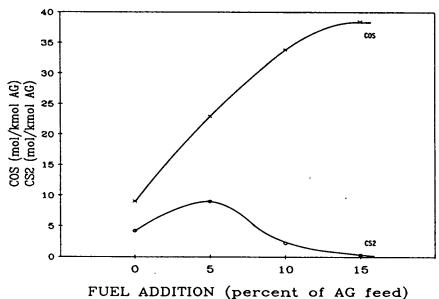




If fuel gas is added to the reaction furnace, the hydrocarbon has the effect of increasing the COS and CS_2 initially, but as the temperature increases further with added fuel gas, the CS_2 content drops off significantly (Figure 8).

Figure 8

EFFECT OF FUEL GAS ADDITION ON COS AND CS2 FORMATION

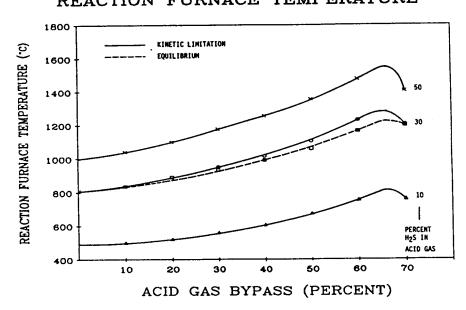


Because equilibrium is not achieved in the reaction furnace, the temperature can vary significantly from values predicted using free energy minimization routines.

In general, the kinetically limited reaction furnace will produce less elemental sulphur and CO than predicted and more COS and CS $_2$. For $_{\rm H}_2$, the quantity existing in the effluent depends on the furnace temperature. In the higher temperature range, $_{\rm H}_2$ production will be less than predicted and in the lower temperature range, more than predicted.

The variation between predicted equilibrium temperatures and actual temperatures is demonstrated (6) in Figure 9 which also shows the relationship between temperature and acid gas bypass for very lean, lean and medium acid gases. These temperatures are based on no preheat of the acid gas or air streams, and since 925°C is regarded as a minimum operating temperature for a stable flame, clearly the very lean acid gas cannot be processed without significant preheat.

EFFECT OF ACID GAS BYPASS ON REACTION FURNACE TEMPERATURE



3.2 Preheaters

Preheaters are used to boost the reaction furnace temperature and usually consist of fired heaters exchanging heat to the air or acid gas by means of banks of tubes. The air can be preheated considerably, however the practical limit is about 400°C. Acid gas cannot be heated above 320°C for fear of H₂S attack on the carbon steel piping. Only the acid gas going directly to the burner is preheated.

3.3 Wasteheat Exchanger/Condenser

The formation of sulphur in the reaction furnace is an endothermic reaction resulting in a temperature drop towards the back of the

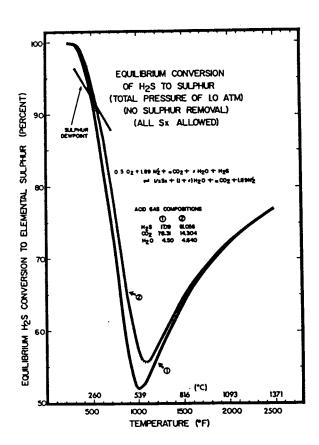
chamber. Generally the residence time in the chamber is 0.7 to 1.5 seconds, and the gas exits the chamber at temperatures in excess of 1000°C. The wasteheat exchanger removes valuable heat from the process generally in the form of high pressure steam. The gases exit the exchanger at approximately 300°C which is above the sulphur dewpoint and therefore minimal sulphur condensation occurs in the tubes.

In rich feed plants where significant sulphur has been produced in the reaction furnace, a condenser is added downstream of the wasteheat exchanger to remove the sulphur as a liquid product. This requires further cooling and the condenser will generally operate at a 150 to 180°C outlet depending on the steam requirement on the shell-side. Lean feed plants that have not produced much sulphur in the thermal stage can pass the wasteheat exchanger effluent directly to the first catalytic stage.

4.0 CATALYTIC STAGES

Free flame conversion of $\rm H_2S$ to sulphur is thermodynamically limited to less than 75 percent at the high operating temperature of the reaction furnace as is shown by the classic Gamson-Elkins curves presented here for a rich and a lean acid gas (Figure 10). Higher conversions are obtained by lower temperatures in subsequent converters.

Figure 10



The catalyst, the low temperature and the removal of sulphur product all enhance the attainment of an overall conversion efficiency greater than 98 percent. The conventional modified-Claus plant operates subsequent converters at lower temperatures, but all above the sulphur dewpoint.

The catalytic stage consists of a reheater, a catalytic converter and a condenser, and most sulphur plants operate two or three of these stages to achieve acceptable recoveries.

4.1 Reheaters

Because the process gas leaves an upstream condenser saturated with sulphur vapour, the stream must be reheated before further sulphur is produced on the next catalyst bed, so that the bed is always maintained above the sulphur dewpoint. Liquid elemental sulphur is also often entrained from the condenser and this must be removed from the stream by means of a mist eliminator (mesh pad), as it will generally not be vapourized in the reheater and will therefore deposit on the bed causing a potential blockage or at least coating the active catalyst.

There are two general categories of reheat (7): direct reheat methods that rely on the mixing of a hotter gas stream with the process stream, and; indirect reheat methods that use heat exchangers.

Capital and operating costs can differ significantly for the various types of reheat available as can the flexibility of operation and the effect on plant recovery efficiency.

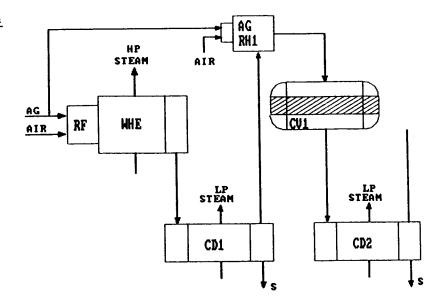
Direct Reheat Methods

The direct methods of reheat have in the past been the most popular because of relative cost and flexibility. The three methods are shown schematically in Figure 11. The most common is perhaps the in-line burner which can be fired with a slip-stream of acid gas or with fuel gas.

Clearly the use of acid gas means that sulphur bearing gases bypass the reaction furnace and some of the catalytic stages and are therefore not taking part in the upstream sulphur recovery process. This results in some loss in plant efficiency. The acid gas fired burner is very flexible in its operation and for rich feed plants the amount of acid gas bypassed is relatively small, hence efficiency losses can be limited to 0.1 to 0.2 percent.

Acid gas burners are fired with sufficient air to allow maximum heat release but minimize the chance of oxygen breakthrough. The typical operating range is 60 to 75 percent stoichiometry, which affects the upstream Claus stoichiometry, forcing slightly deficient operation in the reaction furnace.

Figure lla

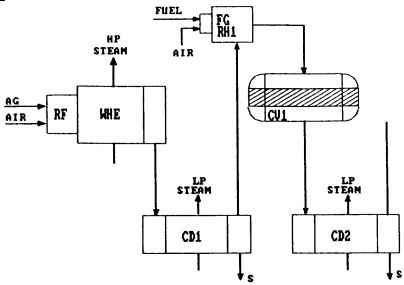


ACID GAS-FIRED REHEATER

Where the acid gas is lean, fuel gas is often used in the in-line burner. This avoids the problem of bypassing sulphur bearing gases, however, complete combustion is now required to prevent unburned hydrocarbon or soot contacting the catalyst. Perfect burn stoichiometry is difficult particularly if the composition of the fuel gas is not stable.

The combustion products from the fuel gas burn dilute the process stream further hindering the Claus reaction. With fuel gas burners the chance of oxygen breakthrough to the catalyst is very high and for this reason this style of reheat can be damaging to the process. Installation of a better quality burner can remove some of the concern but since air to fuel gas control is usually a direct ratio the problem of variable composition is significant.

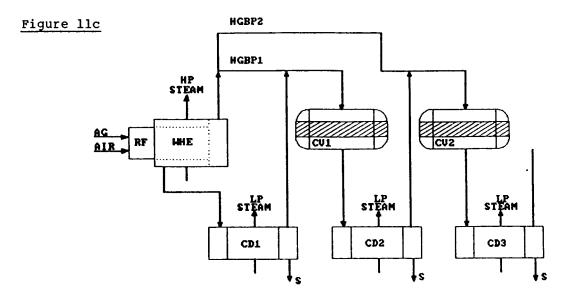
Figure 11b



FUEL GAS-FIRED REHEATER

The third method of direct reheat is the hot gas bypass technique that takes a hot process stream from the first pass of a multi-pass wasteheat exchanger and mixes this stream with the catalytic stage feed. This method although potentially the least expensive presents some serious operating problems.

The hot gas bypass stream, because of its relatively low heating value (typically 600°C) represents a significant portion of the total process stream (as much as 15 percent per reheat application).



HOT CAS BYPASS REHEAT

For a plant using hot gas bypass to reheat the second and third catalytic stages large quantities of uncondensed elemental sulphur and sulphur gases bypass upstream stages, thereby seriously hampering the plant efficiency. Any COS or CS₂ in the bypassed stream usually are not converted in second and third converters causing a direct reduction of recovery efficiency. The method also has serious limitations when it comes to plant turndown because of heat losses, requiring an even greater bypass fraction.

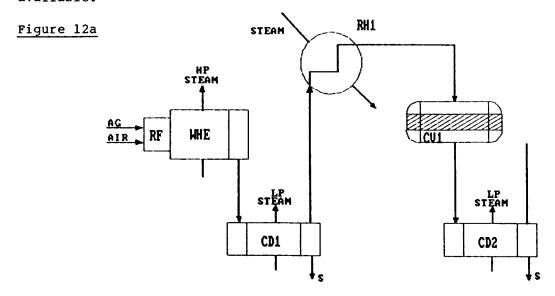
All three methods of direct reheat rely on efficient mixing of the hot stream with the process stream and static mixers are often included downstream of the reheater.

Indirect Reheat Methods

These methods are shown schematically in Figure 12 and are in general preferred to direct methods of reheat because they do not in any way affect the potential recovery efficiency of the plant. All involve heat exchangers and this can be relatively expensive when compared with the direct methods.

Operating flexibility is somewhat restricted in the steam reheater and the gas/gas heat exchanger: the former, because sufficiently high pressure steam is often not available to give the degree of reheat necessary for the first bed (inlet temperature of 240°C) or

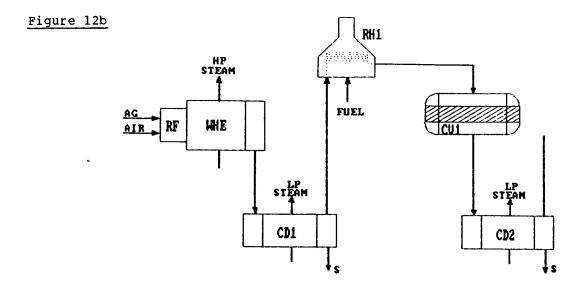
to do a heat soak; the latter, because the gas/gas heat exchanger relies on recovering heat from the first bed effluent and if this bed loses activity during operation, that heat is no longer available.



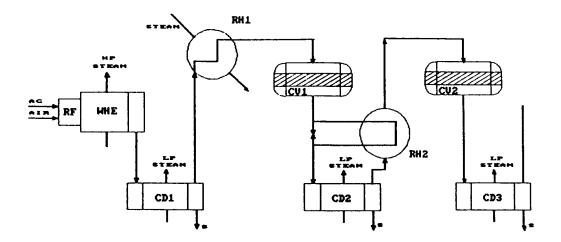
INDIRECT STEAM REHEATER

The other indirect option is a simple fired heater using fuel gas and exchanging heat to the process gas by means of banks of tubes in the fire chamber. This method of reheat is costly in terms of fuel consumption since the flue gas exits at temperatures as high as 600°C, but it has very good flexibility and does not hamper sulphur recovery.

Many plants use a combination of reheat methods, opting for the more flexible acid gas fired in-line burners on the first catalytic stage and indirect (generally steam) reheaters on subsequent stages.



INDIRECT FUEL FIRED HEATER



GAS-GAS HEAT EXCHANGER

4.2 Catalytic Converters

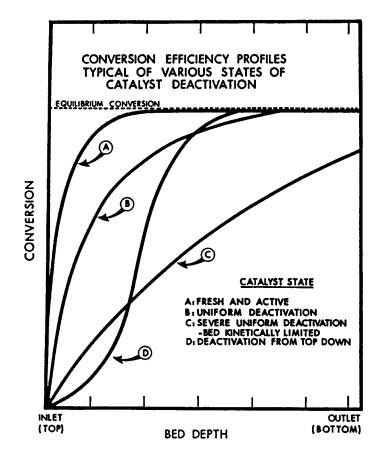
The catalyst beds are operated in a region at the left of the Gamson-Elkins curve, allowing closer approach to the high conversion efficiencies.

Catalyst bed performance is dependent primarily on the catalyst activity, and maintaining high activities is a significant concern in any sulphur plant. The activity is dependent on both physical and chemical properties of the catalyst and damage to any one of these properties will result in reduced activity.

Catalyst bed performance is also dependent on temperature, pressure, residence time and concentration of the reactants and products.

Typically, equilibrium conversion is reached in the first 30 cm of bed depth with truly active catalysts.

The effects of catalyst deterioration are best illustrated by Curve A schematically represents Figure 13. the conversion efficiency across an active catalyst bed, indicating equilibrium is achieved very quickly. As the catalyst becomes deactivated by uniform degradation, equilibrium is achieved further down into the bed, (Curve B) until such time as the bed becomes kinetically limited, (Curve C) that is, unable to achieve equilibrium in the depth of catalyst present. When this happens, it is quite evident that a problem exists, as conversion loading will move to the next converter accompanied by a larger temperature rise across that bed and a higher sulphur production from its downstream condenser. there are no downstream converters, a distinct increase in emissions will be noted.



If deactivation occurs by fouling of the catalyst, it will usually occur from the top down. This generally means that the catalyst under the surface will remain fully active. In this instance, the performance of the bed will have the characteristic shape of Curve D, with full equilibrium being achieved further into the bed.

Deactivation of the catalyst is caused by many different mechanisms and these can be categorized as, those that produce structural changes in the catalyst material and those that block access to the active sites with an external agent.

The first category includes thermal aging, phase changes, sintering and general attrition. These are all irreversible.

The second category includes such mechanisms as sulphation and fouling by sulphur, carbon and other salts or corrosion products.

Sulphation is by far the most common and is caused by oxygen contacting the hot sulphur-laden catalyst. The presence of oxygen is most often due to breakthrough from in-line burners or poor start-up and shutdown procedures.

The O_2 reacts with the sulphur adsorbed on the alumina destroying an active catalyst site and thereby reducing the capability of the catalyst bed to attain equilibrium conversion efficiencies. This process is reversible by a procedure known as rejuvenation whereby the catalyst is heat soaked and subjected to a deficient air operation for a period of 24 to 48 hours. This procedure has been adopted by many plants as a part of a regular catalyst maintenance program and has resulted in extended life at acceptable conversion efficiencies.

The temperature of operation for the catalyst beds is dependent on many factors and no two plants are identical in this respect. In general, temperature control of converters is on the inlet gas stream by whichever reheat method is used at the plant. A plant with a rich feed will have to operate the converters hotter than a lean feed plant. This is due to the lower sulphur dewpoint in the lean feed case, as a result of CO₂ dilution (refer to Figure 10).

Each plant operator must decide on a safe operating margin at which to run each converter, to maintain it above the sulphur dewpoint at all times. In well controlled plants, dewpoint margins of 5 - 8 degrees (Celsius) are regarded as sufficient margin for operating safety.

The first converter is a special case at most plants, as it must handle the hydrolysis of COS and CS_2 which, if not hydrolyzed, will contribute to a lower overall plant conversion efficiency. Although COS and CS_2 are not usually present in acid gas streams, they are common by-products of combustion in the reaction furnace. These two compounds hydrolyze over catalyst at higher temperatures than is desirable for the Claus reaction. For this reason, the first converter is run at a temperature sufficiently high to accomplish 95 to 100 percent hydrolysis of COS and CS_2 , in the 315 to 340°C range. (9)

Subsequent converters are operated at temperatures that are too low for the hydrolysis reaction to proceed to any acceptable degree. The high temperature in the first converter does reduce the conversion level for the Claus reaction, but this is compensated for by the lower operating temperatures downstream.

Typical operating temperatures for rich and lean feed plants are listed on the following page.

Catalyst life prediction is a difficult task as inevitably it is a major plant upset that destroys the activity, such as the carryover of significant quantities of amine. This is particularly true for lean feed plants operating with a split-flow configuration.

Temperature profiles are a good indicator of catalyst activity provided sufficient thermocouples have been installed in the beds. The profile will closely resemble the curves of Figure 13, and can forewarn of gradual deactivation before the bed becomes kinetically limited.

Catalytic Converter Operating Temperatures (°C) (Typical)

		Rich Feed (85% H ₂ S-dry)	Lean Feed (18% H ₂ S-dry)
Converter 1	Inlet	240	240
	Outlet	316	316
Converter 2	Inlet	196	200
	Outlet	218	212
Converter 3	Inlet	176	171
	Outlet	180	173

Source: SULSIM R (10)

A more accurate method of determining the current state of the catalyst is to sample the inlet and outlet streams of the bed and using chromatographic analyses of the samples, compare the measured conversion efficiency with that predicted by equilibrium (11). This technique also allows the operator to study minor species such as COS and CS₂ to check for degree of hydrolysis.

The stream analyses together with the temperature profiles give sufficient information on the catalyst status to allow scheduling of catalyst changeout or rejuvenation, barring unexpected upsets.

4.3 Condensers

The function of the condenser attached to each catalytic stage or to the thermal stage, is simply to recover in liquid form, the elemental sulphur vapour produced in the reactors.

The vapour is comprised of S_1 to S_8 polymers and the liquid is a mixture of S_8 -rings and long-chain polymers. The sulphur vapour pressure at the outlet temperature means that a residual amount of sulphur vapour will be carried out with the effluent. This is an equilibrium amount and can be calculated in terms of percentage efficiency loss for the plant, at the final condenser. Figure 14 presents a nomogram (12) by which the losses can be estimated based on the final condenser temperature and the acid gas H_2S concentration. Vapour losses in lean feed plants are much more serious than in rich feed plants due to the quantity of inert gas comprising the process stream. Pressure also plays a role in the vapour loss, being less, at higher pressures.

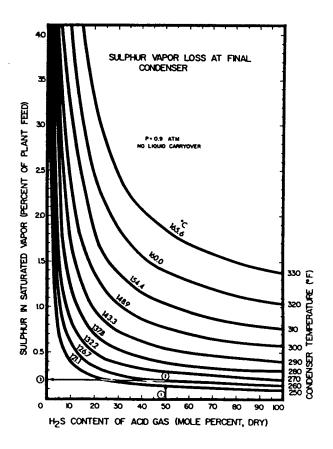
Liquid sulphur entrainment can be a problem with sulphur condensers and has been noted to be prevalent at high and low mass velocities through the tubes.

The recommended design mass velocity range to avoid significant sulphur entrainment is 10 to $26 \text{ kg/(s.m}^2)$. The minimum is 5 and the maximum is $30 \text{ kg/(s.m}^2)$ (13, 14). When the mass velocity is too low relative to the heat exchange rate, condensation starts in the gas phase as fog, rather than as a film on the condenser tube surface. Small fog droplets behave as an aerosol and cannot be removed from the gas stream by normal mist elimination methods.

On the other hand, if the linear gas speed through the tubes is too high, re-entrainment of the sulphur running along the bottom of the tubes can occur. In this case, the mist droplets are usually of sufficient size that they can be removed by properly sized and placed mist eliminators.

Entrainment will typically account for an efficiency loss of less than 0.2 percent, however, many plants have been tested where the loss is significant. Thus with the equilibrium sulphur vapour and the entrainment the difference between conversion efficiency and recovery efficiency is typically 0.4 percent for rich feed plants and 0.7 percent for lean feed plants.

Figure 14



5.0 INCINERATOR

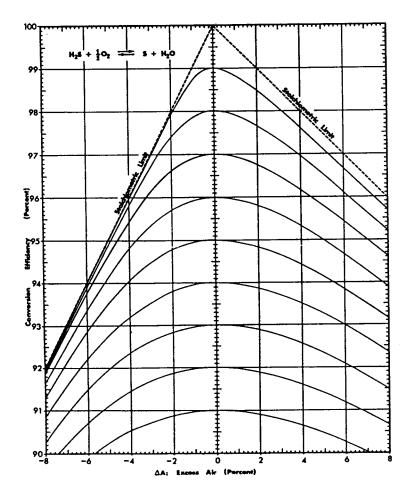
The incinerator is designed to oxidize the remaining reduced sulphur compounds ($\rm H_2S$, COS and $\rm CS_2$) and any elemental sulphur present in the Claus tail gas. This can be achieved by thermal free-flame oxidation or oxidation over a catalyst.

6. CONTROL

The principal control variable in a sulphur plant is the air to acid gas ratio. Plant efficiency is quite dependent on the two principal reactive species $\rm H_2S$ and $\rm SO_2$ being in the correct ratio for the Claus reaction.

This dependence becomes more acute in plants where the operating efficiency is high. The more efficient the plant, the more critical the control (15) of the air as is demonstrated by the curves in Figure 15. It can be seen that for a plant operating with an expected conversion efficiency of 98 percent, the loss in efficiency due to operation 2 percent deficient in air is 0.6 percent, and correspondingly the loss is 0.3 percent for 2 percent excess air operation.

Figure 15



The control of the air is by tail gas analysis using an ultraviolet photometric analyzer. This analyzer measures the $\rm H_2S$ and $\rm SO_2$

concentrations in the tail gas and produces a signal consisting of the quantity;

$$2[SO_2] - [H_2S]$$

where $[SO_2]$ is the concentration of SO_2 in the tail gas (16). The output is linear and directly proportional to the percent error in the process air. Coupled with the necessary feed-back loop, modern analyzer systems are capable of controlling the process air within + 0.5 percent resulting in minimal loss in plant efficiency.

7.0 PROCESS CAPABILITY

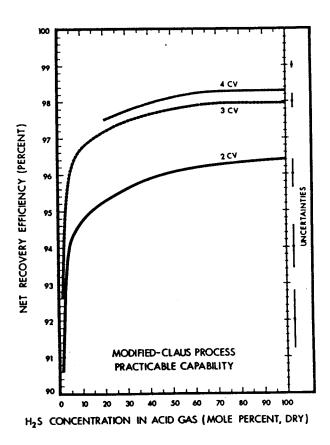
The modified-Claus process is limited in its recovery capability to levels that often fall short of local environmental regulations.

The capability of a particular plant is dependent on its design and feed gas quality as well as the operating variables and catalyst activity.

For an optimized plant, operating will active catalyst, the expected recovery efficiencies (8) are presented in Figure 16.

These efficiencies have been confirmed (1) by field testing and allow for 'real-world' limitation on performance such as, operation above the ideal temperature in the catalyst beds, presence of sulphur fog or mist in the condenser outlets, and operation within the practical limitations of control on the air to acid gas ratio.

Figure 16



The expected efficiencies also allow for an additional loss due to minor operating problems during the long term.

8.0 LITERATURE

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