DESIGN AND OPERATING AMINE UNITS FOR TROUBLE FREE UNATTENDED OPERATION
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Both small and large amine type treating plants are now being operated unattended. However, the degree of success and the amount of downtime can vary widely, depending on how an installation is designed and operated. This paper offers suggestions which have proved to be quite successful in maintaining a high per cent of time on stream.

PLANT DESIGN

1. Equipment Sizing:

Perhaps first and foremost is the necessity of adequate sizing of the plant. A "close" design will perhaps cause more downtime than any other one factor. We normally do not plan any of our smaller installations to operate at more than 60% of capacity. In addition to providing a margin of safety, this also allows for unexpected additional loading should it occur, as well as more flexibility in operation.

In selecting and sizing equipment, the following represent conservative criteria which should be applied:

a. Contactor:

Normally, we prefer a tray type column, particularly when the H₂S exceed 100 gr./100 scf. A tray type column may be more expensive, but the flexibility and performance will more than compensate for the additional cost. In sizing a tray type column, we normally plan no loading to exceed 75% of flood point for the bubble area, and a downcomer volume sufficient to assure a minimum of 7 seconds retention time for the design solution rate. We also have found 24" tray spacing to be far less susceptible to foaming problems than are lesser tray spacings.

A good mist extractor is a must, and we have found a 6" wire mesh type to be quite satisfactory. However, of even greater importance is the vapor dis-engaging space between the top tray and the mist extractor. Our preference is that the distance between the top tray and the mist extractor be equal to the diameter of the column, or equal to 1 1/2 times the tray spacing, whichever is greater.

The method of entry of gas into the bottom of the contactor is also of considerable importance. Normally, the bottom tray will have a longer downcomer with a seal pan. It is extremely important that the inlet gas not be allowed to blow on this seal pan, since it may "blow" the seal on this bottom downcomer, resulting in premature column flooding. If the inlet nozzle (or chimney) may not be located to prevent this, a baffle should be installed to protect this seal pan.

The question of the number of trays to be used is always worthy of close scrutiny. We have one plant treating 630 gr. H₂S/100 scf to an outlet content of 0.05 to 0.10 gr./100 with a 15 tray contactor. However, again applying a conservative design criteria, we would plan a minimum of 18 trays should we build this plant today. Should the inlet H₂S exceed 1000 gr./100 scf, we would recommend 20 trays.
b. Still:

The still deserves attention equal to that given to the contactor. We use the same criteria as to per cent flood point, downcomer size, and tray spacing as we do for the contactor.

One major factor to be considered in addition is the liquid loads in a still. We recommend stills be 12 tray columns, with the feed being on the 15th tray from the bottom. The 16th through the 18th tray will then have only the reflux as a liquid load. Therefore, these trays must be designed realizing that the liquid load on these trays will be much lower, and care must be taken to be sure that these three trays will prime (or seal) properly.

Mist extractors are not necessary in the still. However, the vapor disengaging area criteria described for contactors should also be applied to stills. Also, care should be taken to protect the seal pan on the bottom tray downcomer.

c. Reboiler:

An oversized reboiler will rapidly pay out its additional cost, in decreasing the time necessary to bring a unit up to operating temperatures when it has been down. A close design on a reboiler sometimes results in several hours being necessary in the startup of a unit. In the case of direct fired reboilers, we use a relatively low heat flux of 6000 to 7000 btu/hr.-sq. ft. for design purposes. We also use a long flame burner adjusted to a rich flame and plenty of excess air. This prevents a firetube hot spot adjacent to the burner head. For indirectly heated reboilers, we allow ample fouling factor of 0.004 to 0.005, overall.

We always prefer thermosiphon reboilers where possible, for both direct or indirect heated units. Our second choice is kettle type with an overflow compartment. However, care should be exercised to maintain a level over the heating element at all times.

d. Heat Exchangers:

Solution exchangers should always be selected with the thought that they must be mechanically cleaned on both sides or replaced periodically. Although chemical cleaning is satisfactory on the tube side, our experience is that it is not satisfactory for the shell side of a badly fouled exchanger. The acid will channel, resulting in large parts of the exchanger being untouched.

Solution exchangers should be designed to raise the rich solution temperature to 180°F, minimum, and preferably to 200°F. Any less temperature will result in too much of the still being used to heat the solution, rather than for stripping the H₂S.

There has been much said in the past in favor of high pressure solution exchangers with the contactor dump motor valve downstream of the exchangers. In our experience, we have failed to see the necessity of this procedure. Our preference is to limit linear velocities in the exchangers to 6 fps, and use a rich solution flash tank ahead of the exchangers.
a. Filters:
Good filtration of the solution is a good investment, and is a relatively small cost compared to the total plant cost. We have had the best results with a specially designed activated carbon filter, which cleans the solution by both mechanical filtration, and by adsorption. One filter installed on the rich solution line will perform acceptably, but a second filter, installed in the lean solution line will result in "water white" solution.

f. Surge Tank:
We normally install our surge tanks connected to a tee in the line from the still to the exchangers. This tank is pressure equalized with the still, and is equipped with a purge gas line. The bulk of the solution by passes the surge tank.

This offers several advantages. These include a minimum time to circulate the solution, resulting in a rapid response to any changes made in the operation of the still. Also, no level control or dump valve is required for the still. This system assures no air will contact the solution.

2. Instrumentation
Normal care should be exercised in selection of instruments, controls, and valves, with valves to fail in a "safe" position. We have also found that a small instrument air compressor and batch type calcium chloride air dryer are good investments in preference to using gas for instruments. Even sweetened, dehydrated gas will contain traces of H2S and water sufficient to cause problems periodically. However, in case of malfunctions or plant upsets when dependable instrumentation is most essential, higher H2S levels and/or more water in the instrument gas will compound these problems.

Instrumentation of the rebolier and still deserve more than normal attention, and one must remember certain basic criteria. First, the rebolier kettle operates as if it were a vapor/liquid system boiling in equilibrium, and very little effect is seen due to inerts in the rebolier. For this reason, the kettle temperature is almost totally dependant on kettle pressure.

Second, an amine still, like any other still, works best when loads, vapor rates, pressures, and temperatures are constant. Therefore, this limits the possible methods of control. Three methods which have been used successfully are:

a. Constant Heat Input to Reboiler.
This may be done by a constant firing rate for the burners of direct fired units, or constant steam or hot oil rates to indirect heated units. With this system, the operator must depend on the complete automatic shut down of the rebolier heat source in case of shut down of the unit. This system maintains very uniform steam vapor rates through the still, but provides no adjustment of heat input for changes in loading or ambient temperatures.
b. Still Head Temperature Control.

A temperature controller sensing the temperature of the still head may be used to control the heat input to the reboiler. Since the still head temperature will vary as the steam-acid gas ratio in the still head vapor this offers a very sensitive control of the reboiler heat input, and responds to load changes and ambient temperature changes. However, this type of control is somewhat difficult due to the lag between control point and controlled medium.

c. Still Differential Pressure Control.

A differential pressure controller, sensing the pressure drop across the still and controlling the heat input to the reboiler may be used. This system offers a very uniform vapor rate through the still. However, this rate is the total of both steam and acid gas. Should the acid gas load increase, the steam rate through the still would decrease if the controller is not readjusted upward.

Our preference is the "constant heat input" or the "still head temperature" methods of control, or a combination of these two. We feel this represents the optimum.

3. Control Panel

Every unit should be equipped with a control panel containing the motor starters and an automatic shut-down system, which will shut the unit down and close a master valve on the gas sales line in case of malfunction of the unit. The control panel should be arranged to shut down the unit on any one of the following:

a. Low reboiler level (particularly for direct fired reboilers).

b. Low surge tank level.

c. Low solution circulation rate.

d. Low still head temperature.

e. Power failure, after an adjustable period of up to 120 seconds.

f. Pump or other motor overload.

g. Low instrument air pressure.

A control panel should also include pilot lights to indicate which of the above caused the shut down.

The reboiler low level switch will protect overfiring of the reboiler. The surge tank low level shut down will prevent operation in case of excessive solution loss (such as by leak or foam). The low still head temperature switch will prevent operation in case of low steam vapor rate in the still, resulting in incomplete solution stripping. A time delay relay is normally used to cause shut down in case of an extended power failure. All motor controllers are interlocked to shut the unit down in case any one motor overloads.
OPERATION

1. Solution Care:

Perhaps the most important single factor in good operation of an amine unit is good solution care. We attempt to maintain "water white" solution, principally by the use of both lean and rich solution activated carbon filters, which remove both solid and liquid contaminants. On larger units, we also use side stream reclaimers.

Iron sulfide in our opinion causes most of the problems in amine units. It has been well documented that iron sulfide will cause foaming and fouling of exchangers and equipment. However, we also find that the presence of iron sulfide in the solution causes incomplete stripping of the lean amine, thus making it more difficult to maintain specification gas. Also, if the iron sulfide is removed, we find that higher solution loadings are possible. We have several units operating at loadings as high as 0.75 mol acid gas per mol of amine.

We have recently been successful in descaling a partially fouled unit while it was in operation by adding a new descaling compound to the solution. On one unit, after adding this material for about one month, the pressure drop across the lean solution exchangers dropped from 9 psi to the design pressure drop of 6 psi. We now add this material to all of our units routinely as a scale preventative.

It is also important that only good quality makeup water be used. Particular attention should be given to the pH of the water and its hardness. The water should be neutral, and the hardness should be 100 ppm or less, if the water is not added through a reclaimer.

2. Analytical Procedures:

We recommend one routine solution analysis be performed twice each week. This is an analysis of the solution for per cent MEA. (See Figure 1, Analytical Procedures.) Make-up amine is added according to the result of this analysis, with only water make up to be added routinely each day.

One of the most useful analytical procedures is the test of the lean solution for residual H₂S, listed in the "Analytical Procedures". In general, we find that we must maintain lean solution residual H₂S contents of 40 gr./gal. or less to successfully maintain ¼ gr. or less outlet gas. Thus, this test can rapidly indicate whether the problem is in the stripping operation or the contactor when sour gas is encountered.

3. Routine Operating Procedures:

We insist that all of our plants be checked both morning and evening. With the automatic shutdown system listed above, and in some cases with an H₂S analyzer on the plant tail gas, the unit has little chance to run sour gas. The main purpose for this routine check is to restart the plant in case of automatic shut down. Our operators all work on a contract basis, and are paid on a fee basis according to the gas processed. Most will average $400 to $600 per month, for which they also handle minor maintenance. Sometimes contract operators can be obtained who offer to operate a plant
for less, but if they care for it properly, this will be a reasonable figure.

Summary:
Small amine plants can be designed, built, and operated for near continuous, unattended operation. (One of these plants has exceeded averaging being in operation over 98% of the time, with major downtime cause being power failures). However, the requirements are adequately sized equipment, a complete automatic shutdown system, and care in the operation of the unit.
Figure 1 - Analytical Procedures

PERCENT MEA

1. Measure 1 ml of MEA solution.
2. Add 25 ml distilled water.
3. Add 4-5 drops Methyl Purple Indicator.
4. Add N/10 Sulfuric Acid to colorless end point.

Calculation: Ml N/10 Sulfuric Acid x 0.611= % MEA

HYDROGEN SULFIDE TEST FOR MEA

1. Measure 1 ml sample of MEA solution.
2. Add 25 ml distilled water.
3. Add 2 drops phenolphthalein until pink.
4. Add concentrated HCl to colorless and 5 drops extra until it loses the pink color.
5. Add 1 ml starch solution.
6. Titrate with N/50 Potassium Iodide-Potassium Iodate Solution.

Calculation: Ml Iodide-Iodate x 12.5= GPG H2S

HYDROGEN SULFIDE TEST FOR GAS

1. Dampen filter paper with 5% lead acetate.
2. Hold in gas stream for 30 seconds.
3. No discoloration to a very faint tan indicates 0.25 gr./100 scf or less.
4. Distinct discoloration will indicate more than 0.25 gr./100 scf.
5. Note: A discoloration of yellow, orange, green, or sometimes tan, which will fade in 30-60 seconds in sunlight indicates mercaptans.
SHUT DOWN SWITCH
LFS - Low Flow Switch
FS - Float Switch
LTS - Low Temperature

CONTROLLERS
LLC - Liquid Level Controller
TC - Temperature Controller
PC - Pressure Controller

FIGURE 2
TYPICAL AMINE TREATING UNIT FLOW SHEET
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