

SULPHUR PLANT and TAIL GAS TREATING UNIT - STARTUP and SHUTDOWN

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ABSTRACT

This paper will deal with the considerations, do's and don'ts and risks involved with the startup and shutdown of sulphur plants. The subject matter will pertain mainly to the Claus style of sulphur plant, i.e. those employing a reaction furnace, catalytic conversion stages and sulphur condensers. This paper also discusses startup and shutdown considerations for amine-based tail gas treating units (TGTU).

Although many operators are always in a rush to shutdown and restart a Sulphur Recovery Unit (SRU) due to production schedule constraints, our experience has shown time and time again that skipping the essential steps to save time can result in damage to equipment and catalyst (often necessitating another outage or having to live with reduced sulphur recovery efficiency). Proper conditioning of the SRU prior to shutting it down and proper conditioning (heat-up/dryout) before starting it up again are a must, and take time (*to save time in the end*).

The topics which will be addressed include the following :

SRU Startup :

Cold (a unit which has been opened to the atmosphere) and Hot (unopened)

Check out considerations, prior to startup

- air blowers
- mainburner and controls
- catalyst beds
- sulphur condensers
- inline burners and indirect reheaters
- seal pots
- sulphur storage pit

Refractory dry out considerations

System heat-up ... why you should not rush

Introduction of acid gas

Introduction of ammonia acid gas

SRU Shutdown :

Short term outage (hot) versus long term outage (ready for entry)

Heat soaking requirements

Review of the outdated (high risk) "burnout" or "regeneration" method

Why condition the SRU before it is cooled ?

Natural Gas Sweep method of SRU conditioning

Importance of good natural gas control

Inert Quench (nitrogen) method of conditioning and cooling

When to start introducing air to the system

Shutdown of the sulphur storage pit - risks to be aware of

TGTU Startup

- Precommissioning and Equipment Preparation
- Refractory Dryout
- Hydrogenation Catalyst Presulphiding
- Flushing of Liquid Systems
- Introduction of Tail Gas
- Initial Operations

TGTU Shutdown

- Short Shutdown Considerations
- Long Shutdown Considerations

As with many plant operational procedures, SRU/TGTU startup and shutdown methods can vary from company to company and facility to facility. However, the general guidelines and considerations used as the basis for the various procedures are common, for the most part. And, as is the case for many industrial applications, some procedures entail more risk than others. For this reason we believe it best to address the considerations (i.e. something like the pros and cons along with the attendant risks) so that the operator can make decisions regarding acceptable risk with his “eyes open” for his particular set of circumstances.

In general, following proven methodology for startup and shutdown (mostly learned at the “School of Hard Knocks” by our predecessors) makes life easy. SRUs are very tame when the basic rules are followed, but unforgivingly harsh if shortcuts are taken.

SRU STARTUP CONSIDERATIONS

There are basically two classifications of SRU startups : “cold” and “hot”.

The cold type of startup refers to the first time an SRU is started up and the subsequent startup of a previously run/conditioned (i.e. prepared for opening to atmosphere) SRU.

The hot type of startup refers to the restart of a sulphur plant which has been shutdown quickly for minutes or for a number of days, without prior conditioning of the catalyst or the removal of sulphur bearing process gases from the system. “Hot standby” is also available for processes equipped with a recycle blower (such as the Recycle Selectox and the COPE processes).

Cold Startup :

For the cold startup scenario, the operator should first verify that the SRU's equipment has been checked out thoroughly prior to attempting a startup :

- | | |
|---------------------|------------------------------------------------------------------------------------------------------------------------------------------------------------------|
| Air Blowers | - surge control system operable ? |
| Mainburner | - natural gas control system operable ?
- air purges operable ?
- ignitor operable ? |
| RF/ WHB | - refractory and checker wall inspected ?
- WHB tubes leak checked ?
- tubesheet ferules in good condition ?
- debris removed ? |
| Catalyst Beds | - catalyst depth uniform ? Catalyst ceramic ball support layer in ?
- fresh or used catalyst ?
- grating support in good condition ?
- debris removed ? |
| Condensers | - tubes leak checked ?
- demister pads in place ? using multi-density pads ?
- debris removed ? |
| Inline Burners | - gas control system operable ?
- ignitor operable ? |
| Indirect Reheaters | - tubes leak checked ?
- if fired style, fuel gas control system operable
and ignitor operable ? |
| Seal Pots | - steam jackets on ? do they melt sulphur on the outside?
- are they pre-loaded with sulphur ? or, will they be
loaded on line after startup ? |
| Sulphur Storage Pit | - debris removed ?
- steam coils on ?
- piping steam jackets on ?
- sulphur pumps operable ? |
| Steam Jackets | - are they all on ? do they all melt sulphur on the outside? |

- any new steam piping/ trap systems should be blown to atmosphere for 24 hours for debris removal.

Test the ESD System

If new (fresh) catalyst has been installed in the converter (reactor) beds, it is best to “air blow” each bed in sequence to remove as much of the catalyst fines from the system as possible. This requires blocking off of a converter’s outlet pipe (using a temporary internal wooden cover is quite suitable) and blowing air (using an SRU air blower) through an open manway on the reactor outlet side to atmosphere until no further dust emission is visible. Then sweep up or vacuum the bottom of the reactor vessel (don’t forget to remove the temporary internal cover when done blowing a converter). Some people have also used pressure / depressure cycles to get the dust out.

For a first time startup, it is recommended that the sulphur seals also be air blown (as well as any new sulphur seals installed in existing plants). By opening and closing the valve on the condenser rundown to the seal pot, air can flow through the seal to atmosphere via an open look box to remove light dust and debris (a rag must be stuffed into the sulphur rundown pipe from the look box during this procedure --- don’t forget to remove the rag when done blowing).

The air blows minimize the risk of forming and depositing “sulphur concrete” (mainly sulphur with some catalyst dust and iron in it) in the rundown systems during plant operation, which results in restriction or blockage of liquid sulphur flows in the system.

Next, the whole SRU is first heated up to nominal temperature levels using only the air from the SRU air blower. The discharge air temperature is typically between 160°F and 250°F [70°C and 120°C], depending upon ambient temperature and whether or not an air preheater has been installed. It is desirable to have the catalyst bed temperatures at > 160 °F [70°C] prior to firing any natural gas burners in order to prevent water condensation on the catalyst (*the water dewpoint of a stoichiometrically fired natural gas flame’s flue gas is between 120 °F and 140°F [49°C and 60°C]*).

If the SRU has new refractory or has had major refractory repairs carried out on the mainburner/ reaction furnace area, then a refractory dryout (curing) must be carried out. This can be done using a temporary dry out heater system (much preferred and strongly recommended due to the superior control characteristics) or using the mainburner firing on natural gas with high excess air. The dry out procedure used should be reviewed and agreed to with the refractory manufacturer, and typically involves the following :

- Heat up to 250°F [120°C] at 50 °F [30 °C] per hour and hold for 2 to 4 hours;
- Heat up to 350°F [175°C] at 50 °F [30 °C] per hour and hold for about 20 hours;
- Heat up to 600°F [315°C] at 50 °F [30 °C] per hour and hold for 5 to 10 hours;
- And, heat to 1,000°F [538°C] at 50 °F [30 °C] per hour and hold for 5 to 10 hours. It is actually best to then slowly heat to normal operating temperature and hold there for another 5 hours prior to starting the cooldown.
- Control any cooldowns to 100 °F [55 °C] per hour maximum.

Converter bed and incinerator dryout procedures typically involve the following, and are carried out using natural gas fired inline burners or incinerator burner :

- Heat up to 250°F [120°C] at 50 °F [30 °C] per hour and hold for 2 to 4 hours;
- Heat up to 400°F [204°C] at 50 °F [30 °C] per hour and hold for about 5 hours;
- And heat up to 600°F [315°C] at 50 °F [30 °C] per hour and hold for about 5 hours for converters and up to 1,000°F [538°C] at 50 °F [30 °C] per hour and hold for about 5 hours for the incinerator.
- Control any cooldowns to 100 °F [55 °C] per hour maximum.

The above approaches are a safe ones, i.e. prevent refractory damage or lift off by slowly removing the water present in the refractory materials. *Speeding up the process can and has resulted in refractory failures leading to hot spots on the metal coverings after firing of the mainburner.* This necessitates another SRU outage to affect repairs and another dry out procedure, costing another 4 to 6 days of downtime.

It should be noted that the inline burners (using natural gas) or indirect reheaters should be used as required to maintain the catalyst bed temperatures at > 160°F [70°C] to prevent water condensation onto the catalyst during the dryout procedures. This approach eliminates the possibility of catalyst damage during the SRU heat up step which follows.

SRU heat up to normal operating temperatures is done using the mainburner and inline burners, firing on natural gas using excess air, at a rate of between 50 °F and 100 °F [30 °C and 55 °C] per hour (DO NOT RUSH). Use the mainburner, and inline burners as required (at 30 to 100 to 1 air to gas ratios to keep the flames cool). *For safety reasons, all burner natural gas feed lines should be equipped with an automated double block and bleed system ; all burners should be equipped with a high energy spark ignitor system ; all burner light off sequences should start with a purge cycle (nitrogen purge is preferred) ; and, all should have a 5 to 10 second automated timer limit on a light off attempt (5 seconds is preferred).* I recommend automated natural gas control loops for all burners.

Given the use of DCS systems for operating today's SRUs, it is strongly recommended that the natural gas firing loop for the mainburner be automated ... the cost is low but the benefit is large. The natural gas flow (pressure and temperature compensated) should be brought into the computer and used to determine the air demand for the main air valve controller. This can be done by taking the natural gas flow, multiplying by 9.5 to 10 (check this ratio by calculation), and adding it to the acid gas air demand calculation (which is zero at this time) prior to sending the air flow controller its signal (all by the DCS). This then allows the operator to automatically control the air flow at the desired ratio and is far superior to the more unreliable and very tricky manual control approach.

Along with a good mixing burner (read "not cheap") this approach is most important when burning natural gas at low rates or when co-firing natural gas and acid gas when the acid gas is introduced to the mainburner. An automated system always adds the correct amount of air for the amount of natural gas and acid gas entering the mainburner.

The procedure for introduction of acid gas is fairly straightforward :

1. Ensure that the inline burners are firing at about 95% of stoichiometry and that the

incinerator is at design conditions, requiring higher than normal excess air at about 15% to 25% (using P, T corrected flows is very important).

2. Pull the blinds on the acid gas line(s) using all safety precautions.
3. Increase the natural gas flow and decrease the air flow to the mainburner slowly until 95% to 100% of stoichiometry is reached, adding moderating steam (at a rate range of 4 to 10 pounds of steam per pound (or kilogram per kilogram) of natural gas) in order to keep the reaction furnace temperature at $< 3,000^{\circ}\text{F}$ [1650°C]. Stay above the burner minimum design flow to prevent backfiring.
4. Make sure that the bed temperatures are all above 250°F [120°C] and then introduce amine acid gas to the mainburner (at a rate higher than the low flow shutdown). If the system is automated, as described earlier, the controller will ensure the addition of the correct amount of air for the amount of natural gas and acid gas entering the mainburner ; if not, the operator must calculate the air required and set the controllers airflow setpoint (a very tricky operation).
5. Slowly back out the natural gas flow and increase the acid gas flow to the required level. Shut off the moderating steam. Double block and bleed both the natural gas and moderating steam lines to the mainburner.
6. Switch any acid gas inline burners from natural gas to acid gas.
7. Introduce the tail gas analyzer control (ADA) to the system controls.
8. Introduce any sour water stripper acid gases to the mainburner. Some jurisdictions may require this to happen at step 4.

If the sulphur seals were NOT previously loaded with sulphur, then each condenser's sulphur outlet valve must be opened and closed every 15 minutes until they are filled with liquid sulphur. Since the acid gases that will vent from the look boxes during this procedure can contain up to 10% H_2S , appropriate safety precautions must be taken in those areas. This is why more and more operators are leaning towards preloading the sulphur seals with crushed sulphur prior to startup, even though the work is considered messy and is labour intensive. (a number of Shell and ExxonMobil refineries have installed a sulphur "flush" system, which allows them to flush back into the condensers and down the rundowns, as well as provide the capability to pre-load the seals prior to start-up).

The next and final step is to adjust the bed inlet temperatures to achieve the design bed outlet temperatures and lower the final condenser's outlet gas temperature to the 255°F to 265°F [124°C to 130°C] range (to minimize sulphur vapour losses to the incinerator). Also, a coalescer at the end of the sulphur train will result in 0.1% to 0.2% higher recovery (less sulphur mist entrainment to the incinerator).

Finally, adjust the excess O_2 in the thermal oxidizer (incinerator) flue gas ... typically to 2% (or 10% excess air).

Hot Startup :

This requires light-off of the mainburner on natural gas at below the stoichiometric firing ratio (typically 9.5 air to gas volumetric flow ratio is used to get the desired 95% of stoichiometry target, to leave some margin for safety). Moderating steam is then added to keep the reaction furnace temperature at $< 3,000^{\circ}\text{F}$ [1650°C].

The inline burners are then lit off (on natural gas at the same 9.5 air to gas ratio) and the incinerator started up. *On natural draft incinerator systems, the incinerator can be safely ignited before the mainburner is lit.*

Ensure that the catalyst bed temperatures are greater than 300°F [150°C] ($> 400^{\circ}\text{F}$ [200°C] is preferred) before acid gas is introduced to the mainburner.

Then introduce the acid gas and back out the natural gas/ moderating steam. Then switch all acid gas burners to acid gas from natural gas.

Check the catalyst bed pressure drops to check for sulphur crusting problems. If a bed's pressure drop proves to be high, increase the inlet temperature until the pressure drop decreases to normal levels.

Finally, return to optimum temperatures (usually the design temperatures) throughout the SRU.

NOTES:

Some operating companies allow light-off on acid gas if the Reaction Furnace temperature is still greater than 1600°F [870°C].

Some will also allow lighting off of the gas on hot refractory at above that temperature as well. This is not recommended since a safe approach (i.e. lighting off from a high intensity spark ignitor or a pilot gun) is not difficult or time consuming to use (provided good equipment is supplied to the operator).

SRU SHUTDOWN CONSIDERATIONS

Short Term or “Hot” Shutdown :

This is an emergency shutdown which lasts from minutes to a few days. All burners are down and the system sits and slowly cools off.

It is a good idea to introduce a small nitrogen (or other inert gas -- NOT air) purge flow to the mainburner to continually sweep the system and prevent reverse flow of atmospheric air from the incinerator (*to prevent sulphur fires on the last catalyst bed by preventing air ingress*).

It is recommended that hot shutdowns be limited to 3 to 5 days so that sulphur freezing (or crusting) is prevented from occurring to any large degree. This condition can have a disastrous impact on the ability to restart the SRU.

Another method of hot shutdown is the “hot standby mode”, which involves the circulation of hot process gases through the SRU by use of a recycle gas blower. This type of mode is easily achieved in plants which come equipped with recycle blowers as part of the process (e.g. Recycle Selectox units and COPE units) provided the tail gas line is also tied into the blower suction. Such systems allow for indefinite hot standby operation and have proven useful to operators of such units.

Long Term Shutdowns :

This type of shutdown means to prepare or condition the SRU for entry (open to atmosphere) or for long term lay-up. Long term lay-up would require eventual pressurization with dry nitrogen to prevent internal plant corrosion, following the conditioning process.

The first step is to heat soak the catalyst beds, i.e. operate at 50 °F to 80 °F [30 °C to 45 °C] above normal inlet temperatures for two days (the beds may also be run at H₂S rich inlet conditions, called “rejuvenation”, to remove some sulfate for a while also, if desired ... typically using H₂S:SO₂ ratios of between 5:1 and 6:1). Then lower the acid gas flow, switch from acid gas to natural gas on the inline burners, and finally switch from acid gas to natural gas on the mainburner (reverse of the startup procedure, adding moderating steam flow to the mainburner prior to the addition of natural gas ... use between 2 and 4 pounds [kilograms] of steam per pound [kilogram] of natural gas). The air to gas firing ratios should be about 9.5 (i.e. at about 95% of stoichiometry --- calculate the correct ratio using an accurate natural gas composition) to prevent sulphur fires in the SRU. This process is commonly called a “natural gas sweep” or “fuel gas sweep”.

Maintain the sweep at sub-stoichiometric conditions until all the sulphur rundowns stop flowing - typically takes one to two days. Then rod the rundowns to ensure that the condensers are empty of sulphur. *This precaution ensures that condensers cannot fill with liquid sulphur -- a real fire hazard when air is introduced.*

After twelve hours without sulphur flow from the rundowns, shut down the inline burners and close their associated air purges. Slowly increase the air to gas ratio until the flue gas oxygen concentration reaches about 1% and hold there for at least 6 hours (12 hours is preferred) ... watch bed temperatures and reduce air flow to sub-stoichiometric as required to prevent sulphur fires from continuing should they occur. It is extremely important to have a good control system on the natural gas firing loop for the mainburner and a good burner for this sweep phase to both prevent carbon sooting of the catalyst or the ignition of any sulphur on the catalyst. Shut-off the moderating steam when the Reaction Furnace temperature drops to below 2400°F [1315°C].

When the temperatures are down to under 400°F [204°C] in the catalyst beds (to prevent auto-ignition of sulphur, which can occur in the 445°F to 480°F (230°C to 250°C) range in air), shutdown the mainburner and continue with cooling air flow through the SRU until the plant temperatures are down to the 200°F to 250°F [95°C to 120°C] range; then begin depressuring the waste heat boiler and sulphur condensers on the steam side.

Finally, shut down the blower and open the SRU manways.

An outdated, high risk method (NOT recommended) used in the past to attempt removal of carbon from the catalyst, involving high temperatures (> 600°F [315°C]) and process gas containing 0.5% to 1% excess oxygen, following completion of the natural gas sweep. This method was often referred to as a “burnout” or “regeneration” and has a low chance of success, i.e. a high chance of fires capable of damaging catalyst and equipment due to uncontrollable runaway temperatures. This procedure is not recommended.

A method of rapid cooling of an SRU involves the use of nitrogen, at 100°F to 200°F [40°C to 90°C], purge into the mainburner at a rate of about 20% of normal flow for about one day. This method speeds up a sulphur plant shutdown, but the catalyst will have to be removed and replaced (since it will have sulphur on the surface of as well as inside of the catalyst balls/extrudates). Vessel entry should be under mask only.

Some plants have used a heated nitrogen purge (where moderating steam is not available) at >250°F [120°C] into the mainburner at a rate of about 30% to 40% of normal flow for a number of days (same procedure as the natural gas sweep approach).

Also, some plants have used a natural gas sweep (to remove sulphur) and then followed with a nitrogen purge (after the mainburner is shut down) to cool to the 120°F [50°C] mark in order to speed up the cooldown period.

Another rapid method of shutdown which has been used is the steam purge method. This method is not recommended due to the following drawbacks :

- Catalyst replacement will be required.
- Corrosion concerns for all SRU equipment.
- Water presence in the sulphur rundowns.
- Vessel entry under mask only

Shutdown of the Sulphur Storage Pit :

It is imperative that a good air flow is maintained through the sulphur pit when the sulphur level is to be lowered in the pit in order to prevent the H₂S concentration from building up above the LEL (about 3.5 volume % at the sulphur pit temperature). The concern is a fire in the pit which could result in an explosion if LEL is exceeded.

For pits that normally use an inert vapour space :

- get the H₂S level in the vent gas down to < ~1 volume % (must be < 3.5 volume %) by increasing the purge flow.
- begin introducing sweep air to replace the inert environment.
- have snuffing steam supplies ready for use as the pit level is decreased for entry purposes, since any pyrophoric FeS present can start a fire.

For pits that normally use an air vapour space :

- get the H₂S level in the vent gas down to < ~1 volume % (must be < 3.5 volume %) by increasing purge flow.
- have snuffing steam supplies ready for use as the pit level is decreased for entry purposes, since any pyrophoric FeS present can start a fire.

TGTU STARTUP CONSIDERATIONS

The following discussions apply to amine based Tail Gas Treating Units which use the following major equipment: Preheater (steam or direct fired), Hydrogenation Reactor, Waste Heat Boiler, Quench Column, Amine Contactor, and Amine Regenerator.

The startup sequence for a TGTU depends on the cause and duration of the shutdown. For short shutdowns, typically due to an upset and/or trip of the SRU and resulting loss of tail gas feed, it is common to leave the two liquid systems circulating (quench and amine). The hydrogenation catalyst will stay hot for a few hours (longer in larger units) and in these cases the restart is simply a matter of diverting the tail gas flow to the TGTU once the SRU operation has been stabilized. If the bulk of the hydrogenation catalyst has fallen below the normal operating temperature, it may be necessary to heat the catalyst prior to introducing tail gas.

For initial startups and startups after turnarounds, the following should be considered:

All Vessels	- cleaned and free of debris
Columns	- internals installed correctly
Pumps	- seals full and ready - strainers installed
Preheater (fired)	- BMS full function check - Burner and refractory installation check
Catalyst	- bed depth uniform and leveled

Refractory Dryout

Most modern TGTU's have a provision for recirculating nitrogen using either a blower or a steam eductor. This recirculation system is used for refractory dryout, catalyst presulphiding, catalyst heat-up, and catalyst cool-down. The TGTU process gas is isolated from the SRU and Incinerator, and at the gas inlet to the Amine Contactor. The Quench system must be commissioned if an eductor is used, and it is also a good idea to commission the Quench if a blower is used. Any excess pressure over 5 psig is normally bled off to the Incinerator.

TGTU's using a fired Preheater will require a refractory dryout procedure if the refractory is new or if extensive repairs have been made. The refractory vendor's procedure should be used. Following is a typical heat-up curve. Temperatures are usually based on the process gas outlet temperature, although temporary thermocouples can also be used.

- Heat to 250 F (120 C) @ 50 F (30 C) / hour
- Hold ~ 4 hours
- Heat to 520 F (270 C) @ 50 F (30 C) / hour
- Hold ~ 4 hours

- Cooldown @ 100 F (50 C) / hour maximum

If the Hydrogenation Reactor has refractory, this is typically dried out in conjunction with the fired Preheater refractory. And for new installations, a boilout of the Waste Heat Boiler is usually done at the same time.

Catalyst Presulphiding

Although there are sulfided catalysts available, most hydrogenation catalyst is shipped in the oxidized state. Prior to startup it must be presulphided to convert the catalyst to its reduced (activated) state. The catalyst vendor's specific procedure should be used. The following is a generic procedure that assumes a nitrogen recirculation system and an acid gas presulphiding line from the amine acid gas header to the Preheater or Hydrogenation Reactor inlet.

- Purge the system with Nitrogen to < 0.5% Oxygen
- Establish Quench Water circulation
- Maintain ~ 5 psig at Eductor/Blower suction
- For fired Preheater, 95% of stoich maximum
- Heat catalyst to 390 F (200 C) @ 50 F (30 C) / hour
- Start Acid Gas flow (typ. ~1.0 to 1.5% H₂S)
- If using external Hydrogen source, start Hydrogen flow (typ. ~1.0 to 1.5% H₂S)
- Monitor temperature rise through catalyst
- Monitor Quench pH, add ammonia or caustic as necessary
- After the temperature wave has moved through the bed, increase temperature to 600 F (315 C) @ 50 F (30 C) / hour
- Hold for ~4 hours
- Use stain tubes to verify H₂S at outlet of Reactor >1%
- Cool catalyst to normal operating temperature
- Maintain nitrogen circulation or put system under nitrogen blanket

Flushing of Quench System

The flushing of the Quench loop can be done in parallel with the refractory dryout. Iron sulphide can be formed in the Quench loop during catalyst presulphiding which can plug filters, so it is a good idea to have the Quench system flushed prior to presulphiding.

It is strongly recommended to use a clean water source, preferably condensate or demineralized water. The following are points to consider.

- Install pump strainers (permanent or temporary)
- May take several sets of filters, dirty systems may require large mesh sizes at beginning
- Monitor pump suction pressure
- Alternate pumps

Flushing of Amine System

As the Amine system is usually isolated from the Hydrogenation / Quench system, all Amine activities can be carried out in parallel with refractory dryout and presulphiding. A clean water source (condensate or demin) is critical for the amine system as contamination can cause foaming during startup.

A typical procedure for cleaning the system follows:

- Ensure pump strainers are installed
- Circulate flush water
- Drain and refill as necessary until water is clear
- Heat to 140 F (60 C) using reboiler
- Add soda ash or potash (typically 1% solution)
- Circulate for ~12 hours
- Alternate pumps
- Dump caustic and fill with clean water
- Drain and refill as necessary to remove carbonates
- Some vendors recommend a wash using 3% amine (belts and braces approach)
- Fill system with normal strength amine
- Put system under nitrogen blanket until Startup

Introduction of Tail Gas

The introduction of Tail Gas to the TGTU is straightforward.

First check the following:

- Stable SRU operation
- Stable fired Preheater operation (or steam Preheater up to normal operating pressure/temperature)
- Hydrogenation catalyst temperatures are uniformly at normal operating temperature
- Anti-foam injection system ready for operation and anti-foam available on-site
- Filters are all in-line with spare filter cartridges available on-site

The following is a general procedure for introducing Tail Gas:

- Stop nitrogen recirculation in the Hydrogenation / Quench system (if using a fired preheater, it is typically shut down when the recirculation stops, and started again just before introducing tail gas)
- Line up flow path from Quench overhead to Amine Contactor
- Some plants have a bypass from the Waste Heat Boiler outlet to the Incinerator. This can be used during startup to stabilize the operation of the Hydrogenation Reactor before the process gas is diverted to the Quench Column. It can also be used to keep the catalyst hot during SRU upsets.
- Divert Tail Gas to the TGTU

Initial Operations

The operation of the TGTU is directly related to the operation of the SRU, so all major SRU parameters need to be monitored during the TGTU startup and initial operations. Particular attention should be paid to the output from the Air Demand Analyzer.

The Quench water pH is one of the more important parameters to be watched in the TGTU. A falling pH indicates slippage of SO₂ from the Hydrogenation Reactor. The initial response should be to inject ammonia or caustic but the cause of the SO₂ slippage will need to be addressed quickly. The most common causes for this situation are an SRU upset causing a low H₂S/SO₂ tail gas ratio, low reactor inlet temperatures and low hydrogen flow (or incorrect fired Preheater operation). If the Quench system is carbon steel, significant corrosion will occur quickly at a pH of 4.0 and below.

The following are other TGTU parameters which should be monitored during initial operations:

- The Quench water will turn black (from iron sulphide) during an SO₂ breakthrough from the Hydrogenation Reactor. This is a quick way to verify that the pH meter is correctly indicating a falling pH.
- Catalyst temperatures can give an indication of SRU operation. An increase in the temperature rise across the catalyst typically indicates an increasing SO₂ concentration in the tail gas.
- The fired Preheater operation or hydrogen flow should be adjusted to give 2% to 4% in the Quench overhead (typically there is a hydrogen analyzer in the overhead line).
- The dP across the Amine Contactor and Regenerator packing or trays gives an indication of foaming in the Amine system.
- The Lean Amine should be clear or straw colored. Rich amine should have a greenish tint. Changes in amine color can give an early indication of amine problems (use caution when sampling rich amine).
- The Rich Amine may turn black initially during startup as a layer of iron sulphide is formed in the system. If left in the system, the iron sulphide can cause foaming, so the filters are important during the startup and should not be bypassed.
- Anti-foam is the first line of defense against foaming, but it must be used sparingly. Too much anti-foam can be worse than too little as it will stabilize the foam. Typical injection quantities are 10 ppmw for Silicone based agents, and 100 ppmw for Glycol based agents.
- The testing of the TGTU amine should follow the same schedule as the main Amine Treating Units.

TGTU SHUTDOWN CONSIDERATIONS

The steps to be taken during a TGTU shutdown depend on the cause of the shutdown and the expected duration.

Short Shutdown

A short shutdown lasts a few hours and is many times caused by problems in the SRU or Incinerator. During this time, the Quench and Amine circulation should continue. All temperatures should be maintained at normal operating. As long as the majority of the catalyst is at the normal operating temperature, Tail Gas can be readmitted to the TGTU as soon as SRU operation has stabilized.

Longer Shutdown (no equipment to be opened)

A longer shutdown may occur if there are significant problems in upstream units. If the shutdown is to last several days, the nitrogen recirculation loop should be established to keep the catalyst hot. Liquid circulation should also be maintained. If it appears the shutdown will

last longer than several days, all gas and liquid circulations can be stopped and the unit put under a nitrogen blanket.

Long Shutdown (equipment to be opened)

Shutdowns for turnaround or equipment repair require more elaborate procedures. Some points to consider after Tail Gas flow has been stopped are:

- Maintain amine circulation until the Rich Amine has been fully regenerated (rich loading equals lean loading).
- The nitrogen circulation loop can be used to cool the catalyst.
- The catalyst is in its reduced state and is pyrophoric. It can be removed under nitrogen (usually by a third party) or oxidized in the reactor (refer to licensor or vendor procedures) before removal.
- Follow all site-specific vessel entry procedures.

We hope that this paper has been informative and useful. As with all opinions on matters of unit startup and shutdown, we are sure that some will differ with the opinions expressed herein (but not with the substance I'm sure).