

# The 10 Commandments of Sulphur Recovery

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### Abstract

As licensor of SRU technologies, Comprimo, part of Worley (previously, Jacobs), is frequently contacted by clients for support and advice when the SRU is not running properly. The practical experience from sites all over the world is presented in the form of Ten Commandments: these are guidelines for design, construction, choice of equipment, commissioning, and day-to-day operation.

Common problems that were found were related to refractory, burners, reactors and catalysts, condensers, and process control. Some problems are related to design features but many other are due to improper installation of the equipment. Most importantly, attention must be given to day-to-day operation and handling of conditions that are outside of the normal design parameters

### Introduction

The Sulphur Recovery Unit (SRU) at first sight is not a very complex unit. How is it then possible that SRUs can have so many operational issues? As licensor of SRU technologies, Comprimo is frequently contacted by clients for support and advice when the SRU is not running properly; its technical experts have gathered practical experience from sites all over the world.

Common problems were found to be related to refractory, burners, reactor and catalysts, condensers and process control. Some are related to design features (to be taken care of by the licensor), but more depend on knowing what to install and how to install it. Most importantly, attention has to be given to day-to-day operation and how to handle out of bound operation. This paper presents guidelines for design, construction, choice of equipment, commissioning and day to day operation, illustrated with pictures and operating data.

Taking these Ten Commandments to heart will help operating companies to operate and maintain their SRUs properly and live happily ever after.

## Commandment 1 – You shall install a good burner and pamper it

The main burner is the heart of the Claus process. Select and operate a main burner correctly and your plant will experience good performance and longevity. Select and operate a main burner incorrectly and your plant will be in a world of hurt.

There are essentially two types of burners in use in sulphur recovery units. On the one hand, there are low intensity burners, also called pipe burners which can be found in a lot of older units. These low intensity burners do not mix the combustibles and combustion air well and as a result the burners experience long, extended flames. Some of the components that cause problems in an SRU, such as heavy hydrocarbons, ammonia and BTEX, are not fully destroyed. In addition, due to the poor mixing, there is potential for oxygen slip to the catalytic converters, resulting in catalyst deactivation and lower sulphur recovery efficiencies. Especially during hot standby operation this can cause severe issues if the oxygen reacts with the elemental sulphur in the catalyst.

Comprimo has seen several examples of the impact of poor mixing in a main burner. In one case during hot standby the combustible mixture extended all the way to the tubesheet of the waste heat boiler, where the mixture ignited and damaged the tubesheet and tubes. In addition, it resulted in the deposit of a layer of soot in the tubes and the downstream reaction stages. The real problem was later identified to be related to the injection location of fuel gas for co-firing. In another scenario, the low intensity burner was used with low oxygen enrichment levels (23%), which resulted in a substantial modification of the flame characteristics. As a result, the burner was destroyed in less than a week.

Nowadays high intensity burners are the industry standard. With their short flame and excellent mixing characteristics, the heating is uniform without hot spots. This said though there are still potential concerns with high intensity burners if they are not operated correctly.

The main issue with every burner is backfiring (Figures 1-3). This happens when the flame speed is higher than the velocity of the gas coming out of the burner throat. A sufficiently high pressure drop over the burner throat is a good indication of sufficiently high gas velocity. This means that the pressure drop over the throat can be used to monitor possible backfiring. A low differential pressure alarm helps to keep the pressure drop above the minimum allowed value to prevent back-firing.

But when backfiring does occur it may damage the burner cone. In most cases the pressure drop over the burner will then decrease further. Consequences of a damaged cone for the process are insufficient mixing (which can lead to slip of oxygen, ammonia or BTEX) and possibly flame impingement on the refractory.

### Lessons learned from Commandment 1:

- Install a high intensity burner.
- Prevent backfiring by monitoring the differential pressure across the burner.
- Maintain a low flow trip.



**Figure 1. Burner throat showing backfiring**



**Figure 2. Burner throat damaged by backfiring**



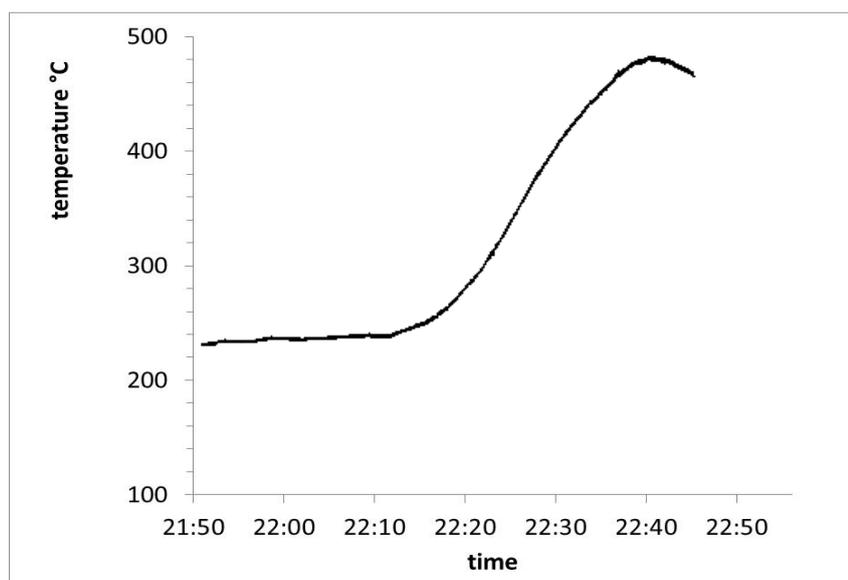
**Figure 3. Undamaged and damaged burner throat**

## Commandment 2 – Do not spoil your air

In normal Claus operation, there will not be any free oxygen present in the process gas downstream of the reaction furnace, provided the SRU is equipped with a high intensity main burner and with steam reheaters. The flow of combustion air is controlled to get the proper  $H_2S-SO_2$  ratio or, in the case of a SUPERCLAUS® or EUROCLAUS® unit, the proper  $H_2S$  concentration. The problem with oxygen slip past the reaction furnace in an SRU is that it deactivates alumina Claus catalyst and, in higher concentrations, can lead to sulphur fires when liquid sulphur has accumulated.

A common scenario where oxygen can be a problem is in hot standby operation, when the unit is taken out of acid gas operation for a short duration and sulphur is present in the unit. To keep the unit hot, natural gas should be combusted, but in many units, fuel gas with varying composition is still used. The stoichiometry of combustion during this mode of operation should be 90-95%. Operation with less than 90-95% of stoichiometric air will cause the formation of soot (typically below 85% for fuel gas and 75-80% for natural gas) and potential deposition on the catalyst beds; operation with excess air will result in oxygen breakthrough to the catalytic converters, where it causes catalyst deactivation and/or sulphur fires.

A common mistake made when firing natural gas is adding too much air. During a recent start-up of an SRU, the unit was being fired on acid gas with natural gas co-firing, when suddenly the acid gas supply was cut. Since the air control was still on MANUAL (because of the start-up), the fuel gas flame became over-stoichiometric. The excess of oxygen caused oxidation of sulphur in the first converter. This is illustrated in Fig. 4, where the bed bottom temperature started to increase from a steady 230°C to almost 500°C. Luckily, in this case the peak temperature was not high enough to cause permanent damage to the catalyst or the reactor.



**Figure 1. Increase of bed bottom temperature of first converter after over-stoichiometric firing of fuel gas**

Common locations for sulphur fires are the catalyst beds and the demisters in the sulphur condensers and coalescer. Especially demisters are very vulnerable. Rapid oxidation of sulphur and iron sulphide, which is always present in the demisters, will quickly destroy the wires (Fig. 5). The oxidation can also become a sulphur fire, destroying the condenser outlet channel, coalescer and tail gas piping.



**Figure 2. Damaged demister pads**

### **Lessons learned from Commandment 2:**

- Prevent the slip of oxygen downstream of the reaction furnace.
- Ensure sub-stoichiometric combustion during hot standby operation.
- Use natural gas with a stable composition instead of fuel gas.
- Limit MANUAL operation of the combustion air controls.

### **Commandment 3 – Operate your furnace hot, but not too hot!**

The reaction furnace is an essential component of an SRU. The temperature in a typical SRU is generally above 1000°C, however depending on the contaminants in the acid gas, higher temperatures may be required for proper destruction of these components. For example, when the acid gas streams contain BTEX, a temperature of at least 1050°C is generally required. This temperature may be even higher depending on the BTEX concentration and available residence time in the furnace. For ammonia destruction the minimum temperature is over 1250°C and a function of the concentration of ammonia in the mixed acid gas and available residence time.

Specifying the correct temperature is not so difficult. But how do we know what the actual temperature in the furnace is? Several papers have been devoted to the difficult task of measuring the furnace temperature<sup>(1-4)</sup>. Comprimo recommends using both thermocouples and infrared pyrometers. In the past

thermocouples were largely avoided for continuous operation due to their short lifespan in the severe conditions of a sulphur plant furnace. Nowadays, there are reliable thermocouples, specifically designed for sulphur plant operation, that can and are used to measure the temperature in the reaction furnace<sup>(5)</sup>.

However even with the installation of temperature elements incidents cannot always be avoided, as will become clear from the following case. This particular SRU was new and had one thermocouple and two pyrometers on the furnace. During start-up on fuel gas the temperature appeared to be too low, at least on both pyrometers which indicated 1000°C. The thermocouple indicated an end of scale temperature around 1600°C. The operator believed that the values of the two pyrometers were correct and increased the fuel gas flow. Because of a problem with the WHB BFW control valve, more boiler feed water was taken in than necessary and consequently the steam pressure did not rise enough, so the fuel flow was increased even more. By the time the operator decided to add steam to quench the fuel gas flame, this was not necessary anymore: a furnace shell rupture occurred after the refractory had failed at a temperature of over 1700°C. This temperature was later estimated from the damage to the refractory. The analysis afterwards showed that the pyrometers were not purged sufficiently, resulting in deposits of water, sulphur or soot on the pyrometer glasses. The purge problem may have been aggravated by a previous power failure when also instrument air and nitrogen had failed.

Another common mistake is applying insufficient or no steam during hot standby operation (fuel gas firing). At near-stoichiometric combustion the temperature of a fuel gas flame can easily be greater than 1700°C. Therefore, steam has to be supplied to cool the flame. We have seen many cases of damaged refractory because of inadequate steam supply. Common causes were found to be:

- Operator not trained for this situation.
- Poor operator instructions.
- Faulty steam gauge.
- Faulty temperature indication.

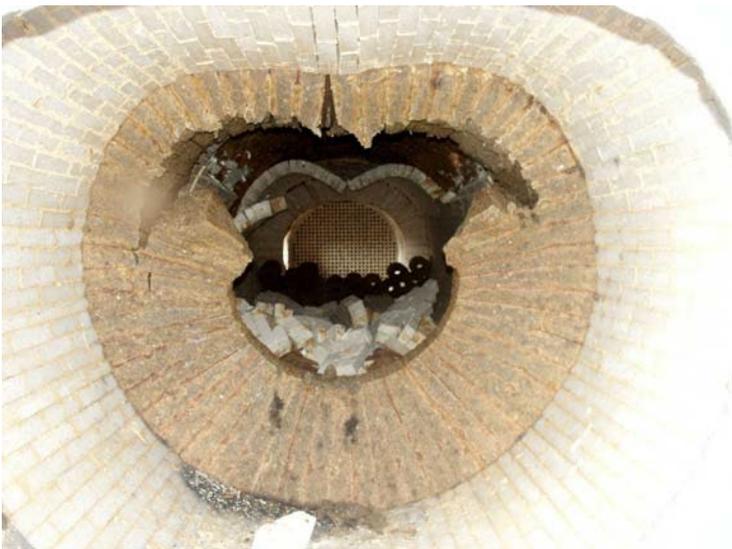
When operating with oxygen enrichment, more awareness is required regarding the operating temperature in the furnace. Oxygen enrichment results in temperatures in the furnace closer to the limitations of the refractory and therefore the margins for error will be lower. Also, oxygen enrichment should NEVER be combined with natural gas firing only, as the temperatures will rapidly exceed the limitations of the refractory.

### Lessons learned from Commandment 3:

- Install reliable and redundant temperature measurement devices.
- When thermocouple and pyrometer show conflicting temperatures, base operational decisions on the highest temperature.
- Purge nozzles sufficiently and check regularly. Do not save on nitrogen!

### Commandment 4 – Refractory design is serious business; ask the experts to do the design

As indicated before, the reaction furnace of an SRU plays a crucial role in the healthy operation of the unit. Due to the high temperatures in the furnace, refractory has to be installed to protect the carbon steel shell of the furnace. The proper design of this refractory is very often overlooked when designing and building a sulphur recovery unit and the results can be catastrophic (Fig. 6, 7). There have been numerous examples of failed refractory systems in sulphur recovery units <sup>(6, 7)</sup>.



**Figure 3. Caved in refractory**



**Figure 4. Damaged shell after refractory failure**

The critical time for refractory failures is always during start-up and shutdown operation. It is during this time that the largest movement of the refractory takes place, and the greatest risk exists that the bricks move to such an extent that they no longer are keyed in. Therefore, on top of a proper design, it is essential to limit the temperature rise during start up and the temperature decrease during shutdown respectively. Refractory vendors supply a temperature-time curve that should be followed.

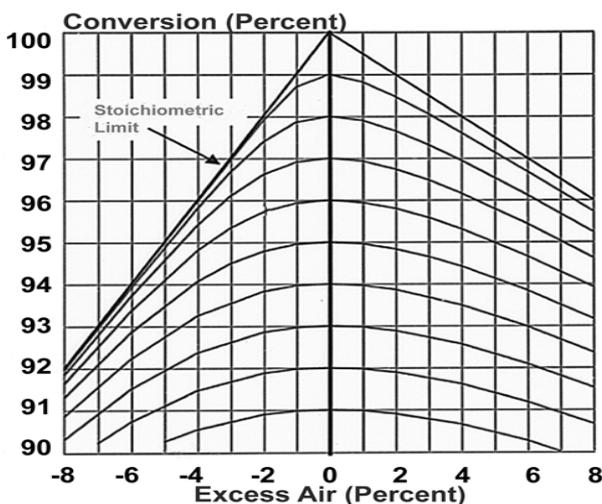
Comprimo recommends that a two-layer brick refractory system is always employed with sufficiently large bricks. The larger the furnace the thicker the refractory layers need to become. We believe that it is essential that the refractory design is done by a specialized refractory company that has specific experience in sulphur recovery units. This ensures that the right materials and right conditions are considered in the design. In the design of the refractory system, the implementation of the weather shield shall be included in the scope of the refractory designer.

**Lessons learned from Commandment 4:**

- Have your refractory designed and installed by experienced companies.
- Use at least a two-layer brick system.
- Limit the temperature rise and fall during start up and shutdown.
- Don't forget about the weather shield.
- Don't install insulation.

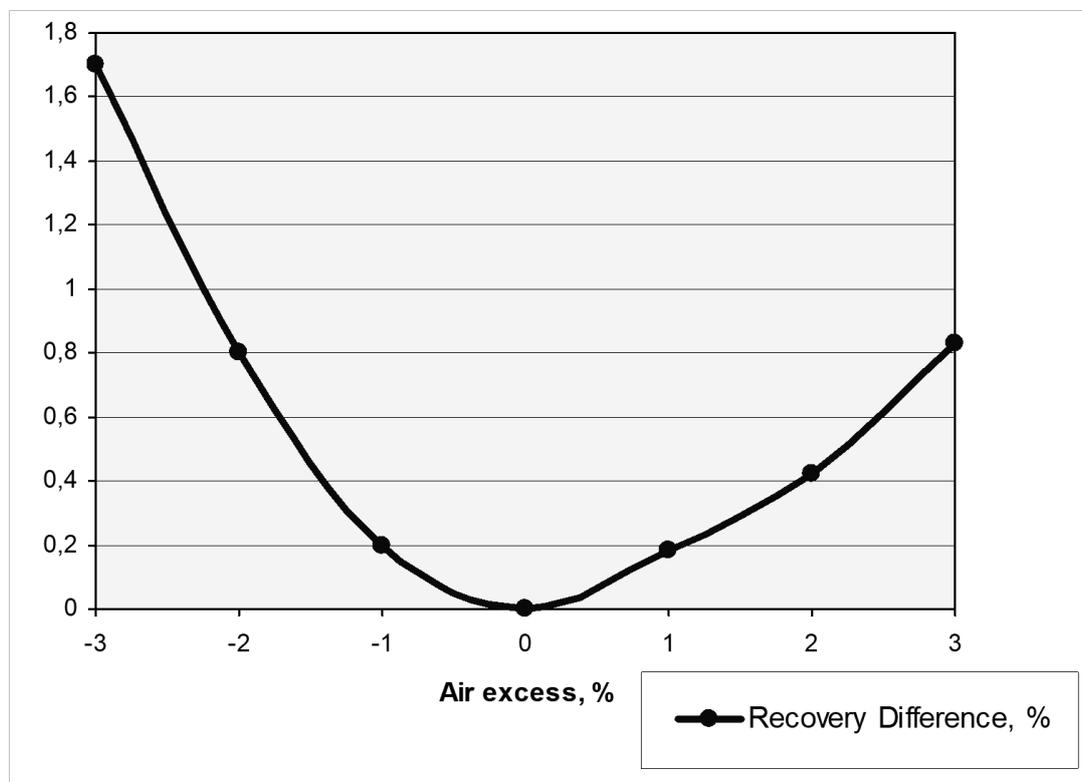
**Commandment 5 – Keep your plant in control**

The control of the air supply to the main burner is crucial in the maintenance of the optimal sulphur recovery in the SRU. In Fig. 8 the relation is shown between stoichiometry and conversion to sulphur.



**Figure 5. Sulphur conversion as a function of main burner stoichiometry**

This corresponds with the impact on overall sulphur recovery efficiency shown in Fig. 9:



**Figure 6. Loss of Sulphur Recovery Efficiency as a function of main burner stoichiometry**

From these graphs a shortage of 2% in combustion air can result in a sulphur recovery efficiency loss of 0.8%. This is especially essential for the operation of a conventional two or three stage Claus unit where the ratio of  $H_2S$  to  $SO_2$  needs to be equal to 2 to get the optimal recovery. The effect becomes less severe with the installation of tail gas treating units such as SUPERCLAUS® and TGTUs. For SUPERCLAUS® operation the main burner is operated off-ratio to minimize the  $SO_2$  concentration into the selective oxidation reactor. In that case operation is on the left-hand side of the graph. For amine based TGUs the right-hand side (excess side) is relevant, as too much air will result in high  $SO_2$  concentrations in the tail gas and a large temperature rise over the hydrogenation reactor.

By applying a dedicated control scheme, such as Comprimo' Advanced Burner Control (ABC) system, the air control can be improved. Even better control can be achieved by implementing a feed forward analyzer such as in ABC+<sup>(®)</sup>, Fig.10. Recently this system was extended to include the analysis of ammonia, so that it now can be used for the control of amine acid gas and SWS gas.

During implementation of this system the value of the ABC+ system was shown in the following incident. An unexpected high carry-over of hydrocarbons occurred that did not cause an upset of the tail gas  $H_2S$  concentration.

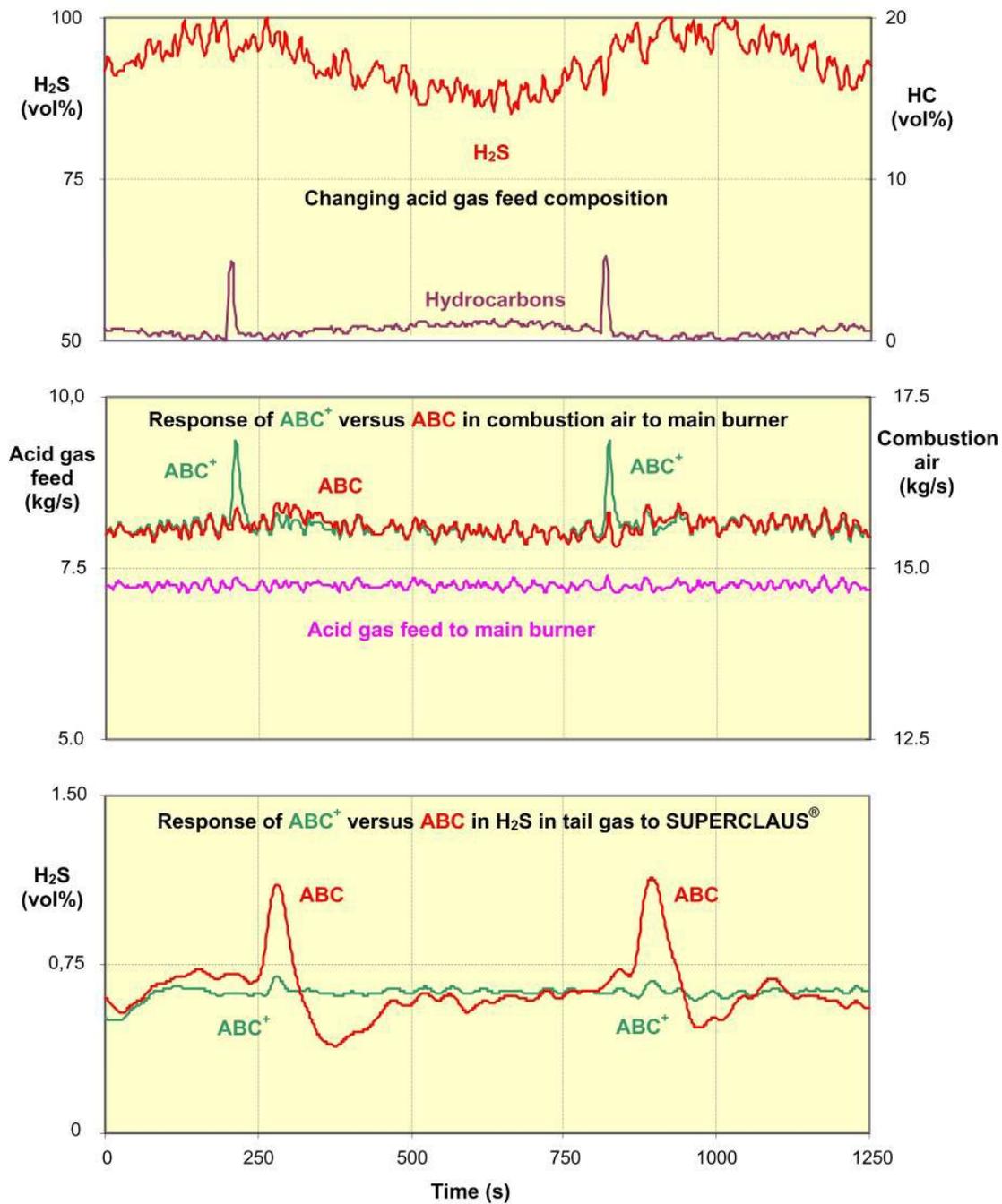


Figure 7. Improved burner control response using ABC+

**Lessons learned from Commandment 5:**

- Install a well-tuned and flexible control system for the combustion air.
- Consider the installation of an acid gas analyzer in the control system.

## Commandment 6 – Maintain your tail gas analyzer

Without any doubt, the tail gas analyzer/controller is the most important piece of instrumentation of a sulphur recovery unit. As was shown in the previous commandment, control of the combustion air flow to the Claus burner is important for an optimum sulphur recovery. Ultimately the control information has to come from the tail gas analyzer, if possible, with the help of an acid gas analyzer.

Both tail gas and feed gas analyzers are rated as complex and require on average four man-hours maintenance per week. Quite often the analyzer spares are overlooked, which is also something to be managed. As an example, we once had to take analyzer parts from one SRU train to get another train running.

But how do you know that your analyzer is working correctly?

Firstly, do not assume that the analyzer is correct and keep your eyes open for inconsistent behavior. When both  $H_2S$  as  $SO_2$  are increasing or decreasing at the same time the analyzer is not working properly. Similarly, when the values do not change or both show zero values, there is something wrong. Also other operating parameters such as reaction furnace temperature, temperature profiles across reactor beds, incinerator temperatures and stack analyzers can be used to determine whether the operation of the tail gas analyzer (or acid gas analyzer if implemented) in the control system is correct.

A striking example is what happened in an SRU with an amine TGTU. The tail gas analyzer sample connection was blocked, and the analyzer still showed a high  $H_2S$  concentration. The operator reacted quickly and increased the combustion air flow to reduce the  $H_2S$  concentration. But he did not look at other parameters and focused only on the indication of the tail gas analyzer, which showed no response. Since the operator did not see an improvement, he continued to increase the air flow until (as was proven later) finally all the  $H_2S$  was being converted into  $SO_2$  and  $SO_3$ . At this high  $SO_2$  concentration the hydrogenation reactor of the TGTU overheated until all  $H_2$  was consumed. Subsequently there was  $SO_2$  breakthrough to the quench column and amine absorber. The solvent became very acidic (HSS), resulting in corrosion in the regenerator overhead system. Within a few hours, holes in the overhead piping were seen and the unit had to be stopped. Also, the hydrogenation catalyst had to be replaced.

Especially when doing a start-up, we always check the analyzer readings by taking gas samples near the analyzer sample point. For instance, when starting up a SUPERCLAUS® unit for the first time we like to convert the catalyst to the sulphated state by gradually increasing the  $H_2S$  concentration. Quite often we have to correct the analyzer reading to get to the desired  $H_2S$  level and associated temperature rise over the catalyst bed. Sometimes the analyzer reading is off by a factor of 2 or more. In such a case calibration is needed and the ranges have to be verified, both in the field as well as in the DCS. A good indication of the validity of the analyzer readings can be obtained by monitoring other parameters, such as catalyst bed temperatures (SUPERCLAUS® and Claus reactor, hydrogenation reactor TGTU) and excess hydrogen concentration (downstream the hydrogenation reactor in case of a TGTU).

As a last comment to proper operation of a tail gas analyzer, the designer shall always remember that there is elemental sulphur present in the process gas that is measured. Ensure that the sample lines are as short as possible and properly heated. Also make sure that sulphur vapor is condensed in the sample probe before it hits the analyzer. Select only well proven analyzers and if possible, select an analyzer that is directly mounted on the tail gas piping. The location of the sample point is also very important: substantial problems were experienced at one plant where the analyzer sample point was located in the outlet channel of the final condenser next to the demister box. In that case, the process gas did not actually flow by the sample point. So, the sample was withdrawn from a stagnant zone where the composition of the gas was not representative of the actual operation. A common problem is plugging of sample lines by sulphur because of poor tracing/jacketing.

### Lessons learned from Commandment 6:

- Select a reliable tail gas analyzer.
- Use short sampling lines.
- Keep the sample line hot.
- Do not blindly trust your tail gas analyzer.
- Regularly perform maintenance.

### Commandment 7 – Keep your equipment and lines warm

A key success factor for good operation of an SRU is keeping equipment and lines in the unit hot. A sulphur recovery unit contains gas streams that are saturated with elemental sulphur and contain high concentrations of water as well. When a sulphur plant is operated above the melting point of sulphur and the dewpoint of water, the corrosion rates on the equipment and piping are very low. Even with the presence of H<sub>2</sub>S and SO<sub>2</sub> in the process gas, it has been Comprimo' experience that a well heated plant can have a long life without substantial corrosion. A lot of research has been done on the mechanisms of corrosion in sulphur recovery units and the main culprit for corrosion has been determined to be wet solid sulphur corrosion <sup>(9)</sup>.

So how does one prevent the presence of wet solid sulphur in a sulphur plant?

Apply heating in the unit, and lots of it. Especially in areas where gases are near the dewpoint of water, it is essential to install proper heating to prevent excessive corrosion from occurring. The main areas that Comprimo considers essential for heat maintenance are tail gas lines and vent gas lines from sulphur pits. In the case of SUPERCLAUS® units, a bypass line is installed around the Selective Oxidation Reaction stage, in which during normal operation gases saturated with sulphur could be stagnant. For all these lines we recommend installing sufficient heating. For smaller units this may be done by jacketing, however for

larger units ControTracing of these piping systems is becoming more and more prevalent. This also includes equipment such as coalescers.

So, what can happen when tail gas and vent air lines are not properly heated? This can be seen in Fig.11.



**Figure 8. Corroded tail gas line**

Comprimo has been in numerous plants over the years where the freezing of sulphur and condensation of water in tail gas piping has caused substantial problems. In one plant, our client indicated that they were fully aware that it was only the insulation on the line that was keeping the line together. In another plant a 40" line was found to be 1/3 full of frozen elemental sulphur. In this plant, the owner employed two welders full time to patch up their 500-meter-long tail gas line to their tail gas unit.

When designing the heating system for lines that may be subject to wet solid sulphur corrosion, it is essential to consider all of the conditions that are present in the plant. This includes items such as ambient conditions, steam pressure, gas flows, etc. In order to prevent corrosion problems from occurring it is essential that the wall temperature of the piping and equipment is maintained above the water dewpoint everywhere in the system. A single steam tracer on the bottom of the tail gas line will not do!

In the following example the SRU (a 2 stage Claus with SUPERCLAUS®) was running in normal operation with the SUPERCLAUS® bypass line installed and isolated. After 6 months, a large leak in the 42" carbon steel bypass line was found due to massive and rapid corrosion (Fig.12).

The failure was along a butt weld starting in the 12 o'clock position. The steam tracing of the bypass line had never been turned on and hot air purge was not available yet.



**Figure 9. Corrosion of SUPERCLAUS® bypass line**

#### **Lessons learned from Commandment 7:**

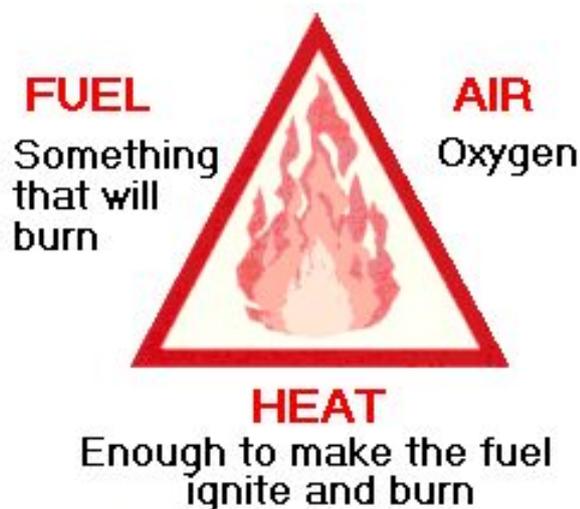
- Assume that condensation will happen, unless you do something about it.
- Assume that even when there is no flow, condensation will happen because of gases diffusing to the cold spot.
- Supply enough heat, most often a few tracers are not enough to keep a big line warm.
- When applying tracing/jacketing, go for engineered solutions.

#### **Commandment 8 – Prevent the accumulation of sulphur, prevent plugging, and do the flush test**

The accumulation of liquid sulphur can lead to a sulphur fire<sup>(10)</sup>. For this to happen, air (or better: oxygen) has to be present in sufficiently high concentrations (>10%). At lower concentrations of free oxygen in the process gas the sulphur can still be oxidized to SO<sub>2</sub> thereby generating extra heat of oxidation but without flame. A sulphur fire can also lead to corrosion that is higher than normally experienced because of the formation of SO<sub>3</sub>.

Besides sulphur vapor (from liquid sulphur) and oxygen (>10 vol.%) there has to be an ignition source (Fig.13). The following sources have been identified:

- High temperature, above the auto-ignition temperature of sulphur. The auto-ignition temperature most often mentioned is 230°C but values ranging from 190°C to 261°C have been reported, possibly linked to the sulphur particle size.
- Pyrophoric iron sulphide (FeS). When exposed to air (oxygen) it will ignite a sulphur/air mixture
- Static electricity generated by agitation of liquid sulphur. Because sulphur is an electric insulating liquid with a high dielectric constant it can easily generate enough static electricity to cause a spark ignition.



**Figure 10. Conditions for a sulphur fire**

In one example (Fig. 14) the 3-stage Claus unit in a refinery was operating without problems.

However, because of a small instrument problem the acid gas was taken out and the unit was operated in hot standby mode. After approx. 30 minutes a sulphur fire was seen in the first reheater. Sulphur had accumulated in the first reheater and could not drain because of an incorrect slope of the reheater. Also, the sliding strips in the reheater shell prevented proper draining of sulphur. In hot standby mode, the air/fuel gas ratio was too high and consequently air contacted the accumulated sulphur. The ignition source is not known, it could be either pyrophoric iron sulphide or hot reheater tubes. Initial oxidation of sulphur by air may have increased the temperature in the reheater to above the auto ignition temperature.

On the first signs of a fire (rising temperature), the operator reacted by admitting more air to the unit, thereby increasing the fire. The way this incident started is similar to the earlier case of a fire in the first converter (Commandment 2). However, because of the extra quantity of sulphur, in this case there was damage to the equipment (Fig. 15, 16).

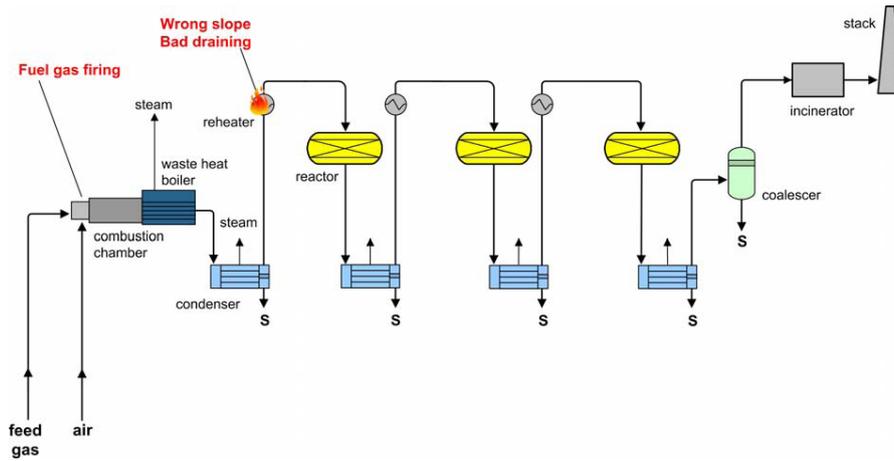


Figure 11. Sulphur fire at the first reheater Case

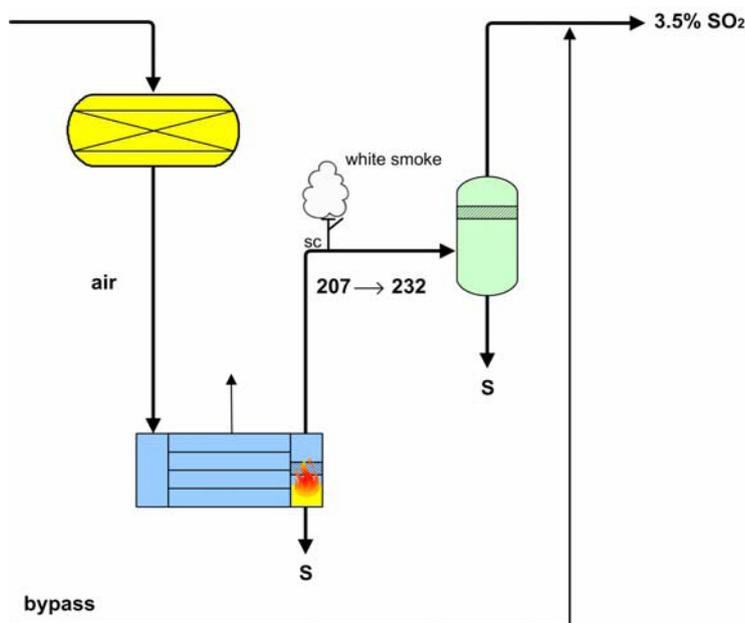


Figure 12. Damaged reheater tubes



Figure 13. Damaged reheater shell

In another example the 2-stage Claus unit in a gas treating plant (followed by a selective oxidation stage) had been running normally but the final condenser outlet temperature was higher than normal. During SUPERCLAUS® bypass operation, hot air was introduced to keep the SUPERCLAUS® stage warm to easily resume the normal SUPERCLAUS® operation. The condenser outlet temperature was seen to rise from 207 to 232°C in 20 minutes. Analysis of the coalescer outlet gas showed an SO<sub>2</sub> content of 3.5 vol.%, indicating a sulphur fire. When the sample valve (Strahman type) in the condenser gas outlet line was opened, white smoke was seen escaping. It was concluded that accumulated sulphur in the final condenser outlet channel was burning (Fig.17). Upon closing the air supply block valve, the fire was extinguished, and normal operation could be resumed.



**Figure 14. Sulphur fire in final condenser**

It was found that partial plugging of the final condenser rundown line had caused liquid sulphur to accumulate in the condenser outlet channel. Because of malfunctioning of the pressure control the condenser temperature had risen to above 170°C, thereby making the sulphur more viscous and making it harder to drain properly. Also, the control valve in the oxidation air line was leaking and causing some oxidation of sulphur. Upon admitting more air, a moderate sulphur fire started, that could be easily extinguished. Luckily in this case no damage to the equipment was observed. However, it might have been worse, see this example of a burnt down coalescer in a similar incident (Fig.18).



**Figure 15. Coalescer damaged by sulphur fire**

### Flush test

To detect partial plugging of rundown lines, locks and funnels the flush test is an excellent tool. It is recommended to do this test preferably every shift but at least every day. If this procedure is carried out consistently a partial sulphur blockage will be detected in an early stage, so that no liquid sulphur build-up can take place. Also, by this procedure partial plugging may be flushed out before it completely blocks the line.

The test is done by closing the run-down line and let a head of sulphur build up. On reopening, the operator observes the flow of sulphur in the look box (or sight glass) and a large flow of sulphur should be seen. If the sulphur is only flowing as normal, then there is a partial plugging. In such a case the flush test is repeated with the waiting time doubled, before reopening. This may help clean the condenser run-down line by flushing with a large flow. When this is not successful, the partial blockage is removed by rodding or by steam pressure.

Nowadays sight glasses have become in use more widely instead of the open look boxes. The gush of flowing sulphur can also be seen with properly dimensioned sight glasses, since during normal operation the flow only partly fills the glass. It may take some experience to judge the relative increase in flow based on the time that the glass stays filled.

### Lessons learned from Commandment 8:

- Ensure proper draining is designed in an SRU.
- Perform a flush test to determine whether partial or full plugging is experienced.

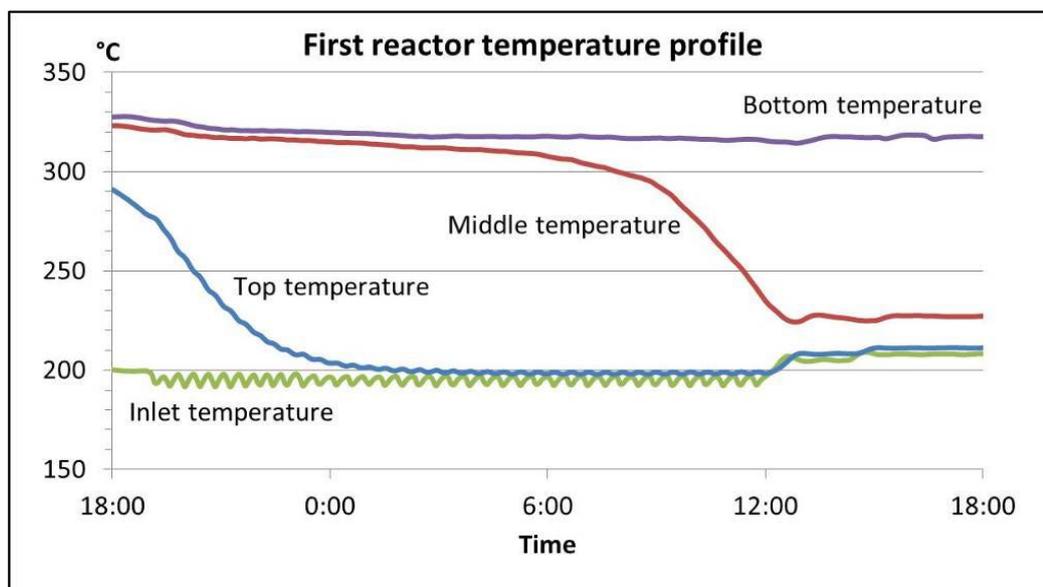
## Commandment 9 – Know your history

In the process industry it has been customary to use control charts to better control the processes. It is a systemized way to analyze how the average and the variation of certain process variables develop over time. As a tool it can be used to monitor process stability and control, but also as an analysis tool. Its use in SRUs has been scarcely documented <sup>(11,12)</sup>.

With the easy access to operating data in the DCS, the technique of a control chart has become less complicated. A download of DCS variables in a spreadsheet can be easily manipulated to give meaningful information. As an example, consider the graph of selected DCS data in Fig. 19.

It shows the temperature profile in the first Claus converter. For no obvious reason the temperatures in the top and middle of the bed decreased. What is effectively shown is that the catalytic activity in the bed decreased. This deactivation was found to be caused by condensation of sulphur on the catalyst. The correct countermeasure is also shown: increase of the inlet temperature; only the desired result was not achieved, a more drastic temperature increase was needed.

Fig. 19 shows that a select set of data can show you what is happening in the SRU.

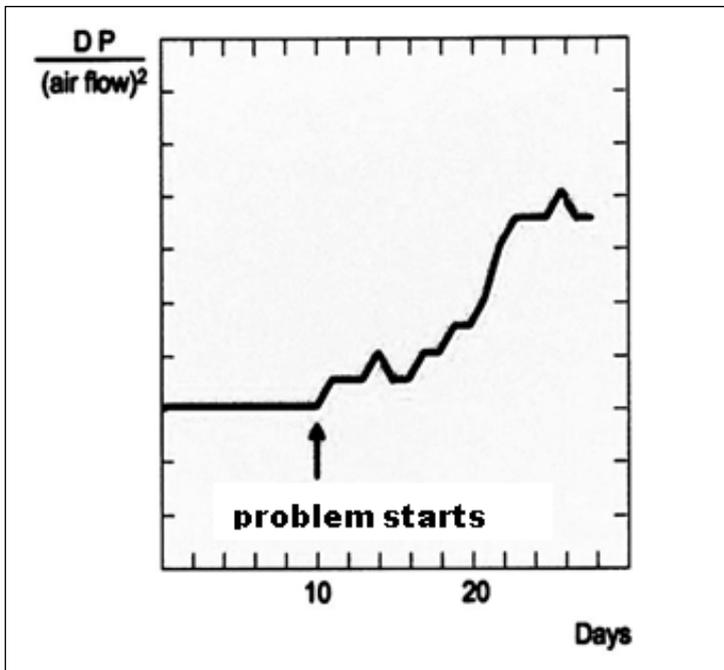


**Figure 16. Temperature profile in first converter**

Also, the temperature difference between catalyst bed thermocouples at the same height can be informative. A difference of more than 5°C is considered a sign of channeling in the bed. This may be caused by soot or other deposits or by 'duning' of the bed after a pressure wave. During turndown of a unit, the risk of channeling is more pronounced.

Another well-known variable to monitor is the pressure drop over the reactor. The pressure drop divided by the square of the gas flow is a measure of the resistance in the reactor (Fig.20). By monitoring this

variable an early indication of trouble can be obtained (e.g. soot). In most cases only the pressure of the combustion air upstream of the burner is monitored, so the resistance would then be given by the combustion air pressure/ (combustion air flow + acid gas flow) ^2.



**Figure 17. Monitoring pressure drop**

For SUPERCLAUS® or EUROCLAUS® operation it is important that there is enough air for the selective oxidation of H<sub>2</sub>S. Prolonged running with insufficient air deactivates the catalyst. Although the catalyst in many cases can be regenerated, this is an operation that is to be avoided. The stoichiometry of the selective oxidation is given by:

$$\frac{OAF * 0.5 * 20.9}{H_2S\% * (AGF + CAF)}$$

where:

H<sub>2</sub>S% is the reading from the tail gas analyzer

AGF is the acid gas flow

CAF is the combustion air flow

OAF is the oxidation air flow

This value should always be > 1, monitoring this value will ensure that the catalyst stays healthy.

### Lessons learned from Commandment 9:

The list of variables that can be monitored is large. But besides the normalized pressure drop, especially the following process variables are worthwhile to monitor:

- Tail gas analyzer air demand signal
- Combustion air pressure / square of gas flow
- Temperature difference over the reactor (bottom bed temperature minus inlet temperature; do not use the reactor outlet temperature since that may be lower than the bottom temperature).
- Temperature difference between catalyst bed thermocouples at same height
- CEMS SO<sub>2</sub> measurement

And for SUPERCLAUS® or EUROCLAUS® operation:

- Stoichiometry of the selective oxidation
- Temperature difference over different layers in the reactor (middle bed temperature minus inlet temperature, bottom temperature minus middle temperature and the ratio between the two).
- Temperature rise as function of the inlet H<sub>2</sub>S concentration.

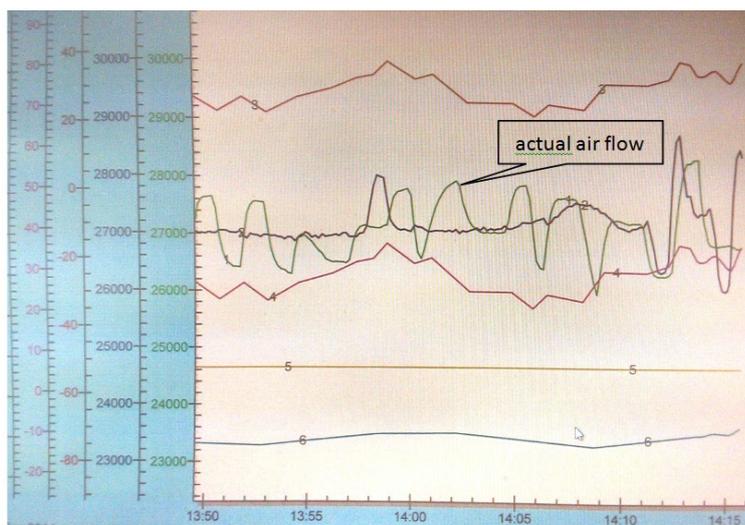
### Commandment 10 – Stabilize your feed

As discussed previously (Commandment 5), an SRU needs a good control of combustion air. But the best control cannot prevent instabilities if the changes in the feed gas are big. Also, here the saying 'garbage in is garbage out' fits.

Examples of instabilities are:

- Variation in the overhead temperature of the amine regenerator or the sour water stripper.  
A ripple of 10°C can cause an uncontrollable air/acid gas ratio.
- Sticky valves, these can be found both in the combustion air line and the acid gas line.
- Sticky blow-off valve of the air blower.
- Pulsation of roots blower.
- Fluctuations in refinery fuel gas composition.

An example of sticky valves is shown in the DCS trace of Fig. 21. The sticky combustion air valve caused a large fluctuation in air flow, which can lead to a lower sulphur recovery. Eventually the burner control was put in MANUAL (with all the risks involved) to get rid of the fluctuations.



**Figure 18. DCS trace showing fluctuating flow of combustion air due to sticky valve**

The overhead systems of the amine regenerator and sour water stripper shall be designed for all operating scenarios. In one case, the addition of a minimum flow protection loop for the amine regenerator reflux pumps had been omitted. So, during turndown of the unit the pumps could not be operated continuously, resulting in unstable operation of the amine regenerator. In addition to this instability, the SRU tripped when the operators forgot to restart the pumps when the high-level alarm came in for the reflux drum; a large quantity of reflux water was dumped in the acid gas KO Drum.

Process upsets such as foaming, and hydrocarbon carry-over shall be minimized. Therefore, the amine circuit shall be kept clean. This means that sufficient lean and/or rich amine filtration shall be considered in the design.

In a gas plant, adding filtration of the rich amine flow increased the time between lean/rich exchanger cleanings from two weeks to more than three months. For the sour water stripper, the main concerns are typically hydrocarbon carry-over and the design of the oil/water separation.

In a refinery, hydrocarbons from the feed of the unit could be eliminated and incidents due to hydrocarbon carry-over to the tower were reduced by installing sour water hydrocyclones between the sour water tank and the sour water stripper. The client indicated that the water from the hydrocyclone was clear as drinking water.

### Lessons learned from Commandment 10:

- Ensure smooth operation of the control valves.
- Proper design and operation of the upstream units is essential to maintain good SRU operation.
- Use natural gas in an SRU.

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