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White Paper.

Oxygen enrichment in Claus plants in view of the production of "clean fuels".

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Contents.

1. Introduction.

The production of clean fuels leads to an increase in ammonia generation as a by-product. The reason for this is that in severe hydrogenation of crude, with the purpose of converting organically bound sulphur into hydrogen sulphide (H_2S), some of the nitrogen also reacts, thus forming ammonia. Both the H_2S and ammonia end up in the Claus plant of the refinery which processes H_2S into elemental sulphur. It can also remove pollutants, particularly by decomposing ammonia. When a Claus plant's load with H_2S and ammonia is increasing, the plant may become a bottleneck. To overcome this problem, oxygen enrichment may be applied and has been applied already in more than 150 Claus units worldwide. So this is a well proven technology. But still myths and prejudices prevail regarding the effects of oxygen. This paper is to describe and quantify what will happen in a Claus plant when oxygen enrichment is applied.

NH_3 determination – sampling procedure of Claus process gas

2. Hardware.

Oxygen concentration near the injector outlet

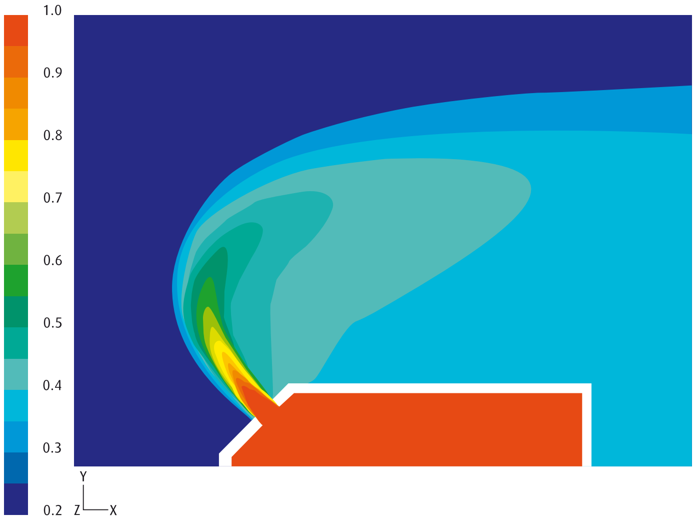

Fig. 1: OXYMIX® oxygen injector and the O_2 concentration in the gas stream calculated by CFD

The hardware required for oxygen enrichment looks simple and straight-forward. But it is worth a closer look to avoid difficulties in operation and safety risks due to faulty design. Therefore, a few important topics on hardware will be discussed here.

The injection of oxygen can be done directly into the pipe for the combustion air to the Claus furnace if the enrichment level is not higher than up to approx. 28 vol%. At higher O_2 levels, the injection usually is directly into the burner. In most cases, the injection is into the air pipe, which is normally made of carbon steel. It is mandatory that high levels of oxygen concentration are safely kept away from the carbon steel wall because, in the long run, the O_2 would react with the carbon and would slowly remove it from the steel. The result then is brittle steel which may fail without warning. The problem here is that the damage becomes visible only when it is too late to prevent it. Therefore, injection has to be done by adequate devices, e.g. the one shown in Fig. 1. It is designed using CFD (computational fluid dynamics) and ensures both good mixing of the combustion air with the oxygen and that a high concentration of O_2 is safely kept away from the carbon steel pipe.

The other important equipment is the control and safety cabinet required to meter the oxygen into the combustion air or into the burner. Of course, the main item there is the flow control valve. But in addition, the safety features are of high importance.

Fig. 2: FLOWTRAIN® for metering O_2 into Claus plants, ensuring fulfilment of the safety requirements

Any major deviation of the actual O_2 flow must be alarmed and – if the situation cannot be corrected – the oxygen flow has to be interrupted in a suitable way. Just switching off the oxygen at high concentrations would almost necessarily lead to a failure of the Claus plant, primarily due to too high temperature in the incinerator. If the oxygen for enrichment is provided from a liquid O_2 tank, temperature control in the piping must be provided to prevent liquid oxygen from entering the air duct. And during shut-down periods, e.g. in turn-around situations, an absolute separation between oxygen source and Claus plant has to be ensured, including safeguarding against creeping gas flows. A device in kind is shown in Fig. 2.

3. Ammonia decomposition.

NH_3 destruction versus temperature

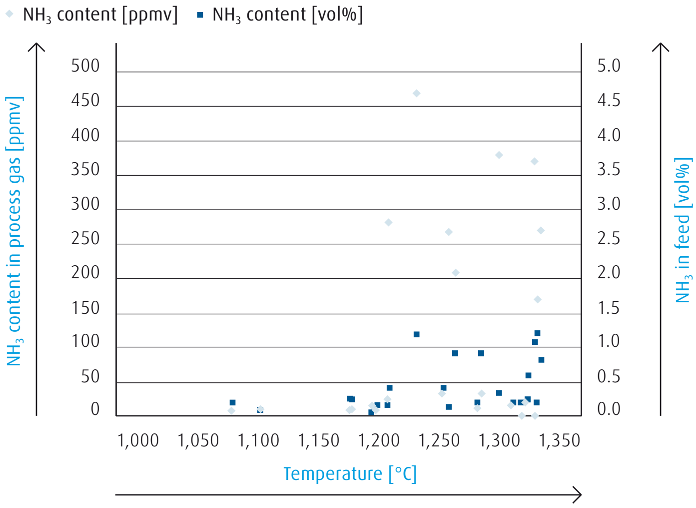

Fig. 3: Efficiency of ammonia destruction as a function of furnace temperature

NH_3 conversion = f(O_2)

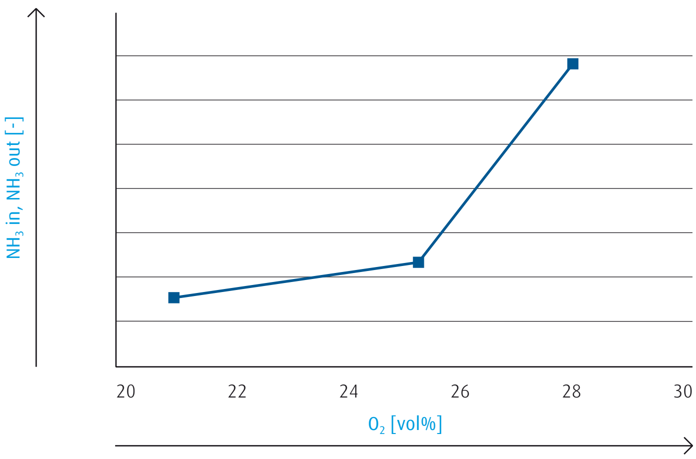

Fig. 3A: Ammonia destruction as a function of oxygen content

3.1. Temperature effects

Literature always claimed that, for efficient destruction of ammonia, a temperature of higher than 1,300 °C is required (see Paskall/Sames, 2003). Our measurements quantify this effect in more detail.

The diagram shows typical results from measurements in a 120 t/d Claus unit. Similar trends we found in other plants as well, though for different NH_3 levels. In this case, at a temperature of 1,100 °C, the decomposition is approx. 100:1, i. e. approx. 0.2 vol% of ammonia at the inlet is reduced to approx. 20 ppm at the outlet. At 1,300 °C, a content of approx. 3.8 vol% can be reduced to 70 ppm, i. e. the reduction is by a factor of 550:1. That means that ammonia decomposition takes place at 1,100 °C. But it can be greatly improved, in this case by a factor of 5.5, when increasing the temperature by 200 °C. At an even higher temperature of close to 1,350 °C, we measured in another plant even NH_3 destruction levels of down to a few ppm residual, i. e. the trend shown in Fig. 3 continues and may reach values up to 40,000:1.

As temperature is coupled with the oxygen enrichment level, the effects of ammonia destruction can be shown as a function of oxygen content (Fig. 3A). The diagram illustrates measurements in several Claus units, where the same tendencies were found, though the numbers varied. Up to 25 % oxygen enrichment, the effect of ammonia decomposition is a lot smaller than at higher levels. This underlines the claims in literature that high temperature favours ammonia destruction, and it does so with increasing efficiency with a higher O_2 content.

The typical operating temperature of a Claus unit with rich H_2S feed is in the range 1,100 °C to 1,200 °C. The conclusion to draw from our measurements is: though ammonia decomposition takes place in Claus plants with no specific means provided, really efficient ammonia destruction requires more. Our measurements in that respect confirm conventional wisdom. But what options does a refiner have? We start with the discussion of the efficiency of Claus burners for ammonia destruction.

NH_3 conversion = f(O_2), burner A

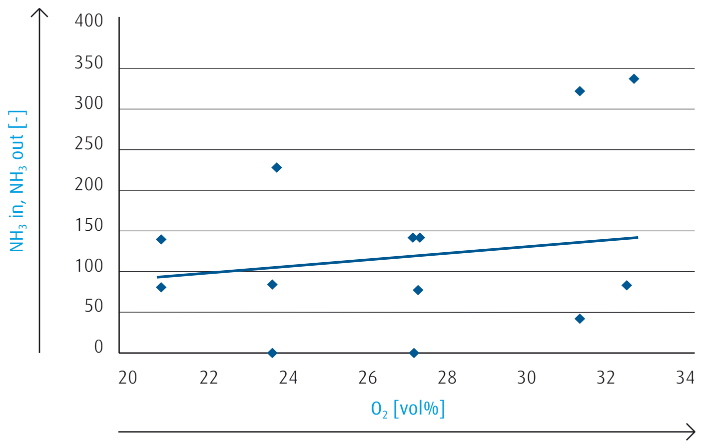

Fig. 4: NH_3 decomposition with burner A

3.2. Burner performance during oxygen enrichment

The selection of the burner and the operating conditions considerably affect the efficiency in ammonia conversion. In order to identify and learn how to control the parameters that influence the operation, Linde carried out tests in different types of Claus plants in a number of refineries worldwide. In these tests, we could quantify the differences between the burners applied and can now provide data on how to achieve an effective treatment of ammonia containing feeds.

During the tests, oxygen was injected in these Claus plants and the reaction of ammonia decomposition was monitored. Measurements of temperature and gas composition were taken and the efficiency measured with NH_3 in/NH_3 out as the parameter. Below, it will be called the ammonia decomposition ratio (= ACR). Results show that ACR increases as the oxygen content increases (Fig. 4, Fig. 5, Fig. 6). This was to be expected in view of literature data and the measurements described above. The combustion air is usually enriched up to 28 vol%, providing the required reaction temperature and the oxygen to be consumed in the exothermic part of the decomposition reactions with ammonia. The amount of excess oxygen not only depends on the required amount for the decomposition but also on several other factors like burner type, load and heater configuration. If the combustion air is enriched above 28 %, a new burner is normally needed to compensate the loss of velocity in the gas flow, thus avoiding damages to the burner.

The test results demonstrate that the highest ACR reached, using burner A (Fig 4.) and burner B (Fig. 5), is 350. In both cases, the combustion air was enriched at least to 28 vol% in oxygen content. In burner C, the ACR is much lower even though the air was enriched up to the same percentage (Fig. 6). With this information, we can conclude that the limitation of ACR is determined to a certain degree by the burner type and also that the greater the oxygen content, the better the ammonia decomposition.

NH_3 conversion = f(O_2), burner B

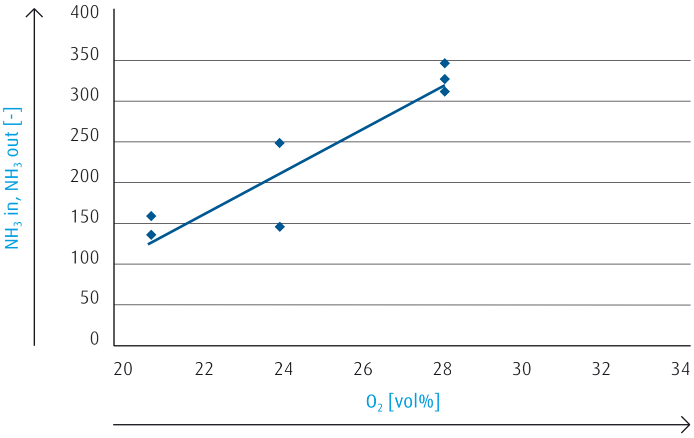

Fig. 5: NH_3 decomposition with burner B

NH_3 conversion = f(O_2), burner C

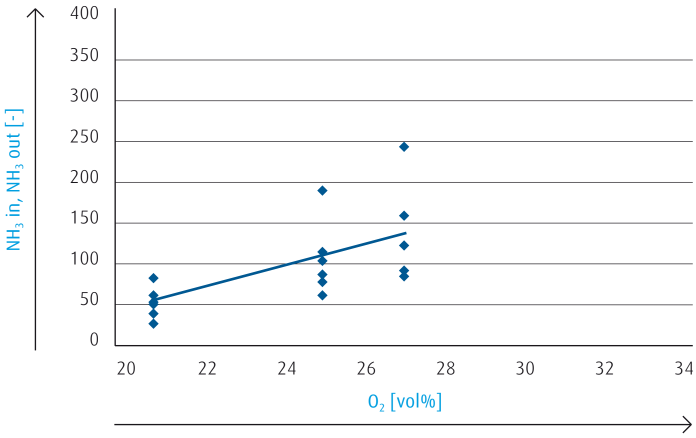

Fig. 6: NH_3 decomposition with burner C

Conventional versus optimised burner: temperature distribution

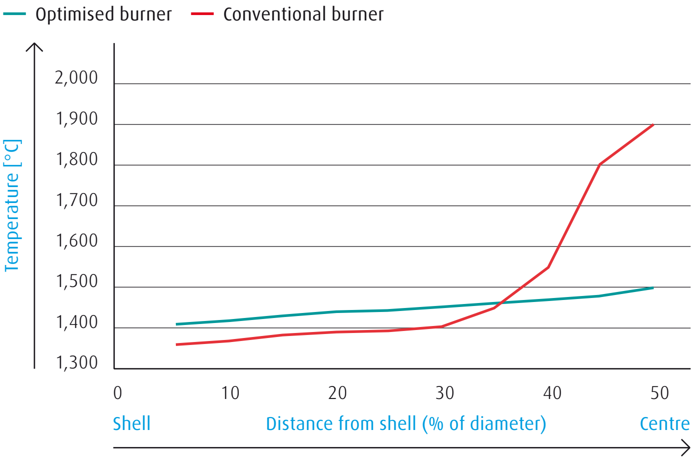

Fig. 7: Temperature distribution in a Claus furnace with a conventional burner with concentric lances and with a modern high-turbulence burner

The main difference between burners is their capability to mix the reaction gases. Some burners consist essentially of concentric lances where the reaction gases mix at the outlet. Other burner types have features improving gas mixture, so that at the outlet a highly turbulent flow is generated. These burners are more expensive. But they are definitely superior in promoting the contact between flows. The temperature distribution within the furnace was found to be more uniform (see Fig. 7). And this also showed better results, as ACR increased even at moderate temperature by one order of magnitude in comparison with the more simple burners (Fig. 8 versus Fig. 6). When the temperature was additionally increased to a higher level, ACRs as high as 35,000 were reached, i.e. virtually complete destruction of NH_3 (Fig. 9).

Temperature is another variable that significantly influences the performance in the furnace. The measurements so far showed that as the temperature increased, so did the ACR. To find the upper limits obtainable, we increased the temperature by air preheat. This resulted in an approx. 70 °C higher furnace temperature and an increase of ACR to more than 35,000 (see Fig. 9).

At high levels of oxygen enrichment, the oxygen content decreases the air flow. This necessarily reduces the energy available to mix the reaction gases. Without the proper mixing system, the ammonia and oxygen molecules do not meet and do not react. Furthermore, all oxygen may be consumed in a small flame zone leaving no free oxygen for reaction outside this zone, i.e. in outer parts of the reaction chamber. This may then lead to strains of ammonia containing gases that reach the outlet of the reaction chamber without having reacted, i.e. contaminating the downstream Claus sections. Therefore, in oxygen enriched operation, the selection of a proper burner becomes increasingly important.

The load plays an important role in the performance of the burner. As the load increases, so does the heat of reaction, while the heat losses remain constant; this is evidenced in the temperature increase in the furnace. As an example of the effects of the load, we measured that a load of 90 % generated an operation temperature of 1,210 °C and an ACR of 4,500, while a load of 65 % generated a temperature of 1,180 °C and an ACR of only 1,500.

In conclusion, efficient ammonia decomposition requires improvements in more than just one parameter. The variables that affect the success of removing ammonia in a Claus plant are: oxygen content, temperature and load. An aspect to take into account is that a burner with good mixing capacity may be several orders of magnitude more effective than a furnace with a more simple burner. A good burner is also more efficient than high temperature. Good mixing plus high temperature allow for virtually complete ammonia decomposition. The target conditions for a successful destruction are high temperature, high load, oxygen enrichment of the combustion air and the proper burner that provides good mixing of the gases. The selection of the right burner is the key to efficiency of ammonia decomposition.

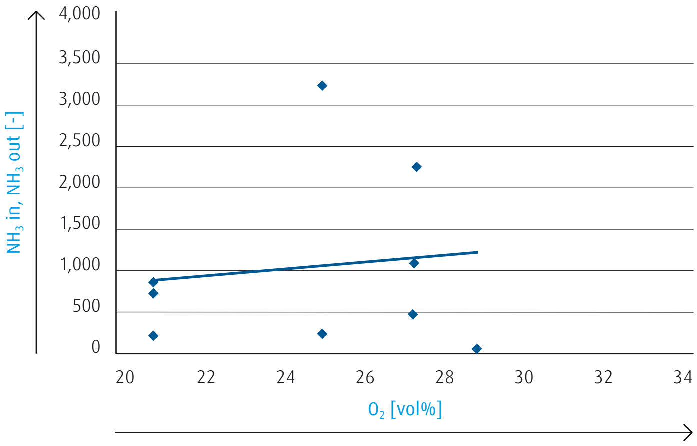

Fig. 8: NH_3 decomposition with burner D without air preheat

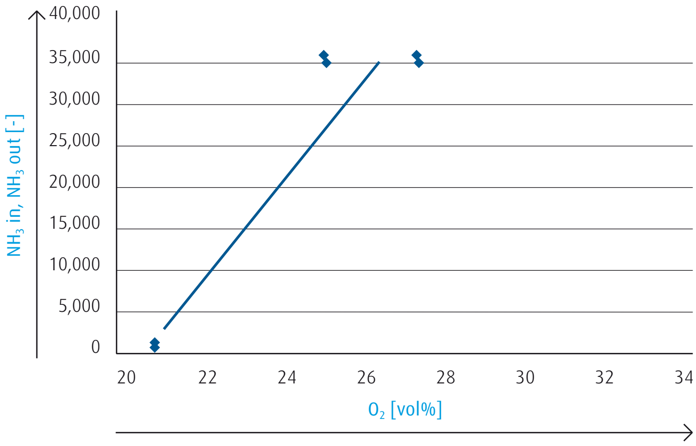

Fig. 9: NH_3 decomposition with burner D and with air preheat

3.3. The chemical route

For efficient ammonia destruction, it is imperative to understand whether ammonia is burned (Eq. 1) by reaction with oxygen or thermally decomposed (Eq. 2).

$4\ NH_3 + 3\ O_2 \longrightarrow 2\ N_2 + 6\ H_2O$, exothermic **Eq. 1**

$2\ NH_3 \longrightarrow N_2 + 3\ H_2$, endothermic **Eq. 2**

While Eq. 1 requires oxygen for the reaction, Eq. 2 just thermally destroys the molecule. Eq. 1 leads to a substantial increase in the furnace temperature while Eq. 2 reduces this temperature. By comparing the measured temperature values with thermodynamic simulation, we found that both reactions occur in parallel. The levels are different for different plants and also depend on the operating conditions. But typically, approx. 60 % of the NH_3 are burned; the balance is destroyed thermally. Altogether, this leads to an only moderate temperature increase in the furnace, much lower than combustion of ammonia would insinuate. This is also the background why oxygen enrichment for ammonia destruction is a realistic option in most Claus plants without having to replace the brick lining of the furnace.

4. Savings due to oxygen enrichment.

4.1. Hydrogen generation

Thermodynamic simulation shows that, due to oxygen enrichment, the H_2 content in the process gas rises substantially. Downstream the furnace, at 28 vol% O_2 and a rich H_2S feed, the H_2 content rises from a typical approx. 1 vol% with air operation to approx. 3 vol% with oxygen enriched operation. But is this effect true?

In a Claus plant with a SCOT tailgas treatment, we measured the H_2 content between the 2-stage Claus and the tailgas treatment, just downstream the 2^{nd} catalytic converter. The results were perfectly in line with the theoretic predictions of the thermodynamic simulation. In essence, for every molecule of O_2 applied in the furnace at 28 % oxygen enrichment, 0.2 molecules of H_2 are generated. When sour water stripper gas is added, the H_2 generation increases by approx. 15 % for each vol% of sour water stripper gas added to the feed.

SCOT is a hydrogenating type of tailgas treatment, i. e. any H_2 arriving at the tailgas treatment reduces the requirement for import of hydrogen from outside sources, e. g. through a reducing gas generator. This then allows to calculate the economic value of the H_2 produced versus the cost of O_2 applied in the furnace. Of course, the values of both H_2 and O_2 vary from site to site. But in general, one can state that savings due to the H_2 generation more or less compensate the cost of O_2 in such a configuration.

Fuel gas to incinerator

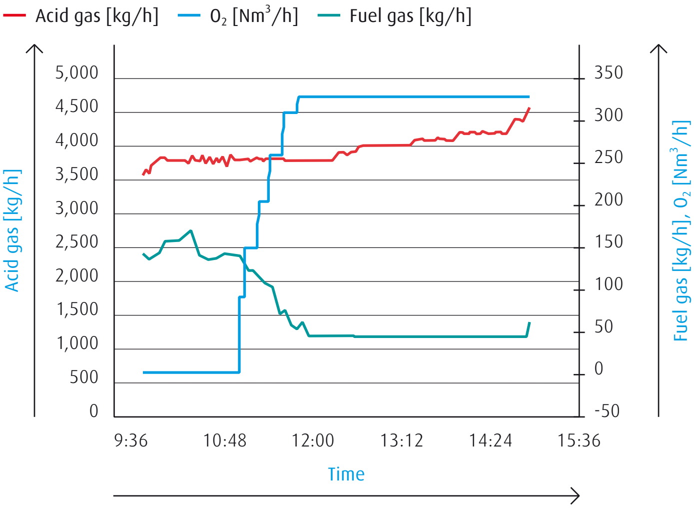

Fig. 10: Fuel gas savings due to oxygen enrichment

4.2. Fuel gas in the incinerator

Due to oxygen enrichment, the total gas flow through the Claus plant is reduced at constant acid gas flow. Subsequently, there is less gas to be heated up and cooled down in the heat exchangers of the plant and also finally in the incinerator. This effect is compounded by increased generation of hydrogen in the Claus furnace, as described above. This additional hydrogen serves as fuel gas in the incinerator if there is no H_2-consuming tailgas treatment in between. In a 3-stage Claus plant with no tailgas treatment, we measured a reduction of fuel gas consumption in the incinerator by approx. 70 %, though typically savings are closer to 20 %.

The increase of the acid gas flow in Fig. 10 is due to test conditions. The goal was to find the maximum sulphur capacity and therefore, at 28 % oxygen enrichment, the acid gas feed was increased to the maximum available level. In parallel, of course, the consumption of fuel gas increased as did the total gas flow.

5. Temperature effects of oxygen enrichment.

Furnace temperature versus oxygen content

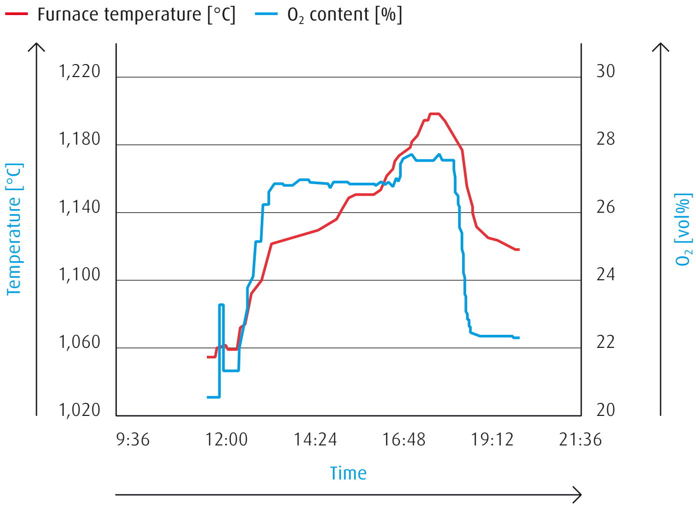

Fig. 11: Temperature increase in a Claus reaction chamber versus oxygen enrichment

5.1. Brick lining

The temperature increase in the Claus reaction chamber due to oxygen enrichment has been described many times in literature. Our measurements were perfectly in line with published data, i.e. per % of oxygen enrichment, the temperature in the furnace rises by approx. 15 to 20 °C, depending on insulation properties of the furnace's brick lining. Fig. 11 illustrates a typical example. It depicts the temperature increase when the oxygen content has been increased from air to 28 vol%.

The diagram shows that the temperature rises slowly with increasing O_2 level. The initial temperature of approx. 1,060 °C rises finally to a level of 1,180 °C. This Δt of approx. 120 °C is typical for oxygen enrichment by 7 vol%.

Brick linings are very different in each furnace. However, the lowest quality bricks usually have a design temperature of min. 1,350 °C. In almost all cases, this is sufficient to tolerate a temperature increase due to oxygen enrichment up to 28 vol%. We recommend to have a Δt of approx. 200 °C between the design temperature of the brick lining and the expected operating temperature. This allows for some fluctuations, e.g. due to controller fluctuations. Also, Claus plants usually have to cope with the gases the refinery delivers and this also adds to fluctuations. Anyway, only in rare cases, oxygen enrichment requires to upgrade or even replace the brick lining. In most cases, the existing lining proved absolutely adequate.

Evaluation of a test with O_2 enrichment

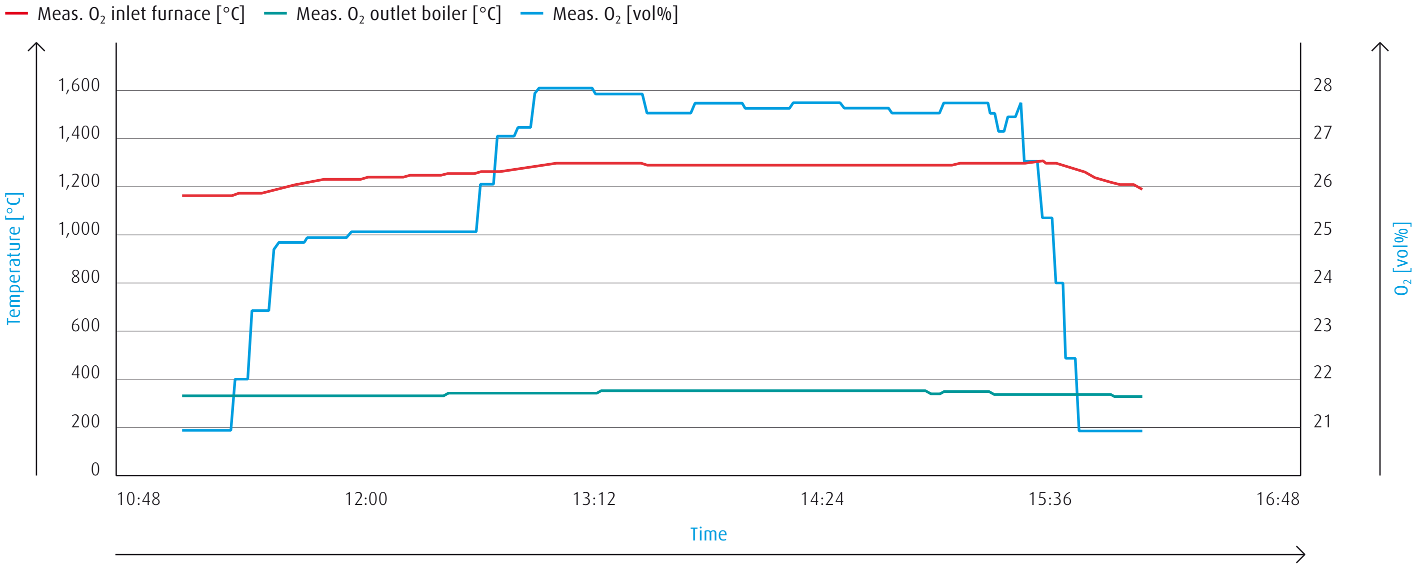

Fig. 12: Outlet temperature of waste heat boiler (WHB) versus oxygen enrichment level

5.2. Heat boiler of the Claus furnace

In the waste heat boiler (WHB), the process gas arrives at oxygen enriched operation with higher temperature. But at constant sulphur load, there is less gas. This makes the task of the WHB more difficult since the Δt increases and the heat transfer coefficient α decreases. In addition, the design has to keep gas velocity and heat transfer in balance to avoid sulphur mist or fogging. Fortunately, in most cases, the WHB was up to oxygen-enriched operation without requiring any changes.

However, this cannot be taken for granted in every case. Therefore, it is recommended to check the capability of the WHB by either measurement or calculation. Measurements will usually show a slight increase of the outlet temperature of the WHB of typically less than 25 °C. If the outlet temperature rises substantially more, there is the risk of film boiling in the heat exchanger and thus the risk of overheating. In such cases, replacing the WHB may become necessary. However, so far we did not come across such a case.

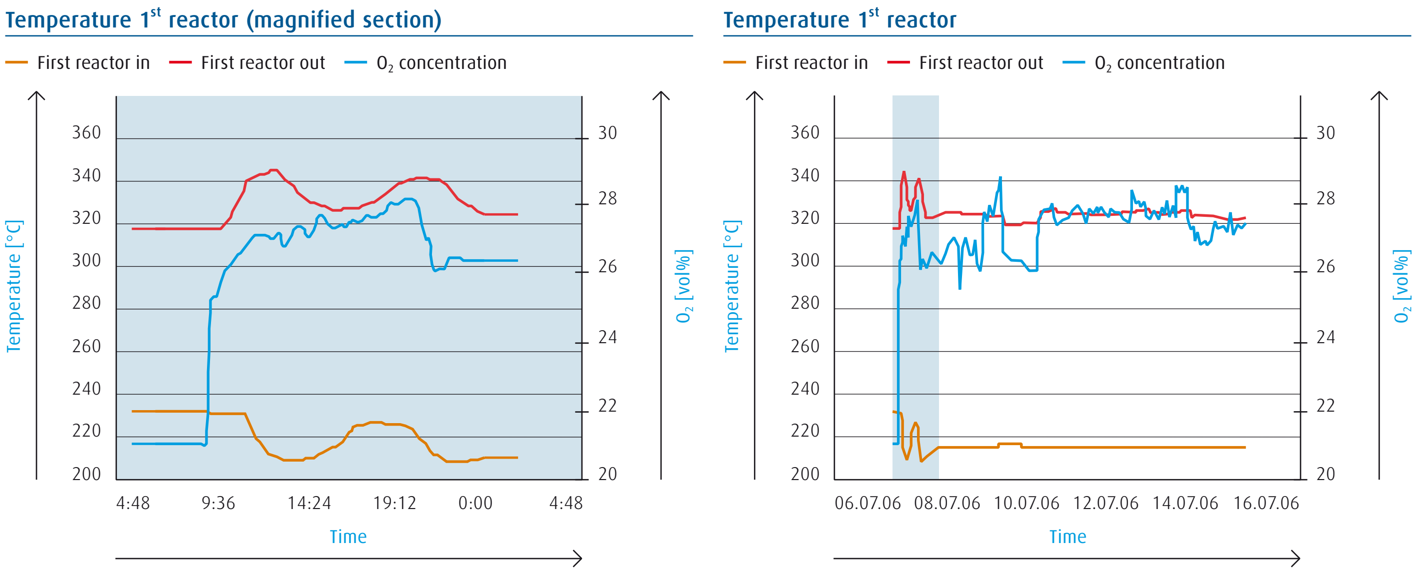

Fig. 13/13 A: Temperature increase in the 1st catalytic converter, physical effect plus side reactions; the left diagram is a magnified section taken from the right diagram.

5.3. The first catalytic reactor

As oxygen enrichment increases, the content of inert N_2 falls. This necessarily increases the concentration of the sulphur species in the process gas. As a consequence, the temperature in the 1st catalytic converter will rise. This effect, which is due to less dilution of the sulphur species, results in a Δt of approx. 20 °C to 25 °C for a rich H_2S feed. A very simple and straight-forward pocket computer calculation can show this. This coincides with our measurements in many Claus plants.

However, in quite a major number of plants, the temperature rise is a lot higher, though only temporarily. In Figs. 13 and 13A, the measured temperature rise was actually approx. 45 °C versus the normal up to 25 °C. This is due to side reactions caused by the higher H_2 content in the process gas. The side reactions cause a rejuvenation of the catalyst. As expected, this effect subsides after a while and does not reappear at later oxygen enrichment to the same level.

6. The cleaning effect of oxygen enrichment.

Evaluation of a test with O_2 enrichment

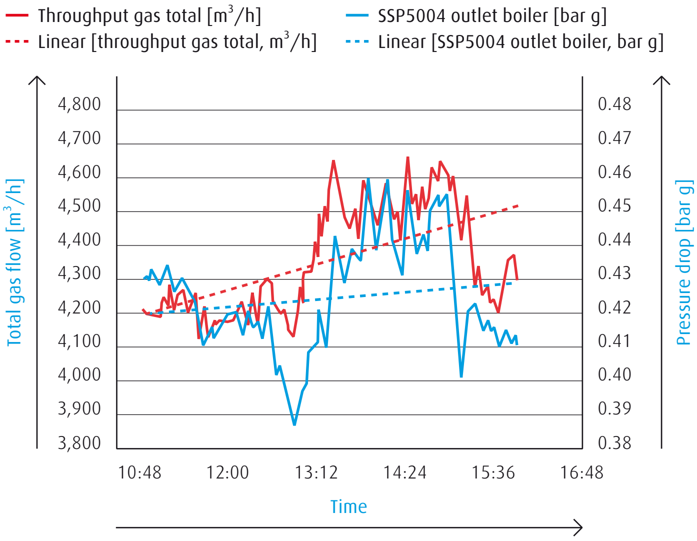

Fig. 14: Pressure drop versus gas flow

When applying oxygen enrichment, after only a few hours of operation, we saw in most plants a decrease in pressure drop through the plant at comparable gas loads. For example, when starting a plant with air operation at 400 mbar Δp, entering then into oxygen enrichment and returning to air operation at the end of the shift, the measured Δp was typically by 20 to 40 mbar lower at 100 % design load. The reduction of pressure drop at identical gas loads in the range 20 to 40 mbar is quite substantial at a total pressure drop of 400 to 500 mbar. Obviously, some of the deposits which formed in the plant operation over the years were removed by the oxygen-enriched operation (see Fig. 14).

One can see clearly that, at a gas flow of 4,200 Nm^3/h, the Δp in the beginning was 430 mbar, a few hours later at the same conditions only 415 mbar, i. e. 15 mbar less. This is a one-time effect, i. e. it cannot be repeated the next day. This, of course, would also be implausible – once the deposits are removed, they cannot be removed a second time. However, it reappears a few months later once new deposits are formed in the plant. Accordingly, in a plant where we measured just shortly after a turn-around, which included cleaning the plant, the reduction of pressure drop due to oxygen enrichment could not be observed.

Explaining the lowered Δp by a cleaning effect of oxygen enrichment is also confirmed by the observation that inner surfaces of the piping and heat exchangers look different after this operating mode.

7. Capacity increase due to oxygen enrichment.

Capacity increase for Δp = constant (conventional wisdom)

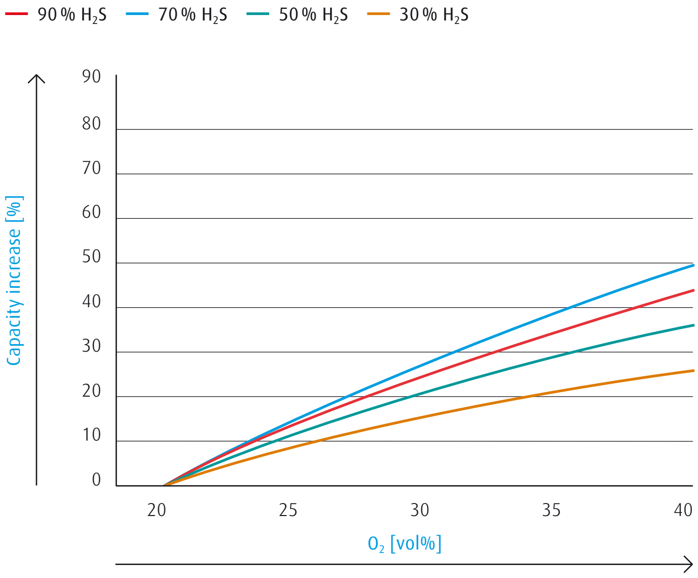

Fig. 15: Capacity increase versus O_2 level (conventional, compare Monnet et al., 2003)

Capacity increase for Δp = constant (measured)

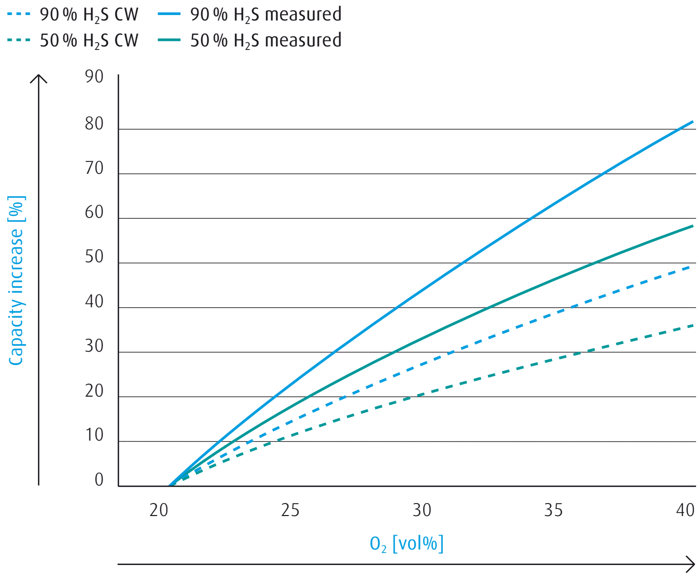

Fig. 16: Capacity increase versus oxygen level (our measurements)

The capacity increase in Claus plants due to oxygen enrichment is a well proven effect. In literature, the above diagram (Fig. 15) is usually published to show the capacity increase as a function of oxygen enrichment level. Our measurements, however, have shown a lot higher capacity increase time and again (Fig. 16).

As oxygen enrichment increases, the gas flow decreases at constant sulphur load. Accordingly, the now lower pressure drop can be filled with additional feed gas and thus the capacity of the Claus plant rises. For Fig. 15, it is assumed that the pressure drop is in proportion to the square of the gas flow. This assumption leads to an increase of the capacity, e.g. of approx. 23 % at 28 vol% O_2. However, under these conditions, our measurements showed invariably higher capacity increases of typically 33 %. The observed capacity increases can be better described by a curve as the uninterrupted lines in Fig. 16. These curves are obtained when assuming an increase of the pressure drop in proportion to the gas flow to the power of 1.35 to 1.5. There are several reasons to explain this behaviour:

1. A Claus plant is a mixture of various elements, such as burners, catalytic converters, piping, heat exchangers, etc. Only for straight pipes, the proportionality of the pressure drop with the square of the gas flow is valid. For other elements, other proportionalities prevail.
2. The Claus reaction is not equimolar. Rather, the sulphur formed is recovered as a liquid and removed from the gas flow. Therefore, the gas flow gets smaller and smaller, the further downstream the gas passes in the Claus plant. In the oxygen-enriched mode, this effect is even more pointed than in the air mode since the lower dilution by inert N_2 means higher concentration of the sulphur species and consequently higher influence of the recovered sulphur on the pressure drop.

In essence, this means that the capacity increase due to oxygen enrichment has been so far substantially undervalued. 28 vol% oxygen enrichment allows for a rich acid gas feed a capacity increase of typically 33 % rather than the 23 % cited in earlier literature (e.g. Monnet et al., 2003).

8. Damages due to ammonia.

Fig. 17: View into process gas pipe with ammonium salt deposits

Unlike the other gas components in Claus plants, ammonia is alkaline. Being prone to acid-base reactions, it has the potential to form salts with all the acidic components of the process gas stream, especially with those being built up in the Claus furnace, like SO_2 and SO_3. Corresponding ammonium salts then solidify in dependence of their volatilities.

Acids like H_2S or CO_2 do not form deposits at the temperature conditions given downstream a Claus furnace. On the other hand, especially ammonium sulphites tend to build up solids preferably at colder spots within the gas path. Sulphates are even less volatile but the process gas concentration of SO_3 being necessary for their build-up is very low due to the sub-stoichiometric oxidation conditions in the thermal step. Therefore, sulphites are much more abundant as deposits and disproportionation takes place over time, forming sulphuric acid underneath the salt layer, thus causing corrosion there. Corresponding damages can be quite substantial. In one Claus unit, after only a few years of operation, a 7-digit amount of euros had to be spent on repairs, especially in the piping and the heat exchangers. In another refinery, the Claus unit had to be stopped and cleaned every three to six months because of plugging by ammonium salts. What these deposits may look like is shown in Fig. 17, taken from a damaged piping in a Claus process gas line.

Of course, every cleaning costs several hundred thousand euros in direct costs, lost production and manpower. These damages are certainly a strong incentive to look for means to avoid them. One of the best and proven ways to do so is oxygen enrichment. But the O_2 does not come for free, of course. A problem many Claus operators face is that managers can easily calculate the cost of oxygen. But it is a lot more difficult to quantify any future damage caused by ammonia. Therefore, we try here a few example calculations.

Installation of oxygen enrichment typically costs 100,000 € to 300,000 €, depending on the size of the Claus plant and the standards applied in the refinery. These costs include the liquid oxygen tank, the vaporiser, the FLOWTRAIN® (metering and safety system), the oxygen injector and the connecting piping.

In addition, there are the costs due to O_2 consumption. Claus plants in refineries usually consume less than 300 Nm^3/h, which may be sufficient for 25 % capacity increase plus ammonia destruction in a 50 t/d Claus plant. At a typical cost of 0.10 €/m^3 per Nm^3/h of O_2, this results in 30 €/h or approx. 250,000 €/a. However, in practice, consumptions fluctuate widely during the year and therefore consumptions are accordingly lower. Experience in a number of plants shows that efficient ammonia destruction can reduce corrosion to almost nil and downtime due to plugging substantially. This leads to the conclusion that there is something like a break-even point if oxygen enrichment avoids one cleaning of a Claus plant per year. Avoiding one downtime per year due to corrosion adds the same amount in favour of oxygen. Based on these assumptions and experience from the operation and outages of the Claus plant considered, one can estimate the economic value of oxygen enrichment in this specific plant.

Scenarios for bigger or smaller plants can easily be calculated from these examples.

9. Conclusions.

Fig. 18: Ammonium salt deposits associated with corrosion

A closer look at oxygen enrichment and ammonia decomposition shows that some of the prevailing plausible opinions need to be revised. Yes, complete ammonia destruction requires at least 1,300 °C. But at 1,100 °C, destruction is also rather efficient. To achieve efficient ammonia destruction, a good burner is essential, and it is even more important than high temperature, since the efficiency of NH_3 destruction varies in a very wide range for different burners. Oxygen enrichment allows to destroy ammonia very efficiently. In addition, it has a number of positive side effects. It cleans the plants by rejuvenating catalysts and reduces the pressure drop by removing salt deposits. This effect reduces corrosion which occurs under salt layers and reduces or even completely avoids downtime due to plugging. Finally, the capacity increase is higher than cited in older literature, i. e. more than 30 % for H_2S-rich feeds at an oxygen enrichment level of 28 vol%.

In conclusion, oxygen enrichment allows to meet the additional requirements caused by "clean fuels" production. Doing it the right way can greatly improve the positive effects.

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- Paskall, H. G. and J. A. Sames, Sulphur Recovery, Calgary, eighth edition, 2003, 2-35.

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