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Safe handling of ammonia synthesis catalysts

Johnson Matthey

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Ammonia synthesis catalysts have long lives and catalyst replacement is an infrequent activity. Many people will go through their careers in the ammonia industry without ever having to replace a synthesis catalyst and the infrequent nature of catalyst replacement means that many plants may not have direct experience of this activity. Ammonia synthesis catalyst can present a range of hazards throughout the replacement process, from transport through loading, reduction, shut down and discharge, but the good practice illustrated in this article, and collaboration between catalyst suppliers and end users can ensure safe and successful catalyst changeouts.

1. Ammonia synthesis catalysts

With the exception of a few plants which use a ruthenium based catalyst, all ammonia synthesis catalysts in commercial operation use promoted iron oxide, which is then reduced to promoted iron prior to use – broadly similar to that developed by Mittasch in the early part of the 20th Century. Whilst it may seem that there has been little subsequent development, this is deceptive in that there is now a much greater understanding about the role of the various promoters and how to optimize their incorporation during catalyst manufacture.

Promoters can be categorized as structural or electronic. Some are added deliberately during production whilst others are found in the iron ore used to produce the catalyst. Electronic promoters increase the activity of the catalyst by changing the electronic nature of the catalyst surface. K_2O is the most important electronic promoter. Structural promoters act to maintain the active structure of the catalyst, in particular its surface area under normal operating conditions. Alumina and calcium oxide are the most important structural promoters.

There is considerable interaction between the various promoters, and this can have a dramatic effect on initial reducibility, initial activity, and long-term stability. A thorough understanding of these interactions is vital in order to optimize the performance of the catalyst and to understand the safe handling of the material. In short, the optimum level of, and ratio between, promoters is more important than the quantity of promoter and may vary depending on the duty. This is illustrated by the fact that the optimum mix of promoters has changed over the years as synthesis gas has become cleaner.

1.1 KATALCO™ series catalysts

The most notable improvement made to iron based ammonia synthesis catalysis was the development of

KATALCO™ 74-1 in 1984. Although over 35 years old, this catalyst remains the highest activity iron based catalyst available in the market it was initially developed for use at low pressure in ICI's AMV and LCA plants, more recently its high activity has been integral to the success of the Uhde Dual Pressure Process. The main difference between **KATALCO 74-1** and standard iron-based catalyst is the incorporation of cobalt a unique manufacturing process ensures optimum performance, in addition to high activity, ease of reduction is also a significant benefit.

Although **KATALCO 74-1** is supplied in oxide form, metallic iron is the active form for ammonia synthesis and the iron oxide needs to be reduced prior to use by reacting with hydrogen to produce metallic iron:



As the reaction is endothermic at low temperature, a source of heat is required to initiate the reduction.

A well-executed reduction takes place with a sharp and well-defined reaction front passing through the converter. Upstream of the reaction front the catalyst is reduced, and hence active. Downstream of the reaction front is unreduced catalyst with little ammonia synthesis activity. In most cases the source of the hydrogen is methanated synthesis gas. The presence of nitrogen in near stoichiometric quantities is beneficial because ammonia synthesis takes place across the reduced catalyst at the same time as reduction continues across the unreduced catalyst. The exotherm created by the formation of ammonia provides heat, which helps drive the reduction reaction.

Water acts as a poison for ammonia synthesis catalyst. Whilst its effects are often reversible, this is not the case during the reduction process and the water that is generated must be kept to a low level to prevent it from poisoning catalyst which has already been reduced. This is achieved by limiting the concentration of water to a level specified by the catalyst vendor (typically 1000 to 3000 ppm) and by performing the reduction at a low pressure to minimise the partial pressure of water.

Plant reduction can be a lengthy and costly process and in many cases it is the plant (start up heater design, circulator design, converter design, operational problems) rather than the catalyst that is the rate determining step.

If the catalyst is indeed the limiting step, an alternative is to use a catalyst with good reduction properties such as **KATALCO 74-1**.

Another very good alternative is the use of pre-reduced and passivated catalysts **KATALCO 35-8** or **KATALCO 74-1R**. These are reduced and then passivated by carefully regulated exposure to controlled amounts of oxygen to give a stable iron oxide skin around the active iron phase. Reduction of this skin is much easier (and faster) than reduction of bulk catalyst allowing a substantial reduction in the time required for activation.

The disadvantages with pre-reduced catalyst are a higher cost and the extra care that is required when handling the material. A common compromise is to use a mixture, with a small quantity of pre-reduced catalyst on top of a larger volume of standard catalyst.

2 Catalyst handling

2.1 Catalyst transport

The primary focus when transporting hazardous chemicals is the hazardous nature of the product. However, the suitability of the labelling and packaging also requires consideration.

In particular the classifications for pre-reduced ammonia synthesis catalyst present challenges

Catalysts are by design very active materials, for some products especially where they are pre-treated this brings to bear additional transport restrictions. Figure 1 provides an example of the transport classification for pre-reduced **KATALCO 35-8A** ammonia synthesis catalyst on a safety data sheet.





SECTION 14: Transport information				
	ADR/RID	ADN	IMDG	IATA
14.1 UN number	UN2881	UN2881	UN2881	UN2881
14.2 UN proper shipping name	Metal catalyst, dry (Iron)	Metal catalyst, dry (Iron)	Metal catalyst, dry (Iron)	Metal catalyst, dry (Iron)
14.3 Transport hazard class (es)	4.2 	4.2 	4.2 	4.2 
14.4 Packing group	II	II	II	II
14.5 Environmental hazards	No.	No.	No.	No.

Figure 1: **KATALCO 35-8A** ammonia synthesis catalyst transport classifications

Normally for planned catalyst deliveries sea freight is used and there are restrictions on what can be safely and legally transported using air freight, e.g. as a pre-reduced catalyst falls under Transport hazard class 4.2 'Spontaneously combustible substance', since the catalyst can self-heat (without exposure to a heat source) when exposed to oxygen. Practically this means it is not usually possible to ship pre-reduced ammonia synthesis catalyst by air.

This relevant transportation information is provided on the MSDS/SDS, an extract from which can be seen in Figure 2. Additionally, there is a requirement to label each package with a HAZ label.



KATALCO 35-8A SAFETY DATA SHEET Conforms to Regulation (EC) No. 1907/2006 (REACH), Annex II (including amendments) - United Kingdom (UK)	
SECTION 2: Hazards identification	
2.1 Classification of the substance or mixture	
Product definition	: Mixture
Classification according to Regulation (EC) No. 1272/2008 [CLP/GHS]	Self-heat. 1, H251 Eye Dam. 1, H318 The product is classified as hazardous according to Regulation (EC) 1272/2008 as amended. See Section 16 for the full text of the H statements declared above. See Section 11 for more detailed information on health effects and symptoms.
2.2 Label elements	
Hazard pictograms	:  
Signal word	: Danger
Hazard statements	: Self-heating: may catch fire. Causes serious eye damage.
Precautionary statements	: Wear eye or face protection. Keep cool. Wear protective gloves and eye/face protection.
Response	: IF IN EYES: Rinse cautiously with water for several minutes. Immediately call a POISON CENTER or physician.

Figure 2: Extract of Safety Data Sheet for **KATALCO 35-8A** pre-reduced ammonia synthesis catalyst

2.2 Catalyst storage

KATALCO series ammonia synthesis catalyst is normally supplied in mild steel drums. These drums must not be stacked on their sides or stacked more than four drums high, even when held on pallets. Stacking of drums introduces both the risk of top drums falling from the stack, and the lower drums being crushed under the weight of the stack.

If the metal drums are to be stored outside, they should be protected against rain and standing water, but this should only be for a maximum of a few months. Longer term storage should be under cover and away from damp. To prevent contamination the lids should be left on the drums until just before charging.

Catalyst drums must not be rolled. Careless handling of the drums risks damage to the catalyst. Catalyst drums are often supplied on pallets, which reduces the likelihood of damage in transit, but requires suitable fork-lift trucks and a paved area to handle the pallets. The fork-lift truck to be used for unloading the pallets should be fitted with rim or body clamps to avoid damage to the drums. It is important not to use standard forks to lift the drums under the rolling hoops, as damage to the drums and catalyst is almost inevitable.



Figure 3: Forklift loading of drums of **KATALCO** series catalyst onto a road truck

2.3 Pre-overhaul checks

Since replacement of the ammonia synthesis catalyst is an expensive and time-consuming activity with long periods between scheduled replacements, and maintenance in and around the converter is difficult once the catalyst is in its reduced state, it is worth taking care to ensure that the ammonia converter is returned to service in good condition after a catalyst change. If the cartridge is not being replaced during an overhaul, it is worthwhile analysing the performance of the converter to diagnose any potential issues which can be addressed during the overhaul.

Johnson Matthey can assist in performing such an evaluation using **KATALCO PERFORMANCE** software and CFD modelling capabilities. If this proves inconclusive a **TRACERCO™** diagnostic check could also be carried out. This might involve injecting a radioisotope to check whether there is internal leakage through an ammonia converter. These activities should be carried out sufficiently in advance of the overhaul to implement contingency plans should any defects be highlighted.

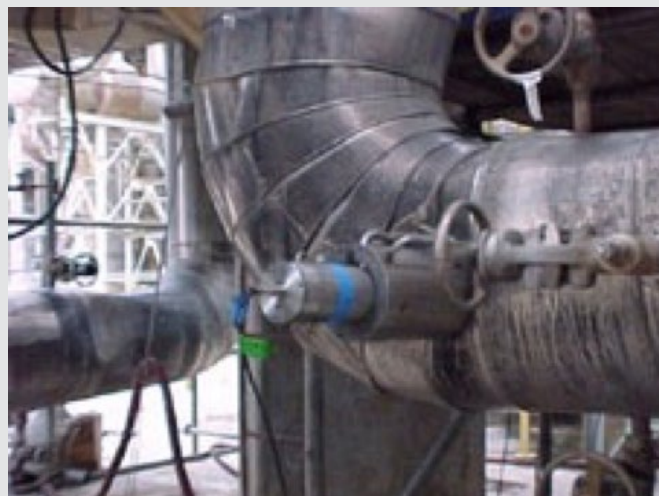


Figure 4: Testing for ammonia converter internal leaks

2.4 Catalyst installation

Once in the overhaul, and in addition to recommended mechanical inspections, typical pre-charging checks include:

- Catalyst support grid integrity
- Cleanliness of internal heat exchangers
- Checking internal heat exchangers for leakage and possible bypassing (**TRACERCO** process diagnostic techniques can again assist with this)
- Quench and bypass valve operation
- Thermocouple positioning and operation (warm each in turn, check corresponding indication in the control room)
- Ensure the vessel is dry before any catalyst is charged
- Clean any residual dust and debris from the catalyst beds prior to charging
- Ensure the catalyst is in good condition.

An even gas flow through the converter is key to ensuring optimum converter performance, therefore catalyst loading needs to be carried out in a manner that ensures uniform bulk density and hence uniform pressure drop characteristics. This is especially important with radial flow beds to avoid the presence of voids in the bed and the resulting bypassing of gas around sections of the catalyst. The density of ammonia synthesis catalyst is high and any equipment set up to handle the catalyst must be capable of withstanding its weight.

As with any process activity, a rigorous and effective risk assessment should be carried out prior to catalyst charging. This should take on board advice from the

catalyst supplier, the cartridge supplier and the plant designer. Protective clothing should be specified with due regard to the Material Safety Data Sheet (MSDS), local legislative requirements and operating company standards. As ever, special attention should be paid to the safety of confined space entries and to the asphyxiation hazards associated with nitrogen.

There is potential for the exposure to dust during catalyst loading. Catalysts should be handled in well ventilated areas and in manners that limit the formation of dust. Appropriate protective clothing, gloves and goggles should be worn, and respiratory protection should be used. All personnel involved in catalyst handling should wash afterwards, and clothing should be changed at the end of each shift as a minimum to prevent contamination.

2.4.1 Pre-reduced catalyst

Whilst pre-reduced catalyst can be charged safely, and there are many references of **KATALCO 35-8** and **KATALCO 74-1R** being safely loaded into converters, there are a few features which should be considered when generating risk assessments and job methods. These are highlighted below.

If the oxide film around the reduced material is damaged, reduced catalyst is exposed to air. The reaction with air is highly exothermic and hence the catalyst particle heats up. As the temperature increases the oxide film becomes a much easier barrier for oxygen to penetrate and hence a self-heating phenomenon occurs. For any individual particle this is not a problem because the particle has a large surface area and can transmit the heat of reaction away easily. However, in large packed beds, such as an ammonia converter, the heat of reaction is conducted to adjacent particles which then heat up allowing oxygen to penetrate to the core of these particles and further increasing the temperature.

If there is no air flow through the packed bed the oxidation reaction is limited by the availability of oxygen. As the concentration of oxygen falls the reaction can no longer be self-sustaining and ceases. If oxygen is readily available a temperature runaway is possible. Once the bed starts to heat up a chimney effect will occur. As the density of the hot air in the bed falls the air will rise out of the bed, drawing in fresh air which allows the oxidation reaction to continue.

The protective oxide layer can be damaged by attrition and by moisture. The use of good quality catalyst, which does not require sieving, is recommended. Excessive vibration should also be discouraged. Excessive moisture can also affect the protective layer and dehumidified air has also been used with success.

Additional precautions include keeping the catalyst cool (< 50 °C, < 120 °F) during handling. If exposed to hot tropical sun the catalyst can act as a black body and absorbed the heat from the sun until it became self-heating. It is recommended that catalyst charging be carried out under shade under such conditions.

As well as heating, oxygen deficiency is another potential hazard to loading personnel within the converter. If the catalyst begins to react with air it will consume oxygen, and hence there is the potential to generate an oxygen deficient atmosphere during catalyst charging. It is recommended that the vessel is charged from outside if possible. If it is necessary to enter the vessel, breathing apparatus (BA) is recommended.

Standby personnel should also be wearing breathing apparatus. BA should be used even if no N₂ purge is activated since the pre-reduced catalyst will slowly consume oxygen within the confined space and the catalyst may evolve some residual ammonia. No ventilation should be provided, to avoid excess oxygen ingress promoting the exothermic oxidation reaction.

During catalyst charging, the vessel thermocouples must be fitted and operational to allow the temperature of the catalyst to be monitored. This will enable any catalyst heating to be observed. The temperature should be monitored throughout the charging process until the converter is boxed up and under nitrogen.

If caught in time, the effects of catalyst heating can be accommodated by applying a nitrogen blanket. It is therefore necessary to have a readily available source of nitrogen and to keep a careful watch on the temperatures during the charging process. If it is necessary to have people in the vessel during the charging, they must be fully aware of the possible hazards and a rescue plan should be established to enable them to leave the converter quickly and safely should temperature rise occur. Once it is safe to do so, a nitrogen blanket can then be established.

2.5 Reduction

Whilst the reduction procedure is quite simple, the effect of the synthesis loop means that a simple change in conditions takes much longer to settle down than would be the case with a once through process.

Water evolved during reduction should never pass over catalyst that has been already reduced, and this is achieved by keeping the unreduced beds at a lower temperature. The water concentration leaving the reactor should be monitored and controlled in accordance with the recommendations of the catalyst supplier.

The water is removed via the loop catchpot. This may be difficult at low loop pressure and it can freeze in the chillers if the refrigeration system is running. As ammonia solution freezes at a much lower temperature than water, ammonia is sometimes added to the loop before reduction commences. This means that the refrigeration system can be brought online earlier thereby minimizing the water concentration in the circulating gas.

Gradually, the reduced catalyst at the inlet of the reactor will start to make more and more ammonia. At this stage, catalyst temperatures need to be controlled carefully (often by increasing the circulation rate), otherwise the reduction will become too rapid and may result in the catalyst temperature falling. Rapid temperature changes are always bad for a catalyst bed but, due to differential expansion, the catalyst is particularly vulnerable if it is partially reduced during such a change.

As the catalyst becomes reduced and produces ammonia, the reduction water contains an increasing level of ammonia. The ammonia liquor condensed from the reduction loop requires safe handling, storage and disposal.

As the reduction proceeds, the heat of the ammonia synthesis reaction gradually becomes the dominant heat input as more and more catalyst is reduced. This allows the circulation rate to be increased tremendously, and so each bed takes roughly the same length of time to reduce even though the downstream beds contain much more catalyst.

The circulation rate is normally increased by opening the converter inlet valve a little. This will put more cool gas to the first bed and cool/hold the bed temperature. It can also affect the flow through shot/bypass valves and therefore the flow through the various beds and interchangers. Adjustments to the inlet valve should be very small until the "feel" of the converter has been established. If a plant trip occurs a forward flow through the converter must be maintained by purging downstream of the converter to prevent reduced catalyst standing in a stagnant atmosphere containing water as this will reduce the final activity of the catalyst. If it is necessary to de-pressure the loop the catalyst should be held under a dry nitrogen atmosphere.

2.6 Shut down

When an ammonia converter is taken offline it is important to exclude air, water and other possible catalyst poisons. The simplest way of doing this is to leave the loop at pressure, the only complication being that liquid ammonia may then condense in sections of the pipework

which are normally above the condensation temperature. This may not be important, but if the pressure is subsequently reduced and the ammonia boils it can produce very low temperatures on pipework which may not have been designed for it. A blow through with warm nitrogen or synthesis gas can avoid this problem.

In the right circumstances it is possible to reach temperatures much lower than the boiling point of ammonia (-33 °C, -27 °F, at atmospheric pressure) and, in the limit, it is possible to reach the adiabatic saturation temperature (-72 °C, -98 °F).

If the pressure in the loop is to be reduced to atmospheric for maintenance, a positive nitrogen purge must be kept through the catalyst. It is wise to minimize the flow of nitrogen even if the nitrogen contains only a few ppm of oxygen in order to avoid re-oxidizing the catalyst.

When the loop pressure is reduced there is a possibility that water or steam may enter the synthesis loop due to any leaking heat exchangers. Special care is required under such circumstances, and if there is any doubt it is as well to make sure that the water side of these heat exchangers are also de-pressured.

Re-oxidation will lead to a temperature rise and it is good practice to check bed temperatures every hour or so. Bed temperature should be checked more often when intrusive work, which can lead to oxygen ingress, is in progress. Another risk that can arise during a shutdown when the nitrogen system is purging several vessels at the same time. Under these conditions it is sometimes possible for the contents of one vessel to enter another via the nitrogen pipework, and thought must be given to this possibility when planning the shutdown.

2.7 Unloading

Ammonia synthesis catalyst must be handled with great care when in the reduced state. In extreme circumstances, oxidation of ammonia synthesis catalyst can generate an exotherm of up to 1600 °C. Air must not be allowed to flow through reduced catalyst (e.g. by transporting in open containers) as this can create a chimney effect and draw more air into the catalyst.

Stabilisation is best carried out by slow oxidation. This can be achieved by keeping the catalyst wet and exposing it to air for a long period (several days) outside of the vessel. The catalyst should be kept in a thin layer between 150 and 300 mm (6 and 12 inches) deep to allow it to cool easily. Self-heating can occur if the catalyst is dumped in heaps. Hydrogen is evolved by the reaction with water and the area should be well ventilated. Sources of ignition in

and around this area should be identified and eliminated to prevent the evolved hydrogen from igniting.

Simple vessels are usually discharged by sucking out the material which is blanketed with nitrogen to prevent excessive oxidation. Some air is usually drawn into the top of the vessel and possibly through the top part of the catalyst, but if there is sufficient nitrogen flow upwards through the remaining catalyst the quantity of hot material is kept to a minimum and trouble is unlikely. Nevertheless, temperatures must be watched carefully and if they become too high, the sucking must be stopped and the nitrogen blanket re-established.

Ammonia synthesis catalyst can be stabilised before discharge by circulating nitrogen with a suitable compressor and adding air carefully to the circulating system. Complete re-oxidation is not necessary, and it is best to keep the catalyst temperatures between about 70 and 100 °C. At lower temperatures reaction is very slow, and at higher temperatures the re-oxidation will proceed further than required. Even when stabilisation has been carried out it is advisable to keep a nitrogen supply available and to monitor bed temperatures carefully, as there is a possibility that some pockets of the catalyst will not have been stabilised and if a hot spot develops it can spread quickly.

Another method of stabilisation is to fill the ammonia catalyst basket with demineralised water. Demineralised water is used because converter internals are usually stainless. As mentioned above, when reduced ammonia synthesis catalyst is immersed in water it evolves hydrogen slowly, and care must be taken to avoid the hydrogen accumulation giving an explosion hazard. Not all ammonia synthesis cartridges are suitable for filling with water. They may not be strong enough or they may contain insulating material, which should not be wetted. This feature should be checked with the manufacturer before the procedure is agreed.

It is not recommended to use steam in place of water to stabilise the catalyst as the hydrogen production is more rapid and effective stabilisation is not achieved.

Another point to note is that any unreduced iron left within the converter can potentially react with air to produce an oxygen deficient atmosphere and this possibility should be addressed as part of the confined space entry procedures and precautions.

3. Case studies

3.1 KATALCO 74-1R loaded into Casale revamped reactor

Two converters on a Kellogg ammonia plant, were revamped with Casale internals. **KATALCO 74-1R** pre-reduced ammonia synthesis catalyst was selected for use in all three of the beds in both converters.

JM not only supplied the catalyst and the technical support services during catalyst reduction, but also arranged for inspection of the baskets during their fabrication and acted as a validator for the project.

To unload the previously installed catalyst, the converter was filled from the bottom with water to oxidise the catalyst. After the catalyst had been flooded for 36 hours, the basket was removed, and the oxidation of the catalyst began. The catalyst remained stable, but after two hours it caught fire due to high wind. After containing the fire, the catalyst was emptied into bins where it again began to glow after coming into contact with air.

Although the previous charge of catalyst had been flooded and no longer appeared active, there was enough residual activity to present a fire risk on exposure to air.

In preparation for loading initially the charge of **KATALCO 74-1R**, was sieved, but as there was no dust collected, the **KATALCO 74-1R** was considered suitable no need to sieve, as recommended by JM.

The loading was started under dehumidified air. Due to hot work on the baskets, and the flammable nature of the rope ladder used in the converter, there was a fire during the loading. The air flow was stopped and nitrogen flow was started from the bottom of the converter. Following this, the rope ladder was replaced with an aluminium ladder. Measurement of the catalyst temperature showed that the catalyst had reached 160 °C, and therefore work was stopped and the catalyst cooled under a nitrogen purge.

Contributory factors to the incident

- Damage to the passivation layer of the pre-reduced catalyst can result in oxidation exotherms, the unnecessary sieving of **KATALCO 74-1R**
- lack of isolation leading to a chimney effect in the converter.
- Flammable materials should not be present inside the converter during loading.

Following the rapid reduction of the **KATALCO** 4-1R charge, the converter effluent was vented to ensure that there was no residual dust that might damage the compressor. No dust was observed in the venting gas.

The customer was extremely happy with Casale and Johnson Matthey for the workmanship, product quality and technical support services during the operation.

3.2 Remote support for loading and reduction of **KATALCO** 35-4 into Haldor Topsøe converter

A new S-50 ammonia converter was installed during a plant turnaround in July 2020. The catalyst for the new converter was **KATALCO** 35-4 supplied by Johnson Matthey.

Since it was not possible to travel to customer sites due to COVID-19 travel restrictions in July 2020, JM were able to provide remote support to the customer in the loading and delivery of catalyst.

The quantity of catalyst supplied by JM was calculated based on the information provided during the tender. However, since there was information lacking regarding the dimensions and the exact volume of the catalyst beds, the supplied volume was approximately 20 m³ less than the volume available to fill.

Since the new converter was a radial bed, JM emphasised the point that it is very important that the catalyst bed is completely filled up to the bed covers and that it must be filled in such a way that loaded catalyst density for the entire bed is consistent and meets the target density to avoid voids in the catalyst bed and insufficient contact time for the required ammonia make.

JM recommended that the catalyst bed must be fully loaded with catalyst up to the cover plates and to the target density. Since additional catalyst needed to be procured to fill the bed as soon as possible, JM was able to offer available UK stock of **KATALCO** 35-4A for either sea freight or airfreight for delivery.

As part of the remote support for the loading, JM provided catalyst operating manual, loading manual and log-sheets to the customer and offered advice on loading methods and vibration methods to ensure the correct loaded density of the catalyst.

JM also provided remote support to the reduction of the **KATALCO** 35-4 catalyst. Guidelines for the reduction of the catalyst were provided and a dialogue maintained between JM and site personnel. JM offered recommendations on reduction temperatures and

reviewed reduction log sheets to assess the progress of the reduction.

Following completion of the reduction, the S-50 converter containing **KATALCO** 35-4 has operated successfully.

3.3 Loading **KATALCO** 35-8 and **KATALCO** 35-4 catalyst into KBR horizontal converter

The loading of the ammonia synthesis converter was to be carried out by a EPC Engineering crew under the guidance of both EPC and KBR engineers. Johnson Matthey was requested to provide additional support and was present for catalyst loading activities and training to ensure JM's loading standards were achieved. The loading was monitored by JM, KBR and EPC and inspections were carried out after each distinct layer was loaded.

Before the loading commenced, all pre-loading checks and requirements were completed. The vessels were inspected, the internal dimensions were checked against the mechanical drawings and the bed dimensions used for the loading calculations were confirmed to be accurate. The bed mechanical integrity was confirmed and the clearance to load was issued following the initial level markings.

The **KATALCO** series catalysts were inspected prior to loading and found to be in exceptional condition with no particles outside of the size range and very little dust. The drums were periodically inspected by JM and KBR to ensure the quality of the catalyst was maintained and all drums were found to be in very good condition.

The **KATALCO** 35-4 series catalyst was transported to site and kept in a designated shelter area close to the loading platform. From the shelter area the drums were lifted to the loading platform. All drums were always kept shaded in between layer loadings.

The **KATALCO** 35-8A and **KATALCO** 35-8C catalysts were supplied as pre-reduced and stabilised metallic iron. A meeting was held between JM, KBR, EPC and the site safety team regarding the loading of the pre-reduced catalyst **KATALCO** 35-8 in the Ammonia Synthesis Converter. Although Johnson Matthey pre-reduced catalysts are specially designed to be stable when loaded in air, KBR specify in their loading documents that that loading of pre-reduced catalyst must be done under nitrogen purge. JM supported KBR's decision to load the catalyst under a nitrogen blanket.

With expertise in the loading of catalyst in a nitrogen or oxygen deficient atmosphere, JM provided training and advice and recommended the use of breathing apparatus

(BA) to eliminate the risk of a compromised atmosphere in a confined space. Detailed loading procedures and safety recommendations were communicated clearly to all participants.

Prior to commencing the loading, the nitrogen source for maintaining the inert atmosphere was identified and checked to be secure and reliable. All other nitrogen consuming activities were considered to ensure that adequate supply to the vessel blanket could be maintained.

Access and egress routes from the converter were assessed and established and the area around the converter was designated an exclusion zone with hazard barricading and signage placed around 1 m from the top cover and the base of the scaffolding. Atmosphere checks were carried out on a routine basis and multiple bed temperature measurements were taken and constantly monitored.

This extra precaution allowed the Bed 1 catalyst to be loaded in less than 12 working hours, without any temperature build up or safety issues.

The loaded bulk density of the catalyst was checked after the loading and vibration of each distinct layer by the JM/KBR/EPC supervisor in order to ensure that the required target density had been achieved. All beds successfully achieved the target densities to within 1% of the target and the loading was confirmed to be very successful.

A close working relationship between JM, KBR and EPC and the site management team ensured a well-managed, safe and successful loading of the new build plant.

4. Conclusions

Ammonia synthesis catalysts can present a number of hazards which can be well mitigated with good handling practice. Appropriate packaging and labelling of materials for transport and well managed storage, installation, reduction, shut down and discharge of catalyst can ensure the safe handling and optimal operation of ammonia synthesis catalysts.

As demonstrated by successful case studies of catalyst discharges, loadings and reductions, operators are encouraged to make use of the knowledge and experience of catalyst suppliers when planning to replace their synthesis catalyst.

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