

# Plant Reliability Improvement Through the Replacement of Ammonia Converter Baskets

*The Dyno Nobel ammonia plant at St Helens, Oregon, USA, has been in operation for more than 40 years. This plant is a 1960's vintage plant that operates at very high synloop pressures of up to 5000 psig. As the old axial flow path baskets were approaching end of life, Dyno Nobel decided to replace those baskets with a new design of Casale axial-radial baskets in all three vessels that were in series.*

*Some issues related to mechanical integrity of the vessels and nozzles were noticed during replacement of the baskets and they are explained in this paper with a focus to help those plants in the industry that operate similar vintage plants.*

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## Introduction

**D**yno Nobel operates an ammonia plant at St. Helens, Oregon, USA, designed by J.F. Pritchard that originally produced 250 STPD (227 MTPD). Some upgrades have been made since the original commissioning in the early 1960's, which have increased the operating rate to more than 300 STPD (272 MTPD).

The site also has a 400 STPD (363 MTPD) urea plant designed by Chemico (150 STPD, 136

MTPD) original nameplate, a Weatherly designed 50 STPD (45 MTPD) nitric acid and 70 STPD (63.5 MTPD) ammonium nitrate plant.

## Synthesis Loop Configuration

The St. Helens ammonia plant synthesis loop had three converters in series with original axial flow path baskets. The syngas from two trains of reciprocating compressors driven by electric motors is sent to the converter bottles after heat exchange in the converter feed/effluent exchanger, E621. The recycle stream is compressed in a separate motor driven centrifugal Hypercirc

compressor. Figure 1 (at the end) shows the schematic flow diagram of the synthesis loop.

Unique features of this plant are a low pressure front-end, operating at 450 psig (32 barg), a high pressure synthesis loop operating at 5000 psig (345 barg), a medium pressure saturated steam system operating at 475 psig (33 barg), and a closed loop refrigeration system.

The synthesis loop reaction takes place in a three reactor configuration with a manually controlled quench gas stream of cool syngas. The quench gas provides cooling in the annulus between the baskets and the pressure vessels, as well as between the sleeves of the connecting nozzles and the internal diameter of the nozzles to reduce the likelihood of external skin hot spots.

The original baskets were designed with an axial gas flow path. The unreacted syngas runs up through a center pipe, out the top, and back down through the catalyst from the top to bottom. There is an electrical start up heater in the first converter.

## Converter Vessel Repairs

The 40 year old Pritchard baskets and pressure vessels had not been internally inspected since a catalyst change in 2001. During the previous 1999 and 2001 inspections, nitrided and cracked thermowell guides on all three converter bottom nozzles were found (Figure 2). The thermowell guides were constructed of 304 stainless, while the thermowells were 316 stainless. New thermowells and thermowell guides were installed in each, which involved heat treating the basket-to-guide welds.



Figure 2. Cracked Thermowell Guide (2001)

In a previous outage in 1987, significant work was done to repair all three catalyst baskets. Severe nitriding of the 304 stainless internal pipes and walls of the basket was seen, which made welding difficult, and impossible in some cases. New Schedule 40 internal pipes were installed to replace the brittle, broken Schedule 5 original pipes. Inconel 600 catalyst support screens replaced the original 304 stainless support screens as well.

It was expected that similar repairs would be needed for the next catalyst change that was to take place in 2012, making welding and repairs nearly impossible. In all previous inspections the carbon steel pressure vessels had shown no indications of high temperature hydrogen attack (HTHA) but had experienced shallow cracking in the basket support lugs and centering pin to the pressure vessel weld. The repairs required grinding and a replacement of one centering pin. Numerous repairs had been made to the nozzles and sleeves that connected between the three converters because of the threaded sleeves and awkward disassembly and re-assembly required for catalyst changes and inspections. Company mechanical reliability specialists risk ranked the baskets with a high likelihood of failure if left in the existing condition for the 2012 campaign, recommending that the baskets be replaced. From previous experience, new nozzle sleeve

construction was also recommended by the plant to ensure a smooth fit up with the new baskets and to remove the need for sleeve repair or re-build during the shut down.

During the 2012 shut down, the Pritchard baskets were extracted and given a visual inspection. Cracking was seen in the outlet nozzle of the A converter basket (Figure 3). Bypassing of gas from the A and C converter had been suspected by looking at the temperature profiles during catalyst surveys. The condition of the 40 year old Pritchard baskets is shown in Figure 4.



Figure 3. Cracking seen in V-616A outlet nozzle



Figure 4. Pritchard catalyst basket V-616A

The pressure vessel top heads of the A and C converters had cracking indications on the ring joint gasket groove that needed to be machined, heat treated, and sent off-site for additional weld repair (Figure 5).



Figure 5. Cracks noted in ring joint gasket groove of the top head of V-616A

An inconsistency with original pressure vessel design drawings was found during the detailed engineering phase of the project. As built measurements of each pressure vessel did not exist. The bottom head dimensions were critical to the design of the new baskets since each basket would be anchored by the bottom centering pin and the basket supports. In preparation for an accurate fit, ultrasonic thickness analyses as well as an external laser scan were conducted to verify the thickness of the shell of the bottom heads and other critical dimensions of the vessels. The original drawings indicated the bottom head shells would be 5.75 inches thick. By repeated UT measurements, the actual thickness was found to be 4.9 to 5 inches at the bottom, increasing gradually to 5.75 inches as the shell moves upward toward the tangent line. This discovery was a critical finding that altered drawings and fabrication plans, ensuring a proper fit could be made in the field.

The heavy wall carbon steel piping exiting the converter gas/gas heat exchanger, E-621, was found to be insufficient for the high pressure hydrogen and temperature combination, based on its service life because of HTHA. Ammonia Casale had simulated a heat and material balance for the revamped conditions and it showed that even though the pressure would decrease, the temperature was expected to increase by at least 40 °F (4 °C). Failure of piping at pressures

greater than 3500 psig (240 bar) is very serious; therefore, it was decided to replace this piping with 1.25Cr - 0.5Mo steel material.

## Design of Internals

The design of the internals (axial-radial beds) was developed by Ammonia Casale based on their previous experience and on the latest innovations developed from prior revamp projects. Ammonia Casale also checked the suitability of the equipment of the synthesis loop after the re-vamping. Complete simulation of the synthesis loop equipment, together with the analysis of the Hypercic compressor (circulator) was performed. The following characteristics are specific to the Dyno Nobel converters which make them unique:

- The main inlet gas coming from the upstream converter is fed to the following reactor through the connecting pipe where it is mixed with quench gas coming from the nozzle N3, located in the top of the vessel. The cold quench gas, before entering the pipe, flows through the annulus between the vessel and the cartridge, keeping the vessel temperature and nozzle connections safely below the design.
- The flow path through the catalyst is inward, from the outer to the inner part of the bed with an axial-radial path (See Figures 6 or 7 below).

### First Bottle – V-616A

Figure 6 shows the flow path of the reacting gas through the converter, V-616A (first bottle, which incorporates the existing start-up heater). At the converter entrance (nozzle N1), the gas is diverted to the bottom part of the cartridge, and then flows upstream through the existing start-up heater. On top of the bed, the gas flowing downward through the outer collector, then enters the catalyst bed. Most of the gas enters radially with the rest axially from the upper part of the bed.

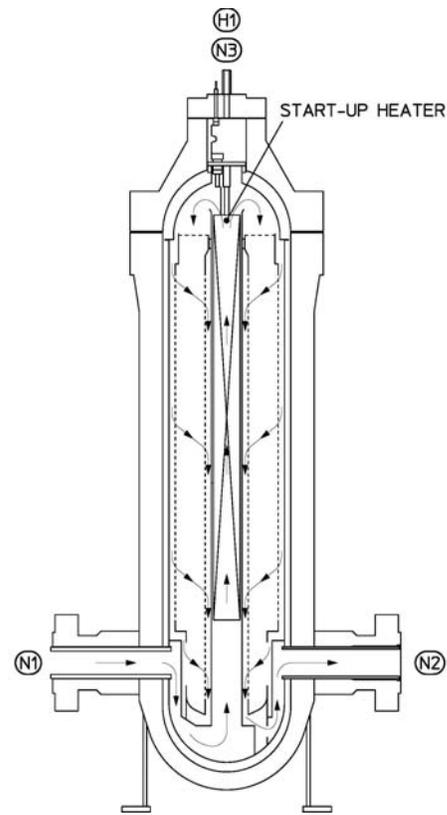


Figure 6. First bottle, V616A converter layout

After leaving the catalyst bed, the gas flows through the inner collector to the bottom part of the cartridge and exits the converter through the nozzle N2.

### Second and Third Bottle – V-616B & V-616C

Figure 7 shows the flow path of the reacting gas through converter bottles, V-616B and V-616C. At the converter entrance (nozzle N1), the gas is diverted to the outer collector, to the top part of the cartridge. The gas flowing upstream through the outer collector then enters the catalyst bed. Most of the gas enters radially with the rest axially from the upper part of the bed. After leaving the catalyst bed, the gas flows through the inner collector (central pipe) to the bottom part of the cartridge and exits the converter through the nozzle, N2.

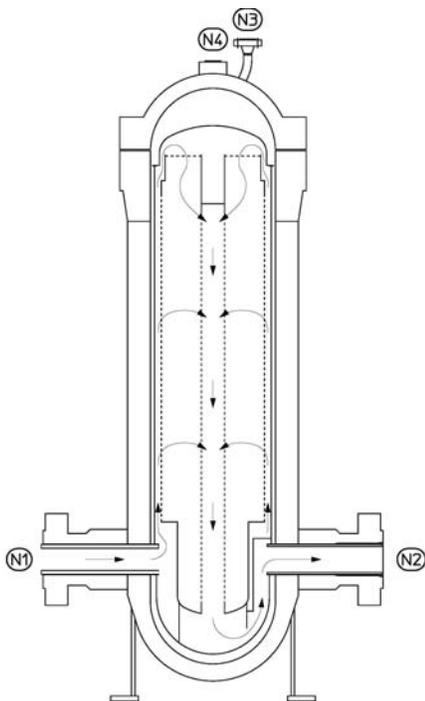


Figure 7. V-616B & C

### Unique features of internals design

The design specifically developed for the above-described converters has been studied to achieve the following goals:

- Full exploitation of catalyst, with optimization of the gas path through the converters.
- High reliability.
- Easy access to internal baskets for maintenance or catalyst replacement.
- Reduced pressure drop across the converters from 110 psi to 35 psi (7.6 - 2.4 bar).
- Reduced operating pressure by about 1000 psig (69 bar).
- Increased ammonia conversion per pass from 15.70 – 17.75 mol% NH<sub>3</sub>.

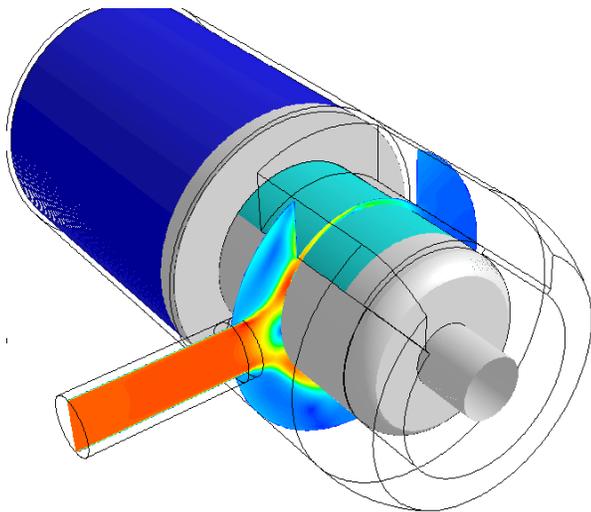
As described in the previous paragraph, the reacting gas flowing through the catalyst bed has an axial-radial path which utilizes all of the catalyst loaded into the baskets. Due to the particular design of the converter (inward flow, from top to the bottom – first bottle – and inward flow from bottom to the top – second and third bottle – so with the axial part in the opposite of inlet opening), a thorough analysis of gas motion was un-

dertaken with fluid dynamic simulation tools. The particular design developed for the gas distribution ensured balanced distribution between axial and radial flow, exploiting the full potential of the catalyst installed, including the top part of the second and third bottles.

The catalyst is contained and supported between two cylindrical walls, made by slotted plate. These plates, constructed of AISI 321 stainless steel, have the double function of containing the catalyst (the slot dimensions are designed for this purpose) and supporting the catalyst (the wall thickness is calculated based on the catalyst properties). The construction procedure used to make these slotted plates is such that it does not affect the metallurgical structure of the metal (no crack formation), resulting in high strength of the cylindrical walls.

In the top of the catalytic bed, a protection screen is installed to prevent migration of the catalyst outside the baskets. This design prevents direct axial impingement of the catalyst by the gas while allowing it to flow axially through the top part of the bed.

In the second and third bottles, the feed gas has to be routed to the top part of the cartridge in order to achieve the up and inward gas flow path. The bottom part of the bottles was properly designed and simulated by the Casale fluid dynamic department, as shown in Figure 8. The picture in Figure 8 illustrates the gas distribution (velocity) of the incoming gas.



*Figure 8. Simulation results in the bottom part of the second and third bottles: feed gas and outer collector*

## Catalyst Loading

Since the Clariant AmoMax-10 RS catalyst for the three axial radial baskets was in the pre-reduced form, loading was done under supplied breathing air due to the recommended nitrogen purge.

AmoMax-10 RS catalyst was loaded into the three new Ammonia Casale baskets on June 27 and 28, 2012, using a Casale developed dense loading method. The newly installed catalyst is a wüstite based, small particle (1.5-3 mm) converter catalyst, whereas the spent catalyst was a conventional magnetite based, large particle (6-10 mm) catalyst. This smaller particle size and higher iron/lower oxygen content of the AmoMax 10 RS was coupled with the axial-radial design to get the highest exposure of the gas to the catalyst, allowing for an increased conversion with lower pressure drop. In addition, there was less water make during reduction.

A special stationary hopper, transition hopper, and loading tool were constructed to get an even distribution of the catalyst and allowed for smooth loading of the pre-reduced catalyst (Figure 9). North American Industrial Services (NAIS) performed the catalyst loading activities

and fabricated the new hoppers and loading tools using Ammonia Casale drawings. The preparation, screening, coordination with the crane operator and setup took much longer than actual loading. The actual loading took between two and three hours because of the small volume of each vessel. The loaded bulk densities of AmoMax 10 RS averaged 2.5 kg/l (156 lb/ft<sup>3</sup>).



*Figure 9. Catalyst loading with hoses attached to special tool*

After loading was complete, the slotted catalyst basket cover (Figure 10) was bolted down and the cartridge cover (Figure 11) was also installed, which enabled keeping a nitrogen blanket until the head could be installed, as two out of three heads were out for repair. Thermocouples were inserted into their thermowells.



Figure 10. Catalyst basket cover shown

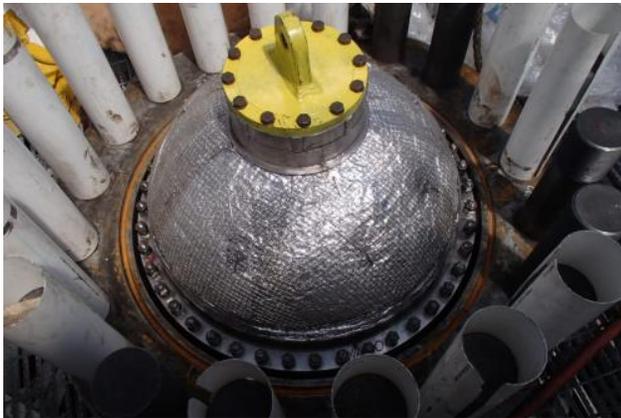


Figure 11. Cartridge cover

## Catalyst Reduction

On July 9, 2012, reduction of the ammonia synthesis catalyst was started. Details are shown on the following graphs.

With the pre-reduced wüstite catalyst, a low amount of water was generated during the reduction as shown in Figure 12. The maximum measured from sampling of the third converter exit was 1100 ppm. The sequence of catalyst reduction was the same as when reducing conventional magnetite catalyst.

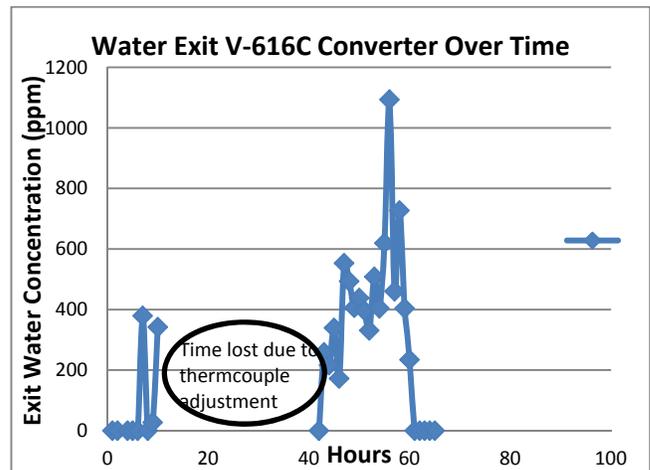


Figure 12. Catalyst reduction chart showing exit water vs. time



Figure 13. Catalyst reduction chart – V-616A, B, C reduction temperatures vs time

The total reduction time was approximately 2.5 days (Figure 13). The first day it was discovered that the outlet gas thermocouples in the bottom of converter A were not installed correctly because the temperatures were reading too low. The synthesis loop reduction was shut down and purged to cool the converter vessels down. Re-adjusting the thermocouple bundle required the removal of a flange from the bottom of the vessel, which because of the skirt and presence of nitrogen and ammonia, the task required a confined space entry with supplied air. The thermowell has a U shaped path, which requires the

thermocouple bundle to make a 90 degree turn to place it correctly in the outlet gas flow path.

Upon its removal, it was discovered that the thermocouple bundle was severely kinked (Figure 14). The bundle was straightened, bound with additional wire, and placed carefully back into the well. Even after these efforts, it was found that the reading was still too low. Ammonia Casale determined that the outlet gas thermocouples were slightly too short (~21 mm, ~3/16 inches) to reach the proper location. Another thermocouple bundle was ordered with the correct length wires and was received within the first 3 months of operation. Subsequent efforts to remove the thermocouple were unsuccessful because the existing bundle was stuck in its position. The plant decided to leave it as is until the next extended shut down.



Figure 14. Thermocouple bundle that was bent going in

Table 1 describes the performance of the ammonia loop before and after AmoMax-10 RS catalyst was installed. The production rate shown is related to the make-up gas availability during process data collection. The higher than expected synthesis loop pressure is most likely due to the unpredictability of the circulating compressor performance at the new conditions. The Hypercirc compressor performance curve was not accurate even for the base operating case as the plant was operating at 122% of name plate

design. When the operation of the Hypercirc compressor was checked during the study phase of the revamping project, the actual compressor operation did not match with the original performance curve. As a result, the final operating conditions (pressure and circulation) of the synthesis loop after the revamp was affected by the discrepancies with the original performance curve.

Survey Date	12/11/2009	4/18/2011	11/9/2011	9/25/2012	5/20/2013
Yrs on Stream	8.53	9.88	10.47	0.21	0.85
Rates (stpd)	306	305.98	300.46	300	309
Converter Outlet Pressure(psia)	4665	4535	4535	3835	3815
Converter DP (psi)*	225	170	180	40	60
Synloop DP (psi)	350	260	270	212	180
<b>Converter Inlet Gas Comp</b>					
NH3	1.73	2.66	2.74	2.505	2.9
Inerts	13.73	11.66	12.3	11.862	10.9
<b>Converter Outlet Gas Comp</b>					
NH3	14.40	12.66	12.95	18.2	17.8
Inerts	17.25	14.29	15.01		12.8
* Measured after quench control valves					

Table 1. Loop performance summary - magnetite vs AmoMax-10 RS

## Project Milestones

The following are the project milestones:

- Commenced study with Ammonia Casale in August 2009.
- Casale study was completed in September 2010.
- Purchase order for catalyst baskets in August 2011.
- Catalyst received at plant site in April 2012.
- Received all baskets and parts between April and May 2012.
- Catalyst was loaded in June 2012.
- Catalyst reduced and activated in July 2012.
- Plant performance tests of catalyst completed in September 2012.

## Conclusions

The project was mainly justified based on the end of life of the old axial flow path baskets and by the possibility of achieving a power reduction by lowering the operating pressure of the synthesis loop. The pressure vessels and nozzles were inspected, and the cracks that were found in the top heads were repaired. The old converter bas-

kets were replaced with new axial-radial flow design baskets supplied by Ammonia Casale. Also, a new wüstite-based Clariant AmoMax-10 RS catalyst was installed in all 3 baskets. By reducing the pressure of the synthesis loop and replacing the catalyst baskets and old piping, the safety and integrity of synthesis loop has improved significantly as well as the conversion efficiency.

The synthesis loop equipment (static and rotating) were checked and simulated by Ammonia Casale under the new operating conditions achieved after the revamping. This activity provided Dyno Nobel with an important tool for a better process control understanding during the operation of the synthesis loop.

The performance of new axial-radial baskets and of the Clariant AmoMax-10 RS catalyst in the synthesis loop has been excellent as seen by the stable and smooth operation of the plant during the last 9 months.

The exceptional performance of new baskets coupled with the Clariant AmoMax-10 RS catalyst has helped to reduce synloop pressures significantly by about 1000 psig (69 bar). Also, electrical usage of the makeup gas and circulating compressors has decreased.

If front end constraints did not exist, the synthesis loop improvements would allow for additional plant capacity.

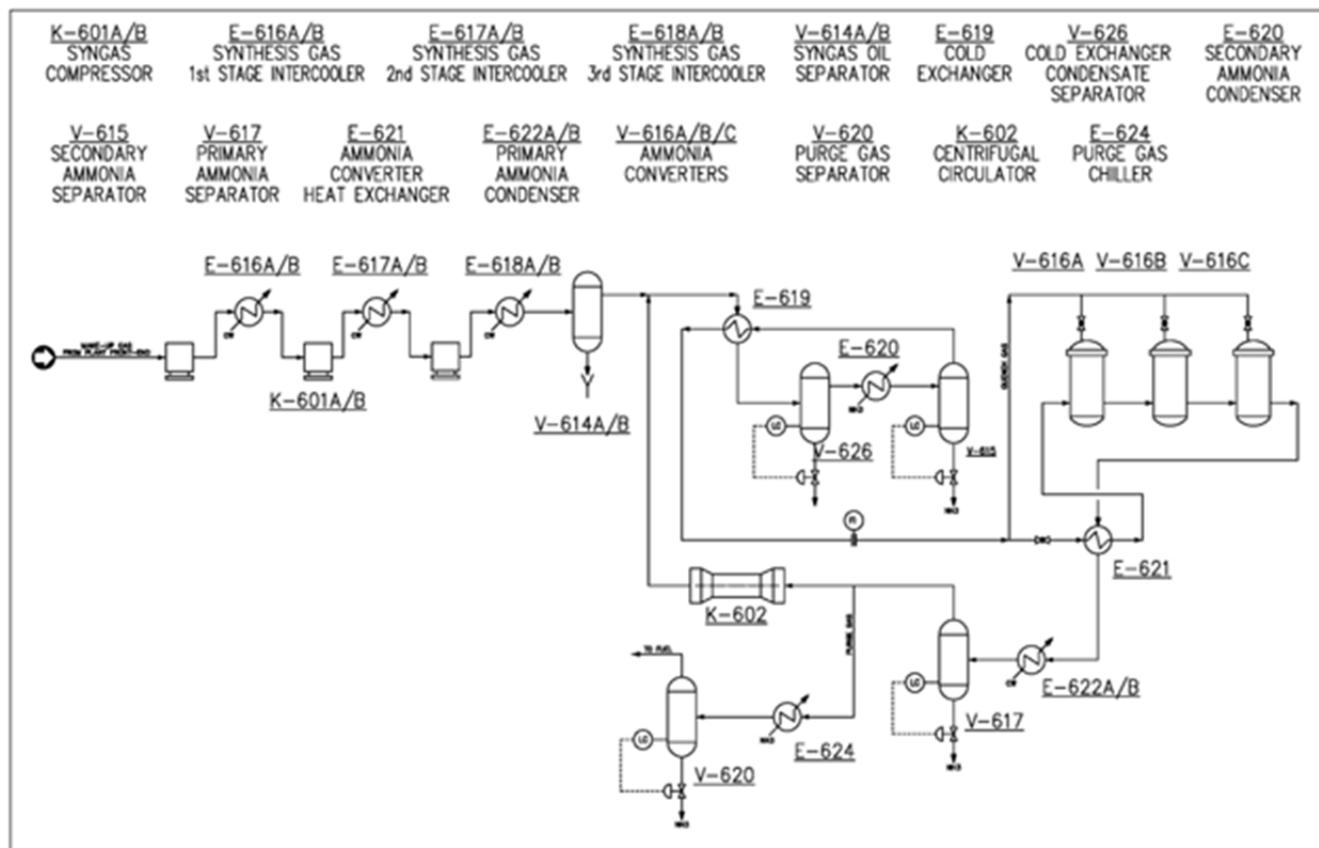


Figure 1. Ammonia synthesis loop

