Failure and Repair of Ammonia Converter Basket

How Shahpur Chemical's fertilizer manufacturing complex in southern Iran solved a major defect problem caused by chloride stress corrosion cracking

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This is a report on a failure of the 1,000-metric ton/day ammonia plant at Shahpur Chemical Co. in 1971, specifically a result of cracking that developed in the internal stainless steel basket of the ammonia converter. Cause of the problem was identified as chloride stress corrosion cracking. The repair methods developed and the precautionary measures adopted are discussed in this article.

The ammonia converter in the plant was fabricated in Japan by Babcock-Hitachi to ASME Section VIII and the M. W. Kellogg Co.'s purchase specification F40-1FS. Design pressure is 2,250 lb./sq. in. and design temperature 400°F. It was commissioned in the plant late in 1970. Within eight months of the initial output of ammonia, the plant was shut down for major repairs to the internal basket. Details of the process history follow.

After a series of commissioning problems, ammonia converter catalyst reduction started on September 11, 1970, and ammonia was first sent to storage 19 days later. However, within one hour of first making product, the start-up heater was taken out of service and reaction was lost on the converter. The outlet temperature on the first two beds dropped by approximately 300°F within five minutes. The heater was re-started and the bed temperatures restored with the exception of one bed, which remained at 600°F. The temperature of the re-cycle gas through the annulus of the converter was 27°F high, at 148°F. At this stage, the production rate was 480 metric ton/day. Relevant flow sheet details are given in Figure 1.

The temperature in the first bed was increased to activation temperature by reducing the flow to the first bed through the converter interchanger 122C. The temperature profile across the converter was now acceptable and the pressure drop was 13 lb./sq. in. When the start-up heater was taken out of service, the converter outlet temperature dropped from 540°F to 380°F and did not recover. With the annulus gas temperature still higher than design, it appeared that gas was by-passing the converter. The rupture disc protecting one of the loop exchangers 121C, was checked but was not the cause.

The syn gas circulation rate and make-up gas rate were increased to make more product. The temperature rise across the first bed dropped to about 50°F, the converter pressure drop increased to 60 lb./sq. in.; the converter outlet temperature dropped to 345°F (538°F normal) and the temperature of the annulus cooling gas increased to 160°F (117°F normal). Production rate was 600 metric ton/day.

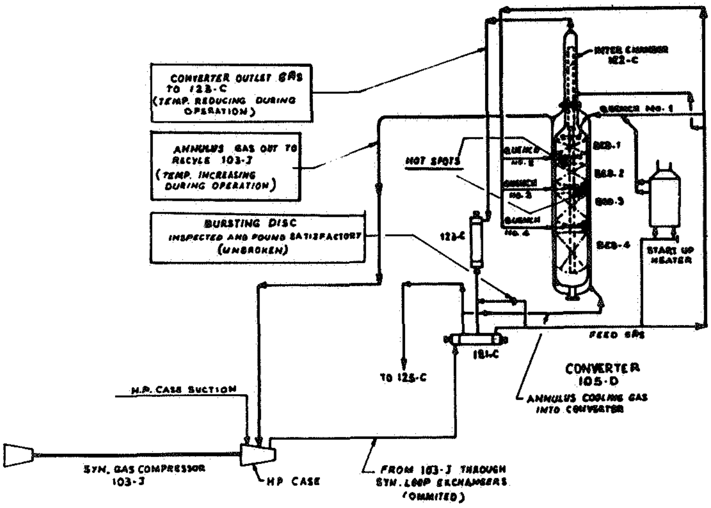


Figure 1. Simplified flow sheet of ammonia converter system.

However, within 24 hr. on October 7, 1970, with the annulus gas temperature still increasing, a hot spot appeared on the converter pressure shell at the top of the third catalyst bed. The hot area was discolored and the metal temperature, as measured with "temple" sticks, was between 415 and 500°F. At this point the plant was shut down.

The initial investigation then got underway. The converter was purged with nitrogen and the outlet piping from the interchanger 122C was removed. The expansion joint gasket on the interchanger was in good condition. The top manway on the converter was removed. The manway at the top of the basket was loose and catalyst dust had escaped into the annulus. This manway was secured in preparation for a basket pressure test. It proved impossible to maintain a nitrogen pressure inside the basket. A balloon fitted to the pressure shell manway inflated and deflated upon admitting and shutting off nitrogen to the catalyst beds. The basket was therefore proved to be leaking and the plant was re-assembled.

Before the plant went back into operation, the following changes were made:

1. Shell hardness tests were carried out around the hot spot area and revealed no metal deterioration.
2. An additional 47 metal temperature-recording thermo couples were installed on the pressure shell.
3. The shell was painted with a temperature indicating paint.

4. A new thermocouple was installed in the re-cycle line carrying the converter annulus gas to the compressor. Re-cycle temperature was not to exceed 250°F.

5. The differential pressure across the basket was not to exceed 35 lb./sq. in. initially and later 50 lb./sq. in.

6. A shell temperature limit of 480°F was imposed.

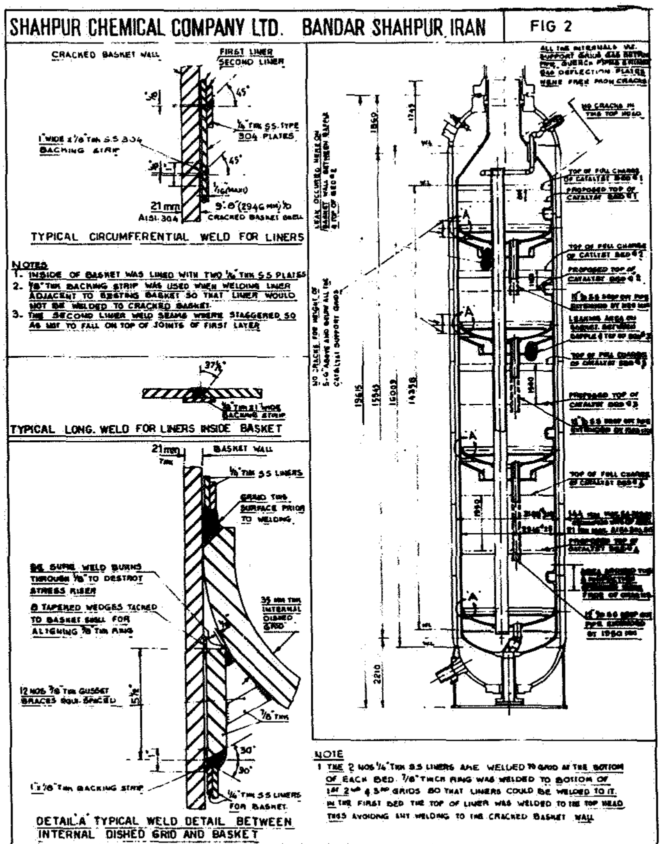
Appearance of a second hot spot

The plant was producing ammonia again by November 12, 1970. Production was limited to 600 metric ton/day by the converter shell temperatures. A second hot spot appeared at the top of the second catalyst bed. Both hot spots were approximately 3 ft. in diameter. Circulation rate was decreased to reduce the shell temperature to 480°. The temperature of the annulus re-cycle gas was 210°F. After four days' production, a series of other plant failures prevented further production until February 5, 1971.

The production rate on re-start in February was 800 metric ton/day, but this figure had reduced to 485 by May 25, 1971, when the plant was finally shutdown for basket repair. During the final stages of this period, the shell temperatures had on occasions exceeded 500°F and the temperature of the recycle gas from the annulus had been as high as 258°F. Throughout the operating period, the safe operation of the unit and possible repair procedures were under constant discussion with appropriate authorities, including the vessel manufacturer.

How converter basket was repaired

The converter was isolated, purged and catalyst removed to gain access to the inner basket. A repair team from the vessel manufacturer carried out the welding repairs.



NOTES

1. INSIDE OF BASKET WAS LINED WITH TWO 1/4" THK S.S PLATES
2. 7/8" THK BACKING STRIP WAS USED WHEN WELDING LINER ADJACENT TO EXISTING BASKET SO THAT LINER WOULD NOT BE WELDED TO CRACKED BASKET.
3. THE SECOND LINER WELD SEAMS WHERE STAGGERED SO AS NOT TO FALL ON TOP OF JOINTS OF FIRST LAYER

NOTE

1 THE 2 NOS 1/4" THK S.S LINERS ARE WELDED TO GRID AT THE BOTTOM OF EACH BED. 7/8" THICK RING WAS WELDED TO BOTTOM OF 1st 2nd & 3rd GRIDS SO THAT LINERS COULD BE WELDED TO IT. IN THE FIRST BED THE TOP OF LINER WAS WELDED TO THE TOP HEAD THUS AVOIDING ANY WELDING TO THE CRACKED BASKET WALL

Figure 2. Details of the internal basket, showing cracks and welds.

Referring to Figure 2, visual examination of the basket showed cracks in the basket wall on each of the four beds, 360° round. Most of the cracks were vertical. The two hot spots on the pressure shell at beds two and three were caused by large vertical cracks. Ultrasonic examination of the basket showed extensive cracking in the basket wall between the catalyst support grids. Many cracks were not visible from the inside basket wall. A band of cracks mostly in the horizontal direction was found in the second bed extending around the circumference of the basket. The cracking is illustrated in Figures 3, 4 and 5.

No cracks were found where the four catalyst support grids were welded to the basket wall for an area 5 or 6 in. above and below the weld. The area around the four 12-in. diam. inspection openings were free of cracks, as was the top head of the basket. The portion of the bottom head, which was examined, showed no cracks. The internals of the basket, i.e. the support grids, gas return pipe, quench pipes and rings, thermowells, and gas deflection plates and distributors, were also free from cracks.

Again referring to Figure 2, a "window" cutout was made in the basket wall at the hot spots in the second and third beds. An air arc was used to cut out the basket wall and the insulation and shroud behind it. Examination of these windows confirmed that the cracks had started on

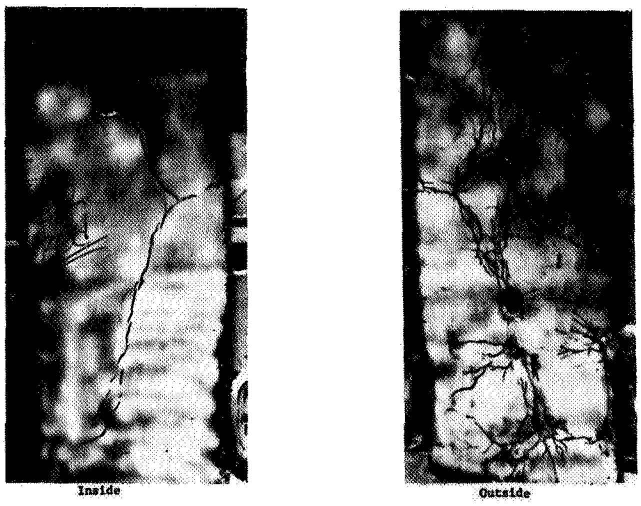


Figure 3. Photographs of the gross sample from the basket shell in bed 2. Dye penetrant applied to reveal smaller cracks. Left is inside and right is outside surface.

Figure 4. An etched section of the sample from bed 2, illustrating cracks originating at the outside surface.

Figure 5. Photomicrograph of a section from bed 2 sample, illustrating nitrided branching transgranular cracks. Oxalic acid electrolytic etch. Total magnification 200x (exposed at 100x, enlarged 2x).

the outside, and had propagated inward.

Examination of the external pressure shell at these two locations (dye penetrant, ultrasonic and hardness) detected no damage to the external vessel. However, the inside of the pressure shell had a thin coating of catalyst dust and insulation. The two windows were repaired by replacing the shroud and insulation and welding a ⅞-in. thick rolled stainless steel plate in position.

The repair of the basket wall was accomplished by lining the inside with two layers of ¼-in. thick plate. Three ¼-in. plates were installed at the second bed to give additional strength to the basket wall in the region of the horizontal band of cracks.

A liner was welded to the grid at the bottom of each bed. A ⅞-in. thick ring was welded to the bottom of the first, second and third grids so that the liner could be welded to it. In the first bed the top of the liner was welded to the undamaged top head.

The liner had to be installed in narrow strips (18 in.) about 5 ft. long so as to fit through the 20-in. manways. Backing strips ⅛-in. thick were used to avoid welding the liner to any part of the cracked basket. The second liner weld seams were staggered with the joints of the first layer.

All plates were of AISI-304 stainless steel except for the two layers of bed one and the inside layer of bed two, which was AISI 321.

The gas deflection plates and distributors were cut loose from the wall and lowered to give room to install the ⅞-in. thick ring and the two layers of ¼-in. plate. The catalyst drop-out pipes in each bed were extended to accommodate the reduced volume of catalyst loaded.

On completion of the repair, the vessel was cleaned with demineralized water and pressure tested to 100 lb./sq. in. with nitrogen. Converter was charged with 56% of design volume, i.e. 1,400 cu. ft. of Topsoe catalyst.

After repair the vessel was recommissioned with a pressure drop limitation of 90 lb./sq. in. across the basket. The ammonia production rate was thus reduced to approximately 90% of design capacity.

Temperature limits on shell

In making the decision to operate the converter with hot spots on the pressure shell, the following factors were considered:

1. Temperature limitation due to pressure stresses and the stress problem created by localized heating.
2. Thermal fatigue due to temperature cycles.
3. Hydrogen attack.

The manufacturer advised that a temperature at the center of the hot spot of 600°F could be tolerated if the radial temperature gradient did not exceed 400°F over a distance of 1.5 meters. He further advised that the temperature limit is 700°F due to stress resulting from internal pressure alone with no account for stressed induced by thermal gradient. The effects of thermal cycling were considered insignificant in this case due to the low number of cycles involved.

The design shell temperature of 400°F is based on an operating hydrogen partial pressure of 1,470 lb./sq. in. and on the assumption that the shell material is carbon steel. However, the actual material used on this vessel was ASTM A302 Grade C having 0.5% Mo and 0.7% Cr. Based on the Nelson curve, this material resists hydrogen attack at higher temperatures than carbon steel and 630°F was suggested as a safe operating temperature. A temperature drop across the shell wall of 30°F was assumed. It was also stated that a temperature of 800°F would result in very shallow hydrogen embrittlement over a prolonged period. The temperature limit therefore was attributable to the stress present in the shell rather than hydrogen attack considerations.

Cause of basket failure

The following is an extract from a report on the cause of cracking by Shilstone Testing Laboratory Inc., Texas:

"The basket shell materials were found to be Type 304 stainless steel, with the plate from Bed No. 3 actually complying with the requirements for type 304L. Zinc was found to be present at the outside surface of the sample from Bed No. 3; no other metallic contaminants were found. Cracking originated at the outside surfaces and was predominantly transgranular. The crack surfaces were nitrided to the same degree as the external surfaces. Chlorides were present in all samples of insulation, including the new, replacement material. The chloride levels varied considerably in the samples taken from the converter. The ratio of magnesium to calcium in the water soluble portions of the insulation samples from the converter was found to range from 1:3 to 1:5; that of the new, replacement insulation was 1:10. The unused catalyst was free of chlorides."

With this report of the findings of an independent consultant, consideration can now be given to the mode of failure. In the absence of any other contaminant except zinc, which is mentioned later, the failure can be attributed to chloride stress corrosion cracking. It can be established that the environmental conditions required to produce this defect existed on the outside surface of the basket.

The magnesium-calcium ratio of sea waters is approximately 3.5:1. The water soluble portions of the insulation samples from this basket exhibited magnesium-calcium ratios of 1.3 to 1.5. The new, unused insulation had a magnesium-calcium ratio of 1:10. These findings reveal that wetting of the basket insulation was not by sea water. Analysis of the new insulation indicated that water soluble chlorides were present to a max. of 8 ppm. A comparison with the chloride content of the used insulation established that additional chlorides had been in contact with the insulation. Based on the magnesium-calcium ratio, the analysis further concluded that the additional chlorides had arisen from contact with "fresh" water. The insulation had concentrated these chlorides towards the outer surface of the shell.

Records show that the water used for the final pressure test of the shell with the basket insulation in position contained 11 ppm. chloride. The Kellogg specification allows for 15 ppm. chloride.

The residual plate-rolling stresses in the basket material, and later the pressure stress occurring during catalyst reduction, contributed to the stress factor. As nitriding of the crack surfaces had developed to the same degree, cracking had obviously occurred prior to, or very soon after, commissioning.

The temperature increase above ambient usually required to initiate cracking occurred either during the drying process after hydrostatic test or during catalyst reduction. The temperature used for drying after hydrostatic test was 260°F.

The only other contaminant found was zinc, an unlikely source of cracking, since cracking by zinc occurs only at elevated temperatures and the 950°F service temperature probably was too low to result in intergranular penetration of the zinc.

Recommendations

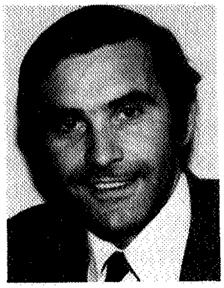
1. There are six similar design converters in operation, one of which has failed in a similar manner to that at Shahpur. With these six converter baskets, damage will be revealed by shell hot spots due to the gas leakage from inside the basket to outside. However, other converters operate with the differential pressure gradient in the opposite direction. Basket defects, therefore, will not be so readily detected. Converter baskets should be inspected closely at the next catalyst change.

2. In all maintenance work associated with converter internals, particular attention should be paid to controlling the ingress of chlorides.

3. Hydrostatic testing should be carried out with water containing less than 1 ppm. chloride. There are, however, many specifications which allow up to 25 ppm. for pressure testing of stainless steel vessels. Subsequent drying should be carried out at temperatures below 150°F.

4. The use of moisture retaining insulation for the basket should be avoided. Insulation should not contain chlorides. #

A. Kusha



D. Lloyd

DISCUSSION

BILL SALOT, Allied Chemical: Does your new ammonia converter design have the same kind of insulation around the basket?

KUSHA: No.

SALOT: Does it have insulation at all?

KUSHA: Yes, a one inch thick insulation blanket of MAX 5 ppm chloride retained by 316 stainless steel sheets.

KUSHA: Does not have any insulation at all.

SALOT: May I make a few comments then, based on that? You mentioned there are six of these "slim-jim" converters, and three have failed with cracks. My company has two that have not failed yet. That leaves one other that I don't know about. The two that Allied Chemical has in this country were the first two, and therefore the oldest. During the design stages, a question was raised about the insulation. Rock wool insulation was proposed to be put around the basket. We requested that it be eliminated in favor of multiple layers of reflective insulation. Later this year, one of those converters will have its catalyst replaced. We will go in and ultrasonically inspect for cracks. (SALOT has since learned that the insulation change was not made. The inspection has been completed and no cracks were found.)

KUSHA: You said three converters have failed. I said one I think was in Gulf, and the second one was ours. The third one, I understood yesterday, has failed during the last few days, but I am not sure which converter it was.

SALOT: The third one to fail was at Commercial Solvents, in Sterlington, La.

KUSHA: Thank you very much for all cooperation that has been given by Allied Chemical to Shahpur Chemical in design, construction and operation. We do have very good and close cooperation with Allied Chemical.

E.J. LEMIEUX, M.W. Kellogg Co.: The paper is very factual and I just want to make a few comments about our Specification FCSP-D which is our Ammonia Converter Special Precautions to try to minimize chloride contamination in ammonia converters. The last issue is Number 4, the first issue was in 1972 and the water for hydrotesting is now less than 2 parts per million for the basket and the shell. We do not allow any more heating of the drying gas and, of course, during shipping of the vessels they are continuously under pressure, nitrogen pressure, during the sea voyages.

The swabbing materials, rust inhibitors, dye penetrants and marking materials are tested for chloride. We do not allow any field hydrostatic tests of our converters.

KUSHA: Thank you very much, again, I hope that all these precautions will be taken care of in the construction of our new ammonia plant.

JAN BLANKEN, UKF: We operate two converters with a higher pressure in the basket than outside the basket. One since 7 years and one since four years.

Until I heard about the failure of the basket of the third convertor I did not worry too much because the failures of the first two baskets happened rather shortly after start-up.

Am I right in saying that the third basket failed after 6 or 7 years of operation?

KUSHA: Yes.