Operating experience on ammonia water-exchange system at Heavy Water Plant Talcher

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Introduction:

The Heavy Water Plant at Talcher employs bithermal ammonia hydrogen exchange process for the production of heavy water. The plant is integrated with the ammonia plant of Fertilizer Corporation of India (FCI). The synthesis gas ($3H_2$ + N_2) from the ammonia plant is passed through exchange towers of heavy water plant before entering the ammonia converter of FCI. In the exchange towers deuterium from the synthesis gas is extracted into a liquid ammonia stream containing potassium amide as catalyst. This ammonia stream is further enriched in deuterium in 3 enrichment stages of heavy water plant. In the third enrichment stage the liquid stream attains an concentration of 17.5 % in deuterium. Part of this enriched ammonia is withdrawn as a intermediate product and after removal of potassium is fed to a ammonia-water exchange column. In the ammonia-water exchange column enriched ammonia in vapour form is brought in contact with a stream of water containing approximately 10.6 % deuterium, and thus exchange of deuterium takes place between ammonia and water. Depleted ammonia with a deuterium concentration of 13.8%, after removal of water, is fed back to the third enrichment stage. Enriched water with deuterium concentration of 16.8%, after removal of ammonia is distilled in vacuum distillation column to get nuclear grade heavy water.

The ammonia-water exchange tower together with the heavy water distillation column and associated piping, equipment & machinery is supplied as a package unit by M/s Sulzer of Switzerland.

Description of existing ammonia-water exchange column:

The ammonia water exchange column is made up of stainless steel having an inside diameter of 250 mm and designed for a maximum working pressure of 20 kg/cm2. It is a packed column consisting of 3 sections. The upper section containing approximately 9 meters of packing serves to separate water from ammonia to a purity level of 1 ppm of water. The middle section containing approximately 12 meters of packing serves as the deuterium exchange section where deuterium from the rising ammonia vapour is transferred to the stream of water trickling down the packing. The lowest section containing approximately 6 metre of packing serves to separate ammonia from water to purity level of maximum 1 ppm ammonia in water. The column is provided with a head condenser (ammonia chiller) at the top and a thermosyphon reboiler at the bottom. The packings are made of stainless steel (316 L) wiremesh. The column operates at a pressure of 2 kg/cm2 absolute. The top and bottom temperatures are maintained at -20 Deg. C and +120 Deg. C respectively.

Process description:

A schematic diagram of ammonia water exchange column together with major controls is shown in figure 1. Enriched ammonia from IIIrd stage of enrichment unit after removal of catalyst is first vapourised in the feed evaporator and then introduced into the column in the form of vapour. The top product of heavy water distillation column together with required quantity of make up demineralised water is fed at the top of exchange section. Uncondensed gases from the top of the column are vented to atmosphere after bubbling through water and then passing through a cold trap to minimise loss of deuterium. Withdrawal of top product i.e moisture free depleted ammonia is controlled by a temperature controller (TIC) which keeps the ammonia hold up of the column constant. Ammonia free enriched water from the sump of the column is withdrawn with the help of a level controller.

Start up of the column:

Based on our experience in the commissioning stage the start up procedure for the ammonia water exchange column has now been streamlined and involves following major steps in the mentioned order of sequence.

1. Stabilisation of cooling water, chilled water and refrigeration loops.

2. Feeding of required quantity of water and ammonia into the ammonia water exchange column and raising the boil up in the reboiler to the designed value.

3. Stabilisation of water cycle in closed loop.

4. Establishing required water and ammonia hold up of the column by operating the column in total reflux with respect to ammonia and by maintaining desired temperature profile.

5. Stabilisation of ammonia cycle in closed loop and ensuring the purity of top and bottom products.

6. Coupling of column with the enrichment section and heavy water distillation column after approximately 2 to 3 hours stable operation of the column in closed ammonia/water cycle.

Operating experience at heavy water plant Talcher:

Operating experience at Heavy Water Plant Talcher shows that performance of ammonia water exchange column is very sensitive to the temperature profile of the column. Any change in ammonia hold up of the column and consequential contamination of top and bottom products with water and ammonia respectively is immediately reflected in the temperature profile. Thus proper monitoring and control of the temperatures at different locations serves as an excellent means to ensure stable operation and desired performance of the ammonia water exchange column.

When the column operates in stable condition coupled with heavy water distillation column and the enrichment section the following temperature profile is attained.

Temperature point	Location (approximate distance from top)	Temperature
TR-7703	Column head	-20 Deg. C
TIC-7701	Mid-column temp. 11 m (2m below water feed point)	30 Deg. C
TI-7726	12.2 m	80 Deg. C
TR-7752	14.2 m	83 Deg. C
TR-7727	21.0 m (at ammonia vapour inlet)	110 Deg. C
TR-7704	27.0 m (bottom)	120 Deg. C

The relative locations of these temperature measuring points are shown in figure 2. As can be seen from this figure, in the 1.2 meter zone between TIC-7701 and TI-7726 the temperature changes sharply from 50 Deg. C to 80 Deg. C whereas at TIR-7752 i.e 2 meters below the temperature is 83 Deg. C - a rise of only 3 Deg. C in 2 meters.

Stable operation of the ammonia water exchange column at Talcher is achieved with the mid-column temperature set between 20 Deg. C and 30 Deg. C. The temperatures at TI-7726 and TR-7752 are observed to come down occasionally upto 50 Deg. C and 53 Deg. C respectively without seriously affecting the operating stability or the purity of top and bottom products. Similarly increase in mid-column temperature even upto 95 Deg. C (at TIC-7701) does not affect the column top temperature of -20 Deg. C (at TR-7703) or the purity of the top product. However, a prealarm at 60 Deg. C and an alert alarm at 85 Deg. C for TIC-7701 have been provided.

As expected, the operating experience confirms that the temperature at the top of the column (TR-7703) is very sensitive to the presence of water in the top product. Similarly the temperature at the bottom of the column (TR-7704) is sensitive to the presence of ammonia in the bottom product. Close monitoring and control of these temperatures therefore serves an effective means to ensure desired purity of bottom and top products. It is also observed that the mid-column temperature at TIC-7701 and the temperature at ammonia inlet section (TR-7727) give useful advance information about the trend in operating stability and possible contamination of top and bottom products.

Thus when the mid-column temperature TIC-7701 goes above 85 Deg. C the column is immediately put in close loop operation with respect to top product i.e. ammonia, anticipating contamination with water. Similarly when the temperature at the ammonia inlet section (TR-7727) falls below 110 Deg. C, the water cycle is put into close loop operation due to possible contamination with ammonia.

Problems encountered in operation of the column:

i) The original scheme had provisions for operating only the ammonia stream in closed loop. There was no provision to put the water stream in closed loop operation. The column was commissioned when the main plant was operated with only pure ammonia containing no catalyst (Potassium), and thus there was no enrichment either. Demineralised water could, therefore, be fed continuously and drained from the column to put the column in stable operation. Also whenever the temperature at the bottom of the column dropped below 90 Deg. C, due to excess ammonia inventory in the column, a greenish precipitate containing chromium and nickel compounds was observed to contaminate the sump water. This was due to release of coating provided on certain portions of the packings in the bottom section by contact with hot ammonia. However, this did not affect the column performance.

During normal operation also, when the column is stabilised with deuterium rich ammonia column, the water at the column sump contains appreciable amount of ammonia. This water can neither be taken to heavy water distillation tower as it will damage the copper packing of the distillation tower nor it can be drained. Hence, the system has been modified whereby the sump water at 120 Deg. C is cooled in a double pipe heat exchanger and recycled to the ammonia water exchange column through a filter. The filter helps removing out the precipitate. This arrangement has now become an integral part of the Unit and without this start-up/stabilisation of the column would be extremely difficult.

ii) It is also observed that prolonged operation of the column in closed loop with respect to ammonia results in water flowing back to the ammonia feed evaporator and forming a solution with cold ammonia. Potassium collected in the feed evaporates also dissolves in this solution. The effectiveness of feed evaporator is lost and this solution finds its way into the column as liquid and adversely affects the columns stability. The conductivity of water in the sump also shoots up sharply due to the presence of potassium. Keeping the above experience in view the feed evaporator is now washed free of potassium with demineralised water after the column is shut down. Also in the event of ammonia feed not being available for more than three hours, operation of the column in closed loop with respect to ammonia is avoided and the column is shut down.

iii) During one of the operations, the pressure drop across the hot column of the third enrichment stage was observed to increase sharply indicating choking of the trays. On close scrutiny it was observed that ammonia water exchange column top temperature had gone upto + 10 Deg. C instead of the normal value of -20 Deg. C though the mid column temperature was steady around 40 Deg. C. An immediate investigation revealed that one of the bellows in the electro pneumatic transducers in the temperature indicator loop had been leaking resulting in loss of signal. Thus even though the actual mid-column temperature went beyond 100 Deg. C the indication was only 40 Deg. C and alarms set at 85 Deg. C did not get actuated. This resulted in the top product ammonia getting contaminated with water. This water got carried to the enrichment stages where it reacted with potassium amide solution to form solid deposits of KOH and choked trays. The bellows were replaced and as an additional safety, a prealarm set at -10 Deg. C has been provided for the column top temperature. It is also proposed to provide an independent mid-column temperature indication with alarm to improve reliability.

Conclusion:

The existing arrangement of the column has given excellent performance. There was no problem with regard to deuterium exchange between the ammonia and water streams. The purity of the ammonia and water streams are extremely critical with regard to process safety of the exchange section and D2O column respectively. The temperatures at the various points in the column in stable operating conditions give a very sure indication of the top and bottom products purity. By maintaining these temperatures in the desired operating range reliable performance of the column has been achieved. The operating experience gained and the data collected at heavy water plant Talcher over the last few years can be utilised for design and operation of new ammonia water exchange column. This will not only be useful for integration with existing heavy water plants down stream of the exchange sections, but also is a prerequisite for considering operation of future heavy water plants independent of ammonia plants.

AMMONIA WATER EXCHANGE COLUMN AT HWP TALCHER WITH AMMONIA AND WATER FEED DRAW OFF SYSTEMS AND BASIC CONTROLS

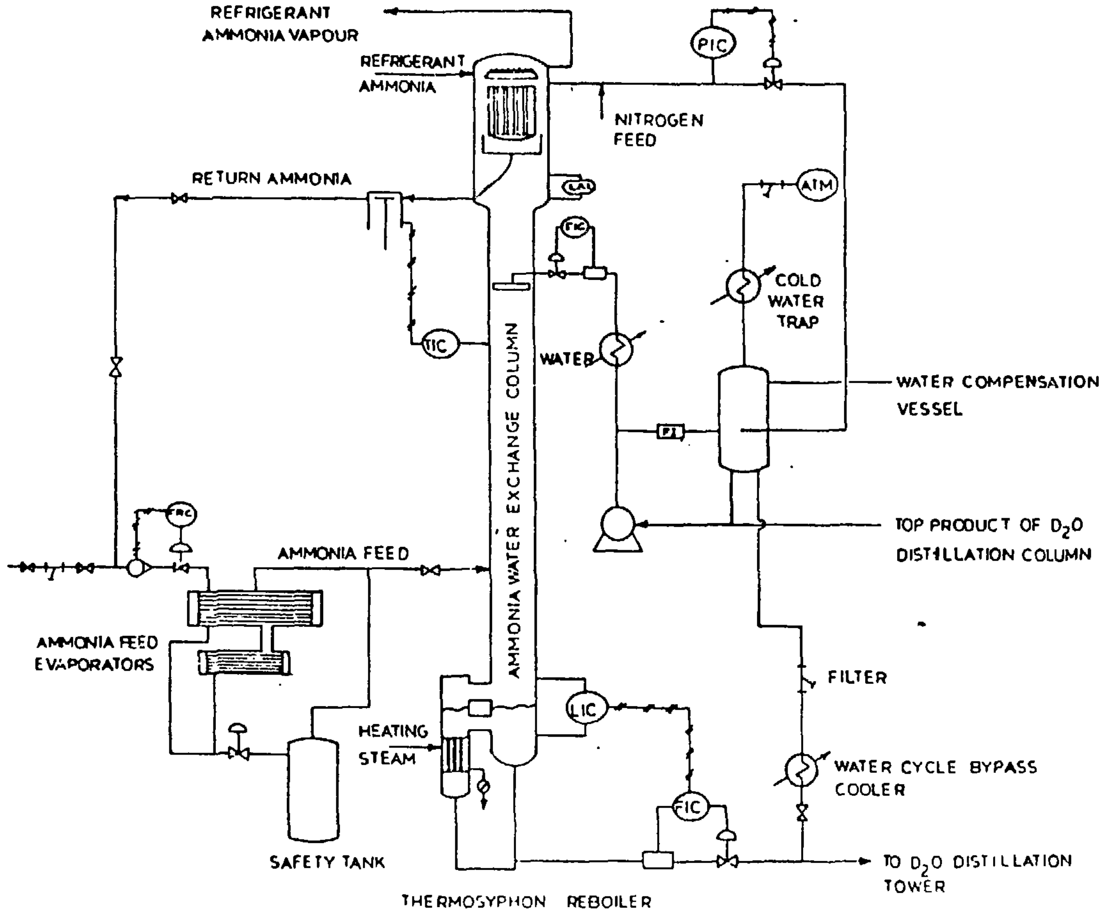

FIG.1

TEMPERATURES IN AMMONIA-WATER EXCHANGE COLUMN

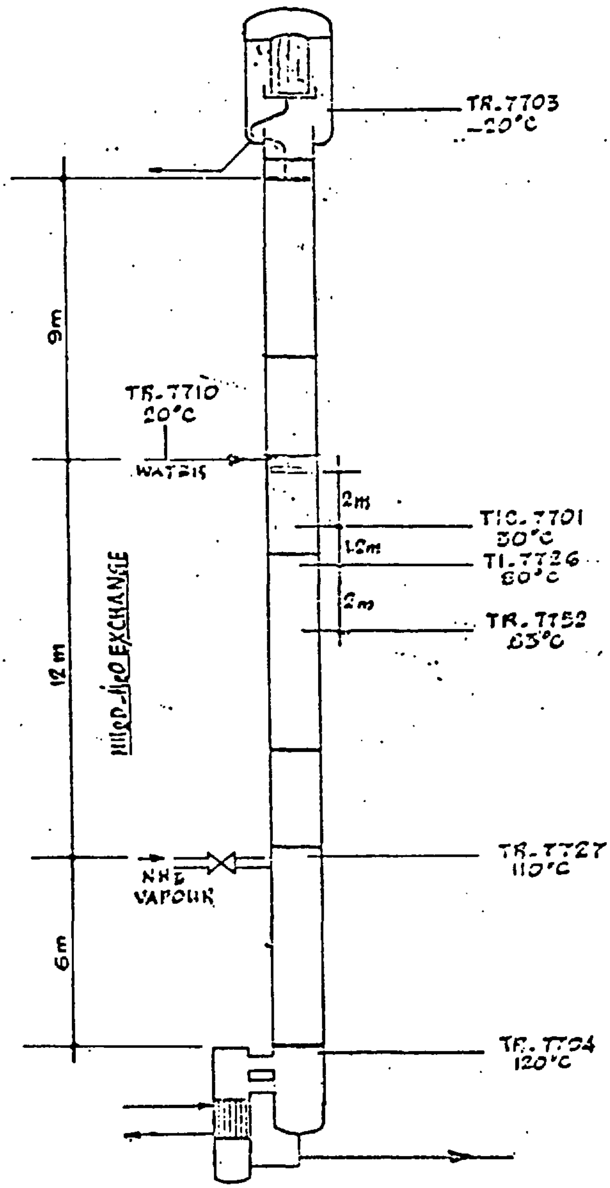

FIG. 2