

Hydrogen Isotope Separation in the JET Active Gas Handling System during DTE1

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1. INTRODUCTION

During the Deuterium-Tritium-Experiment (DTE1) the JET Active Gas Handling System (AGHS) supplied tritium and deuterium to the JET tokamak and processed the exhaust gases of the torus with the main intention to recycle tritium and deuterium, to detritiate impurities containing tritium and to discharge detritiated gas mixtures via the stack.

The gas processing in the AGHS was performed in various subsystems which were partially discussed previously /1, 2/. Two important subsystems were the preparative Gas Chromatographic (GC) system /3/ and the Cryogenic Distillation (CD) system /4, 5/ which were used for hydrogen isotope separation and enrichment of deuterium and tritium.

This paper will present the use and performance of both systems during DTE1 and the subsequent clean-up phase until the start of the Remote Tile Exchange (RTE) at JET. It will be shown that the operation of both systems was fundamental for the successful re-processing of the gases in AGHS and the supply of pure tritium and deuterium. The preparative GC system built for 100% enrichment of large amounts of T₂ and D₂ must be distinguished from the analytical GC (AN-GC) system /6/ which is used for quantitative determination of gas contents in various gas mixtures.

2. THE JET CD SYSTEM

In distillation the principal mechanism in the separation process is the difference in vapour pressure between the individual component liquids: the less volatile isotopes, mainly the heavier hydrogen molecules in comparison to the lighter ones, are enriched in the liquid phase flowing downwards in a packed distillation column operating at around 21 K, whereas the more volatile isotopes are enriched in the vapour phase flowing upwards.

The CD system was designed for a daily throughput of 30g T₂, 60g D₂ and 150g H₂ and for purity higher than 99.989% in the case of H₂ and T₂ (assuming an optimum tritium inventory of 33g) and higher than 99.998% for D₂. The requested impurities were smaller than 0.001% for H₂ and T₂ and smaller than 0.01% for D₂. Three columns (COLs) and two equilibrators for the removal of HD, DT and HT were installed. A few design parameters for the columns are listed in table 1

The use of the CD system requires a preliminary cooldown period of between 2-3 days to achieve an operational state. This action is done remotely using an automated control sequence such that operator intervention is only optional. Further automated operational modes were available again to minimise manual intervention, and they were used to some extent although manual control was at times more convenient for specific feed and product control. This was especially the case with inventories containing little protium component (e.g. in column 1). The use of CD under these conditions was not envisaged in the original design and manual control was sometimes necessary.

Table 1:

| DESIGN PARAMETERS | COL-1 | COL-2 | COL-3 |
|-------------------------------------|-----------|----------|-----------|
| Number of stages | 80 | 80 | 100 |
| Feed Locations | 60/79 | 40/79 | 45 |
| Sidestream drawoff | — | — | 42 |
| Intermediate boiler location | — | — | 60 |
| HETP (mm) | 60 | 60 | 75/60 |
| Packing height (m) | 4.8 | 4.8 | 6.9 |
| Inner diameter (mm) | 16 | 16 | 17/13 |
| Column top pressure (mbar) | 1005-1200 | COL 1 +5 | COL 1+ 10 |
| Typical column pressure drop (mbar) | 4 | 5 | 8 |
| Condenser temperature (K) | 18-20 | 20-24 | 23-26 |
| Reboiler temperature (K) | 21-24 | 21-24 | 23-26 |
| Condenser duty (W) | 10 | 13 | 15 |
| Reboiler duty | 8.5 | 9 | 7/4.5 |
| Reflux ratio | 6 | 12 | 35 |

The main purposes of the control system are: i) to maintain separation performance even during process perturbations, ii) to react quickly to load changes and set point variations initiated by operator, and iii) to establish and maintain pre-set operational modes to minimise operator input.

The columns have several variable inputs (flows, heat loads, etc.) and subsequent variable states (pressure, concentration, internal gas flows, etc.) that must be kept between permissible limits. A single change in input can affect several system parameters in any of the three columns.

The control strategy is therefore based on the need to maintain mass and energy balance in each of the columns under a transparent mode of control which incorporates single and cascaded loops with PID algorithms. There are different methods of achieving this control regime, however with the JET design the 3 columns are coupled together by their feed and recycle streams thus requiring minimum interference between them for stable operation especially as the flow rates between the columns vary only slightly. Feed rates to the columns are kept at a constant rate using fixed power syphon pumps.

It is the sump level change of each column that indicates the mass balance of that column. As the feed rates of each column are already fixed, any change in sump reboiler duty affects primarily the distillate (top) flow and its purity. A direct cascade control of sump level from product flow is not desirable due to the long dynamic response time. The sump level is directly controlled by the reboiler to a specific level irrespective of system perturbations. Hence the

product flow purity is affected by this process. Any mass change in the column will affect the sump level and the response of the reboiler is to compensate.

Energy balance within each column is reflected by individual pressure rises. A steady pressure in each column is essential for establishing a steady separation profile. In normal operation the pressure is set at one point only: in case of the JET CD system at the top of COL1 as governed by its condenser power. A higher pressure in the column induces a higher helium cooling power by valve control. No direct pressure control exists for COL2 and 3 and so a designed imbalance exists between the reboiler and the condenser duties.

The CD system was operated between week 22, 1997 and week 8, 1998 almost continuously. First, the CD volume of 800L was filled with 1720 barL of hydrogen from uranium (U) beds in Intermediate Storage (IS) system and cooled down to about 21K. Then 11920 barL of hydrogen gas mixture were added over the weeks mentioned above. The main part of this gas injected into the torus was not supplied by the AGHS. The AGHS and the Torus were a closed gas loop only with respect to tritium. Due to the limited storage capacities of AGHS these additional gases had to be detritiated and discharged. This was achieved with the CD system. 9480 barL with 410 GBq were discharged mainly from the top of COL1 into the stack. The tritium concentration at the exit of top of COL1 was in average 0.5 ppm because COL1 was mainly filled with deuterium which restricted severely the separation capability of COL1 designed for handling protium. The 9480 barL gas were released in daily batches $< 0.5\text{m}^3$ to stay within the self-imposed management limit for daily discharges of less than 40 GBq. 700 barL with 97 GBq from top of COL1 and 320 barL with 214 GBq from top of COL3 were discharged to the Exhaust Detritiation (ED) system. The whole tritium activity of these 1020 barL was collected in ED as water. Further transfers from CD to other subsystems of AGHS: 750 barL with 173 GBq were moved from top of COL3 to PS-D2 U-beds and 710 barL with 773 TBq from bottom of COL3 to IS and GC U-beds. The residual gas in CD after warm-up to room temperature was finally stored in IS U-beds for safety reasons.

In summary: 2.2g (8.8 barL) of tritium were enriched in CD, maximum tritium concentration in the T2 product was about 35.2%. Higher tritium concentrations in the T2 product are expected with higher tritium inventories in CD. 9480 barL of hydrogen were directly discharged into the stack and 1020 barL to ED. Only D2 molecules were detected in the D2 product from top of COL3 with the thermal conductivity detector (lower detection limit: 50 ppm).

3. THE JET GC SYSTEM

Hydrogen gas mixtures injected into a column filled with Pd deposited on Al_2O_3 and forced through the column by H_2 are separated into the product T2, interfraction T2+TD+D2, product D2, interfraction D2+DH+H2 and product or eluant H_2 due to the large isotopic effects of hydrogen in contact with Pd.

Approximately 30 barL of hydrogen gas mixture to be separated were injected into the helium filled Pd column with a total hydrogen absorption capacity of about 130 barL and forced through the column by addition of protium. At the outlet of the columns automatic valves were switched as a result of the hydrogen concentrations seen by katharometers and ionisation chambers. The products T2 and D2 were switched into different volumes where their purity and amounts were assessed. The interfractions were collected in GC U-beds and injected again. When protium was observed at the exit, further addition was stopped and the regeneration of the columns started. Careful regeneration of the columns was important for very good T2 product quality to reduce H2 content.

During after DTE1 more than 160 separation runs were performed. In 40 runs pure tritium was cut for re-use. Most of these runs were performed during and shortly after DTE1 when the tritium concentration in the gas mixture was still high. The largest tritium amount produced in one separation run was 8g of very pure tritium. Approximately 1 barL and 0.4 barL of gas were needed for the interfractions T2+TD+D2 and He-T2, respectively. This showed that for the production of pure tritium the injected hydrogen gas mixture must have a tritium concentrations higher than 3% because otherwise the tritium will only contribute to build-up of the interfractions. 105 runs were performed to pre-enrich tritium. The tritium interfractions were collected, stored in an internal GC U-bed and finally injected into one of the four columns for pure tritium production. 15 runs were done to remove tritium from the eluant gas. The whole column was filled with the eluant gas and optimum use made of the whole column.

Approximately 160g of tritium were processed in 4700 barL of hydrogen gas in the GC columns. 96g of tritium and 440g of deuterium were produced. The best tritium purity achieved was: 99.96% T, 0.01% D and 0.03% H. A typical deuterium product purity was: 99.70% D, 0.17% H and 0.13% T. The analytical technique used a thermal conductivity detector and ionisation detectors.

4. COMBINED USE OF JET CD AND JET GC SYSTEMS

Due to historical reasons two isotope separation systems were installed and commissioned at JET. The operation of both systems during and after DTE1 was necessary. The GC system performed the enrichment of the tritium to almost 100% as requested for neutral beam injection, but was not able to detritiate gas mixtures to dischargeable low tritium concentrations. The CD system was not able to produce tritium concentrations higher than 35% due to the limited tritium inventory, but produced the low tritium concentrations (<1 ppm) in gas mixtures for direct discharge into the stack.

The GC and CD systems were used in the following way: Deuterium product and eluant gas of the GC system were transferred to CD for further detritiation and T2 product from the bottom of COL3 of CD was moved to GC for final enrichment to 100%.

5. CONCLUSIONS

Operation of the GC system was very simple. Changes in flow rate, column temperature, column pressure, etc., seemed not to influence the product quality in an easily detectable way when between certain limits. The GC system consistently produced far better tritium than the required specification for recycling to the J1 machine

Operation of the CD system was more difficult than GC, but could be handled very efficiently by experienced operators. Perturbations of the system could occur, but were stopped often by small changes of set points. The CD system produced the low tritium concentrations which allowed direct discharge of large gas quantities into the stack. In principle, the CD system should also produce very pure tritium if the tritium inventory is increased to 33g in CD.

In summary: The CD and GC systems performed very well during and after DTE1 and were both necessary for the successful management of the AGHS and of DTE1.

6. REFERENCES

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