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Chromatographic System of the  
JET Active Gas Handling System –  
Tritium Commissioning and  
Use during and after DTE1

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# The Preparative Gas Chromatographic System of the JET Active Gas Handling System – Tritium Commissioning and Use during and after DTE1

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Preprint of a Paper to be submitted for publication in  
Fusion Engineering and Design

February 1999

## ABSTRACT

The preparative Gas Chromatographic (GC) system at JET was designed to produce deuterium and tritium of purity > 98%. Displacement gas chromatography with Pd as the packing material is used. The paper explains briefly this batch method and explains in detail the tritium commissioning as well as the full use and performance of the GC system during the Deuterium-Tritium Phase and the subsequent clean-up phase at JET. More than 160 g of tritium were handled in 160 GC isotope separation runs. The best tritium and deuterium qualities achieved were in the range of 99.9 and 99.7 %, respectively. Finally, a few possible improvements of the GC system are discussed.

## 1. INTRODUCTION

The preparative Gas Chromatographic (GC) system is part of the JET Active Gas Handling System (AGHS), the performance of which is described in an overview elsewhere in this volume [1].

The objectives of the JET Active Gas Handling System (AGHS) are:

- a) Continuous evacuation of the torus and of equipment connected to the torus,
- b) Separation of the pumped gases (mainly hydrogen, small amounts of helium, and impurities (hydro-carbons created by the interaction of the plasma with the wall materials of the torus and nitrogen, oxygen, water from leaks, etc.)) into streams of hydrogen, helium and impurities,
- c) Detritiation of impurities and recovery of hydrogen and especially of tritium,
- d) Isotopic separation of hydrogen mixtures ( $Q_2$ ) into product streams of tritium ( $T_2$ ) and deuterium ( $D_2$ ),
- e) Storage of  $T_2$  and  $D_2$ ,
- f) Re-supply of  $T_2$  and  $D_2$  to the users,
- g) Discharge of detritiated gas streams into atmosphere,
- h) Achievement of the tasks given above in a safe manner with minimum risks for the operators and for the environment.

The JET AGHS is a tritium gas processing plant which contains the necessary measures for the safe handling of tritium. These are:

- a) All primary process containments are surrounded by secondary containments and in some cases by tertiary containments. Tritium leaked or permeated through the first primary wall is captured in multiple containments.
- b) The AGH-plant and the torus constitute an almost closed gas loop with respect to tritium. Detritiated gases (helium, protium, nitrogen,  $CO_2$ , CO, etc.) which are to be discharged have to pass through the Exhaust Detritiation (ED) system which has a detritiation factor larger than 1000.

Furthermore, tritium-hydrogen-impurity gas mixtures are processed in the AGHS and  $T_2$  and  $D_2$  are re-used which means that transport of these gases to other tritium processing plants is not necessary. Also the transport of new tritium to JET (in the absence of a tritium breeding blanket) is minimised.

- c) A Distributed Control System (DCS) controls and monitors the various processes inside the AGHS. The DCS can observe unsafe operational parameters and can stop equipment and processes and even shut down the subsystems or the whole AGHS.
- d) Safety of the system is provided by a special hardwired interlock system which is capable of recognising the generation of unsafe process conditions and of shutting down part or the whole AGHS. This hardwired interlock system is independent of the DCS and contains only simple diagnostic tools and simple electric circuits (no integrated cards or computers) with a very high reliability.
- e) During tritium transfers the AGH building is constantly manned by a team which has to follow Local Rules specifying many aspects of management of a tritium plant such as: management structure; operating directives; design; installation; commissioning; operation; maintenance; modification; radioactive material control; waste management; radiological protection; and training. Operating Instructions are also followed for the processing of the various gases within the AGHS.

The JET AGHS [1] is shown in block form in Fig. 1 and can be split into ten subsystems:

- a) The Cryogenic Forevacuum (CF) system [2] which pumps all gases from the torus. Helium is cryo-adsorbed on charcoal and all other gases are cryo-condensed on surfaces cooled with liquid helium. These cryo-pumps can be regenerated such that separation of the pumped gases into helium, hydrogen and impurities is possible with a very high degree of purity.
- b) The Mechanical Forevacuum (MF) system which is used to pump the torus and other connected systems down from atmosphere, to supply a certain sub-atmospheric pressure to the torus in case of a large leak, to transfer gases between various AGH subsystems and to discharge gases from AGH-subsystems.
- c) Impurities from CF and other subsystems of the AGH-plant are sent to the Impurity Processing (IP) system for detritiation. The operational experience with this system is described in a companion paper [3].
- d) The hydrogen gas collected in CF is sent to the Intermediate Storage (IS) system [4] for temporary and safe storage in JET designed uranium (U)-bed before being injected into one of the two isotope separation systems at JET.
- e) The Cryogenic Distillation (CD) system [5].
- f) The preparative Gas Chromatographic (GC) system [6].
- g) The  $D_2$  and  $T_2$  products of these two isotope separation systems are stored in U-beds in the Product Storage (PS) system [4].

- h) From PS  $D_2$  and  $T_2$  are sent via the Gas Introduction (GI) and Gas Distribution (GD) systems to the various users around the torus.
- i) Gases are discharged through the Exhaust Detritiation (ED) system where hydrogen and gas species containing hydrogen are catalytically oxidised and the resulting water vapour is trapped on very dry molecular sieve beds.
- j) Gas samples from the different subsystems can be sent to the Analytical Laboratory (AN) [7] for characterisation of their composition. The analytical techniques available are mass spectrometry, analytical gas chromatography and ionisation chambers.

This paper will present the design of the preparative GC system at JET, explain various safety measures installed for the handling of tritium and will discuss active commissioning results obtained with tritium gas mixtures for the first time as well as the use of the GC-system during the first Deuterium-Tritium Experiment (DTE1) [8] and the subsequent clean-up phase [9] at JET.

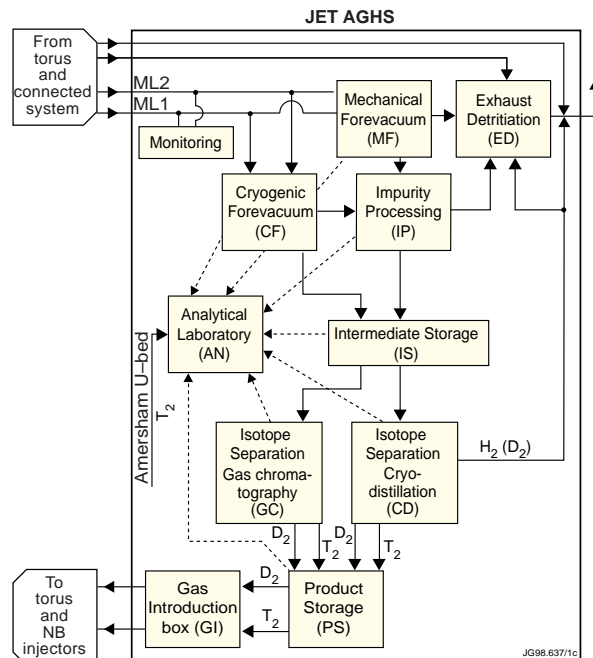


Fig. 1. Schematic block flow diagram of the AGHS showing the various subsystems and their main connections.

## 2. DESCRIPTION OF THE PROCESS OF THE JET GC SYSTEM

### 2.1 Physical principles

The solubility of hydrogen in Pd shows a very large isotope effect [10]: the lighter hydrogen has a higher affinity to Pd than the heavier one, consequently to absorb the same amount of tritium in Pd as of protium or deuterium a far higher pressure is needed.

If a hydrogen isotope mixture ( $Q_2$ ) is forced through a Pd filled column, the heavier hydrogen molecules prefer to stay in the gas phase while the lighter ones absorb in the Pd. At the  $Q_2$  front there is a region where the tritium concentration in the gas phase and that absorbed in

the Pd increases to almost 100%. As the isotope mixture is forced through the column this region containing the high tritium concentrations grows absorbing tritium from the tritium gas mixture. Similar effects occur at the front between the  $Q_2$  mixture and the protium gas used for displacement. Deuterium forces tritium into the gas phase and protium replaces deuterium and tritium. As a consequence after many absorption and desorption processes of the displaced hydrogen in the Pd the gas mixture just in front of the  $H_2$  gas is enriched in deuterium up to nearly 100%. At the same time the tritium is removed which means that the tritium concentration decreases. If 100% deuterium is reached in a very thin region of the column, this region will increase in width. This process of enrichment of the deuterium and of the tritium regions continues until the  $Q_2$  gas mixture is used up. From then on the separated batches of tritium, deuterium and protium move with their small interfractions through the columns with no further improvement of the achieved gas purity. When they reach the column exit the gas contained in Pd is desorbed into the gas phase by the forcing  $H_2$  eluant gas. In this process tritium gas will exit the column first followed by deuterium and afterwards by protium.

If protium is used to force the gas mixture through the column, the technique applied is called *displacement gas chromatography*.

## 2.2 Displacement gas chromatography

Figure 2 shows a simplified flow diagram of the JET preparative GC system (only one of the four parallel columns is shown schematically).

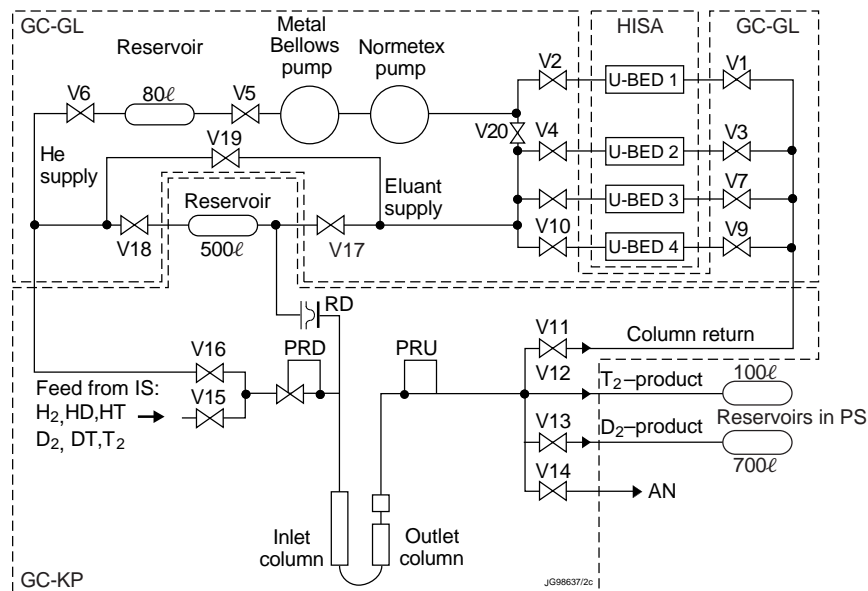


Fig. 2. Schematic flow diagram of the GC-system at JET: V1 to V20 are automatic valves, PRD (PRU) down (up) stream pressure regulators at the column inlet (outlet), RD rupture disc, U-bed uranium bed, IS intermediate storage system, PS product storage system, AN analytical system.

Displacement gas chromatography is a batch process. The various steps of this process are discussed below.

*a) Starting conditions:*

At the start, the Pd is in a thoroughly regenerated condition. The column is filled with helium and heated to 320 K, the valves V11, V1, V2 and V5 are open (all other ones are closed), and the bellows and Normetex pump are running. U-bed 1 removes all reacting gases (mainly  $Q_2$  and during the separation run the interfractions) and the pumps compress the helium gas into the 80L reservoir to a pressure of about 0.13 MPa.

*b) Injection of gas mixture to be separated:*

The hydrogen isotope mixture  $H_2+HD+HT+D_2+DT+T_2$  from the Intermediate Storage (IS) System enters the helium filled column via valve V15. When approximately a quarter of the column is filled, valve V15 is closed. Isotope separation occurs even during this stage.

*c) Injection of  $H_2$  displacement gas:*

Protium from U-bed 3 or 4 is injected into the column by opening the valves V8 or V10 and V19 and V16. This eluant displaces the gas mixture and helium. The separation continues until the interfractions and products exit the column in the following sequence:

‘pure’ helium;

interfraction He+ $T_2$ ;

‘pure’  $T_2$ ,

interfraction  $T_2+DT+D_2$ ;

‘pure’  $D_2$ ;

interfraction  $D_2+DH+H_2$ ; and

‘pure’ eluant.

For simplicity only the main hydrogen molecules are listed.

*d) Switching of valves to select the products tritium and deuterium:*

The ‘pure’  $T_2$  and  $D_2$  gases are transferred to the 100 L and 700 L reservoirs of PS by opening only valve V12 and V13, respectively, whereas all interfractions are moved by the open column return valve V11 into U-bed 1. Only one valve at the column exit is open at any time. The switching of the valves is controlled by various diagnostic signals (see Section 3.2).

*e) Regeneration of the Pd:*

If ‘pure’ eluant is observed at the exit, the addition of further eluant is stopped by closing the valves V8 or V10 and V19 and V16. If no further separation runs are planned (at JET four



parallel columns of equal size and of very similar properties are available) the U-bed 3 or 4 supplying the eluant is cooled down to room temperature.

The protium in the Pd packing is desorbed by heating the column to 473 K with valve V11 and one of the inlet valves to U-bed 3 or 4 open. Valves V1 and V3 are closed. U-bed 3 or 4 absorbs the eluant gas that is both in the pipe work and released by the Pd packing. Residual amounts of protium in the packing can contaminate the product quality of T<sub>2</sub> and D<sub>2</sub>. As pumping efficiency on the Pd packing is poor, helium is circulated at 473 K through the column (V6, V16, V11, V1, V2, V5 are open, all other valves are closed) to remove the low protium equilibrium pressures above the Pd and scrubbed by the cold U-bed 1. After at least 1 hour of He circulation at 473 K the columns are cooled down to room temperature and are ready for the next injection and the cycle begins again.

The process above describes the isotope separation of gas mixtures supplied from the IS system. In addition, the GC system can also be used to perform GC internal separation runs. When U-bed 1 contains enough interfractions from previous separation runs, the content of U-bed 1 is transferred to U-bed 2. By simply heating U-bed 2 the hydrogen gas mixture can be injected into the column via valves V4, V19 and V16 and displaced with eluant as discussed above. Internal GC runs have the advantage in that the IS system can be used at the same time for a different task and the interfractions do not need to be moved to the IS system for storage.

### **3. DESIGN OF THE JET GC SYSTEM**

#### **3.1 Mechanical structure**

The preparative GC system can be split into three main parts (outlined in Fig. 2):

*a) A cylindrical vessel (designated GC-KP):*

This vessel (overall dimensions: 1.2m diameter, 5m high), with many flanges in the upper part for the electrical connections for the various diagnostics, contains the 4 Pd filled columns and the pipe work which connects the inlet and outlet heads mounted on top of the columns with various ring manifolds plus a 500L reservoir.

*b) A rectangular box (designated GC-GL):*

This box (overall dimensions: 0.83m x 1.3m x 2.9m) houses the pumps, an 80 L reservoir, various valves, small volumes containing gas at the correct pressure for the reference sides of the up- and down-stream pressure regulators and pipe work not shown in the simplified flow diagram.

*c) An evacuated vessel (designated HISA):*

This vessel (overall dimensions: 0.66m diameter, 1m long) contains four JET U-beds. Identical HISA units are used in IS and PS and have been discussed previously [4].

The GC-KP vessel is separated into an upper and lower containment. The upper containment is filled with nitrogen to about 75 kPa and contains all process pipe work and diagnostics. The columns stand in tubes whose inner diameter is only 0.2 cm larger than the outer diameter of the column. The space between these tubes is made so small in order to achieve good heat transfer between them. The outer tubes are heated by oil circulated by a commercial oil heater. The oil is supplied to the outer tubes via metal flexible hoses located in the lower containment which is evacuated to avoid heat losses due to convection. There is no direct connection between the boundaries of the column and the oil system in order to minimise contamination of the oil due to permeation of tritium at higher temperatures. The columns can be heated to 573K for reconditioning, in most cases 473K is high enough for simple desorption of the eluant. During these heating cycles the columns are filled with protium containing only traces of tritium. Hence, tritium permeation of this system is of minor concern.

A further advantage of this design is that thermal expansion of the tubes causes almost no vertical movement of the inlet and outlet heads on top of the columns because both tubes expand simultaneously. As a consequence stress on the pipe work due to vertical movement of the column heads is drastically reduced.

Each column consists of two tubes of 2.7m length with an inner diameter of 3.4 cm. The lower ends are connected via a 0.63 cm outer diameter pipe.

The 500 L reservoir is located between the four columns in the lower containment and is used to check the inventory of the GC U-beds via (PVT)-measurements.

### 3.2 Diagnostics

Each column is equipped with diagnostics at inlet and outlet heads. A flow meter, down-stream pressure regulator, pressure gauge and a set of thermocouples are mounted to the inlet head, whilst the outlet head has thermopile, ionisation chamber, katharometer, pressure gauge, thermocouples, and an up-stream pressure regulator. The katharometer and ionisation chamber are mainly used for isotope identification and control of the valve switching.

Figure 3(a) shows a cross-section of the outlet head. The stainless steel block (4.4 x 4.4 x 29 cm<sup>3</sup>) functions as a manifold with a 0.4 cm diameter bore hole in the middle for the gas flow. This design achieves a very low dead volume of less than 30 cm<sup>3</sup>.

The cross section shows clearly the middle and outer electrodes of the ionisation chamber (IC) as well as the filaments of the katharometer (TCD). The outer diameter of the middle electrode and the distance between middle and outer electrode are both 0.4 cm.

Three of the four valves (V11, V12, V13, and V14 of Fig. 2) used to switch the gases exiting the column to different destinations can be seen at the top of Fig. 3(a). The fourth valve is not shown. The seats of these valves are machined into the stainless steel and the valve bodies containing the valve stem (commercially available product from Nupro Company) are welded to the block. The actuator of a further valve is seen behind the block near the katharometer

filaments. Via this valve which is not shown in the simplified flow diagram of Fig. 2 the column can be easily pumped empty by bypassing the down-stream and up-stream pressure regulators. The female Cajon connector on the left side accepts a pressure gauge which again is not shown in the simplified flow diagram.

The diagnostics mentioned above are described in more detail elsewhere [6]. The position of the inlet and outlet heads in the upper part of GC is shown in the engineering drawing of Fig. 3(b).

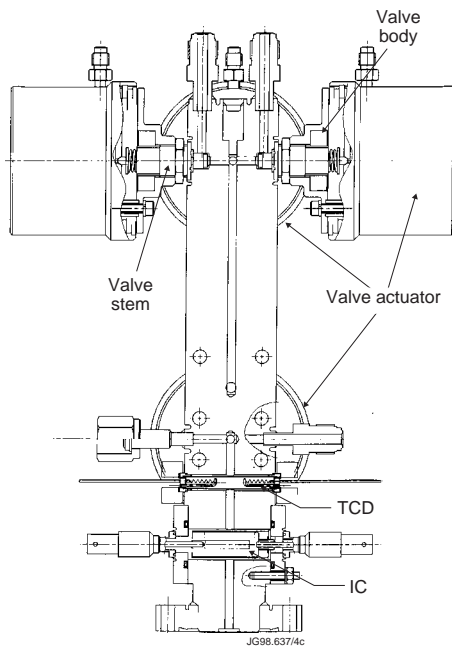


Fig. 3(a). Cross section of outlet head showing ionisation chamber (IC), katharometer (TCD), and valves.

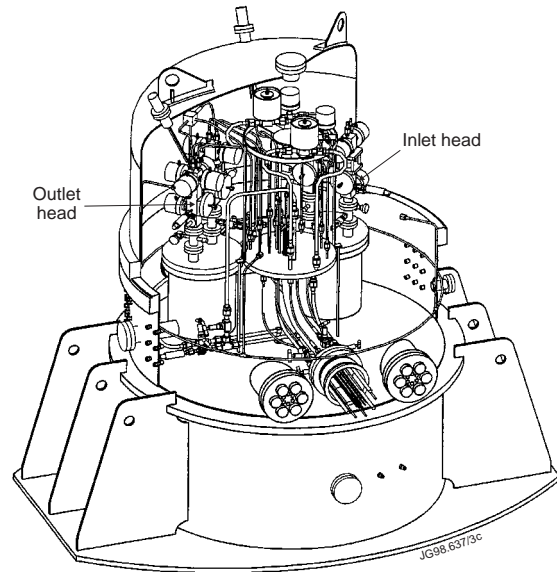


Fig. 3(b) Engineering drawing of upper part of GC showing the inlet and outlet heads of two columns and pipe connections.

### 3.3 Pd packing

The columns and the thermopiles were filled with 18 to 20 weight% palladium deposited on porous  $\alpha$ -Al<sub>2</sub>O<sub>3</sub>. 1.57 Pa m<sup>3</sup> of hydrogen can be absorbed in 1g of the Pd packing [11].

The protium and deuterium storage capacities of the four columns are between 13.4 and 14.53 kPa m<sup>3</sup> and 12.48 and 13.68 kPa m<sup>3</sup>, respectively, measured at 320 K and approximately 0.1 MPa. The higher absorption capacity of some columns is due to a slightly higher weight percentage of Pd in the packing material.

Palladium was chosen as isotope separation material because: it has a very large isotope separation factor; the oxide layers can be easily reduced in the presence of hydrogen; poisoning of Pd by other gases happens very rarely; it has a very high diffusion coefficient; the  $\alpha$  and  $\beta$  phases of PdQ<sub>x</sub> have the same lattice structure; and the equilibrium pressures at room temperature are in an easily accessible range.

### 3.4 Hardwired interlocks

Nineteen hardwired interlocks are installed for the GC system and are checked at regular intervals.

These interlocks turn off the heating power to the U-beds and close the inlet and outlet valves when temperatures higher than 823 K or pressures higher than 0.25 MPa are reached or the pressure in the HISA-vessel increases; they stop the pumps when the pressure in the 80 L reservoir is higher than 0.14 MPa; they switch off the power to the oil heater when the pressure in one of the Pd filled columns reaches values above 0.17 MPa; and they shut down the GC-KP or the GC-GL systems and turn off the valve actuator compressed air supply when tritium concentrations higher than  $4 \times 10^{10}$  Bq/m<sup>3</sup> are observed in the N<sub>2</sub> filled secondary containments or when the pressure in the upper dome of the GC-KP exceeds 0.12 MPa.

### 3.5 Passive safety features

The ultimate safety of the GC system operation is assured by passive safety features.

The 500 L reservoir in the lower containment of GC-KP serves also as an expansion volume if one of the four rupture discs (RD in Fig. 2) bursts due to overpressure (>0.3 MPa) in the Pd filled column caused e.g. by the heating of partly or fully loaded Pd with all column valves closed.

The upper secondary containment is connected via an up-stream pressure regulator to a 10 m<sup>3</sup> expansion tank. In case of a failure of the pipe work in the upper dome the pressure will rise. Then the pressure regulator opens and allows the gas to expand into the buffer tank.

The size of the lower containment was chosen such that even the expansion of the whole hydrogen content absorbed in the four columns would only lead to subatmospheric pressure within the containment.

### 3.6 Control system

The GC system is controlled by a Distributed Control System (DCS) with special micro-Distributed Control Interfaces (DCIs).

Two modes for operation were configured for GC: Single Mode Operation (SMO) where the operator performs all actions step by step via a keyboard and automatic mode where automatic sequences are run in the Distributed Control Unit (DCU) and the operator only answers a limited number of questions.

In a DCU the closing of a valve occurs within 2 to 3 seconds after a SMO command. This response time increases even further when an automatic sequence has to perform measurements and calculations and data are to be transferred between different DCUs.

To avoid unacceptable switching times special micro-DCIs were installed in the DCUs of the GC-system. The time the micro DCI needs to perform the necessary measurements and calculations for the correct valve switching takes less than 0.3 seconds. Thus, in most cases the valves operated by the micro-DCI will be switched in far less than 0.3 seconds.

## 4. COMMISSIONING OF THE GC SYSTEM

### 4.1 Non-active commissioning

The non-active commissioning was performed with various mixtures of protium and deuterium and a clear separation into pure streams of deuterium and protium with a very small interfraction was achieved [6].

### 4.2 Active commissioning

#### 4.2.1 Commissioning with trace tritium amounts

Trace tritium gas had been collected during the Preliminary Tritium Experiment (PTE1) at the end of 1991 [12] and was stored in four U-beds in the JET U-bed safe store. These U-beds were moved into the AGH-building and connected via a long 0.63 cm outer diameter tube to the process pipe work inside the Make-up box of the Analytical System [13]. The highest tritium concentration of the trace tritium before any enrichment was approximately 0.05 %. This low tritium concentration was high enough to test most tritium specific instruments for correct functioning. The protium and deuterium content in the residual gas mixture were 20% and 80%, respectively.

Due to the low trace tritium concentration the method of internal GC separation runs as discussed in Section 2.2 was slightly modified. The whole column was filled with the Q<sub>2</sub> gas mixture and no eluant gas (H<sub>2</sub>) was added. The injected gas mixture acted as its own displacement gas. In this way the amount of enriched tritium gas was maximised. A further advantage of this method was that improper functioning of the GC system or operator errors could not lead to mixing of trace tritium gas with inactive eluant gas and therefore further dilution of the trace tritium gas mixture was excluded. After a column was full and the enriched tritium peak absorbed into a U-bed, the gas content of the whole column was desorbed into the same U-bed. This yielded the same trace tritium gas mixture as before the injection and therefore separation runs under equal and easily comparable conditions could be performed.

These test separation runs allowed the first check of the process ionisation chambers and the connected electronics and also the selection of a suitable range. The process ionisation chambers are so small (active volume about 2.5 cm<sup>3</sup>) and are surrounded by stainless steel walls of such dimensions that even the strongest gamma sources available could not generate a measurable IC-current. Checking of the ICs before the tritium exposure would thus only have been possible with other radioactive gases.

Figure 4 shows signals of the GC diagnostics for the injected trace tritium gas mixture as a function of time. The tritium enrichment is most clearly seen by the ionisation chamber response which shows a very sharp increase at the front from helium to tritium. The maximum tritium concentration was estimated to be approximately 20% tritium in deuterium. At the same time the katharometer signal shows the characteristic change of signal response from helium to

deuterium. There is a small peak seen in the katharometer signal coinciding with the IC peak which is attributed to the enriched tritium in the deuterium gas.

The thermopile response is seen in the top part of Fig. 4. There is an arrangement of twelve thermocouples in two planes of six with the flow direction perpendicular to the planes. As long as helium is moved through the twelve thermocouples which make up the thermopile the signal is constant. A very sharp increase is seen when the first six thermocouples are heated by the exothermic heat generated by Pd absorbing Q2. After reaching a maximum the signal height decreases slowly due to the fact that the hydrogen absorption front is moving away from the first six thermocouples and slow cooling occurs. When the hydrogen absorption front hits the next six thermocouples of the thermopile which are 6 cm away from the first ones, a sharp decrease occurs because the signals of the two arrays of six thermocouples cancel when both arrays see the same temperature. The decreasing signal of the thermopile undershoots the baseline because in the meantime the first six thermocouples were cooled further. The time between the increase and decrease of the thermopile signal shows how fast the hydrogen front moves through the thermopile.

The ionisation chamber and katharometer respond slightly later than the thermopile because the gas has to pass through a small amount of Pd packing on top of the thermopile before reaching the other diagnostics, but the ionisation chamber and katharometer respond almost simultaneously because they are separated only by a volume of 1 cm<sup>3</sup>.

#### 4.2.2 Commissioning with tritium gas pre-enriched by the CD system

The Cryogenic Distillation (CD) system at JET was filled with 0.08 g trace tritium gas. The highest tritium concentration achieved with this low total inventory was about 4 % of tritium in deuterium at the bottom of column 3 of the CD system. This low tritium concentration is not surprising because the CD system was designed to produce the best tritium product purity with a total inventory of about 33 g tritium [5].

This pre-enriched tritium was injected into each of the 4 columns of the GC system and displaced with “pure” eluant gas (H<sub>2</sub>) from one of the U-beds 3 or 4 of Fig. 2. In total about 2.4 kPa m<sup>3</sup> of a deuterium-tritium mixture were injected into the columns with an average tritium concentration of approximately 1.4%.

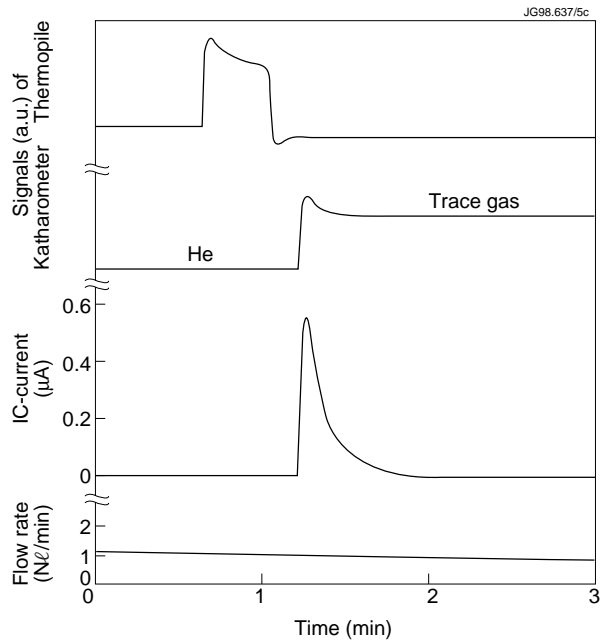


Fig. 4. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded during a separation run with the trace tritium gas.



Figure 5 shows the responses of the GC diagnostics. The enrichment of tritium is most clearly seen by the IC-signal (please note that the current range of Fig. 5 is one order of magnitude higher than in Fig. 4), but now also the katharometer signal shows a clear tritium peak. The tritium concentration in the peak is about 90% in deuterium. No tritium plateau was achieved because 0.08 g (800 Ci) tritium in a single GC column are not enough to enrich tritium to 100%. This is principally because the column diameter chosen for the maximum feedthrough of the GC system is too large to enrich 0.08g of tritium to 100%. Assuming homogeneous hydrogen charging of the Pd to 70 atomic percent the 0.08 g of tritium is already absorbed in the Pd packing at a height of 1.2 cm. Because gas is also needed to build the interfractions, insufficient gas is left to reach 100% purity.

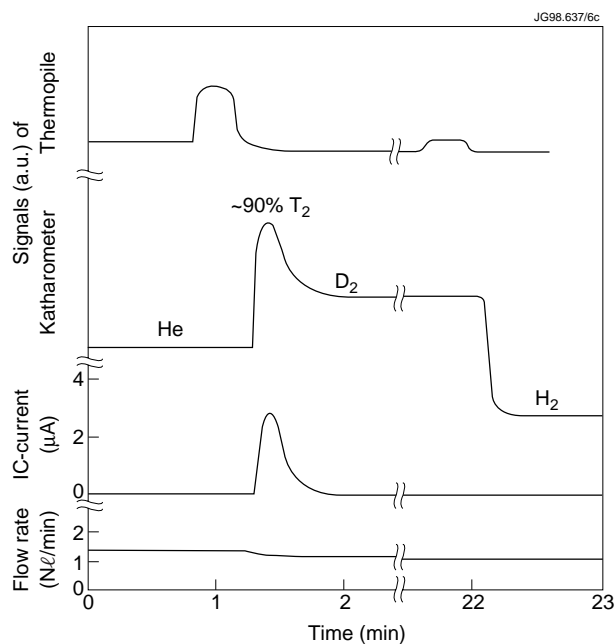


Fig. 5. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded during a separation run with the trace tritium gas enriched by the Cryogenic Distillation system. To emphasise the interfractions the constant signals between the 2<sup>nd</sup> and 22<sup>nd</sup> minutes are not shown.

The katharometer signal reacts very clearly on the various gases: helium, 90% tritium in deuterium, deuterium and protium as indicated next to the horizontal lines. The transitions of helium to tritium and of deuterium to protium are very sharp. As a consequence the interfractions are very small. This requires very fast and accurate switching of the valves for cutting the products and interfractions.

The top trace of Fig. 5 shows again the response of the thermopile. The first peak is caused by the absorption of the tritium-deuterium mixture in the fully regenerated palladium as explained above. The second peak which occurs before the change of deuterium to protium seen by the katharometer is much smaller than the first one and is caused by the isotopic difference of the absorption heat between deuterium and protium. Protium (as explained in Section 2.1) has a higher affinity to palladium than deuterium, thus more energy (heat) is released by the absorption of protium than by deuterium. When the deuterium in the palladium is finally replaced by protium this leads to the small signal in the thermopile seen just before the 22<sup>nd</sup> minute in Fig. 5.

These tritium experiments demonstrated that the JET GC system was capable of separating hydrogen isotope mixtures of the six molecules into tritium, deuterium and protium.

Further commissioning with larger tritium amounts was however needed to demonstrate the correct switching of the valves and the achievement of the required purity of the tritium product.

#### 4.2.3 Commissioning with gas mixtures of 1 g tritium

1g tritium was mixed with various amounts of deuterium. These mixtures were injected into the four GC columns and displaced by eluant gas ( $H_2$ ). The result of a typical separation is shown in Fig. 6.

Long and flat plateaux of the enriched tritium were seen for the first time by the ionisation chamber and katharometer. This clearly demonstrated that the tritium amount used during the trace tritium phase had not been large enough to achieve 100% 'pure' tritium.

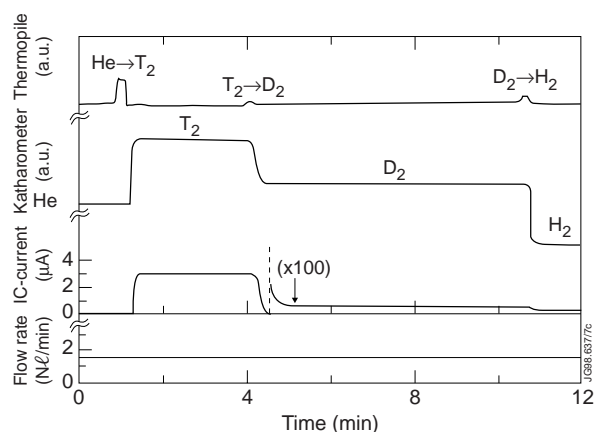


Fig. 6. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded during a separation run with an hydrogen isotope mixture containing approximately 1 g tritium.

The observed interfractions seen in Fig. 6 are very sharp, the transition from tritium to deuterium being less sharp than the transition from deuterium to protium. This is an expected result from the fact that the isotope separation factor between tritium and deuterium is smaller than that between deuterium and protium which means that it is easier to separate deuterium and protium than tritium and deuterium. Samples taken during the separation runs by opening valve V14 (Fig. 2) to the analytical equipment revealed tritium purity higher than 99.8 % demonstrating far better performance than the minimum design requirement of 98 % tritium purity.

The IC signal change from  $10^{-6}$  to  $10^{-8}$  A range reveals that the tritium concentration in the deuterium product quickly reaches levels below 0.2 %.

The thermopile response is again seen in the top trace of the figure. This time three peaks are clearly seen, the first large one is due to the transition of helium to tritium, the second one, the smallest, is due to the small isotopic difference in the absorption heats between tritium and deuterium and the third one, slightly larger, is due to the larger isotopic difference between deuterium and protium.

A few separation runs were necessary to optimise the automatic switching of the valves. Special micro-DCIs are used to achieve fast switching of valves V12, V13 and V14 in Fig. 2. These micro DCIs are enabled by the first peak of the thermopile signal. This guarantees that automatic switching of the product valves V12, V13 and the column return valve V11 starts only just before the separated products exit the columns. The valve V12 (to allow tritium product to enter the 100 L reservoir in Product Storage (PS)) opens only automatically when the micro DCI is enabled and the katharometer, ionisation chamber signals and the slope of the plateaux are between pre-defined values. The valve only closes again if all conditions specified for further interfractions or products are fulfilled. All these pre-defined parameters can be changed during



a separation run by an operator. In this way the operator can modify the parameter values to achieve very pure products by adjusting the parameters very closely to the experimentally observed values because the katharometer is slightly flow sensitive and the ionisation current depends on the pressure in the columns. Normally the parameters are set so that the interfractions  $\text{He}+\text{T}_2$ ,  $\text{T}_2+\text{DT}+\text{D}_2$  and  $\text{D}_2+\text{DH}+\text{H}_2$  are sent via the open column return valve to U-bed 1 for absorption, and pure  $\text{T}_2$  and pure  $\text{D}_2$  to the 100 L and 700 L reservoirs of PS, respectively. However the parameters can be easily changed to send e.g. the interfraction  $\text{He}+\text{T}_2$  and the product  $\text{T}_2$  to the 100 L reservoir, because tritium can be separated from helium by the simple absorption of tritium in uranium.

During the commissioning phase of the GC system with gas mixtures containing 1 g tritium eleven separation runs were performed and 5 g of tritium processed in the four columns of GC.

## **5. USE OF PREPARATIVE GC SYSTEM**

### **5.1 Comparison between CD and GC at JET**

The JET CD system can produce far purer protium and deuterium than the GC system [5]. Tritium can be removed so efficiently from the protium and deuterium product streams of CD that their direct discharge into the environment is possible. Tritium levels far lower than 1ppm can be achieved in the protium and deuterium streams, whereas the tritium levels of deuterium and of the eluant in the GC system are approximately 0.1% and so far too high to be discharged in large quantities.

The JET CD system is designed to produce very pure tritium only with an inventory of about 33 g of tritium. With only 0.08 g of tritium present in the CD system the maximum tritium concentration observed was approximately 4%. In comparison, with the same gas mixture, an enrichment to over 90% tritium was achieved by means of GC.

The JET CD system needs 2-3 days for cooling down from room temperature to 19 K, the total gas amount to fill CD is approximately  $176 \text{ kPa m}^3$  and the correct operation of CD with its many control loops is not simple. In contrast, the GC system is almost immediately ready for separation and its operation is much simpler.

The two hydrogen isotope separation systems, GC and CD, complement each other which can also be seen by the interaction of these two systems discussed in Section 5.3.

### *5.2 Use of the GC system during DTE1 and experiences gained*

More than 160 separation runs were performed with the preparative GC system during DTE1 and the consequent clean-up phase up to the start of the remote tile exchange. Tritium was produced for operational use no fewer than 40 times. 'Pure' tritium as well as 'pure' deuterium were generated in 24 runs. Only tritium was produced in 16 runs. Under these conditions the enriched deuterium product was added to the  $\text{T}_2+\text{TD}+\text{D}_2$  interfraction. 105 separation runs were

performed to pre-enrich tritium from low tritium-hydrogen gas mixtures and to cut deuterium and 15 runs to clean-up the eluant gas protium.

Approximately  $470 \text{ kPa m}^3$  of hydrogen isotope mixtures with  $64.6 \text{ kPa m}^3$  (160 g) of tritium were injected and processed in the columns of the JET GC system. The recovered tritium and deuterium amounts were 96 g and 440 g, respectively. This demonstrates again that large amounts of tritium had to be re-cycled through the GC-columns to achieve the final enrichment. The largest tritium amount separated in a column was 8 g. These numbers are to be compared with the total amount of tritium of 20 g which was purchased and available for DTE1. A large fraction of these 20 grams was retained in the torus, especially in carbon tiles and flakes, in a not easily releasable form. 6.2 g of tritium remained in the torus at the beginning of the Remote Tile Exchange [9].

Tritium production runs with the GC system were preferably done with large tritium amounts because correct switching of the three valves at the column outlet head was less important as more tritium was present, and also in automatic mode pre-selected parameters could be changed if required.

Figure 7 shows the responses of the various diagnostics of a GC separation run where  $3.3 \text{ kPa m}^3$  of a tritium-deuterium mixture were injected. The three peaks of the thermopile are clearly visible. The flow rate shows small fluctuations caused by manual control of the power to the U-bed supplying the eluant gas. These fluctuations cause minor changes in the katharometer signal as the katharometer reacts with a decrease of its signal on increased flow rate due to stronger cooling. The katharometer signal of helium does not show these changes because the flow rate of the displaced helium leaving the column is very small. The ionisation chamber signal shows a perfect plateau during the whole tritium production time which shows that the up-stream pressure regulator controls the pressure very well. The parameters for the automatic run were chosen that the He+T<sub>2</sub> interfraction and the 'pure' tritium were moved to the 100 L reservoir in PS (see Fig. 2) but the interfraction T<sub>2</sub>+TD+D<sub>2</sub>, 'pure' D<sub>2</sub> and interfraction D<sub>2</sub>+DH+H<sub>2</sub> were transferred to U-bed 1 of GC. In this way the maximum amount of tritium is collected and no deuterium product is transferred to the 700 L reservoir.

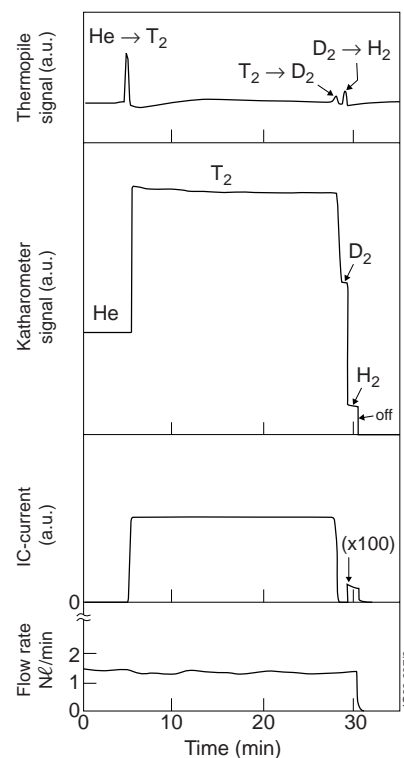


Fig. 7. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded during a separation run with an hydrogen isotope mixture containing approximately 8 g tritium.

Normally, the IC is switched into the  $10^{-8}$  A range when the tritium concentration is below 1 % tritium in deuterium (see Fig. 6). In Fig. 7 the switching occurs due to the chosen parameters when protium is recognised by the diagnostics. At the end of the separation run the IC is switched back into the  $10^{-6}$  A range. Helium also transferred to the 100L reservoir will not be absorbed by the uranium and can be pumped away.

Table 1 lists gas compositions of the  $D_2$  and  $T_2$  produced by GC. The values given are obtained after absorption and desorption in a JET U-bed. The analysis was performed with the JET analytical gas chromatographic system [13].

Table 1: Required and achieved purity of deuterium and tritium

<b>REQUIRED GAS PURITY</b>			
	H / %	D / %	T / %
$D_2$ gas	<1	>98	<0.25
$T_2$ gas	<0.5	<1	>98
<b>ACHIEVED GAS PURITY</b>			
	H / %	D / %	T / %
$D_2$ gas	0.17	99.70	0.13
$T_2$ gas	0.03	0.01	99.96

The tritium and deuterium concentration in the eluant can increase due to contamination in the column and in the pipe work. Part of the eluant injected during a separation run is cleaned-up during the run which keeps the contamination at low levels. A few runs were made to study if the GC system can clean-up the eluant gas. Eluant gas was injected into the whole column in the same way as for the trace tritium gas mixture (see Section 4.2.1). Similar responses as shown in Fig. 4 were observed for the eluant gas which means that the GC system can also be used to purify its own eluant gas.

The GC system contains four columns filled with Pd packing. The original intention was to inject the gas mixture to be separated into these columns simultaneously. But due to the limited heating power of the U-beds of only 2 kW high flow rates in all four columns could not be achieved at the same time. In addition, due to the down-stream regulators (see Section 7.1) any change of a flow in one column influences the flows in the other columns. Those changes cannot be tolerated during the time when the products are extracted because they can cause rapid changes of the katharometer signal which are interpreted wrongly by the automatic sequence as changes of the product purity. Thus, during DTE1 the columns were used sequentially.

When enough gas for the injection into 3 or 4 columns was available, three or four separation runs were performed during normal working hours. The regeneration was in most cases performed by the night shift. A typical tritium and deuterium production run lasts about 1.5 hours. A further hour is needed to heat the U-beds. The regeneration of the columns, which

includes heating of the column to desorb hydrogen, circulation of the helium to remove hydrogen by a scavenging U-bed and cooling of the column to room temperature, takes about 4 to 6 hours. Longer and more thorough removal of the protium from the Pd packing lowers the final protium content in the tritium and deuterium product.

The 80 L reservoir shown in Fig. 2 is filled with helium to a pressure of about 0.13 MPa. The helium is used to fill the columns to achieve a sharp front between He and T<sub>2</sub> and to remove the partial pressure of hydrogen above the Pd packing by simple helium circulation through a cold U-bed. During GC separation runs hydrogen can reach the 80 L reservoir when the efficiency of hydrogen absorption in the cold U-bed is not 100%. The presence of tritium is clearly indicated by an ionisation chamber in the 80 L volume. If hydrogen is expected and/or tritium seen by the ionisation chamber, it is good practice to circulate the helium-hydrogen mixture by means of metal bellows pump through a cold U-bed before any injection into the Pd filled columns.

No noticeable increase of the flow resistance through the columns could be observed, a clear indication of the good quality of the Pd packing used.

The whole GC-system worked very reliably during the full tritium, the DTE- and the subsequent clean-up phase. No opening of any secondary containments was necessary during the whole time.

The only equipment which needed replacement during DTE1 was the oil circulation pump. This was a conventional piece of equipment. The oil was verified not to contain any tritium (as expected from the design). The change of the pump was a straightforward undertaking. No delay of torus operations was caused by this failure.

### 5.3 Use of GC and CD

Normally gas mixtures with tritium concentrations > 0.2 % were sent to GC for manual pre-enrichment separation and those with tritium concentrations < 0.2 % to the CD system.

Many manual separation runs were performed with the GC system to enrich tritium and to cut deuterium into the 700 L reservoir. After injection into a column the gas mixture was displaced by eluant, the pre-enriched tritium with the interfractions He+T<sub>2</sub>, T<sub>2</sub>+TD+D<sub>2</sub> was absorbed in U-bed 1 (see Fig. 2) and deuterium was cut into the 700 L reservoir of PS. Protium present in the gas mixture was added to the eluant gas. The interfractions He+T<sub>2</sub>, T<sub>2</sub>+TD+D<sub>2</sub> and D<sub>2</sub>+DH+H<sub>2</sub> were collected in U-bed 1 until enough tritium was available to perform an automatic run for T<sub>2</sub> and D<sub>2</sub> production. Those pre-enrichment runs were performed manually as perfect cutting was not necessary and often no tritium plateau was achieved due to a very large deuterium fraction.

Tritium pre-enriched by CD was moved to GC and enriched to almost 100 % in GC whereas protium and deuterium from the top of 'column 1' of CD [5] were mainly discharged to stack subject to daily discharge limit. Deuterium from the top of 'column 3' of CD was transferred to U-beds of PS-D<sub>2</sub> for further re-use. The deuterium product of CD was preferred to the D<sub>2</sub> prod-

uct of GC because its protium and tritium levels were much lower, although  $D_2$  product of GC fulfilled the requirements of the  $D_2$  users at JET.

The deuterium product of GC system was sent to the 700 L reservoir of PS, then absorbed in one of the PS- $D_2$  U-beds, moved from the PS- $D_2$  U-bed to one of the IS-U-beds and then injected into the CD system for further detritiation and subsequent discharge.

#### 5.4 Interfractions $He+T_2$ , $T_2+TD+D_2$ and $D_2+DT+H_2$

Figures 8, 9, and 10 present the responses of thermopile, katharometer, ionisation chamber and flow meter as a function of time for the interfractions  $He+T_2$ ,  $T_2+TD+D_2$  and  $D_2+DT+H_2$ , respectively.

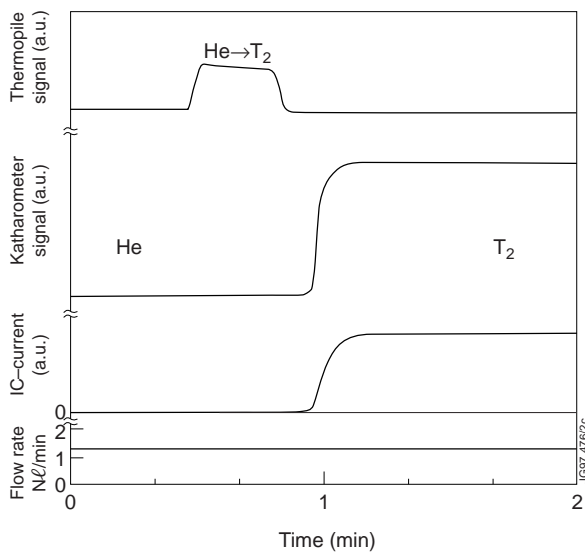


Fig. 8. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded for the transition between helium and tritium.

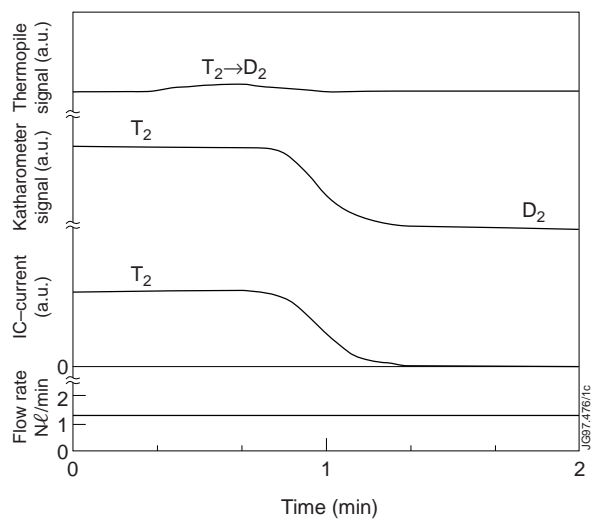


Fig. 9. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded for the transition between tritium and deuterium.

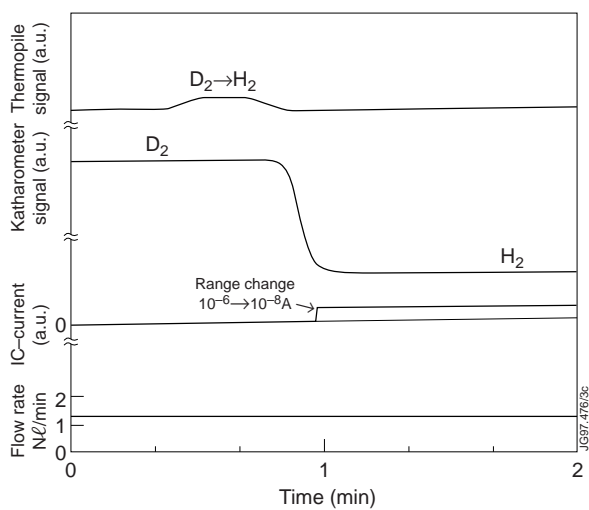


Fig. 10. Signals of thermopile, katharometer, ionisation chamber (IC) and flow meter recorded for the transition between deuterium and protium.

The switching from pure tritium to deuterium occurs in 40 sec and from pure deuterium to protium in 12 seconds. The fastest change occurs for helium to tritium which happens in 10 seconds and reflects the very small degree of mixing in the column and the fast responses of the ionisation chamber and of the katharometer.

The size of the  $He+T_2$ ,  $T_2+TD+D_2$  and of  $D_2+DH+H_2$  interfractions is approximately 0.3 NI, 0.9 NI and 0.4 NI, respectively.

This again explains why with the trace tritium gas containing 0.08g pure tritium (0.32

NI), a pure tritium stream could not be created because the tritium was needed to create the interfractions.

The katharometer and ionisation chamber responses seem to be equally fast. Both detectors are built so that small gas streams are continuously flowing through them.

The change from tritium to deuterium requires a longer time than from deuterium to protium, a clear reflection of the different isotope separation factors.

The switching time between deuterium and protium is almost the same as between helium and tritium. This means that the separation quality must be even better than the plot indicates because a large fraction of the switch time is caused by mixing/diffusion and/or by the response times of the diagnostics. Shorter switch times can be achieved by higher flows with the disadvantage that the valve switching must be done very fast.

It is important to note that the size of the interfractions observed in a GC separation run using Pd as packing material is fully independent of the amount of mixed hydrogen molecules HD+HT+DT in the gas mixture due to the fact that hydrogen has to dissociate before its absorption by Pd. The mixed hydrogen molecules are split and contribute finally to the main products T<sub>2</sub>, D<sub>2</sub>, and H<sub>2</sub>. This behaviour is very different from the separation achieved with the analytical gas chromatographic system [7] where the hydrogen molecules are not split and are separated due to different retention times in the column used.

## 6. INFLUENCES ON HYDROGEN ISOTOPE SEPARATION QUALITY

### 6.1 Flow rate

Good hydrogen isotope separation is achieved with gas flows > 1.2 NI/min in the JET GC system. The recommended flow is 3 NI/min. The influence of flowrate on isotope separation is shown in Fig. 11.

No sharp profiles are found with gas flows < 0.9 NI/min as seen in the Fig. 11a. The IC and katharometer already show a significant increase in signal before the main peak at 2.5 minutes which is not seen in the other curves obtained with higher flow rates. This precursor behaviour is attributed to interdiffusion of He and Q<sub>2</sub>.

This interdiffusion will also affect the separation between the hydrogen isotopes and must be reduced by the choice of sufficient hydrogen flows through the column.

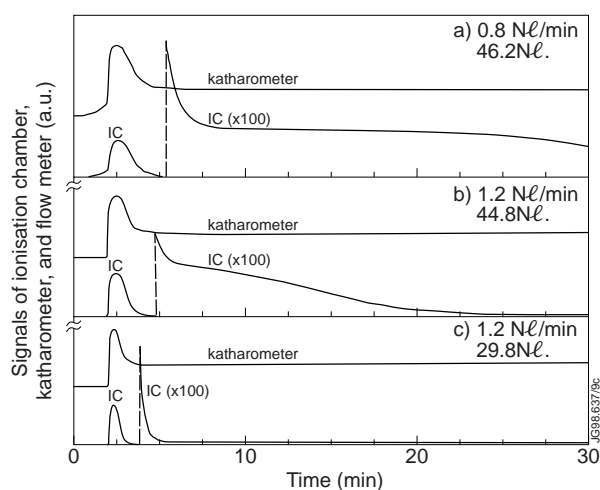


Fig. 11. Signals of katharometer, ionisation chamber (IC) and flow meter recorded for various flow rates and various injected gas amounts: a) 0.8 NI/min and 46.2 NI, b) 1.2 NI/min and 44.8 NI and c) 1.2 NI/min and 29.8 NI.



## 6.2 Size of gas mixture batches to be separated

There is a maximum batch size for the injected hydrogen isotope mixtures above which poor separation behaviour sets in. The gas batch size to be separated needs to be tailored to the amount of Pd packing in the column and the composition in the mixture.

Figure 11 shows the response of the outlet diagnostics after the injection of different gas amounts (11a: 46.2 NI, 11b: 44.8NI, 11c: 29.8NI), but of the same  $Q_2$  composition, into two columns. Note that the IC-range sensitivity was increased by a factor 100 during runs. Only the IC curve in graph (c) decreases rapidly after the main tritium peak whereas the two other IC signals approach the zero line very slowly. In the two upper figures too much gas was injected into the column. Tritium injected in the latter part of the gas batch could not be moved by the enrichment process to the main tritium peak. This means contamination of the deuterium with too high levels of tritium and therefore poor hydrogen isotope separation.

Clear isotope separation is obtained for any gas mixture when the injected gas amount is smaller than a quarter of the capacity of the Pd packing. The disadvantage of the injection of a too small gas amounts with low tritium concentrations is that the enriched tritium concentration does not reach 100% as already noted.

The time-integrated flow rate measured at the column inlet gives the injected  $Q_2$  gas amount. In addition, two 0.05 cm diameter shielded thermocouples are mounted in the 0.1 cm gap between the oil and column tube and are fixed to the outer surface of the Pd filled column at a quarter way along its length to detect the temperature increase upon arrival of the  $Q_2$  front. They determine when the column is filled to a quarter.

Figure 12 shows a typical temperature response of this batch-fill monitor. The data originate from the same separation run as the ones shown in Fig.7. The first sharp temperature rise is due to the absorption of tritium in Pd, the second smaller one due to the replacement of tritium by  $D_2$ /protium gas. Afterwards the temperature falls slowly to the value of the surrounding oil.

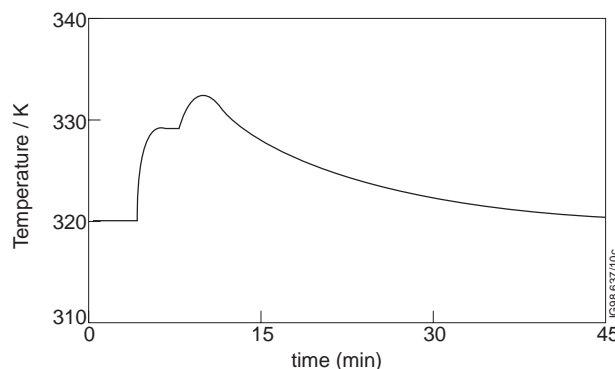


Fig. 12. Temperature response of the batch-fill monitor.

## 6.3 Desorption of protium from the Pd packing

The displacement protium gas and low levels of deuterium and tritium absorbed in Pd are to be removed as completely as possible from the Pd packing before the injection of a new gas mixture. Undesorbed protium, deuterium and tritium will lead to contamination of the product gases  $T_2$  and  $D_2$ , and of the eluant gas, especially if absorbed near to the column outlet.

In the JET GC system the Pd packing can be heated up to 573 K, although in most cases only 473 K was used. During the heating the liberated hydrogen is absorbed by a cold U-bed.

Finally, helium is circulated through the heated column and a U-bed at room temperature (see Fig 2, V5, V6, V16, V11, V1, V2, V5 are open) to generate very low hydrogen equilibrium pressures and therefore low hydrogen concentrations in the Pd and to scavenge desorbed hydrogen from helium before its re-injection into the column.

#### **6.4 Temperature of Pd packing during separation**

The hydrogen isotope separation factor decreases with increasing temperature.

Most separation runs at JET were performed at 320 K. In the design phase it was decided that cooling of the columns to temperature below room temperature - although beneficial with respect to separation behaviour - did not warrant the extra equipment expense. The temperature of 320 K was chosen to be independent of seasonal temperature fluctuations.

The exothermic absorption heat leads to an additional temperature rise of the Pd packing (see e.g. Fig. 12).

#### **6.5 Dead volumes at column outlet**

The size of dead volumes between the Pd packing and the valves used for switching the products should be minimised because mixing of the separated hydrogen products can occur due to temporary storage of the gas followed by slow exchange. Furthermore the volume between the diagnostics and the valves should be small for correct timely switching. Due to these reasons no flow meter was installed in the column outlet head despite the good diagnostic reasons for doing so (see Section 7.2).

#### **6.6 Clean-up of the eluant gas**

Deuterium and/or tritium contamination of the eluant gas occurs with time. Under normal conditions part of the eluant gas is cleaned up during well performed separation runs.

Eluant gas diluted by tritium and/or deuterium can be cleaned up especially by filling the whole column with the eluant and cutting the enriched tritium and/or deuterium peaks. These separation runs correspond to the ones discussed in Section 4.2.1 for trace tritium.

### **7. RECOMMENDATIONS OF IMPROVEMENTS FOR FUTURE SIMILAR GC SYSTEMS**

#### **7.1 Down stream pressure regulators**

Mechanical down-stream and up-stream pressure regulators [14] are installed at the inlet and outlet of the columns (see PRD and PRU in Fig. 2). The up-stream pressure regulator (PRU) is necessary to keep the pressure in the column above 90 kPa because gases from the column are either absorbed in U-beds or compressed by a Normetex pump and a metal bellows pump in



series into a 80 L reservoir. The hope was that the flows through the columns could be controlled by the pressure difference between the column inlet and outlet and by the flow resistance of the Pd packing. The required pressure difference can be adjusted by changing the reference pressure of the down-stream pressure regulator.

At the start of a separation run the column return valve V11 is opened. The helium pressure in the column is determined by the PRU. This pressure is below the controlled pressure of the regulator PRD. Therefore the pressure regulator PRD is open. When the column inlet valve is opened for hydrogen injection large flows are observed because the PRD is fully opened and the hydrogen entering the column is immediately absorbed by the Pd packing at its low equilibrium pressure and only a negligible pressure drop is created along the short flow path even with very high flows. This happens also with a helium filled column. The PRD stays fully open and does not control the flow.

The flow into the column is not controlled by the two pressure regulators so long as the pressure drop of the hydrogen gas flowing through the Pd packing is smaller than the pressure difference between the PRD and PRU. During this time the flow is dependent on the pressure at the column inlet (made by the U-bed supplying the hydrogen) and the flow resistance of the Pd packing. Even under the assumption that the supplying U-bed pressure is kept constant the flow through the column would constantly decrease due to the increasing flow resistance caused by a longer passage of the hydrogen through the packing until it is absorbed and by a temperature increase of the packing material due to the absorption heat and by expansion of the Pd during absorption. The down-stream pressure regulator PRD only starts to control the pressure at the inlet of the column when the pressure drop caused by the flow is higher than the pressure difference between the two pressure regulators. If at this moment the column is not completely filled with hydrogen, the flow will continue to decrease due to the increasing flow resistance.

All separation runs showed that flow rate changes between 1.2 and 10 NI/min had no easily observable effects on the separation quality. High flow rates (higher than 10 NI/min) especially after first opening to the supplying U-beds were often observed. Normal filling was done with flow rates around 3 to 5 NI/min (the flow rates were reduced to approximately 1.5 NI/min only during product extraction for the purpose of exact valve switching). The flow rate of a gas mixture to be separated can become very low when the U-bed is to be emptied as far as possible. Sometimes the eluant was not added immediately after the gas mixture injection. None of this seemed to have any noticeable influence on the separation quality. Therefore this displacement technique is considered to be very robust and reliable.

When T<sub>2</sub> od D<sub>2</sub> products are expected at the column exit, a constant flow of hydrogen is an important precondition for the correct interpretation of the katharometer signal because it depends on the flow. A special katharometer was developed which minimised this flow dependence [6].

A fairly constant hydrogen flow through the Pd packing is achieved at JET by controlling the power to the U-beds supplying the hydrogen isotope mixture and the eluant gas and by keeping the flow rates such that the created pressure drop is smaller than the pressure difference between the down-stream and up-stream pressure regulators. This means that during a separation run the PRD is most of the time open.

The pressure regulator PRD is very useful when helium is circulated through the column to remove residual eluant gas from the Pd packing, when the pressure drop caused by the flow in the column is higher than the pressure difference between the regulators and when the Pd packing is fully empty or fully saturated with hydrogen. Under these conditions constant and reproducible flows are obtained with the down- and up-stream pressure regulators.

To improve this system the proposed instrument replacing the down-stream pressure regulator would be a flow controller. At the time of design and construction of the GC system all commercially available flow controllers were partially sealed with O-rings. This disallowed their use as no O-rings in primary tritium containments were permitted at JET. Fully metal sealed flow controllers were not available at that time.

## **7.2 Flow meter**

Flow meters capable of measuring flows up to 10 NI/minute are mounted at the inlet of the column to measure the gases injected into the column.

The amount of tritium and deuterium product is measured in the 100 L and 700 L reservoirs of PS respectively by (p, V, T, c) determination. The size of the interfractions and the flow rate of the helium is unknown because no flow meter is used at the outlet of the columns.

A flow meter installed at the column exit would be very useful to determine the size of these interfractions and the flow rate of helium. This is necessary because the flows measured at the inlet of the column can be very different from the ones at the outlet. For example during the whole time of addition of the hydrogen gas mixture to the Pd packing a large flow is measured at the inlet whereas the flow at the outlet is only due to the small amounts of displaced helium. When the first hydrogen products leave the column the flow measured at the inlet is not the same as the flow at the outlet because the Pd packing warmed up by the exothermic heat of absorption is slowly cooling and further absorption of eluant gas occurs.

## **7.3 Exchange of helium by nitrogen in upper dome of GC-KP**

The upper dome of the GC-KP system was filled with helium to achieve the best thermal contact between the oil filled tubes and the columns. Furthermore, tritium potentially leaked or permeated into the secondary containment could be very simply re-gained by circulation of the helium through cold U-beds.

The flow meters employed are based on the principle that a symmetrical temperature profile without flow is modified into an asymmetrical shape by a gas stream. The measured asym-

metry gives the unknown flow. These flow meters were found to react sensitively on the atmosphere surrounding the instrument.

Normally these instruments are calibrated in a surrounding atmosphere of air and give satisfactory results when used in air.

Flow meters located in the helium atmosphere of the upper containment of the GC-KP system were found to give such unreasonable flows that the helium had to be exchanged with nitrogen which reduced the thermal contact between the oil filled tubes and the columns.

The only other possibilities would have been to re-calibrate the flow meters in a helium atmosphere or to exchange the flow meters with another type.

## **8. SAFETY**

No tritium leaks were observed in the various secondary containments of the GC system with the large JET ionisation chamber [15]. Parts of these containments are non-purged, ultra leak tight volumes which means that any possible leak was integrated over the whole time of DTE1.

High standards were applied during the design and construction phase: design generally in agreement with ASME VIII, Div.1, and ASME B31.3, manufacture and assembly of the equipment in 'clean' rooms, use of only tritium compatible material, an all metal construction of the primary and secondary containments, stringent leak test procedures with a final global test requirement of no leak indication in the  $10^{-10}$  Pa m<sup>3</sup>/s range, etc.

## **9. CONCLUSIONS**

The preparative GC system at JET was commissioned successfully with tritium and performed very well during the DTE and subsequent clean-up phases.

The GC-system was used for more than 160 tritium hydrogen isotope separation runs and functioned very reliably. 160 g of tritium passed through the columns. Good product quality for tritium and deuterium was obtained for flows between 1.2 and 10 Nl/min and temperatures between room temperature and 340 K.

The GC-system was used to pre-enrich tritium and to produce very high tritium concentrations ( $\approx 99.9\%$ ). The tritium content in the eluant and in the helium gas could be kept at low levels ( $< 0.1\%$ ).

No tritium leaks from primary into secondary containments or into atmosphere were observed.

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