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# Gas Recovery System for the First JET Tritium Experiment

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# ABSTRACT.

Gas recovery during the first JET Tritium Experiment was mainly based on the use of a cryopump designed earlier for collection of torus exhaust gas. This cryopump system was extended to include collection of gas from the neutral injectors and to provide sampling and diagnostic facilities such as an ionisation chamber for on-line tritium accounting, a mass spectrometer for gas analysis and a sampling manifold for off-site tritium accounting. The cryopump was backed by a collection system comprising a reservoir, recirculation pump and four uranium beds originally designed for the JET Active Gas Handling System.

During operation of the system from start of the experiment on 04.11.91 until 20.12.91 a total of 1450 barl of gas with a total tritium content of  $4.3 \times 10^{13}$  Bq (1162 Curies) were collected.

A small exhaust detritiation system was installed to reduce emissions in case of accidents such as major torus or neutral injector air or water leaks. As no major problems were encountered during the experiment, it was not used.

# Introduction

The main technical aspects of the first JET tritium experiment are covered by M. Huguet et al [3]. The very stringent time schedule from decision to execution of the experiment made it necessary to make full use of existing equipment and to assemble it into a system fit for the purpose. This was successfully achieved by combining an existing cryopump with components developed for the JET Active Gas Handling System.

# **1** Overall System Description

The overall system layout is schematically given in Figure 1. Gas arriving from torus and neutral injectors passes through an inlet manifold which contains various diagnostic elements, such as

- ionisation chamber )
   ) for on-line analysis
   mass spectrometer )
- sampling manifold for off-site analysis.

The gas is then transferred via a cryopump into the receiving reservoir of the gas storage system. This reservoir (calibrated volume of  $345 \ell$ ) is used to measure the exact batch size (PVT) and the tritium content with an ionisation chamber. Subsequently the gas is stored permanently on uranium beds.

Gases not absorbed in this process (helium, argon, nitrogen, in some cases also hydrocarbons with low specific activity) are discharged via the gas disposal system to stack.

# 1.1 <u>Inlet manifold</u> - details and operation

A detailed schematic of the inlet manifold is given in Figure 2. Before start of the experiment, the system was assembled and fully leak tested to meet the criteria of JET Tritium Class 1 equipment: no detectable leak on a helium leak detector in the most sensitive range of  $10 \times 10^{-10}$  mb/s<sup>-1</sup> full scale. This can only be achieved by using components fully compatible for Ultra High Vacuum (UHV) applications, i.e. exclusive use of stainless steel components with either welding or all-metal gaskets or (for electrical feedthroughs) metal-ceramic joints. For internal seals (valve seats), organic gaskets were permitted: Viton gaskets in large angle valves and Vespel stem tips in small sample valves.

Gas arriving from the torus is typically pumped as follows: once the system is fully evacuated, all valves are closed with the exception of V3 (torus crown, ie vacuum duct from torus high vacuum pumps, to inlet manifold). After a JET pulse, the torus turbopumps exhaust into the evacuated torus crown and inlet manifold for a fixed duration (typically 10 minutes). The pressure rise measured on P1 and P3 together with the known torus crown volume permits quantitative assessment of torus gas release. Tritium concentration can be measured by admitting gas to the ionisation chamber IC1 via V9. Tritium accounting in detail is described by G. Saibene et al [4].

The residual gas composition is monitored with the mass spectrometer with the main emphasis on potential air leak detection which might create an explosion hazard of hydrogen-air mixtures after compression with the tubular cryopump (Cold Finger). During a series of JET pulses, at

this stage also pre-evacuated sample bottles were filled for off-site tritium concentration analysis [5].

Subsequently, the torus crown is evacuated by opening V1 to the Cold Finger until start of the next JET pulse, where the above described cycle is repeated.

For pumping of neutral beam injectors, the procedure is similar: the neutral beam injector boxes (NIB's) collect all gases on their own large cryopumps [6] during operation. By warming up the LHe cooled cryopanels to 77 K, the pumped gas is released. This regeneration is done periodically: during the main tritium experiment each evening after completion of the experimental program, during the subsequent clean-up experiment in increasing intervals, typically once per week.

Once the NIB cryopanels are regenerated, the NIB is connected via V2 to the evacuated inlet manifold. The total amount of gas can be measured via P2 and P3 with the known NIB and NIB pumping duct (crown) volume. Activity measurement via IC1 and sample bottles and mass spectrometer checks for potential air in-leakage are performed as during torus gas measurements.

The NIB is then evacuated to the Cold Finger by opening V1, followed by isolation of the NIB and re-cooling of its cryopumps. Typical NIB gas batches ranged from 11.5 barl after the main experiment to 270 barl during the clean-up experiment.

# 1.2 Cryopumping

The cryopump connected to the inlet manifold is shown in Figure 3.

A "Cold Finger" consisting of a corrugated metal hose of 50 mm ID and 60 mm OD is inserted in a LHe dewar (250  $\ell$  capacity). At its lower end, the Cold Finger contains 100 grams of activated charcoal to permit pumping of helium both during the main experiment and for clean-up tests with tokamak pulses in <sup>4</sup>He. The total capacity for <sup>4</sup>He was found to be ~ 15 bar $\ell$  at an equilibrium pressure of 1 Pa.

The capacity of the cryopump for crycondensation of gases condensible at 4 K is far in excess of the requirements of the experiment: batches of 600 bar $\ell$  (nearly 1  $\ell$  of solid hydrogen) can be pumped without any problems to pressures below 0.1 Pa. The dead-end design with counterflow heat exchange between evaporating helium gas outside the Cold Finger, make this design highly efficient and economical: pumping speed at 30 Pa inlet pressure of  $0.35 \text{ m}^3\text{s}^{-1}$  makes it equivalent to a medium sized roots blower of ~ 1200 m<sup>3</sup>h<sup>-1</sup> pumping speed. Simultaneously, LHe consumption is low: during the first three weeks of the main experiment including the major part of the clean-up experiment, only 200  $\ell$  LHe were consumed - the major part of it due to thermal cycling of the Cold Finger between 4 K and 300 K (~ 50 batches were transferred from torus and NIB's to the gas recovery system).

Once a gas batch is cryopumped on the Cold Finger, V1 is closed, V13 in the gas recovery system is opened and the Cold Finger is warmed to room temperature by lowering the lift platform with the LHe dewar. A highly flexible polymer bellows between Cold Finger top and LHe dewar neck

prevents air ingress (and freeze-out) into the LHe dewar. Regeneration proceeds rapidly: if the Cold Finger contains only a gas batch of less than 5 barl (compatible with the imposed safety limit of 15 mb in the reservoir in order to avoid any possibility of obtaining a potentially explosive mixture), the transfer can be completed in two minutes by lowering the dewar fully. Three minutes later (with V13 closed and the dewar raised) the Cold Finger is ready to pump. This device is a good example for a fast regeneration cryopump required in future fusion systems for gas processing.

# 1.3 Gas Storage System

The schematic of the Gas Storage system is shown in Figure 4. Gas is admitted from the Cold Finger into a reservoir with a calibrated volume of 345*ℓ*. In order to protect the Cold Finger against overpressure in case of a loss of coolant accident (e.g. inadvertent lowering of the dewar with a large batch condensed in CF), an overpressure relief device with 200 mbar  $\Delta P$  is installed in parallel to V13 - with a valve V14 (always open) in series.

The volume was calibrated in earlier experiments:

- using high-accuracy capacitance manometers by comparison with smaller calibrated volumes (which in turn were calibrated by filling with water and weighing);

using direct weighing: transferring D2 from the volume to a large JET U-Bed, the inventory transferred (27 moles by PVT measurement) was found to be in full agreement with the U-bed weight gain of 108 grams.

The pressure gauge P8 used for absolute gas measurements is a capacitance manometer with 13.3 KPa (100 Torr) full scale pressure, with high accuracy and linearity (deviation  $\leq 0.25$  %). Prior to absorption of the transferred batch, the gas was recirculated to achieve full mixing and stable readout on the ionisation chamber (IC2) signal. This was done by running the Normetex pump PUN (15 m<sup>3</sup>h<sup>-1</sup>) for several (typically five) minutes with V15, 16, 18, 21 open. This mixing run is necessary, since the Cold Finger (during defrosting) releases gases in sequence of volatility; typically the hydrogen isotopes would be followed by helium (desorbing from activated charcoal) and impurities like argon and hydrocarbons. Without mixing, additional errors would have been introduced in the tritium accounting [4]. After gas batch size and tritium content are measured, the gas is absorbed on uranium. Two uranium beds built for prototype tests for the JET AGHS [2] were installed in series with the recirculation pump PUN.

Two further uranium beds (U-B3 and U-B4) were added at a later stage of the clean-up experiment when it became evident that decontamination of NIB 8 beam dumps could only be achieved by intensive beam operation. This resulted in large gas batches, typically in the order of 100 to 300 bar $\ell$  with activities up to  $1.5 \times 10^{11}$  Bq. These additional uranium beds were connected in a spur after the Normetex pump (previous location of a pressure gauge).

Bypass valves V18 and V21 and shut-off valves V19, 19A, V20, 20A and V22, 22A, 23, 23A permitted gas circulation through U-beds 1 or 2 or both in series. This recirculation was also necessary to achieve rapid absorption. Many batches containing helium, argon, methanes and other inert gases could otherwise not be processed, since accumulation of these gases in the U-bed prevent further ingress of hydrogen ("blanketting").

The uranium beds were also used for impurity processing:

- during regeneration of NIB 8 cryopumps after initial tests with D<sub>2</sub> 1% T<sub>2</sub> beam injection an air leak developed, admitting approximately 10 barl of air to the system. Recirculation of the air/deuterium mixture through U-bed 1 at room temperature resulted in absorption of oxygen and deuterium (including tritium). Nitrogen could subsequently be discharged to stack.
- during the initial cleanup phase immediately following the main experiment, it was found that the hydrocarbons formed by plasmawall interaction in the torus (also pumped by NIB's through leaking fast shutters) were at an activity level at which direct discharge to stack could not be justified ( $\geq$  100 mCi per batch). The residual gases were recirculated through both U-beds, with the heater on U-bed 2 activated. When U-bed 2 reached 410 °C, a decrease in activity was observed, and at 450 °C, the activity was virtually removed (more than 99.7 % of IC2 signal). A check with the mass spectrometer showed that the remaining gases were only <sup>3</sup>He (from torus cleanup pulses with <sup>3</sup>He plasma) and some traces of argon. This demonstration shows that it is possible to process <u>all</u> plasma exhaust on a fully activated U-bed containing pyrophoric

uranium powder at a temperature well below 500 °C. During later stages of the cleanup experiment, also U-bed 2 had to be used for hydrogen isotope storage, thus making further impurity processing impossible. However, at this stage, the activity level of residual impurities had dropped to permit discharge to stack.

### 1.4 Gas Disposal

The gas disposal unit is schematically shown in Figure 5.

The only component used during the experiment was the rotary pump PUR (35 m<sup>3</sup>h<sup>-1</sup> pumping speed) to discharge waste gases to a stack which is continuously monitored for tritium [8].

For major potential upset conditions, such as a large torus air leak or a water leak inside torus or NIBs, a small exhaust detritiation system was on standby: one branch consisting of two 200  $\ell$  drums filled with molecular sieve 5 Å for water collection, the other branch containing a recombiner catalyst for hydrogen oxidation and subsequent absorption.

This system was not used, as none of the incidents for which it was foreseen did occur. Since minor activities arising from normal operation were well within approved routine discharge limits [7], it was decided to discharge directly to stack. This was in accordance with the principle of "Best Practicable Means" (BPM) as it avoided the generation of tritiated waste (in the form of molecular sieve drums) with very difficult and expensive disposal formalities.

# 2 Calibration and measurement of collected tritium

The JET ionisation chambers (IC1 and IC2) were originally calibrated at the Tritium Systems Test Assembly (TSTA) in Los Alamos (USA) with tritium in air at activity levels up to  $3.7 \times 10^{10}$  Bq m<sup>-3</sup> at a pressure of 75 kPa (local atmospheric pressure).

Throughout the operations during the tritium experiment, the operating pressures were much lower : typically 1 to 10 Pa in IC1 after torus pulses and between 0.5 to 3.3 kPa in the gas storage reservoir after compression of batches with the cryopump. Under these conditions, the IC signal is not simply proportional to specific activity for the following reasons:

- In the "normal" mode, ie at sufficiently high pressure, the mean free path of the tritium decay  $\beta$ -particle is small compared to chamber dimensions and all its energy is lost inside the active chamber volume in ionisation and excitation; hence, for a given gas composition with its characteristic cross sections for ionisation and excitation, the number of ion pairs generated per event, ie the ion current measured, is practically independent of pressure.
- At very low pressures, virtually no secondary ionisation can take place and only the low energy  ${}^{3}$ He<sup>+</sup> recoil nucleus from tritium decay will contribute to the current. In this "ion collection" mode, observed by Barnes and Gibson earlier [9], the signal is again pressure independent, ie only proportional to the specific activity and the chamber volume. Since it is also independent of gas composition, the signal can be defined from basic principles: we should obtain 1.6 x 10<sup>-19</sup> A Bq<sup>-1</sup>, ie collect one elementary charge for each event inside the chamber volume.

Virtually all our operation pressures were in an in intermediate range, where the number of ion pairs generated per event is a function of pressure. The only way to accurately measure specific activity (lacking a full pressure -dependent calibration) was to top up IC1 with nitrogen to 75 KPa, thus operating it in the normal mode. However, as this mixture could then not be absorbed on U-Beds, it had to be discharged to stack. Hence, this method was used only a few times during operations and only for acceptably low activities in order to limit stack discharges.

The final calibration of IC2 was performed after installation of U-beds 3 and 4. The gas collected in U-beds 1 and 2 was desorbed into the 345*l* reservoir in batches of 11.33 kPa. The signal of IC2 was measured during absorption on U-beds 3 and 4 as a function of pressure. During this transfer, only pure hydrogen isotope mixtures were present.

At a sufficiently low pressure (eg 3.33 kPa), gas was admitted to IC1 (via V15, V35, V7). Subsequently IC1 was filled to 75 kPa with nitrogen. For this mixture, the sensitivity was known from calibration at TSTA to be  $1.243 \times 10^{-20}$  [A Bq<sup>-1</sup> m<sup>3</sup>] for a fixed bias voltage of 115 volts.

The signal J(P) of the ionisation chamber IC2 at any pressure P is proportional to the tritium inventory P.C<sub>T</sub>. We can define a pressure dependent sensitivity S(P) as follows:

$$J(P) = S(P).P.C_{T}$$
<sup>(1)</sup>

With the current J(P) in [A], the pressure P in [Pa] and the tritium concentration  $C_T$  in [Bq Pa<sup>-1</sup> m<sup>-3</sup>] we obtain

$$S(P) = \frac{J(P)}{P.C_T} [A Bq^{-1} m^3]$$
 (2)

Or, with the calibrated data from IC1,

$$S(P) = \frac{J(P)}{P} \times 7.56 \times 10^{-8} [A Bq^{-1} m^3]$$
(3)

Fig 6 shows the IC2 current vs pressure for a calibration run with a tritium concentration of  $1.323 \times 10^7$  Bq Pa<sup>-1</sup> m<sup>-3</sup> and the sensitivity S as a function of pressure.

In this calibrated range, for pure hydrogen isotope mixtures, the tritium inventory can now be obtained at any pressure by reading S(P) at the pressure P. The activity A is then

$$A = \frac{J(P)}{S(P)} [Bq m^{-3}]$$
 (4)

and the inventory I is

$$I = A \times V_{RES} = \frac{J(P)}{S(P)} \cdot V_{RES} [Bq]$$
(5)

Where  $V_{RES}$  is the volume of the reservoir in  $[m^3]$ .

During the transfer from U-beds 1 and 2 to U-beds 3 and 4 the total amount of gas and the total tritium inventory could be accurately measured: from start of the experiment on 4 November 1991 until 22 December 1991 a total of 1450 bar $\ell$  of gas containing  $4.3 \times 10^{13}$  Bq (1162 curies) of tritium.

The accuracy of the gas measurement is  $\pm 2\%$  (volume calibration, pressure gauge P8 and some minor temperature fluctuations during the gas transfer).

The accuracy of the tritium inventory measurement depends on resolution and stability of the pico-ammeter ( $\pm 1 \times 10^{-12}$ A) but primarily on the accuracy of the initial calibration of TSTA which has been given as  $\pm 10\%$ . This results in an overall systematic error of  $\pm 10\%$ . Relative errors caused by the pico-ammeter are quite small for high activities and high batch pressures, typically  $\leq 0.1\%$ .

# 3 Safety during Operations

The general safety features and precautions are described in detail by M Huguet et al [3] and A C Bell et al [7].

The main subunits of the Gas Collection System, ie the inlet manifold together with the cryopump and the gas storage system, were enclosed in secondary containments constructed from rugged plastic sheet, supported by a steel frame. Access for all operations such as manual valves and sample bottles was through standard glove ports. Posting ports (again plastic, weldable by a standard thermal foil welding set) were used to introduce or remove sample bottles and tools as required.

Both containments were connected to a ventilation system discharging to the centrally monitored stack [8]. Each containment discharge line with a continuous flow of typically  $15\ell s^{-1}$  was also individually monitored for tritium. After any operation involving the manipulation of process connections (sample exchange, installation of U-beds 3 & 4), the plastic isolators were tested for contamination by taking smear samples through posting ports. Throughout the operations, no indication of tritium release was found. This can be attributed to

the very careful and conscientious work of all operators involved and to the meticulous selection and leak testing of all components during assembly.

# 4 Recommendations for future experiments

The smooth operation of the experiment left little to be desired apart from improvements in equipment calibration. In particular, measurements of activity at low pressures are affected by:

- background noise of current measurement
- background noise due to surface contamination of ionisation chamber
- low sensitivity due to low yield in secondary ionisation.

It is therefore essential to perform all measurements at a sufficiently high pressure (where the sensitivity is practically pressure-independent), typically at a pressure  $\geq$  50 kPa.

Since the addition of nitrogen precludes absorption on uranium beds after measurement, the gas to be used for topping up must be hydrogen. This also requires a new, precise calibration of the ionisation chamber with hydrogen as carrier gas for tritium.

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Fig 1 Gas Recovery System schematic







Fig 3 Cryopump



Fig 4 Gas Storage : Uranium Beds 1 and 2 are used for primary storage and impurity processing. When full, their inventories are transferred to Uranium Beds 3 and 4.



Fig 5 Exhaust Detritiation : drums filled with molecular sieve 5Å for water absorption and a recombiner for catalytic oxidation were foreseen for major Torus/NIB leaks. Only the mechanical pump PUR was used to discharge low activity waste gas (helium, argon, nitrogen, some methane) directly to stack via V36.



Fig 6 IC2 current vs pressure for a calibration run with a tritium concentration of 1.323x10<sup>7</sup> Bq Pa<sup>-1</sup> m<sup>-3</sup> and sensitivity 5 as a function of pressure

#### ANNEX

P.-H. REBUT, A. GIBSON, M. HUGUET, J.M. ADAMS<sup>1</sup>, B. ALPER, H. ALTMANN, A. ANDERSEN<sup>2</sup>, P. ANDREW<sup>3</sup>, M. ANGELONE<sup>4</sup>, S. ALI-ARSHAD, P. BAIGGER, W. BAILEY, B. BALET, P. BARABASCHI, P. BARKER, R. BARNSLEY<sup>5</sup>, M. BARONIAN, D.V. BARTLETT, L. BAYLOR<sup>6</sup>, A.C. BELL, G. BENALI, P. BERTOLDI, E. BERTOLINI, V. BHATNAGAR, A.J. BICKLEY, D. BINDER, H. BINDSLEV<sup>2</sup>, T. BONICELLI, S.J. BOOTH, G. BOSIA, M. BOTMAN, D. BOUCHER, P. BOUCQUEY, P. BREGER, H. BRELEN, H. BRINKSCHULTE, D. BROOKS, A. BROWN, T. BROWN, M. BRUSATI, S. BRYAN, J. BRZOZOWSKI<sup>7</sup>, R. BUCHSE<sup>22</sup>, T. BUDD, M. BURES, T. BUSINARO, P. BUTCHER, H. BUTTGEREIT, C. CALDWELL-NICHOLS, D.J. CAMPBELL, P. CARD, G. CELENTANO, C.D. CHALLIS, A.V. CHANKIN<sup>8</sup>, A. CHERUBINI, D. CHIRON, J. CHRISTIANSEN, P. CHUILON, R. CLAESEN, S. CLEMENT, E. CLIPSHAM, J.P. COAD, I.H. COFFEY<sup>9</sup>, A. COLTON, M. COMISKEY<sup>10</sup>, S. CONROY, M. COOKE, D. COOPER, S. 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