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# Tritium Off-gassing Trials on Dust and Flakes from the JET MkIIA Divertor

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## **ABSTRACT**

In 1998 100 grams of highly tritiated carbon dust and flakes containing an estimated 1 gram of Tritium were removed from the JET machine using a vacuum cleaner with a cyclone dust separator during the Remote Exchange of the divertor tiles. Two pots of flakes were filled and transferred, in drums, to the Active Gas Handling System where they were stored and ventilated to the Exhaust Detritiation System (EDS). A ventilated glovebox allowed the material to be handled safely and undergo calorimetric tritium assay. The glovebox had an air atmosphere with a continuous purge to the EDS with a facility for argon purging of isolators within the glovebox. Design features of the glovebox, operational experiences in handling tritiated carbon with a specific activity in excess of 1TBq/g and results of the tests are discussed.

## **1. INTRODUCTION**

In 1998 100 grams of highly tritiated carbon dust and flakes containing an estimated 1 gram of Tritium were removed from the JET machine using a vacuum cleaner with a cyclone dust separator during the Remote Exchange of the divertor tiles. Two pots of flakes were filled and transferred, in drums, to the Active Gas Handling System where they were stored and ventilated to the Exhaust Detritiation System (EDS). A ventilated glovebox allowed the material to be handled safely and undergo calorimetric tritium assay. Design features of the glovebox, operational experiences and results of the tests are discussed.

## **2. GLOVEBOX DESIGN**

The AGHS glovebox is designed to operate with a continuous air purge of 10-20 m<sup>3</sup>/h to the Exhaust Detritiation System (EDS). Inlet and exhaust streams are HEPA filtered and tritium concentration is continually monitored via a JET ionisation chamber (IC). The IC has a local display and a two-stage hardwired alarm set at 400 and 800MBq/m<sup>3</sup>. Under emergency conditions, such as a ruptured glove, a fast acting exhaust valve opens to EDS to maintain the glovebox depression. Flow increases to 100 m<sup>3</sup>/h and ensures an inward air velocity greater than 1m/s at the breach point. Exhaust flow is HEPA filtered at all times. Operating rules dictate that if the IC upper alarm level is reached work within the glovebox is stopped and gloveport bungs replaced. Work is not resumed until the tritium concentration falls below the IC lower alarm level. The emergency exhaust route can be opened manually under such conditions to increase flow through the box and reduce down time. Other features of the glovebox include a 300mm posting port and a drum port for transfer of items into the glovebox. The drum port doubles as a bagging out facility for waste and contaminated items.

### 3. OPERATIONAL EXPERIENCE

The entire operation was rehearsed and refined under clean conditions to train operators prior to committing the box to active work. Each step of the operation was covered by a detailed operating instruction. The material for assay was contained in two identical cyclone pots, numbers 1 and 2. The estimated inventory of the pots was 100g of carbon containing 2g of beryllium and 1g of tritium [1]. As 90% of the material was estimated to be in pot 2, pot 1 was handled first to allow any shortcomings in procedures or equipment to be assessed and improved before tackling the high inventory pot 2.

The cyclone pots had been stored for over six months following collection resulting in a build up of decay helium. In pot 2 the over-pressure was calculated to be up to 1.8 bar. Venting assemblies were engineered that allowed the helium to be released slowly from the pot whilst being retained for analysis. The assemblies comprised a bayonet adapter that mated to the cyclone pot, and a stainless steel sample cylinder. The cylinders were evacuated and then partially filled with argon prior to use. 0.5 $\mu\text{m}$  filters were installed to prevent carry-over of particulate into the cylinder. During initial venting of cyclone pot 1, 40GBq was released into the glovebox with a peak concentration of 8GBq/m<sup>3</sup> due to a poor seal between the bayonet adapter and the cyclone pot. The operation was stopped and the filter gaskets replaced with 10 $\mu\text{m}$  elements to reduce the resistance to flow. A further 87GBq was released into the glovebox on subsequent venting.

The off-gassing rate was measured using an aluminium sample pot modified to include a valved inlet hose and an outlet hose direct to the glovebox outlet. This was attached to the cyclone pot with a bayonet sample pot adapter. This arrangement allowed an air purge to pass over the flakes and directly to the EDS. The off-gassing rate was measured over a 38 minute period. A constant rate of 150GBq/h was achieved after 25 minutes.

The flakes were transferred from the cyclone pot to an aluminium sample pot. The sample pot is a sacrificial container designed to fit inside the precision-machined stainless steel calorimeter can. After decontamination and smearing, the sample pot was sealed into a PVC bag, and transferred to a ventilated enclosure where it was removed from the bag and inserted into the calorimeter can. The complete assembly was weighed and the mass of flakes determined to be 56.8g. This gave an off-gassing rate of 2.6GBq/h per gram of flakes.

The assembly was leak tested before being sealed in the JET calorimeter [2]. Due to the low mass of the assembly, thermal equilibrium was expected to be reached within a few hours. However initial data showed a rapidly decaying heat source characteristic of a chemical reaction and consistent with oxidation of hydrogen, see fig.1. The heat from the reaction diminished exponentially as the

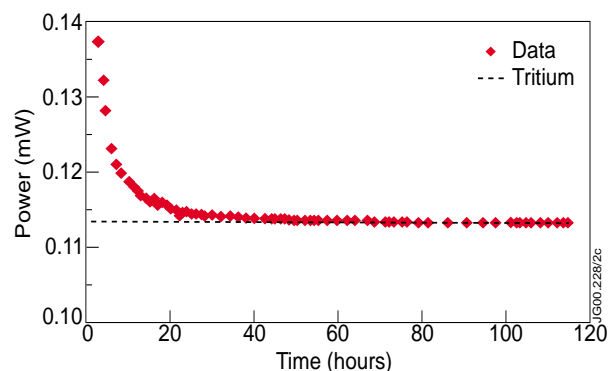


Fig.1: Results from the calorimetry of pot 1.

finite source of oxygen within the sealed can was consumed. The sample was resident in the calorimeter for 7 days. The tritium inventory was 0.17g giving a specific activity of 1.06TBq/g [3].

Following calorimetry the sample was posted back into the glovebox and the flakes returned to the cyclone pot for storage. During this whole operation involving pot 1, 550GBq were released in the glovebox.

#### **4. MODIFICATIONS FOLLOWING THE ASSAY OF CYCLONE POT 1**

A review of the handling of the first sample identified two key issues. Firstly, activity levels within the glovebox needed to be reduced. The high tritium levels seen during the first operation, up to 13GBq/m<sup>3</sup>, slowed the operation down considerably as operators withdrew from the box at 0.8GBq/m<sup>3</sup>, and also increased contamination of the glovebox internal surfaces and equipment. Secondly, the residence time in the calorimeter was prolonged by the oxidation reaction that occurred. The reaction needed to be suppressed.

To reduce contamination, and allow continuous operation, an argon supply system was installed in the glovebox. To comply with the glovebox safety case the system was engineered to provide a maximum flow of 0.1m<sup>3</sup>/hr during normal operation. Failure of the pressure relief valve and/or regulator would result in a flow of 0.3m<sup>3</sup>/h, much less than the EDS flow rate and thus not compromising the glovebox ventilation. A number of isolators and adapters were fabricated to facilitate purging and blanketing. The suite of operating instructions covering glovebox operations were revised, validated and formally re-issued before operations resumed.

#### **5. OPERATIONAL EXPERIENCE II**

The assay of the second pot followed the same methodology as the first with the addition of the argon purge facility being used at various stages.

An argon-purged isolator was fitted around the connection between the cyclone pot and the venting assembly. The exhaust from the isolator fed directly to the glovebox outlet. During venting 20GBq was released into the isolator and exhausted to the EDS. Activity levels within the glovebox remained below 50MBq/m<sup>3</sup>. The total activity released from the operation was six times lower than pot 1 due to a better seal between the cyclone pot and the venting assembly. Glovebox activity levels were reduced by a factor of 160 with the use of the argon-purged isolator.

Before transferring the flakes from the cyclone pot to the sample pot both containers were purged extensively with argon. The sample pot was purged to remove air that could react with the flakes, the cyclone pot was purged for 50 minutes to obtain a steady state off-gassing rate. A constant off-gassing rate of 150 GBq/h was achieved after 35 minutes with an argon purge rate of 0.5 NI/minute. The argon atmosphere negated the increase in inventory to give the same off-gassing rate as pot 1.

After transfer to an aluminium sample pot, and further purging to ensure an inert atmosphere, the second batch of flakes was weighed and assayed in the JET calorimeter. 0.35 grams of tritium in 97.5 grams of flake material gave a specific activity of 1.27TBq/g and an off-gassing rate of 1.5GBq/h per gram of flakes.

During the whole operation involving pot 2, 410GBq were released. Most of this activity was purged directly to the EDS.

## **6. PRODUCTION OF 1 GRAM SAMPLES**

The final operation in the programme, prior to box clean-up, was to produce two 1 gram samples for chemical analysis and further off-gassing tests. A calibrated spatula was produced to extract a 1.5cc sample, the density of the tritiated flakes being assumed to be the same as that of deuterated flakes from earlier campaigns. The intention was to briefly open the second sample pot, remove the samples and re-seal the pot. Due to the high off-gassing rates a novel PVC isolator was built to allow the sampling to be performed, with the calibrated spatula, in an argon atmosphere. The isolator had no rigid support structure and instead used inflatable limbs that were filled with argon. The use of inflatable supports was favoured as it allowed a large isolator to be built and then collapsed for posting into the glovebox. Rigid supports would either have limited the available working volume or necessitated cumbersome collapsible limbs. Once the limbs were inflated the pots and spatula were sealed inside the isolator and the argon supply was then used to purge the atmosphere directly to the glovebox outlet. The pots destined to receive the samples were pre-filled with argon.

This was the only operation where the flake material was actually visible to the operators. The flakes appeared to be glossy and were seen to have a blue/green hue. During this operation 270GBq were released into the isolator and purged to the EDS.

The pots containing the 1 gram samples were decontaminated and posted out of the glovebox. They were sealed in PVC and aluminized bags and then inside stainless steel transfer pots that were purged with argon before being placed inside the outer, approved transport packaging. Out-gassing trials were performed on the two outer layers of packaging before shipment.

The mass of flake material received by AEAT at Winfrith was 0.928g with a measured density of 1.6 g/cc. All flake handling operations were performed in a nitrogen environment.

Three individual flakes were removed from the bulk sample and weighed before being heated to 800°C in an airflow for 8 hours. All off-gasses were trapped by a water bubbler.

Flake 1 (5.5mg) released 6.54 GBq giving a specific activity of 1.19TBq/g. Flake 2 (8.3mg, 8.77GBq) gave 1.06TBq/g and flake 3 (5.1mg, 6.50GBq) 1.27TBq/g. The results agree with the JET figures and also show there is little variation across the sample.

Figure 2 shows the results of the Thermal Desorption Analysis (TDA) performed on a flake weighing 0.9mg. The flake was slowly heated in a furnace tube with an argon gas purge. The off-gas passed through a heated copper oxide catalyst and then through a bubbler to collect tritium. The bubbler water was analysed with a scintillation counter.

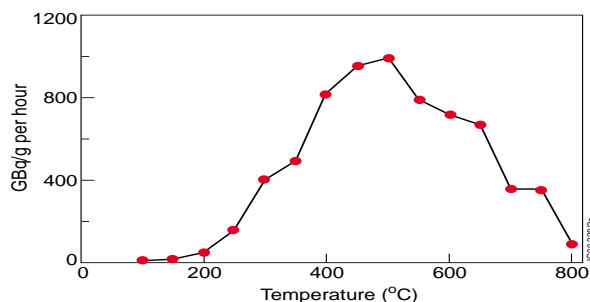


Fig.2: Temperature dependence of off-gassing.

Following TDA to 800°C the flake underwent total oxidation in air at 900°C. In total 1.12GBq was released from the flake giving a specific activity of 1.24TBq/g. 89% of the total tritium was released during the TDA.

## 7. GLOVEBOX CLEANUP

The flake samples were returned to their original cyclone pots and then removed from the glovebox, sealed in PVC and returned to a ventilated drum for storage. Other equipment was cleaned and removed with the operational waste. The internal surfaces of the glovebox were then decontaminated, first with alcohol wipes and then with a proprietary PVA decontaminant. Surface smears taken before and after application of the PVA decontaminant gave a decontamination factor above 100 but surface activity was still above 100Bq/cm<sup>2</sup>.

## 8. FURTHER WORK

A new facility is being designed to study tritium retention in carbon flakes and tiles and its ultimate recovery. The facility will be installed in a new purpose built glovebox.

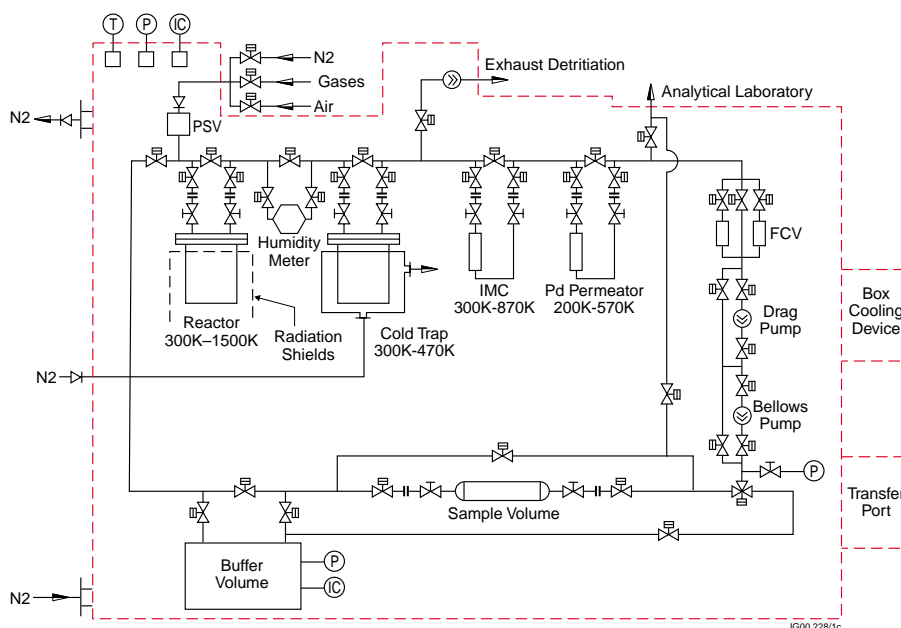


Fig.3: Schematic of new flake facility.



The existing glovebox is to be modified to operate with a nitrogen atmosphere and will be used for sample preparation. Initial studies will use samples of 5g (6TBq) that will be accurately weighed and then assayed in the JET calorimeter. Torus venting conditions can be simulated by passing air or nitrogen over the samples at temperatures up to 600K. Gases released from the flakes will be collected and undergo chromatographic analysis. The study will evaluate the rate and chemical forms of tritium released from flakes at ambient and elevated temperatures (up to 770K) as a function of tritium inventory. Figure 3 shows the main components of the facility.

## **9. CONCLUSION**

Methods for safely handling and assaying particulate carbon with specific activities > 1TBq (tritium) per gram have been developed at JET. Using a secondary argon purged isolator within the primary glovebox containment reduced operational activity levels by two orders of magnitude and significantly reduced the total activity released from the samples. Results from the chemical analysis of flake samples agree with the results from the JET calorimetric assay.

## **REFERENCES**

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