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Tritium Commissioning and
Use during DTE1

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

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ABSTRACT

The analytical gas chromatographic system (AN-GC) was the main tool used to analyse gas mixtures from various subsystems of the Active Gas Handling System (AGHS) and to characterise the functioning of the different processes during the Deuterium-Tritium Experiment (DTE1). Samples were transferred to AN-GC via long $\frac{1}{4}$ " lines. Calibration factors for different gas species were determined for the thermal conductivity detectors, flame ionisation detectors and ionisation chambers. The performance of the AN-GC is discussed by means of various chromatograms and the experience gained with these detectors as well as their limits are mentioned. The AN-GC worked very reliably and about 320 analyses were performed during DTE1 and the subsequent cleanup phase.

1. INTRODUCTION

The Active Gas Handling System (AGHS) at JET is used for processing gas mixtures pumped from the torus and connected systems. The gases are separated into hydrogen and impurities streams. Impurities are detritiated to re-gain tritium. Hydrogen gas mixtures are isotopically separated into pure tritium, deuterium and protium. Tritium and deuterium are re-supplied to the various users. This gas processing is done in various subsystems which are described in a companion paper [1]. A central analytical facility (designated Analytical Laboratory (AN)) exists which is connected to the other subsystems of AGHS via long stainless steel tubing of 0.63 cm outer diameter. The three analytical tools used in AN are: analytical gas chromatographic system (AN-GC), mass-spectrometers (Omegatron and quadrupole), and ionisation chambers.

This paper will focus on modifications and enhancements made to the previous design of the AN-GC and will discuss the tritium commissioning and results obtained during the deuterium-tritium experiment (DTE1). Inactive calibration results of AN-GC were partly presented previously [2]. Information already discussed in the previous paper will be repeated here only if necessary for the understanding of the new information.

2. DESCRIPTION OF THE JET ANALYTICAL GAS CHROMATOGRAPHIC SYSTEM

2.1 Purpose of the JET AN-GC system

The AN-GC was constructed to determine the concentrations of the gas species pumped from the torus and handled in AGHS. These gas species are the six hydrogen molecules, helium (no distinction between helium-3 and helium-4 is required), N_2 , O_2 , Ar, CO, CO_2 , CH_4 and higher hydrocarbons as well as their tritiated compounds.

A detailed description of the specification and the mechanical configuration of the AN-GC was given previously [2]. Here only the main principles will be given again. The main emphasis will be on the new detectors added and results obtained with tritiated gas mixtures.

2.2 Flow diagram of the JET AN-GC system

A updated schematic flow diagram of AN-GC is shown in Fig. 1. This system can be split into three main parts: the compression/injection stage; system 1; and system 2.

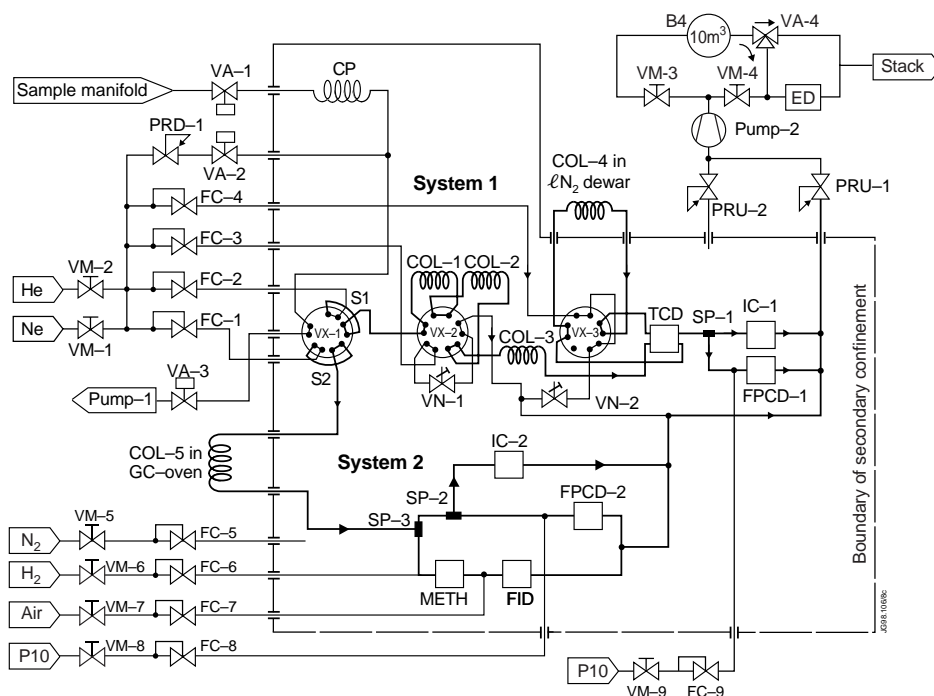


Fig.1. Schematic flow diagram of the AN-GC system: a) compression stage consists of VA-1, VA-2, VA-3, PRD-1, S1, S2, VX-1; b) System 1 consists of FC-2, FC-3, FC-4, FC-9, VX-2, VX-3, VN-1, VN-2, VM-9, COL-1, COL-2, COL-3, COL-4, TCD, FPCD-1, IC-1, and SP-1; c) System 2 consists of FC-1, FC-5, FC-6, FC-7, FC-8, VM-5, VM-6, VM-7, VM-8, METH, FID, FPCD-2, IC-2, SP-2, and SP-3; d) common equipment: VM-1, VM-2, VM-3, VM-4, PRU-1, PRU-2, and Pump-2. COL stand for column, VA for actuated valve, VM for manual valve, VN for needle valve, VX for Valco valve, FC for mass flow controller, PRD for down-stream pressure regulator, PRU for up-stream pressure regulator, TCD for thermal conductivity detector, IC for ionisation chamber, FID for flame ionisation detector, FPCD for flow proportional counter detector, METH for methaniser, SP for splitter, CP for inlet compression capillary, S1 and S2 for sample volumes, P10 for gas mixture of 10% methane and 90% argon, B4 for buffer volume B4 of 10m^3 , ED for Exhaust Detritiation system.

2.2.1 Compression/injection stage

Samples of different pressures between 30 and 330 kPa can be compressed to the pressure of 330 kPa by means of the down-stream pressure regulator PRD-1. This allows the injection of reproducible sample quantities and easy comparison of the peak areas of different chromatograms. Via switching VX-1 the samples in the volumes S1 and S2 are injected into the systems 1 and 2, respectively. The size of the injected volumes is approximately 0.05 cm^3 .

2.2.2 System 1

The main purpose of system 1 is to determine the concentrations of the six hydrogen molecules H_2 , HD, HT, D_2 , DT, T_2 . These molecules are separated by COL-4 which is kept at 77 K in a liquid nitrogen filled dewar. The physical separation of an injected gas sample into He, ΣQ_2 (sum of the six hydrogen species), Ar, O_2 , N_2 , CH_4 and CO is achieved with COL-1 and 2. On

the other hand, many gas species such as higher hydrocarbons, water, etc. are trapped in COL-1 and 2. For their detection use of system 2 or bypassing of the COL-1 and 2 is necessary. COL-3 is mainly installed for the analysis of water and Valco-valve VX-2 for the bypassing of COL-1 and 2.

The purpose of Valco-valve VX-3 is to switch the gas stream after He and ΣQ_2 have passed the first time through the Thermal Conductivity Detector (TCD) so that the other gas species bypass COL-4. In this way these gas species are not trapped in COL-4 at 77 K and any deterioration of COL-4 is excluded.

Three detectors are used in system1 for the analysis of the separated gas species: a thermal conductivity detector (TCD); an ionisation chamber (IC-1); and a flow proportional counter detector (FPCD-1). The TCD detects the various gas species by their different thermal conductivity. IC-1 and FPCD-1 react to radioactivity in the gas mixtures, in our case mainly to the tritium.

Details of the columns and detectors used can be found in Table 1 and 2, respectively.

Table 1: Details of columns used in the JET analytical gas chromatographic system

Number of columns	packing material	mesh	column wall	column length/m	column diameter/cm
COL-1	molecular sieve 13X	45/60	stainless steel	1.8	0.32
COL-2	molecular sieve 13X	45/60	stainless steel	1.8	0.32
COL-3	HayeSepD	60/80	stainless steel	1.2	0.32
COL-4	AL ₂ O ₃ doped with Fe	100/120	Cu	1.5	0.32
COL-5	HayeSepQ	60/80	stainless steel	3.6	0.32

Table 2: Details of detectors used in the JET analytical gas chromatographic system

Detector	Supplier	active volume/cm³	further information
TCD	Gowmac	—	filament temperature: 250° C, filament current: 160 mA
FID	Varian	—	250 V
FPCD 1 and 2	CFFTP, Ontario, CA	20	2100 - 2300 V
IC-1 and 2	JET designed	5.8	26 V

2.2.3 System 2

System 2 was constructed to measure CO, CH₄, CO₂, and higher hydrocarbons, their tritiated fractions and the sum of the tritiated hydrogen molecules (HT+DT+T₂). The three detectors used for that purpose are: flame ionisation detector (FID); ionisation chamber (IC-2); and flow proportional counter detector (FPCD-2). CO and CO₂ are converted into methane by a heated catalyst (called methaniser (METH)) with addition of hydrogen and detected by the FID indirectly via the methane.

2.2.4 JET specific ionisation chamber

FPCDs are known to be very sensitive. Therefore, already at the stage of construction of AN-GC a splitter was installed in front of them to reduce the sample size by a factor of 100. Nevertheless highly tritiated gas samples could not be analysed with the FPCD due to overflow problems of the readout electronics.

A schematic of the special ionisation chambers (IC-1 or IC-2) added to the AN-GC is presented in Fig. 2.

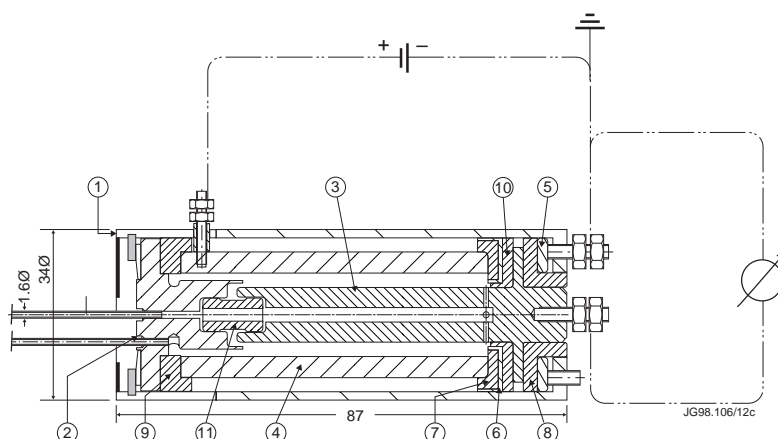


Fig. 2. Schematic of the ionisation chamber (IC1 and IC-2 of Fig. 1): the various items labelled by numbers are: 1: casing; 2: end electrode; 3: centre electrode; 4: outer electrode; 5: thrust plate; 6: guard ring; 7 to 11: various isolators.

The gas stream enters the IC between the outer and centre electrode and exits the IC via the middle bore in the centre electrode. The small gap between the end plug and the outer electrode acts as an ion trap for charges entering the IC. The outer cylinder is kept at a constant voltage of 26 V. The centre electrode is separated from the other parts of the IC by insulating material and a guard ring with a guard ring isolator and collects the amplified charges induced by the electrons of the tritium decay. The electrode material is OFHC-copper electroplated with 1 µm of nickel followed by 10 µm of gold. Teflon (PTFE) is used as insulating material. The use of teflon as construction material was accepted because even pure tritium injected into system 1 or system 2 will be diluted to concentrations less than a few percent in the carrier gas.

At JET the detectors and columns with the exception of COL-4 and COL-5 are located inside a stainless steel secondary containment which is purged by a small N₂ gas flow. The

inside of this secondary containment can become hot, especially the volume very near to the FID. The new ionisation chambers were mounted on a 1 cm thick Cu plate which acts as a heat sink and prevents temperature changes affecting the IC.

2.2.5 Discharge routes of the JET AN-GC system

The gas pressure at the exits of the various detectors is kept at 98 kPa by the up-stream pressure regulator PRU-1 (see Fig. 1). The carrier gas plus the injected sample is transferred by the metal bellows pump (pump-2) normally to Exhaust Detritiation (ED) with VM-4 open and VM-3 closed. All combustible gases such as the protium for the methaniser and the methane of the P10 gas as well as the hydrogen and tritium in the injected sample are converted inside the ED system to water and trapped in the molecular sieve of the driers [3].

A further discharge route was installed as a modification to the AGHS which allows under normal conditions the discharge of the various gas streams of the AN-GC by by-passing the ED system (see Fig. 1). The gas is moved by the pump Pump-2 with VM-3 open, VM-4 closed into the 10 m³ buffer tank B4 which is equipped with a JET designed ionisation chamber of an active volume of 0.57 litre [4]. The tank B4 is connected via an automatic three-way valve either to ED or directly to the stack. The connection is usually open to the stack. The automatic three way valve is switched to open the path to ED and to close the direct discharge to the stack by a hardwired interlock when the IC detects high tritium concentrations in B4.

3. THE DETECTORS OF THE JET AN-GC SYSTEM

3.1 The thermal conductivity detector (TCD)

The response of a TCD for the same injected sample size depends on the difference of the thermal conductivity between the gas to be analysed and the carrier gas. For the analysis of hydrogen a carrier gas of small thermal conductivity should be used because hydrogen (protium) is the gas with the highest thermal conductivity, but for the detection of argon, oxygen, nitrogen, methane, etc., a carrier gas with high thermal conductivity gives a low detection limit, e.g. hydrogen or helium.

To change the sensitivity of the JET TCD the choice for two carrier gases exists for the AN-GC-system by opening/closing VM-1 and VM-2 in Fig. 1: neon is used when high sensitivity for the six hydrogen molecules is required, helium if accurate determination of argon, oxygen, nitrogen, methane, is requested.

Many other factors influence the response of a TCD: flow rate of the carrier gas; temperature of the filaments of the TCD; temperature of the TCD housing; and temperature of the gas entering the TCD. These parameters have to be kept constant and checked at regular intervals.

In the case of the six hydrogen molecules the signal heights or peak areas decrease with increasing molecular mass due to the use of neon as carrier gas. The TCD is not only a gas specific detector, but in the case of hydrogen can even distinguish the six isotopically different

molecules due to the large isotopic dependence of the thermal conductivity of these gases. This fact complicates the analysis of the concentrations in a hydrogen isotope mixture because calibration factors for every hydrogen molecule have to be determined and the individual Q_2 concentration cannot be calculated from a TCD peak to which different hydrogen molecules contribute.

3.2 Ionisation chamber (IC)

The response of an ionisation chamber depends mainly on the voltage (normally chosen in the voltage plateau), the composition and the pressure of the gas mixture, and the physical dimensions of the ionisation chamber.

In the case of the AN-GC the ionisation chamber bias is supplied by a battery and the gas pressure is kept constant by an up-stream pressure regulator (PRU-1); under these conditions the signal is mainly a function of the composition. If the same type of molecules are to be analysed in the same carrier gas, e.g. HT, DT and T_2 in neon, then the only difference in response expected is that a T_2 molecule causes twice the signal of a HT or DT molecule. In addition, as mentioned previously, the dilution in the carrier gas is so high that charge recombination effects can be neglected.

For the same reason, the assumption is made that for singly and doubly tritiated hydrocarbons the IC signal ought to be the same as for HT/DT and T_2 , respectively. This assumption, however, could not be proven for lack of fully characterised test gas standards.

3.3 Flame ionisation detector (FID)

The outstanding features of a FID are: high sensitivity for almost all organic compounds; insensitivity to pressure and flow fluctuations of the carrier gas, and to temperature variations; and good linearity over a wide concentration range.

At JET the hydrogen and the synthetic air used to create the flame of the FID as well as the carrier gas are of purity better than 99.99 % to avoid introduction of hydrocarbons into the FID.

A pure hydrogen-air flame does not contain ions. In the presence of organic molecules ionisation occurs in the flame and the positive ions created are proportional to the number of carbon atoms in the flame.

3.4 Flow proportional counter detectors (FPCD)

FPCD were installed in the AN-GC by the supplier. These detectors were found to be far too sensitive for most analyses in the AGHS, even after installation of a 1:100 flow splitter in front of them [2].

As a consequence ionisation chambers were installed in the main flows of system 1 and 2 and the FPCD were not used in practical situations.

4. CALIBRATION RESULTS FOR THERMAL CONDUCTIVITY DETECTOR, IONISATION CHAMBERS AND FLAME IONISATION DETECTOR OF THE JET AN-GC SYSTEM

Only the calibration of the TCD, the IC and the FID will be discussed here. The FPCD was not used for quantitative analysis.

4.1 The thermal conductivity detector (TCD)

As mentioned in Section 3.1 the TCD reacts differently to various gas species as well as to the six hydrogen species due to their different thermal conductivity. Individual calibration factors have to be determined for each species.

Table 3 lists the calibration factors of gas species to be analysed by the JET TCD. These factors are used to normalise the measured peak areas to obtain correct concentration ratios. The calibration factors listed are relative to deuterium.

Table 3: Calibration factors of TCD for various gas species referenced to D_2 .

Gas species	Calibration factor
He	0.95
ΣQ_2	—
Ar	0.98
O_2	1.37
N_2	1.38
CH_4	2.40
CO	1.24
H_2	0.588
HD	0.783
HT	0.976
D_2	1.000
DT	1.126
T_2	1.454

When the calibration factors of all gas species detected are known, the concentration of each gas species of interest can be calculated from the peak area of this gas species multiplied by its calibration factor, divided by the sum of all peak areas multiplied by their calibration factors.

No calibration factor is given for the peak of ΣQ_2 in Table 3 because in most cases the relative concentrations of the six hydrogen molecules will not be known. The peak can be quantitatively used when only one type of hydrogen molecule is present or as an additional check of

the total hydrogen analysis by comparing the area of the ΣQ_2 peak at 1.81 minutes retention time with the sum of the other hydrogen peaks exiting COL-4 with retention times higher than 14 minutes. Due to the very sharp peak far smaller amounts of hydrogen can be detected by the ΣQ_2 peak than by the broad hydrogen peaks eluting from COL-4.

The linearity of the TCD was presented in a previous paper [2].

4.2 Ionisation chamber (IC)

One way to measure the linearity of a detector is to inject different gas amounts of the same calibrated gas mixture into the gas chromatograph. Different gas amounts can be obtained simply by changing the pressure in the injection volume.

In the case of tritium well known calibrated gas mixtures are difficult to obtain and their composition changes with time due to chemical reactions induced by ionisation and excitation. Furthermore, measurements with calibrated tritium mixtures lead to an unwanted additional tritium discharge or tritium load to ED.

To check the linearity of the AN-GC ionisation chamber in situ, the response of the IC was simply compared with the TCD. The linearity of the TCD was checked previously and found to be satisfactory. Chromatograms performed during the full tritium commissioning phase of the AGHS and during the first phase of the Deuterium-Tritium Experiment (DTE) [1] were used and the IC peak areas for HT, DT and T_2 compared with the normalised TCD peak areas. No new measurements were performed for this purpose.

4.2.1 The ionisation chamber IC-1 of system 1

The area counts of the single peaks HT, DT and T_2 of IC-1 are plotted in Figs. 3, 4, and 5 as a function of the corresponding TCD area counts, respectively. The ionisation chamber shows very good linearity for the three tritiated hydrogen species in the measured three decades. The

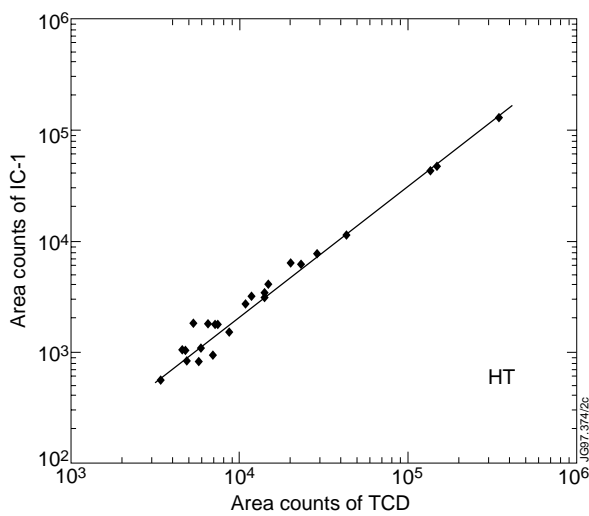


Fig. 3. Area counts of ionisation chamber IC-1 as a function of the TCD area counts for HT (normalised to deuterium).

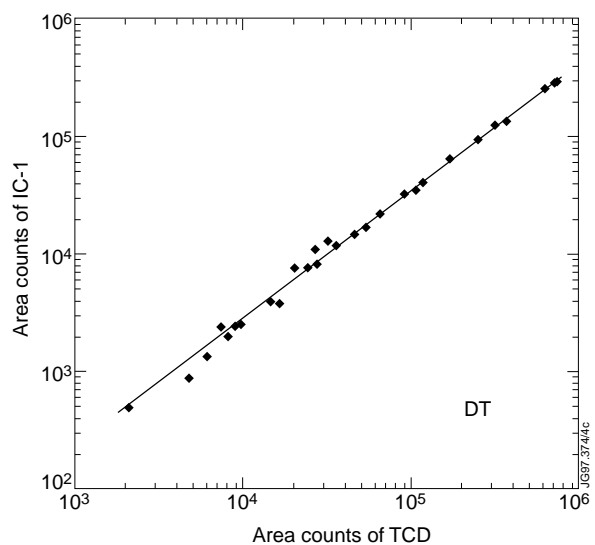


Fig. 4. Area counts of ionisation chamber IC-1 as a function of the TCD area counts for DT (normalised to deuterium).

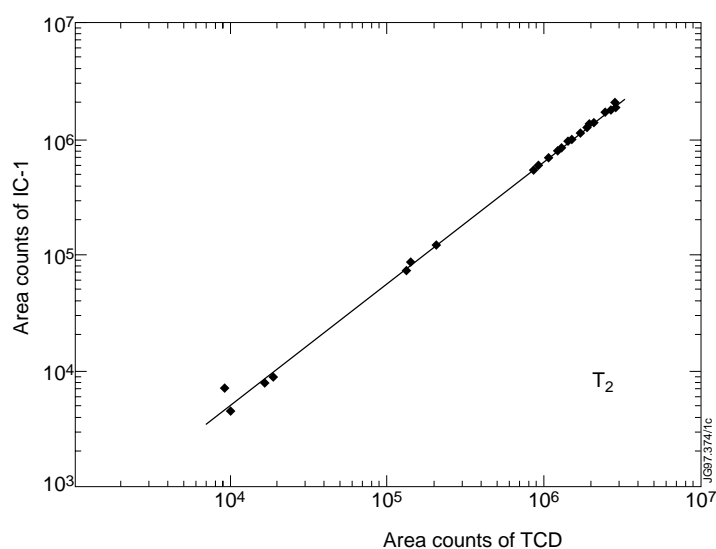


Fig. 5. Area counts of ionisation chamber IC-1 as a function of the TCD area counts for T_2 (normalised to deuterium).

agreement of the data for larger area counts in all three figures is excellent, larger scatter is observed in the lower area count region as expected due to the background noise of the detectors. The highest area counts are obtained for T_2 because ‘pure’ T_2 is supplied by the Product Storage (PS)- T_2 U-beds of the AGHS [1], but from other AGH U-beds containing hydrogen gas mixtures large DT or HT peaks are always mixed with significant fractions of D_2 and T_2 or H_2 and T_2 , respectively. The maximum possible area counts for HT and DT are always far smaller than those for T_2 .

If the difference in the sensitivity of the TCD for HT and DT is taken into account (see table 3) the data sets for HT and DT fit the same best straight line. This is also true for the T_2 data shown in Fig. 5 after halving the IC-1 data.

4.2.2 The ionisation chamber IC-2 of system 2

IC-2 was installed to determine the tritiated fraction of hydrocarbons in the gas mixtures. Hydrogen which is also injected in system 2 is separated by COL-5 from the other gas species. All six hydrogen gas species exit COL-5 with the same retention time. Thus IC-2 is capable to detect the sum of HT, DT and T_2 fractions in the gas mixture. Hydrogen elutes with a short retention time, thus its peak is sharp and a high sensitivity for HT+DT+ T_2 is obtained.

For lack of calibrated gas mixtures for tritiated hydrocarbons the linearity of IC-2 was checked with tritiated hydrogen.

Figures 6 and 7 show the IC-2 response for HT+DT+ T_2 as a function of the sum of the TCD as well as of the IC-1 signals for HT, DT and T_2 , respectively. In the case of the TCD the areas for HT and DT were divided by two. Very good linearity of IC-2 is found again which was already expected from the behaviour of IC-1.

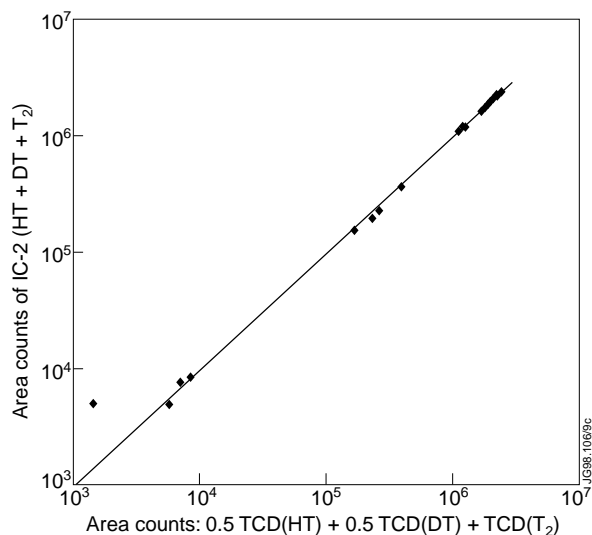


Fig. 6. Area counts of ionisation chamber IC-2 for HT+DT+T₂ as a function of the sum of the TCD area counts: 0.5 TCD(HT) + 0.5 TCD(DT) + TCD(T₂) (normalised to deuterium).

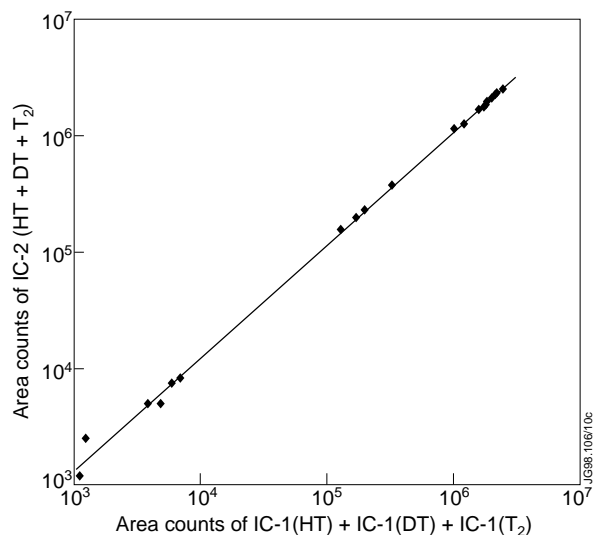


Fig. 7. Area counts of ionisation chamber IC-2 for HT+DT+T₂ as a function of the sum of the IC-1 area counts: IC(HT) + IC(DT) + IC(T₂).

4.3 Flame ionisation detector (FID)

The gas mixture listed in Table 4 was used to determine the retention times of the hydrocarbons for COL-5 and their response in the FID. The calibration factors calculated as the ratio of the peak area for methane to the hydrocarbon of interest is given in Table 4. The statement made in section 3.3 that the FID signal is proportional to the number of carbon atoms is proven generally correct. The peak area for C₂H₂ and for C₂H₆ is twice and the one for C₃H₈ about three times larger than the methane area for the same injected sample size. Therefore, the calibration factors for C₂H₂ and for C₂H₆, referenced to methane, are approximately 0.5, the one for C₃H₈ about 0.33. The mixture contained a large number of gas species and the hydrocarbons were at low concentrations, which hampered the accurate determination of their calibration factors. Even a small error in absolute concentrations can introduce a large error in the calibration factor. This is believed to be the case for the high calibration factors obtained for CO, CO₂ and C₂H₄ which should be 1.0, 1.0 and 0.5, respectively. Therefore, these values are

Table 4: Calibration factors of FID for various gas species referenced to CH₄ Gas mixture used for determination of calibration factors was 3.000% He; 5.030% Ar; 4.994% N₂; 4.989% H₂; 5.014% D₂; 0.102% CO; 0.199% CO₂; 0.104% C₂H₄; 0.097% C₂H₆; 0.099% C₃H₈; 0.998% CH₄; 0.100% C₂H₂; balance Ne

Gas species	Calibration factor
CO	(1.04)
CH ₄	1.000
CO ₂	(1.17)
C ₂ H ₄	(0.69)
C ₂ H ₂	0.498
C ₂ H ₆	0.492
C ₃ H ₈	0.321

Values in parenthesis are believed to be inaccurate. The calibration factors should be equal to 1/n where n is the number of carbon atoms per molecule.

listed in parenthesis in Table 4. A gas mixture with hydrocarbons of higher concentrations was not available. In addition, such a gas mixture would carry the risk of poisoning the catalyst in the methaniser by excessive amounts of unsaturated hydrocarbons.

During the inactive commissioning [1] the FID showed only positive peaks which were attributed to hydrocarbons, CO and CO₂.

With the first tritium commissioning a negative peak of the FID was observed with the same retention time as the HT+DT+T₂ peak of IC-2. Finally negative peaks were also observed in connection with tritiated hydrocarbons. Thus it is clear that the FID reacts sensitively to charges created by radioactivity in the gas, in our case tritium or tritiated compounds. The electrons of the tritium decay (β^-) and their amplification are the cause of this signal. Part of the amplification occurs via the neon carrier gas. Neon atoms promoted from ground state to an excited metastable level by absorption of the β^- decay energy can ionise the gas molecules other than neon present in the carrier gas and are detected when passing through the FID. In fact this is the main principle of a helium-ionisation detector [5] where β^- particles created in a tritiated target irradiate continuously the helium carrier gas, but signals are only detected when gas species are present in the carrier gas and are ionised by the excited helium atoms.

In addition, the flame was of no importance for the tritium induced FID peaks, because the peaks were also observed with the flame off.

The area under this negative FID peak (caused by HT+DT+T₂) is shown as a function of the calculated TCD-peak areas ($0.5 \text{ TCD(HT)} + 0.5 \text{ TCD(DT)} + \text{TCD(T}_2)$) in Fig. 8. The TCD areas for HT and DT are divided by 2 because only one tritium atom is present in these molecules. A smooth curve can be fitted to the plotted data representing a clear relationship between the results of the two detectors.

It can thus be seen that a FID can also be used to characterise the tritium content in hydrogen. Gas species normally not recognised by an FID can be seen when they are radioactive.

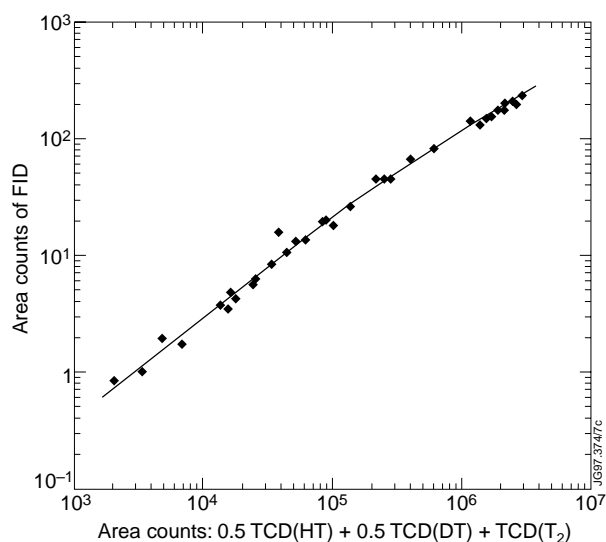


Fig. 8. Area counts of FID for HT+DT+T₂ as a function of the sum of the TCD area counts: $0.5 \text{ TCD(HT)} + 0.5 \text{ TCD(DT)} + \text{TCD(T}_2)$.

5. DISCUSSION OF RESULTS MEASURED WITH THE JET AN-GC

This section presents various results obtained with the different detectors during the various tritium phases of the AGHS.

5.1 Deuterium with and without HD

Two TCD chromatograms for deuterium are shown in Fig. 9. The upper chromatogram is obtained for commercially available deuterium. The deuterium characterised by the lower spectrum was extracted from the top of column-3 of the JET Cryogenic Distillation (CD) system [6]. In both cases samples compressed to 0.3 MPa were injected.

The same attenuation factors are used in both plots to allow easy comparison.

The main difference between the two chromatograms is the clearly visible HD peak in the upper picture. No H₂ is seen.

Very pure deuterium is produced by the CD system. The only peak observed is deuterium which means that the HD content must be below the lower detection limit of 50 ppm. Furthermore, no tritium peaks such as DT are seen by IC-1. This type of deuterium was supplied to some of the D₂ users (principally the Neutral Beam System) during DTE1 [1], [7].

In contrast, HD of easily measurable concentrations is found in the commercial deuterium.

5.2 Variations in the retention times of the hydrogen species

The two TCD-spectra shown in Fig. 10 are typical for hydrogen gas mixtures with all six hydrogen species. A small peak of helium at the retention time of 1.47 minutes is seen.

Both spectra are obtained from an un-compressed sample. Under these conditions the injection pressure is equal to the supply pressures of the gas in the manifold which were 87.2 and 75.2 kPa for the upper and lower figures, respectively.

The peaks in Fig. 10a are more symmetrical than the D₂ peak of Fig. 9a. The reason for this is that the compressed D₂ sample of Fig. 9 was too large with respect to the capacity of

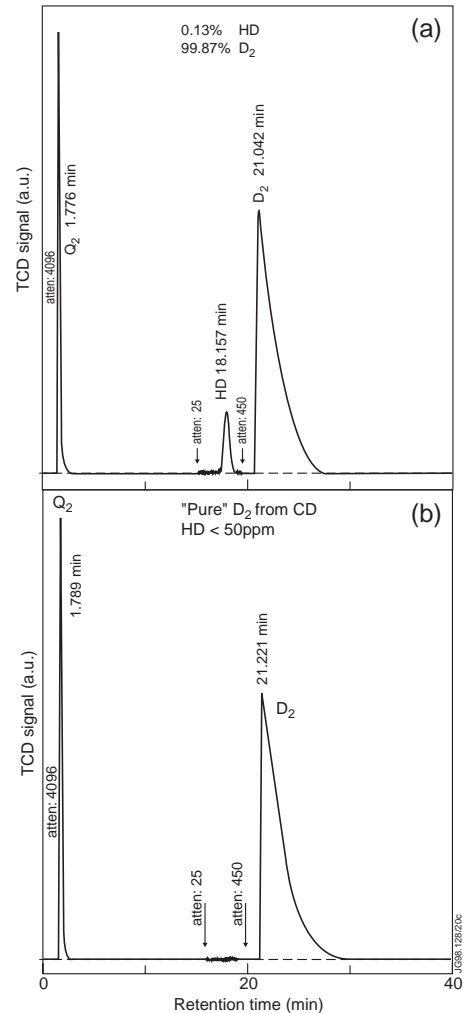


Fig. 9. TCD chromatogram of deuterium: a) for commercially available deuterium; b) for deuterium extracted from the top of COL-3 of the JET Cryogenic Distillation system.

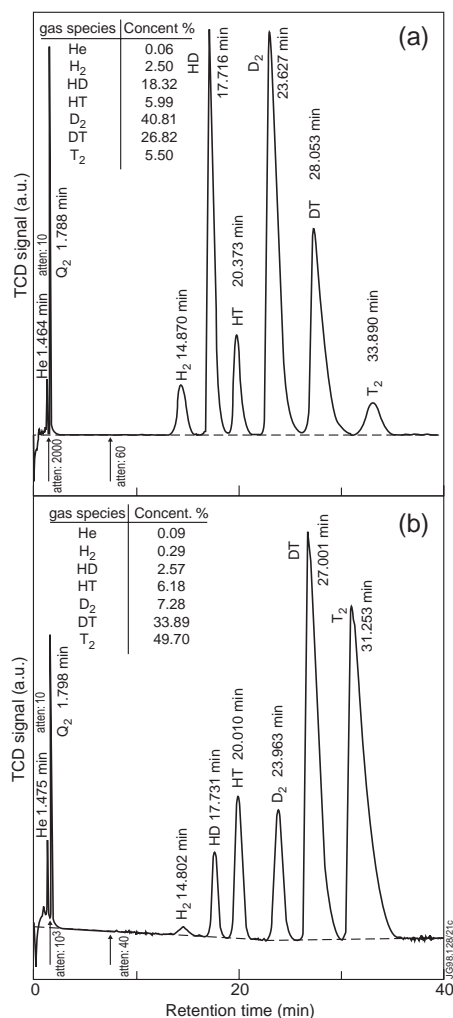


Fig. 10. TCD chromatograms of two different hydrogen mixtures with all six hydrogen gas species showing different retention times for the six hydrogen species.

to 5 minutes. Similar shifts can be caused between peaks with very large and very small concentrations (see e.g. D₂ peak in Fig. 9 and in Fig 10). Peaks with very large concentrations usually elute with shorter retention times and peaks with very small concentrations with longer ones. Furthermore, the retention time depends on the temperature of the liquid nitrogen cooled column COL-4. Changes of the height of the liquid nitrogen level above the column may also influence the temperature of the column and the density in the pipe work below the level of liquid nitrogen and consequently the retention times.

The correct identification of hydrogen peaks with different concentrations can become complicated when one or more of the six hydrogen molecules are absent from the mixture or below the detection limit. An ionisation chamber in series with the TCD helps to solve this problem because the tritiated gas species are seen by the IC and they exit the column at same time, a further aid to clear identification.

Small negative peaks are seen at almost zero retention times in Fig. 10a and 10b. They are visible due to the very small attenuation factor used to show the small helium peaks and are

COL-4. During the movement of the D₂ gas through the column a certain fraction of the trapping centres in the column is already occupied by D₂ molecules. Further D₂ molecules have to pass longer distances before being trapped again by free trapping centres. Thus some of the D₂ molecules pass faster through the column. More weight is added to the side of the D₂ peak with the lower retention time. The result is a very asymmetrical peak. This is the case for the compressed D₂ sample in Fig. 9a with a retention time of 21.0 minutes. For comparison the uncompressed D₂ peaks with a concentration of 40.8% and 7.3% exit after 23.6 or 24.0 minutes, respectively, almost 3 minutes later.

Another shift in the retention times is seen in Fig.10 from 31.2 to 33.9 minutes for the T₂ peaks with concentrations of 49.7 % to 5.5%, respectively. The shift in the retention times increases with increasing difference in concentrations.

The difference between the retention times of adjacent peaks in Fig. 10 is about 2.5

caused by flow fluctuations in the TCD as the injection volume S1 was filled with a gas pressure lower than the pressure present at the inlet of COL-1. This causes a small instability of the TCD signal. This instability is not observed when the injection volume is filled with gas of a pressure equal to the head pressure of the AN-GC which is the case when compressed samples are used. In Fig. 11 this TCD behaviour is not seen due to the very large attenuation factor of 2500.

5.3 'Pure' tritium measured with TCD, IC-1, FID, and IC-2

The chromatograms of 'pure' tritium are presented in Fig. 11 for the four main detectors. Please note that the attenuation is set to unity in the TCD and IC-1 spectra to show the small impurity peaks HT and DT. The DT and HT peaks are clearly seen in the IC-1 spectrum, whereas the TCD peaks show a significant noise level. No H₂, HD or D₂ peaks were detected by the TCD.

The chromatograms shown in Fig. 11 are not representative of the best tritium purities achieved during DTE1. Indeed tritium purities of 99.92% T₂, 0.06% HT, 0.02% DT were achieved, but they are not presented here because their HT and DT peaks are almost not visible.

Figure 11 also shows the concentrations of the gas mixture calculated for the TCD only by the TCD analysis and for the IC by the IC analysis. The concentrations listed in the TCD spectrum are obtained with the calibration factors of Table 3. The values of the IC-1 spectrum are calculated by simply dividing the T₂ area by two, by adding the three peak areas up and by calculation of the relative percentages of the various peak areas. The two concentration sets agree very well and give confidence in the calculation procedures.

The sample was injected at a pressure of 92.9 kPa. The T₂ peaks look very asymmetrical. The difference in retention time between the DT and the T₂ peaks is only 2 minutes because the very small DT peak elutes relatively late, but the T₂ peak is early. This is about three times smaller than the difference in retention times between T₂ and DT plotted in Fig. 10a. The negative FID peak and the IC-2 signal appear at the same retention time. The peak due to HT+DT+T₂ in IC-2 is far sharper than the HT, DT, and T₂ peaks of IC-1. This is due to different columns and very different retention times. The peak width increases with retention time.

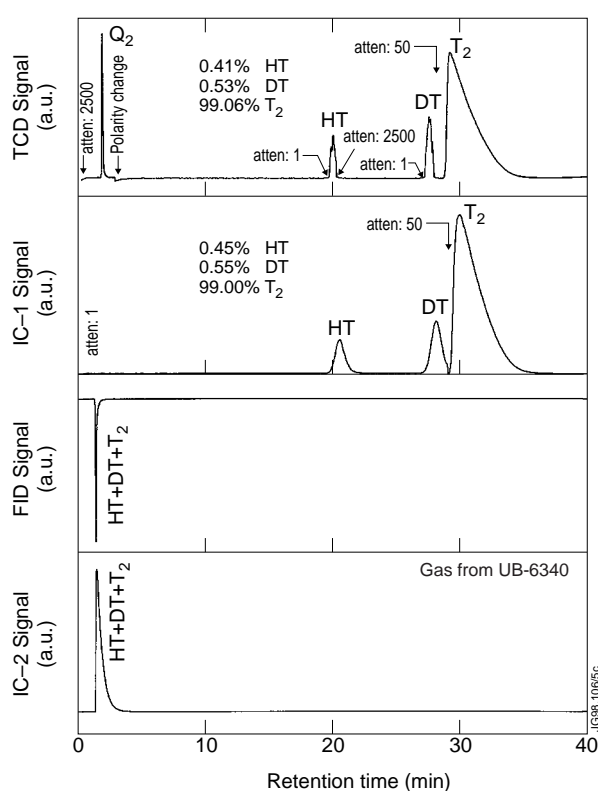


Fig. 11. TCD, IC-1, FID and IC-2 chromatograms for 'pure' tritium.

5.4 Advantages of multi-detector evaluation

5.4.1 Sensitivity enhancement by combining results of TCD and IC-1

Table 3 shows that the detection of T_2 by a TCD is approximately 2.5 times more difficult than H_2 . The width of the tritium peak is about twice the one of H_2 . As a consequence the lower detection limit for T_2 is approximately 5 times higher than for H_2 .

The ionisation chamber is most sensitive to the T_2 peak. Figure 11 shows that the HT and DT peaks measured with IC-1 are far easier to analyse than the TCD peaks.

By using the clear linear relationship shown in the Figs. 3, 4 and 5 between IC-1 and TCD response, the peak areas of the TCD and the IC-1 can be combined in such a way that the HT, DT and T_2 data of the TCD are substituted by the ones of IC-1. In this way the advantages of both detectors are used in one analysis.

Comparison between pure TCD and combined TCD and IC-1 analysis for mixtures with easily observable TCD peaks for HT, DT, and T_2 revealed that these two methods give very similar answers, but the combined method was better when the HT, DT or T_2 concentrations were small.

5.4.2 Sensitivity enhancement by combining results of TCD and FID

The FID shows a much greater sensitivity and lower detection limit for methane and higher hydrocarbons than the TCD. In addition, COL-1 and COL-2 absorb higher hydrocarbons so that they can not be detected with the TCD.

The TCD and FID response for methane were measured with the gas mixture listed in Table 4 for various gas amounts injected via S1 and S2. A very good linear relationship was observed. Using the linear equation the FID response can be converted into a more accurate TCD result for methane than the one actually measured by the TCD. By finally using the TCD-calibration factor between methane and deuterium (see Table 3) the TCD area for methane was transformed into a deuterium equivalent area and then used in the same way as discussed in Section 4.1 to calculate the percentage of methane in the mixture.

The above procedure can also be used for CO, CO₂ and other hydrocarbons after their FID areas are multiplied with the calibration factors of Table 4 to reduce the higher sensitivity for higher hydrocarbons C_nQ_m . Theoretically the measured calibration factors of Table 4 should be equal to $1/n$ with n counting the number of carbon atoms per molecule.

5.4.3 Determination of tritiated hydrocarbons

The IC-2 areas for hydrocarbons were converted into TCD areas using the data shown in Fig. 6 and normalised to deuterium. The ratio of the TCD area calculated using the IC-2 area to the TCD area obtained from the FID area (see Section 5.4.2) gives the ratio of doubly tritiated hydrocarbons $C_nR_{m-2}T_2$ (R for H or D) to the total number of C_nQ_m molecules. Multiplication of this ratio with the concentration of hydrocarbons (obtained in Section 5.4.2) gave the concentration of doubly tritiated hydrocarbons.

5.4.4 Comparison between IC-1 and IC-2

IC-1 measures the three hydrogen molecules HT, DT and T₂ time resolved at long retention times (> 20 minutes), whereas IC-2 detects the three mixed molecules simultaneously at a retention time of approximately 1.2 minute. The IC-1 peaks are about 3 times broader than the single one of IC-2. Therefore, the sensitivity of the IC-2 is far higher, any influence of background correction in case of IC-2 far lower and only one peak needs to be analysed.

The IC-2 response after conversion of the HT+DT+T₂ area with the linear relationship of Fig. 6 into a TCD area (normalised to deuterium) can be compared with the IC-1 data after the conversion of the HT, DT, T₂ IC-1 areas by means of the Figs. 3, 4, and 5, respectively, into TCD areas (again normalised to D₂) and addition of these TCD areas. With these TCD areas the concentrations of HT+DT+T₂ can be calculated from the IC-2 peak and compared with the sum of the concentrations obtained from IC-1 for HT, DT and T₂ as discussed in Section 4.1.

The concentration calculated with IC-2 for HT+DT+T₂ is more accurate than the sum of the concentrations obtained from the three peaks seen by IC-1 and can be used as an additional check of the IC-1 results.

At very low concentrations this check or comparison between IC-1 and IC-2 does not work as well because the IC-2 peak can be affected by contamination due to the inactive gases H₂, HD and D₂ eluting with the same retention time. This is discussed in the next section.

5.5 'Contamination peaks' in ionisation chambers caused by surface contamination

As there are only three known tritiated hydrogen species not more than three tritium peaks are expected in an IC-chromatogram. But in reality IC chromatograms often show six or even more peaks, as for example in Fig. 12. The six IC peaks show the same retention times as the corresponding TCD peaks. In Fig. 12 no T₂ peak is observed in the TCD-chromatogram due to the low tritium concentration.

One simple explanation for the appearance of the three peaks in connection with the three inactive hydrogen species is that tritium trapped on the surfaces of the tubing in front of the IC and of the IC itself was exchanged or replaced by inactive hydrogen. Therefore, tritium was released from these surfaces into the gas and was detected by the IC. The tritium amount desorbed or exchanged increases with the size of the separated gas species H₂, HD or D₂.

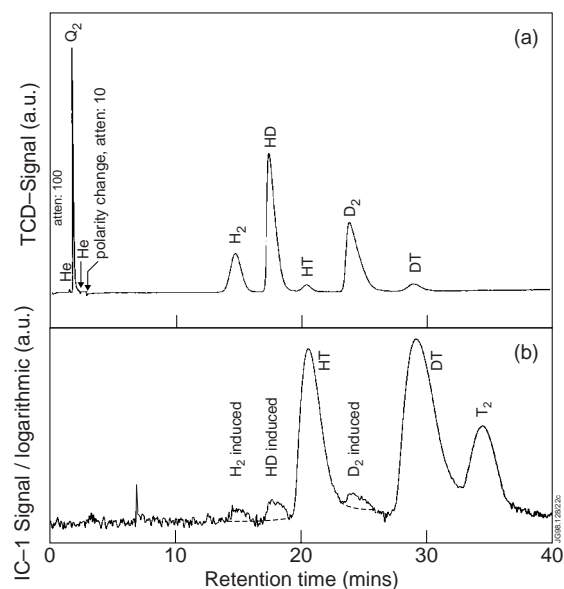


Fig. 12. TCD and IC-1 signal for a hydrogen gas mixture. This shows 'contamination peaks' of IC-1 at the same retention times as H₂, HD and D₂.

These ‘contamination peaks’ were larger when high tritium samples were analysed shortly before, leading to a high tritium coverage of the inner surfaces of the pipework and of the IC.

These ‘contamination peaks’ were not caused by tritium liberated in the columns in front of the IC because tritium desorbed in the various sections of the column exited the column at various times. This could only lead to a slightly higher tritium background in the IC-reading, but not to a well defined peak.

It is worthwhile to note that any gas, not only the presence of H₂, HD, D₂, can cause these ‘contamination peaks’ to appear. Therefore not only the exchange seems to be important, but also simple liberation. IC-2 saw similar contamination peaks to IC-1 induced e.g. by CO or CO₂. The peaks appear generally at retention times different from those of tritiated gas species, do not influence their analysis and can often be just simply ignored.

In IC-2 this contamination effect is more difficult to analyse because the IC-2 peak at 1.2 minutes caused by HT+DT+T₂ can not be distinguished from contributions due to liberation of tritium contamination by H₂, HD and D₂. The largest contributions in IC-2 seen from tritium contamination were well below 50 ppm and can therefore be neglected.

A further, more likely explanation for these contamination peaks follows the arguments mentioned in Section 4.3. Tritium bound to inner surfaces of the ionisation chamber irradiates continuously the passing neon carrier gas. These excited neon atoms can ionise all other gases with the exception of helium. If other gas species than helium or neon are present in the ionisation chamber, their ionisation can be easily detected with the ionisation chamber. The detected current increases with increasing concentration of the gas species in the carrier gas and with increasing coverage of the inner surfaces with tritium. The fact, that contamination peaks were observed in connection with the inactive gas species such as H₂, HD, D₂, N₂, CO, and CO₂, but never with He, can simply be explained by the neon amplification effect (analogous to the helium amplification in a helium-ionisation detector), because excited neon atoms are not capable of ionising helium, but not by the model, given above, of a simple transfer of tritium from the inner walls into the gas phase. If the neon amplification effect is combined with the first model, and transferred tritium in the gas phase also gives rise to an amplification effect, then it is no longer possible to distinguish between the two models. This shows that the neon amplification effect plays an important role in any ionisation chamber where neon is used as carrier gas. Analogous statements are true for ionisation chamber filled with helium.

These ‘contamination peaks’ were a nuisance caused by internal tritium contamination. Their concentration if mistaken for a tritiated peak was smaller than 50 ppm. They were generally identified by their asymmetric peak form caused by the asymmetrical distribution of the large gas amounts passing through the ionisation chamber and by their retention times which coincide with the retention times of strong peaks of tritium free compounds seen by the TCD and/or FID.

5.6 Tritium induced negative peaks in the FID chromatograms

The main and most clearly visible negative peak induced by tritium is presented in the FID chromatogram of Fig. 11 and is simply due to the sum of HT, DT and T₂.

Figures 13 and 14 show TCD, IC-1, and FPCD-1, FID chromatograms with FID flame on and off measured for a gas mixture obtained after performance of accountability measurements for the PS-T₂ U-beds. This gas was scrubbed through PS-T₂ U-beds and contains mainly helium-3 and hydrocarbons. The hydrocarbons are expected to be strongly tritiated because they are mainly generated by the interaction with the 'pure' tritium gas.

The TCD spectrum (Fig. 13a) shows only the positive and negative signal of helium-3 when passing through both sides of the TCD. No Q₂ signal is seen arising from COL-1 to 3 or

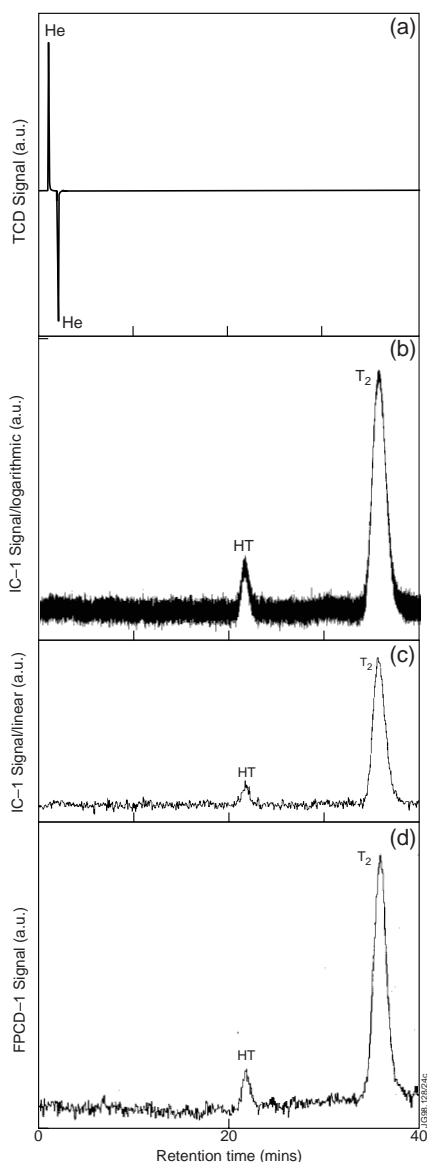


Fig. 13. TCD, IC-1 and FPCD-1 chromatograms of a gas mixture obtained after handling pure tritium and scrubbing this gas through a cold U-bed. The IC-1 chromatograms are shown in linear and logarithmic presentation.

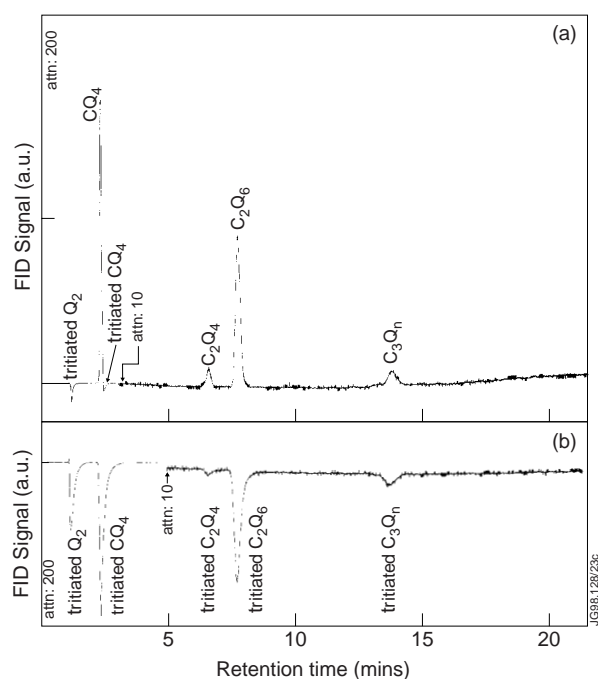


Fig. 14. FID chromatograms (a: with flame ignited, b: with flame off) of a gas mixture obtained after handling pure tritium and scrubbing this gas through a cold U-bed.

from COL-4. Only IC-1 and FPCD-1 detect the very small HT and T₂ concentrations of a few ppm. Figures 13b and 13c present the IC-1 chromatogram in logarithmic and linear presentation. The logarithmic presentation is directly obtained from the pico-amperemeter and shows the noise level in an enhanced way, whereas the linear response (Fig. 13c) is calculated and the noise is reduced by the height of the T₂ peak. As expected (see Fig. 13d), the FPCD-1 detects also the small HT and T₂ concentrations in the gas mixture.

The FID chromatogram with flame ignited (Fig. 14a) shows not only the negative peak at 1.1 minutes retention time, but a sharp negative peak of small height next to the CQ₄ peak. The cause of this negative peak is again tritium in tritiated CQ₄. Such a negative peak can appear when the retention times of CH₄ and CT₄ (the two extreme cases of untritiated and fully tritiated gas species) are slightly isotope dependent. The retention time of CQ₄ in COL-5 seems to increase in the sequence CH₄, CD₄ and CT₄.

For gas mixtures with large fractions of protium and deuterium in CQ₄ and only small fractions of protium and/or deuterium exchanged by tritium, only one major positive FID peak is expected which is the difference of the FID peak obtained with no tritium present in CQ₄ and of the negative peak created by tritium in CQ₄.

Figure 14b shows a chromatogram for the same gas mixture, but with the FID flame not ignited. No positive peaks are seen, but many negative ones which all occur with the same retention times as the hydrocarbons peaks in the figure above. All these peaks are caused by the amplification of the electrons of the tritium β⁻ decay discussed in Section 4.3.

The negative peaks (labelled ‘tritiated’) in Fig. 14b obtained with the flame off are far higher than corresponding ones (similarly labelled in Fig. 14a) with the flame ignited because the density of the gas contributing to the signal inside the FID is inversely proportional to the effective temperature. Figure 15 shows this clearly on two measured FID chromatograms with flame on and off. The ratio of the areas is 6.1:1, which means that the areas of Fig. 14b are to be divided by this factor before their addition to the positive peaks in Fig. 14a to obtain the ‘correct’ peak area for the total amount of hydrocarbon molecules under the assumption that these two effects can be superimposed in a linear way. Assuming that the temperature of the gas entering the FID with flame off is 330 K the effective temperature inside the FID collector with flame on is estimated to be 2000 K which seems reasonable comparing the hot tip of the hydrogen flame (<3000 K) with the active collection volume of the FID.

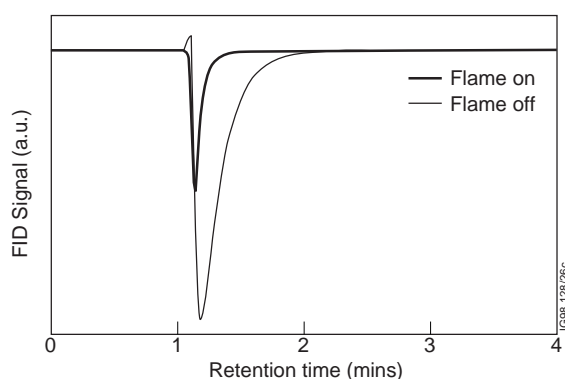


Fig. 15. FID chromatograms of the negative peak due to HT+DT+T₂ for FID flame on and off.

This means that the measured FID peaks will always give undersized FID-areas if tritiated species are present in the gas. Therefore, the concentrations of the hydrocarbons will be underestimated using FID for the analysis of tritiated gas species.

The presence of these negative peaks also often suppressed by large positive contributions from the FID leads to underestimation of the concentrations of hydrocarbons in the gas mixture especially if the fractions of tritiated hydrocarbons is large. Furthermore, it is not clear if the two measured areas can be simply added or a possible interference between the two effects exists. The main conclusion is that the use of an FID for unambiguous analysis of radioactive gases should be minimised or avoided if possible.

5.7 Chromatograms of gas mixtures collected and treated in Impurity Processing (IP) system

TCD, IC-1, FID, IC-2 chromatograms of a gas mixture collected from various volumes of the Cryogenic Forevacuum (CF) system are shown in Fig. 16. The peaks of the TCD spectrum (Fig. 16a) can be attributed to He, Q₂, N₂, CQ₄, CO and H₂. No other hydrogen species are recognised by the TCD, therefore the Q₂ peak is caused by H₂ alone. The IC-1 signal (Fig. 16b) supports this analysis because the observed HT, DT and T₂ peaks are so small (recognisable by the height of the signal with respect to the noise level) that these gases are not seen by the TCD. The FID detects CO, CQ₄, CO₂, C₂Q₄, C₂Q₂, C₂Q₆ and C₃Q_n (n=4, 6, 8). See Fig. 16c. In addition, a very small negative peak due to tritiated hydrogen is seen in the original data, but not recognisable in Fig. 16c. The peaks C₂Q₄ and C₂Q₂ are not resolved in this case although C₂H₄ and C₂H₂ were normally resolved [2]. This could be due to the fact that Q_n (n=2, 4) stands here for all kinds of isotopic combinations of protium, deuterium and tritium and with slightly different retention times for these various gas species only one peak is observed. The peaks with retention times at 17.5 and 27.5 minutes are not identified. The gas mixture for various hydrocarbons available for calibration studies did not contain hydrocarbons higher than C₃H₈ (see Table 4). These unidentified peaks are probably caused by higher hydrocarbons (C_mQ_n with m>3). From the IC-2 chromatogram (Fig. 16d) it can be clearly seen that all hydrocarbon peaks detected by the FID are tritiated. Due to the relatively large size of the ionisation chamber the structure of the C₃Q_n peaks as seen by the FID is smeared out in the IC-2 chromatogram.

The spectra presented in Fig. 16a-d were obtained after collection of the gas and before any treatment in the Impurity Processing (IP) system. The purpose of IP is to regain tritium from any tritiated compound [8]. Various techniques such as cracking of hydrocarbons on hot uranium or burning of hydrocarbons with oxygen to water are used in the JET AGHS.

Figure 16e shows the chromatogram after the gas was circulated a few hours over a uranium bed heated to 770 K. The peaks of the higher hydrocarbons (C_nQ_m with n>1), CO and CO₂ disappeared almost completely. Note that they are shown with an attenuation factor equal 1. The negative FID peak caused by tritium was a prominent structure and demonstrated that tritium

and hydrogen were liberated by cracking of hydrocarbons on hot uranium. The CQ_4 and the Q_2 concentrations were probably almost in equilibrium, so only absorption of hydrogen by a cold U-bed would lead to further reduction of the CQ_4 peak. The IC-2 spectrum (not presented) showed clearly only one peak for tritiated hydrogen.

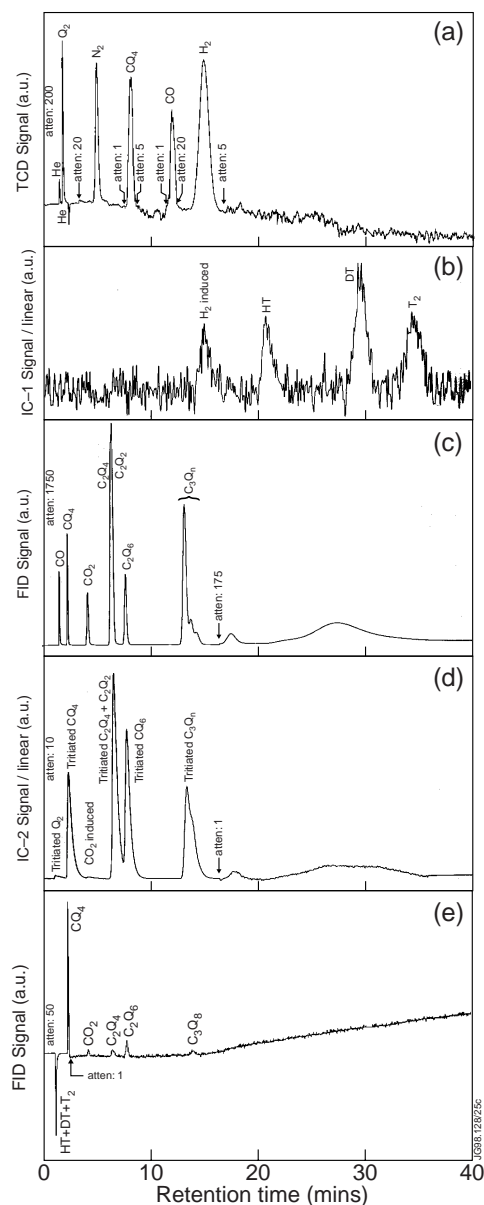


Fig. 16. TCD, IC-1, FID, IC-2 chromatograms (a-d) of gas collected in the Impurity Processing (IP) system and FID chromatogram (e) of the gas after circulation through hot uranium bed.

6. GENERAL PERFORMANCE OF THE AN-GC SYSTEM

The AN-GC has been in practice the workhorse of the analytical laboratory. The other analytical tools, Omegatron, residual gas analyser, and ionisation chambers, were rarely used. The number of analyses performed with AN-GC for the various subsystems of AGH-plant during DTE1 is given in Table 5. The total number of analyses is 318.

Table 5: Analyses performed with the analytical GC system for various AGH-subsystems from week 21, 1997 to week 6, 1998. PS stands for Product Storage, IS for Intermediate Storage, CF for Cryogenic Forevacuum, IP for Impurity Processing, GC for preparative Gas Chromatography, and CD for Cryogenic Distillation.

System	PS	IS	CF	IP	GC	CD	AGHS
No. of Analysis	88	58	10	93	48	21	318

Figure 1 shows that the operator can manually choose between two carrier gases, Ne and He. Due to the fact that the majority of analyses was performed for hydrogen isotope mixtures, Ne was the main carrier gas used. For analysis of samples from IP the FID was used to characterise the hydrocarbon concentrations. N₂, O₂ and Ar were of no great importance, were analysed with neon as carrier gas and did not require the use of He for a more accurate analysis.

The AN-GC is in principle capable of performing water vapour analysis when VX-2 is switched to bypass the molecular sieve filled columns COL-1 and COL-2. No water analysis was performed during and after DTE1 because in most cases the samples to be analysed were supplied from U-beds and were expected to be ultra-dry. IP and ED are the AGHS subsystems where water is handled. In IP the water vapour is trapped in a cold trap and processed in hot U-beds. In ED special humidity gauges exist for water vapour detection down in the low ppm range. The transfer of water vapour from these systems to AN was not done to avoid contamination of the long pipe work with water.

Valco valve VX-3 was installed for injection of helium and hydrogen only into COL-4. All other gases bypass the column to avoid adsorption and condensation of these gases on the Fe doped Al₂O₃ at 77 K in COL-4. During and after DTE1 VX-3 was not operated. All gases eluting from COL-3 were allowed to enter COL-4 because no decrease of efficiency of COL-4 was observed during trace and full tritium commissioning. This statement remained correct at the end of the DTE1 clean-up phase. The reason for this behaviour is that COL-4 was often allowed to warm up. In fact COL-4 is only cooled to 77 K when needed for analysis. During these warm-up periods trapped impurities are released again albeit at a slow rate.

Most analysis during and after DTE1 with AN-GC were run in the simplest configuration, making as few changes to the operating system and software as possible and operating the smallest number of valves possible with the intention to receive accurate analysis and to preserve the existing equipment as long as possible.

The AN-GC has worked very reliably since its assembly at JET in 1991 and was in almost continuous use. The data control system of the AN-GC was improved by adding a PC which controls the gas chromatograph, collects the data and allows the usual data manipulations.

Only the inner parts of the FID and the catalyst in the methaniser had to be exchanged once.

The catalyst lost its efficiency in converting CO and CO₂ to methane after exposure to larger amounts of unsaturated higher hydrocarbons.

All columns and detectors worked as expected with the exception of the FPCDs.

7. DISCHARGE ROUTES FOR THE GASES FROM AN-GC

7.1 Discharge to ED

The gases used to operate AN-GC were transferred via a metal bellows pump to ED. The inlet of ED is equipped with a 1 L ionisation chamber. The response of this ionisation chamber is plotted as a function of time in Fig. 17 for gases moved from AN-GC to ED during the analysis of a 'pure' T₂ sample. Two peaks are easily seen which are attributed to tritium leaving system 1 and 2 of AN-GC. The time difference between these two peaks is equal to the time between the main peaks of IC-1 and IC-2 in Fig. 11.

7.2 Discharge into stack

The discharge route to stack reduces the tritium collection in ED. This was especially important during the trace tritium and full tritium commissioning and the first part of the DTE1 [1] phase when the AN-GC was the main source of the tritium collected in ED. Due to this discharge route into the atmosphere the tritium injection into ED could be kept so low that the water collected in ED was partially discharged within the JET discharge authorisation [9].

The tritium discharged via B4 into the stack existed as tritium or hydrocarbon gas in the carrier gas because any tritiated water was gettered in COL-1 and 2 of system 1. Tritium as gas gives a lower dose than in water form and this route was therefore the best practical environmental option (BPM).

Figure 18 shows the signal of the tritium detector in the stack during the analysis of a pure tritium sample in AN-GC. Two peaks are clearly visible. The first peak is due to the HT+DT+T₂ exiting from system 2 and the second peak is due to mainly T₂ eluting at far higher retention times from COL-4 of system 1. The time difference between the two peaks in Fig. 18 is equal to the difference in retention times of the main peak for IC-1 and IC-2 shown in Fig. 11.

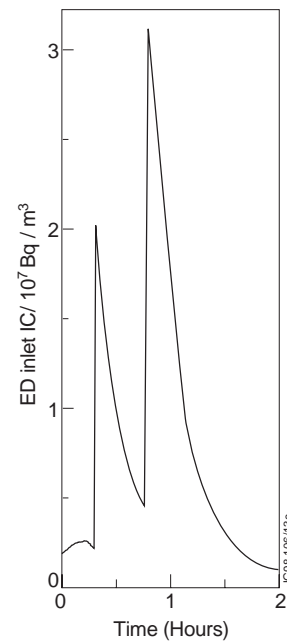


Fig. 17. Response of ED inlet ionisation chamber to exhaust gases from the AN-GC during the analysis of a 'pure' tritium sample.

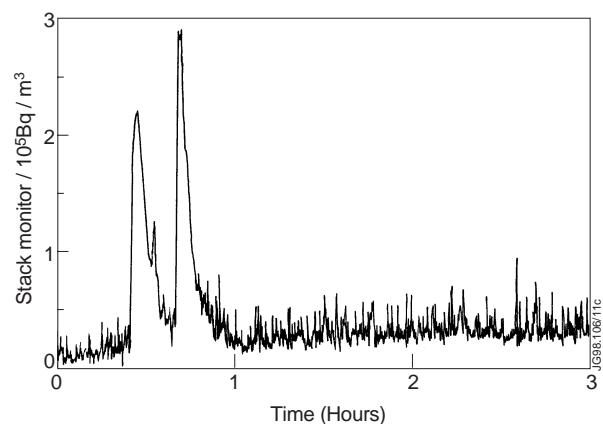


Fig. 18. Response of tritium monitor in the stack of J-25 to exhaust gases from the AN-GC during the analysis of a 'pure' tritium sample (ED system bypassed).

7.3 Further reduction of tritium released into stack or ED

The tritium discharges to ED or into the stack could be stopped by the addition of a dedicated clean-up system to the AN-GC to recover the tritium. Due to the gas streams of synthetic air, H₂ and P10 gas (10% CH₄/ 90% Ar) for the FID and FPCD, respectively, such a clean-up system would require a major modification of the pipe work of the AN-GC and connected systems to treat the various exhaust gases separately.

The simplest way of reducing the injection of tritium into ED or stack was found to be the use of small pressures in the injection loop if they still allowed adequate sensitivity. In most cases only un-compressed samples with pressures below 0.1 MPa were analysed. The pressure of compressed samples is approximately 0.3 MPa.

8. CONCLUSIONS

This paper presents calibration factors for the gas species detected by TCD and FID which were used for quantitative analysis. In addition, methods and calibration curves obtained during the DTE-1 phase are given.

The installation of two new ionisation chambers was extremely successful with respect to the higher sensitivity, the lower noise levels and the easier identification of the hydrogen molecules.

The negative FID peak for Q₂ can also be used for calibration purposes of the total tritium amount. The negative peaks for tritiated hydrocarbons seen very clearly with the flame off lead to underestimation of the true concentrations of hydrocarbons.

All gas species of interest could be detected and analysed by the AN-GC. The only exception was Q₂O which for most diagnostics is a very difficult gas to be analysed quantitatively. Furthermore, very broad peaks were detected for gas mixtures processed in IP with the FID which are detected at very long retention times. Their identification has not been possible so far and would require the preparation of calibration mixtures with far heavier hydrocarbons.

A further advantage of the AN-GC system in comparison to Omegatrons or quadrupoles is that the composition of the gas mixture is not changed by the analysis itself as e.g. cracking products are created by the very hot temperatures of filaments in residual mass spectrometers.

The main disadvantage is that an AN-GC run takes about 40 minutes.

The gases processed in the various AGH-subsystems were mainly analysed by the AN-GC. The AN-GC played a very important role in checking the various processes and the quality of the different products made in the AGHS during tritium commissioning, DTE-1, the clean-up phase [10] and the return to D-D Operations in JET experiments.

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