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BY

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PLASMA PHYSICS LABORATORY



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HYDROGEN ISOTOPE ANALYSIS BY QUADRUPOLE MASS SPECTROMETRY

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ABSTRACT

The analysis of isotopes of hydrogen (H, D, T) and helium (3 He, 4 He) and selected impurities using a quadrupole mass spectrometer (QMS) has been investigated as a method of measuring the purity of tritium qas for injection into the Tokamak Fusior Test Reactor (TFTR). A QMS was used at low resolution, m/Am + 150, for quantifying impurities from m/q = 2 to 44, and at medium resolution, m/ Δ m \sim 600, for determining concentrations of HD in 3 He, and 4 He in H m/D₂. gases to be studied were introduced into the QMS housing through a molecular leak from a 3-liter inlet volume. A linear relation between peak height (and area) and component partial pressure was verified and QMS sensitivities for various ions were determined. Increases in background hydrocarbon and water vapor were measured whenever hydrogen (any isotope) was present in the QMS housing. Baking and H2 glow discharge conditioning (GDC) minimized these effects. Low resolution, computer-controlled QMS analysis of D, and T_2 standard mixtures after H_2/GDC treatment demonstrated

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the capability to verify tritium purity at the 98 at. % level as required in the TFTR application. Detection limits quoted were 0.005 mole % for Ar; C.02 mole % for CT₄, O₂ and CO₂; 0.05 mole % for N₂/CO; and between 0.03 and 0.25 mole %, depending on the sample, for hydrogen and helium species.

INTRODUCTION

Assurance of low impurity levels in tritium for injection into the Tokamak Fusion Test Reactor (TFTR) requires analytical instrumentation. Tritium purity specifications 1 limit the $z \le 2$ impurities to <2.0 at. %, the $z < z \le 10$ impurities to <0.1 at. %, and the z > 10 impurities to <0.01 at. % in order to minimize plasma radiation losses from non-hydrogenic species. 2

An analytical system using a quadrupole mass spectrometer (QMS) has been developed to sample and analyze tritium gas at critical points in the Tritium Storage and Delivery System (TSDS). The response of the QMS to the various isotopic species of hydrogen and helium and impurities was studied in a tritium mass spectrometry laboratory concurrent with the construction of the TSDS unit. The results of this QMS evaluation are reported here.

The variety of ion species in the mass range from m/q=2 to 6 is shown in Table I. The principal impurities with $2 \le 2$ expected in high-purity tritium are: helium-3 as a natural decay product of tritium; HT as a contaminant accumulated during storage and handling of the gas; and DT remaining from the original isotopic separation. With these species dominant at m/q=3, 4 and 5, respectively, a low resolution QMS operation is acceptable for routine analysis of impurities with $2 \le 2$. Whenever HD, $\frac{4}{2}$ He, or $\frac{1}{2}$ Dis expected as the component of a mixture, operation of the QMS at high resolution

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(Table I) is desired to identify species at m/q = 3 and 4. Impurities with Z > 2 expected in high-purity tritium gas are N_2/CO , Ar, CO_2 , CT_4 , HTO, and T_2O .

In this paper we demonstrate that a QMS operated in a low-resolution mode, under computer control, can analyze tritium within the defined specifications. Also discussed are the high resolution capabilities and limitations of the QMS in separating the various ion species at m/q = 3 (3 He and HD) and 4 (4 He, HT and D_2).

EXPERIMENTAL ARRANGEMENT

The QMS unit consists of an Extranuclear Laboratories, Inc. quadrupole mass spectrometer having a specially selected mass filter with 9.5-mm rod diam which is operated at 5.1 MHz. This spectrometer is capable of producing high resolution or high transmission over the mass range of 0-64 amu. The QMS is operated as a residual gas analyzer in a 10-cm diam housing. Ions are produced with an axial ionizer—ing a tungsten filament. The transmitted ions are detected with a 14-stage CuBe box-dynode multiplier. The vacuum system with test gas manifold is shown schematically in Fig. 1. Pure gases and standard mixtures are introduced into the 3-liter holding volume using the capillary sampling manifold or the gas pipette.

Standard mixtures not containing tritium were prepared by a partial-pressure mixing system^{4,5} and standard tritium-containing mixtures were made by a partial volume mixing system.³ Compositions of tritium standards are computed for each day used and purity is monitored using routine quantitative mass spectrometry.⁶

The quadrupole and housing were conditioned for high vacuum operation by baking to >100°C followed by a combination of baking and glow discharge conditioning (GDC) 7 using a continuous flow of $\rm H_2$ through the housing at a nominal pressure of 13.3 Pa (0.1 Torr). A discharge current of 250 mA was produced by applying a voltage of 400 Vdc through a 500% load resistor to the anode (Fig. 1). All other components of the QMS except the CuBe detector were grounded. Background pressures between analyses of hydrogen-helium mixtures following baking and $\rm H_2/GDC$ are in a low $10^{-6}\rm Pa$ (10^{-8} Torr) range.

Low-resolution operation of the quadrupole unit was achieved by adjusting the ion focusing potential and resolution to obtain minimum peak full-width-at-half-maximum (FWHM) consistent with minimal ion attenuation for argon at mass 40. This resulted in Δm values of 0.25, 0.6, and 0.7 amu for m/q=4, 28, and 40 amu, respectively, thus providing complete separation of all masses for independent measurement of peak intensities. In this low resolution operation, 100-eV ionizing electrons with a total emission of 0.85 mA were used. The ion energy was set at 15 eV. All other conditions, such as detector voltage, were kept constant during day-to-day, quantitative measurements.

High resolution operation of the QMS required sweeping the mass range with a voltage ramp from an oscilloscope over a narrow mass range. Resolution and ion energy were adjusted to obtain optimum peak separation. Ion energies and currents of <2 eV and 2.4 mA,

respectively, were required to achieve the best resolution and sensitivity.

RESULTS AND DISCUSSION

High Resolution

High resolution scans at mass 3 and at mass 4 are shown in Figures 2a and 2b-2d, respectively. The spectrum in Fig. 2a is a typical high resolution scan of a 75-25 mole % ³He/HD mixture excited by 100-eV ionizing electrons. The ³He/HD peak height ratio was measured to be 1.7. Thus, the ratio of the ionization probabilities of hydrogen to helium is calculated to be 1.8, which agrees well with the low resolution QMS operation. The resolution and peak shape allow for a quantitative diagnostic measurement of ³He and HD with abundance ratios of <20 that often occur with D/T mixtures. Helium-3 sensitivity for the signal-to-noise ratio shown in Fig. 2a is 0.004 V per Torr of inlet pressure compared with 70 V per Torr measured in the low resolution QMS operation described in the next section of this paper. Sensitivity decreases rapidly with increasing resolution 8 such that the resolution available for a particular diagnostic application is limited by the partial pressure in the QMS housing of the component being studied.

The resolving power of the QMS at m/q = 4 was tested by preparing mixtures of 4 He, HT and D_2 in the inlet system. The resolution requirements for component separation are given in Table I. No equilibrated mixture high in HT was available; instead, HT was prepared by mixing 113 Pa (0.85 Torr) of T_2 with 113 Pa (0.85 Torr)

of H2 in the 3-liter inlet volume. This produced an approximately 0.04 Torr inlet equivalent of HT by exchange reactions within the ion source; to this, 13 Pa (0.1 Torr) of 4He and small amounts of D_2 were added, giving the spectra shown in Fig. 2b. The D_2^{+} shoulder on the HT can be seen in reference to the spectrum of pure D, and 4He shown in Fig. 2d. As D, is added to the inlet, the spectrum in Fig. 2c clearly shows the broadened peak of an unresolved doublet. The resolution [m/Am(FWHM)] shown in Fig. 2d was calculated to be 650. An ion energy of 1.5 eV was used; a sensitivity for ⁴He was measured to be 0.035 V/Torr. Higher resolutions are achievable, but the m/Am of 650 was chosen so as to produce useful signal-to-noise ratios for examining this low-level HT-D doublet expected in TFTR tritium specimens. The sloping baseline in Figures 2b and 2c is caused by a beta-induced current from the multiplier that is directly proportional to the partial pressure of tritium: as the tritium sample is depleted, the baseline decreases.

A limitation of high resolution by QMS is the cutoff of ion transmission that occurs with the next mass higher than the one under study; for example, maximizing on mass 4 causes mass 5 to disappear.

High resolution analysis also requires manual adjustments between each peak making this mode of analysis impractical as a routine analytical procedure for all masses. The resolution capability demonstrated here for the 9.5-mm diam rod size is arbitrarily classified as "medium resolution" from previous work³ on other mass spectrometers. Medium resolution (~600) denotes separation of ³He from HD and ⁴He from HT/D₂. If higher resolution diagnostic work is needed

where HT and D_2 must be uniquely identified, the 16-mm diam rod size mass filter with a 0-23 amu mass range has been shown $\frac{10}{100}$ to produce a resolution in excess of 1000 at mass 4 and would be a better choice.

Low Resolution

a. Background

The background mass spectrum of the QMS following approximately 140 hours of baking and GDC is shown in Fig. 3a. The base pressure in the QMS housing was 1.6×10^{-6} Pa $(1.2 \times 10^{-8}$ Torr) primarily from residual H₂ with low levels of H₂O, CO and CO₂ at less than 10^{-9} Torr partial pressure. However, Figures 3b through 3d show that exposure of this clean system to an inlet pressure of 80 Pa (0.6 Torr) of H₂, D₂, and T₂, respectively, produces large increases in background components. Exposure of the system to helium or argon does not produce these increases. There are at least four processes occurring when hydrogen is introduced:

- (1) Displacement of adsorbed gases, mainly $\mathrm{H}_2\mathrm{O}$, CO, and hydrocarbons.
- (2) Isotopic exchange between the adsorbed species and the hydrogen isotope introduced.
- (3) Hydrogenation and displacement of hydrocarbons as seen in the C_1 , C_2 , and C_3 hydrocarbon mass regions.
- (4) Trimer formation from molecular collisions in the gas phase seen at mass 6 (D_3^+) for D_2 and masses 7, 8, and 9 for the D/T mixture.

These effects have been noted in a number of previous studies involving hydrogen isotopes. Effects (1)-(3) are particularly troublesome if the mass spectrometer ion source is fabricated from 300 series stainless steel. 11,12 Recent studies of the surface chemistry of stainless steel under low energy 0 + $^{+}$ bombardment have shown that the surface carbon hydrogenates to form volatile molecules (CH₄, C₂H₄, and CO) and certain components of the surface oxides (Fe₂O₃, FeO and Fe(OH)_X) are reduced to liberate $^{+}$ 2O. $^{13-17}$

The mass spectra in Fig. 3 show the residual gas production in the ion source with the introduction of various hydrogen isotopes, in addition to showing the result of isotopic exchange between the introduced gas and the previously adsorbed isotope. Conditioning of this ion source (i.e., with the $\rm H_2/GDC$ procedure) can reduce these effects substantially. The total residual gas production from ion source effects with hydrogen present was reduced from $\cdot 100\%$ of the partial pressure of $\rm H_2$ introduced to $\cdot 5\%$ after 140 hours of baking and $\rm H_2/GDC$ cleaning. We note that no significant residual gas production ($\cdot 50$ ppm) was seen in a cycloidal mass spectrometer used for similar studies; that instrument employed an ion source fabricated from a Ni-Cr alloy, Nichrome-V. 18

The fourth ion source effect noted above, the formation of the trimer, is also a subject of previous studies. ¹⁹ We find the D_3^+ formation is linearly dependent on D_2 pressure with a production ratio, D_3^+/D_2^+ , of 2 x 10⁻⁵ per each 10⁻⁶ Torr of D_2 pressure in the QMS housing.

b. Computations

Composition of the gas analyzed is calculated assuming the total gas pressure as the sum of partial pressures

$$x_{i} = 100^{\circ}PP_{i}/\Sigma PP_{i} , \qquad (1)$$

where \mathbf{X}_{i} is the mole-percentage of the ith component, and its partial pressure, PP_i, is computed from the mass spectral ion intensities, instrument sensitivities, \mathbf{S}_{i} , and other calibration factors, given in the equation below.

$$PP_{i} = \left[\left[PA_{i} \left(\text{net} \right) - BG_{i} - \Delta PS.e^{\frac{Lt_{i}}{\sqrt{6}}} \right] e^{\frac{Lt_{i}}{\sqrt{6}}} - \sum_{j} C_{ij} \right] / S_{i}. \quad (2)$$

In Eq. 2, the net peak height measured with the sample present is corrected for background, BG_{i} , and tritium induced baseline shift, ABS, then decayed using the leak rate for mass 6, L/ 6, and time, t_{i} , and next exponentially corrected for flow from the inlet for a time, t_{i} . Finally, the correction for fragments and multicharged ions from higher mass components is made with C_{ij} of the form

$$C_{ij} = R_{ij}.PH_{j} (corrected).$$
 (3)

The factor R_{ij} is the fragmentation or multi-charged production ratio for ion intensity at mass M_i due to parent mass M_j . All this implies that computations begin at high mass and proceed toward low mass.

A measure of stability and quality of calibration factors is given by the closure equation

$$C/M = \sum_{i} PP_{\underline{i}}/P_{IN} \quad (4)$$

which is the ratio of the sum of partial pressures to the inlet pressure, P_{IN} , for a given sample. This "calculated to measured", C/M, value is theoretically unity for perfect calibration.

c. Calibration Factors

The leak rate for the 'an-hole molecular leak was determined by monitoring P_{1N} for D_2 as a function of time as the gas leaked out. The slope of the ln P_{1N} versus time curve gives a value of L/2 from which L = 0.0016 (amu^{1/2} . sec⁻¹) was determined.

Sensitivity values are calculated by solving Eqs. 1 and 2 for S_1 using known gas compositions, usually pure gases. Explicit in the form of Eq. 2 is the assumption that S_1 is a constant, relating corrected peak height to component partial pressure over a wide range of partial pressures. This linearity assumption was checked for D_2 , T_2 , ^3He , and N_2 . Sensitivities for D_2 and D_3 showed random variations of less than 2%, relative over the range from 0.05 to 0.8 Torr of inlet pressure; D_3 showed the same random variation over the range from 0.05 to 0.5 Torr. Delium-3 sensitivity monotonically incressed over the range from 0.05 to 0.6 Torr with the sensitivity at 0.6 Torr 5.5% higher than at 0.05 Torr. This may

indicate a decrease in ${}^{3}\text{He}$ pumping speed of the turbomolecular pump at housing pressures of 3 x 10^{-5} torr and higher.

Fig. 4 shows two sets of sensitivities as functions of mass profiles for two different preparations of low resolution QMS operation. Each set of sensitivity factors was stable, while each set of QMS control potentials remained fixed. Over a period of 12 days, a day-to-day variation of absolute sensitivities of less than 8% relative standard deviation was measured for H₂, D₂, T₂, ³He, ⁴He, Ar, and N₂. Furthermore, the variations in sensitivities were highly correlated, implying that the cause of day-to-day shift in sensitivity is a factor common to all species. This correlated behavior suggests that, once a sensitivity profile is determined, the relative sensitivities will vary more as the 2% within-day fluctuations than as the 8% absolute variation. The definition of mole percentage in Eq. 1 is referenced to the sum of partial pressures rather than inlet pressure, such that only relative sensitivities are required for accurate analysis.

Fragmentation ratios of importance to high-purity tritium analysis are $D^+/D_2^+ = 0.0124 \pm 0.0005$, and $T^+/T_2^+ = 0.010$ (estimated). A ratio for $Ar^{2+}/Ar^+ = 0.25 \pm 0.01$ was also measured. The D_3^+ trimer described earlier also acts as an interference peak.

d. Sample Analysis

Standard mixtures were analyzed to test accuracy and calibration stability. A general observation was the need to condition the spectrometer after it sat idle or when the sample composition was changed. After two or three replicates of a sample, an asymptotic value close to the standard composition was reached. Examples of

analyses of standard mixtures are given in Table II. Standards A and B are high-purity D, with known amounts of N, and Ar impurities added. The HD and DT values measured are higher than the standard, indicating exchange reactions between sample D2 and the adsorbed H and T present in the source region. At the time of this analysis, the surfaces inside the OMS housing had been randomly exposed to all isotopes of hydrogen for four months so that they held a significant amount of tritium. The peak at mass 6 is D3 at a level consistent with a sample pressure of 0.3 Torr in the inlet. A small air leak known to be in the gas pipette (Fig. 1) produced a 0.05 mole % oxygen contamination and associated 0.18 mole % nitrogen. The corrected 28 peak (from air) is still biased high by 40% relative for Standard A, and Ar is high by a similar amount; the trend is repeated for Standard B. The implication is that sensitivities determined at high pressures for N2 and Ar may not apply at low levels. Standard C samples were drawn using the capillary sampling system which contributes no measurable contamination. The standard consists of high purity tritium with 211 days of helium ingrowth added to the 0.23 mole % of helium present at fill time. The levels of HT and methane present in the mix are typical of tritium stored in a "clean" stainless steel system for 200 days. 20 Again, the minor hydrogen species HT and DT are biased high with respect to the standard, implying some exchange reactions. However, the level of bias is tolerable for qualifying tritium purity. Accuracy and detection limits of 0.005 mole % for Ar and 0.02 mole % for the ${\rm CT_4}$, ${\rm O_2}$,and ${\rm CO_2}$ components satisfy program goals. The only problem

is mass 28. Formation or displacement of CO, N_2 , or C_2H_4 from surfaces at a level of 0.08 mole % occurs whenever pure D_2 , T_2 , or H_2 is introduced, but a value of 0.003 is measured when 3He or 4He is analyzed. Using a systematic correction of 0.08 \pm 0.05 mole % for mass 28, a detection limit of 0.05 mole % is quoted for mass 28 in the presence of hydrogen. The detection limit for H, D, and T molecular species and He in the presence of N_2 or Ar is estimated at 0.03 mole %. For samples containing high purity D_2 or T_2 , isotopic exchange reactions producing HD, HT,or DT from QMS surfaces limit detection to 0.25 mole % for species in the actual sample.

CONCLUSTONS

Operating a quadrupole mass spectrometer as a residua gas analyzer is appropriate for analyzing within the specifications of TFTR, the tritium purity of gas leaked into the QMS housing. After initial calibration, recalibration is not needed until changes are made in resolution or other control settings. After such a change, pure qases or a stable multi-component standard mixture plus tritium could be used to establish the sensitivity profile for the new settings. Stability can be monitored using the C/M ratio, where C is the calculated sum of partial pressures, and M is the measured inlet pressure. Variations in C/M within the range of 0.92 and 1.08 are expected from day to day with no loss of analysis quality. Larger deviations warrant recalibration. Isotopic exchange effects observed in this study make the monitoring of water vapor and hydrocarbon peaks for process gas information highly questionable. Detection limits for Ar and CO, are limited by hydrocarbon spectra fluctuations in the presence of hydrogen. The only effective solution to the

fluctuations is reduction of hydrocarbon contamination by \mathbf{H}_2 glow discharge conditioning.

Medium resolution operation providing useful separation of 3 He from HD and 4 He from HT/D $_2$ is achievable using the mass filter tested by manual adjustment of resolution and ion energy controls for each nominal mass.

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m/q	ION SPECIES*	RESOLUTION (PAIR)		
2	D ⁺ , (H ₂ ⁺)	1302 (D-H ₂)		
3	$^{3}\text{He}^{+}$ + $^{+}$ $^{+}$ $^{+}$ $^{(HD^{+})}$ $^{(H_{3}^{+})}$	512 (³ He - HD)		
4	$(^{4}He^{+})$, HT^{+} , (D_{2}^{+}) , $(H_{2}D^{+})$	189(⁴ He - HT), 930 (HT - D ₂)		
5	DT ⁺ , (H ₂ T ⁺), (D ₂ H ⁺)	856 (DT - D ₂ H)		
6	T2 ⁺ , (D3 ⁺)	591 (T ₂ - D ₃)		

^{*}Ion species in parentheses are not expected in high purity tritium samples.

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TABLE II

TYPICAL ANALYSES OF STANDARD MIXTURES

Mass-	Standard A		Standard B		Standard-C	
Component	QMS (±S)	Standard	QMS (±S)	Standard	QMS (<u>+</u> S)	Standard
2- н ₂	0(0.02)	0	0.01(0.64)	0	0 (0.02)	O
3- ³ He/HD	0.23(0.06)	0.08(HD)	0.34(0.05)	(DH) 80.0	6.1(0.08)	6.40 (³ He
4-HT/D ₂	97.48(0.15)	98.53(D ₂)	99.14(0.14)	99.78(D ₂)	0.54(0.07)	0.54(HT)
5-DT	0.09(0.06)	0	0.02(0.01)	0	0.12(0.02)	0.08
6-T ₂	0.03(0.02)	0	0.06(0.02)	0	92.70(0.09)	92.94
24-ct ₄	0(0.02)	0	0(0.02)	0	0.04(0.02)	0.035
28-co/n ₂	2.06(0.12)	1.35	0.36(0.14)	0.135	0.09(0.04)	0.002
28-(Air)	[1.88(0.12)]	[1.35]	[0.16(0.14)]	[0.135]		
32-0 ₂	0.050(0.002)	<0.002	0.055(0.004)	<0.002	0 (0.006)	<0.002
40-Ar	0.059(0.005)	0.044	0.005(0.004)	0.004	0.002(0.004)	<0.002
44-co ₂	0.002(0.002)	<0.002	0.010(0.006)	<0.002	0 (0.01)	<0.002
Replicates	6		4		4	

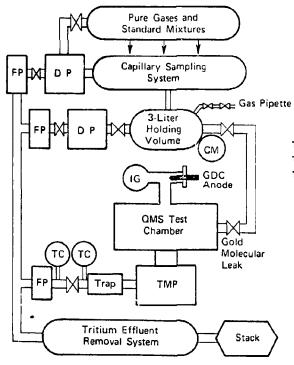
Figure Captions

- Shows the vacuum system and test gas manifold arrangement Fig. 1 for evaluating QMS response to all hydrogen and helium isotopes and selected impurities. Gas pressure in the inlet holding volume is measured with a 133 Pa (1 Torr) full scale capacitance manometer. For gas pressures greater than 133 Pa, necessary for high resolution studies, a variable reluctance pressure transducer is used. Gas flows into the QMS housing through a tenhole gold molecular leak with a conductance of 2.0 cm^3/sec for T_2 . For a T_2 sample of 40 Pa (0.3 Torr) in the inlet, the flow rate through the leak is 6 x 10^{-4} Torr-liter/sec which produces $= 9 \times 10^{-4}$ Pa (7×10^{-6} Torr) housing pressure. Using an ion gauge correction factor of 2.2 for hydrogen, a pumping speed of 40 liters/sec for T2 is computed for the nominal 110 liters/ sec turbomolecular pump. Two-stage rotary forepumps sealed for tritium service are used as roughing pumps.
- Fig. 2 Illustrates the high resolution scan of a 75/25-3He/HD mixture with calculated resolution M/ΔM (FWHM) of 750.

 This spectrum was made using a 133 Pa (1 Torr) inlet pressure of the mixture which produces a 7 x 10⁻³ Pa (5 x 10⁻⁵ Torr) pressure in the QMS housing. The ion energy was set at 1.12 V and ion focusing potentials were adjusted to produce the best ³He-HD separation and intensity.

 Deconvolution of the peaks indicated a resolution, m/Δm, of approximately 750, where Δm is the FWHM. High resolution scans of mixtures of ⁴He, HT and D₂ are shown in spectra (b) and (c), and ⁴He-D₂ in spectrum (d). Calculated resolution is 650.

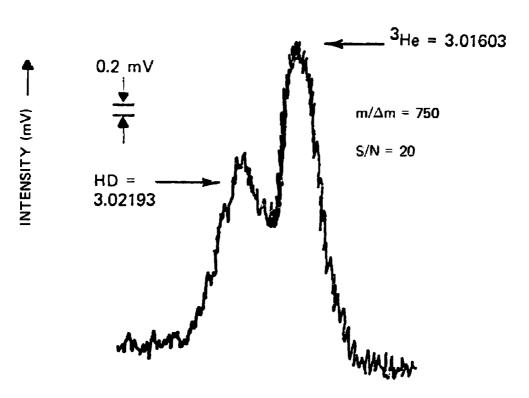
- Fig. 3 Shows the low-resolution mass spectrum of background after 140 hours of $\rm H_2/GDC$; and mass spectra during exposure to 0.6 Torr inlet pressure of $\rm H_2$, $\rm D_2$, and D/T mix (b, c, and d, respectively). All spectra are on the same scale; arrows indicate off-scale peaks.
- Fig. 4 Illustrates the sensitivity versus m/q profile for two different QMS control settings (x and ●). Helium sensitivities are denoted by circles, and estimated or interpolated sensitivities are indicated by squares.



THIS QMS EVALUATION UTILIZED
THE FACILITIES OF MOUND'S
TRITIUM MASS SPECTROMETRY LAB

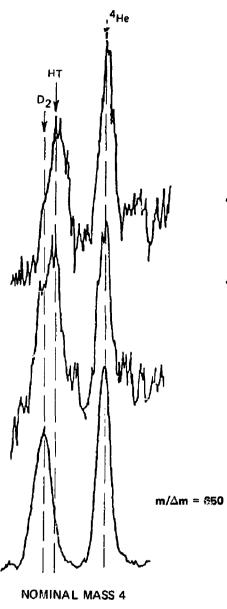
Fig. 1. (PPPL-81X2046)

USEFUL HIGH RESOLUTION AT MASS 3 IS PRODUCED



NOMINAL MASS 3

Fig. 2a. (PPPL-81X2047)



- BASELINE SEPARATION OF
 4He -- HT/D2 IS PRODUCED.
- → THE SHAPE OF THE HT/D₂ PEAKS INDICATE PRESENCE OF BOTH HT AND D₂ IN DIFFERENT AMOUNTS

Fig. 2b,c,d. (PPPL-81X2048)

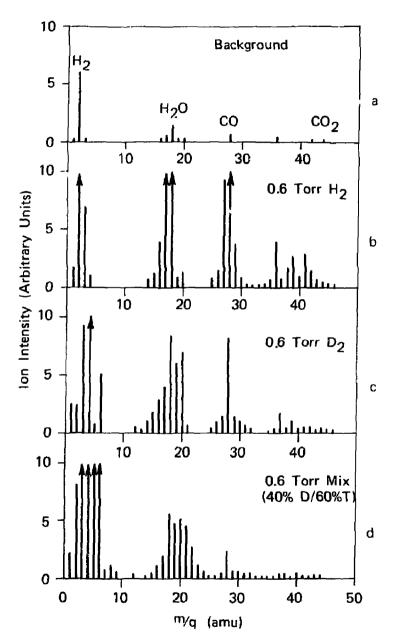


Fig. 3. (PPPL-81X2045)

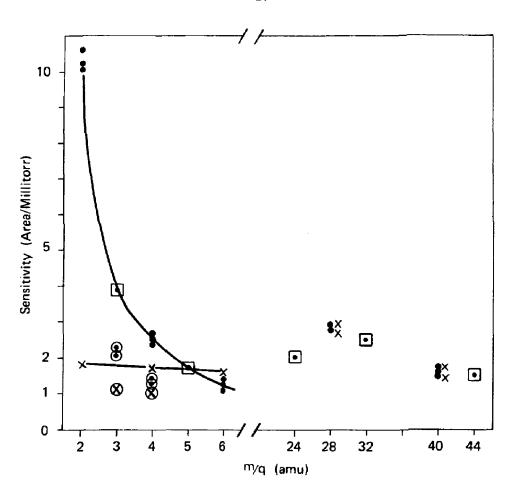


Fig. 4. (PPPL-81X2049)