United Kingdom Atomic Energy Authority

RESEARCH GROUP

Preprint

# THE ABSOLUTE CALIBRATION OF GAUGES FOR TRITIUM GAS IN THE PRESSURE RANGE $10^{-8}$ TO $10^{-5}$ TORR

M. R. BARNES
A. GIBSON

Culham Laboratory
Abingdon Berkshire

1968

CLM – P 169

# THE ABSOLUTE CALIBRATION OF GAUGES FOR TRITIUM GAS IN THE PRESSURE RANGE $10^{-8}$ TO $10^{-5}$ TORR

by

M.R. BARNES

A. GIBSON

(Submitted for publication in Vacuum)

## A B S T R A C T

A procedure for the absolute calibration of vacuum gauges for tritium gas is described. The method depends upon measuring the radioactive decay current generated by a known volume of tritium. Calibrations, in the pressure range $10^{-8}$ to $10^{-5}$ torr, are presented for a mass analyser and an ionization gauge which are commercially available.

# C O N T E N T S

# 1. INTRODUCTION

The calibration of pressure measuring devices, for hydrogen and its isotopes, in the range $10^{-8}$ to $10^{-3}$ torr is of special interest to workers in the field of nuclear fusion[1]. In particular, experiments using the method of Rodionov[2] to investigate the containment of single particles in magnetic traps, require devices calibrated for tritium gas in the range $10^{-8}$ to $10^{-4}$ torr. This paper describes a method for the absolute calibration of pressure devices for tritium. The method is applied to the calibration of a quadrupole mass analyser and to a Bayard Alpert ionization gauge. The importance of any systematic errors in the calibration procedure is assessed by cross calibration (at around $10^{-4}$ torr) with a McLeod gauge, and with a ZnS(Ag) scintillation counter which in turn is calibrated against a standard source.

# 2. THE BASIS OF THE CALIBRATION

The number of tritium atoms/unit volume can be determined from the known decay rate of tritium[3] by measuring the current $(I_R)$ of $^3\text{He}^+$ ions generated in a known volume (V) by the radioactive decay of tritium. Conventionally this number density is expressed as a pressure $(P_t)$ at $273^{\circ}\text{K}$. Thus:

$$P_t = \frac{273k}{2\lambda e} \cdot \left( \frac{I_R}{V} \right)$$

where

    k   is the Boltzmann constant

    $\lambda$   is the tritium decay constant ($1 \cdot 791 \times 10^{-9}$ $\text{sec}^{-1}$)

    e   is the electronic charge

    $P_t$   is the equivalent partial pressure of molecular tritium, and will include contributions from all molecules in the system which contain tritium. The most important tritiated species are $T_2$ and HT; in our system all other tritiated species are negligible so that $P_t = P_{T_2} + \frac{1}{2} P_{HT}$.

If $I_R$ is the amps, $V$ in litres and $P_t$ in torr this equation may be written

$$P_t = 0 \cdot 494 \times 10^8 \left( \frac{I_R}{V} \right) \qquad \text{... (1)}$$

A suitable collection chamber for measuring this current is shown in Fig.1. The central rod (A) is made sufficiently negative so that it collects essentially all the $^3$He ions (maximum energy 3·3 eV) born in the cylindrical volume. The β-particles born in the decay (maximum energy 18 keV) will be essentially unaffected by the electric field and will be collected on the outer cylindrical electrode (B). The effect of any secondary electrons generated by the β's at the rod or at the cylinder can be made small by keeping the ratio of the rod radius to cylinder radius small. There will be a background current in the chamber due to tritium adsorbed on the electrodes, the largest effect being due to $^3$He$^+$ ions released from the cylinder and reaching the rod.

## 3. APPARATUS AND EXPERIMENTAL PROCEDURE

The gauge to be calibrated is compared with the collection chamber on a vacuum system with conductances such that no pressure differential can exist between the gauge and the chamber. The mass analyser can be calibrated directly, but the calibration of the ion gauge requires a separate estimate of the fraction of the total pressure which is due to tritium. The vacuum system is constructed of stainless steel with electrolytically polished vacuum surfaces. The joints are all metal using either gold rings, or copper gaskets. The system is shown schematically in Fig.2. Tritium gas is admitted to the system from a pyrophoric uranium source through a nickel leak. The gas pressure is usually established by varying the leak current with only the

1 litre/sec pump operating, but at the higher pressures it is possible to admit the required amount of gas, with no pumps operating, and then to switch off the leak current.

The collection chamber (Fig.1) has a stainless steel body electrically insulated from the vacuum system by a glass to metal seal. The collector rod is insulated from the body by a glass to metal seal incorporating a guard ring; the seal resistance is around $3 \times 10^{14} \Omega$. The collector current is measured with a vibrating capacitor electrometer capable of measuring less than $10^{-15}$A (input resistance = $10^{11} \Omega$). The chamber is operated with a collection voltage of 300 V, this being sufficient to collect substantially all the ions generated. Fig.3 shows the variation of collector current with voltage on the chamber. The collection chamber has a residual background current of $\sim 10^{-13}$ A, which increases to an equilibrium level on exposure to tritium, recovery takes from 12 to 24 hours. On a time scale of minutes this background is essentially constant, it can be elliminated by measuring the change in collector current when the tritium is pumped out of the system. The tritium is pumped out in a time of about one minute using the 50 litre/sec pump. Even at the lowest pressures measured the 'gas out' pressure was 10 times smaller than the 'gas in' pressure. The electrometer can be backed off to facilitate measuring this small change in current, the errors introduced by this procedure can be determined by repeating the, gas in-gas out, measurement several times, these errors are included in the error bars on Fig.5 and Fig.6. The absolute pressure is then determined from:

$$P_t = \frac{I_c}{S_{cc}} \qquad\qquad .. \ (2)$$

where $I_c$ is the change in collector current when tritium at pressure $P_t$ is pumped out of the chamber and $S_{cc}$ is the sensitivity of the

collection chamber calculated from (1) to be $2 \cdot 8 \times 10^{-8}$ A torr$^{-1}$ for our chamber.

The percentage of tritium in the vessel is typically, $50 \rightarrow 80\%$ with the 1 litre/sec pump in operation, and 30% with no pumps operating, the remaining gas is almost entirely hydrogen. The 1 litre/sec pump was on during the calibration of the ion gauge and mass analyser, but not during the McLeod gauge comparison (to avoid pressure gradients due to the impedance of the gauge connection) or during the scintillator comparison (because of sensitivity to the light emitted from the pump discharge). Fig.4 shows a typical mass spectrum.

The McLeod gauge, and scintillator probe comparison were made against a base pressure of $\sim 10^{-7}$ torr (total pressure). The ion gauge and mass analyser were calibrated against a base pressure of $\sim 2 \times 10^{-9}$ torr produced after bake-out of the vacuum system to $200^{\circ}$C for 24 hours and outgassing the gauges for 15 to 20 minutes.

## 4. VARIAN QUADRUPOLE MASS ANALYSER

The resolution $(M/\Delta M)$ of the mass analyser can be varied from 0 to 100, the sensitivity of the instrument decreasing as the resolution increases. The calibration reported here is for $M/\Delta M = 26$. The analyser ion current is measured directly on an electrometer. The sensitivity $(S_t)$ is determined from:

$$S_t = \frac{I_c}{P_t \cdot I_e}$$

where

$I_e$ = emission current.

$I_c$ = analyser current (including contributions from $T_2$ and HT).

$P_t$ is the tritium partial pressure obtained from (2).

The data is shown in Fig.5. The solid line is the best linear least squares fit. The sensitivity is $(2 \cdot 56 \pm 0 \cdot 02) \times 10^{-2}$ torr$^{-1}$ at pressures below $10^{-6}$ torr and falls to $(2 \cdot 24 \pm 0 \cdot 02) \times 10^{-2}$ torr$^{-1}$ at $10^{-5}$ torr. The error estimates are based solely on the scatter of the data about the best fit.

## 5. MULLARD IOG 13 ION GAUGE

The sensitivity $(S_t)$ of the ion gauge for tritium is determined from

$$S_t = \frac{I_c \cdot R}{P_t \cdot I_e}$$

where

$I_c$ = ion current

$I_e$ = emission current

$P_t$ = tritium partial pressure from (2)

$R$ = fraction of total pressure due to tritium

The quantity $R$ is determined from the mass spectrometer (A in Fig.2), the contribution to the total pressure from gases with masses greater than 6 is negligible, and we have assumed the instrument is equally sensitive for masses 2, 4 and 6.

Two calibrations were made at an interval of 3 months (the gauge being stored at atmospheric pressure for this time); the change in sensitivity between calibrations was about 4%, and some of this is accounted for by the fact that the two calibrations are over differentr pressure ranges. Both sets of data are shown in Fig.6. The solid and dashed lines are the best linear least squares fit to the two sets of data. For the combined data the sensitivity is $(4 \cdot 95 \pm 0 \cdot 06)$ torr$^{-1}$ at pressures below $10^{-6}$ torr and falls to $(4 \cdot 27 \pm 0 \cdot 06)$ torr$^{-1}$ at $10^{-5}$ torr, again the error estimates are based solely on the scatter about the best fit.

## 6. McLEOD GAUGE

The gauge was connected to the vacuum system through a liquid nitrogen trap and an all metal valve. The valve allowed the vacuum system to be isolated from the gauge when the trap was empty, to avoid

any possible entry of mercury vapour from the gauge. The conductance of the valve was sufficiently high, however, to make its contribution to the overall impedance of the gauge connection negligible.

The McLeod gauge is a total pressure gauge, the method of comparison is, therefore, to determine the proportion of tritium in the system as for the ion gauge calibration. The partial pressure of tritium $(P_{m_t})$ is then

$$P_{m_t} = Pm. \; R \quad \text{where } Pm = \text{McLeod gauge indicated pressure}$$

This quantity is to be compared with the tritium pressure $(P_t)$ obtained from (2).

Over the pressure range covered, the errors in measurement come mainly from the McLeod gauge. The results have been corrected for the effects of mercury vapour streaming[4] but capillary depression is expected to produce a 10% error at $P \sim 5 \times 10^{-4}$ torr.

The data is shown in Fig.7; the dashed line is for $P_{m_t} = P_t$ and the solid line is the best linear least squares fit to the data, it is:

$$Pm = (0 \cdot 9 \pm 0 \cdot 25) \, P_t + (0 \cdot 7 \pm 2) \times 10^{-5}$$

which is consistent with the two gauges indicating the same pressure within the experimental error.


## 7. ZnS(Ag) SCINTILLATOR PROBE

A scintillator on a quartz rod 25 cm long × 1 cm diameter, was prepared and calibrated using a standard tritiated polymer source[5]. The scintillator efficiency $(n_p)$ was measured before and after cross calibration with the collection chamber and was 15%. The probe was mounted on the vacuum system together with a geometry tube. The tube,

arranged concentrically around the probe, determines its working volume. The efficiency of the probe is then determined from:

$$n_g = \frac{Sg}{C}$$

where $Sg$ is the count rate, due to tritium gas, measured by the scintillator, and $C$ is the $\beta$ arrival rate at the probe (calculated from the known geometry and decay rate, using $P_t$ from (2)).

There is a possibility of error in the measurement due to 'prompt' adsorption on the tube and scintillator which is proportional to the tritium pressure in the system and which is removed immediately the tritium is pumped out. Such surface adsorption would produce a count which is independent of radius of the geometry tube, whereas the count due to the gas is approximately proportional to radius. Measurements with geometry tubes of varying radius are consistent with this hypothesis and provide an estimate of the magnitude of the absorption. Extrapolating the data to infinite tube radius gives an estimate for $n_g/n_p$. Measurements at a tritium pressure of $1 \times 10^{-4}$ torr give:

$$\frac{n_g}{n_p} = (1 \cdot 05 \pm 0 \cdot 3)$$

consistent with the chamber and polymer calibrations being the same within the experimental error.

## 8. SPURIOUS EFFECTS

There are a number of effects which might in principle give rise to systematic errors in the calibration procedure. We have considered: ionization effects due to the $\beta$-particles and any secondaries they generate, various types of tritium adsorption on the walls and collecting rod, effects due to X-ray production. All of these

are entirely negligible, except for ionization produced by $\beta$ particles (and subsequent secondary electrons) which arise from tritium adsorbed on the chamber wall. We have assessed this effect experimentally by adding hydrogen gas to the tritium; this increases any ionization effect but does not change the current arising from decay in the tritium gas. These measurements indicate that the sensitivities we quote should be increased by $(2 \pm 1)\%$.

## 9. CONCLUSIONS

We have made an absolute calibration of a Varian quadrupole mass analyser and a Mullard IOG 13 ion gauge for tritium gas in the range $3 \times 10^{-8} \rightarrow 10^{-5}$ torr. In both cases the sensitivity is essentially constant below $10^{-6}$ torr the values being $0 \cdot 026$ torr$^{-1}$ for the Varian instrument $(M/\Delta M = 26)$ and $5 \cdot 0$ torr$^{-1}$ for the IOG 13 gauge. These values differ by less than 10% from those obtained from the manufacturer's data. Both instruments show a decrease in sensitivity by about 14% from this value, in the range $10^{-6}$ to $10^{-5}$ torr. Since others[6] find no variation of sensitivity with pressure for hydrogen at pressures less than $10^{-4}$ torr, it is tempting to suppose that the observed fall off is due to an error in the calibration procedure; however, we are not aware of any effect which could cause a departure of this magnitude.

The internal accuracy of the sensitivities determined here is about $\pm$ 1% and the measurements with the McLeod gauge and scintillator suggest that any systematic errors are not greater than about 10%.

## 10. ACKNOWLEDGEMENT

The authors would like to thank Mr. G.W. Reid for his help and advice.

# 11. REFERENCES

1. Proc. I.A.E.A. Conference on Plasma Physics and Controlled Nuclear Fusion Research, Culham, 1965. Vienna, I.A.E.A., 1966. Publication STI/PUB/111.

2. RODIONOV, S.N. 'An experimental test of the behaviour of charged particles in an adiabatic trap', J. Nucl. En. Part C, 1, 1960, pp.247-252.

3. JONES, W.M. 'Half-life of tritium', Phys. Rev., 100, 1955, pp.124-125.

4. ISHII, H. and NAKAYAMA, K. 'A serious error caused by mercury vapour stream in the measurement with a McLeod gauge in the cold trap system', Trans 8 AVS Nat. Vac. Symp. 1961, 1, pp.519-24, Pergamon, 1962.

5. POWELL, B.A. 'The counting efficiency for $\beta$-particles of ZnS(Ag) and glass scintillators and the adsorption of tritium on the scintillators and on stainless steel', Plasma Phys. 10, 1968, pp.79-82.

6. SCHULZ, G.J. 'Characteristics of the Bayard-Alpert ionization gauge at pressures above $10^{-5}$ mm Hg', Appl. Phys. 28 (10), 1957, pp.1149-1152.

Vibron 62A electrometer amplifier

Input resistance

Glass to metal seal

Guard ring

Ion collector rod

300V

15·2 cm.

0·54cm.dia.

Ⓐ

Ⓑ

Baffle

10·8cm.dia

Screening

Glass to metal seal

Vacuum system
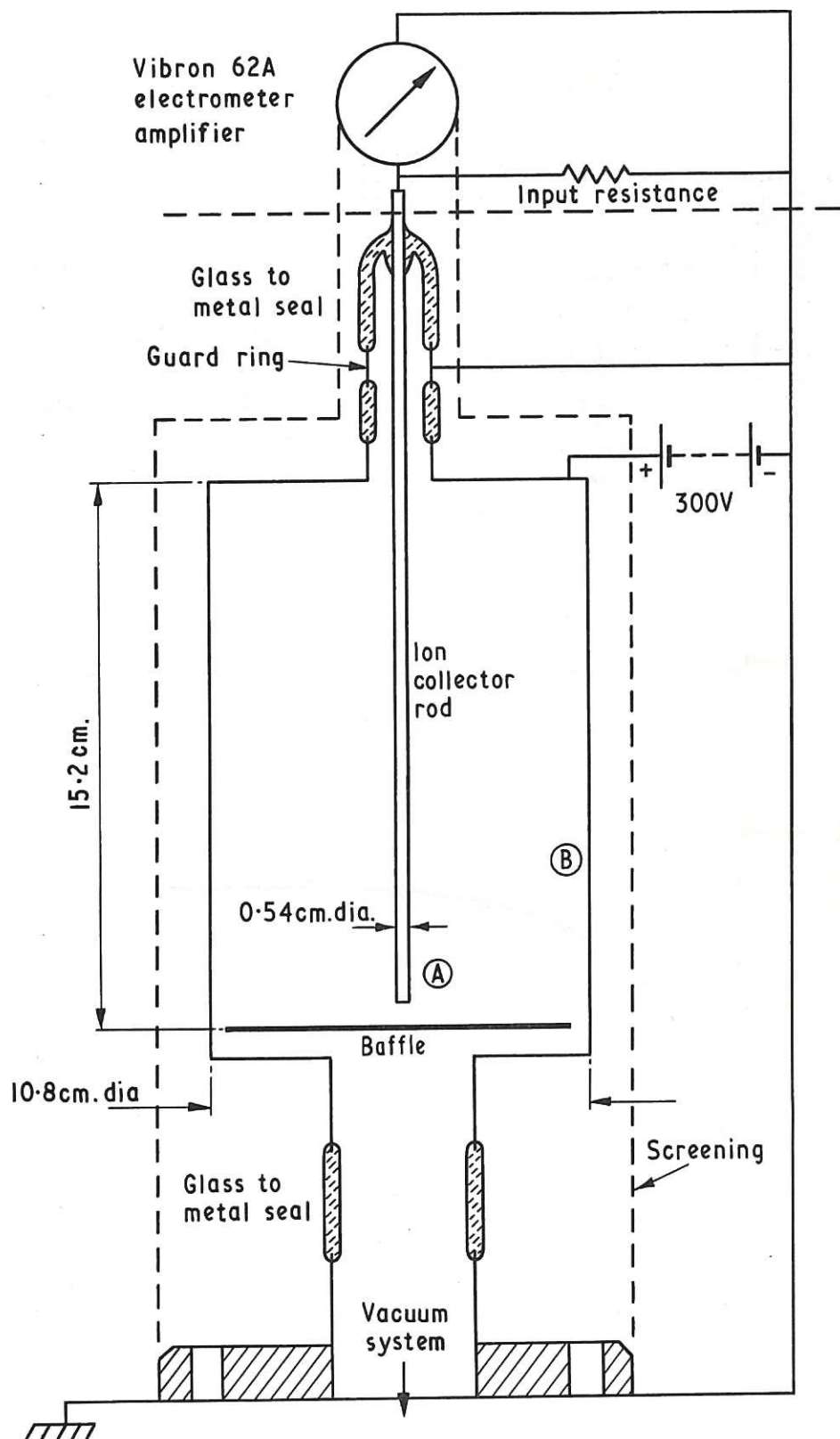
Fig. 1    Collection chamber    (CLM-P 169)
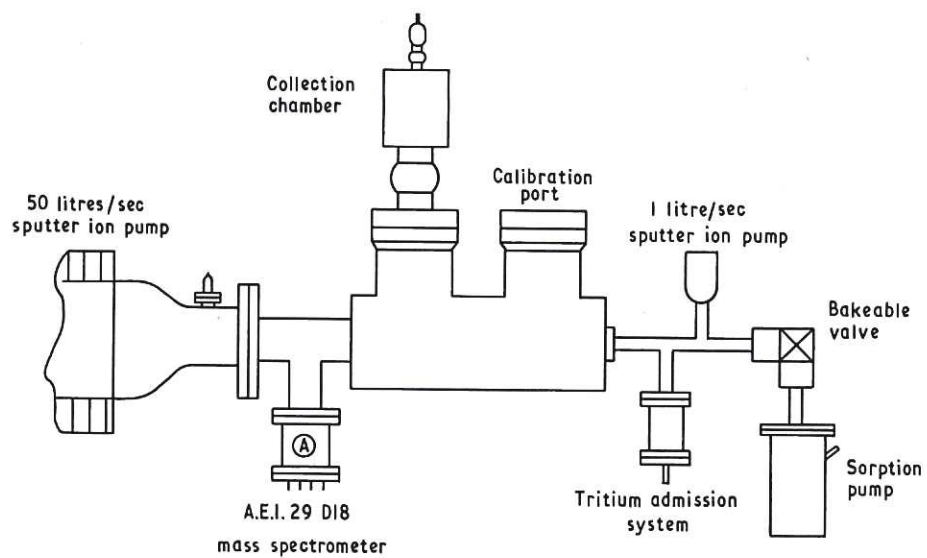
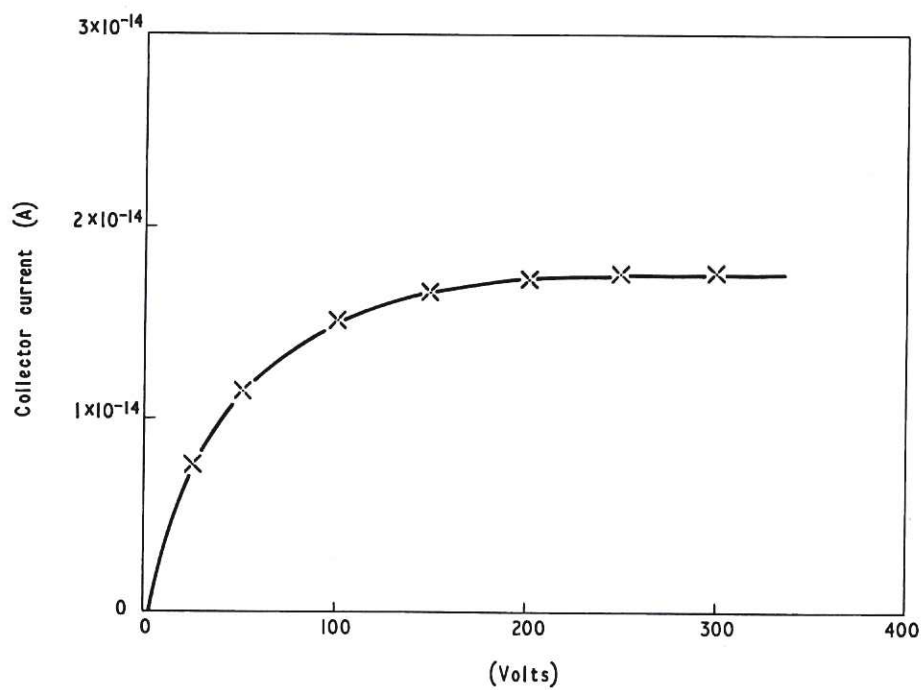Fig. 2   Vacuum system   (CLM-P 169)



Fig. 3                                                      (CLM-P 169)

Collector current (corrected for background)  vs  chamber voltage
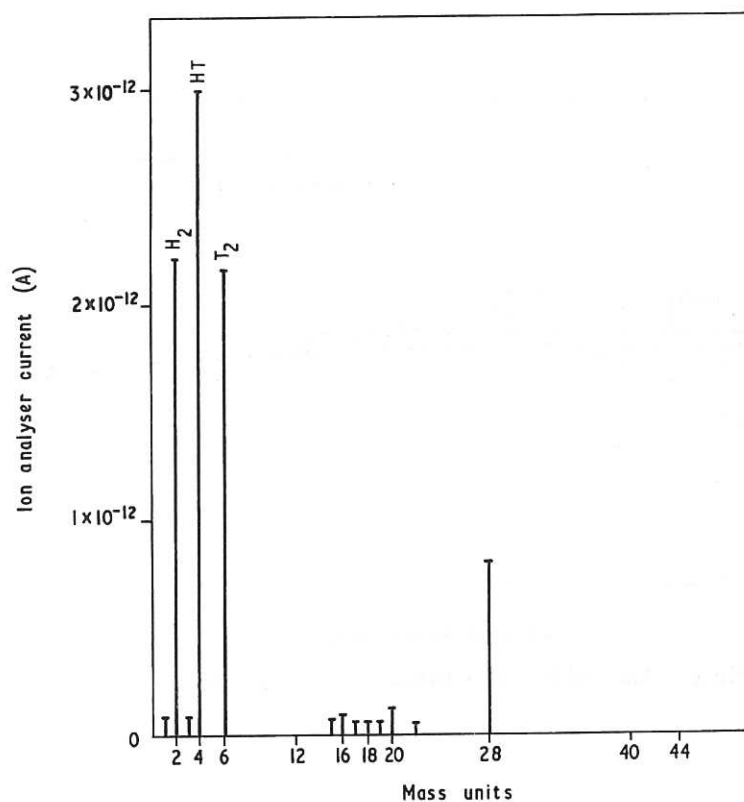for a tritium pressure of $6 \times 10^{-7}$ torr

Fig. 4                                                    (CLM-P 169)

Mass spectrum at a total pressure of $5 \times 10^{-7}$ torr. Note that the sensitivity, on the diagram, increases progressively with mass; the increase amounting to a factor of 10 between Mass 2 and Mass 28
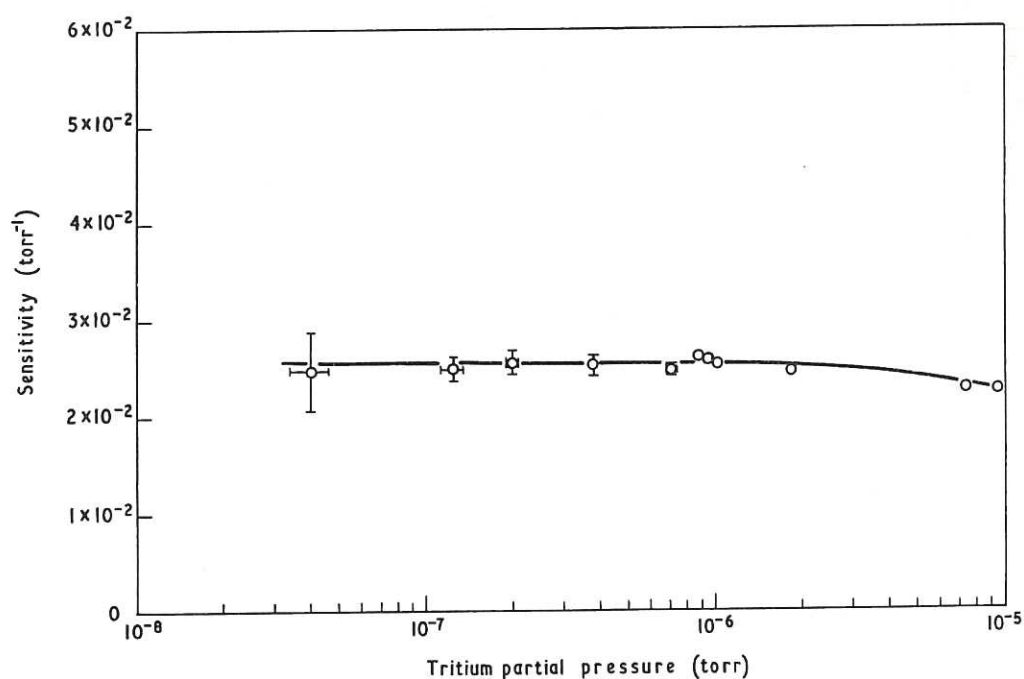


Fig. 5                                    (CLM-P 169)

Quadrupole mass analyser calibration (M / $\Delta$M = 26)
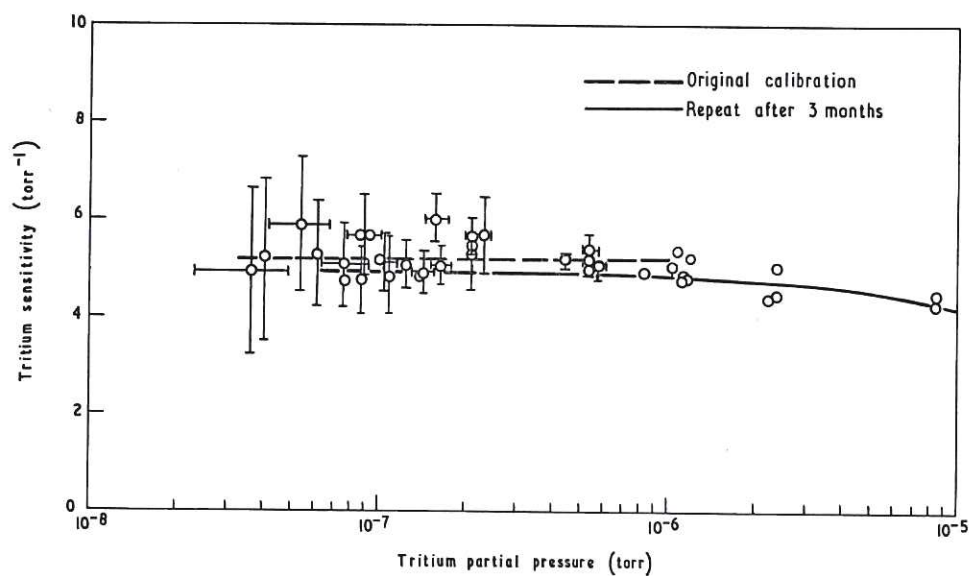
Fig. 6   Mullard IOG 13 calibration   (CLM-P 169)
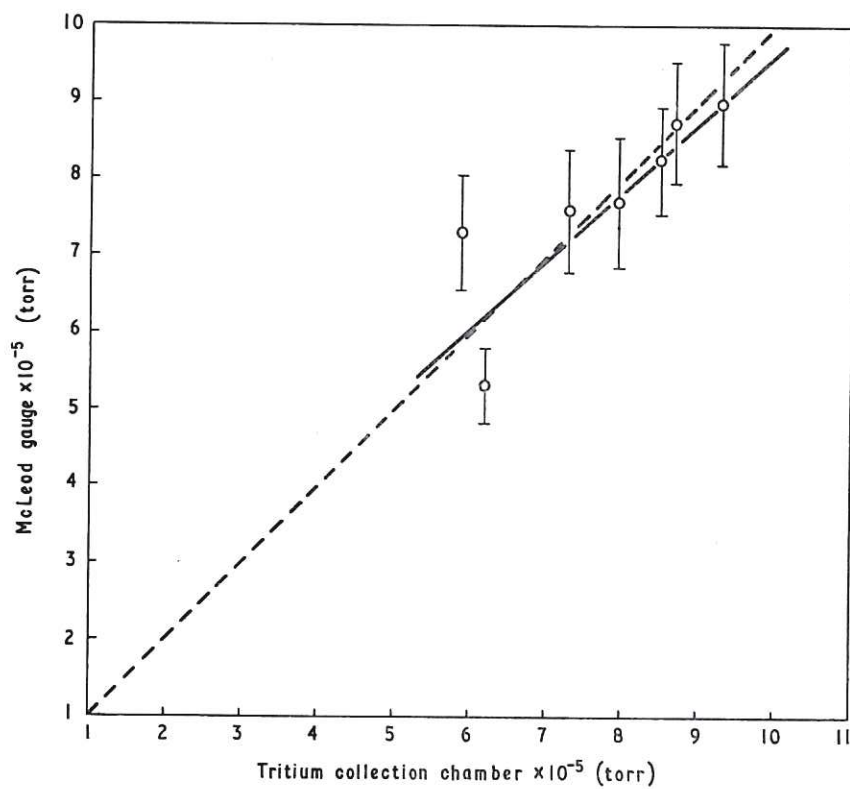


Fig. 7                                    (CLM-P 169)
Comparison of collection chamber with McLeod gauge