## CHROM. 3712

## A helium ionization detector of high sensitivity

It is widely accepted that the principle underlying the operation of the argon and helium ionization detectors is based upon the "Penning effect". Despite this there are a few instances where, using a helium ionization detector<sup>1-3</sup>, the effect is incapable of accounting for the results. The impurity in each instance, neon, hydrogen, argon and nitrogen, gave either a wholly or partly negative response with a helium carrier gas of extreme purity.

In most of the work in which the "Penning effect" has been invoked to account for experimental results the detector pressure was either below about 50 torr<sup>4-6</sup> or above 700 torr<sup>2,7</sup>. It was, therefore, desirable to investigate the change in the detector response over a much wider range of pressures. A Berry detector was installed in an assembly and operated at pressures from about 5 torr to values in excess of 700 torr; this detector assembly is to be fully described elsewhere. The detector was connected on the upstream side, to a molecular sieve 5A column gas chromatograph via a flow restrictor and on the downstream side to a vacuum pump. The pressure was measured close to the detector outlet on the downstream side and the pressures recorded were not therefore the actual pressures within the detector; it is unlikely, however, that they will differ greatly from them. The gas chromatograph operating conditions were:

- (i) carrier gas flow rate 70 ml/min at ambient pressure,
- (ii) sample size 10 ml,
- (iii) detector anode voltage set to give a current of  $4.7 \times 10^{-8}$  A with the carrier gas using a decade voltage supply, W. G. Pye & Co., Ltd., Cat. No. 12363,
- (iv) radioactive source 100 mC tritium foil.

## TABLE I

Absolute pressure (torr)	Anode voltage (V)	Detector current $\times$ 10 <sup>-8</sup> A for impurity				
		$\overline{O_2}$	Ar	CH4	$N_2$	$H_2$
5	297	58.5	32.3	14.9	8.6	5.3
10	284	90.2	67.3	31.9	16.7	10.4
14	268	91.8	87.0	46.3	23.9	14.1
16	255	74.2	82.4	45.9	25.I	14.5
20	243	35.1	55.0	32.8	17.7	11,6
26	228	9.2	28.9	19.0	10.6	7.5
33	218	0,8	11.0	8.4	4.3	3.1
40	218	— I.8	4.9	4.3	2.2	1.2
53	224	- 2.4	1.2	2.1	0.6	0.48
67	241	- 2.4	1.3ª	1.9	0.3 <sup>8</sup>	0.6ª
86	265	- 2.0	1.3ª	1.6	0.3"	0,9ª
110	295	- I.3	1.6ª	1.8	0.4ª	0.9ª
213	394	1.9	2.3ª	3.3	0.6ª	I.2 <sup>8</sup>
320	460	2.5	2.I <sup>%</sup>	3.5	0.5ª	I.2 <sup>8</sup>
757	680	2.4	1.6ª	4.2	0.4ª	I.O <sup>B</sup>

VARIATION OF DETECTOR CURRENT WITH ABSOLUTE PRESSURE

<sup>a</sup> These results are anomalous. The current quoted is the sum of the positive and negative parts of the peak.

NOTES

Using a helium carrier gas of extreme purity<sup>3</sup> and a helium-impurity gas blend containing 12 v.p.m. of each of nitrogen, methane, argon and hydrogen and 23 v.p.m. of oxygen, a series of analyses was carried out at different pressures. In each case the standing current of the detector was maintained at a constant value by adjustment of the applied voltage when pure helium was flowing through the detector. The molecular sieve column was regenerated in situ at 400° for 72 h with a continuous purge of the helium carrier gas and was capable of separating oxygen and argon<sup>8</sup>.

The results given in Table I show that: (1) anomalous responses are not obtained for any of the impurities tested when the pressure is less than about 40 torr, (2) a wholly negative response is obtained for oxygen in the pressure range 40-110 torr, and (3) in all cases a maximum is reached at about 14-16 torr, which is 10-60 times more sensitive than at atmospheric pressure.

It would appear therefore that if the "Penning effect" is able to account for the detector current obtained at 14-16 torr, and the rapid fall in current from 16 torr to about 40 torr is due to the loss of metastable atoms, then the current obtained at values in excess of this pressure must be associated with either the surviving metastable atoms or another process.

The increase in sensitivity obtained at low pressures is to be further investigated for a wider range of impurities and operating conditions, and a detector will be built based upon these findings.

M.G.O. Inspectorates, Chemical Inspectorate, Headquarters Building, Royal Arsenal, East, Woolwich, S.E. 18 (Great Britain)

R. T. PARKINSON R. E. WILSON

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