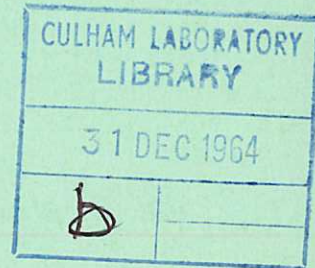


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SOME IMPROVEMENTS IN THE GAS EXPANSION METHOD OF GAUGE CALIBRATION

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SOME IMPROVEMENTS IN THE GAS EXPANSION METHOD OF GAUGE CALIBRATION

by

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A B S T R A C T

The paper describes the gas expansion method of gauge calibration and the techniques which have been developed to enable operation to be extended into the high vacuum regime. Some results are included from experimental calibrations of a Bayard Alpert gauge for argon and hydrogen down to below 10^{-6} Torr.

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C O N T E N T S

	<u>Page</u>
INTRODUCTION	1
ION GAUGE CALIBRATION	2
Argon	2
Hydrogen	3
CONCLUSIONS	4
REFERENCES	5

INTRODUCTION

The calibration of vacuum gauges has usually been carried out by one of the following methods:

- Comparison with a McLeod gauge
- Orifice method
- Gas expansion technique.

Calibration against a series of McLeod gauges gives useful results over the pressure range from a few Torr down to 10^{-5} Torr although considerable care is required in the design and operation of equipment in the lower decades of the range where the accuracy is influenced by the pumping effects of mercury vapour streaming from the exposed mercury surfaces to the liquid nitrogen trap, usually placed between the McLeod gauge and the gauge under test. The technique for comparative calibration against a McLeod gauge has been amply described in the literature by such authors as C.G. Jansen and A. Venema¹, H. Ishii and K. Nakayama² etc.

The orifice technique for calibration uses either the combination of a known gas flow and a known pumping speed to provide a calculable pressure or a sequence of orifices of known flow conductance to establish a calculable pressure ratio between a standard gauge and the one to be calibrated. This technique may be used only for calibration of gauges within the molecular flow region as only in this regime is it possible to calculate sufficiently accurately the conductance of the flow restricting orifices. The technique has the advantages of providing a known pressure continuously and of being suitable for use in the ultra high vacuum regions as shown, for example, by Davis³. It does however rely upon the accuracy with which small gas flows can be measured, and conductances and gas streaming effects predicted.

The gas expansion technique has previously been mainly confined to the medium and coarse (down to 10^{-3} Torr) vacuum regions, using apparatus with greased stop-cocks and joints. The apparent advantages of the method lie in its dependence upon a simple application of Boyle's Law. The aim of the present paper is to

describe the construction of an apparatus employing the gas expansion technique and to demonstrate its capabilities for ion gauge calibration for both inert and active gases down to pressures of 10^{-6} Torr.

APPARATUS

Fig.1 shows diagrammatically an apparatus which is of glass except for the metal isolation valves, and is fully bakeable. It consists of three small volumes, A, C, and E, each approximately 0.05 litres capacity, and three large volumes, B, D, and F, of about 10 litres each, arranged alternately, each volume separated from its neighbour by a bakeable metal isolation valve. Gas is introduced into the first small volume, A, at a pressure measured by a mercury manometer, H, made from 1 cm bore glass tubing to minimise meniscus effects. It is then expanded into BC. C is then isolated from B and the gas expanded into DE. After isolation of E, the gas is introduced into the final volume, F, which houses the gauge, G, awaiting calibration. By this means a reduction in pressure of about 10^7 is attainable. Photographs of the apparatus are seen in Figs. 2 and 3. Although the facility for bakeout at 400°C exists, it has in practice been found that after the initial bake at 400°C , baking at about $120-130^{\circ}\text{C}$ for 24 hours has been sufficient for base pressures of 10^{-8} to be attained, and enables the gauge under test to be attached by an indium seal to avoid the stress problems associated with hard metal gaskets. At the end of the period of bake, the gauge electrodes are thoroughly outgassed while the system is still hot and the liquid nitrogen trap is filled as soon as possible to avoid condensation of hydrocarbons, mainly backstreaming from the diffusion pumps.

ION GAUGE CALIBRATION

Argon

It is proposed to deal first with the technique for the inert gases, for example argon, for which no chemical pumping is expected. The type of gauge used for the experimental work was a Bayard Alpert gauge having a centrally mounted hairpin filament and a tin oxide conducting layer on the inside of the

gauge envelope to avoid oscillation effects in the gauge. Following bakeout, the gauge is calibrated by the following procedure. A sample of test gas is expanded to fill the middle large volume with a reservoir of gas. A succession of gas samples is then transferred through a further stage of gas expansion from the middle reservoir to the final large volume, which is isolated from the pumps and contains the gauge to undergo calibration. During the injection of the succession of gas samples into the final volume, a record is kept of the variation of the gauge ion current with time and has the form shown in Fig.4. The object of using a succession of samples to build up the gas pressure in the final volume through a series of steps is to avoid the surface adsorption effects which might reduce the apparent magnitude of a single gas sample injected into a vessel, initially at a low pressure. By such measurements of the increments in ion current, the sensitivity of the gauge may be calculated from the known initial pressure and the expansion ratio for each of the gas sample injections. Further calibration points may be obtained at other pressures using different initial filling pressures. When these calculated values of sensitivity are plotted against pressure, a graph is obtained of the form shown by the full line in Fig.5. It was suspected that the upward trend of this sensitivity curve at the lower end of the pressure range studied was due to the effect of background contamination introduced into the final volume from the middle gas reservoir along with the gas. The effect was confirmed by observation of the variation of pressure in the final volume during dummy gas sample injection from the middle reservoir volume, leaving the volume uncharged with test gas. Correction of the experimental argon measurement for this background effect is shown in the broken line in Fig.5. Within experimental error, the gauge sensitivity factor remains constant over the pressure range 5×10^{-7} to 10^{-4} Torr.

Hydrogen

In calibrating an ion gauge by the gas expansion technique for an active gas such as hydrogen, strong pumping effects may be observed which make it difficult to determine the true pressure produced within the final volume by the gas sample injection. An example of the variation of the pressure during injection of a

series of samples of hydrogen is seen in Fig.6. Since the ion current may change by 6% of the peak value per second due to the pumping effects, it is necessary to use an ion current amplifier with a time constant no greater than a second or so. In view of these rates of current change it is also an advantage to use a recording oscillograph instead of manual recording to facilitate the accurate extrapolation back to the time of gas injection. A tracing from a typical oscillograph record is seen in Fig.7 together with the method of extrapolation to correct for gauge pumping effects assuming that the rise time of the ion current wave form is determined by the ion current amplifier rather than by the flow of the injected gas sample. Fig.8 shows the sensitivity values for hydrogen calculated from these figures. Below about 5×10^{-6} Torr it is necessary, as for argon, to make a correction for background gas effects. In the case of hydrogen, however, the correction required must involve subtraction of the time variation of the current due to background gas from that for hydrogen. This is shown in Fig.9. The correction technique assumes that there is no interaction between the injected gas sample and the background contamination.

CONCLUSIONS

By suitable attention to design and method of use of the apparatus, the range of the gas expansion method of gauge calibration can be extended well into the high vacuum range. This enables it to be used for the calibration of ionization gauges for both inert and active gases down to better than 10^{-6} Torr, provided precautions are taken at the low pressure, and to allow for errors caused by gas adsorption by the walls of the final chamber, pumping by the gauge and gas sample contamination.

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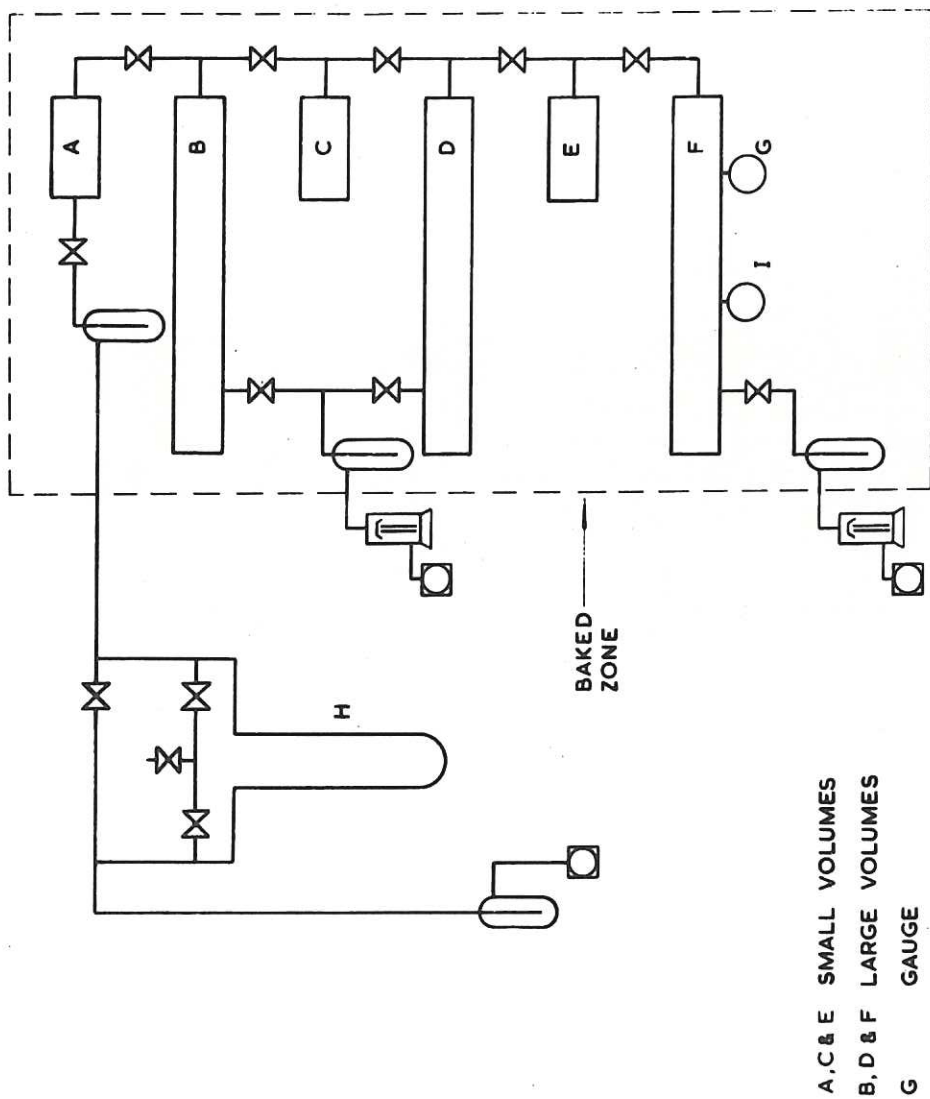


Fig. 1
 (CLM-P.67)
 Schematic representation of calibration apparatus

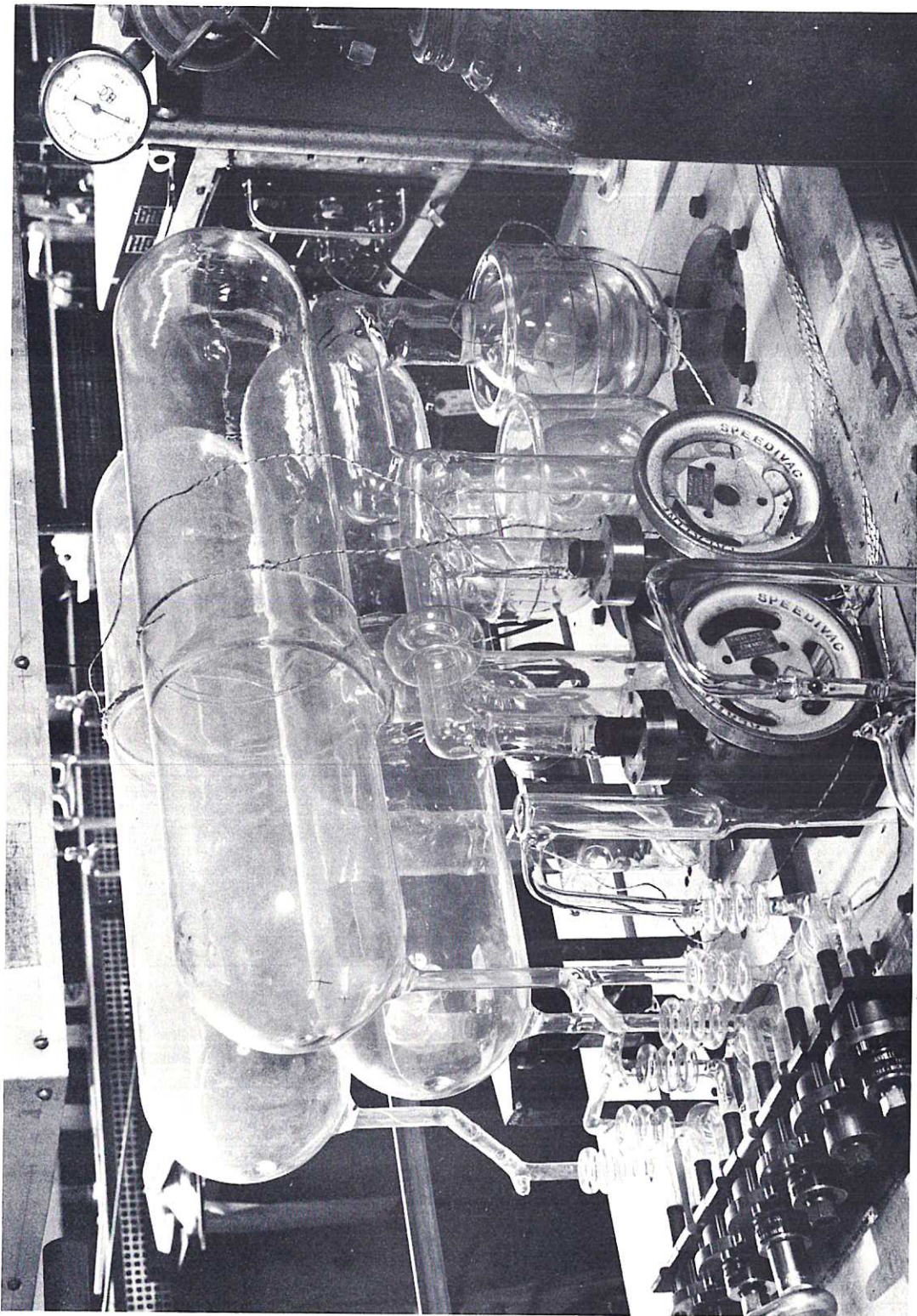


Fig. 2
(CLM-P 67)
Apparatus showing bakeable isolation valves

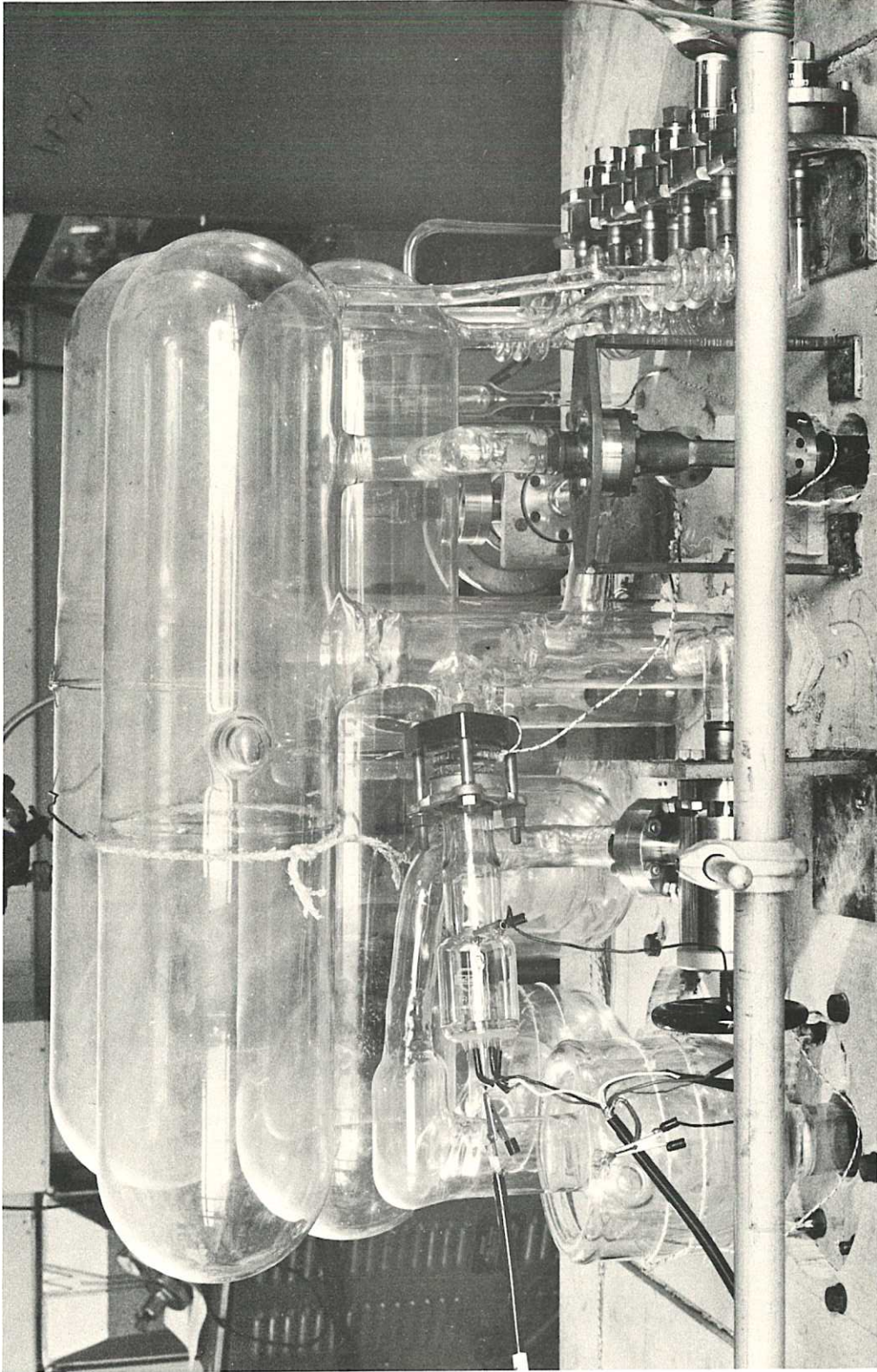


Fig. 3
(CLM-P 67)
Apparatus showing gauge for calibration

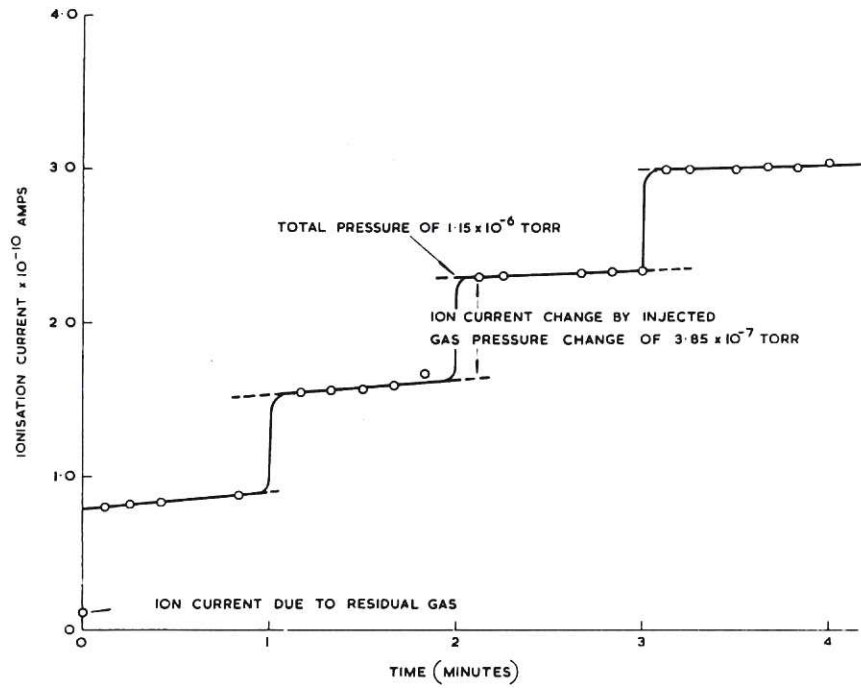


Fig. 4 (CLM-P 67)
 Argon : variation of ion current with successive gas sample injections

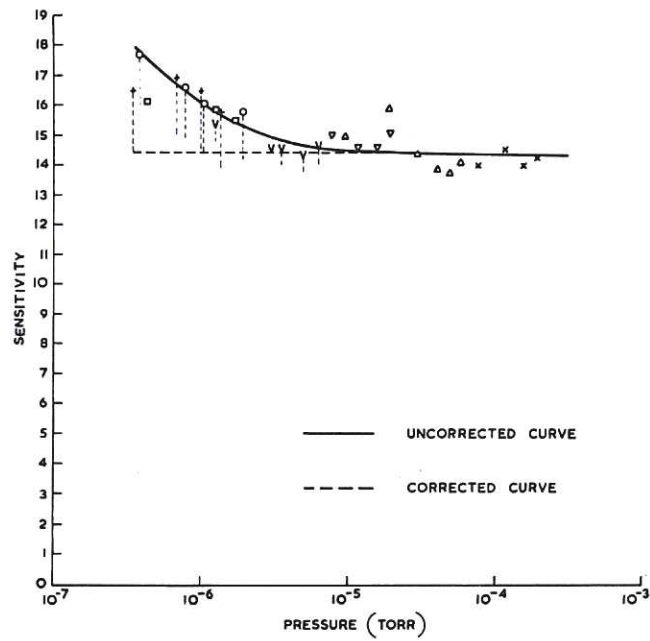


Fig. 5 (CLM-P 67)
 Argon : dependence of gauge sensitivity on pressure

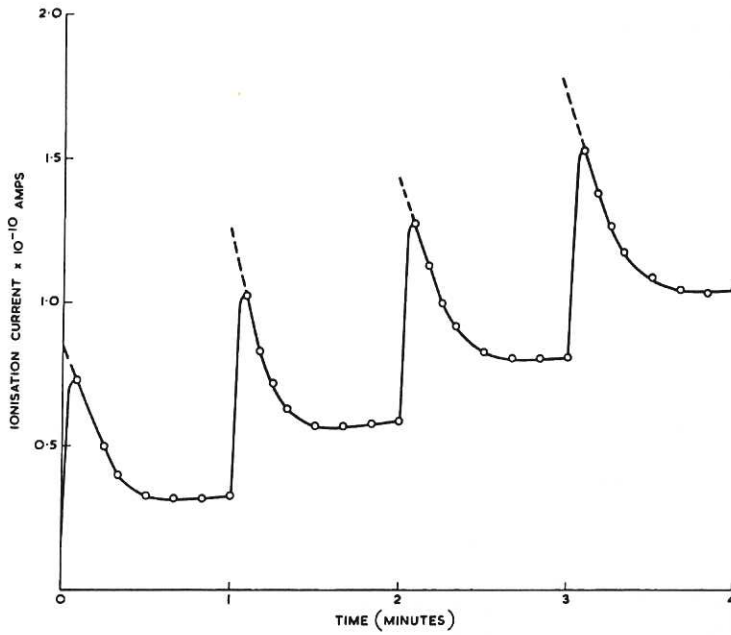


Fig. 6 (CLM-P 67)
 Hydrogen : variation of ion current with successive gas sample injections

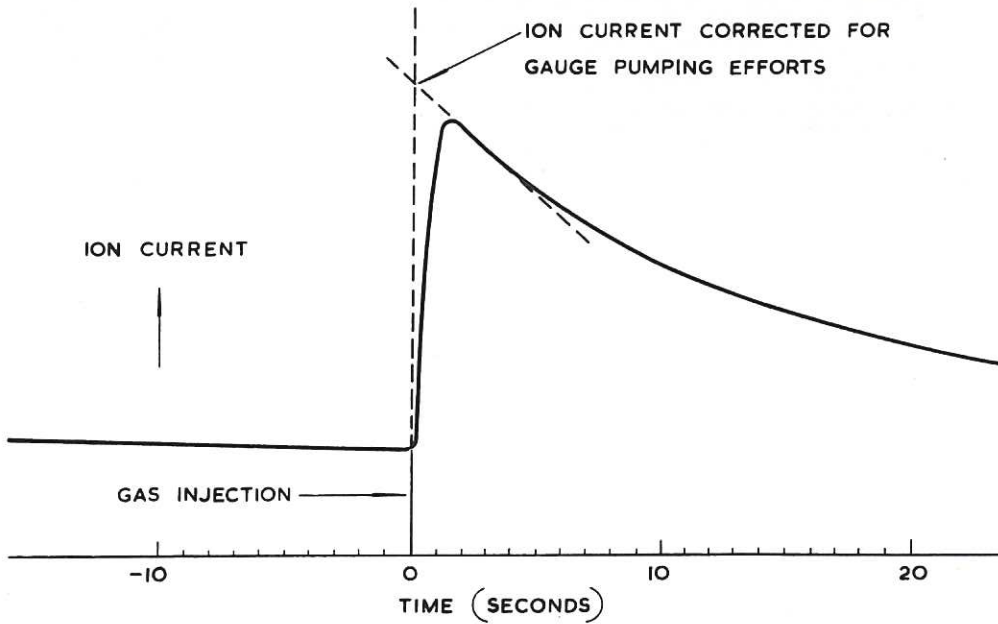


Fig. 7 (CLM-P 67)
 Hydrogen : typical oscillograph record of ion current variation during gas injection

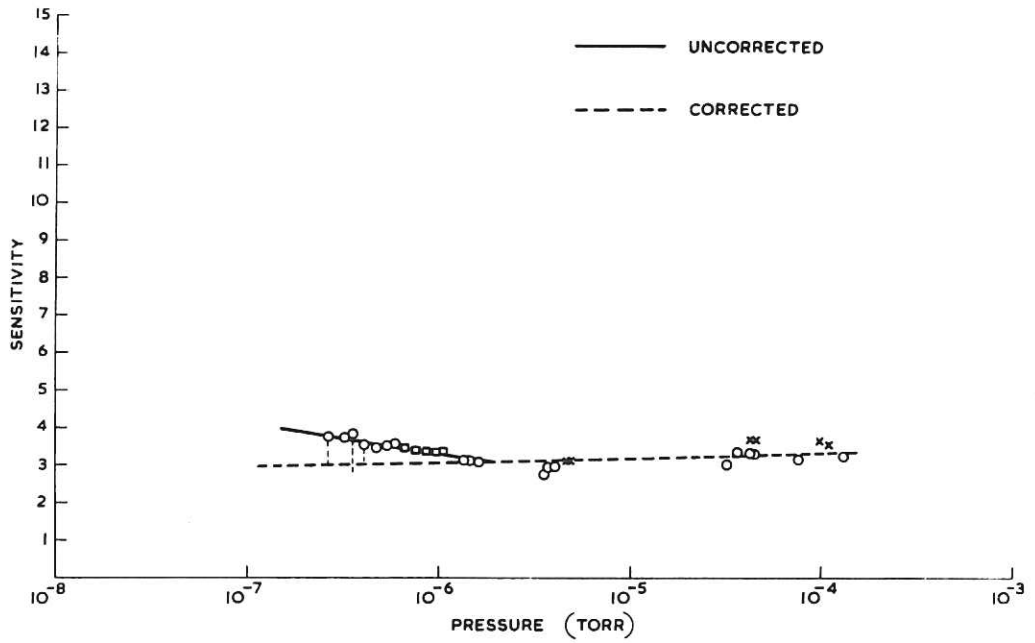


Fig. 8 (CLM-P 67)
Hydrogen : dependence of gauge sensitivity on pressure

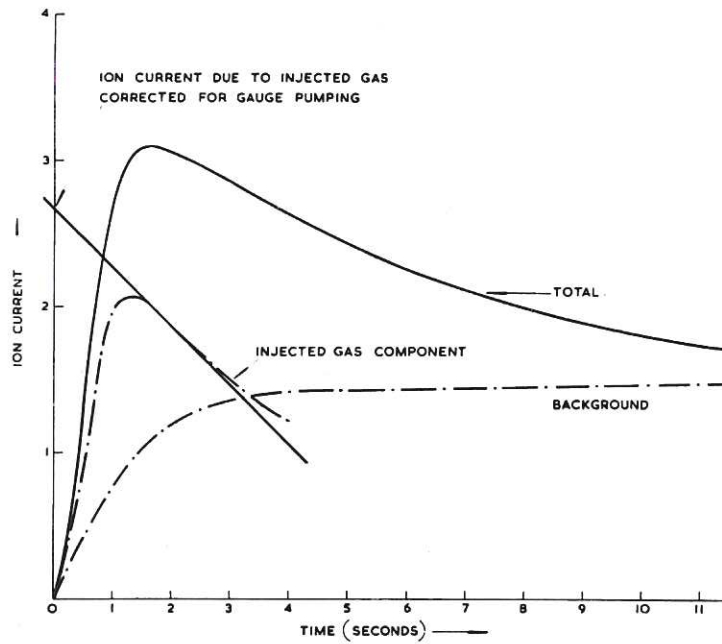


Fig. 9 (CLM-P 67)
Calibration for hydrogen : subtraction of ion current variation due to background gas injection from that during hydrogen injection

