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SOME ERRORS I N GAUGE CALIBRATION

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SOME ERRORS IN GAUGE CALIBRATION

Introduction

This investigation is concerned with the experimental error in gauge calibration caused by gas flow within and into vacuum systems.

The calibration technique first used included the "variable volume technique"[1] to determine "Q", the gas flow rate, and the "limiting orifice technique"[1,2] to determine "S", the pumping speed at the plane of the gauges to be calibrated. Because of early difficulties in accurately measuring small flow rates, the full pumping speed of the vacuum system was measured without an orifice by the "rate of rise of pressure technique."[3] The higher pumping speed allowed the use of a correspondingly higher gas flow rate that could be more accurately measured. The "rate of rise of pressure technique" was investigated to determine if it was a suitable method to calibrate gauges and that investigation forms the basis for this paper.

There were two major problems. First, the experimental methods for measuring "S" had to be studied so that consistent and accurate data could be recorded. Second, when the speeds as experimentally determined by the "rate of rise technique" were compared to the speeds calculated by the

*Work done at Veeco Instruments Inc., Plainview, New York.

"limiting orifice technique," there was serious disagreement.

When the gauge factors for nitrogen, helium and oxygen relative to argon are compared to the literature, good agreement is obtained by the "rate of rise technique" and by the "limiting orifice technique." However, the absolute gauge factor as determined by each method can differ by as much as 16%, a number much greater than the expected absolute accuracy of each technique.

Experimental

The vacuum system used is shown schematically in Fig. 1. It consists of a standard 6-in. oil diffusion pumped system with or without a limiting orifice, calibration dome, and gas metering system.

A basic variable volume "Q" measuring device and associated formulae are shown in Fig. 2.

The problem associated with the measurement of leak rates by the "variable volume technique" below 1×10^{-4} torr ℓ/sec is not due to error in measuring small volume changes, but rather to the effects of atmospheric pressure and temperature on the volume and gas pressure in the "Q" measuring device.

The effects of changes in atmospheric pressure may be transmitted through small leaks in the "Q" device. These leaks can, of course, be found and sealed.

By far, the most persistent error is caused by small (less than $1^{\circ}K$) changes in ambient temperature.

For example, the colume change necessary to measure a leak rate of 1×10^{-5} torr ℓ/sec over a ten-minute interval is about 1×10^{-5} ℓ.

The "Q" device shown in Fig. 2 has a leak and reference volume of about .03 liters each. If, during the ten-minute test interval, a $1/100^{\circ}K$ temperature differential develops between the leak and reference volume, it will

cause a differential volume change of 1×10^{-6} ℓ. This is enough to cause a 10% error in a "Q" measurement of 1×10^{-5} torr ℓ/sec. A larger leak or reference volume will cause a correspondingly larger error.

A suggested method to overcome this problem is to reduce the pressure in the "Q" manifold so that a proportionally larger volume change can be measured for the same leak rate. This can, of course, be done. However, it means that an absolute gauge has to be attached to the "Q" manifold, and a more sophisticated differential manometer and leak valve have to be used. These devices are usually attached to the "Q" device with tubing, and in the end this extra tubing just serves to amplify any error caused by temperature differentials. Most important the ion gauges cannot be stabilized because the test gas pressure is continuously changing due to low pressure and volume of "Q" device. What appears to happen in this case is that a complicated device is constructed whose accuracy can be seriously questioned.

An ideal device would be one that is small and made from a solid block of metal of high heat conductivity with a minimal of appendages.

A "Q" device has been fabricated for these tests from a solid block of aluminum and it has been consistent in measuring leaks of 1×10^{-4} torr ℓ/sec to better than 1%.

The rate of rise of pressure technique to measure pumping speed is useful with any calibration system that has a valve above the pumps and can reach an ultimate in the low 10^{-8} torr scale. The schematic of a system that can use this technique is shown in Fig. 3 along with the associated formula.

The equilibrium gauge reading is usually set high on the 10^{-7} scale when no orifice is used so that a measurable rate of change through the mid 10^{-5} scale can be recorded. The rate of change through the 10^{-6} scale is much too fast to make an accurate reading.

The main advantages of this technique to measure, "S", are it uses the full pumping speed of the vacuum system to experimentally measure the pumping speed at the plane of each gauge to be calibrated, is independent of gas species and the gauge factor.

Before showing the results of the speed runs, the vacuum technique used will be briefly described:

1. The gauges and dome were baked out at $125^{o}C$ for 8 hours.
2. After bakeout, liquid nitrogen was added to the trap and the gauges I^2R outgassed at the system's base pressure for 15 minutes.
3. The gauges were stabilized with the test gas at a pressure of 4×10^{-4} torr for 1 hour at 1 milliampere emission current.
4. The order of calibration was from the 10^{-4} scale down to the 10^{-7} scale.
5. The equilibrium pressure for the determination of pumping speed was usually on the high 10^{-7} torr scale.
6. The test gas used was argon unless otherwise noted.
7. One electrometer, a standard laboratory model with a reported absolute accuracy of ±2% was used to read the ion current and rate of change of ion current from all the gauges with their respective emission regulators. That is, one electrometer was moved position by position about the done, first to note all the equilibrium gauge readings, second to record all the rate of change of electrometer readings. This test was repeated four times.

These results are shown in Table I. The main point to note is the consistency in the speed number for each gauge and the consistency between the speed readings for all the gauges. However, it can be noted that there is a non-random spread in the speed numbers, that is tube no. 5 is the highest, tube no. 1 is next, then tube no. 4, then tube no. 2 and finally tube no. 3.

This systematic spread is small and could be a real effect. However, it has not been defined and could be caused by non-uniform gas flow, the emission controllers, or the gauges themselves.

Comparative Tests

A natural extension of these experiments was to compare the "rate of pressure technique" for measuring pumping speed with the "limiting orifice technique." This was done by placing a 1/2" orifice just below the gauges as indicated in Fig. 1. This orifice had a theoretical speed for argon of 10.7 ℓ/sec at 20°C. The pumping speed at the plane of the gauges was corrected for the system with the limiting orifice by the formula:

$$\frac{1}{S_g} = \frac{1}{S_o} + \frac{1}{S_{system}}$$

The "rate of rise of pressure technique" was also used to determine the pumping speed at the plane of the gauges and the results of these tests were compared with the "limiting orifice technique" in Table II.

Several points should be noted. First, the spread in the pumping speed for the rate of rise method is about equal to the spread when no orifice is used. Second, the experimental pumping speed by the rate of rise method is higher than the theoretical speed by the limiting orifice method in every case and, in particular, there is little agreement for the helium speed. Third, the spread between the average pumping speed for a particular gas and the theoretical speed is not a constant indicating the discrepancy may be due to a characteristic of that particular gas.

When these speed runs were made by the rate of rise method, both with and without an orifice, the gauge factor was usually determined for each gauge.

Table III is a comparison of the gauge factors as determined with or without an orifice by the rate of rise method. The agreement is usually

within 2%. However, if the gauge factors as determined by the limiting orifice are compared to these numbers the argon factor would be 11% high; the nitrogen 9%; the helium 17% and the oxygen 6%. Several of these differences are much greater than the expected absolute accuracy of either technique.

It is straightforward to experimentally check the rate of rise method by substituting different orifices to limit the pumping speed. This is what was done in the above example. However, the parameters involved in checking the pumping speed as determined by the limiting orifice technique are mostly theoretical. They have to do with the system pumping speed, orifice corrections, corrections for calibration dome geometrics and gauge positioning.

Table IV gives the average gauge factors relative to argon for the ion gauges used. This work is in general agreement with much of the published data on the relative gauge factors, even though the experimental pumping speeds were not in agreement with theoretical speeds as calculated by the orifice technique.

Conclusion

Some of the problems associated with the laboratory use of the "variable volume technique" of measuring small leaks have been discussed.

The problems associated with gas flow in vacuum systems and the measurement of pumping speed to the accuracies necessary for gauge calibration have been investigated. The results show that the pumping speed can be consistently measured to be within 2% by the "rate of rise of pressure technique".

The results of calibrating by the "variable volume technique" to measure "Q" and the "rate of rise technique" to measure "S" are in excellent agreement with previously published data on gauge factors relative to argon. However, there is serious disagreement between this technique and the limiting

orifice technique to measure pumping speed. Further experiments are needed to resolve this discrepancy.

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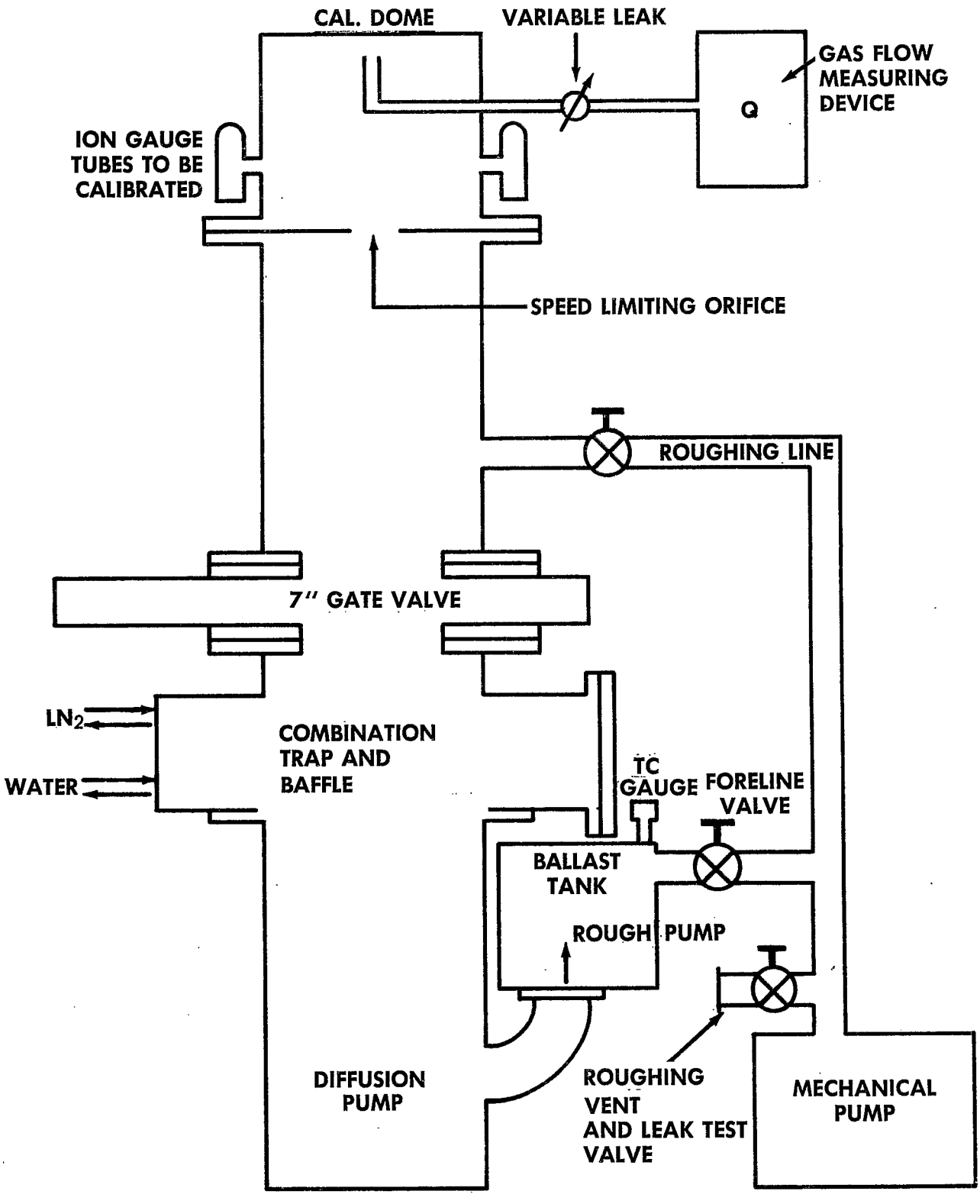
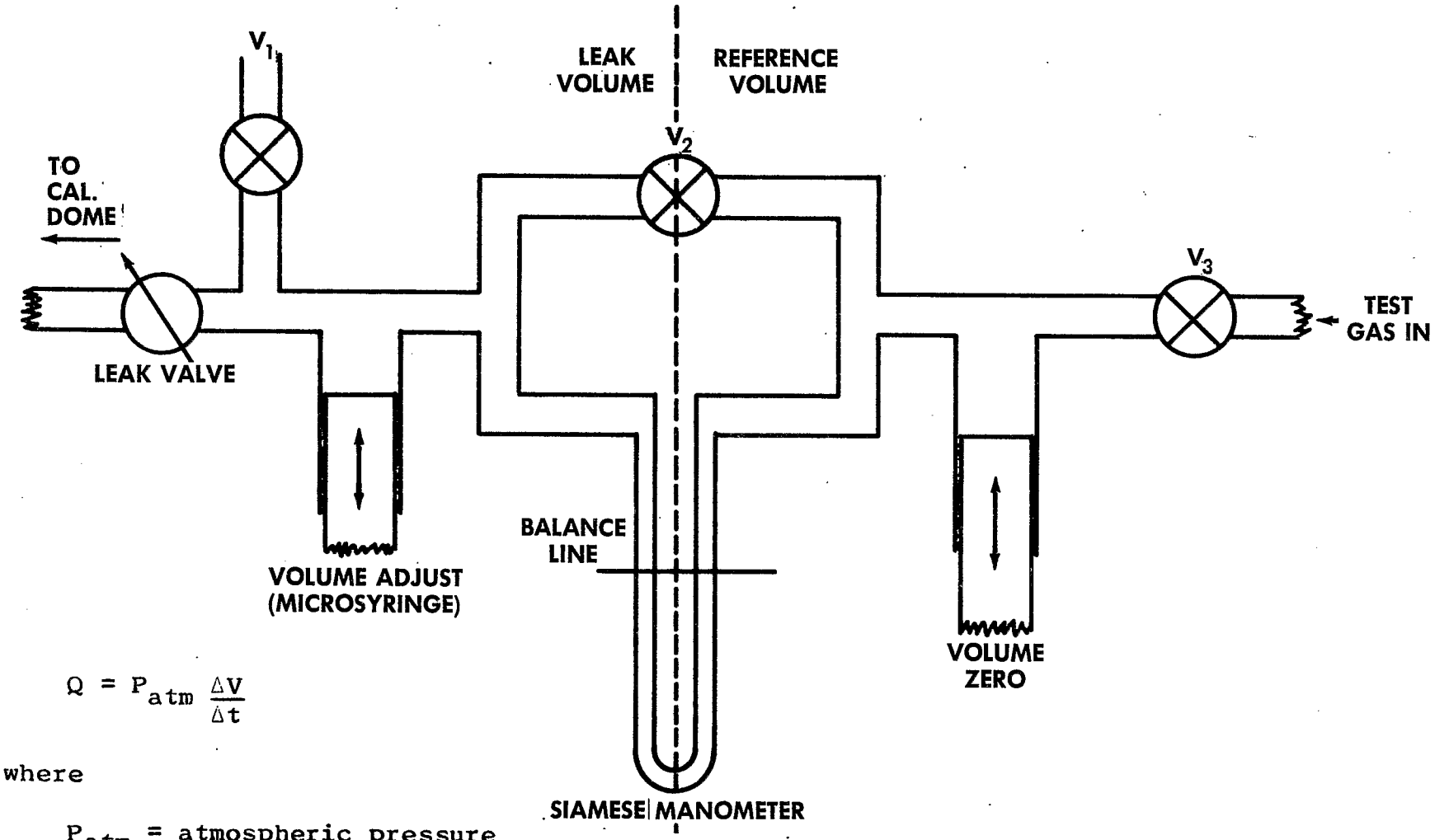


Figure 1 Calibration Vacuum System.



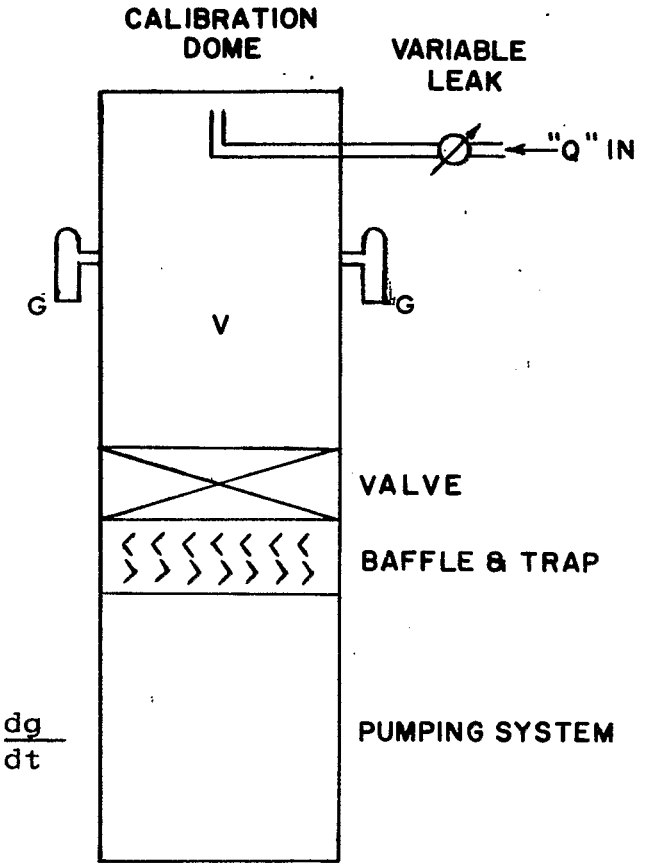
$$Q = P_{atm} \frac{\Delta V}{\Delta t}$$

where

P_{atm} = atmospheric pressure

$\frac{\Delta V}{\Delta t}$ = rate of change of leak volume at constant pressure

Figure 2. "Q" Measuring Device Schematic



$$S = \frac{v}{g}\frac{dg}{dt}$$

where

v = volume of calibration dome between the hi-vac valve and leak valve.

g = equilibrium gauge reading in calibration dome with the leak valve cracked and the hi-vac valve open.

$\frac{dg}{dt}$ = rate of change of gauge reading in the calibration dome with the leak valve cracked and the hi-vac valve closed.

Figure 3 Calibration System Schematic

TUBE #	SPEED RUN #				AVERAGE	% SPREAD
	1	2	3	4		
1	320	325	329	323	326	.9
2	314	318	328	320	320	1.3
3	304	312	316	316	312	1.3
4	314	329	336	319	325	1.8
5	325	329	332	338	331	1.2
AVG	315	323	328	323		
% SPREAD	1.8	1.9	1.5	1.9		

TABLE I

Pumping speed in ℓ/sec measured at the plane of the gauges. Each test was repeated four times.

TUBE #	PUMPING SPEED (l/sec)							
	Ar	THEO Ar	N_2	THEO N_2	He	THEO He	O_2	THEO O_2
1	11.5	10.3	13.6	12.3	37.8	32.1	12.4	11.6
2	11.5		13.3		37.1		12.2	
3	11.0		13.0		36.2		11.9	
4	11.5		13.5		37.7		12.4	
5	11.7		13.8		38.4		12.6	
AVG	11.4		13.4		37.4		12.3	
% SPREAD	1.8		1.8		1.7		1.6	

TABLE II

Comparison of the pumping speed in ℓ/sec determined experimentally by the "rate of rise of pressure technique" and calculated by the "limiting orifice technique".

TUBE #	GAUGE FACTORS $\frac{\text{True Pressure}}{\text{Gauge Reading}}$							
	ARGON		NITROGEN		HELIUM		OXYGEN	
	no aperture	aperture	no aperture	aperture	no aperture	aperture	no aperture	aperture
1	.58	.61	.80	.82	4.47	4.36		1.20
2	.70	.74	.93	1.00	5.00	5.55		1.27
3	.63	.61	.82	.82	4.58	4.60		.99
4	.88	.91	1.15	1.23	6.37	6.67		1.34
5	.78	.76	1.01	1.04	5.83	5.67		1.27

TABLE III

Comparison of the gauge factors determined by using the "rate of rise of pressure technique" for the vacuum system with and without a limiting orifice.

	Rate of Rise	Limiting Orifice	Rothe	Schulz	Duchman	Reynolds	Waterbuck
Ar	1	1	1	1	1	1	1
N_2	.74	.75		.67	.84	.73	.71
He	.14	.13	.13	.14	.13	.13	.13
O_2	.63	.65					.62

TABLE IV

Comparison of the ion gauge sensitivities relative to Argon determined by several different techniques.