Leak Calibration Method for any Gas at a Variable Pressure
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INTRODUCTION

The leak detection is nowadays widely used in various fields as the automotive and the refrigeration industries. The leak tightness of installations charged with refrigerants must be controlled periodically by refrigerant detectors, qualified via refrigerant leaks. However the latter are not traceable yet.
Environmental concerns are the source of increasingly stringent requirements with regard to the containment of fluorinated gases with greenhouse effect.
In this context, the European community established the requirement for periodic inspections of all equipment with more than 3kg of gas of such gases. These inspections should be carried out with calibrated detectors sensitive to 5g/year leak of fluorinated gas and checked every 12 months.
The qualification of the detectors is totally based on the accuracy with which the reference leaks can be calibrate.

Theory

A very small portion of a know mixture should pass through a sniffer (a capillary with known conductance), into the vacuum chamber where the mass spectrometer is. The intensity of the selected peak will vary on exactly the same proportion in which the concentration of this gas varies. Thus, it is possible to obtain a calibration for the concentration of the mixture inside the volume at required pressure. This calibration remains valid, as long as the conditions under which it was obtained remain.

Fig. 1 – Experimental Apparatus

The preliminary tests were perform using a known helium leak. Several measurements were made using deferent volumes: 20, 25, 30, 35 e 40cc. That was accomplished by using a volume control, like a big syringe. After the gas entrance, there was a stabilization time of about 15 minutes. The data aquired before that period was neglected.

Fig. 3 – Data acquired after stabilization period the showing the inversely proportionality between the concentration rate measured by the quadrupole and the volume between leaks.

EXPERIMENTAL

To achieve a measure of an unknown leak flow shall be as follows: the test volume is evacuated and filled with nitrogen to the test pressure (about atmospheric pressure) with the leak mounted. The gas concentration will increase continuously due to migration of gas from the leak. This concentration can now be measured in absolute values by the mass spectrometer through the sniffer. The concentration derivative in time corresponds exactly to the flow. Its value can be easily obtained through a linear adjustment of the value of concentration against time.
The concentration rate showed by the quadrupole is given by

$$m = \frac{Q_f \cdot C}{S \cdot V}$$

Where the m is the concentration rate, C is the sniffer conductance, V is the volume between leaks and S is the pumping speed.
It is possible to verify the inversely proportionality between the concentration rate measured by the quadupole and the volume between the leaks.
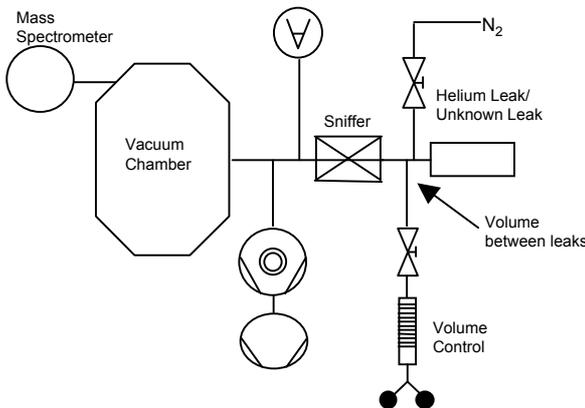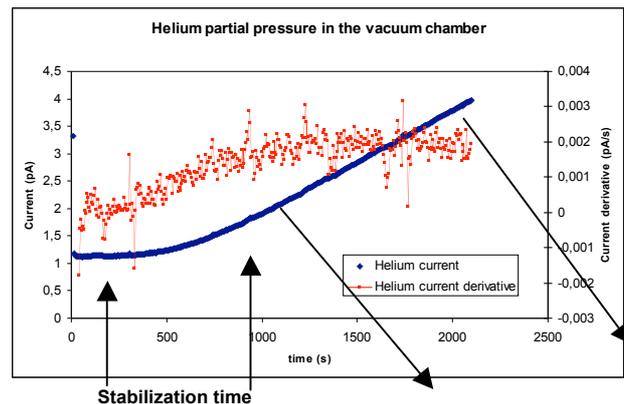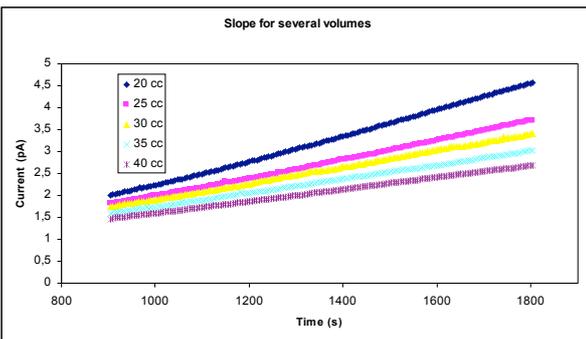The preliminary measurements were made using a known helium leak, for several volumes to test this proportionality.

Fig. 2 – Data obtain shows the stabilization time and the slope showing the proportionality between the leak and the concentration rate.

Stabilization time

Slope showing the proportionality between the leak and the concentration rate measured by the quadrupole.

As the excepted, it was possible to realized that the slope was inversely proportional to the volume.
By increasing the volume the slope was decreasing.

CONCLUSION

The preliminary results have been acquired with helium showing the inversely proportionality between the concentration rate measured by the quadupole and the volume between the leaks. With the system used, leaks in the 10^{-5} and 10^{-6} mbar.L/s range where able to be detected. The lower limit depends on the volume, the mass spectrometer sensibility and the sniffer conductance. Further studies will be performed in order to define with more accuracy the volumes to be used, the sniffer conductance, the inlet pressure range and finally the uncertainty calculations.
In the case of molecular gases like those used as refrigerants, the only difference in the procedure is that a characteristic mass peak should be selected to measure the concentration.

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