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Measurement systems development for the calibration of leaks and holes in dynamic pressure (microflow) at CENAM, Mexico

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Abstract

The pressure and vacuum group of the Force and Pressure Division, at the Centro Nacional de Metrologia (CENAM, Mexico) has developed two measurement systems for calibration, one for leaks and another for holes in dynamic pressure.

The leaks calibration system is based on the method of pressure increment at constant volume (and constant temperature) within the measurement range starting at 1×10^{-6} $Pa \cdot m^3 \cdot s^{-1}$ up to 5 $Pa \cdot m^3 \cdot s^{-1}$ with volumes of 0.5 L or 1 L (according to the leak range to be calibrated), with an operation pressure from 1×10^{-5} Pa up to 130 kPa, using helium, nitrogen or air as manometric fluids.

For the calibration of holes in dynamic pressure, the method PVTt is used. This is, pressure increments at constant volume and constant temperature during a given time. The volume used is 3.75 L with a gas flow up to 20 $cm^3 \cdot min^{-1}$ using nitrogen as operation fluid.

The procedure of holes calibration consists on measuring the flow through a small hole by measuring the pressure increment due to the gas accumulation in a known volume during a period of time with controlled temperature conditions. The PVTt system is formed by pressure gauges, a gas collection tank with a known volume, temperature sensors, a chronometer for time measurement, a vacuum pump, a set of valves to control the gas flow direction and the hole which will be calibrated.

Keywords: Vacuum, leaks, dynamic pressure

1. Introduction

A leak is an instrument that consists basically of an element that restricts the gas flow which is inside of a small container; the flow rate of the leak is calibrated and then its value is used as a reference. There are basically two types of leaks:

a) Permeation leaks, which allow gas to permeate through an element, which is made of glass, plastic, elastomer, metal or other materials.
b) The other type is physical leaks, where the leak element causes a physical restriction of the gas flow. The leak restriction can be capillary tubing, a drawn glass capillary, crimped tubing, sintered powders and micro holes.

The difference between the two types of leaks is that while physical leaks allow the flow of many fluids, the permeation leaks allow the flow of only one or a few types.

The two measurement systems developed are based on the measurement of fundamental quantities, such as length, mass, time and temperature. The calibration principle of both systems is based on the pressure increment which occurs in a calibrated volume (constant volume) within a given time, taking into account the gas temperature.

2. Measurement Principle

2.1 Measurement Principle for Leaks

There are two techniques for the calibration of leaks [1, 2], the primary techniques are based on the measurement of mass, pressure, volume, temperature and time. The secondary techniques use a leak of known leakage rate to generate a signal on an instrument which is then compared to the signal due to the gas emitted by the leak under testing.

The primary technique used for leaks calibration is by measuring the pressure increment at a constant volume [1, 2]. The gas from the leak enters an originally evacuated volume, making the pressure in the known volume increase. Obtaining the differential of the ideal gas law with respect to time, at constant temperature, the molar leakage rate can be evaluated.

$$P \cdot V = n \cdot R \cdot T \tag{1}$$

$$n = \frac{P \cdot V}{R \cdot T} \tag{2}$$

$$Leak = Q_m = \frac{dn}{dt} = \frac{V}{R \cdot T} \cdot \frac{dP}{dt} \tag{3}$$

Where:

P is the absolute pressure of the gas (Pa),
V is the volume that contains the gas (m^3),
n is the number of moles contained in the volume *V,*
R is the constant of the ideal gases 8.314 51 $Pa \cdot m^3 \cdot mol^{-1} \cdot K^{-1}$
T is the absolute temperature of the gas (K).

2.2 Measurement Principle for Holes

For the calibration of holes a PVTt measurement system is used. In this system, the gas flow that goes through the hole at standard conditions is measured, using the technique of pressure increment at constant volume, the calculation of the flow is made by means the following equations:

$$Q = \frac{\dot{m}}{\rho_{N_2,st}} \tag{4}$$

$$\dot{m} = \frac{V \cdot (\rho_2 - \rho_1)}{t} \quad (5)$$

$$Q = \frac{V \cdot (\rho_2 - \rho_1)}{t} \cdot \frac{1}{\rho_{N_2,st}} \quad (6)$$

Where:

- Q is the volumetric flow (m^3/s),
- $\rho_{N2,st}$ is the standard density of the nitrogen 1,250 46 kg/m^3
- $\dot{m}$ is the mass flow (kg /s),
- ρ_1 is the density of the nitrogen in the initial condition (kg/m^3),
- ρ_2 is the density of the nitrogen in the final condition (kg/m^3),
- t is the time from the initial condition to the final condition (s).

With the ideal gas law, we can obtain the gas density for the initial and final conditions using the equation,

$$\rho = \frac{P \cdot M}{z \cdot R \cdot T} \quad (7)$$

Where:

- P is the pressure (Pa),
- z is the compressibility factor,
- M is the molecular weight of the gas (kg/kg·mol),
- ρ is the nitrogen density (kg/m^3),

The compressibility factor z depends on the gas that is used during the calibration, for nitrogen is determined by the following equation:

$$\begin{aligned} z &= 1 + B \cdot \rho_M + C \cdot \rho_M^2 \\ \rho_M &= \frac{P}{R \cdot T} \\ B &= 131{,}21 + 0{,}651\ 12 \cdot T - 7{,}636E - 04 \cdot T^2 \\ C &= 3\ 454{,}2 - 11{,}35 \cdot T + 1{,}5E - 02 \cdot T^2 \end{aligned} \quad (8)$$

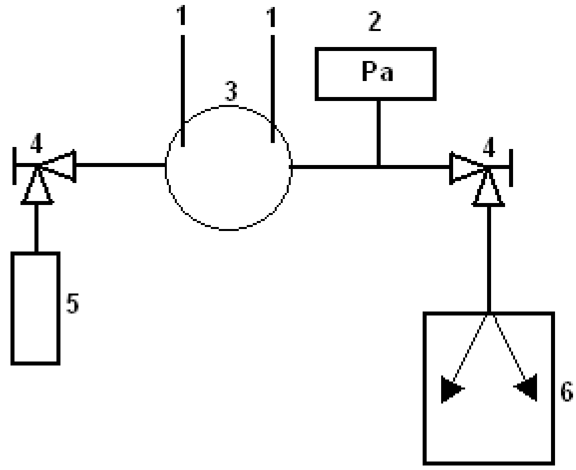
Where:

- B *and* C are the virial coefficients,
- ρ_M is the molar density (mol/m^3).

3. Measurement Systems

3.1 Measurement System for Leaks Calibration

Figure 1 shows the measurement system for leaks calibration. This system has two temperature sensors, an absolute manometer or vacuum measuring sensor (with an appropriate range), two valves for high vacuum, a pumping system which consists of a mechanical pump and a turbo molecular pump, and the leak to be calibrated.



1) Temperature sensors, 2) Absolute manometer CDG, 3) Volume, 4) Valves for high vacuum, 5) Leak to be calibrate, 6) Pumping system, mechanical pump and turbo molecular pump.

Figure 1. System for the calibration of leaks.

The calibration procedure consists on evacuating the system by means of the pumping system having the leak valve closed until a low residual pressure is reached. If a good residual pressure cannot be reached, the system should be baked to a temperature up to 150 °C for 12 h with the vacuum pumps working; this procedure should reduce the out gassing (to minimize virtual leaks) and clean the internal surface of the volume.

The virtual leak Q_0 due to the outgassing of the internal surface of the measurement system should be measured. The leak should be removed and a plug should be installed instead, with the two valves open to evacuate until the residual pressure is reached. Then, the valve of the pumping system should be closed and the measurement of the pressure increment in the absolute gauge in function of the time should start; with these data and with equation 3, Q_0 can be calculated.

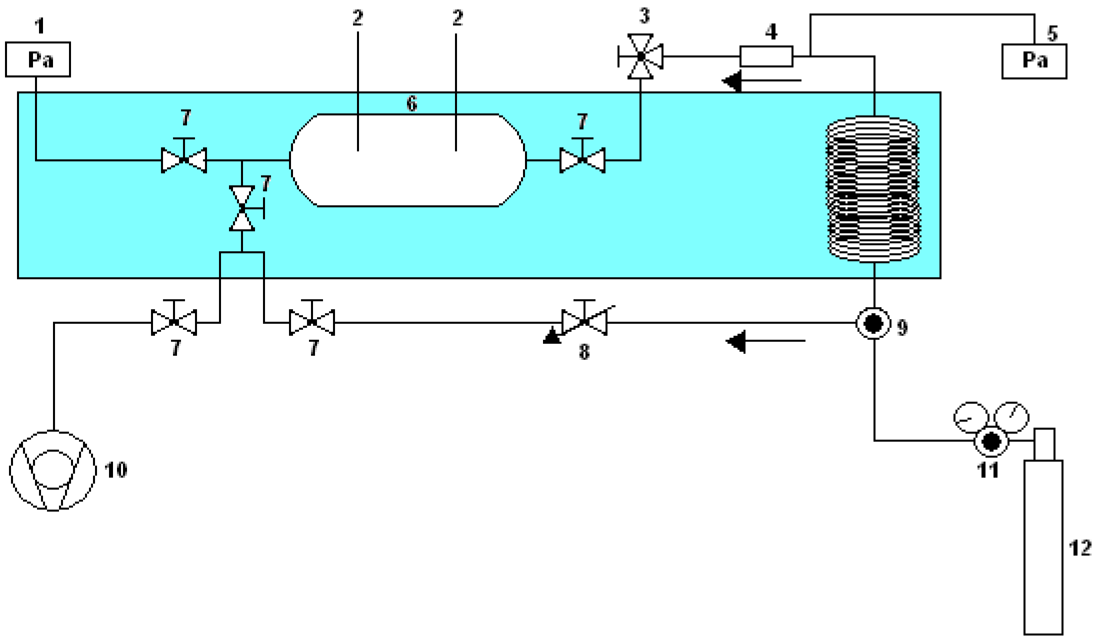
To measure the leak to be calibrated, the leak is installed in the system. With the leak valve opened we evacuate until the minimum pressure is reached, then the valve of the pumping system is closed and the measurement of the pressure increment with the time begins; this measurement is repeated six times. And the leak Q_0 is measured again. The leak value is calculated from:

$$\overline{Q}_m = \frac{\sum_{i=1}^{6} Q_{m\,i}}{6} - \left[\frac{Q_{0_{inicial}} + Q_{0_{final}}}{2}\right] \quad (9)$$

3.2 Measurement Holes Calibration System

Figure 2 shows the components of this system. It has an absolute manometer that is connected to a cylinder to measure its pressure increment due to the gas flow coming from the hole, two temperature sensors (connected to the cylinder), a two way valve that directs the gas flow towards the volume or to the atmosphere, a set of valves to help the operation of the system, the hole to be calibrate, a manometer which registers the pressure applied to the hole, a vacuum pump for evacuating the calibrated volume (cylinder), a system container tank (filled with fluid) used for the calibration, two pressure regulators are used to reduce and adjust the correct pressure applied to the calibrated volume or cylinder.

The nitrogen supplied by a cylinder passes through a spiral tubing which is submerged in the fluid of the tank (a thermostatic water bath) together with the calibrated volume to maintain temperature stability of the system.



1) Absolute manometer, 2) Temperature sensors, 3) Two way valve, 4) Hole to be calibrate, 5) Manometer, 6) Volume, 7) Valves, 8) Flow control valve, 9) Low pressure regulator, 10) Vacuum pump, 11) High pressure regulator, 12) Nitrogen tank.

Figure 2. System for holes calibration.

For holes calibration, these should be installed in the calibration system using the necessary fittings with flow direction toward the volume, initially we should evacuate the system with a vacuum pump, after the system is filled with an adequate gas for the calibration by means of the thank until the specific pressure that the hole need for calibration. The inlet pressure of the hole is adjusted using the low pressure regulator fixed to thank and with the three way valve open to atmosphere, the stabilization of the leak is allowed for the necessary time and it is registered by the increments of pressure during the time.

4. Conclusions

The Pressure and Vacuum group at CENAM has established two measurement systems, one for the calibration of leaks and another one for the calibration of holes. Both are based on the method of pressure increment at constant volume at a given time. The first system was designed for leaks calibration with a range from 1 x 10^{-6} $Pa \cdot m^{-3} \cdot s^{-1}$ up to 5 $Pa \cdot m^{-3} \cdot s$, using a spherical volume of 0.5 L. The second measurement system for holes calibration or micro flows has a measurement range up to 20 cm^3/min, with a volume of 3.75 L.

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