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# The determination of volume ratios by gas depletion through multiple expansions

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**Abstract**

We present a new technique for the determination of the volume ratio of two vacuum chambers connected through a valve. The method is based on the measurement of the pressure in a chamber filled with a gas that is repeatedly depleted by expansion in a second chamber that was previously evacuated. Our calculation shows that under the reported measurement conditions, this technique has an uncertainty comparable to that obtained from the gas accumulation technique [Elliott KWT, Clapham PB.The accurate measurement of the volume ratios of vacuum vessels. NPL Report MOM 28, January 1978]. An excellent equivalence between the results obtained with this new technique and the measurements obtained by the gas accumulation technique is demonstrated.


*Keywords:* Volume ratio; Static expansion

## 1. Introduction

Volume ratio determination is a key characterisation used in the traceability of static expansion systems that are becoming popular for small pressure generation [1–4]. The methods used for the determination of the volume ratio can be gravimetric, dimensional or related to pressure measurement [2,5–7]. We present a new technique based on pressure measurement that provides an alternative method to establish the traceability of a static expansion system and thus, improves the confidence in the results of the characterisation of the system.

## 2. Experimental procedure

The basic element of a static expansion system is a pair of chambers, a large one with a volume $V_l$ and a small one with a volume $V_s$, which are connected through a valve called the expansion valve. The small chamber is closed at the other end by a valve called the inlet valve that is normally used to introduce a gas at a known pressure, but, in this work, it is used to generate a vacuum after the expansion.

The determination of the volume ratio of the pair of chambers is performed by first filling the large chamber with a known gas at a determined pressure. Typically we use nitrogen and argon at 100 kPa. The expansion valve is closed at this stage and the inlet valve is opened, creating a vacuum in the small chamber. The inlet valve is then closed and the expansion valve opened, causing a fast but limited drop of the pressure in the large chamber. After a thermalisation time of typically 5 min, the expansion valve is closed slowly to avoid a non-equilibrium condition due to the movement of the piston in the valve. The pressure in the large chamber and the temperatures are recorded at this stage. The inlet valve is then opened to evacuate the small chamber. The cycle is repeated a number of times that is of the order of the ratio between the two volumes $V_l/V_s$. This means that for a large chamber of 30 l and a small chamber of 0.3 l about 100 cycles are needed.

The static expansion system of METAS (MSE1) that was used for the experimental part is schematically shown

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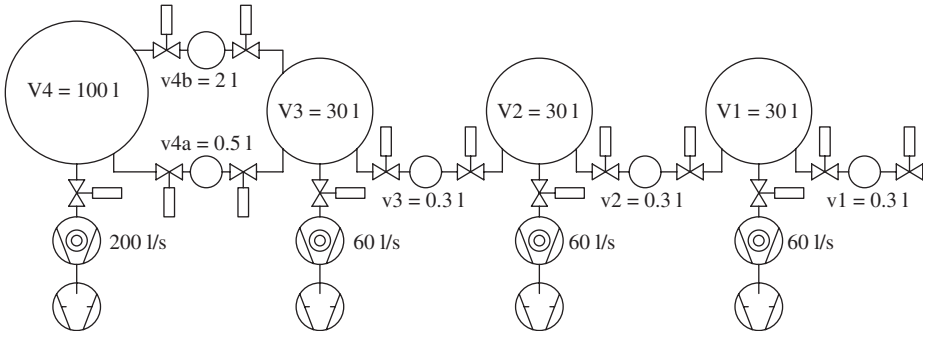


Fig. 1. Front view of the static expansion system.

on Fig. 1. The whole static expansion system consists of four pairs of chambers mounted in cascade in order to achieve a pressure reduction ratio up to $5 \times 10^{-9}$. The first two pairs of chambers in the MSE1 have a volume ratio of about 100. The third and fourth large chambers communicate through small chambers of 0.5 or 2 l, allowing the selection of two different expansion ratios for the last expansion stage. Depending on which expansion valve is opened with the next chamber, chamber 3 has a volume ratio of, respectively 100 or 105. Chamber four has a volume ratio of about 200 respective to the 0.5 l chamber and a volume ratio of 50 respective to the 2 l chamber.

The pressure in the large chamber is measured with a quartz resonant pressure sensor. During the measurement by gas accumulation, another quartz resonant pressure sensor is used to measure the pressure in the small chamber before expansion. A set of negative thermal coefficient (NTC) resistances connected to an electronic readout system interfaced through RS232 with a computer is used for the determination of the average temperature of the chambers. Four temperature sensors are placed on the chambers of 30 l and eight sensors on the chamber of 100 l. The temperature of each small chamber is measured by two sensors.

The MSE1 is fully automatic, allowing overnight measurements without the attendance of an operator.

## 3. Estimation of the time needed for the thermalisation of the gas after expansion

The total energy of the gas is not affected by the expansion from a chamber in a previously evacuated volume. However, during the expansion, some of the energy of the gas remaining in the initial chamber is needed to accelerate the gas going in the previously evacuated chamber [8]. Right after the expansion there is a difference of temperature between the gas and the wall of the chamber. The thermalisation is estimated by modelling a gas enclosed in a spherical chamber. The thermal capacity of the gas enclosed in the sphere is given by the product of the volume and the molar specific heat at constant volume divided by the molar volume:

Table 1
Thermalisation time of nitrogen enclosed in a sphere

| Radius (m) | Volume ($dm^3$) | $P = 1$ kPa Thermalisation time (s) | $P = 100$ kPa Thermalisation time (s) |
|---|---|---|---|
| 0.3 | 113 | 21 | 2100 |
| 0.2 | 33 | 9.4 | 940 |
| 0.08 | 2 | 1.5 | 150 |
| 0.05 | 0.5 | 0.6 | 60 |
| 0.04 | 0.3 | 0.4 | 40 |

Values for different volumes of sphere and two value of pressure are given.

$$\chi = C_v \frac{4}{3}\pi r^3 \frac{p}{RT} \tag{1}$$

for a diatomic gas ($N_2$) we have: $C_v = (5/2)R$ which allows to simplify (1):

$$\chi = \frac{10}{3}\pi r^3 \frac{p}{T}. \tag{2}$$

The average heat conductivity from the gas enclosed in the sphere to the wall is given by the product of the heat conductivity of the gas $K$ by the average area of contact and divided by the average length to the wall. The average area of contact is assumed to be the area of a sphere of radius $0.5r$ and the average distance of contact to the wall is assumed to be $0.5r$.

$$\kappa = K\frac{4\pi(r^2/4)}{0.5r} = 2K\pi r. \tag{3}$$

The time constant of the thermalisation process is then given by the ratio of the thermal capacity by the heat conductivity:

$$\tau = \frac{\chi}{\kappa} = \frac{5r^2 p}{3KT}. \tag{4}$$

The results of a numerical application for nitrogen ($K = 0.024\,\mathrm{W\,m^{-1}\,K^{-1}}$) at a temperature of 295 K are shown in Table 1 for various radius of sphere and various pressures.

The volumes of less than 2 l at 100 kPa correspond to the situation of the small chamber in the gas depletion technique. It appears that the worst-case thermalisation