**Method.** Pour 5 ml of water at 36.5 ± 0.5 °C into the inner tube (A), introduce a suppository with the tip downwards and onto that, place the inset (C1 or C2). Note the time which elapses between this moment and the moment when the lower, rimmed end of the glass rod (C1) or the steel rod (C2) reaches the narrowed part of the inner glass tube. Melting or dissolution is then considered as complete.

**01/2008:20923**

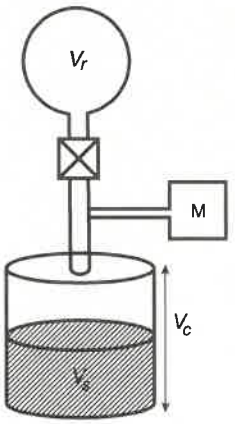
## 2.9.23. PYCNOMETRIC DENSITY OF SOLIDS

The test for pycnometric density of solids is intended to determine the volume occupied by a known mass of powder by measuring the volume of gas displaced under defined conditions. Hence, its pycnometric density is calculated.

APPARATUS

The apparatus (see Figure 2.9.23.-1) consists of the following:

- a sealed test cell, with an empty cell volume ($V_c$), connected through a valve to a reference cell, with a reference volume ($V_r$),
- a system capable of pressurising the test cell with the measurement gas until a defined pressure ($P$) indicated by a manometer,
- the system is connected to a source of measurement gas, which is preferably helium, unless another gas is specified[5].




$V_r$ = reference volume

$V_c$ = cell volume

$V_s$ = sample volume

M = manometer

Figure 2.9.23.-1. – *Schematic diagram of a gas pycnometer*

The temperature of the gas pycnometer is between 15 °C and 30 °C and must not vary by more than 2 °C during the course of measurement.

The apparatus is calibrated which means that the volumes ($V_c$) and ($V_r$) are determined, using calibrated, polished steel balls having a total volume (around 6 $cm^3$) known to the nearest 0.001 $cm^3$. The procedure described below is followed in two runs. Firstly, with an empty test cell and secondly with the steel balls placed in the test cell. The volumes ($V_c$) and ($V_r$) are calculated using the equation for the sample volume taking into account that the volume is zero in the first run.

METHOD

Weigh the test cell of the pycnometer and record the mass. Fill the test cell with a given mass of powder of the substance to be examined. Seal the test cell in the pycnometer. Remove volatile contaminants in the powder by degassing the powder under a constant purge of gas; occasionally, powders may initially have to be degassed under vacuum. Record the system reference pressure ($P_r$) as indicated by the manometer while the valve that connects the reference cell with the test cell is open. Close the valve to separate the reference cell from the test cell. Pressurise the test cell with the gas to an initial pressure ($P_i$) and record the value obtained. Open the valve to connect the reference cell with the test cell. Record the final pressure ($P_f$). Repeat the measurement sequence for the same powder sample until consecutive measurements of the sample volume ($V_s$) agree to within 0.5 per cent. The sample volume is expressed in cubic centimetres. Unload the test cell and measure the final powder mass ($m$) expressed in grams.

EXPRESSION OF THE RESULTS

The sample volume ($V_s$) is given by the expression:

$$V_s = V_c - \frac{V_r}{\frac{P_i - P_r}{P_f - P_r} - 1}$$

The density ($\rho$) is given by the equation:

$$\rho = \frac{m}{V_s}$$

**01/2008:20925**

## 2.9.25. DISSOLUTION TEST FOR MEDICATED CHEWING GUMS

PRINCIPLE

The test is used to determine the dissolution rate of active substances in medicated chewing gums. This is done by applying a mechanical kneading procedure to a piece of gum placed in a small chamber designed to simulate the process of chewing.

APPARATUS

The chewing apparatus (Figure 2.9.25.-1) consists of:

- 1 chewing chamber,
- 1 vertical piston,
- 2 horizontal pistons with O-rings and sealing rings.

The chewing chamber consists of 4 individual parts:

- 1 central chamber,
- 1 funnel (Figure 2.9.25.-2),
- 2 guides with bushes (Figure 2.9.25.-3).

Funnel and guides are mounted on the central chamber. The O-rings are incorporated in the piston recess with the sealing ring round it; the sealing rings ensure that the chamber is watertight. The horizontal pistons are placed in the chewing chamber through the guides.

The gum is artificially chewed by the horizontal pistons, and the vertical piston ensures that the gum stays in the right place between chews.

Machine speed is controlled to ensure a constant cycle. One cycle (chew) is defined as follows: the horizontal pistons start from their outermost position, move to their innermost

(5) If gases other than helium are used, it would not be surprising to obtain values different from those obtained with helium, since the penetration of the gas is dependent on the size of the pore as well as the cross-sectional area of the penetrating molecule. For example, the pycnometric density of porous materials will be overestimated by a measure using nitrogen by comparison with helium.