

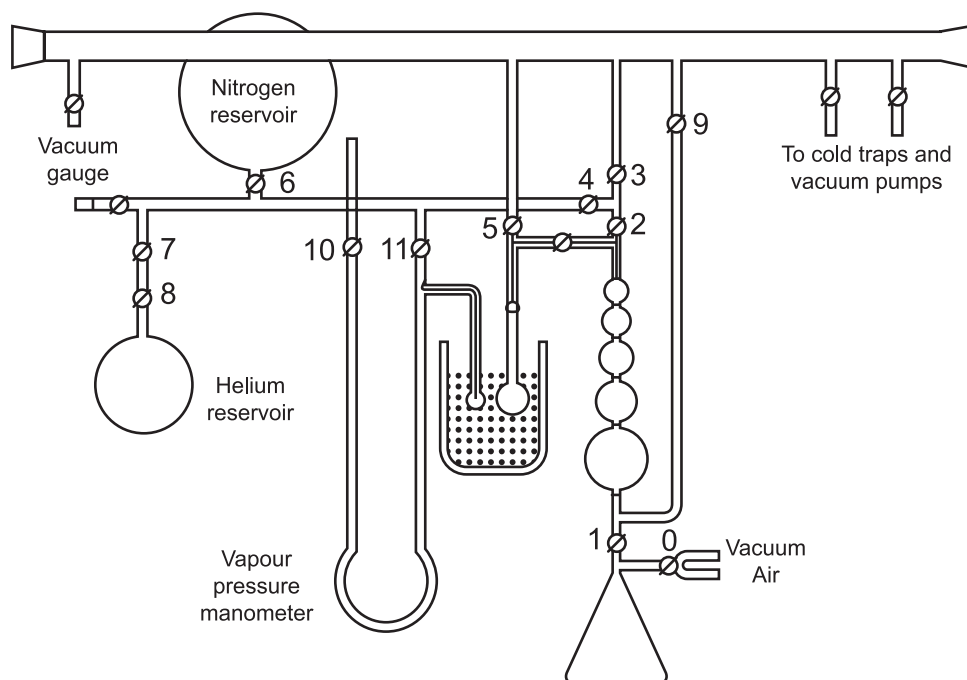


Figure 2.9.26.-2. – Schematic diagram of the volumetric method apparatus

the gas. The use of a diluent gas, such as helium, is therefore unnecessary, although helium may be employed for other purposes, such as to measure the void volume.

Since only pure adsorbate gas, instead of a gas mixture, is employed, interfering effects of thermal diffusion are avoided in this method.

III.2.2.2. Procedure

Admit a small amount of dry nitrogen into the sample tube to prevent contamination of the clean surface, remove the sample tube, insert the stopper, and weigh it. Calculate the weight of the sample. Attach the sample tube to the volumetric apparatus. Cautiously evacuate the sample down to a pressure of 2.66 Pa or less.

If the principle of operation of the instrument requires the determination of the void volume in the sample tube, for example, by the admission of a non-adsorbed gas, such as helium, this procedure is carried out at this point, followed by evacuation of the sample down to 2.66 Pa or less. The adsorption of nitrogen gas is then measured as described below.

Raise a Dewar vessel containing liquid nitrogen at $-196\text{ }^{\circ}\text{C}$ up to a defined point on the sample cell. Admit a sufficient volume of nitrogen gas to give a relative pressure, P/P_0 , equal to 0.10 ± 0.02 . Measure the volume adsorbed, V_a . Repeat the measurement of V_a at P/P_0 values of 0.20 ± 0.02 and 0.30 ± 0.02 .

A minimum of three data points is required. Additional measurements may be carried out, especially on those rare occasions when non-linearity is obtained at a P/P_0 value close to 0.3. Since non-linearity is often obtained at P/P_0 or below 0.05, values in this region are not recommended. The test for linearity, the treatment of the data, and the calculation of the specific surface area of the sample are described above.

IV. REFERENCE MATERIALS

Periodically verify the functioning of the apparatus using appropriate reference materials of known surface area which should have a specific surface area similar to that of the sample to be examined.

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2.9.27. UNIFORMITY OF MASS OF DELIVERED DOSES FROM MULTIDOSE CONTAINERS

The following test is intended for oral dosage forms such as granules, powders for oral use and liquids for oral use, which are supplied in multidose containers provided at manufacture with a measuring device.

Weigh individually 20 doses taken at random from one or more containers with the measuring device provided and determine the individual and average masses. Not more than 2 of the individual masses deviate from the average mass by more than 10 per cent and none deviates by more than 20 per cent.

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2.9.28. TEST FOR DELIVERABLE MASS OR VOLUME OF LIQUID AND SEMI-SOLID PREPARATIONS

The test applies to liquid (solutions, emulsions and suspensions) and semi-solid preparations supplied in single-dose containers where only part of the contents is used.

LIQUID PREPARATIONS

Empty as completely as possible the contents of one container and determine the mass or volume of the contents as appropriate. In the case of emulsions and suspensions, shake the container before the determination. The mass or volume is not less than the amount stated on the label.

SEMI-SOLID PREPARATIONS

Empty as completely as possible the contents of one container. The mass of the contents is not less than that which is stated on the label.