VACUUM FOR BALANCES*

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Abstract

This paper deals with the design of gravimetric apparatus with regard to the requirements of vacuum. Items discussed include the calculation of suction speed and ultimate vacuum, the choice of the pump and of the method of pressure control, and the design of the balance and the balance stand.

Keywords: balance, vacuum, weighing

Introduction

Mass determinations are carried out in vacuum for various reasons [1]:

- to avoid re-contamination of a freshly-prepared surface or cleaned sample before the measurement starts,
- to remove gases acting as diffusion barrier and slowing down evaporation or the reaction of a gas component with the sample surface,
- to examine mass variations due to physical or chemical reactions below atmospheric pressure, e.g. the measurement of adsorption isotherms.

The different tasks require different qualities of the vacuum and this should be considered in the choice of the pump. The balance needs to be suitable for vacuum, and the tubes of the apparatus should be designed in such a way that the gas flow is not restricted. For this purpose, simple rules should be observed, but these seem to be unknown for many balance manufacturers and experimenters. The present paper deals with the design of gravimetric apparatus with regard to the requirements of vacuum.

We have several possibilities to weigh a sample in vacuum [2] (Fig. 1):

a) The sample can be weighed in a closed, evacuated bottle. This may be used to check the sample mass after enclosure.

The main application is in the determination of the mass of a gas filled in an empty cylinder. From the pressure and mass of the gas in a calibrated volume, the density and the molecular mass can be derived.

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* Dedicated to Dr. ir. Carel H. Massen on his 60th birthday
Fig. 1 Methods of mass determination in vacuum. a – Weighing in evacuated bottles, connected by means of flexible tubes to the vacuum pump and the gas supply, b – weighing by means of a vacuum balance arranged in the vacuum vessel, c – weighing of a sample suspended by means of a magnetic coupling and enclosed in a separate bulb.

In order to control the gas pressure, the bottle can be connected by means of a flexible tube to a vacuum pump and gas supply. In this arrangement, weighings are not affected by buoyancy, but disturbances by the flexible tube will decrease the sensitivity. If the counterweight vessel is connected simultaneously, the gas mass does not influence the weighings either. Such arrangements are applied for the investigation of catalytic processes, in particular at high pressure and with occasional evacuation for gas exchange. As conventional balances are used in all such cases, we abstain from discussing these methods.

b) The balance with pans and sample is arranged in a vacuum vessel. For this purpose, special vacuum balances are needed.

c) The sample is in a separate vacuum vessel, suspended by means of a controlled magnet from the balance, which is in the atmosphere.

Theory

The basic equation describing the pressure in a vacuum chamber is

$$p S_{\text{eff}} = -V \frac{dp}{dt} + q$$  \hspace{1cm} (1)

where $p$ is the pressure, $t$ is the time, $S_{\text{eff}}$ is the effective pumping speed and $q$ is the influx of gas. To obtain a low ultimate pressure, it is necessary to have a high pumping speed and a low influx. It might appear from Eq. (1) that the volume of the chamber affects only the pumpdown speed. However, since the desorption of gas contributes to the influx, the extent of the walls and the geometry of the chamber will influence the ultimate pressure as well. The ultimate pressure is reached at the steady state between gas influx and removal, i.e. $dp/dt=0$. In this case, Eq. (1) reduces to

$$p = \frac{q}{S_{\text{eff}}}$$  \hspace{1cm} (2)

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