DISTURBANCES IN WEIGHING - PART II

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ABSTRACT

   Continuing the survey of disturbances which affect the accuracy of weighing
in vacuum or in controlled atmosphere (published in Thermochimica Acta 82
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- convective currents in the balance case
- buoyancy
- sorption effects and contamination
- electrostatic effects.

INTRODUCTION

   In our paper at the Plymouth Conference (see ref.1) we surveyed the
influences of disturbances due to Brownian motion, Knudsen forces, cavity
forces, unequal thermal expansion of balance arms, magnetostatic effects,
radiation pressure and building vibrations. The present paper deals with
buoyancy, sorption effects and contamination and electrostatic effects.

BUOYANCY

   The most typical error in all mass determinations is Archimedes'buoyancy.
When weighing under varying pressure, changing temperatures or in different
gases, the effect of buoyancy may far exceed mass changes to be determined.
Buoyancy therefore has either to be compensated or to be corrected for (see
ref.2-16).On the other hand, buoyancy can be used for density or pressure
measurements (see ref. 8-10).

Using a force measuring device as a mass sensor the effect of buoyancy equals the mass of the gas displaced by suspension, pan and sample and is proportional to the density of the gas.

For a gas obeying the equation of state

$$p V = n R T \tag{1}$$

the force difference ΔF is

$$\Delta F = g M p V/R T \tag{2}$$

where p is the pressure,

   n = number of moles,

   V = displaced volume,

   R = universal gas constant,

   T = absolute temperature,

   M = molecular mass of the gas,

   g = gravitational acceleration,

With an equally armed balance we have the possibility to reduce the buoyancy error because instead of the total volume displaced only the difference of volume on either side of the fulcrum is indicated and this volume difference can be minimized by using materials of the same density. For a thermobalance matters are complicated due to the sample, counterweight and beam being at different temperatures. Furthermore, with sorption experiments volume and mean density of a porous sample is changing because at high relative pressure remarkable amounts of liquified gas are taken up. To avoid errors in evaluation, the buoyancy of the beam should be eliminated completely using gold and aluminium riders at the opposite balance arms, the mass of which can be calculated on the basis of equation (2). The residual, pressure proportional buoyancy $m_B(p)$ results as the sum of individual effects from the different parts:

$$m_B(p) = - V_a(p/p_o)\,\rho(T_s) - V_s\rho(T_s) - V_p\rho(T_s) + V_c\rho(T_c) + V_p\rho(T_c) \tag{3}$$

The actual mass seems to be reduced by the buoyancy of the adsorbate which depends on the relative gas pressure p/p_o and the density ρ of gas at the local temperature (neglecting that at the counterweight), the buoyancy of the sample and that of pan and suspension and to be increased by the buoyancy of counterweight and its pan and suspension.

As a practical consequence, pan and suspension at each side should be equal in material and mass, the sample should be counterbalanced by a weight of the sample's density and both counterweight and sample should be held at equal temperature. In general the counterbalance consists of a mixture of different materials, e.g. quartz and gold or a hollow glass sphere so that the thermal expansion differs from that of the sample. This produces an error however an order of magnitude smaller.

In sorption measurements, the buoyancy effects of the adsorbate is mostly negligible. For accurate measurements, however, even density anomalies as a result of packing effects of the adsorbate have to be taken into account, which are in the range below permille (see ref. 11).

SORPTION EFFECTS AND CONTAMINATION

Disturbances as a result of dissolved water or organic vapour in glue for knife edges or in the coil insulation were often discussed   at earlier VMT Conferences. Meanwhile most vacuum and thermobalances were improved in this respect. Only amalgamation by mercury vapour and corrosive gases continue to be a problem solved only by the quartz spring and the magnetic suspension balance.

Sorption (see ref. 17-24) takes place at the sample and at all parts of the balance not only during the measurement but also in the high vacuum at the beginning and end affecting the zero point.

At roomtemperature and a pressure of 10^4Pa the outer surface of the sample is covered with a monolayer within 1 min (if the sticking probability is 1). Residual gases in an evacuated balance include hydrogen, helium, oxygen, nitrogen, water, methane and higher organics. With exception of helium all the species should be considered with regard to chemisorption. Physisorption mostly takes place with water and organics. Frost-like water adsorbate may be harmful at cryogenic temperatures on account of its large surface. Countermeasures are better vacuum, time-saving preparation and baking. To avoid readsorption after baking the balance tubes should be cooled quickly, so that they can act as baffles, and the measurement should be started as quickly as possible.

During measurements, in particular sorption at the counterweight of water vapour originating from ambient air or insufficiently dried gases or of organic vapours from pumps or sealing material may be sources of error. In general, the influence is negligible because up to a relative humidity of 0.8 only about one monolayer is adsorbed, the mass of which amounts to 0.01 µg per cm^2. Near the saturation point with eight monolayers, the correction is only 0,1 µg. More serious are fingerprints consisting of salts and grease which take up remarkable amounts of water and organic vapours (see ref. 20). Fingerprints can be removed using water and acetone.

With sorption experiments near the saturation point condensation in the counterweight pan can be avoided by keeping the temperature there somewhat higher than at the sample, e.g. by adding some permille of oxygen to the nitrogen bath.

ELECTROSTATIC EFFECTS

Electrostatic charges may originate from several causes during the weighing procedure. Typical examples occur when a powder is filled in the pan, when at the outer side of the hangdown tube the nitrogen bath is removed and when ice is generated from the water vapour out of the air. It is advisable to ground both the hangdown wire and the hangdown tube. If only one of them is grounded this part may, similar to a grounded condensator plate, collect influention charge. The resulting force can be estimated by assuming the primary and the influenced charge to be equal point charges of opposite sign and using Coulomb's law.

$$F = \frac{Q^2}{4\pi\varepsilon_o r^2} \qquad (1)$$

and

$$V = \frac{Q}{4\pi\varepsilon_o r} \qquad (2)$$

where Q is the charge

    r is the mutual distance

    F is the force

    V is the potential difference and

    $\varepsilon_o = 10^{-11} \frac{As}{Vm}$

From equations (1) and (2) it follows:

$$F = 4\,\mathcal{E}_c\,V^2 \approx 10^{-10}V^2 \tag{3}$$

Equation (3) makes estimation of the mass error m easy as F is only
dependent upon V

$$m \approx 10^{-11}V^2 \quad (\Delta m \text{ in kg})$$

The voltage differences accompanying transport of powders, gases or
liquids can easily amount to thousands of volts. The error m can then
amount to more than 10 mg. As mentioned before the effect can be eliminated
by grounding both balance and balance case.

A METHOD TO ELIMINATE MECHANICAL VIBRATIONS

   In order to eliminate erroneous forces caused by vibrations, harmonic
as well as stochastic ones, an additional sensor to measure the acceleration
can be combined with a circuit, of determined transfer function, whose output
is fed to the electrodynamic force meter of the balance. If the parameters of
this device are properly chosen, the influence of vibrations will be con-
siderably reduced.
   In balances with magnetic suspension coupling, the coupling itself can
be used as a accelerometer. In this case, the transfer function depends on
load. It is possible to correct the deviation by adaptive control (ref. 25).

Compensating of Errors by Vibrations using
the Magnetic Coupling as Accelerometer

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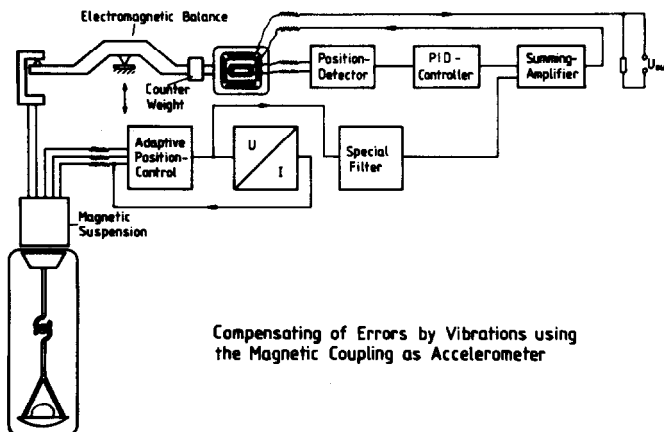
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