110. *An Electrical Sorption Balance.*

By S. J. Gregg.

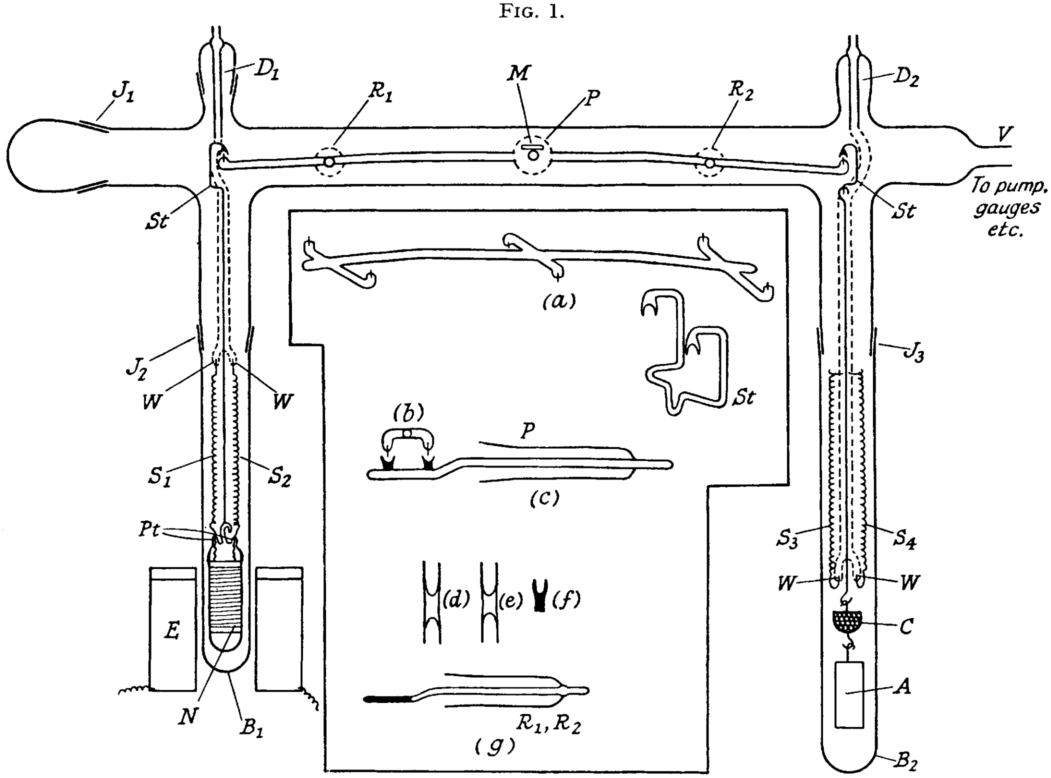
The sorption balance, which utilises an electromagnetic principle, has a sensitivity of *ca.* 0·0003 g., which is independent of the total load (10—20 g.). It is suitable for measuring adsorption, rate of decomposition, rate of evaporation, rate of effusion, etc. Its maximum range is about 0·4 g. of adsorbate.

The sorption balance here described has been designed to overcome the drawback, inherent in most existing designs, that a high sensitivity demands a low total load. Its sensitivity, which is of the order 0·0003 g., is independent of the total load, which may be of the order 10—20 g. The change in weight of the adsorbent due to uptake of adsorbate is measured by counterbalancing it electromagnetically. In principle it is similar to the instrument described by Blewett (*Rev. Sci. Instr.*, 1939, **10**, 231) in which an external solenoid interacts with a magnet attached to the balance arm, except that the magnet is replaced by a solenoid carrying a current, thereby eliminating any magnetic hysteresis effects. Anderson (*Trans. Faraday Soc.*, 1915, **11**, 69) used a very similar idea, having one solenoid hung from the arm of an ordinary balance, and two stationary solenoids; the balance was housed in a bell-jar which could be evacuated. This arrangement is not very convenient for out-gassing and similar operations, however, and not suitable for high-vacuum work.

Experimental.

In the present model the deflection of the balance arm is followed by an optical lever, and the arm is brought back to an arbitrary zero by adjusting the currents in " exterior " solenoid E and if necessary in " interior " solenoid N (Fig. 1); the force exerted between E and N is proportional to the product of the currents they carry. The balance can be

Fig. 1.



calibrated by hanging known weights on the right-hand side, and measuring the external and the internal current, $I_{ext.}$ and $I_{int.}$, when the arm has been brought back to its zero position. Small changes in weight can be followed by the scale deflection, a separate calibration for weight versus scale deflection having been carried out.

The balance beam is of Pyrex tubing (*ca.* 5 mm. in diameter) with small side tubes sealed on and then cut off short, forming sleeves which accommodate cut-off sewing needles serving as bearings (Fig. 1*a*). There are two such needles in the middle and two at each end; they bear on hemispherical depressions in glass, made by sinking in a piece of Pyrex tubing, about 4 mm. in diameter, near one end [Fig. 1(*d*)], then blowing gently [Fig. 1(*e*)], and finally cutting off [Fig. 1(*f*)]. The main glass bearings, *i.e.*, those for engaging the two middle needles, are fused on to a glass support P which is joined on to a standard ground glass joint by an internal seal [Figs. 1, 1(*b*), 1(*c*)]. Small glass stirrups *St* are provided at each end of the beam to accommodate the glass suspension rods which carry the loads [Figs. 1 and 1(*a*)].

The needles are fitted into their sleeves by means of vacuum wax or Wood's metal; by gently moving the arm and the glass bearings relatively to one another whilst the cementing material is still warm (before erecting the balance) it is possible to " feel " the position of minimum potential energy of the needle points with relation to the glass hemispheres;

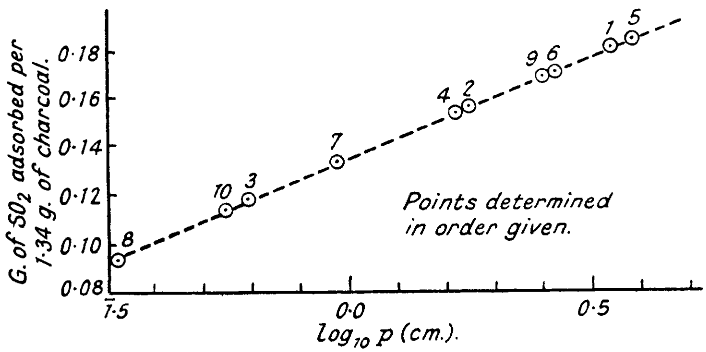
they are then held in this position till the cement solidifies. This procedure ensures that in use the needle points shall always return to the same zero position after adventitious displacements due to jarring. A galvanometer mirror M is mounted at the centre of the beam and reflects a spot of light on to a scale. The sensitivity of the balance is fixed, by trial and error, by suitable bending of the arm in the flame before erection. The balance is then mounted in a " case " of Pyrex tubing about 40 mm. in diameter with No. B34 standard ground glass joints at J_1, J_2, and J_3 (Fig. 1).

The inner solenoid N, of 1000 turns of 36 S.W.G. silk-covered copper wire, is enclosed in a soda-glass container provided with a hook at the top, the two leads being taken through platinum seals (Fig. 1). To conduct the current into the solenoid from the outside of the balance case, two springs S_1 and S_2 of fine wire are used. In the present model each spring consists of 80 turns of 42 S.W.G. copper wire, the turns being about 4 mm. in diameter; platinum wire can be used, but the maximum permissible current is thereby reduced. An identical pair of springs (S_3 and S_4) is attached to the other arm and mounted in such a sense that the two sets mutually compensate, in that both of the sets stretch together and both contract together. This compensating device ensures that the springs shall not appreciably reduce the sensitivity of the balance or introduce excessive damping. The compensation springs can be used as leads to a resistance thermometer incorporated in the adsorbent container, when it is desired to measure heats of adsorption (see next paper); when not so used, they may be connected in series with the solenoid springs S_1 and S_2, thereby compensating any effect on the elasticity of these springs due to the heating effect of the small current, *ca.* 0·05 amp., which they carry. Connection from the four springs through the balance case is made by mercury–tungsten seals W (platinum seals through Pyrex glass are not vacuum-tight) attached to the lead-in tubes D_1 and D_2 which accommodate copper leads.

The outer solenoid E is 1000 turns of 22 S.W.G. copper wire, and will carry a maximum current of about 1·2 amp. without overheating.

In addition to the adsorbent container hung from the right-hand arm, there is a counterpoise C; a small sealed bulb containing mercury, or a small glass bucket which can be charged with lead shot, is suitable. The weight of the counterpoise is such that the balance beam can, at the commencement of the experiment, be brought to its zero position by a very small current in the outer solenoid. The container is hung on to the counterpoise by means of a small glass hook on the latter.

FIG. 2.



At R_1 and R_2 there are arrestments of glass rod, fixed by sealing through small side tubes joined horizontally and at right angles to the axis of the top tube of the case. The side tubes may be provided with ground glass joints if desired, thereby permitting some adjustment. When not in use, the balance beam can be gently lowered on to arrestment R_1 by gradually altering the current in E; after thus lowering and then re-floating the beam again, the value of the weight can be reproduced within $\pm 0·0002$ g.

For out-gassing the adsorbent, the lower end of B_2 is surrounded with a heater at the desired temperature, and pumping commenced through the connection V. When out-gassing is complete the heater is replaced by a thermostat at the required temperature; before commencing measurements a period of ½—1 hour should be allowed to elapse so that the adsorbent may take up the temperature of the thermostat.

The balance is rapid in operation, and readings of the weight may be taken every 15 seconds or so if desired. The maximum adsorption it can measure is of the order of 0·4 g., but by increasing the number of turns in the solenoids this could be increased. Steps are now being taken to render the balance automatically recording.

It has been in use for some time and has been found suitable for the following types of work: (1) Adsorption isotherms: the result for a typical example—sulphur dioxide on sugar charcoal at 25°—is shown in Fig. 2. (2) Rate of decomposition of a solid in a controlled atmosphere—*e.g.*, dehydration and rehydration of a crystal hydrate, or decomcomposition of a carbonate in an atmosphere of carbon dioxide at known pressure. (3) Rate of evaporation in a vacuum of a crystalline substance. (4) Determination of small vapour pressures by an adaption of the effusion method.

In interpreting the results of measurements under headings (2), (3) and (4), it is necessary to bear in mind the variation in temperature of the working substance due to heat of decomposition, of evaporation, etc. In many cases it is probable that a steady state is soon set up in which the heat effect of the reaction just balances the gain or loss of heat by the working substance from or to the surroundings; the measured rate of reaction then corresponds to isothermal conditions with the temperature of the working substance differing by a definite amount, $\Delta T°$, from that of the thermostat. In favourable cases it is possible to make a reasonably good estimate of $\Delta T°$. In ordinary adsorption work, however, where a charge of gas is suddenly admitted to or withdrawn from the adsorbent, the temperature of the latter may rapidly change by several degrees (see next paper); the balance still accurately measures the rate of adsorption, but it is not an isothermal rate.

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