An Electromagnetic Sorption Balance.

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An electromagnetic sorption balance is described which permits one to follow the change in weight of a solid either *in vacuo* or when exposed to a gas at a temperature between −200° and 1200° c. A change in weight of up to 4 g. (±0·1 mg.) on 25 g. may be measured.

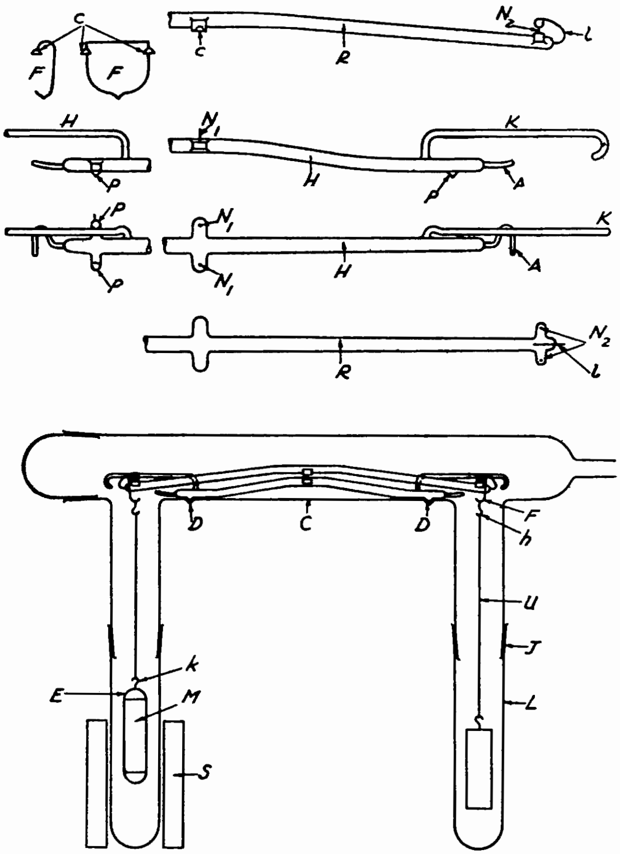
Earlier (*J.*, 1946, 561), an electrical sorption balance was described, which has proved useful for following the change in weight of a solid, in a vacuum or a gas, maintained at a fixed temperature or subject to a rising temperature, between −200° and 1200°. Such balances have been useful in sorption experiments (*e.g.*, Gregg and Sing, *J. Phys. Colloid Chem.*, 1951, **55**, 597), in thermogravimetric analysis in air or in a vacuum (Gregg and Stephens, *J.*, 1953, 3951; Gregg, Parker, and Stephens, *J. Appl. Chem.*, 1954, **4**, 666), and in measurements of the rate of reactions of the type Solid *A* ⟶ Solid *B* + Gas, or Solid *B* + Gas ⟶ Solid *A* (Britton, Gregg, and Winsor, *Trans. Faraday Soc.*, 1952, **48**, 63, 70). Nevertheless, certain drawbacks were encountered; in particular, (1) it is troublesome to take it down for repairs or cleaning, and to reassemble it; (2) the numerous ground-glass joints considerably increase the likelihood of leakage in vacuum or sorption experiments; (3) the range of weight variation that can be studied is rather limited (*ca.* 0·5 g.); (4) the use of the springs for leading in the current to the inner solenoid results in reduced sensitivity. The modified design described now reduces or overcomes these disadvantages.

Points (1) and (2) are dealt with by incorporating the main bearing and the arrestments in a single holder of Pyrex glass, such that the balance arm, already mounted on its holder, can be slid as a single unit into position in the balance case. Details will be gathered from the Figure. The holder *H* is provided at each end with the hooks *K* for ease of handling, and it has three pips *P* which register with the corresponding depressions *D* in the balance " case " *C* and so fix the holder *H* in position. The needles N_1 are held in small cylindrical holes in the glass by means of Wood's metal or wax; alternatively, if sapphire needles are used they can be fused into the glass. The arrestments *A* are formed by drawing out the end of *H* and bending it so that the movement of the ends of the balance arm *R* is restricted to 0·8 to 1·2 mm. The balance arm (*ca.* 25 cm. long) is of Pyrex glass and has cups *c* at the centre and needles N_2 at the two ends, each carrying a stirrup (*F*) from which hang the leads. The stirrups are made as small as possible, and are prevented from falling off the arm by the loops *l* of platinum wire, which still permit free movement of the stirrups when the balance is in use. The small platinum hooks *h* facilitate the otherwise rather delicate operation of hanging the suspension rods *U* on to the stirrups when the arm is in position.

The inner solenoid of the earlier model is replaced by a cylindrical magnet *M* (5 cm. × 1 cm.), magnetised along its length and made of Alcomax III, an alloy chosen because its magnetic characteristics are such as to give no appreciable hysteresis effects at the field strengths employed; it is contained in a glass envelope *E* provided with a hook *k*. By use of the magnet it is possible to dispense with the lead-in springs of the earlier model, and also to increase the range to several grams with a current through the solenoid *S* of a few mA at the most. A suitable specification of the solenoid is as follows: a cylindrical former of Paxoline, 80 mm. long by 31 mm. diameter, is wound to a depth of 8 layers with 35 S.W.G. copper wire, double silk covered or enamelled, producing a total resistance of about 100 ohms; with two 2-volt accumulators this coil gives a total range of 4 g. or so. The current should be measured potentiometrically to give a sufficient degree of precision; an ammeter is useful for purposes of indication.

For determination of the null position of the arm an optical projection system has been found more convenient than an optical lever. An enlarged image of some suitable moving part, *e.g.*, the hook *k* of the glass magnet case, is formed on a ground glass screen carrying a small graduated scale; the null position is then chosen at a fixed distance from one or other of the two readings obtained when the arm is touching the arrestments. In this way errors due to slight shifts in the optical system or in the position of the balance case are eliminated. Suitable arrangements are: projection lamp 40 w, with a lens of 20 cm. focal length (f) placed approximately 20 cm. in front of it and distant 25 cm. from the hook k; after passing k the light beam passes through a lens (f = 10 cm.) placed 6 cm. in front of k, and is brought to a focus on a screen 60 cm. from k; a plane mirror is placed immediately behind k—outside the case—so as to turn the beam into a convenient direction.

The precision of the balance is *ca.* 10^{-4} g., and the zero is reproducible after several days; the calibration, in milliamp. per gram (of the order of 1 mA per g. for the above specification),



in general varies by only one per cent. or so over a period of months, but should be checked at frequent intervals. The total load on each side of the balance can be 50 g. or more, so that the weight of the working substance may be as high as 25 g. or so if necessary.

The now disused lead-in connections on the " bucket " side of the balance may be used for a resistance thermometer or a thermocouple to measure the temperature of the sample. Up to about 450° limb L may be of Pyrex glass, but between 450° and 1000° fused silica or mullite should be used. From 1000° to 1350° mullite is satisfactory, and for making connection with the rest of the balance case may be fused on to a standard joint of Pyrex. At high temperatures it is necessary to protect the joint J by a water-cooled aluminium baffle.

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