A Simple Robust Ultramicrobalance

By

H. Asbury, R. Belcher and T. S. West

With 13 Figures.

(Received April 12, 1955.)

Several balances are available commercially which can be used for weighing ultramicro quantities of material. In the course of an investigation in which extremely small amounts of material had to be analysed, such a balance was required, but the cost was prohibitive. Accordingly, a suitable balance was designed and constructed. The simple construction and robust nature of this balance should be of general interest.

Historical

In 1841, Weber\(^1\) constructed a beam microbalance of the elastic filament type, using thin metal strips for the purpose. A fused quartz beam was employed by Steele and Grant\(^2\) in 1909. This latter balance used the familiar knife edge as a pivot, but silica suspension fibres were used for the scale pans. Pettersson\(^3\) subsequently modified the latter balance by suspending the beam itself from vertical silica fibres. This balance detected a mass difference of 10^{-9}g under a load of 20 mg. Naturally such small differences in weight could not be measured by the use of a series of conventional type weights, and other means of measurement had to be employed. Pettersson, and Steele and Grant used buoyancy bulbs and variable air pressure for this purpose, but such a device is clumsy and is not suited to weighing materials which may lose weight under partial vacuum.

In 1901, Salvioni\(^4\) constructed a displacement type balance consisting of a glass thread fixed rigidly at one end and carrying a minute scale-pan near the other end. The deflection of the free end of the thread under various loads was measured by means of a travelling microscope. The original balance was suitable for microanalytical determinations, but other workers subsequently evolved much more sensitive balances of the same type. It was on a Salvioni type of balance that Cunningham and Werner\(^5\) weighed the first amounts of plutonium to be isolated.

In 1903 Nernst and Riesenfeld\(^6\) devised a torsion displacement balance using a fused quartz beam cemented at right angles to a horizontal torsion...
fibre. One end of the beam carried the scale-pan attached to a fine suspension fibre, whilst the other carried a quartz counterpoise and a pointer moving over a graduated scale. The displacement of the pointer was proportional to the load placed on the scale-pan. The original Nernst balance had a total load capacity of 2 mg, and a sensitivity of 2 \( \mu \)g was obtained by using a cathetometer to observe the movement of the pointer. Modifications by Riesenfeld and Moeller, and by Emich and Donau increased the load capacity to 15 mg, and the sensitivity to 0.1 \( \mu \)g. The Nernst balance was examined by Steele and Grant, but they preferred to evolve a knife-edge balance, because they failed to obtain sufficiently reproducible weighings with the former.

One of the most notable contributions to the evolution of a simple yet accurate balance was made fairly recently by Neher when he introduced the application of variable torque to the quartz beam by a torsion fibre. The angle of rotation of the free end of the torsion fibre necessary to restore the beam under load to a predetermined zero position was directly proportional to the load. Neher's balance had a sensitivity of ca. 0.001 \( \mu \)g with a load capacity of 1 mg.

_Kirk, Craig, Gullberg, and Boyer_ used various features from the balances of Steele and Grant, Pettersson, and Neher in designing an instrument more suited to ultramicro analytical work. In this balance a fused quartz rod beam of cantilever construction was mounted on a quartz torsion fibre. The free end of this fibre was attached to a calibrated torsion head. Constant tension on the torsion fibre was achieved by inserting a flexible quartz bow at the other end of the torsion fibre. The amount of torsion necessary to restore the beam to its original position was read from the calibrated dial.

In 1950, Wilson and El-Badry described an ingenious method for the construction of a Kirk-Craig type balance. The beam of the Kirk-Craig balance was made entirely of fused quartz, all the joints being made by fusion of the various members. This rather delicate operation was achieved by using a special jig assembly to hold the fibres in place. The balance devised by Wilson and El-Badry differed only in minor details from that of Kirk, Craig and co-workers, except that an entirely new method was used in assembling the beam. The construction of a fused beam requires complicated manipulation and strict cleanliness, because contamination of the quartz with foreign material of any kind leads to the formation of brittle joints. Wilson and El-Badry simplified the construction of this type of balance greatly by showing that the beam could be joined together very simply with an organic water-repellent adhesive such as "Durofix". The joints thus obtained were strong and neat, being almost invisible to the eye. The beam assembled by this method was rigid and could survive relatively rough treatment.

_Carmichael_ subsequently described the design and performance of a fused silica microbalance in which the beam was essentially similar to those used by Kirk, Craig and co-workers and by Wilson and El-Badry. Various refinements in construction were made, but the most notable feature of its construction was the use of a metal block (channeled to hold the various moving parts) to house the whole beam assembly. By this device the undesirable effects introduced through thermal convection (caused by temperature differences in the large volume of air enclosed in the cases of earlier balances) were avoided.

The total load capacity of most of the balances already described is rather limited, but quartz fibre balances of the suspension-torsion type have a much larger load capacity. Thus the Garner microbalance has a