Chemical Instrumentation

. . . a ChemEd feature

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In this, and the subsequent articles in this series, the basic principles, characteristics, and limitations of chemical instrumentation will be surveyed. We define chemical instrumentation as comprising all those devices that have found important uses in chemical laboratories, ranging from balances and burets to servomechanisms and spectrometers. Our emphasis will be on commercially available equipment, and we shall attempt to provide summaries of the design features of the current offerings of manufacturers. Approximate prices will be quoted in order to indicate the order of magnitude of cost of these various features. The special advantages and/or disadvantages inherent in these designs will be critically discussed to provide the reader with the kind of knowledge he should have in order intelligently to choose and use his equipment.

1. Balances

The analytical balance is probably the most generally used and most important of all chemical laboratory instruments. It represents the foundation stone upon which all exact chemical knowledge rests, and in the past the rate of progress of chemistry has closely paralleled advances in the art of balance construction. Several significant developments in instrumentation for weighing have taken place in recent years, and the chemist now has a considerable variety of balance types from which to choose. The following discussion is intended to provide the scientist or teacher with a basis for deciding which of the available instrument designs is best suited for the particular requirements of his own situation.

In order to measure any physical property it is necessary to have a detector—in instrumentation terminology, a *transducer*—that will respond in a useful way to that property. In modern balances, four basic types of detectors are employed: the cantilever (or flexure spring), the torsion bar, the extension spring, and the lever. These are illustrated schematically in Figure 1.

The mass to be measured is placed on the balance at a distance from the center of support or suspension, and produces a characteristic type of deflection. In the cantilever balance, a horizontal support may bend in the vertical plane; in the torsion bar type, a rod or plate rigidly clamped at one end twists about its long axis; in the spring type, the coil extends; and in the lever type, the beam tilts about its fulcrum. The cantilever, torsion and spring types find application in the most sensitive microbalances, as well as in some of the grosser, larger capacity scales and certain special purpose balances (e.g., the du Nouy tensiometer), but generally not in the intermediate category of macro- and semimicro-analytical balances. For these latter balances, the lever principle has been universally adopted.

Measurement of a Deflection

Given a deflection of the detector, there are two fundamentally different approaches to what shall be done with it. In *direct-deflection* balances, the deflection is directly measured on some convenient scale, and this reading is converted to mass units by reference to the deflections produced by standards. The scale is usually marked in mass units, but its calibration must be checked periodically. The accuracy of the weighing is limited by the reproducibility of the deflection and the correctness of the calibration. Furthermore, the deflection is generally assumed to be a linear function of the mass between calibration points, and since this is almost never strictly true for large deflections, it represents another limitation of this type of balance for work of the highest accuracy.

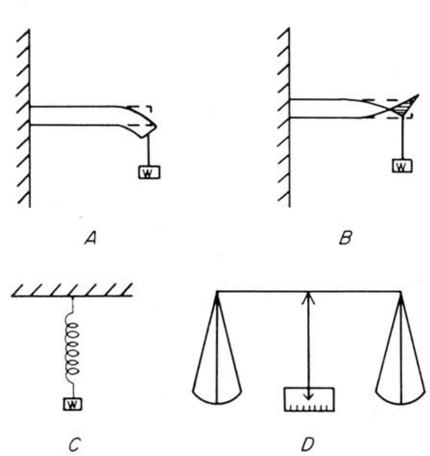

Figure 1. Basic types of weight transducers. A. Cantilever, or flexure spring, B. Torsion bar, C. Extension spring, D. Lever.

The second basic approach consists in applying an equal and opposite force to the detector sufficient to bring the system back to its original position. In other words, the signal (in this case, torque or force) produced by the unknown is compared with and opposed by the signal produced by a series of knowns until the difference in signals (in instrumentation terminology, the *error signal*) is zero. These are, therefore, called *null-balance* instruments. A null-balance device has the advantage that the absolute values of the masses determined with it are completely independent of the relationship, which is generally nonlinear, between force and deflection. The precision and sensitivity depend upon how much unbalance force is necessary to produce a detectable difference from the null condition; i.e., on the magnitude of the minimum detectable error signal. The fundamental difference between weighing by direct deflection and by null balance is illustrated diagrammatically in Figure 2.

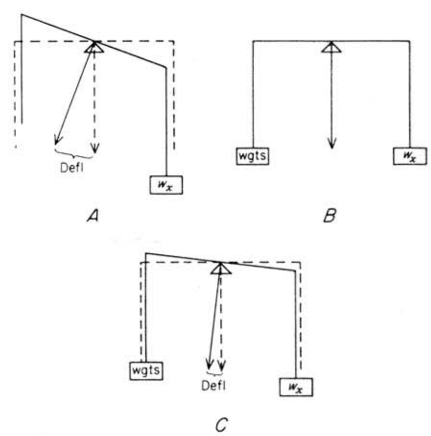

Figure 2. Weighing by direct deflection, null-balance, and combination of both. A. Direct deflection: $w_x \propto$ Defl., B. Null balance: w_x = wgts., C. Combination: w_x = wgts. + (Defl.) × (Sens).

To bring a balance containing an unknown weight exactly to a null balance would be an exceedingly tedious operation. On the other hand, the direct-deflection type of instrument is generally neither precise nor accurate enough for analytical work of the highest accuracy. Therefore, practically all chemical balances are used in a fashion that is intermediate between the two types; that is, an approximate null balance is obtained

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using the larger weights (often including weights down to the milligram level), and the relatively small deflection from the null condition that remains is determined, converted to mass units, and added to yield the total mass (Figure 2*C*). Among modern laboratory balances, models are currently available in which the direct deflection part of the weighing ranges from as much as the last 1000.0 mg of the total mass, to as little as a few units in the last decimal place.

The greater the proportion of the total weight that is determined by the direct deflection, the faster is the weighing process, but the accuracy and precision may be correspondingly diminished. The single-pan balances, to be discussed later, in which speed is a major consideration, and some of the "projection-type" two-pan balances, utilize direct deflection for a greater part of the total weight determination than does the conventional, "old-fashioned" two-pan balance. Where direct deflection is utilized over a large weight range, the scale that is read must be nonlinearly divided if a uniform accuracy is to be maintained for all deflections from small to large. By employing sufficient care in the ruling of the scale or graticule, it is possible to produce direct deflection instruments that are as accurate over their ranges as are the null-balance devices.

Nearly all commercial balances having sensitivities and reproducibilities suitable for analytical chemical work are based upon the lever principle. This is because the lever lends itself to the null balance design more readily and reliably, and with greater sensitivity, than does the cantilever, torsion bar, or spring. Commercial laboratory balances are available with capacities ranging from only a few milligrams up to several kilograms, and with sensitivities from one microgram to 0.1 gram per minimum detectable deflection. [The minimum detectable deflection is usually taken to be one-quarter of a scale division; it will be noted that the sensitivity defined in this way is not the same as the sensibility reciprocal which is commonly used in the teaching of quantitative analysis, namely the weight necessary to produce a change in rest point of one scale division.]

Lever Balances

The following discussion, although general with respect to principles, will be applied, for convenience, specifically to balances for macro-quantitative analysis; i. e., to models having a sensitivity of 0.1 mg, and a capacity of about 200 g.

Lever balances can be classified into two main types: (*a*) the *two-pan*, and (*b*) the *single-pan* instruments.

The two-pan balance is the classical macro-analytical balance; it is the ubiquitous equal-arm, three knife-edge device that is familiar to every student of quantitative analysis. Traditionally, for precisions of 0.1 mg, the unknown mass has been balanced to the nearest 0.5–1.0 mg with the weights and rider, and the remaining few tenths of a milligram obtained from the difference between the rest point and the zero point. Most of the recent developments in two-pan balance instrumentation have been directed toward improving the speed and convenience with which a complete weighing can be performed. Models of this type of balance are currently available at prices ranging from about \$130 to over \$700. This rather large range of prices reflects differences in quality of workmanship, in the capacity of the balance, and in the number and kind of conveniences or accessories built into the instrument.

The quality of the workmanship in a new balance cannot readily be judged by a nonspecialist; this is a factor that becomes apparent only after years of use, and limits the effective life of the instrument. Thus, a \$300 balance that is so well made as to deliver 0.1 mg accuracy for many years is a better buy than a \$150 instrument that deteriorates appreciably after a few years. This is not to say that all higher-priced balances are necessarily better bargains than the lower-priced ones of similar tolerance. The only reasonable criterion here is the experience of others over the years with balances of the make being considered for purchase, as well as the amount of use (and abuse) it is anticipated the balance will see.

It may be noted, in this connection, that the useful life of a well-made balance appears to be improved by the use of sapphire, which is considerably harder than agate, for the bearing surfaces upon which the knife edges rest, and by a beam arrestment mechanism that contacts the beam by moving along a curved path concentric with the arc through which the terminal knife edges travel as the beam deflects, so that the knives are not dragged across the bearings when a swinging beam is arrested. The difference between the straight-fallaway and the arc-arrestment types is shown schematically in Figure 3. Such features as these, however, add con-

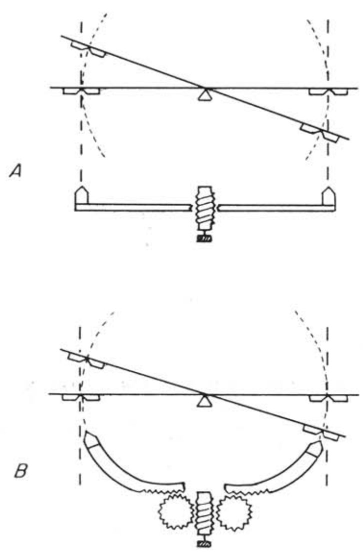

Figure 3. *A.* Straight fall-away arrestment. If the beam is arrested when not horizontal, the supports do not seat properly, and tend to drag beam across its fulcrum. *B.* Arc arrestment. Supports seat properly for any orientation of the beam.

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siderably to the total cost of the instrument.

Some balances, e.g., those now made by Oertling, Ltd., and Stanton Instruments, Ltd., incorporate special devices for automatically fixing the rate at which the beam is dropped onto the knife edges, thus eliminating wear of the edges resulting from careless operator manipulation.

Detailed discussions of the physics of lever balances, including the significance of the precise positioning of the terminal knife edges, coplanarity and exact parallelism of all three knife edges, sharpness and hardness of the knife edges, proper design of the beam and pan arrests, and the maintenance and testing of balances can be found in the publications listed in the bibliography at the end of this article.

The speed and convenience with which an approximate balance point is achieved with the classical type of two-pan balance has been increased by the use of several mechanical and optical devices, including multiple built-in riders operated from an external keyboard or dial control, a pendant gold chain in place of the milligram weights and rider, air or magnetic dampers attached to the beam or pans, and optical projection systems for reading the pointer deflection.

The built-in weights and riders with external control represent simply a manipulative convenience, generally at no loss in accuracy or sensitivity. The most

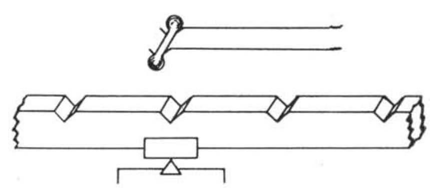

Figure 4. The notched beam and rider.

common form is the notched beam with its 0.5 g or 1.0 g rider, which can be placed at positions on the beam corresponding to 0.1 g increments up to a total of 1.0 g (Figure 4). In these balances there is generally a chain weight device or projection system for the last 100.0 mg of the weight, so that no fractional weights ever need to be placed on the pan. Some balances have weights mounted above the stirrup of the knife edge or pan, so that they may be dropped onto the end of the beam by manipulating an external control,

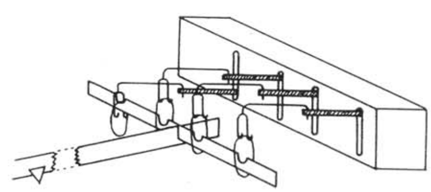

Figure 5. Built-in weights and control for remote loading of beam.

thereby also obviating the transfer of individual weights from a box of weights to the balance pan (Figure 5). The choice of balances so equipped should be based on whether the greater number of weighings that can be performed per working day is worth the materially increased cost of the instrumentation.

The suspended chain also represents a manipulative convenience, but this is obtained at the expense of some accuracy. It is difficult to produce a chain with the great uniformity and mechanical stability that is necessary for analytical balances, and any chain inevitably picks up enough dust and grease with time to change its effective torque significantly, and to affect the linearity of its calibration. Therefore, chain-equipped balances tend to be less accurate than comparable instruments employing riders, especially after they have been in use for some time, but they are not much more expensive, and are much faster to operate.

Damped balances can be as accurate as, and considerably faster than undamped instruments, and they are not much more expensive. Magnetic damping involves the presence of a magnet inside the balance case, and this may lead to significant errors if traces of magnetic materials are present in any of the parts of the balance or in the sample being weighed. Air-damped instruments do not suffer from this disadvantage, but some care must be taken to keep the cylinders from picking up enough dust to cause frictional effects that may disturb the balance point. The amount of magnetic or air resistance necessary to give critical damping, such that the beam neither overshoots the rest point, nor approaches it too slowly, depends upon the total mass of the balance. Therefore, the position of the damper should be somewhat different for a light load than for a heavy one. A desirable feature (found, however, in only some of the damped balances) is a scale for the position of the damper, so that its location can be varied in a reproducible way to fit the load being weighed (Figure 6). Finally, it should be

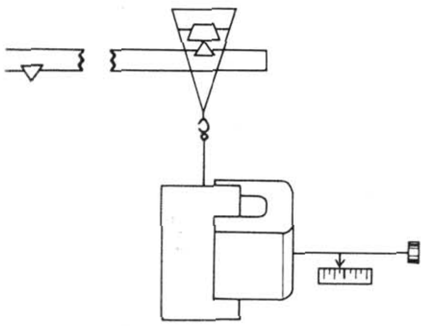

Figure 6. An adjustable damper, with a scale for reproducing settings to give optimum damping for different loads.

recognized that the use of a damper effectively cuts the sensitivity of a balance in half, since the minimum change in rest points that can be detected corresponds to a free-swinging deflection that would be twice as large.

Optical projection systems for facilitating the determination of the residual, direct-deflection part of the total mass appear to be gaining in popularity. The actual deflection of the beam from its

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zero point is kept small, to minimize the errors inherent in the nonlinearity of large displacements, and the deflection is magnified optically. If the optical system is well designed and free of distortions, the accuracy of this type of balance can be as good as the nonprojection types for ±0.1 to 0.05 mg work. The fact that a light source is necessary for optical projection systems can represent a limitation on accuracy beyond this level, as a consequence of the thermal and convection effects produced by the radiant energy. These effects are minimized in well-designed projection balances by mounting the lamp outside the balance case, interposing infrared absorbing filters between the lamp and the balance, and using low wattage lamps. Projection-type balances with damping are very fast; a weighing to 0.1 mg can be completed in a minute or less.

It should be clear from the foregoing that each type of balance has special advantages and disadvantages with respect to cost accuracy, speed, and effective lifetime. It is necessary fully to appreciate the factors involved in balance design, and to assess the type of use to which the balance will be subjected, before one can intelligently decide on the most suitable instrument for any particular laboratory or course of instruction.

The accompanying Table presents a survey of the design characteristics and

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Table 1. Characteristics of Macro-Analytical Equal-Arm Laboratory Balances

Make & model	List price	Chain	Damped	Read-out	Arrestment
Ainsworth LC	$215	..	..	...	Arc
Ainsworth LCB	325	Yes	..	Notched beam	Arc
Ainsworth LCB–M3	367	Yes	Mag.	Notched beam	Arc
Amer. Bal. Co. JR–10	215	..	..	...	Arc
Amer. Bal. Co. JR–201	275	Yes	..	Notched Beam	Arc
Amer. Bal. Co. JR–2012	314	Yes	Mag.	Notched beam	Arc
Christian Becker AB–1	695	..	Mag.	Notched beam & projection	Arc
Christian Becker AB–2	449	Yes	Mag.	Notched beam	Arc
Christian Becker AB-4	386	Yes	Mag.	Notched beam	Arc
Christian Becker AB–7	194	..	..	...	Straight
Fisher, Stud. Anal.	125	..	..	...	Straight
Fisher, Stud. Anal.	278	Yes	Mag.	Notched beam	Straight
Oertling FO5[a]	675	..	Air	Built-in wgts to 10 g & projection	Straight, with gravity plunger control of rate
Oertling HO1[a] (Single Pan)[b]	940	..	Air	Built-in wgts to 200 g & projection	Straight, with gravity plunger control
Oertling HO3[a] (Single Pan)[b]	1050	..	Air	Built-in wgts to 100 g & projection (0.05 mg sens)	Straight, with gravity plunger control
Sartorius Selecta (Single Pan)[b,c]	890	..	Air	Built-in wgts to 200 g & projection	Straight
Sartorius Projecta[c]	645	..	Air	Built-in wgts to 100 g & projection	Straight
Sauter 10[d]	395	..	Air	Built-in wgts to one g & projection	Straight
Sauter 11[d]	445	..	Air	Built-in wgts to 1 g. & projection	Straight
Seederer-Kohlbusch 20	169	Yes	..	...	Straight
Seederer-Kohlbusch 6SA	260	Yes	Mag.	Notched Beam	Straight
Stanton A42[e]	630	..	Air	Built-in wgts to 1 g & projection	Straight
Stanton B16[e]	790	..	Air	Built-in wgts to 200 g & projection	Straight
Voland 100-N	125	..	..	...	Straight
Voland 200	140	..	..	...	Straight
Voland 120-N	223	Yes	..	Notched beam	Straight
Voland 220-D	345	Yes	Mag.	Notched beam	Straight
Voland 320-D	278	Yes	Mag.	Notched beam & digital reader	Straight
Voland 340-D	385	Yes	Mag.	Notched beam & digital reader	Straight
Voland 640-D	445	Yes	Mag.	Notched beam & digital reader	Arc
Voland 750-D	650	Yes	Mag.	Built-in wgts to 100 g digital reader	Arc

[a] Imported to U.S.; available through J. & G. Instrument Corp., P.O. Box 6, Milltown, New Jersey.

[b] These balances are of the equal-arm lever type, but only one pan is provided for the use of the operator. In lieu of the other pan, all necessary weights are contained within the balance case and are dropped onto extensions of the beam by means of external controls.

[c] Imported; available through C. A. Brinkmann & Co., Great Neck, L. I., N. Y.

[d] Imported; available through August Sauter of New York, Inc., Albertson, L. I., N. Y., and several U. S. laboratory supply houses.

[e] Imported; available through Burrell Corporation, 2223 Fifth Avenue, Pittsburgh, Pa.

comparative list prices[1] of the currently most widely distributed commercial macro-analytical balances of the two-pan, three knife-edge, equal-arm lever type available in the United States.

The conventional undamped two-pan balance is probably still the best where highest precision over a long effective life-time are the most important considerations. The speed and convenience of weighing are improved by the notched beam and built-in weights, and if these are calibrated periodically, there is no loss in accuracy. However, the use of damping, chain-weights, and projection reading of direct deflections may entail a loss of precision and accuracy in the measurements, particularly after the balance has been in use for some time, and are to be recommended only where the working time gained is important enough to compensate for this, and for the increased cost.

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The next article in this series will treat single-pan, constant-load balances, as well as other recent approaches to weighing, including automatic and electronic balances.

[1] The prices quoted here and in subsequent articles of this series are to be taken *cum grano salis*, and are given solely to provide an order of magnitude and frame of reference. The price of a given instrument may vary considerably from region to region, dealer to dealer, and time to time.