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ACCURATE WEIGHING WITH THE MICROCHEMICAL BALANCE

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FOREWORD

The original intent of the author was to prepare a manual for instructing personnel in the detailed procedure for accurately weighing with the microchemical balance. Eventually it seemed worthwhile to fill the need for a more comprehensive presentation which would serve those planning the acquisition and use of such an instrument, as well as those desiring background information. Accordingly, the procedural description of a technique for sixth place weighing was supplemented with sections on balance installation and maintenance, a discussion of the factors influencing apparent weights, and a program for balance testing.

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ACCURATE WEIGHING WITH THE MICROCHEMICAL BALANCE

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THE MICROCHEMICAL BALANCE

There are two types of beam microbalances¹ as the term is conventionally understood: one, usually of quartz construction, provides sensitivities as low as 10^{-9} g, but with capacities limited to milligrams; the other, known as the "microchemical" balance, is designed for multigram capacities compatible with a precision and accuracy of 10^{-6} g. The latter is similar in appearance to the familiar analytical balance.

The microchemical balance built by W. H. F. Kuhlmann, Hamburg, Germany, has been widely used in quantitative microanalysis, partly because of the warm recommendation given it by F. Pregl, one of the pioneers in this field. It was constructed with a brass beam 7 cm long and a scale-reading telescope to measure pointer deflection. Thus the precedent was set about 1910 for shortening the microchemical balance beam to reduce its mass and period, and, in consequence, for making the balance inconveniently small. Since then several other companies have undertaken to compete with Kuhlmann, including Wm. Ainsworth & Sons, Inc. (Denver), Christian Becker (Clifton, N.J.), Paul Bunge (Hamburg), C. Longue (Paris), J. Nemetz (Vienna), L. Oertling, Ltd. (London), Pfaltz and Bauer, Inc. (New York), Sartorius-Werke, A.G. (Göttingen), Seederer-Kohlbusch, Inc. (Englewood, N.J.), Starke-Kammerer-A.G. (Vienna), and, recently, with a single-pan balance, Mettler Instrument Corp. (Hightstown, N.J.). Of these balances several have notable improvements and convenient features. Since it is impractical to present the case for each instrument, a representative long-period type of balance will be described.

The Ainsworth microchemical balance has several features which make it more usable than the short-beam balance. The beam is 12.7 cm long and is made of hard-rolled aluminum alloy instead of brass. Its movement is detected by means of an optical lever which constitutes an almost weightless pointer of such length that a sweep across ten spaces on the index scale corresponds to an angular swing of only 0.2° .² This device permits weighing by the single-swing method, so that the increase in period

¹Detailed reviews of the types of microbalances are given by G. Gorbach, "Die Mikrowage," *Mikrochemie* 20, 254-337 (1936) and more recently by E. R. Hoffman, "Outline of Microbalances," *Interchemical Review* 9, 75-87 (1950-51).

²The latest model has a projection device by which a finely ruled graticule on the end of the "pointer" is magnified to give equivalent readability with half the angle of deflection. The knife edges and planes have been changed to sapphire, which is non-hygroscopic and much harder than agate.

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resulting from the increase in beam length is not a disadvantage. All of the weights employed, 1 to 100 mg, totaling 221 mg, are transferred by means of a mechanical weight carrier. Heavier objects, up to the balance capacity of 20 g, are tared, best with a tare of almost identical size, shape and material. Because the weights are not handled they remain remarkably constant.

The Ainsworth beam has 201 rider V-notches, located with an accuracy of 0.005 mm according to the manufacturer; each has the equivalence of 0.005 mg when a 0.5-mg rider is used. A 5-mg rider is massive enough to be handled as a rod shape which would obviate the error from off-vertical placement of the 0.5-mg loop-type rider. Nevertheless, the use of the 0.5-mg rider is recommended here because it permits weighing by the single-swing method and reduces by as much as one-half the number of rider settings to be made. To the left of the zero notch is a short blade extension of the beam which supports a 0.5-mg V-shaped rider for adjusting the zero point of the balance. In lieu of this rider, which may move if the balance case is slightly jarred, the round zero-adjusting nuts on each side of the center knife edge can be replaced by nuts having a star shape so that they can be adjusted from outside the balance case.

Microchemical balances are by nature delicate instruments and can be ruined quickly by bad habits acquired through the misuse of analytical balances. With this in mind the operator should always move deliberately when using the balance in order to prevent mistakes which shorten its sixth-place life. The following sections will give an appreciation for the factors which affect the functioning of the balance and, where specific features are involved, will refer to the Ainsworth instrument.

Installation of the Balance

Several balance installations which have proved to be satisfactory have been described in the literature.^{3,4} Ideally, the room should have no outside walls and should be continuously air-conditioned, with a system having diffusing-type outlets to the room so that air currents are not apparent.

If a clean, draft-free ambient atmosphere having the same temperature as that of the balance can be assumed to exist, then the properties of the atmosphere which, in changing, impose limitations on the precision and accuracy of weighing are temperature and relative humidity.⁵

³A. Steyermark, Ind. Eng. Chem., Anal. Ed., 17, 523 (1945).

⁴R. N. Boos, "National Research Council Report on Design, Construction and Equipment of Laboratories," editor H. S. Coleman, Reinhold Pub. Corp., 1951, p. 188.

⁵Pressure changes are sufficiently gradual, except when a front is altering the weather, to make barostating unnecessary in most places.

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The following table shows the control of these factors which is necessary for a given average error of weighing if the volume occupied by the object differs by no more than 0.5 cc from that occupied by its counterweight.

ATMOSPHERIC VARIABLES AFFECTING THE PRECISION OF WEIGHING⁵

A. Relative humidity (temperature constant)

<u>Relative humidity</u>	<u>Composition of knife edges and planes</u>	<u>Variation in relative humidity</u>	<u>Av deviation of weighing</u>
45%	agate	$\pm 2\%$	± 0.002 mg
45%	sapphire	$\pm 2\%$	± 0.0005 mg

B. Temperature (relative humidity constant)

<u>Temperature</u>	<u>Variation in temperature</u>	<u>Av deviation of weighing^a</u>
25 C	± 0.5 C	± 0.002 mg
30 C	± 0.1 C	± 0.0005 mg

^a without a long period of equilibration between the operator and the balance.

For a balance equipped with agate bearings the major effect of variations in relative humidity is the change in zero point caused by the variable hygroscopicity of the agate; with non-hygroscopic bearings, such as those of sapphire, only the lesser effects on air density and weight of adsorbed moisture need to be considered (refer to the table).

The control of temperature remains most demanding because, even if the object and its counterweight occupy equal volumes, the beam arms expand or contract differently depending on strain and other differences remaining after fabrication. The magnitude of the resultant zero point shift at a given temperature varies from balance to balance, but it is of the order of several micrograms per degree centigrade, as shown by studies of weighing precision under closely controlled conditions.

Illumination in the balance room is chosen so that there will be a minimum amount of heat. General lighting should be diffuse and subdued, so that it may be left on while balances with optical levers are used. Direct lighting of each balance for adjustments and preliminary operations can be provided with individual fluorescent fixtures controlled by switches mounted below the table top.³

The support for the balance must keep it from receiving any shocks and vibrations that would cause damage to the knife edges when in contact with their planes. Beyond a heavy rigid support the complexity of the arrangement is dictated by the frequency and amplitude of the vibrations encountered. The problem has been investigated thoroughly by Steyermark, et al.^{3,6} Our arrangement is an adaptation with materials of various densities to damp out vibrations of various frequencies. Each support is rigidly constructed with legs of high-density concrete and a top made of layers of various thicknesses: red fir (2 in. thick between the legs and the top), then brass ($3/4$ in.), aluminum ($3/4$ in.), lead ($1\frac{1}{2}$ in.), cork (1 in.), soapstone (1 in.), and a surface-grounding plate on which the balance rests, all bonded together with two-sided Scotch tape. Individual U-type tables, with electrically conducting tops $4\frac{1}{2}$ ft long and 1 ft wide at the base of the U, separate the operator from each balance support, while providing a working and grounding surface and storage drawers for accessories.

Assembly of a balance on its vibration-eliminating support is done in strict accordance with instructions from the balance maker. The greatest care and, if possible, previous experience are demanded. For handling and cleaning the balance parts selected pieces of chamois and of silk, finger cots and camel hair brushes must be prepared.⁷ Features of procedure are emphasized in the following section.

Maintenance of the Balance

The essential parts of a balance should never be touched with uncovered fingers. Chamois cots, which have been scrupulously washed, rinsed, and dried in air, should be worn.

The balance should be dismantled for cleaning only when its behavior begins to be unsatisfactory or when dust particles are seen on the beam and stirrups. There is greater likelihood for damage to the balance during dismantling and re-assembly than during the other operations with the balance.

The pans should be dusted as frequently as necessary; while being brushed, they can be kept from swinging by holding ivory-tipped forceps against the front edge. Occasionally at the end of a day, each pan should be removed by grasping it with an ivory-tipped forceps, and the base plate cleaned. Since this plate usually is Carrara glass, the brushing and wiping should be done several hours before weighing is resumed. Of course, never try to remove dust and lint by blowing.

⁶A. Steyermark, Anal. Chem. 28, 517 (1956).

⁷Prepare silk and chamois by washing several times in soapy water, rinsing in several portions of ammoniacal water, then in distilled water, and finally drying in air. Clean camel hair brushes similarly but, before drying, dip in acetone to remove traces of oil.

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When the balance must be dismantled, follow the balance-maker's instructions, noting the code symbols of the left and right sides. In particular, check to be sure that the beam arrest is all the way up before the pans, the stirrups, and the beam are removed in that order. As each part is removed, place it on clean glazed paper which is cushioned by something like sheet sponge rubber. Avoid any contact with the beam pointer tip.

Clean all beam and stirrup arrestment contacts by brushing or, if necessary, by wiping with chamois and silk and then brushing. Clean the beam by brushing the main structure and all of the rider notches; glide gently over the knife edges with chamois stretched between the ends of an ivory-tipped forceps, finally brushing the edges, yet all the while avoiding the pointer tip and adjustment nuts. Clean the stirrups by gliding over the bearing planes with chamois and then brushing the planes and the metal parts. Wipe the pans and pan hangers thoroughly with chamois and silk, especially polishing the pan arrest contacts. Finally examine all bearing surfaces, the rider notches and the pointer tip under magnification to insure that no dust particles, brush hairs or lint remain.

Before assembling the balance, level the case. Replace the parts in reverse of the order they were removed, making sure that the beam arrest is up before lowering the beam onto its supports. View the rider on its carrier and then place it in a beam notch; if it appears bent out of one plane or if it tilts at all from a vertical position, straighten it on a glass flat.

Let the re-assembled balance "settle down" for at least a day. Then determine the sensitivity reciprocal and the precision of weighing for comparison with previous values (refer to the sections under Method of Weighing).

METHOD OF WEIGHING⁸

For sequence weighing to the sixth decimal place (on the scale of grams), the procedure of weighing to the zero or center line of the index scale, with subsequent correction for any shift which occurs in the zero point of the balance, is recommended here as faster and less liable to error than

⁸ This outline assumes a background of information on balances, weights, and weighings such as is given by Kolthoff and Sandell, "Quantitative Inorganic Analysis," 3rd edition, Macmillan, 1952, chapter XIII.

The fundamentals of microbalance technique are developed in detail by H. Sternberg, "Über die Grundlagen der Mikrowagetechnik," *Mikrochemie* 22, 187 (1937).

The terminology used here accords with the recommendations of the ACS Committee on Balances and Weights, "Terminology for Describing the Performance of Analytical and Other Precise Balances," *Anal. Chem.* 26, 1190 (1954).

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weighing to the zero point of the balance. A further reduction in time is possible when the balance is operated so that the single-swing method of estimating equilibrium points is applicable. Since the retardation of a swing caused by air resistance and bearing friction is approximately proportional to amplitude (case of damped harmonic motion), it is only necessary to keep the swings short enough so that the retardation can be neglected.⁹

The method of determining equilibrium points from several swings requires more time at the balance and is warranted only when the deflection sum must be large or when the greatest reliability is necessary.

Finding the Zero Point

Half an hour before a series of weighings, the optical lever and room lights are turned on and the balance door is opened a few inches to equilibrate atmospheric conditions inside and outside the case. During a weighing sequence no changes in the air pattern should be permitted. After such changes the zero point, or equilibrium point of the unloaded balance, may return to its original value, but meantime it usually wanders noticeably.

Just before starting at the balance the operator should contact an electrical ground to eliminate any accumulated electrostatic charge. The balance is operated from the greatest convenient distance in order to reduce as much as possible the operator's effect on it. While the beam is supported by the beam arrest, the pan arrest is partially released several times until the pans hang without free motion. The beam arrest is lowered, slowly at first, while the knife edges and planes are being brought together. With the beam rider positioned vertically in its zero location, the carrier weights up and the pans empty, the pan arrest is released. If it appears that the sum without regard to sign of two successive half-swings or deflections will be small enough to neglect their retardation, the mean of the second and third deflections is taken as the zero point of the balance at that particular time.

Beam motion should be stopped only with the pan arrest and only as the moving line approaches or passes through the zero line of the index scale.

Determination of the Sensitivity Reciprocal (SR)

In order to calculate approximate rider locations, the third decimal place of milligrams, and corrections for changes in zero point, a factor which is the reciprocal of the balance sensitivity, is needed. To correct for zero point changes this factor must be known reliably within two percent of its actual value. Being the reciprocal of a balance characteristic it does

⁹ For sixth place weighing with the Ainsworth balance this is the case when the difference between two consecutive turning points of a swing is less than twenty spaces on the floor scale.

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not change appreciably unless the knife edges are deteriorating; accordingly, the sensitivity reciprocal should be redetermined at intervals to serve as an index to the condition of the balance.

After several zero point determinations have shown that ambient conditions are acceptably uniform, the beam rider is moved to the right and located vertically in the notch corresponding to 0.050 mg. The rest point with this 0.050-mg excess load is found by the method of several swings. From these data the weight equivalent to one space of the index scale is calculated and recorded as the sensitivity reciprocal (SR) for small loads. The procedure is repeated systematically for 0.050-mg excess load with balanced loads of 1, 2, 4, 8 and 16 grams on the pans.

An example from an Ainsworth balance will make several points clear:

Deflections with no load

	+	-
(1)		1.7
(2)	11.8	
(3)		1.1
(4)	11.1	
(5)		0.5
(6)	10.5	
(7)		0.0
Av.	<u>+11.1</u>	<u>-0.8</u>

Deflections with 0.050-mg load

	+	-
(1)		41.0
(2)	0.0	
(3)		38.6
(4)	-2.3	
(5)		36.3
(6)	-4.5	
(7)		34.1
Av.	<u>-2.3</u>	<u>-37.5</u>

Zero point, using all deflections: +5.15
 using deflections 1,2,3: +5.2
 using deflections 2,3,4: +5.2
 using deflections 2,3: +5.35

Rest point: $\frac{(-2.3) + (-37.5)}{2} = -19.9$

$$\text{SR for small loads} = \frac{0.050 \text{ mg}}{(5.15 + 19.9) \text{ spaces}} = 0.002 \text{ mg/space}$$

Note that in the zero-point determination the sum of second and third deflections is 12.9 and that the error arising from the use of these two deflections only (single-swing method) is 0.2 space, equivalent to 0.0004 mg. This is about the readability error in an equilibrium point determination with the Ainsworth instrument.

Weighing

For a single weighing made to the zero point of the balance, the zero point should be brought to near coincidence with the zero or center line of the index scale by means of the zero-adjusting rider.

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For weights obtained by difference over an interval of time the weighings can be made faster to the zero line of the index scale; subsequently corrections are made for any shifts which occur in the zero point.

Containers are provided with tares, preferably "identical" and previously adjusted on a rough balance to a weight difference less than 200 mg. Before loading on the microchemical balance, objects must be allowed to stand at the balance and come to its temperature.

With relatively large glass objects a test should be made for the presence of electrostatic charge with a pith ball suspended from a silk thread; if attraction occurs, showing that an electrostatic charge has accumulated despite the usual precautions, it can be eliminated by supporting the non-conductor symmetrically in a high-frequency electrical discharge.

Loading the balance requires deft use of long-handled forceps to minimize the effect of the hands when the balance is open. While the beam remains supported by the beam arrest, the pan arrest is operated slightly until the pans hang motionless when free of their arrest. Then the beam is released and the carrier weights are added in a systematic fashion, based on testing for balance with the pan arrest.

Note that the usual preliminary testing with the beam arrest is not necessary on the microchemical balance because the tare or counterbalance for the object is adjusted elsewhere to within the weight which the pan arrest will support. Another feature of technique which influences the sixth place life of the knife edges derives from sure-fingered control of the pan arrest and from familiarity with the rates at which deflections start under various degrees of imbalance. The pan arrest should be released just enough to show the deflection direction; in other words, the excursion of the moving line from the zero line of the floor scale should not be permitted to exceed a few spaces until a free swing would remain on scale. With an SR of 0.0025 mg/space this point is not reached on the Ainsworth instrument until the object is balanced within 0.125 mg by means of the beam rider.

Finding the rider position in sixth place weighing requires no more than three preliminary trials at most. If it is not within 0.125 of the 0.500 mark on the beam, it will be so when relocated at either the 0.250 or 0.750 marks. At this point the two turning points of a free swing are used with the SR to figure mentally where to relocate the rider. Then the rest point is determined by the single-swing method. If the weight equivalent of the rest point is more than 0.01 mg, a last rider adjustment is made; if less than 0.01 mg, this amount can be used reliably to adjust the indicated weight of the object without further trial.

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For example: On a balance with SR equal to 0.0025 mg/space a certain lightweight object is counterbalanced by its tare plus 43 mg plus the rider at the 0.500 mg mark on the beam; the turning points of the free swing are +2 and -50, corresponding to $\left(\frac{-48}{2}\right)(0.0025) = -0.06$ mg. Accordingly the rider is relocated at the 0.440 mg mark and the rest point found from the second and third deflections of a swing: $+4.7, -2.5; \frac{+2.2}{2} = +1.1$. Therefore the final weight of the object relative to the tare is 43.443 mg, uncorrected for errors in the weights and rider.

Correction for Zero Point Changes

When there is an interval of time between related precision weighings made to some arbitrarily chosen zero, it becomes necessary to check zero points and to correct for any change which occurs during the interval. The milligram equivalent of the zero-point shift is calculated by means of the sensitivity reciprocal appropriate for the load; it is added to a given weight when the shift from the reference zero point is in the minus (or "subtract-weight") direction, and it is subtracted when in the plus (or "add-weight") direction. This inverse rule can be derived in several simple ways, one of which relates the shift to the equivalent rider position.

Of course, the zero point, when determined, must be representative of the zero point at the time the weight was taken. Procedurally this means either establishing a steady state, with the operator as part of the system, or operating in a standardized fashion so that the two equilibrium points of a given weighing, rest point and then zero point, are taken at corresponding times in the zero-point cycle. The latter procedure is more convenient and is quite satisfactory for relatively rapid sequence weighing to the sixth place of grams.

Evaluation of the Precision of Weighing

The ultimate precision of a balance depends on the mechanical perfection of its knife edges and planes. Ideally, the edges would be straight, sharp, parallel, equidistant, and in the same horizontal plane. Practically, the deviations from perfection in these respects place an overall limit on weighing accuracy beyond which it is useless to set the sensitivity by raising the center of gravity. On the other hand, a skilled maker achieves such high standards that usually the limit is imposed by the technique of the user and the variability of the environment in which the balance is used. In any case the precision or reproducibility of weighing should be appraised realistically with the technique and under the conditions expected in regular use.

A series of at least ten weighings of the same object are made, each by the complete sequence of operations: loading, rest-point determination, unloading, and zero-point determination. Either the standard deviation or

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the probable error is computed on the basis of one less degree of freedom than the number of weighings, since one is used in finding the average. If the probable error of a single weighing is found to be $\pm 0.00x$ mg, this means that there is an even chance that one weighing in one, in three, in five, and in twenty repeated weighings will have an error exceeding $(1)(0.00x)$, $(1.5)(0.00x)$, $(2)(0.00x)$, and $(3)(0.00x)$ mg, respectively.

With usual precautions in locating and using a balance the probable error in a single weighing may be as low as ± 0.002 or 0.003 mg. To reduce this and approach the limit imposed by good balance construction, increasingly elaborate control of environmental and operator effects is required.

Calibration of the Weights

For all results derived from weight ratios, and not requiring knowledge of the absolute values of the weights, internal calibration by the T. W. Richards substitution method will suffice. It is recommended that the beam rider be assumed correct and that relative corrections for the carrier weights be determined on this basis. All values should be checked several times with the best control of influencing factors that can be maintained. These corrections are applied when consideration shows that they may affect a given result significantly.

FACTORS INFLUENCING THE APPARENT WEIGHT OF OBJECTS

Temperature

If an object is at a temperature higher than that of the balance, the resulting upward currents of air lift the balance pan and decrease the apparent weight. Additionally, if the object is a closed vessel, the air within may be less dense than that surrounding the vessel, so that its weight appears to be less than it actually is. There is also the possibility that a warm object may cause the ratio of beam arm lengths to change. It follows that every object must be brought to the temperature of the balance before it is weighed.

Humidity

The amount of water adsorbed on an object depends on the composition and extent of its surface, as well as the humidity and temperature of the atmosphere. Therefore, the effect on weighing vessels can be reduced by making them as small as possible and of non-hygroscopic material. A platinum container that has been ignited and then cooled in a good desiccator will not gain weight appreciably on exposure to the atmosphere. On the other hand, moisture-free glass will become noticeably heavier while being weighed in the usual atmosphere.

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Since equilibrium with respect to surface adsorption of moisture is reached much faster from the wet side than from the dry side, large glass apparatus such as absorption tubes and filter beakers are treated by a "wet-wiping" technique. This involves wiping the surface in a standard way with a lint-free cloth (not chamois) which has been dampened evenly with a solution of equal parts methanol and water. After standing 15 min at the balance the object is weighed. Obviously, even after a reliable technique has been developed, the apparent weight will vary with humidity. This variation can be eliminated by the use of an identical tare treated identically.

If a sample material is sufficiently stable to be weighed in air, it should not be placed in a desiccated atmosphere before weighing; instead, the results of analyses performed on it are corrected to dry basis from a moisture determination. For this reason a desiccant is seldom needed in the desiccator.

A sample which is so hygroscopic that it cannot be weighed reliably in air, even with the indication from preliminary approximate weighings, must be handled and weighed under moisture-free conditions. Apparatus and technique which have been applied successfully to this troublesome case are referred to in a review by L. T. Hallett, *Ind. Eng. Chem., Anal. Ed.*, 14, 956 (1942).

Buoyancy of the Atmosphere

The buoyant effect of dry air at 25 C and 760 mm Hg is 1.19 mg per cc displacement. A one degree centigrade variation in temperature changes the buoyancy 0.004₄ mg. A 3-mm variation in barometric pressure changes it 0.005 mg. A variation in the water vapor content of the atmosphere also changes the buoyancy. These considerable changes in apparent weight can be reduced by keeping the time between initial and final weighings as short as possible. They can be made negligible by using a tare of nearly identical displacement.

Electrification

A dry glass vessel when brushed or (dry) wiped can be given such a charge of static electricity that its apparent weight may be changed by milligrams. The effect is most troublesome with Pyrex-type glassware and with relative humidity below 40 to 50 per cent. Dissipation of the charge can be accomplished harmlessly by introducing the non-conducting vessel into a zone of high-frequency discharge.¹⁰ Flaming will eliminate the charge but will also remove the film of equilibrium moisture adsorbed on the surface. Exposure to ultraviolet light is fairly effective, but slowly so. A radioactive

¹⁰ F. W. Van Straten and W. F. Ehret, *Ind. Eng. Chem., Anal. Ed.*, 9, 443 (1957).

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source may be used to ionize the air so that a surface charge will leak off quickly, but the level of activity required is intolerable without shielding. Clearly the simplest approach is to arrange not to brush or (dry) wipe a weighing vessel.

Electrification of the operator and thus of the balance can be very troublesome. Therefore one should wear cotton clothing and should wet the hands at a raw water tap just before starting to use the balance. This process may have to be repeated after a few minutes to get rid of isolated charges held in the clothing.

It is worthwhile to ground a balance system which has conducting contact throughout when the beam is arrested, and it is especially helpful to replace the balance door with one made of electrically conducting glass. Still, the simplest solution to the problem of electrification is to prevent its occurrence by observing the precautions noted. Only by attention to details such as these can rapid reliable sixth-place weighing be achieved.

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TEST PROGRAM FOR EVALUATION OF
THE ESSENTIAL CHARACTERISTICS
OF A MICROCHEMICAL BALANCE*

Sensitivity

- a. For sixth-place weighing, adjust the position of the center of gravity nut to give a sensitivity reciprocal (SR) of about 2.5 micrograms per space of the magnified scale. Determine the period of the balance at this setting.

- b. Determine the constancy of the SR over a period of days.

[Several determinations should be made each time and the averages compared. The variation should not exceed 20 parts per thousand.]

- c. Determine the SR and the period at loads increasing to capacity.

[The sensitivity should not increase markedly with load; if it decreases, the change should not exceed 10 per cent from no load to capacity. From the data it is possible to compute how much the end edges are out of the plane of the center edge - acceptable if less than 10 seconds of arc.]

Stability

Calculate h, the distance of the center of gravity below the point of oscillation of the beam, for the unloaded balance.

[Practically, the limit of stable behavior is imposed by the precision of the balance. It is useless to decrease h, for greater sensitivity, if behavior of the balance becomes erratic.]

Precision

- a. Examine the knife edges and planes for visible imperfections by edge-lighting and under magnification.

*The tests and procedures which involve adjustments of the balance should not be attempted without the necessary experience and skill, for which enthusiasm is no substitute.

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- b. Determine the precision of the balance, as measured inversely by the probable error of repeated weighings of the same mass. Take a series of at least ten pairs of equilibrium points at each load, e.g. 0.05 mg, 1 g, 2 g, 4 g, 8 g, and 16 g, with the gram loads centered on the pans.

[To determine the precision of the balance, as distinct from the precision of weighing, environment and operator effects on the balance must be excluded or constant. Rider placement and index scale reading are done as precisely as possible.]

- c. Determine the precision of the balance under controlled conditions when a half-capacity load is placed at the front and at the back edges, respectively, of each pan in turn.

[If an appreciable change is found, the knife edges are not in parallel vertical planes.]

- d. Evaluate the constancy of balance precision with various swing amplitudes.

[Variation shows that the knife edges are worn, not clean, or otherwise faulty.]

- e. Find the magnitude of the rider placement error. Hang an extra 0.5-mg beam rider on the rider carrier; take alternate zero points and then rest points with the extra rider placed in the mid-notch, arresting the beam after each determination. Deduct the readability error.

[The change in sensitivity from adding the extra rider will not affect this test.]

- f. Determine the variation in the factor of merit (sensitivity divided by the period squared) as the center of gravity is raised stepwise 3 or 4 times until seventh-place sensitivity is indicated. Note that environmental and operator effects on the balance must be constant.

[Since the balance sensitivity varies inversely with h and the period varies inversely with the square root of h , this factor should remain constant as h is decreased. Actually a balance is worthy if the variation is less than ± 0.0005 when sensitivity in $\text{mm}/0.050 \text{ mg}$ is divided by the square of the period in seconds.]

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Accuracy

- a. Calculate the ratio of beam arm lengths at small load from weighings of a small mass made under controlled conditions by the transposition method, i.e., first on one pan and then on the other.

[Equality of the beam arm lengths to within at least 4 parts per million is shown by microchemical balances from competent makers. The V-notches for the rider should be located with an accuracy of 0.0005 in.]

- b. Calibrate the weights internally with the rider as standard.

This calibration by the T. W. Richards substitution method suffices for all results derived from weight ratios and not requiring knowledge of the absolute values of the weights.

- c. Determine the absolute value of the rider or of one of the weights by comparison with a standard weight. Compute the values of the other weights from the ratios established by internal calibration.

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APPENDIX

STEPWISE SUMMARY OF WEIGHING PROCEDURE

A microchemical balance must be adequately cared for and intelligently used if it is to give reliable indications of weight to the sixth place of grams. Reasons for the following essentials of procedure are noted when unusual or not readily obvious.

1. Make the load as light in weight as possible -
 - a. to keep the balance period at a minimum;
 - b. to permit the correction for zero-point shift to be calculated with the sensitivity reciprocal (SR) of the unloaded balance.
2. Make the load as small in volume as possible, or use an identical tare to reduce variations in buoyancy caused by variations in temperature, pressure and/or humidity.
3. Arrange to let the room and balance atmospheres equalize for half an hour after changes have been made, such as switching on optical lever and room lights. Note the range of temperature change during weighing by means of a 0.1 C thermometer hung near the front of the balance case.
4. Allow objects to stand and come to temperature at the balance before weighing. After heating, objects of platinum must be cooled at least 2 min, porcelain and glass at least 20 min.
5. Prevent accumulation of static electricity.
 - a. Wear cotton clothing.
 - b. Eliminate rubbing the hands and brushing or dry-wiping objects to be weighed.
 - c. Discharge large glass objects in a zone of high-frequency discharge if a pith ball test shows that a charge has accumulated despite the usual precautions.
 - d. Wet the hands at a raw water tap and grasp the tap firmly for a few seconds just before starting to use the balance.
6. Operate the balance at the maximum distance which is still convenient. Adjust the rider carrier without laying the hand against the side of the case.
7. Operate the balance carefully and skillfully.
 - a. Move deliberately to prevent mistakes which shorten the life of the balance.
 - b. Lower the beam arrest slowly through the first part of its arc.
 - c. Weigh with the pan arrest, permitting only minimal deflection of the moving line while testing for balance until a free swing will stay on-scale. Prevent off-scale deflection.
 - d. Stop beam motion with the pan arrest only, and only as the moving line approaches or passes through the center line of the index scale.
8. Establish a convenient but uniform procedure so that in a direct weighing the rest point and then the zero point are taken at corresponding times in the zero-point cycle.
9. Use the single-swing method of weighing; find the rider position by mental calculation with the sensitivity reciprocal rather than by a succession of trials. Reposition the rider until it remains vertical.
10. Develop a quantitative wet-wiping technique, with 50 per cent methanol, for large glass objects which must be weighed before and after heating.