CHAPTER 3

Weighing on the Microchemical Balance

Almost all microchemical procedures begin at the balance. Therefore, it is of utmost importance that the beginner in this field first master the technique of weighing. It must be stressed at this point that any bad weighing habits that might have been acquired through the misuse of analytical balances must be stopped immediately. Otherwise, the beginner will not be successful and, what is more important, the balance will be ruined within a short time. The delicate nature of this instrument was pointed out in Chapters 1 and 2 and this point must be constantly in the mind of the operator, regardless of his experience. In fact, the more experienced the chemist is in this field, the greater will be his respect for his balance, as he realizes his great dependency on it and the manner in which it is functioning. All movements by the operator should be slow and deliberate as this will prevent accidents which shorten the life of the balance.

It should be needless to state that carelessness has no place around this instrument. Dust particles, if allowed to accumulate on the balance pans, weighing vessels, weights, or tares, often will represent weighing errors or contamination representing large percentages, since the weights and amounts of material dealt with are relatively small.

Four methods are used for weighing objects on the ordinary analytical balance, namely, the so-called (a) ordinary, (b) deflection, (c) transposition, and (d) substitution. However, only one of these, the deflection method, is used when working with a microchemical balance. Consequently, this method alone will be described.

**Determination of Zero Reading*** and Deflection†

The determination of the zero reading of the balance should be the first operation performed by the beginner. If an unloaded balance (rider in the 0.0-mg. notch and both pans empty) is allowed to swing freely, it would eventually come to rest and the position on the scale, at which the pointer would remain, is called the rest point of the unloaded balance or the zero point. However, the balance is allowed to swing only until equilibrium is reached.

* Please see references 7, 8, 22, 44, 45, 59, 62.
† Please see references 7, 8, 11, 18, 22, 24, 25, 41, 42, 44–46, 48, 51–54, 62.
The deflection of the balance, under these conditions, is called the deflection of the unloaded balance or the zero reading. The zero reading is two times the zero point. Similarly, a loaded balance swinging in equilibrium would eventually come to rest and the position on the scale, at which the pointer would remain, is called the rest point of the loaded balance or merely the rest point. The deflection of the balance under these conditions is called the deflection of the loaded balance or merely the deflection—(deflection = 2 × rest point). The following method of obtaining the zero reading and the deflection deals with the free-swinging (undamped) types of balances, but also applies (with obvious variations) to all varieties. [On balances with damping devices (chapter 2), the pointer actually comes to rest. Obviously, on these balances the zero point and rest point are obtained. However, the scale has been altered so that the zero reading and the deflection are read off directly.]

**METHOD**

Before a balance is used each day, the doors of the case should be left partly open for 15–20 minutes. This permits air to circulate, equalizing the atmospheric conditions inside and outside the case. Likewise, during the day when the balance is not in use, the case doors should be left partly open so that the balance can then be used without delay. However, the balance is never allowed to swing when the doors are open.

With the rider in the zero notch (0.0 mg.) and the doors of the case closed, the beam- and pan-arresting mechanisms are released (in the order named, if separate). The amplitude of swing should be small, but great enough so that the balance is actually swinging; the pointer should swing an arc equivalent to about 3 to 8 scale divisions, that is, 30 to 80 deflection units.* If on releasing the arresting mechanisms the pointer does not swing a large enough arc, lock the beam-arresting mechanism just as the pointer is swinging towards the center and when it is as near as possible to it. This produces the minimum jar. Where the pan arrests act separately from that of the beam, finally lock the pans in place. Then set the balance swinging again as above—first releasing the beam arrest and then that of the pans.† The first few swings to each side should be ignored so as to be certain that equilibrium is closer to being obtained. The

* Readings on the models similar to the Ainsworth FH, Becker EM-1, and Bunge 25 MPN balances (Chapter 2) are always recorded as whole numbers instead of as fractions. Consequently, if the deflection is 3.5 divisions to the left (each division = 0.01 mg. = 10 deflection units) the reading is recorded as —35 (35 μg. or deflection units)—see diagrams under cases 1 to 6 below. However, on the Ainsworth FHM, direct readings are in μg.

† Caution: The balance should never be set to swinging by the practice of releasing the arresting mechanisms and gently fanning one pan with the hand.
3. Weighing on the Microchemical Balance

scale is then observed by whatever means is provided on the balance—lens, telescope, microscope or projection device. The readings are recorded (separating those to the left and to the right) until the decrement, or decrease, on each side is equal as shown in the following examples. All values to the left of the zero are negative (—) and all values to the right of the zero are positive (+). The zero reading is simply the algebraic sum of the average of the last three readings to the left and the average of the last two readings to the right.

<table>
<thead>
<tr>
<th>Rider at 0.0 mg.</th>
<th>swing to left deflection units or μg.</th>
<th>swing to right deflection units or μg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>-28</td>
<td>-25</td>
<td>+30</td>
</tr>
<tr>
<td>-22</td>
<td>-20</td>
<td>+28</td>
</tr>
<tr>
<td>-20</td>
<td>-18</td>
<td>+24</td>
</tr>
<tr>
<td>Average of last three</td>
<td>-20</td>
<td>Average of last two</td>
</tr>
<tr>
<td>'. Zero reading = +5 deflection units or μg.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Rider at 0.0 mg.</th>
<th>swing to left deflection units or μg.</th>
<th>swing to right deflection units or μg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>-35</td>
<td>-33</td>
<td>-15</td>
</tr>
<tr>
<td>Average of last three</td>
<td>-35</td>
<td>Average of last two</td>
</tr>
<tr>
<td>'. Zero reading = -14 deflection units or μg.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Rider at 0.0 mg.</th>
<th>swing to left deflection units or μg.</th>
<th>swing to right deflection units or μg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>-40</td>
<td>-37</td>
<td>-11</td>
</tr>
<tr>
<td>-35</td>
<td>-33</td>
<td>-13</td>
</tr>
<tr>
<td>Average of last three</td>
<td>-35</td>
<td>Average of last two</td>
</tr>
<tr>
<td>'. Zero reading = -49 deflection units or μg.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Rider at 0.0 mg.</th>
<th>swing to left deflection units or μg.</th>
<th>swing to right deflection units or μg.</th>
</tr>
</thead>
<tbody>
<tr>
<td>+4</td>
<td>+7</td>
<td>+40</td>
</tr>
<tr>
<td>+8</td>
<td>+9</td>
<td>+37</td>
</tr>
<tr>
<td>Average of last three</td>
<td>+8</td>
<td>Average of last two</td>
</tr>
<tr>
<td>'. Zero reading = +46 deflection units or μg.</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
The zero reading should be taken at least six times in immediate succession to determine the reproducibility of the balance (see Determination of Sensitivity and Precision below). During the course of each day, it changes about 5–10 deflection units due to temperature changes and various other causes. Consequently, correction has to be made for these changes if an object is weighed several times during the course of a determination and there is a time lapse of hours or days between weighings. If, however, all of the weighings necessary can be accomplished within a matter of minutes, such as when weighing a sample, the zero reading is disregarded and, in fact, is assumed to be zero.

The deflection of the loaded balance (objects and weights on the pans and rider usually in some notch other than 0.0 mg.) is done in the same manner as that used for the zero reading. For the weighings where the zero reading may be assumed to be zero, a deflection to the right (+) of the zero signifies that not enough weight is on the rider and, therefore, the deflection is added to the weight of the rider; likewise, if the deflection is on the left (—) of the zero, too much weight is on the rider and the amount must be subtracted. In other words, the deflection is added algebraically to the rider. For example, if the rider is in the 6.2 mg. notch and the deflection is —21, the rider reading is 6.179 mg. Similarly, if the rider is in the 1.3-mg. notch and the deflection is +2, the rider reading is 1.302 mg.

The beginner usually has little trouble with the deflections until he is confronted with the problem of correcting for the zero readings when an object must be reweighed several times at later intervals. The author believes that no confusion will result if the beginner records in his notebook the weights corrected for the zero reading each time a weighing is made rather than to deal with the change in zero reading. To make this correction the zero reading is subtracted algebraically from the deflection, observing all signs and the result is added algebraically to the rider quantity. If the following diagrams of the scale and examples are kept in mind, no confusion will exist regarding how the zero reading is to be used in the correction. All possible combinations are represented. Let us suppose that the zero reading (unloaded balance, rider at 0.0 mg.) is +20 and the deflection for a weighing (loaded balance) is +42:

Case 1

\[
\text{Correction} = +42 - (+20) = +22.\]
3. Weighing on the Microchemical Balance

This means that not enough weight is on the rider and 0.022 mg. must be added to the rider weight. If the deflection is +20 and the zero reading is +42,

Case 2

\[
\begin{array}{c}
\text{Deflection} \\
+20
\end{array}
\]
\[
\begin{array}{c}
\text{Zero reading} \\
+42
\end{array}
\]

(Correction = +20 — (+42) = —22),

the opposite would apply, 0.022 mg. would be subtracted from the rider weight. Using similar reasoning, the following diagrams are self-explanatory.

Case 3

\[
\begin{array}{c}
\text{Deflection} \\
-47
\end{array}
\]
\[
\begin{array}{c}
\text{Zero reading} \\
+40
\end{array}
\]

(Correction = —47 — (+40) = —87) \therefore 0.087 mg. to be subtracted from rider weight.

Case 4

\[
\begin{array}{c}
\text{Zero reading} \\
-47
\end{array}
\]
\[
\begin{array}{c}
\text{Deflection} \\
+40
\end{array}
\]

(Correction = +40 — (—47) = +87) \therefore 0.087 mg. to be added to rider weight.

Case 5

\[
\begin{array}{c}
\text{Deflection} \\
-46
\end{array}
\]
\[
\begin{array}{c}
\text{Zero reading} \\
-20
\end{array}
\]

(Correction = —46 — (—20) = —26) \therefore 0.026 mg. to be subtracted from rider weight.
Determination of Sensitivity and Precision

Case 6

Zero Deflection
reading —20

(Correction = —20 — (—46) = +26) : 0.026 mg. to be added to rider weight.

In the examples shown, it will be noticed that no deflection of 50, or more, is recorded. If the deflection is 50, or more, the rider should be moved over one notch so that one of less than 50, of opposite sign, will be obtained.

Determination of Sensitivity and Precision*

The sensitivity and precision of the balance should be determined frequently, since it is an indication of the manner in which the instrument is performing. The zero reading is first determined as described above. The rider is then moved to the 0.1-mg. notch and the deflection obtained. The displacement caused by the load of 0.1 mg. is called the sensitivity. The value is obtained by subtracting, algebraically, the deflection (rider at 0.1 mg.) from the zero reading. Table 9 shows three different examples which include the various possibilities with a balance having a sensitivity of 98.

**TABLE 9**

<table>
<thead>
<tr>
<th>Sensitivity (No. of deflection units equal to 0.1 mg.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zero reading (rider at 0.0 mg.)</td>
</tr>
<tr>
<td>---------------------------------</td>
</tr>
<tr>
<td>Example 1 +10</td>
</tr>
<tr>
<td>Example 2 0</td>
</tr>
<tr>
<td>Example 3 —5</td>
</tr>
</tbody>
</table>

In each of these examples, the addition of 0.1 mg. caused a displacement of 98 units or 1 unit = 0.00102 mg. The sensitivities of good balances are rarely 100 (1 deflection unit = 0.001 mg. or 100 units = 0.1 mg.), although the manufacturers' specifications often claim this. By raising or lowering the center of gravity of the beam, by means of the weight on the pointer (see Chapter 2), it is possible to obtain this figure with a good instrument. However, this is not necessary as a balance with a sensitivity between 95

* Please see references 7, 8, 22, 44, 45, 50, 62.
and 105 gives excellent results without applying a correction\textsuperscript{44,45} (too small to be significant). When it is not within these limits, correction should be made as shown by the following examples. If the sensitivity is only 75, 0.1 mg. is equal to 75 units or each unit equals \(\frac{0.1}{75}\) or 0.00133 mg. instead of 0.001 mg. Similarly, if the sensitivity were 125, each deflection unit would represent \(\frac{1.0}{125}\) or 0.0008 mg. Obviously, the deflection units must be multiplied by these factors to yield the correct values.

The procedure of taking the sensitivity should be repeated at least six times in rapid succession to determine the precision or reproducibility.\textsuperscript{44,45,50,62} This will include a zero reading, followed by the deflection when loaded with 0.1 mg., followed by a zero reading, and so on. The values obtained for the six determinations will usually vary and the standard deviations are calculated. For example, let us suppose the following set of readings in Table 10.

**TABLE 10**

**DETERMINATION OF PRECISION OF BALANCE**

<table>
<thead>
<tr>
<th>Zero reading</th>
<th>Deflection</th>
<th>Sensitivity</th>
<th>Deviation of zero readings from mean</th>
<th>Deviation of deflections from mean</th>
</tr>
</thead>
<tbody>
<tr>
<td>+20</td>
<td>-78</td>
<td>98</td>
<td>3</td>
<td>3</td>
</tr>
<tr>
<td>+15</td>
<td>-81</td>
<td>96</td>
<td>2</td>
<td>0</td>
</tr>
<tr>
<td>+17</td>
<td>-76</td>
<td>99</td>
<td>1</td>
<td>1</td>
</tr>
<tr>
<td>+21</td>
<td>-76</td>
<td>97</td>
<td>4</td>
<td>5</td>
</tr>
<tr>
<td>+15</td>
<td>-82</td>
<td>97</td>
<td>2</td>
<td>1</td>
</tr>
<tr>
<td>+16</td>
<td>-85</td>
<td>101</td>
<td>1</td>
<td>4</td>
</tr>
</tbody>
</table>

Mean +17
Mean -81
Mean 98

\[ s = \text{standard deviation}^\text{60,72} \]

\[ s = \sqrt{\frac{\sum (x - \bar{x})^2}{n - 1}} \]

where \(n\) is the number of readings, \(x\) is equal to the individual readings, and \(\bar{x}\) is the mean of the individual readings.

It can be seen from Table 10 that this balance cannot be expected to reproduce either the zero readings or the deflections closer than approximately 3 deflection units. Since the balance has a sensitivity of 98, each unit is equal to 0.00102 mg. (see above). Therefore, the precision is approximately \(3 \times 0.00102\) mg. = 0.00306 mg. or simply, 0.003 mg. (3 μg.). This figure coincides with that established\textsuperscript{20,28,50} years ago, as being acceptable.* (If the

* In spite of their being used constantly for periods ranging from six to twenty-two years, all of the balances in the author's laboratories have precisions under 2.7 μg. (from 1.1 to 2.6 μg.).
reproducibility of the balance is poor, it might be due to poor rider placement which can be determined by taking a series of zero readings without moving the rider between determinations. Also for the zero reading a number of successive determinations with the rider on the left-hand pan instead of on the beam will prove to be informative. If the reproducibility is poor when the rider is in the zero notch on the beam, but is good when the rider is on the left-hand pan, the zero notch on the beam allows the rider to move.

After obtaining the sensitivity and precision with a load of 0.1 mg., the procedure should be repeated with a 10-gram weight in the center of each pan.* The zero reading cannot be taken when weights are on the pans, so, instead two deflections are determined. Since the two 10-gram weights will vary somewhat, the rider is placed in the notch giving the closest conditions to equilibrium and the deflection determined. The rider is then moved over 0.1 mg. on the beam so that a deflection of opposite sign will be obtained and the procedure repeated. This will give the number of divisions equivalent to 0.1 mg. with a 10-gram load in each pan. To determine the reproducibility of the balance under these conditions, the above should be repeated at least six times moving the rider back and forth as was done above. A balance will rarely have the same sensitivity and precision or reproducibility under a load of 10 grams as it does under one of 0.1 mg. (except the constant load type) but the decrease, with a good instrument, will not be over 5%.

Calibration of Weights

Only a few weights out of the ordinary set receive constant use in this field of work as will be explained later in this chapter. However, those few which are used must be accurately calibrated. For this calibration, the rider of the balance is used as the standard. (If necessary, this could be checked against a Bureau of Standards 10-mg. weight.) During the process of making a weighing, the weights, W, are always placed on the right-hand pan. Consequently, they should be similarly placed during calibration and, therefore, the substitution method is recommended. A second set of weights, T, is used and these are always placed on the left-hand pan. It is only necessary to calibrate two 10-mg., one 20-mg., and one 50-mg. weights.

The tare 10-mg. weight, T_{10}, is placed on the left-hand pan and the rider is placed in the 10-mg. notch. The deflection* is determined, checked several

* All weights, tares, and objects to be weighed are always centered.
† Errors due to inequalities in the length of the beam arms may be determined in a manner similar to that used for analytical balances.
‡ The zero reading of the balance need not be determined since the weights are merely being compared to the rider.
times, and the average taken. (Note: For the calibrations, all values should be checked several times, using the average.) The rider is then placed in the zero notch (0.0 mg.) and the 10-mg. weight, to be calibrated, $W_{110}$, is placed on the right-hand pan and the deflection again determined. The difference between these is the calibration to be applied to the weight. Suppose, for example, the deflection with the rider in the 10-mg. notch is $+8$, and when the weight, $W_{110}$, is on the right-hand pan (rider in the zero notch) the deflection is $+10$. This would mean that the weight, $W_{110}$, is 0.002 mg. less than the rider weight. Consequently, the exact weight of $W_{110}$ is 9.998 mg.

The weight $W_{110}$ is then replaced by the weight $W_{210}$ (on the right-hand pan), the rider kept in the zero notch, the deflection obtained, and the correction applied. Again suppose for example that the deflection is $-4$. This would mean that the weight $W_{210}$ is 0.012 mg. heavier than the rider, or 10.012 mg.

Next, the 10-mg. tare weight, $T_{10}$ is replaced on the left-hand pan by the 20-mg. weight, $T_{20}$. On the right-hand pan is allowed to remain the 10-mg. weight, $W_{210}$, and the rider is placed in the 10-mg. notch. The deflection is again taken. For example, suppose it is $+5$. The weight $W_{210}$ is then replaced by the 20-mg. weight, $W_{20}$, and the rider placed in the zero notch. The deflection is again determined. Let us suppose it is then $+15$. This would mean that the weight, $W_{20}$, is 0.010 mg. lighter than the rider plus the weight, $W_{110}$, or,

$$W_{20} = \text{Weight of rider} + \text{weight of } W_{210} - 0.010 \text{ mg.}$$

$$= 10.000 + 10.012 - 0.010$$

$$= 20.002 \text{ mg.}$$

Using the rider and calibrated weights $W_{110}$, $W_{210}$, and $W_{20}$, the 50-mg. weight, $W_{50}$, is then calibrated. If desired, the 100-mg. weight, $W_{100}$, may be checked against the rider $+$ $W_{110} + W_{210} + W_{20} + W_{50}$.

### Apparatus

**COMBUSTION BOATS**

(See Table 11)\(^{62-64}\)

**Size A**

This boat (Fig. 23), referred to in the literature as the Hayman boat\(^{31,62,63,65}\) weighs approximately 0.45 gram. Because of its small size, it is used for samples weighing from 1–5 mg. For drying procedures, it should be used with the weighing bottle, pig type, metal, size A (Fig. 31).\(^a\)

\(^a\) For some procedures, such as those described in Chapters 7, 8, and 15, porcelain boats of comparable size are recommended (Fig. 22); usually the dimensions are 17 mm long, 6 mm wide, and 4 mm high.
**Fig. 22.** Porcelain combustion boat.

**Fig. 23.** Platinum combustion boat, size A—details of construction.

### TABLE 11
**Combustion Boats**

<table>
<thead>
<tr>
<th>Size</th>
<th>Figure</th>
<th>Use</th>
<th>Approximate weight (grams)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>23</td>
<td>For samples of 1 to 5 mg.(^a)</td>
<td>0.45</td>
</tr>
<tr>
<td>B</td>
<td>24</td>
<td>For samples of 5 to 25 mg.(^a)</td>
<td>0.7</td>
</tr>
<tr>
<td>C</td>
<td>25</td>
<td>For samples over 25 mg.(^a,b)</td>
<td>1.5</td>
</tr>
</tbody>
</table>

\(^a\) For drying procedures, the size A boat is used with the weighing bottle, Fig. 31. Sizes B and C can be used either with the metal weighing bottles (Figs. 32 and 33, respectively) or with the glass weighing bottles, pig-type (Figs. 29 and 30).

\(^b\) The size C boat is especially suitable for bulky materials and explosive substances, and for holding glass capillaries containing liquid samples.

**Size B**\(^6,62-64\)

This boat (Fig. 24) weighs approximately 0.7 gram. It is the most commonly used size for handling samples in the range of 5 to 25 mg.

**Fig. 24.** Platinum combustion boat, size B—details of construction.
3. Weighing on the Microchemical Balance

Size C

This boat (Fig. 25) weighs approximately 1.5 grams and is used for semimicro purposes with samples that weigh up to 50 mg. and for micro purposes with bulky material or explosive substances. Capillaries containing liquid samples are placed in this boat, and the combination is introduced into a combustion tube.

![Diagram of boat size C](image)

**FIG. 25.** Platinum combustion boat, size C—details of construction.

**WEIGHING CUP**

This item (Fig. 26) is designed as a sample container to fit into the weighing bottle, outside cap (Fig. 28). This combination is useful for weighing hygroscopic materials.

![Diagram of weighing cup](image)

**FIG. 26.** Weighing cup—details of construction.

**WEIGHING BOTTLE**

This type of weighing bottle was originally recommended by Roth. It may be inserted directly into such an item as a Carius combustion tube. It should be made of borosilicate glass, so that it will withstand elevated temperatures and may be used repeatedly (Fig. 27). It may be used for either non-hygroscopic or hygroscopic samples, liquid or solid (see Table 12).

**WEIGHING BOTTLE, OUTSIDE CAP**

The microweighing bottle originally was described by Hayman (Fig. 28). It should be made from soda-lime glass in order to reduce accumulation of electrostatic charges. The weighing cup (Fig. 26) should fit inside for use as a liner, if so desired. The bottle has been designed with an outside cap,
which permits the use of a lubricant with less danger of contaminating the sample than if an inside stopper were used. For the sake of simplicity, the capillary is straight (see Table 12).

![Diagram of weighing bottle](image)

**Fig. 27.** Weighing bottle—details of construction.

### TABLE 12

<table>
<thead>
<tr>
<th>Figure</th>
<th>Approximate capacity (ml.)</th>
<th>Approximate weight (gram)</th>
<th>Material</th>
</tr>
</thead>
<tbody>
<tr>
<td>27</td>
<td>0.05</td>
<td>0.6</td>
<td>Borosilicate glass</td>
</tr>
<tr>
<td>28</td>
<td>0.75</td>
<td>2.5</td>
<td>Soda-lime glass</td>
</tr>
<tr>
<td>29</td>
<td>3</td>
<td>5.0</td>
<td>Soda-lime glass</td>
</tr>
<tr>
<td>30</td>
<td>3</td>
<td>5.7</td>
<td>Soda-lime glass</td>
</tr>
<tr>
<td>31</td>
<td>0.5</td>
<td>1.5</td>
<td>Aluminum alloy</td>
</tr>
<tr>
<td>32</td>
<td>1</td>
<td>4.5</td>
<td>Aluminum alloy</td>
</tr>
<tr>
<td>33</td>
<td>2</td>
<td>6</td>
<td>Aluminum alloy</td>
</tr>
</tbody>
</table>

*a* This bottle is intended for introducing samples into containers of small diameters.

*b* The cap of the bottle shown in Fig. 28 may be used instead of the caps with rod handle shown in Figs 29, and 30.

*c* This bottle (Fig. 30) is used for extremely hygroscopic materials; only the small cap is removed for vacuum drying, and it is replaced as soon as the vacuum is released. The bottle containing the combustion boat with sample may be attached to a combustion tube by means of a rubber adapter, and the boat pushed into position in the tube by passing a wire through the small joint.

*d* These bottles are designed for use in conjunction with the combustion boats (Figs. 23 to 25), as follows:

<table>
<thead>
<tr>
<th>Bottle</th>
<th>Use with boat</th>
</tr>
</thead>
<tbody>
<tr>
<td>Fig. 31</td>
<td>Fig. 23</td>
</tr>
<tr>
<td>Fig. 32</td>
<td>Fig. 24</td>
</tr>
<tr>
<td>Fig. 33</td>
<td>Fig. 25</td>
</tr>
</tbody>
</table>

The combination is useful for weighing and drying extremely hygroscopic material. Sample, boat, and bottle are weighed, and the combination, without removing the cap, is placed in the drying apparatus (Fig. 65). During drying, vapors escape through the capillary. After drying, the combination is reweighed.
3. Weighing on the Microchemical Balance

WEIGHING BOTTLE, PIG-TYPE, WITH OUTSIDE CAP

This is the so-called Friedrich-type weighing bottle (Fig. 29). It should be made preferably from soda-lime glass in order to reduce accumulation of electrostatic charges. The outside cap for the weighing bottle (Fig. 28) may be used instead of the cap with the rod handle. The capacity of this bottle is approximately 3 ml. (see Table 12).

WEIGHING BOTTLE WITH TWO CAPS

This bottle (Fig. 30) should also be made preferably from soda-lime glass. The approximate capacity of the main body is 3 ml. This weighing bottle is used for extremely hygroscopic materials; only the small cap is removed for vacuum drying, and it is replaced as soon as the vacuum is released. For the determination of hydrogen or oxygen in extremely hygroscopic materials, the
weighing bottle containing the combustion boat with sample is attached to a combustion tube by means of a rubber adapter, and the boat pushed into position in the combustion tube by passing a wire through the small joint. The outside cap for the weighing bottle (Fig. 28) may be used instead of the cap with the rod handle (see Table 12).

NOTE: THE TWO FEET AND THE BEAD OF THE LARGER CAP SERVE AS A 3-POINT SUPPORT.

NOTE: GRINDINGS—GROUND SURFACES OF INNER MEMBERS MUST NOT EXTEND OUT FROM CAPS MORE THAN 1 MM.

Fig. 30. Weighing bottle with two caps—details of construction.

WEIGHING BOTTLE, PIG-TYPE, METAL

Size A

This weighing bottle (Fig. 31), referred to as the Hayman type, is designed for use in conjunction with the boat, size A (Fig. 23), as shown in the assembly drawing. The bottle should be made of an aluminum alloy, and the cap should be lapped to fit the body joint. The combination is useful for weighing and drying extremely hygroscopic material. Sample, boat, and bottle are weighed, and the combination, without removing the cap, is placed in the modified Abderhalden drying apparatus (Fig. 65). During the drying

NOTE: DIMENSIONS TO HAVE TOLERANCE OF ±0.010" UNLESS OTHERWISE SPECIFIED. DIMENSIONS IN MILLIMETERS ARE APPROXIMATE.

Fig. 31. Weighing bottle, pig-type, metal, size A—details of construction.
operation, water leaving the sample passes through the capillary. The combination is finally removed from the dryer and again weighed. The approximate capacity is 0.5 ml.; the approximate weight is 1.5 grams (see Table 12).

**Size B**

This weighing bottle (Fig. 32) should be made from an aluminum alloy, and the cap should be lapped to fit the body joint. It is designed for use with the combustion boat, size B (Fig. 24), as shown in the assembly drawing. The manner in which it is used is described under weighing bottle, pig-type, metal, size A. The approximate capacity of the bottle is 1 ml.; the approximate weight is 4.5 grams (see Table 12).

![Fig. 32. Weighing bottle, pig-type, metal, size B—details of construction.](image)

**Size C**

This weighing bottle (Fig. 33) should be made of an aluminum alloy, and the cap should be lapped to fit the body joint. It is designed for use with the combustion boat, size C (Fig. 25), as shown in the assembly drawing. The manner in which it is used is described under weighing bottle, pig-type, metal, size A. The approximate capacity is 2 ml.; the approximate weight is 6 grams (see Table 12).

![Fig. 33. Weighing bottle, pig-type, metal, size C—details of construction.](image)
Three types of tare flasks are recommended, one with, and two without a ground-in stopper (Figs. 34, 35, and 36). They should be made preferably of soda-lime glass. On all three flasks the serial numbers should be etched in order to avoid rough surfaces. They are used as counterweights for such items as absorption tubes, crucibles, filter tubes, etc. Their weights are adjusted by adding small lead shot or glass beads until these closely approximate those of the objects which are to be counterbalanced. The tare flasks are marked and stored in the balance case.

**Fig. 34.** (Left) Tare flask, without stopper, small—details of construction.  
**Fig. 35.** (Right) Tare flask, without stopper, large—details of construction.  
**Fig. 36.** Tare flask, with stopper—details of construction.
3. Weighing on the Microchemical Balance

**SPATULA, METAL**

**Type A**

This type of spatula (Fig. 37), in addition to being useful as a general spatula, can be used as a preparative tool, the bottom end for crushing crystals and the bent blade for scraping containers. It is to be made preferably of stainless steel.

**Type B**

This spatula (Fig. 38) has a flat, bent portion at one end, and a V-shaped scoop at the other. It is made preferably of stainless steel and is particularly useful in the weighing of samples.

**Type C**

The spatula (Fig. 39) is suitable for the larger samples commonly encountered in semimicro and preparative work. It has a U-shaped scoop at one end and a V-shaped scoop at the other, and is made preferably of stainless steel. The spatula can be used for adding lead shot or beads to the tare flasks.
(Figs. 34, 35, and 36) and for measuring and introducing the solid reagent into the combustion tube of the apparatus for the manometric determination of carbon\(^62,66\) (see Chapter 18).

![Diagram of apparatus](image)

**Fig. 39.** (Left) Spatula, metal, type C—details of construction.
**Fig. 40.** (Right) Spatula, metal, type D—details of construction.

**Type D\(^6,63\)**

This spatula (Fig. 40), which is actually a dental spatula, has been found useful by the author and other experienced analysts. It is made preferably of stainless steel.

**STEEL FORCEPS (NICKEL PLATED) WITH PLATINUM–5% RUTHENIUM TIPS\(^6,63\)**

The tips are sturdy and are made from the platinum–5% ruthenium alloy. A pin has been included. This serves as a stop to prevent the forceps from being depressed to such an extent that the tips can open, allowing the held object to drop. When the forceps are pressed together, the tips make contact for a distance of 0.25 to 0.5 inch. Figure 41 shows the forceps with platinum–5% ruthenium tips. The forceps may be made without rare metal tips, using any other metal such as nickel-plated or stainless steel, depending upon their in-
tended use. The construction and over-all dimensions should be identical to those shown in Fig. 41.

Fig. 41. Steel forceps (nickel plated) with platinum-5% ruthenium tips—details of construction.

STEEL FORCEPS (NICKEL PLATED) WITH CONICAL TAPERED HOLDERS

Until the introduction of the forceps of the type shown in Fig. 42, absorption tubes, filter tubes, etc., were handled with the fingers covered with chamois cots, with metal forks, or with forceps prepared by soldering sections of metal tubing to the tips of dissecting forceps. The forceps (Fig. 42), which have been commercially available for a number of years, are of superior construction and provide a better means of handling objects. Because they are made in one piece from spring steel, they are also more durable.

DESICCATORS

Either of the desiccators shown in Figs. 43, 44, and 46 may be used for cooling, storage and safe transportation of microweighing equipment. They are used in combination with the metal cooling block which is made of
a metal or alloy with a high heat conductivity and whose surface is highly resistant to abrasion and corrosion (Fig. 45). One desiccator (Figs. 43 and 46) consists of an aluminum body and a borosilicate glass cover, and the other desiccator (Fig. 44) consists of a complete glass desiccator with cover and contains an aluminum insert for holding a metal cooling block. In general,

**Fig. 42.** Steel forceps (nickel plated) with conical tapered holders—details of construction.

**Fig. 43.** Metal crucible container with glass cover (desiccator)—details of construction (*left*, glass part, *right*, metal part).
the author prefers the use of the former, except in such cases where the use of desiccants is required.

FIG. 44. Glass desiccator with metal insert.

FIG. 45. (Left and center) Metal cooling block, including details of construction.
FIG. 46. (Right) Metal crucible container with glass cover and metal cooling block.

WEIGHING (OR CHARGING) TUBES

Three types of weighing or charging tubes (Figs. 47, 48, and 49) are used for weighing samples by difference. The two types with ground caps are used for hygroscopic samples. These may be inserted into the drying apparatus for additional drying purposes.

RACK

A rigid wire rack of the type shown in Fig. 50 is used to support such items as absorption tubes (Chapter 9) and filter tubes (Chapters 11 and 16) while waiting for them to come to equilibrium before weighing.
A metal fork (Fig. 51) prepared from twisted wire is used for transferring absorption tubes and filter sticks from the rack to the balance. It catches the end of the object whose weight holds it in place.

Krūnel'd iron wires (Fig. 52) about 1 mm. O.D. and 120–150 mm. in length are used as swab sticks with cotton tips for cleaning out the ends of various objects previous to weighing.

Small lead shot of various sizes are used in the tare flasks (Figs. 34, 35, and 36) to increase the weight of the latter to within a few milligrams of that desired. Glass beads may be used instead.
3. Weighing on the Microchemical Balance

Fig. 49. Weighing (charging) tube with vented cap—details of construction.

Fig. 50 (Left) Wire rack.

Fig. 51 (Right) Fork, showing method of use.

Fig. 52. Knurled iron wire.
Weighing of Samples

NON-HYGROSCOPIC SAMPLES
WEIGHED IN BOATS

A large portion of the samples dealt with are weighed in small combustion boats, either platinum⁶⁻⁶²⁻⁶⁴ (Figs. 23–25) or porcelain⁴⁴,⁴⁵,⁶² (Fig. 22). If the samples are not hygroscopic, these may be placed directly on the left-hand pan. If, however, the sample is hygroscopic the weighing is accomplished as described below. The boats are first cleaned by immersion in boiling dilute nitric acid, washed with distilled water, and then heated to redness in the flame of a burner. The boat is best held by means of a platinum wire⁴⁴,⁴⁶,⁶² hook (sealed into a glass rod, Fig. 53) which fits into the small hole in the former.

![Fig. 53. Platinum hook (sealed into glass rod).](image)

After ignition, the boat is allowed to cool on a metal block in a small desiccator⁶⁻⁴¹,⁴²,⁴⁴,⁴⁵,⁶²,⁶⁴ (Figs. 43–46). Platinum boats may be weighed after about 10 minutes, but porcelain boats should not be weighed for at least 30 minutes. Tare counter weights may be prepared for each boat from aluminum, bronze, or glass rod by carefully filing or grinding away material until the weight is within a milligram of that of the boat.⁵²,⁵⁴,⁵¹–⁵⁴ The tare and boat then should be kept together. The exact weight of the boat need not be known, since the weight of a sample is obtained by the gain as described below.

The boat is placed on the left-hand pan of the balance by means of a platinum-tipped forceps (Fig. 41), and the tare counter weight placed on the right-hand pan using ordinary bone-tipped weight forceps. The balance is brought as close to equilibrium as possible by moving the rider to the nearest notch in a manner similar to that employed with an analytical balance. Each time the beam is set down on its knife edges, it should not be allowed to actually swing. The release should be only enough so that the direction of swing of the pointer can be noted. After the rider has been placed in the notch necessary for equilibrium, the balance is allowed to swing and the deflection noted as described above. If it is found to be ±50, or greater, the rider should be moved one notch so that a value of less than ±50 is obtained. The weight as shown on the rider plus or minus the deflection* is recorded

* It is not necessary to take the zero reading of the balance since it will not change during the course of the few minutes required to weigh the sample—it is assumed to be zero in these weighings.
3. Weighing on the Microchemical Balance

as the weight of the boat, as for example, 3.068 mg. The boat is then removed from the left pan and placed on the metal block of the microdesiccator.* The sample is then introduced into the boat with the aid of a microspatula61, 42,44,52,63 (Figs. 37–40). (Care must be exercised so that the outside of the boat does not have any attached particles of sample. If so, these are removed by either tapping the boat on the metal block or by holding it with the forceps and carefully brushing the outer sides and bottom with a small camel’s-hair brush, making certain that no hairs become detached from the brush and adhere to the boat.) The boat containing the sample is placed on the left-hand pan and the rider again moved to obtain equilibrium as above. Again, the combined rider and deflection are recorded, as for example, 9.873 mg. Subtracting the weight of the empty boat from this, i.e., 3.068, gives 6.805 mg. as the weight of the sample. Besides the commonly used size B platinum combustion boat (Fig. 24), two other sizes may be used as shown in Table 11.

HYGROSCOPIC SAMPLES WEIGHED IN BOATS

When a hygroscopic sample is weighed in a boat, it must be protected from the moisture in the atmosphere. The boat is placed in the weighing bottle, pig-type, with outside cap (Fig. 29; also see Table 12).6 (This weighing bottle or pig should be made preferably from soda-lime glass in order to reduce accumulation of electrostatic charges after being wiped clean with a chamois skin. Practically all glass articles that are to be weighed are prepared from this glass in preference to borosilicate glass, because the latter retain such charges†).

The pig should be wiped with a chamois skin, grounded against a light fixture or pipe, and then let stand for about 15 minutes before being placed on the balance. (During this time the charge will be lost.‡) The pig must be handled by means of a chamois skin from this point, but should not be stroked lest it again becomes charged. It is then placed on the balance along with a boat. The combination is weighed, removed from the balance, sample added to the boat as described previously in this section, the boat inserted into the stoppered pig and the combination reweighed and then preserved in this

* Boats should always be removed from the balance before attempting to add samples. Samples should never be added to a boat on the balance pan.
† All glassware develops a charge (when wiped with the chamois skin), detectable on the microchemical balance. As stated in Chapter 1, when the relative humidity is kept at 40–50%, 20,54,62 the charges are rapidly lost. Grounding exposure to U.V. light, 45,49,61,62 contact with a high-frequency discharge or with radioactive material also accomplishes the same aim.
‡ If the electrostatic charge is not lost, on the balance the object decreases in weight so rapidly that weighing is impossible. The rate of decrease is often as much as 0.3 mg. in several minutes.
condition until analyzed. If the sample is very hygroscopic it will be necessary to weigh the boat and pig, correcting for the zero reading of the balance. An approximate amount of sample is then added to the boat, and the open pig, boat, and sample are dried several hours in a drier (see Chapter 4, Preparation of Sample). The combination is then handled with a chamois skin and weighed, taking into account the zero reading of the balance. This same technique is used with samples which readily absorb carbon dioxide from the air, as for example, some amines.

Very hygroscopic samples are best handled by means of either the combination of platinum boats and the various weighing bottles shown in Figs. 30–33, or the Friedrich-type (Fig. 29) but with a cap having a capillary opening such as that of the weighing bottle shown in Fig. 28. The boat and container are first accurately weighed. The approximate amount of very hygroscopic sample is added and the unit, with the stopper attached, is placed in a drier. Moisture leaves the sample by way of the capillary opening. After drying, the unit is again weighed. The capillary opening, being small, does not allow an appreciable amount of moisture to re-enter the system during the weighing time. (A series of experiments established the following: (a) Water vapor diffuses through a dry ground joint and (b) There is little difference in the amount of diffusion whether the cap or stopper is closed or contains a capillary, straight or with bulbs. Benedetti-Pichler and Bromund recommended lubrication and the Committee on Microchemical Apparatus of the Division of Analytical Chemistry, American Chemical Society, established that the only way to prevent absorption of moisture is by means of a lubricated closed cap. However, the author does not recommend the use of lubricants in any drying procedure due to possible contamination.)

**WEIGHING OR CHARGING TUBES**

Three types of so-called weighing (charging) tubes are used for weighing samples. For non-hygroscopic samples the open end variety (Fig. 47) may be used but for hygroscopic samples the glass-stoppered types (Figs. 48 and 49) are necessary. The charging tubes are generally used, for samples that are to be placed eventually in Carius combustion tubes (see Chapters 10 and 11) or Kjeldahl digestion flasks (see Chapters 9 and 12). With charging tubes, samples are weighed by difference. The charging tube is wiped with the chamois skin, grounded against a light fixture or pipe, and set aside on a wire rack (Fig. 50) for about 15 minutes.* It is then held with a chamois skin or special forceps (Fig. 42), the approximate weight of sample added

* If the electrostatic charge is not lost, on the balance the object decreases in weight so rapidly that weighing is impossible. The rate of decrease is often as much as 0.3 mg. in several minutes.
by means of a microspatula,* and then placed horizontally on the hooks on the left-hand hanger of the balance. (As a tare counterweight, either aluminum, glass or brass rod may be used—see paragraph above regarding weighing with boats.) The charging tube plus sample is accurately weighed (zero reading of balance need not be determined—it is assumed to be zero in this case). By means of the special forceps or chamois skin, the charging tube is removed from the balance, the sample dumped out into the desired container (making certain that no particles adhere to the edges) and the empty charging tube reweighed.

**LIQUID SAMPLES**

(a) High-boiling liquids with a low vapor pressure should be weighed in boats in the same manner as non-hygroscopic solids. (b) Low-boiling liquids may be handled either by (1) or (2).

1. **Capillaries.** Low-boiling liquids must be weighed in glass capillary tubes of the type shown in Fig. 54. These are prepared by drawing out a piece of molten tubing so that the inside diameter is about 1 mm. They are cut in lengths of 2.5 to 5 cm. depending upon the purpose for which they will be used (5 cm. for Carius combustions, 2.5 cm. for all others—see following chapters). One end is sealed by heating in a flame. To the tubes which are to be used in connection with the carbon-hydrogen and Dumas nitrogen determinations, about 2–3 mm. of powdered potassium chlorate should be added by the method used for filling melting-point capillaries. After the chlorate is in the bottom (sealed end), the powder is melted by warming over a small flame and then immediately allowed to resolidify. This prevents loss on handling and subsequent weighing errors. Tubes both with and without added chlorate may be further constricted at the open end to a finer capillary if so desired. The additional constriction is desirable when handling very low-boiling liquids, but introduces the danger of breaking the fragile tip while handling. The tube is wiped clean with a chamois skin and allowed to set some minutes before placing on the balance pan. It is then placed on the left-hand pan with the aid of the forceps and accurately weighed (assume zero reading to be zero), using the fractional weights as tares. The tube is removed from the balance with the aid of the forceps (and chamois skin) and held for a few seconds about an inch above a heated hot plate or burner of a combustion furnace to expand the air inside. The open end is immediately placed below the surface of the sample and held there until the liquid ceases to rise in the

* Caution: Remove any adhering particles on outside as above described.

† The chlorate decomposes on heating, yielding oxygen. This furnishes an atmosphere of oxygen in the capillary and also sweeps out the volatilized sample.
capillary. The tube is then placed, closed end down, in a centrifuge and whirled for a few seconds to force the liquid to the bottom. The tube is gently wiped and returned to the balance for an approximate weighing. If too little sample is present, the tube may be gently warmed again, making certain not to vaporize the liquid already in the capillary, and the above process repeated as many times as necessary. If, however, too much sample is obtained the capillary may be partly emptied by shaking while inverted or by warming to the extent that some sample volatilizes. After the desired approximate amount is in the tube, it is sealed by holding the open end in a flame, first making certain that no liquid is near this end. The tube is again wiped as above and accurately weighed. Before placing such samples in the necessary apparatus for the determination, the tip is scratched with a file and broken off—then both portions are introduced.

![Diagram of capillaries](image)

Fig. 54. Glass capillaries. (A) Plain capillary tube, unconstricted. (B) Tube containing \( \text{KClO}_3 \), unconstricted. (C) Constricted tube containing \( \text{KClO}_3 \). (D) Sealed off tubes, both unconstricted and constricted, containing \( \text{KClO}_3 \) and sample.
(2) \textit{Capsules.} Low-boiling liquids may also be weighed in methylcellulose or gelatin capsules, depending, of course, whether or not these capsules contain the element(s) or group to be determined. They do, however, present the problem of destroying considerably more organic material than ordinarily encountered. They are best weighed when placed in the type of small aluminum cup shown in Fig. 55. The cup and \textit{two caps} are weighed. All parts should be handled by means of forceps, preferably those shown in Fig. 42. To the one cap, supported in the aluminum cup is placed the required amount of sample and then the second cap, \textit{closed end down}, is forced into the first cap (Fig. 56). This forms a tight seal, preventing escape of the sample. The combination is reweighed to obtain the weight of sample.

\begin{figure}[h]
\centering
\includegraphics[width=0.3\textwidth]{fig55.png}
\caption{(Left) Aluminum cup for holding methylcellulose (or gelatin) capsules. Enlarged (approximately 2 × actual dimensions) to show details.}
\end{figure}

\begin{figure}[h]
\centering
\includegraphics[width=0.3\textwidth]{fig56.png}
\caption{(Right) Methylcellulose (or gelatin) capsules showing method of fitting together for effective closure. Enlarged (approximately 2 × dimensions) to show details—preferably capsule size No. 4 or No. 5.}
\end{figure}
ADDITIONAL INFORMATION FOR CHAPTER 3

Alber\textsuperscript{3,28,62} described the system shown in Fig. 57 for filling capillary tubes with liquids, particularly hygroscopic ones. The system is momentarily evacuated causing a reduced pressure in the capillary. On breaking the vacuum, the liquid is forced up into the tube. A similar system is described by Furman\textsuperscript{22}.

\begin{table}[h]
\centering
\caption{ADDITIONAL INFORMATION ON REFERENCES\textsuperscript{*} RELATED TO CHAPTER 3}

Following the system used in the preceding chapters, this table lists additional references the author wishes to call to the attention of the reader. (See statement regarding such information at top of Table 4 of Chapter 1.)

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\textsuperscript{*} The numbers which appear after each entry in this table refer to the literature citations in the reference list at the end of the chapter.
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