3.2 Siting the balance

For best results, the balance should be placed on a level and stable surface in an environment not subject to chemical fumes or rapid changes in temperature. In addition, the balance should not be close to any source of variable stray magnetic field, such as a conventional magnetic susceptibility balance using an electromagnet, or an e.s.r. spectrometer. If in doubt, check whether the zero reading of the balance changes when the external magnetic field is varied. If the balance is placed on a bench with drawers, ensure that large ferromagnetic objects are not situated directly below.

3.3 Packing the sample tube

With solid samples, considerable care should be taken in packing the powder into the sample tube since the major error in the Gouy method normally arises from inhomogeneous packing. The sample should be in the form of a reasonably fine and uniform powder. Large crystals will not only pack in an inhomogeneous manner but may also result in an error due to magnetic anisotropy and may require an air correction. Very fine powders, on the other hand, can pack unevenly. If the substance is available in a reasonably fine crystalline form, then lightly crushing any aggregates with a plastic spatula is advised.

A small amount of solid is then introduced into the weighed sample tube, and the bottom of the tube gently tapped on a wooden bench a number of times to settle the solid particulates. This procedure is repeated, until a sufficient amount of sample is added, corresponding to a sample length, \( l \), in the range 2.5cm - 3.5cm are recommended. The minimum value of \( l \) is 1.5cm, above which the reading is usually not affected. A narrow bore tube can be used to ensure this length when there is not much sample.

Even packing can be ensured by taking readings in between tapping the sample tube until the balance readings become constant. Further proof of the sample being homogeneous and well packed can be obtained by taking readings while rotating the tube containing the sample and noting the readings in different positions.

After the first measurement, it is advisable to empty out the sample, repack the tube and repeat the procedure several times, to ensure the measurement is reproducible.

The dimensions of the standard sample tubes are:

<table>
<thead>
<tr>
<th></th>
<th>Outside Diameter</th>
<th>Inside Diameter</th>
<th>Cross-sectional Area</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>0.400 ± 0.0013 cm</td>
<td>0.324 ± 0.0013 cm</td>
<td>0.08245 ± 0.00066 cm²</td>
</tr>
</tbody>
</table>
3.4 Operation of the balance

1. Turn the RANGE knob to the x1 scale and allow a 10 minute warm-up period before use. If the balance is to be used frequently it should preferably be left on continuously.

2. Adjust the zero knob until the display reads 000. The zero should be adjusted on each scale used.

3. Place an empty sample tube of known weight into the tube guide and take the reading, R_0.

4. Pack the sample as suggested in section 3.3 above and note the sample mass m in grams and the sample length, l, in cm.

5. Place the packed sample tube into tube guide and take the reading, R.

N.B. A negative reading indicates that the tube plus sample have a net diamagnetism.

6. If the display goes off scale turn the RANGE knob to the x10 scale, re-zero and multiply the reading by 10.

The mass susceptibility, \( \chi_g \), is calculated using:

\[
\chi_g = \frac{C_{bal} \cdot l \cdot (R - R_0)}{10^9 \cdot m}
\]

Where:
- \( l \) = the sample length (cm)
- \( m \) = the sample mass (gm)
- \( R \) = the reading for tube plus sample
- \( R_0 \) = the empty tube reading
- \( C_{bal} \) = the balance calibration constant

With the recommended sample tubes R_0 will vary only slightly and for most purposes a constant value can be assumed. A value of R_0 should, however, be determined each time a thicker walled sample tube is used or for each tube is sample susceptibility values are very low. Since glass is diamagnetic R_0 will be negative. Some samples have their readings vary with ambient temperature. In such cases, the samples and balance should be allowed to come to temperature equilibrium before measurement and the temperature of the balance room should be recorded with the reading.

An acceptable method of operation is to zero the balance with an empty tube in place, thus R - R_0 in the equation will be replaced by a single reading R, when the tube containing the sample is introduced.
WORKED EXAMPLE

Table 1, which follows, gives a selection of experimental results.

Taking MnSO₄·4H₂O as an example:

\[ \chi_g = \frac{2.086 \times 3.8 \times [2856 - (-17)]}{10^9 \times 0.3431} \]

Thus,

\[ \chi_g = 66.38 \times 10^{-4} \text{ (c.g.s)} \]

Both cgs. and SI units can be used to describe magnetic susceptibility, but since most data in the literature on the subject are expressed in cgs. units, these are used exclusively in this manual.

Using Sample Tubes of other internal diameters

The reading displayed by the balance is proportional to the sample’s volume susceptibility and the volume of sample present in the measuring region of the balance. \( C_{Bal} \) is a function of tube internal diameter. Since most measurements are made using standard tubes (0.4 cm. OD x 0.324 cm. ID) the reference and calibration use this size.

If non-standard tubes are used the volume of sample in the measuring region will differ from that used for calibration. There are two ways to correct for this.

1. Repeat the calibration procedure using a substance of known susceptibility in a tube of the non-standard size.

2. Multiply the reading with the non-standard tube by the ratio of the squares of the internal diameters, standard/non-standard.

   e.g. using a narrow bore tube of inside diameter 0.2 cm. the ratio is \((0.324 \times 0.324)/(0.2 \times 0.2) = 2.624\). The sample reads +216 and a similar empty tube -046. The calibration constant with a standard bore tube is 1.115. Thus

\[ \chi_v = \frac{1.115}{10^-} \times (216 - (-046)) \times 2.624 = 0.767 \times 10^{-6} \text{ cgs.} \]

Care also has to be taken when converting to mass susceptibility. To be sure of correcting for the non-standard tube, this must be done using the fundamental formula:

\[ \chi_g = \frac{\chi_v \times l \times A}{m} \quad \text{where} \ A = \text{the internal cross-sectional area of the tube} \]

3.5 Conversion of cgs. units to SI units

- Volume susceptibility \( \chi_v \) multiply cgs. value by 4π
- Mass susceptibility \( \chi_g \) multiply cgs. value by 4π x 10⁻³
- Molar susceptibility \( \chi_m \) multiply cgs. value by 4π x 10⁻⁶