NOTE

THE BEAM OF THIS BALANCE IS FITTED WITH A RESTRAINING CLAMP TO PROTECT THE SUSPENSION ELEMENTS DURING TRANSIT. THE CLAMP MUST BE REMOVED BEFORE USE. REFER TO CALIBRATION AND SET UP INSTRUCTIONS IN APPENDIX G.
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1. OPERATING GUIDELINES

The magnetic susceptibility balance is a sensitive instrument that, if used in accordance with the guidelines given in this manual, should give trouble-free service.

A number of important rules with regard to the use and maintenance of the balance should be adhered to if best results are to be achieved.

1. The removal of the transit clamp may alter the zero setting of the beam. Should it not be possible to zero the display using the ZERO knob please refer to Appendix G for full setup procedure.

2. The balance should be set up on a flat, stable surface free from vibration, and should be positioned away from the influence of stray magnetic fields.

3. Ferromagnetic materials should not be placed inside or near the balance. Ferromagnetic impurities introduced in sample preparation can lead to considerable errors. The use of plastic or non-magnetic stainless steel spatulas is therefore recommended.

4. The sample guide is designed such that a glass tube broken while using the balance will fall through the base of the unit. Care should be taken not to damage the thin wall of the guide tube when cleaning.
2. DEFINITION OF MAGNETIC SUSCEPTIBILITY

“The ratio of the intensity of magnetism induced in a substance to the magnetising force or intensity of field to which it is subjected”.

Volume Susceptibility \[ \chi_v = \frac{I}{H} \]

Where: \( I \) = the intensity of magnetism induced  
\( H \) = the intensity of magnetic field applied

Mass Susceptibility \[ \chi_g = \frac{\chi_v}{d} \]

Where: \( d \) = the density of the substance

Molar Susceptibility \[ \chi_m = \chi_g \times MW \]

Where: \( MW \) = the molecular weight of the substance.
3. THE MAGNETIC SUSCEPTIBILITY BALANCE

3.1 Introduction

The SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance (M.S.B.) is the result of collaboration with Professor D. F. Evans of Imperial College, London, and is designed as a replacement for a traditional Gouy balance system. The Evans method uses the same configuration as the Gouy method (see Appendix A) but, instead of measuring the force which a magnet exerts on the sample, the equal and opposite force which the sample exerts on a suspended permanent magnet is observed. The apparatus is shown diagrammatically in Figure 1.

The M.S.B. works on the basis of a stationary sample and moving magnets. The pairs of magnets are placed at opposite ends of a beam so placing the system in balance. Introduction of the sample between the poles of one pair of magnets produces a deflection of the beam that is registered by means of phototransistors. A current is made to pass through a coil mounted between the poles of the other pair of magnets, producing a force restoring the system to balance. At the position of equilibrium, the current through the coil is proportional to the force exerted by the sample, and can be measured as a voltage drop.

The following general expression for mass susceptibility, $\chi_g$, in cgs. Units may be derived in the same manner as for the traditional Gouy method (Appendix A):

$$\chi_g = \frac{l}{m} \left( C \left( R - R_0 \right) + \chi_{vair} \cdot A \right)$$

Where:

$C = \text{a constant of proportionality}$

$R = \text{the reading obtained for tube plus sample}$

$R_0 = \text{the empty tube reading (normally a negative value)}$

$l = \text{the sample length (in cm)}$

$m = \text{the sample mass (in gm)}$

$A = \text{the cross-sectional area of the tube (in cm}^2\text{)}$

$\chi_{vair} = \text{the volume susceptibility of the displaced air}$

For powder samples the air correction term, $\chi_{vair} \cdot A$, may normally be ignored. $C$, the constant of proportionality is related to the calibration constant of a given balance by the formula

$$C = \frac{C_{bal}}{10^9}$$
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>Description</th>
<th>Value</th>
<th>Standard Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>Outside diameter</td>
<td>0.400</td>
<td>± 0.0013 cm.</td>
</tr>
<tr>
<td>Inside diameter</td>
<td>0.324</td>
<td>± 0.0013 cm.</td>
</tr>
<tr>
<td>Cross-sectional area</td>
<td>0.08245</td>
<td>± 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:

$$ \chi_g = \frac{C_{\text{Bal}} \cdot l \cdot (R - R_0)}{10^3 \cdot m} $$

Where:
- $l$ = the sample length (cm)
- $m$ = the sample mass (gm)
- $R$ = the reading for tube plus sample
- $R_0$ = the empty tube reading
- $C_{\text{Bal}}$ = 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 MnSO$_4$.4H$_2$O as an example:

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

Thus,

\[
\chi_g = 66.38 \times 10^{-6} \text{ (c.g.s.)}
\]

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

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

   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:

   \[
   \chi_g = \frac{\chi_v \times \text{density} \times A}{m}
   \]

   where $A$ = 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 $4\pi$
- Mass susceptibility $\chi_g$ multiply cgs. value by $4\pi \times 10^{-3}$
- Molar susceptibility $\chi_m$ multiply cgs. value by $4\pi \times 10^{-6}$
TABLE 1

<table>
<thead>
<tr>
<th>Compound</th>
<th>Sample Mass m (gm)</th>
<th>Typical Experimental Values</th>
<th>Literature Value</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$l$ (cm)</td>
<td>$R$</td>
<td>$10^6 \chi_g$</td>
</tr>
<tr>
<td>MnSO$_4$.4H$_2$O</td>
<td>0.3431</td>
<td>3.8</td>
<td>2856</td>
</tr>
<tr>
<td>CoCl$_2$.6H$_2$O</td>
<td>0.1743</td>
<td>2.5</td>
<td>1344</td>
</tr>
<tr>
<td>Hg[Co(SCN)$_4$]</td>
<td>0.4106</td>
<td>3.8</td>
<td>830</td>
</tr>
<tr>
<td>CuSO$_4$.5H$_2$O</td>
<td>0.2018</td>
<td>1.7</td>
<td>318</td>
</tr>
<tr>
<td>Empty tube</td>
<td>-</td>
<td>-17</td>
<td>-</td>
</tr>
<tr>
<td>H$_2$O (liq)</td>
<td>0.2950</td>
<td>3.6</td>
<td>-45</td>
</tr>
</tbody>
</table>

Notes

Values of $\chi_g$ are in cgs. units

Literature values are from work indicated in Appendices E & F

Temperature of the laboratory = 20°C

For the Balance used in the above Table, $C_{Bal} = 2.086$ (In the past, this constant would vary from balance to balance. Current manufacturing procedures allow us to make the $C_{Bal} = 1.0$ and. should it be required, to adjust this constant in the laboratory. See Appendix G, Stage III).
3.6 Measurements on liquids or solutions

Liquid samples can be treated in the same way as solids. The full expression allowing for the susceptibility of the displaced air should be used, and a correction applied for the volume of the meniscus. Alternatively, if the density \(d_S\) in gm/cc of the solution is known, a convenient expression for the mass susceptibility \(\chi_S\) of the solution is:

\[
\chi_S = \frac{C_{Bal} \cdot I \cdot (R - R_0)}{10^9 \cdot A \cdot d_S} + \frac{\chi_{V, air}}{d_S} \quad \text{(terms are as defined in section 3.1)}
\]

The mass susceptibility of a solution can be obtained from the Weidemann additivity relationship. For \(m_1\) grams of solute and \(m_0\) grams of solvent:

\[
\chi_S = \frac{m_1}{m_1 + m_0} \chi_g + \frac{m_0}{m_1 + m_0} \chi_0
\]

Where \(\chi_S\) = the mass susceptibility of the solution
\(\chi_g\) = the mass susceptibility of the solute
\(\chi_0\) = the mass susceptibility of the solvent.

\(\chi_0\) is found by a separate measurement.

In measurement on liquids, it is important to ensure that there are no air bubbles on the side of the tube.

3.7 Calibration of the balance

Each balance is individually calibrated and the calibration constant, \(C_{Bal}\) printed on the rear of the case. The calibration can be checked from time to time using a standard substance. If the balance is to be used mainly for solid samples, then a solid calibrant (preferably HgCo(SCN)_4) is recommended since some of the systematic errors in packing may cancel. The constancy of the calibration can also be checked using a sealed-off sample of MnCl_2 solution.

Note that the Curie-Weiss Law applies and the reading will be related to the tube temperature, \(T^\circ\text{C}\), by the factor: -

\[
\frac{1}{T + 255}
\]

Details of standard substances for calibration of magnetic balances are given in Appendix E.
3.8 Uses for the SHERWOOD SCIENTIFIC M.S.B.

1. **Assignment the oxidation state** of the metal in complexes of the transition, lanthanide and actinide elements. Examples are given in Appendix C.

2. **Stereochemical information** - for example square planar \( \text{Ni}^{2+} \) complexes are diamagnetic, while octahedral \( \text{Ni}^{2+} \) complexes are paramagnetic with two unpaired electrons.

3. **Information concerning ligand field strength** - transition metal complexes can be high-spin or low-spin, depending, in part of the field strength of the ligands. Therefore the calculation of magnetic moments and hence the number of unpaired electrons can be used to assess ligand field strength.

4. **Antiferromagnetic interactions in dimers and polymers** - antiferromagnetic interactions between neighboring metal atoms or ions in dimeric or polymeric complexes (e.g. cupric acetate) will lead to magnetic moments which are smaller than expected.

5. **Complexation by ligands** - for a number of transition metal species, complexation by ligands alters the magnetic behaviour. Thus, a number of square planar \( \text{Ni}^{2+} \) complexes are diamagnetic when dissolved in non-coordinating solvents such as benzene and chloroform but paramagnetic in coordination of two ligand pyridine. This is due to axial coordination of two ligand molecules to give a 6-coordinate \( \text{Ni}^{2+} \) complex.

6. **Criterion of purity** - pure \( \text{Y}_2\text{O}_3 \) is diamagnetic, but contamination with lanthanides such as erbium or dysprosium can cause samples to be paramagnetic.

7. **Measurement on air-unstable compounds** - the balance is ideally suited for compounds which decompose when exposed to the air since almost anything that is not ferromagnetic or too bulky or heavy can be sealed into the sample tubes. For example, a glass B10 socket and a stopper can be used to seal the end of a sample tube.

8. **Measurement of solutions** - liquid samples are readily handled and magnetic titrations can be performed.
APPENDIX A

THE TRADITIONAL GOUY METHOD

The most commonly used method for measuring magnetic susceptibility is that introduced by Gouy in 1889 which is shown diagrammatically in Figure 2, page 14.

The sample, normally contained in a suitable tube, is suspended from a balance such that the bottom of the sample is in a region of high and uniform field strength \( H \), while the top is in a region of negligible field.

In cgs. units, the force \( F \) acting on the sample is given by: 

\[
F = \frac{1}{2} \chi_v \cdot A \cdot H^2
\]

Where:
- \( \chi_v \) = the volume susceptibility of the sample
- \( A \) = the sample cross sectional area.
- \( F = g^*\delta(m) \)
- \( \chi_g = \chi_v \cdot l/d \)
- \( A = m/l^*d \)

\( \delta(m) \) = apparent change in mass in grams on application of the magnetic field
- \( g = \) acceleration due to gravity (981 cm s\(^{-2}\))
- \( m = \) sample mass in grams
- \( d = \) sample density in g cm\(^{-3}\)
- \( l = \) sample length in cm.

Hence,

\[
\chi_g = \frac{2 \cdot g \cdot l \cdot \delta(m)}{H^2 \cdot m}
\]  

(ii)

If \( H \) is kept constant, this can be written as:

\[
\chi_g = \frac{C \cdot \delta(m) \cdot l}{m}
\]  

(iii)

Where: \( C = \) a constant

Subtracting the reading of the empty sample tube from the same tube containing the sample does not correct for the susceptibility of the air displaced by the sample. The 20.9% oxygen in air is paramagnetic - \( \chi_{V\text{air}} = 0.029 \times 10^{-6} \text{cgs. at } 20^\circ\text{C and 760 mm pressure, contributions from other constituents being two orders of magnitude less.} \) At room temperature the full statement of equation (i) to include the air correction is:

\[
F = \frac{1}{2} \left( \chi - 0.029 \times 10^{-6} \right) \cdot A \cdot H^2
\]  

(iv)
Leading to the corrected expression:

\[
\chi_g = \frac{0.029 \times 10^{-6} V + C \delta(m) \delta}{m}
\]  

(v)

Where: \( V = \) volume of the sample in cm\(^3\)

For moderately paramagnetic materials, the difference between the values of \( \chi_g \) calculated using (iii) and (v) is negligible. With diamagnetic or weakly paramagnetic materials the correction term becomes significant. However, it should be noted that equation (v) takes no account of the air trapped when solid samples are packed in the tube. The packed density of a powder is often less than half the true solid density, in which case greater error is introduced by using equation (v) rather than the simple equation (iii). For paramagnetic solids the use of the simple formula (iii) is recommended.

**Figure 2.**
APPENDIX B

BASIC MAGNETOCHEMISTRY

Substances may exhibit three main types of magnetic behaviour, ferromagnetism, paramagnetism and diamagnetism and some characteristic properties are listed below.

<table>
<thead>
<tr>
<th>Behaviour</th>
<th>Properties</th>
<th>Typical Examples</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ferromagnetism</td>
<td>Sample powerfully attracted to the strongest part of an inhomogeneous magnetic field. Magnetic properties dependent on magnitude of the applied field. Becomes paramagnetism above the Curie temperature.</td>
<td></td>
</tr>
<tr>
<td>Paramagnetism</td>
<td>Sample attracted to the strongest part of an inhomogeneous magnetic field. In many cases the magnetic susceptibility is, to a reasonable approximation, inversely proportional to the abs. temp. (Curie Law).</td>
<td></td>
</tr>
<tr>
<td>Diamagnetism</td>
<td>Sample very weakly repelled from the strongest part of an inhomogenous magnetic field. Susceptibility normally temperature-independent.</td>
<td>Always present, but outweighed by any paramagnetism or ferromagnetism</td>
</tr>
</tbody>
</table>

Other types of behaviour include temperature-independent paramagnetism (TIP) and antiferromagnetism.

TIP arises from the mixing-in of excited state(s) of the compound, under the influence of the applied magnetic field, to give a weak paramagnetism that is independent of temperature. In some spin-paired transition metal compounds with low-lying excited states, such as KMnO₄ and K₂Cr₂O₇, the TIP is sufficient to outweigh the underlying diamagnetism and a small overall paramagnetism results.

Antiferromagnetism, like ferromagnetism, is a cooperative phenomenon and involves the interaction of unpaired electrons on neighbouring metal atoms or ions. In contrast to ferromagnetism, the antiparallel arrangement of spins is the more stable, and the resulting paramagnetism is normally less than would be observed in the absence of this interaction.
If a substance is placed in a field of intensity $H$ gauss then $B$, the magnetic induction of the field within the substance, is given by:

$$B = H + 4\pi I$$

Where: $I$ = intensity of magnetism induced by the field

$I / H$ is called the volume susceptibility of the substance, and is given the symbol $\chi_V$. In most cases, a more useful quantity is the magnetic susceptibility per unit mass or mass susceptibility, $\chi_g$, equal to $\chi_V / d$, where $d$ is the density of the substance in gm/cm$^3$. It is conventional to regard $\chi_V$ as dimensionless and $\chi_g$ as having the dimensions of reciprocal density.

The molar susceptibility, $\chi_m$ is $\chi_g \times$ the molecular or formula weight of the substance.

**N.B.** For diamagnetic compounds $\chi$ is negative and for paramagnetic compounds $\chi$ is positive.

For compounds containing a paramagnetic ion, $\chi_m$ will be less than the susceptibility per gram atom of the paramagnetic ion, $\chi_A$, because of the diamagnetic contribution of the other groups or ligands present. Since magnetic moments are additive, $\chi_A$ can be obtained from $\chi_m$ by the addition of the appropriate corrections. Diamagnetic corrections for a variety of ions and ligands are given in Appendix D.

For a paramagnetic metal ion, it is customary to quote, not $\chi_A$, but the effective magnetic moment, $\mu_{\text{eff}}$, of the ion in Bohr magnetons (BM). $\mu_{\text{eff}}$ and $\chi_A$ are related by the expression:

$$\left(\mu_{\text{eff}}\right)^2 = \frac{3kT\chi_A}{NB^2}$$

Where: $N$ = Avogadro's number
$B$ = the Bohr magneton
$k$ = Boltzmann's constant
$T$ = absolute temperature (K).

Hence: -

$$\mu_{\text{eff}} = 2.828\sqrt{T^* \chi_A}$$

**N.B.** $\mu_{\text{eff}}$ will be independent of temperature for a substance obeying the Curie Law.
For compounds containing unpaired electrons, both the spin angular momentum and the orbital angular momentum of the electrons can contribute to the observed paramagnetism. However, for complexes of transition metal ions, the orbital contribution is largely “quenched” by the field due to the surrounding ligands. In this case, we have the simple “spin-only” formula: 

\[ \mu_{\text{eff}} = \sqrt{n(n+2)} \text{ BM} \]

Where: \( n \) = the number of unpaired electrons.

Values of \( \mu_{\text{eff}} \) as a function of \( n \) are given below.

<table>
<thead>
<tr>
<th>( n )</th>
<th>( \mu_{\text{eff}} ) (BM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>1.73</td>
</tr>
<tr>
<td>2</td>
<td>2.83</td>
</tr>
<tr>
<td>3</td>
<td>3.87</td>
</tr>
<tr>
<td>4</td>
<td>4.90</td>
</tr>
<tr>
<td>5</td>
<td>5.92</td>
</tr>
<tr>
<td>6</td>
<td>6.93</td>
</tr>
</tbody>
</table>
# APPENDIX C

## EXAMPLES OF OXIDATION STATES AND MAGNETIC MOMENTS

<table>
<thead>
<tr>
<th>Metal and oxidation state</th>
<th>Configuration</th>
<th>Number of unpaired electrons</th>
<th>Magnetic moments $\mu_{\text{eff}}$ (BM)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ti$^{3+}$</td>
<td>d$^1$</td>
<td>1</td>
<td>1.7 – 1.8</td>
</tr>
<tr>
<td>Ti$^{4+}$</td>
<td>d$^0$</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>V$^{3+}$</td>
<td>d$^2$</td>
<td>2</td>
<td>2.7 – 2.9</td>
</tr>
<tr>
<td>V$^{4+}$</td>
<td>d$^1$</td>
<td>1</td>
<td>1.7 – 1.8</td>
</tr>
<tr>
<td>V$^{5+}$</td>
<td>d$^0$</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Cr$^{2+}$</td>
<td>d$^4$</td>
<td>4 (2)</td>
<td>4.8 – 5.0 (3.0 – 3.3)</td>
</tr>
<tr>
<td>Cr$^{3+}$</td>
<td>d$^3$</td>
<td>3</td>
<td>3.7 – 3.9</td>
</tr>
<tr>
<td>Mn$^{2+}$</td>
<td>d$^5$</td>
<td>5 (1)</td>
<td>5.7 – 6.0 (ca. 2.0)</td>
</tr>
<tr>
<td>Mn$^{3+}$</td>
<td>d$^4$</td>
<td>4 (2)</td>
<td>4.8 – 5.0 (3.0 – 3.3)</td>
</tr>
<tr>
<td>Mn$^{4+}$</td>
<td>d$^3$</td>
<td>3</td>
<td>3.7 – 3.9</td>
</tr>
<tr>
<td>Fe$^{2+}$</td>
<td>d$^6$</td>
<td>4 (0)</td>
<td>5.0 – 5.6 (0)</td>
</tr>
<tr>
<td>Fe$^{3+}$</td>
<td>d$^5$</td>
<td>5 (1)</td>
<td>5.7 – 6.0 (2.2 – 2.5)</td>
</tr>
<tr>
<td>Co$^{2+}$</td>
<td>d$^7$</td>
<td>3 (1)</td>
<td>4.3 – 5.2 (2.0 – 2.7)</td>
</tr>
<tr>
<td>Co$^{3+}$</td>
<td>d$^6$</td>
<td>0</td>
<td>0*</td>
</tr>
<tr>
<td>Cu$^{1+}$</td>
<td>d$^{10}$</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>Cu$^{2+}$</td>
<td>d$^9$</td>
<td>1</td>
<td>1.8 – 2.1</td>
</tr>
<tr>
<td>Ag$^{1+}$</td>
<td>d$^{10}$</td>
<td>0</td>
<td>0</td>
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<tr>
<td>Ag$^{2+}$</td>
<td>d$^9$</td>
<td>1</td>
<td>1.7 – 2.1</td>
</tr>
<tr>
<td>Ru$^{2+}$</td>
<td>d$^6$</td>
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<tr>
<td>Ru$^{3+}$</td>
<td>d$^5$</td>
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<tr>
<td>Eu$^{2+}$</td>
<td>f$^7$</td>
<td>7</td>
<td>7.8 – 8.0</td>
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<tr>
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<td>f$^6$</td>
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<td>3.4 – 3.6</td>
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<td>3.4 – 3.6</td>
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<tr>
<td>U$^{6+}$</td>
<td>f$^0$</td>
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</tr>
</tbody>
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Notes: * Small Temperature-Independent Paramagnetism. Numbers in parentheses represent less common situations.
## APPENDIX D

### DIAMAGNETIC CORRECTIONS FOR IONS AND MOLECULES

<table>
<thead>
<tr>
<th>Cations</th>
<th>$10^6 \chi_m$ (cgs)</th>
<th>Anions</th>
<th>$10^6 \chi_m$ (cgs)</th>
<th>Molecules</th>
<th>$10^6 \chi_m$ (cgs)</th>
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<tbody>
<tr>
<td>Li$^+$</td>
<td>1</td>
<td>F$^-$</td>
<td>9</td>
<td>H$_2$O</td>
<td>13</td>
</tr>
<tr>
<td>Na$^+$</td>
<td>7</td>
<td>Cl$^-$</td>
<td>23</td>
<td>NH$_3$</td>
<td>16</td>
</tr>
<tr>
<td>K$^+$</td>
<td>15</td>
<td>Br$^-$</td>
<td>34</td>
<td>en</td>
<td>47</td>
</tr>
<tr>
<td>Rb$^+$</td>
<td>22</td>
<td>I$^-$</td>
<td>50</td>
<td>pyridine</td>
<td>49</td>
</tr>
<tr>
<td>Cs$^+$</td>
<td>33</td>
<td>CH$_3$COO$^-$</td>
<td>29</td>
<td>(C$_6$H$_5$)P</td>
<td>167</td>
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<td>NH$_4^+$</td>
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<td>C$_3$H$_5$COO$^-$</td>
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<td>Mg$^{2+}$</td>
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<td>CN$^-$</td>
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<td>Sr$^{2+}$</td>
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<td>CNS$^-$</td>
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<td>Ba$^{2+}$</td>
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<td>ClO$_4^-$</td>
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<td>Cu$^+$</td>
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<td>CO$_3^{2-}$</td>
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<td>Ag$^+$</td>
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<td>C$_2$O$_4^{2-}$</td>
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<tr>
<td>Zn$^{2+}$</td>
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<td>HCOO$^-$</td>
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<td>Cd$^{2+}$</td>
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<td>NO$_3^-$</td>
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<td>Hg$^{2+}$</td>
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<td>O$_2^-$</td>
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<td>OH$^-$</td>
<td>11</td>
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<tr>
<td>Pb$^{2+}$</td>
<td>32</td>
<td>S$_2^-$</td>
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<tr>
<td>First row transition metals</td>
<td>13</td>
<td>SO$_4^{2-}$</td>
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</tr>
<tr>
<td></td>
<td></td>
<td>S$_2$O$_3^{2-}$</td>
<td>46</td>
<td></td>
<td></td>
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<tr>
<td></td>
<td></td>
<td>acac$^-$</td>
<td>55</td>
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</tr>
</tbody>
</table>

Notes:  
*acac = acetylacetonate*  
*en = ethylenediamine*
APPENDIX E

STANDARD SUBSTANCES FOR THE CALIBRATION OF MAGNETIC BALANCES


This is probably the best solid calibrant. It is easily prepared in a pure state, packs well and has a high volume susceptibility.

\[
\chi_g \text{ at } 20^0C = 16.44 \times 10^{-6} \text{ c.g.s.} \quad \chi_g \text{ at } T^0C = \frac{4981}{283 + T} \times 10^{-6} \text{ c.g.s.}
\]


A useful secondary standard.

\[
\chi_g \text{ at } 20^0C = 11.04 \times 10^{-6} \text{ c.g.s.} \quad \chi_g \text{ at } T^0C = \frac{2759}{230 + T} \times 10^{-6} \text{ c.g.s.}
\]

(NH$_4$)$_2$Fe(SO$_4$)$_2$.6H$_2$O

Readily available, but less satisfactory than the above two calibrants.

\[
\chi_g \text{ at } 20^0C = 32.3 \times 10^{-6} \text{ c.g.s.} \quad \chi_g \text{ at } T^0C = \frac{9500}{274 + T} \times 10^{-6} \text{ c.g.s.}
\]

CuSO$_4$.5H$_2$O

Readily available, but the susceptibility is not known accurately.

\[
\chi_g \text{ at } 20^0C = 6.0 \times 10^{-6} \text{ c.g.s.} \quad \chi_g \text{ at } 20^0C = \frac{1758}{273 + T} \times 10^{-6} \text{ c.g.s.}
\]

H$_2$O

The standard for diamagnetic liquids in water.

<table>
<thead>
<tr>
<th>$T^0C$</th>
<th>$10^6\chi_g$ (cgs)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10</td>
<td>-0.719</td>
</tr>
<tr>
<td>20</td>
<td>-0.720</td>
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<tr>
<td>30</td>
<td>-0.721</td>
</tr>
<tr>
<td>40</td>
<td>-0.722</td>
</tr>
</tbody>
</table>

The presence of dissolved atmospheric oxygen can for most purposes be ignored.
APPENDIX F

USEFUL REFERENCES


APPENDIX G

CALIBRATION AND SET UP INSTRUCTIONS

This Magnetic Susceptibility Balance (MSB) has been accurately calibrated at our factory. Transit may have altered the calibration. The MSB must be re-calibrated at its intended work place.

Once the MSB has been set up and calibrated, moving the unit may disturb the mechanism such that re-zeroing (Stage II) and/or re-calibration (Stage III) may be necessary.

Re-calibration may easily be carried out instructions are carefully followed.

CAUTION THE MECHANISM OF THIS INSTRUMENT MAY BE SERIOUSLY DAMAGED UNLESS EXTREME CARE IS TAKEN DURING RE-CALIBRATION.

Stage I Post Delivery Preparation

1. Remove the flat lid above the front panel by unscrewing the two screws at the back of the instrument. This exposes the inner tensioned balance strip supporting the magnets, which is held by a travel clamp, which must be removed. A sample guide tube is held on the inside of the top cover. The elements of the travel clamp are shown in Figure 3.

Figure 3
Removal of the travel clamp can be accomplished without touching the torsion wire or any component lying to the left of the brass knob.

2. Remove the Transport Clamp by first loosening the Nylon Locking Screw and then gently unscrewing the Brass Knob of the clamping tube. The lower Black Knurled Knob should be held in position from the right-hand side of the mechanism. As the screw comes free, remove the clamp tube by carefully pulling it up and out of the mechanism.

CAUTION The balance can be seriously damaged if this is not carefully done.

Remove the Upper Support block by sliding it out of the right-hand side of the mechanism. Check that the lower support block drops to its lowest position. Screw the Brass Knob Rod, the Upper Support Block and the Black Knurled Knob together and leave within the instrument for future use if the instrument is being moved in the future, and leave the Nylon Locking Screw in its position to hold the Sample Guide Tube.

3. Place the sample tube guide in the sample-holder bridge so that its collar rests on the bridge surface and fix it in place by tightening the Nylon Locking Screw. Figure 1 on page 6 shows the final configuration (although the Lower Support Block, which remains with the mechanism is not shown.)

Stage II Zeroing of Balance

4. On the front panel, gently turn the zero knob anti-clockwise to the end of its range then turn the knob back clockwise five times. This has centred the zero control.

5. Level the instrument by the circular spirit level on sample-holder bridge, using the two leveling screws at the back of the instrument case.

6. Plug in the electrical power lead and switch on power at the mains plug.

NOTE Generally only minor adjustments will be needed to complete instructions 7 to 9.

7. Coarse Setting

Set RANGE knob to x10 and adjust horizontal counterweight so that numerical display reads and steady value between 015 to -015.

8. Fine Setting

Set RANGE knob to x1. Adjust horizontal counterweight so that numerical display reads a steady value between 075 to -075

9. Carefully replace the lid and screw in place.
Stage III  Determination of Calibration Constant

10. Turn the zero knob until the numerical display shows zero 000. Insert the calibration sample into sample holder and allow reading in numerical display to settle.

11. Record reading and calculate calibration constant $C_{\text{Bal}}$ from the formula.

$$C_{\text{Bal}} = \frac{C_{\text{Tube}}}{(R - R_0)}$$

12. The calibration constant can be adjusted by turning a small potentiometer screw located on the back panel of the Mk 1, between the analogue outputs and power in. Turning the screw fully clockwise will result in the highest readings (lowest calibration constant) and vice versa.

Following an adjustment of the screw, the balance must be rezeroed without the sample, have the empty tube reading and the sample reading re-entered into the formula. This process must be repeated until the desired calibration constant is obtained.

CLAMP RE-FITTING

1. Loosen the Nylon Locking Screw and carefully remove the Sample Tube Guide. Clip into position on the underside of the lid.

2. Slide the Upper Clamp Block into position, above the balance arm, from the right-hand side. The chamfered corner to the front.

3. Carefully place the Brass Knob Rod through the upper clamp block, balance arm and lower clamp block. Then holding the Black Knurled Knob in position, turn the brass knob rod to tighten up the clamp. Tighten it firmly with your fingers, DO NOT USE ANY TOOLS.

4. Re-tighten the nylon clamping screw, finger tight plus a quarter turn.
APPENDIX H

TORSION WIRE REPLACEMENT PROCEDURE
For instruments upto serial number 15247

1. Remove the top cover.
2. Remove the four small bolts that clamp the beam to the torsion wire. Remove the top half of the clamp and leave the bottom in place.
3. Loosen the two bolts on both ends of the torsion wire. This will allow the removal of the damaged piece.
4. Place the new torsion wire in the two end clamps in line with the marks engraved on the clamps. Tighten the bolts on one end of the torsion wire.
5. Very carefully, using pliers, place tension across the wire and tighten the remaining two bolts.
6. Place the top of the clamp in place and snugly fit the four small bolts.
7. To check the tension of the torsion wire, make sure the bottom of the beam swings cleanly.
8. Turn the unit on to x10. Carefully center the clamp until a non-flashing reading is observed.
9. Turn the unit to x1. Adjust the counterweights to obtain a zero reading.
10. Replace the top cover.
11. The unit must now be calibrated. Make sure to note the new value of $C$.

For instruments from serial number 15248 on

Please return the instrument to Sherwood Scientific via your distributor.
Sherwood Scientific Limited
Product Warranty Statement

Warranty Term: 12 Months

Sherwood Scientific Ltd (Sherwood) warrants, subject to the conditions itemised within this document, through either Sherwood personnel or personnel of its authorised distributors, to repair or replace free of all charges, including labour, any part of this product which fails within the warranty time specified above, appertaining to this particular product. Such failure must have occurred because of a defect in material or workmanship and not have occurred as a result of operation of the product other than in accordance with procedures described in the instructions furnished with this product.

Conditions and specific exceptions that apply to the above statement are as follows:

1. End-user warranty time commences on the date of the delivery of product to end-user premises.

2. ‘Free of all charges’ statement applies only in areas recognised by Sherwood as being serviced either directly by its own personnel, or indirectly through personnel of an authorised distributor. Products purchased outside these areas requiring service during the warranty period will incur charges relative to the travel/transit costs involved. However, products purchased in such areas will be serviced during the warranty period free of all charges providing they are returned, carriage paid, to either Sherwood or by pre-arrangement to an authorised Sherwood distributor.

3. All maintenance (other than operator maintenance as described in the instructions), repairs or modifications have been made by Sherwood or Sherwood authorised personnel.

4. This product has where applicable been operated using Sherwood specified supplies and reagents.

5. Sherwood reserves the right to make any changes in the design or construction of future products of this type at any time, without incurring any obligation to make any changes whatsoever to this particular product.

6. Reagents, supplies, consumables, accessories and user maintenance items are not included in this warranty.
Product Warranty Statement (continued)

7. Repairs or replacement of any part failing due to abnormal conditions including the following, are excluded from this warranty:
   a) Flood, lightning, earthquake, tornado, hurricane, or any other natural or man-made disaster.
   b) Fire, bombing, armed conflict, malicious mischief or sprinkler damage.
   c) Physical abuse, misuse, sabotage or electrical surge.
   d) Damage incurred in moving the product to another location.

8. User agrees to permit Sherwood personnel or personnel of its authorised distributor to make changes in the product which do not affect results obtained, but do improve product reliability.

Representations and warranties purporting to be on behalf of Sherwood made by any person, including distributors and representatives of Sherwood, which are inconsistent or in conflict with the terms of this warranty (including but not limited to the limitations of the liability of Sherwood as set forth above), shall not be binding upon Sherwood unless reduced to writing and approved by an officer of Sherwood Scientific Ltd.

Except for the obligations specifically set forth in this warranty statement, in no event shall Sherwood be liable for any direct, indirect, special, incidental, or consequential damages, whether based on contract, tort or any other legal theory and whether advised of the possibility of such damages.

Neither Sherwood nor any of its third party suppliers makes any other warranty of any kind, whether expressed or implied, with respect to Sherwood Products.

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England