

MSB Mk1

MAGNETIC SUSCEPTIBILITY BALANCE

OPERATOR MANUAL

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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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Intended Use

This operator's manual contains complete instructions for setting up and using the Magnetic Susceptibility Mk1 balance.

The MSB Mk1 is intended for use by persons knowledgeable in safe laboratory practices. If the instrument is not used in accordance with these instructions for use, the protection provided by the equipment may be impaired.

With the exception of the simple adjustment of the Zero mechanism, there are no user serviceable parts within the instrument.

Sherwood Scientific Limited and its authorised Distributors and Agents consider themselves responsible for the effects of safety, reliability and performance of the MSB Mk1 only if:-

- assembly operation, extensions, re-adjustments, modifications or repairs are carried out by persons authorised by them.
- the electrical installation of the relevant room complies with IEC requirements or local regulatory code.
- the equipment is used in accordance with the instructions for use.

The information contained in this manual was correct at the time of going to print. However, Sherwood Scientific's policy is one of continuous product improvement and the right to change specifications, equipment and maintenance procedures at any time, without notice, is reserved.

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1 Introduction

The SHERWOOD SCIENTIFIC Magnetic Susceptibility Balance (MSB) 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.

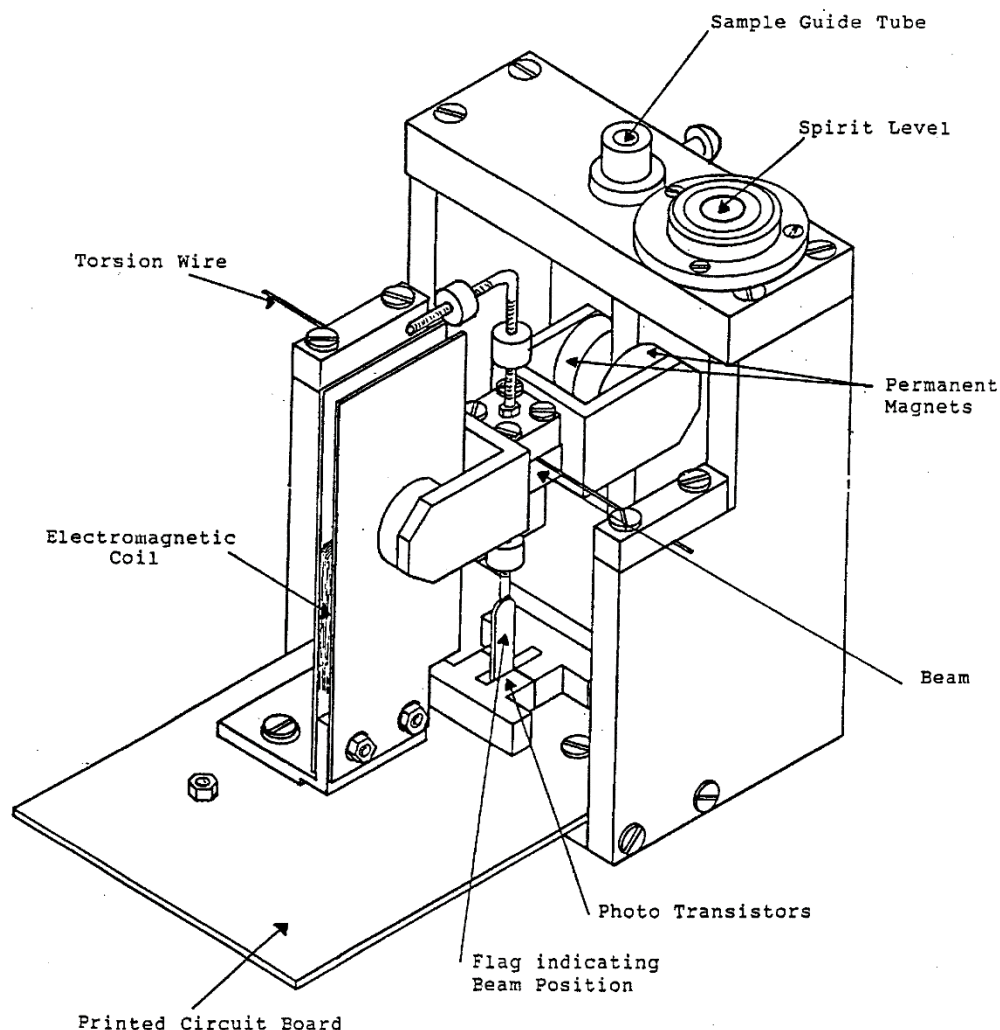


Figure 1

The MSB 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 a paramagnetic or diamagnetic 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.

1.1 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.

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

$$\chi_g = \frac{l}{m} (C * (R - R_0) + \chi_{var} * A)$$

Where:- C = a constant of proportionality.
 R = the reading obtained for tube plus sample.
 R_0 = the empty tube reading (normally a negative value).
 l = the sample length (in cm).
 m = the sample mass (in gm).
 A = the cross-sectional area of the tube (in cm²).
 χ_{var} = the volume susceptibility of the displaced air.

For powder samples the air correction term $\chi_{var} * A$, may normally be ignored.

C , the constant of proportionality is related to the calibration constant of a given balance by the formula.

$$C = \frac{C_{Bal}}{10^9}$$

The balance is configured during production such that C_{Bal} is equal to 1 (to determine C_{Bal} see Appendix B).

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.

Conversion of cgs Units to SI Units.

Volume susceptibility	χ_v	multiply cgs value by	4π
Mass susceptibility	χ_g	multiply cgs value by	$4\pi \times 10^{-3}$
Molar susceptibility	χ_m	multiply cgs value by	$4\pi \times 10^{-6}$

2 Installation

2.1 Unpacking

1. Unpack the instrument and accessories.
2. Check all items for damage.
3. Check all the items on the Accessory List have been delivered. Contact your Sherwood Scientific distributor if you have any problems.
4. The MSB Mk1 is shipped with the following items:

2.2 Accessory List

Cat. Number	Description	Quantity
001 53 313	PSU Uni. Multi-Plug Adapter, 12V DC	1
710 56 000	MnCl ₂ Sample Tube	1
710 86 002	Sample tube Ø4mm	2
710 66 701	Software CD	1
710 91 001	Operators manual	1

2.3 Services required

The MSB Mk1 is supplied with a Universal Power Supply accepting input voltages over the range 100 – 240V AC, 50/60Hz, with output to the instrument of 12V DC.

2.4 Specification

Size	unpacked 220mm x 280mm x 140mm (w x d x h) packed 340mm x 260mm x 450mm
Weight	unpacked 3kg (7 lbs) packed 4.5kg (9.9 lbs)
Measurement range	Approx. $\pm 2 \times 10^{-5}$ to $\pm 5 \times 10^{-8}$ arbitrary units
Amount of sample required for accurate determination of Magnetic Susceptibility	70µl (min) 300µl (max) Approx. 0.04g solid (depending upon density) Approx. 0.025g solution
Detection limits	Approx. 10x lower than above (depending upon Magnetic Susceptibility)
Outputs	Digital display Analogue output
Magnetic Field Strength	3.5 kGauss

3 Assembly

The balance is shipped with a Travel Clamp which protects its delicate mechanism during transit. The Travel Clamp must be removed before use and the Sample Guide Tube fitted, see Section 3.2.

If the balance is to be moved more than a few meters, re-install the Travel Clamp then move the balance, remove the Travel Clamp when the balance is in the new location. If the balance is put away for storage re-fit the Travel Clamp.

Depending on the frequency of use consider re-fitting the Travel Clamp after each use so that the balance is not damaged inadvertently by being moved by personnel who are unaware of the delicate nature of the mechanism.

3.1 Siting of the Balance

Before removing the Travel Clamp it is important to consider the siting of the balance. The balance is sensitive to external magnetic fields; many stray constant fields can be adjusted for, however stray variable fields such as those emitted from conventional magnetic susceptibility balances using an electromagnet, or an Electron Spin Resonance spectrometer cannot be adjusted for and will cause the balance readings to fluctuate.

Other sources of variable magnet fields include computers, mobile phones, as well as more benign-seeming moving metal objects such as doors opening and closing. If the balance is placed on a bench with drawers, ensure that ferromagnetic objects are not situated directly below.

If in doubt, once the balance is installed check whether the zero reading of the balance changes when the external magnetic field is varied. If the variation is too great, refit the Travel Clamp and try installing the balance in another location.

For best results, the balance should be placed on a level and stable surface free from vibration in an environment not subject to chemical fumes or rapid changes in temperature.

3.2 Removal of the Travel Clamp and fitting of the Sample Guide Tube

Once a suitable location of the balance has been selected (see above) the Travel Clamp can be removed and the Sample Guide Tube (710 86 010) fitted following the instructions below.

Keep all the Travel Clamp components safe for re-use.

CAUTION:-

THE MECHANISM OF THIS INSTRUMENT MAY BE SERIOUSLY DAMAGED UNLESS EXTREME CARE IS TAKEN WHILE THE CASE IS REMOVED.

DO NOT INTRODUCE ANY FERROMAGNETIC OBJECTS INTO THE BALANCE.

Removal of the Travel Clamp must be undertaken with great care and without touching any other internal components.

1. Remove the Top Cover from the instrument by removing the two screws at the back, see Figure 2. This exposes the inner Tensioned Balance Strip supporting the magnets, which is held by the Travel Clamp. A Sample Guide Tube is held in a clip on the underside of the Top Cover.



Figure 2

2. Use the Rear Feet to level the instrument, so that the bubble is in the centre of the level, see Figure 3.



Figure 3

3. Looking inside the instrument the individual parts of the Travel clamp can be identified in Figure 4.

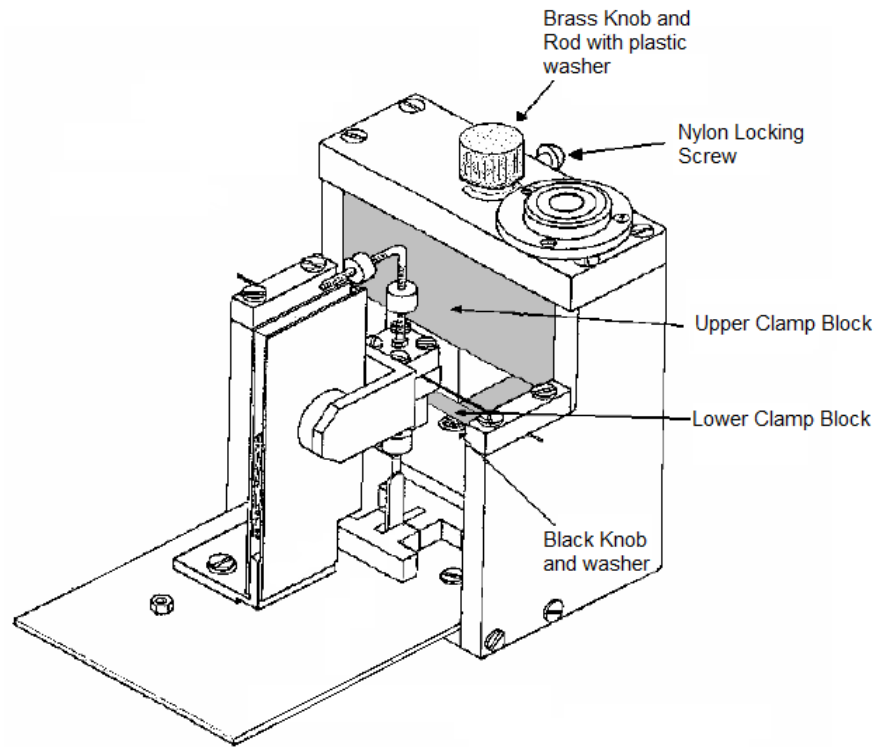


Figure 4

4. Remove the Black Knob from the bottom of the Clamp Rod, Figure 5, and place (with its copper washer) to one side. Loosen, but do not remove, the Nylon Locking screw. Slide the Brass Knob Rod out from the top of the Frame and put to one side. It is important to also remove the plastic washer that sat under the top of the Brass Knob and keep it with the rest of the Travel Clamp components. If the washer is not removed erroneous results will be generated.

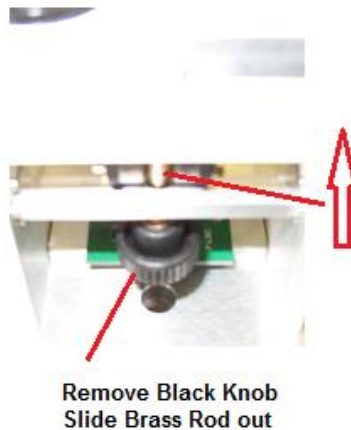


Figure 5

Assembly continued

- Slide the plastic (Upper Clamp) Block out of the right hand side of the Frame and put to one side, keep the Plastic Block, do not discard. Check that the Lower Clamp Block has dropped away from the Yoke, Figure 6.

NOTE: The Lower Clamp Block is not removed from the instrument.

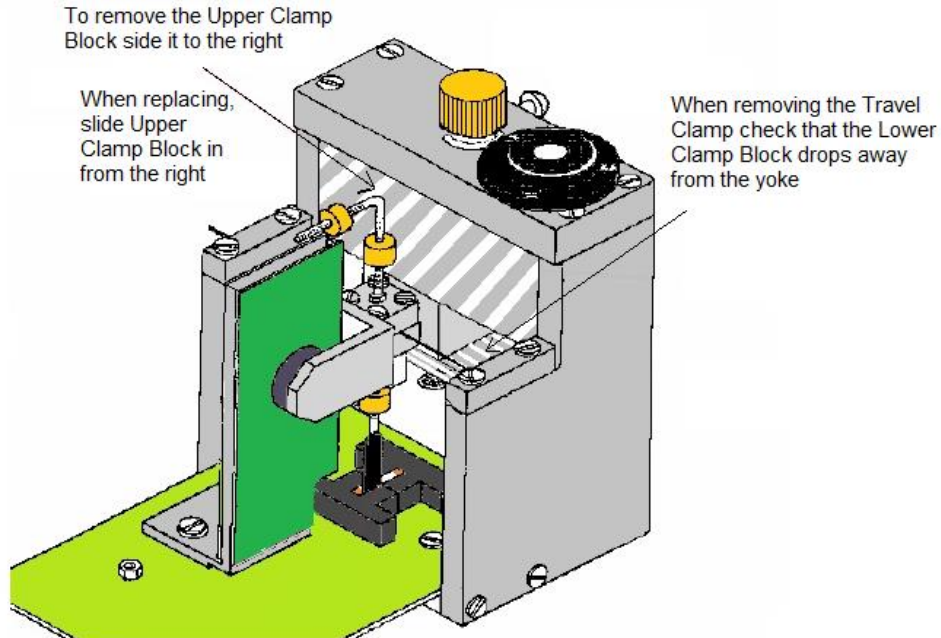


Figure 6

- Connect the power supply to the instrument. Remove the Sample Guide Tube (710 86 010) from the clip on the underside of the Top Cover and place it into the sample hole (where the Brass Rod came out from) so that its collar rests flush with the surface and fix it in place by tightening the Nylon Locking Screw, Figure 7.



Insert Sample Guide Tube into the frame and secure with the Nylon Locking Screw

NOTE: no washer on the Sample Guide Tube

Figure 7

7. The Travel Clamp components (apart from the Lower Clamp Block which remains in the instrument) have now been removed and the Sample Guide Tube fitted. Keep the Travel Clamp components safe for future use.

Sometimes removal of the Travel Clamp can affect the ability to set the display to read zero; and turning the Zero knob as far as it can go still doesn't achieve zero on the display. This is corrected by manually adjusting the position of a weight inside the instrument, see Section 3.4 Manually Adjusting the Zero.

3.3 Removal of Sample Guide Tube and refitting the Travel Clamp Components

If the balance is to be moved the Travel Clamp components **must** be re-fitted. If the balance is to be left un-used for some time it is also wise to re-fit the Travel Clamp. The elements of the Travel Clamp are shown in Figure 4.

1. Remove the Top Cover from the instrument by removing the two screws at the back, Figure 2.
2. Loosen the Nylon Locking Screw and carefully remove the Sample Guide Tube Assy (710 86 010). Store the Guide Tube in the clip on the underside of the balance Top Cover, Figure 7.
3. Approaching from the right with the chamfered corner to the front right, slide the Upper Clamp Block into position above the balance arm, Figure 6.
4. Thread the Brass Knob Rod through the plastic washer and carefully put the Brass Knob Rod into the hole the Sample Guide Tube came out from, making sure it passes through the Upper Clamp Block, Balance Arm and Lower Clamp Block. Then, while holding the Black Knurled Knob and its copper washer in place underneath, turn the Brass Knob Rod to tighten up the clamp. Tighten it firmly with your fingers, DO NOT USE ANY TOOLS.
5. Re-tighten the Nylon Locking Screw, finger tight plus a quarter turn.
6. Refit the Top Cover and secure with the two screws.
7. The mechanism is now secured and the balance can be moved without damage.

3.4 Manually Adjusting the Zero

Sometimes removal of the Travel Clamp can affect the ability to set the display to read zero; and turning the Zero knob as far as it can go still doesn't achieve zero on the display. This is corrected by manually adjusting the position of a weight inside the instrument. Follow instructions 1 & 2 (Section 3.3 Removal of the Travel Clamp) to remove the Top Cover and level the instrument, then with the instrument switched on and the empty Sample Guide Tube fitted:-

1. Turn the Zero Knob to the centre of its travel (5 complete turns from either end of travel), Figure 8.



Zero Knob 5 turns from either side

Figure 8

2. Switch the instrument to range x10, Figure 9.

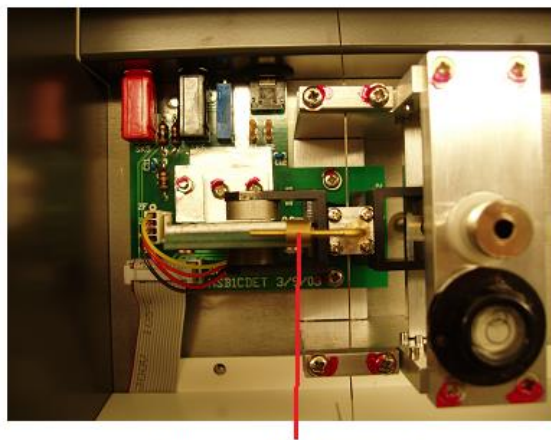


Figure 9

3. With the Balance Arm free to rock from side to side, very gently tilt the Arm to each end of travel, the display should change from -1 to 1. Release and allow the Balance Arm to settle and the display to stabilise.

4. Carefully adjust the counter weight (on the horizontal top arm shown in Figure 10) by screwing it along the thread so the display reads less than ± 40 .

Note:- Only small adjustment to the weight is necessary to give a large reading change.



Adjust this Weight, either left or right to obtain + or - 40 or less

Figure 10

5. When the display reads ± 40 or less, switch the Range knob to x1, then adjust the weight again to read as close to zero as possible. **This is a very fine adjustment so only move the weight by a very small amount each a time.**
6. Allow the reading to become stable, refit the Top Cover and screws.

The Zero knob should now be able to achieve a reading of 000.

3.5 Checking the Balance Calibration

The Mk1 is set up during production such that by use of the sample mass and length a sample in a **standard internal diameter tube** the reading, R, minus the empty tube's reading, R_0 is related to the Magnetic Susceptibility by the equation:-

The mass susceptibility, χ_g , in cgs

$$\chi_g = \frac{\text{Length of tube filled in cm}}{\text{Mass of sample in tube in g}} * C * (R - R_0)$$

Where C is a constant of proportionality:-

$$C = \frac{C_{Bal}}{10^9}$$

C_{Bal} is the Balance Constant which is set at the factory during production to 1.

NOTE:- If a *non-standard internal diameter* tube is used then an additional calculation must be made to account for the non-standard tube diameter (see Section 4.4 Working with Tubes with Non Standard Internal Diameters).

3.6 Checking the Calibration with the Sealed Sample of MnCl_2 Solution

Each instrument is accompanied with a sealed sample of Manganese Chloride solution in a standard sample tube. The tube is labeled with the reading it gave when measured in a calibrated instrument using a tared standard sample tube (C_{std}), the temperature the measurement was made at ($T^\circ\text{C}$) and the reading the tube gave when it was empty (R_0). The tube can be used easily to check the instrument's calibration (if the ambient temperature is greater than that on the tube's label then the reading will be slightly lower and vice versa, Appendix C shows examples of how a typical sealed Manganese Chloride solution tube reading changed with temperature).

Avoid inadvertently warming the sample when handling the tube; hold it at the very top or by the label.

1. Ensure the balance is level (Figure 3). Switch on and allow the balance to warm-up for ten minutes.
2. Turn the Zero knob until the display shows zero.
3. Set the Range knob to 'x1'
4. Insert a clean empty standard tube and adjust the reading to zero with the Zero knob.
5. Check that all the solution is at the bottom of the sealed Manganese Chloride solution tube; whilst holding just the top of the tube force the solution down the tube with a flick of the wrist.
6. Insert the sealed sample of MnCl_2 solution tube and allow the reading in the display to settle, the reading displayed is $R-R_0$.
7. The $R-R_0$ reading should be the same as that shown on the tube's label (C_{std}) (after making allowance for temperature).

If the $R-R_0$ reading differs significantly, then the calibration has drifted, or occasionally it can be due to the buildup of particles on the magnets (refer to Section 5 Troubleshooting). Refer to Section 3.7 Adjusting to the Factory Calibration Setting.

3.7 Adjusting to the Factory Calibration Setting

If the $R-R_0$ reading for the sealed sample of MnCl_2 solution differs significantly (after accounting for the temperature) from that shown on the tube's label (C_{std}) and there are no particles on the magnets (refer to Section 5 Troubleshooting) then the calibration has drifted. To reset the factory calibration:-

The calibration 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.

Avoid inadvertently warming the sample when handling the tube; hold it at the very top or by the label.

1. Follow instructions 1 to 6 in Section 3.6 Checking the Calibration with a Sealed Sample of MnCl_2 Solution.
2. While the sealed sample of MnCl_2 solution is in the balance adjust the display by turning the small potentiometer screw located on the back panel to achieve the reading on the label. Turning the screw clockwise will result in higher readings.
3. Following an adjustment of the potentiometer screw, the balance must be re-zeroed with the Zero knob and the empty tube and the sealed sample of MnCl_2 solution re-measured.
4. Steps 2 and 3 must be repeated until the empty tube reads zero and the sealed sample of MnCl_2 solution reads the C_{std} number on its label.
5. When the reading on the display reads the same as the label and the empty tube reads zero, then the calibration has been successfully adjusted to the production setting and C_{Bal} is 1.

3.8 Adjusting the Calibration to a Standard Substance

If required, the calibration can be adjusted using a standard substance. If the balance is to be used mainly for solid samples, then a solid calibrant (preferably $\text{HgCo}(\text{SCN})_4$) is recommended since some of the systematic errors in packing may cancel.

Details of standard substances for calibration of magnetic balances are given in Appendix D. Follow the procedure outlined in Section 3.7 Adjusting to the Factory Calibration Setting, but using the chosen calibrant and its volume susceptibility reading.

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

$$\frac{1}{T + 255}$$

4 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.

Once installed in a suitable location and the calibration checked the balance is ready to begin measurement. Always allow the instrument ten minutes after turning on to warm up before use.

4.1 Packing the Sample Tube

With solid samples, considerable care should be taken in packing the powder into the sample tube since the major error 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 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 particles. 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. The minimum value of l is 1.5cm, above which the reading is usually not affected. If only a small amount of sample is available then a narrow bore tube can be used to ensure this length (see Section 4.4 Working with Tubes with Non-Standard Internal Diameters).

Uniform 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.

Depending on the accuracy required, after the first measurement, it may be advisable to empty out the sample, repack the tube and repeat the procedure several times, to ensure the measurement is reproducible. If re-zeroing with the original tube then considerable care should be taken to ensure all the sample is removed from the tube to avoid generating a false zero reading (R_0).

4.2 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.
3. Place an empty sample tube of known weight (g) into the tube guide and take the reading, R_0 .

It is wise to note the weight (g) and empty reading (R_0) of each tube at both range settings. To determine R_0 at Range x10, simply remove the tube, turn the Range Knob to x10, use the Zero Knob to set the reading to zero then insert the empty tube and record R_0 for Range x10. Remember to switch back to Range x1 and re set zero.

4. Pack the sample as suggested in Section 4.1 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 (shows 1 or -1 on the far left of the display) turn the RANGE knob to the x10 scale. The instrument must now be re-zeroed with the Zero knob.

NOTE:- Both R and R_0 Readings taken on the 'x10' range must be multiplied by 10 to give the actual reading.

The dimensionless quantity volume susceptibility, χ_v , is calculated using:-

$$\chi_v = \frac{(R - R_0) * C}{\text{area}}$$

Where C is a constant of proportionality:-

$$C = \frac{C_{Bal}}{10^9}$$

C_{Bal} is the Balance Constant which is set during production to 1.

And **area** is the cross sectional area of the tube in cm^2 (for standard tubes the cross sectional area is $0.0824\text{cm}^2 \pm 0.00066\text{cm}^2$).

When using standard tubes the Mass Susceptibility, χ_g in cgs, is calculated using:-

$$\chi_g = \frac{\text{Length of tube filled in cm}}{\text{Mass of sample in tube in g}} * C * (R - R_0)$$

For those not wanting to manually calculate Mass and Molar Susceptibility, a CD accompanies each instrument containing software which will perform calculations.

With the Sherwood 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 if sample susceptibility values are very low. Since glass is diamagnetic R_0 will be negative.

Some samples readings vary with ambient temperature and should be allowed to come to temperature equilibrium with the balance before measurement; the temperature of the balance room should be recorded with the reading.

Operating Guidelines continued

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 when the tube containing the sample is introduced.

4.2.1 Worked example

Taking $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ as an example:-

A standard tube filled to a length of 3.8cm with 0.3431g of $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$. The instrument was set to Range $\times 10$.

An empty standard tube was measured and gave a reading of 4.

The tube filled with $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ read 587.

The Mass Susceptibility therefore is:-

$$\chi_g = \frac{3.8 \text{ cm}}{0.3431 \text{ g}} * 1 \times 10^{-9} * (5870 - (-40))$$

Thus,

$$\chi_g = 65.5 \times 10^{-6} \text{ (cgs)}$$

The literature value for Mass Susceptibility for $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$ is 65.2×10^{-6} (cgs).

Appendix E gives a selection of experimental results.

4.3 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 χ_s of the solution is:-

$$\chi_s = \frac{C_{Bal} * (R - R_0)}{10^9 * A * d_s} + \frac{\chi_{V_{air}}}{d_s}$$

(terms are as defined in Section 1.1 Definition of Magnetic Susceptibility).

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 χ_s = the mass susceptibility of the solution.

χ_g = the mass susceptibility of the solute.

χ_0 = the mass susceptibility of the solvent.

χ_0 is found by a separate measurement.

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

4.4 Working with Tubes with Non Standard 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. The balance's calibration accounts for the standard tube internal diameter.

The standard tube's dimensions:-

External diameter	0.400 ±0.0013cm
Internal diameter	0.324 ±0.0013cm
Cross Sectional Area	0.08245 ±0.00066cm ²

Refer to Appendix F for the Method to Determine the Internal Diameter of MSB Tubes.

If non-standard tubes are used with narrower internal diameters the volume of sample in the measuring region will be smaller. 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, see Section 3.8 Adjusting the Calibration to a Standard Substance.

OR

2. Calculate the Volume Susceptibility (X_v) using:-

$$X_v = \frac{C \cdot (R - R_0)}{\text{area}}$$

Where C is a constant of proportionality:-

$$C = \frac{C_{Bal}}{10^9}$$

C_{Bal} is the Balance Constant which is set during production to 1.

And then calculate Mass Susceptibility (X_g) using:-

$$X_g = \frac{X_v}{\text{Density}}$$

Where:- density = $\frac{\text{mass of the sample}}{\text{Length} \cdot \text{area}}$

Length is the length of the sample in cm and **area** is the cross sectional area of the tube in cm².

4.4.1 Worked example

Using a narrow bore tube of inside diameter 0.1cm filled to a length of 2.3cm with 0.025g of $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$.

R_0 measured -91

R measured 700

$R - R_0$ 791

Firstly calculate the Volume Susceptibility (X_v) using:-

$$X_v = \frac{C \cdot (R - R_0)}{\text{area}}$$

$$\text{Area of a circle} = \pi \cdot r^2 \quad r = \frac{0.1\text{cm}}{2}$$

$$X_v = \frac{1 \times 10^{-9} \cdot (700 - (-91))}{\pi \cdot r^2} = 5.036 \times 10^{-6}$$

Then calculate Mass Susceptibility (X_g) using:-

$$X_g = \frac{X_v}{\text{Density}} = \frac{5.036 \times 10^{-6}}{(0.025\text{g}/2.3\text{cm} \cdot 0.157\text{cm}^2)} = 7.28 \times 10^{-5}$$

Converting χ_g to Molar Susceptibility by multiplying by the molecular mass:-

$$\chi_g \cdot \text{Molecular Mass} = \text{Molar Susceptibility}$$

$$7.28 \times 10^{-5} \text{cgs} \cdot 197.7 = 1.44 \times 10^{-2} \text{cgs}$$

The literature value of Molar Susceptibility for $\text{MnCl}_2 \cdot 4\text{H}_2\text{O}$ is $1.46 \times 10^{-2} \text{cgs}$.

5 Troubleshooting

5.1 Removal of Particles from the Magnets

The Sample Guide Tube is designed such that anything that drops into the sample hole falls straight out of the bottom of the instrument to protect the delicate balance mechanism and makes it easy to keep the instrument clean. However it is possible for ferromagnetic debris to find its way through the hole in the bottom of the case and stick to the magnets and thereby affect the instrument's readings.

If the calibration of the balance has drifted or the results obtained are not as expected it may be that particles have collected on the magnets inside the instrument. Check if particles have collected by:-

1. Turn off the instrument and remove the Top Cover by unscrewing the two screws at the rear.
2. Inspect the permanent magnets for debris which may have become attached, (Figure 1).
3. Very carefully apply some **tacky** tape (do not use regular Sellotape or similar very sticky tape; the tape needs to be 'peel-able') to the surfaces of the magnets where particles have collected and very gently peel it away. Repeat with fresh tape until all particles have been removed.
4. Refit the Top Cover and secure with the two screws.
5. Check the balance calibration with the sealed sample of MnCl_2 before making further measurements.

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 11.

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: -

$$F = \frac{1}{2} * \chi_v * A * H^2 \quad (i)$$

Where: χ_v = the volume susceptibility of the sample.

A = the sample cross sectional area.

$$F = g * \delta(m)$$

$$\chi_g = \chi_v / d$$

$$A = m / l * d$$

$\delta(m)$ = 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,

$$\chi_g = \frac{2 * g * l * \delta(m)}{H^2 * m} \quad (ii)$$

If H is kept constant, this can be written as: -

$$\chi_g = \frac{C * \delta(m) * l}{m} \quad (iii)$$

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 - $\chi_{\text{Vair}} = 0.029 \times 10^{-6}$ cgs. at 20°C and 760mm 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: -

$$F = \frac{1}{2} * (\chi - 0.029 * 10^{-6}) * A * H^2 \quad (iv)$$

Leading to the corrected expression: -

$$\chi_g = \frac{0.029 * 10^{-6} * V + C * \delta(m) * l}{m} \quad (v)$$

Where: V = volume of the sample in cm³.

For moderately paramagnetic materials, the difference between the values of χ_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.

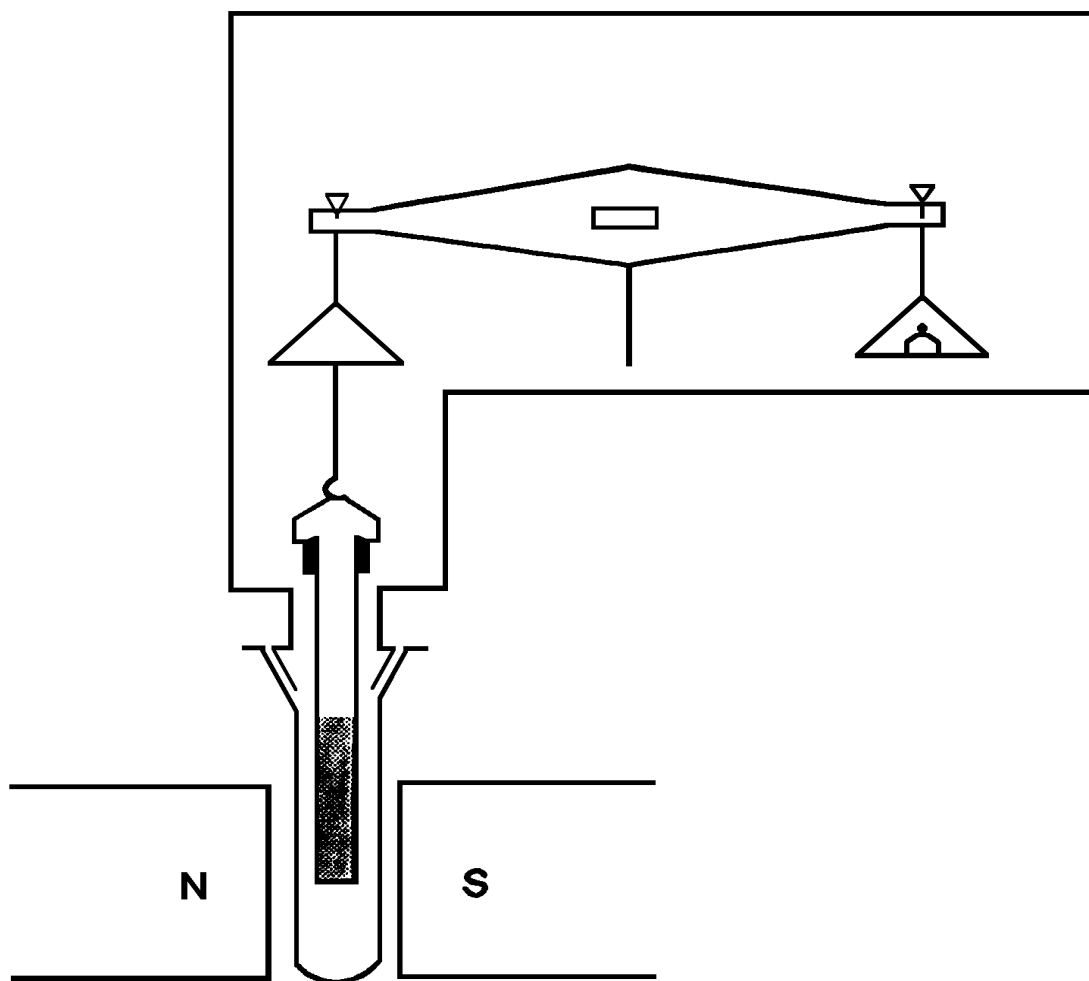


Figure 11

Appendix B

Determining the C_{Bal} for the Balance

C_{Bal} for the balance has been set during production to 1. Sometimes setting C_{Bal} to a different number may be desirable. To determine the C_{Bal} follow the procedure below:-

1. Ensure the balance is level (Figure 3). Switch on and allow the balance to warm-up for ten minutes.
2. Turn the Zero knob until the display shows zero.
3. Set the Range knob to 'x1'.
4. Insert a clean empty standard tube and adjust the reading to zero with the Zero knob.
5. Insert the sealed sample of MnCl_2 solution that accompanied the balance and allow reading in the display to settle, the reading displayed is $R - R_0$.

$$C_{\text{Bal}} = \frac{R - R_0 \text{ from sealed sample of } \text{MnCl}_2 \text{ solution label}}{R - R_0 \text{ from display}}$$

Appendix C

Temperature Effects on Manganese Chloride Solution

The effect of temperature on the readings of a typical sealed MnCl_2 sample tube have been observed between 16 and 24°C.

Ambient Temperature $T^{\circ}\text{C}$	Mean Reading ($R-R_0$)
16	1217
17	1211
18	1204
19	1197
20	1192
21	1185
22	1178
23	1172
24	1165

APPENDIX D

Standard Substances for the Calibration of Magnetic Balances

HgCo(SCN)₄ (Figgis and Nyholm, J. Chem. Soc., 1958, 4190).

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

$$\chi_g \text{ at } 20^{\circ}\text{C} = 16.44 \times 10^{-6} \text{ c.g.s.}$$

$$\chi_g \text{ at } T^{\circ}\text{C} = \frac{4981}{283 + T} \times 10^{-6} \text{ c.g.s.}$$

Ni(en)₃S₂O₃ (Curtis, J. Chem. Soc., 1961, 3147).

A useful secondary standard.

$$\chi_g \text{ at } 20^{\circ}\text{C} = 11.04 \times 10^{-6} \text{ c.g.s.}$$

$$\chi_g \text{ at } T^{\circ}\text{C} = \frac{2759}{230 + T} \times 10^{-6} \text{ c.g.s.}$$

(NH₄)₂Fe(SO₄)₂·6H₂O

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

$$\chi_g \text{ at } 20^{\circ}\text{C} = 32.3 \times 10^{-6} \text{ c.g.s.}$$

$$\chi_g \text{ at } T^{\circ}\text{C} = \frac{9500}{274 + T} \times 10^{-6} \text{ c.g.s.}$$

CuSO₄·5H₂O

Readily available, but the susceptibility is not known accurately.

$$\chi_g \text{ at } 20^{\circ}\text{C} = 6.0 \times 10^{-6} \text{ c.g.s.}$$

$$\chi_g \text{ at } 20^{\circ}\text{C} = \frac{1758}{273 + T} \times 10^{-6} \text{ c.g.s.}$$

H₂O

The standard for diamagnetic liquids in water.

$T^{\circ}\text{C}$	$10^6 \chi_g \text{ (cgs)}$
10	-0.719
20	-0.720
30	-0.721
40	-0.722

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

APPENDIX E

Experimental Results

Compound	Sample Mass m (gm)	Typical Experimental Values				Literature Value
		l (cm)	R	$10^6 \chi_g$	Error	$10^6 \chi_g$
MnSO ₄ ·4H ₂ O	0.3431	3.8	2856	+66.38	+1.8%	+65.20
CoCl ₂ ·6H ₂ O	0.1743	2.5	1344	+40.72	-0.2%	+40.81
Hg[Co(SCN) ₄]	0.4106	3.8	830	+16.72	+1.7%	+16.44
CuSO ₄ ·5H ₂ O	0.2018	1.7	318	+5.89	-1.9%	+6.00
Empty tube	-	-	-17	-	-	-
H ₂ O (liq)	0.2950	3.6	-45	-0.713	+1.0%	-0.720

Notes:

Values of χ_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 Section 3.7 Adjusting to the Factory Calibration Setting.

APPENDIX F

Method to Determine the Internal Diameter of MSB Tubes

Introduction

Determining the internal dimensions of MSB tubes before use enables more accurate measurements to be made and is important when comparing samples measured in different tube sizes.

Equipment

Accurate balance (0.0005g)

Rule

Deionised water

Fine capillary (with a narrower external diameter than the tubes internal diameter)

Method

Accurately weigh the tube and record the mass. Carefully fill the tube, using a fine capillary if necessary, with deionised water making sure all air bubbles are eliminated and that no drops of water are stuck to the inside above the meniscus. Be sure *not* to fill the tube beyond the vertical limits, ie do not fill to the part which begins to widen towards the neck of the tube, Figure 12.

Measure the length of the water in the tube from the top of the base to the bottom of the meniscus, Figure 12.

Wipe the tube with a soft lint free tissue to remove any moisture on the outside and record the new accurate mass.

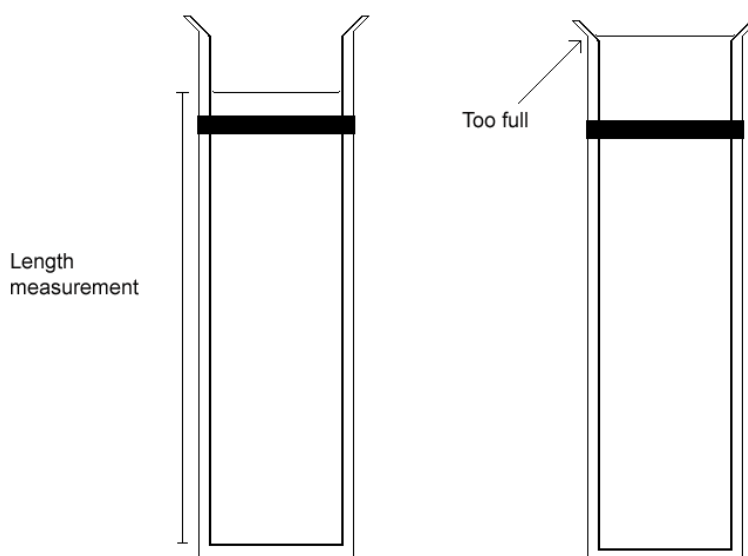


Figure 12

Calculation

Internal volume of the tube is equal to the cross sectional area multiplied by the length. Using water at standard temperature and pressure allows the volume, area and internal diameter to be calculated using the mass and length according to the worked example below.

Worked Example

A 0.1cm nominal internal diameter tube weighed 2.0862g empty and with 7.5cm depth of water added weighed 2.1572g. Therefore the mass of water was 0.0710g.

The mass of the water occupied by this volume is 0.0710cm^3 , as at standard temperature and pressure water has a density of 1g/cm^3 .

As the volume of a cylinder is:-

$$\text{Volume} = \text{Cross sectional area} \times \text{Length}$$

and

$$\text{Area} = r^2 \times \pi$$

so

$$\text{Volume} = r^2 \times \pi \times \text{length}$$

Rearrange, giving:

$$r^2 \text{ cm}^2 = \frac{\text{Volume cm}^3}{\text{Length cm} \times \pi}$$

Enter the values...

$$r^2 \text{ cm}^2 = \frac{0.0710 \text{ cm}^3}{7.5\text{cm} \times 3.14}$$

$$r^2 \text{ cm}^2 = 0.00301 \text{ cm}^2$$

$$r = 0.055\text{cm}$$

Since the diameter of a circle is 2 x radius, therefore the internal diameter is calculated as: -

$$\text{ID} = 2 \times 0.055$$

$$\text{ID} = 0.11\text{cm}$$

Appendix G

Uses for the Sherwood Scientific MSB.

1. Assignment the oxidation state of the metal in complexes of the transition, lanthanide and actinide elements. Examples are given in Appendix H, table 3.
2. Stereochemical information - for example square planar Ni^{2+} complexes are diamagnetic, while octahedral 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 neighbouring 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 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 Ni^{2+} complex.
6. Criterion of purity - pure Y_2O_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 H

Basic Magnetochemistry

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

<u>Behaviour</u>	<u>Properties</u>	<u>Typical Examples</u>
Ferromagnetism	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.	Fe, Co, Ni and many of their alloys. Some other alloys - eg. Pt/Bi, ferrites
Paramagnetism	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).	Compounds with unpaired electrons eg many transition metal, lanthanide & actinide complexes. Also free radicals, O ₂ , NO.
Diamagnetism	Sample very weakly repelled from the strongest part of an inhomogeneous magnetic field. Susceptibility normally temperature-independent.	Always present, but outweighed by any paramagnetism or ferromagnetism

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 χ_v . In most cases, a more useful quantity is the magnetic susceptibility per unit mass or mass susceptibility, χ_g , equal to χ_v/d , where d is the density of the substance in gm/cm³. It is conventional to regard χ_v as dimensionless and χ_g as having the dimensions of reciprocal density.

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

N.B. For diamagnetic compounds χ is negative and for paramagnetic compounds χ is positive.

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

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

$$(\mu_{\text{eff}})^2 = \frac{3kT\chi_A}{N\beta^2}$$

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. μ_{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: -

$$\mu_{\text{eff}} = \sqrt{n(n+2)} \quad \text{BM}$$

Where: n = the number of unpaired electrons.

Values of μ_{eff} as a function of n are given below.

n	μ_{eff} (BM)
1	1.73
2	2.83
3	3.87
4	4.90
5	5.92
6	6.93

Diamagnetic Corrections for Ions and Molecules

Cations	$10^6 \chi_m$ (cgs)	Anions	$10^6 \chi_m$ (cgs)	Molecules	$10^6 \chi_m$ (cgs)
Li ⁺	1	F ⁻	9	H ₂ O	13
Na ⁺	7	Cl ⁻	23	NH ₃	16
K ⁺	15	Br ⁻	34	en	47
Rb ⁺	22	I ⁻	50	pyridine	49
Cs ⁺	33	CH ₃ COO ⁻	29	(C ₆ H ₅) ₃ P	167
NH ₄ ⁺	13	C ₆ H ₅ COO ⁻	71		
Mg ²⁺	4	CN ⁻	13		
Ca ²⁺	9	CNO ⁻	23		
Sr ²⁺	16	CNS ⁻	34		
Ba ²⁺	26	ClO ₄ ⁻	32		
Cu ⁺	15	CO ₃ ²⁻	28		
Ag ⁺	27	C ₂ O ₄ ²⁻	28		
Zn ²⁺	13	HCOO ⁻	17		
Cd ²⁺	20	NO ₃ ⁻	19		
Hg ²⁺	36	O ²⁻	6		
Tl ⁺	36	OH ⁻	11		
Pb ²⁺	32	S ²⁻	28		
First row transition metals	13	SO ₄ ²⁻	38		
		S ₂ O ₃ ²⁻	46		
		acac ⁻	55		

Notes: acac = acetylacetonate
 en = ethylenediamine

Examples of Oxidation States and Magnetic Moments

Metal and oxidation state	Configuration	Number of unpaired electrons	Magnetic moments μ_{eff} (BM)
Ti ³⁺	d ¹	1	1.7 – 1.8
Ti ⁴⁺	d ⁰	0	0
V ³⁺	d ²	2	2.7 – 2.9
V ⁴⁺	d ¹	1	1.7 – 1.8
V ⁵⁺	d ⁰	0	0
Cr ²⁺	d ⁴	4 (2)	4.8 – 5.0 (3.0 – 3.3)
Cr ³⁺	d ³	3	3.7 – 3.9
Mn ²⁺	d ⁵	5 (1)	5.7 – 6.0 (ca. 2.0)
Mn ³⁺	d ⁴	4 (2)	4.8 – 5.0 (3.0 – 3.3)
Mn ⁴⁺	d ³	3	3.7 – 3.9
Fe ²⁺	d ⁶	4 (0)	5.0 – 5.6 (0)
Fe ³⁺	d ⁵	5 (1)	5.7 – 6.0 (2.2 – 2.5)
Co ²⁺	d ⁷	3 (1)	4.3 – 5.2 (2.0 – 2.7)
Co ³⁺	d ⁶	0	0*
Cu ¹⁺	d ¹⁰	0	0
Cu ²⁺	d ⁹	1	1.8 – 2.1
Ag ¹⁺	d ¹⁰	0	0
Ag ²⁺	d ⁹	1	1.7 – 2.1
Ru ²⁺	d ⁶	0	0
Ru ³⁺	d ⁵	1	1.8 – 2.1
Eu ²⁺	f ⁷	7	7.8 – 8.0
Eu ³⁺	f ⁶	6	3.4 – 3.6
U ⁴⁺	f ²	2	3.4 – 3.6
U ⁶⁺	f ⁰	0	0

Notes: * Small Temperature-Independent Paramagnetism. Numbers in parentheses represent less common situations.

APPENDIX I

Useful References

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8. Landolt - Bornstein, Book II, Part 10, Magnetic Properties II. Springer - Verlag, Berlin, 1967. A critical compilation of diamagnetic susceptibilities. Useful for calculating diamagnetic corrections.
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APPENDIX J

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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