MSB - AUTO

MAGNETIC SUSCEPTIBILITY BALANCE

OPERATOR'S MANUAL

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700 91 001 Issue 7 3rd June 2009 ECN 566
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LIST OF ILLUSTRATION

The information in this manual applies to all instruments sold from July 1992 and onwards.

WARNING!

The external magnetic force exceeds 10 Gauss and may effect the operation of pacemakers and devices and instruments sensitive to a strong magnetic field.

CAUTION!

Please refer to the Operating Guidelines in Section 1.5 before unpacking and using the Auto. Special care must be taken in positioning the Auto in order to obtain good results.

Section 1.0 General Information

The MSB-Auto Manual is organised to enable the user to do the following:

- Become familiar with the instrument options, accessories and use.
- Understand the property of magnetic susceptibility and the advantage of its measurement.
- Learn to safely unpack and pack the instrument and to follow the optimal setting up procedures, allowing the user to take full advantage of the portability of the instrument.
- Select a location for operating the unit. This is a basic requirement for obtaining good results, particularly when operating at high sensitivities.
- Operate the MSB Auto, taking full advantage of its advanced features such as auto- zeroing, taring, and auto-calculations.
- Use the balance with external peripherals, such as a computer or recorder.
- Apply the instrument to the measurement of volume and mass susceptibility, throughout a wide range of measurement.
- Extend the use of the instrument to quantitative analysis.
- Apply the instrument to quality and process control monitoring.
- Apply the instrument to experimental problems and research studies.
- Finally, diagnose faults.

A reference section citing a few highly recommended publications is appended in order to allow the user to continue to expand their knowledge of magneto-chemistry.

1.1 Introduction

For many years we have produced a simple laboratory magnetic susceptibility balance, the Mk 1, which has been bought by teaching laboratories throughout the world. We have collected our user’s comments and recommendations and incorporated these ideas into the design of the Auto.

The MSB-AUTO is our top of the range instrument offering enhanced sensitivity and improved versatility. It is more user friendly, reducing the number of operator manipulations required and making magnetic susceptibility measurements easier than ever, but the instrument still remains at an affordable price.

Solids, liquids, or gaseous samples can be measured using the range of accessories supplied with each instrument. This is intended to encourage the user to venture into new application areas, outside of the specific need, which helped to justify the purchase. We feel that there are many new applications out there that will reward the investigator.

Please keep us informed, - we may even be able to help. Since the introduction of the AUTO, we have been collating new applications. Some of these are cited in later sections. We intend to up-date our user’s awareness by circulating information sheets on the more interesting studies. The realisation of this good intention depends on you keeping us informed.

Traditionally, measurement of magnetic susceptibility has been made using the Gouy method. The original instruments were originally made from conventional laboratory balances and large permanent magnets. The magnets were brought towards the sample and the positive or negative change of apparent weight was noted. The balances and sample holder were free of ferromagnetic materials. The systems that evolved were very large relying on heavy magnets, fixed in position, and a moving sample. Neither their size nor their cost fit the current requirements of most research and teaching laboratories.

The late Professor Evans of Imperial College London introduced an innovative step to the measurement. Instead of measuring the apparent weight loss or gain of a sample, the force acting upon a suspended magnet was detected, thereby turning the method on its head. The advantage realised by this step led to the development of the first light and inexpensive magnetic susceptibility balance, which we still offer as the MSB-MKI. The MSB-AUTO is a new state of the art instrument with many improved features, which still relies on the advantage first obtained by Professor Evans.

1.2 Instrument Description

The MSB-Auto incorporates state of the art technology and takes magnetic susceptibility measurements to new high levels of sensitivity. It is particularly aimed at meeting the needs of the research laboratory and industrial quality control. When applicable to an analytical problem, the Auto provides a most simple solution.

The instrument is portable, quick to install, and easy to use, requiring a minimum of sample pre treatment.

The Auto arrives to your lab in a carrying case slightly larger than a portable computer. Keep the case, since it is ideal for safe storage as well as protecting the instrument while transporting it to different locations. The Auto comes fully operational, equipped with sample tubes, batteries, and all necessary accessories. The main components and accessories of the Auto are shown in the illustrations Figures 1A and 1B and 2. The objects shown in Figure 1A and 1B include:

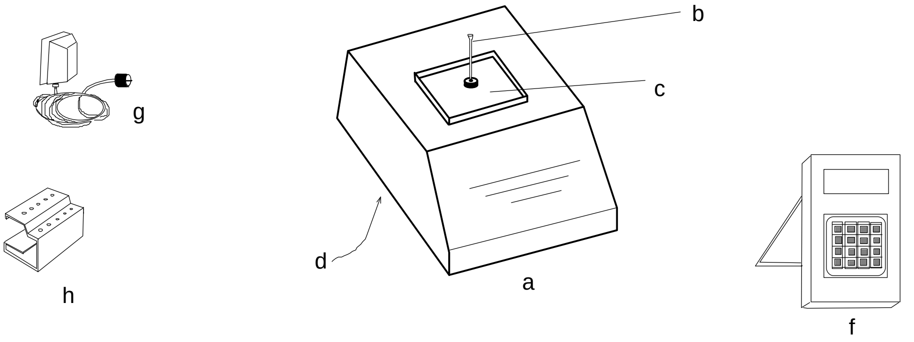


Figure 1A The Auto

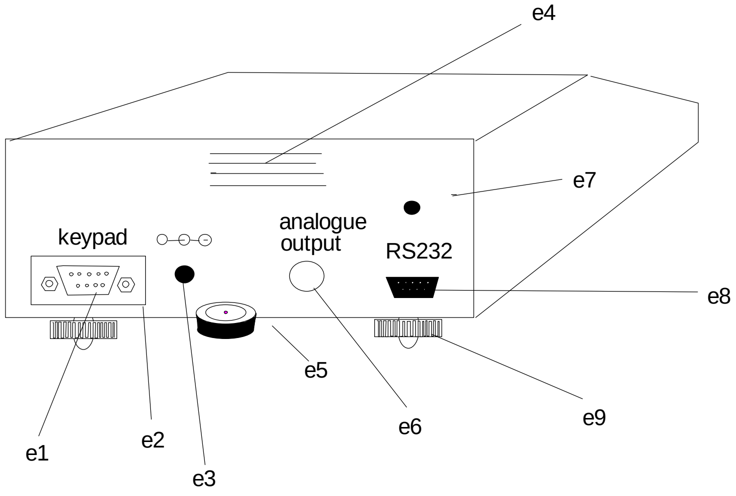


Figure 1B The Back Panel

1.2 Instrument Description continued

a = The sensor housing.

This contains the detector to which the samples are introduced through a sample holder on the top of the instrument.

Note the following positions:

b = A sample tube fitted into a tube adapter which positions the sample within the magnetic field of the detector (within the instrument).
c = The sample tube is surrounded by a removable stainless steel catch tray.
d = The travel clamp is located under the catch tray which can be removed by pulling off the sample tube adaptor and sliding the catch tray towards the back the hole next to the sample introduction port. Turning this screw fully counter-clockwise releases the travel clamp for measurement. Turning the screw clockwise applies the clamp.

Note the following positions on rear panel as illustrated in Figure B:

e1	keypad inlet
e2	serial number location
e3	input from the 9 V dc power supply
e4	address of local supplier
e5	bubble level
e6	analogue output
e7	calibration port
e8	RS232 interface for computer
e9	adjustable feet for levelling the detector

f = The hand held unit

The operator controls are on the keypad located on a handheld unit. The measurements can be read on a digital display, which also serves as a status indicator, informing the operator of the mode in which the instrument is currently operating. The hand held unit also contains the on/off switch, the rechargeable batteries in their holder and the cable and plug, which connects it to the rear of the detector housing.

The following accessories are included with each unit:

the case for moving and storing the unit
the 9V power supply and connector (1g)
the normal and large tube adapters
normal sample tube {0.400 cm OD x 0.324 cm ID} (2b)
large sample tube {0.500 cm OD x 0.420 cm. ID} (2a)
flow cell - normal tube size (2g)
the tube holder (1h)
stoppered gas tight cell -normal tube size (2e)
the tabulation and graphical software package
operator’s manual

1.2 Instrument Description continued

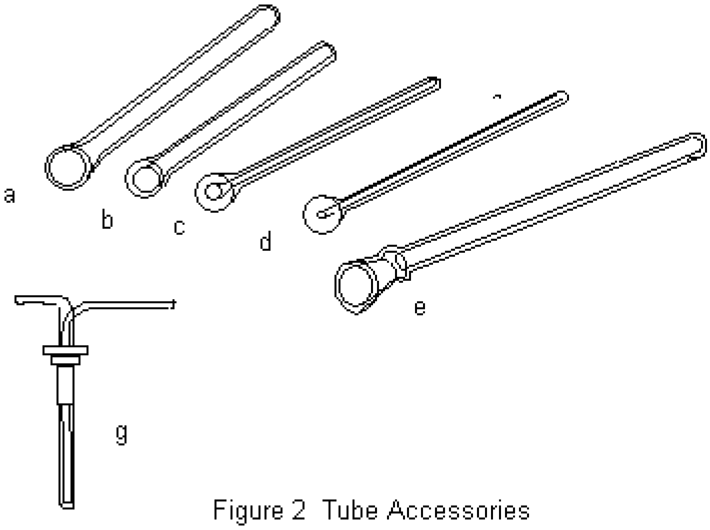


Figure 2 Tube Accessories

Further accessories which are available include:

narrow bore sample tube {0.400 cm OD x .200 cm ID} (2c)
very narrow bore tube {0.400 cm OD x 0.100 cm ID} (2d)

Please refer to Section 3 before removing the travel clamp or attempting to operate the unit for the first time!

A more general discussion follows which is intended to provide a general background to magnetic susceptibility measurement, for those new to magneto chemistry.

1.3 Theory and Definitions

This section is intended to introduce the reader to magneto chemistry and provide a basis for using the Auto.

Basic Principles

Most substances can be classified into one of three groups, dependent on their magnetic properties. Those attracted by a strong magnetic field are known as **paramagnetic**, whereas those repelled are designated **diamagnetic**. The third and most recognised class of materials, **ferromagnetic**, is unique in its ability to retain its own magnetic field and, therefore, they are useful in the construction of permanent magnets. Unlike the ferromagnets, the magnetic properties of the diamagnetic or paramagnetic materials could only be observed and measured when they are placed into an external magnetic field. The external magnetic field becomes more concentrated when passing through a paramagnetic substance and becomes weaker when going through a diamagnetic material.

The behaviour of a substance in an externally applied magnetic field and its resulting classification is dependent on the state of their electrons in their atomic and molecular orbitals. Free electrons, which are unpaired give, rise to high paramagnetic readings while any magnetic effects from paired electron tend to be cancelled out. The overall strength of a paramagnetic or diamagnetic response in a given substance depends on the types of atoms and molecules present, the amount, and the matrix in which they are contained. It is affected by crystal structures, valence states and anything altering the electronic configuration or alignment.

Ferromagnets, able to retain a permanent magnetic field as a solid, become more like a strong paramagnet when made into a fluid, as in a ferro-fluid. Anything effecting the concentration of free electrons in a substance also effects the magnetic susceptibilities; hence free radical formation through chemical reaction and changes in the temperature of a substance can often be found to change the magnetic value.

In practise, the measurement of a magnetic susceptibility value is easy to perform using the Auto and, in most cases, useful results are obtained which can be interpreted without too much difficulty. The magnetic susceptibility of a pure compound can be measured directly at room temperatures, without a correction for temperature, since ambient temperature effects are usually not very great. The determined values are normally within 5% of those quoted in the literature. A mixture of compounds normally has a susceptibility value which is a simple summation of the weight per cent of the susceptibility values of the individual components. This is with the provision that the substances in admixture have not chemically interacted.

1.3 Theory and Definitions continued

What can be measured by the MSB AUTO?

Essentially all diamagnetic and paramagnetic compounds, solids liquids and gases can be measured by the Auto. Diamagnetic forces are usually far weaker than paramagnetic. Their measurements on the MSB-AUTO are indicated by a minus sign before the number, whereas the field liking paramagnetic substances are presented as a plus value.

Ferromagnetic materials can be measured by our balance when in solution or when present in trace amounts in a solid sample, since under these circumstances they are unable to hold a permanent magnetic field and behave like strong paramagnets.

The relative strengths of permanent magnetic fields, held by solid ferromagnets can be measured provided a technique is used which takes account of the field direction, i.e. the direction the sample is introduced, and sample distance.

In addition to using the AUTO for the purpose of characterising a sample qualitatively on the basis of its magnetic properties, the instrument can also be applied to quantitative analyses, making a determination of the amount of an analyte present in a sample.

Analysis by magnetic susceptibility is analogous to the other chemical procedures based on physical parameters, such as spectral absorption or conductance etc. An analyte must be distinguishable from its solvent or solid matrix in order for analysis to be possible. The most favourable combination of components in a sample for a sensitive method to be realised is for the analyte to be a strong paramagnetic substance, giving a strong signal, and the solvent or solid matrix to be diamagnetic, having a weak background. Selectivity and sensitivity can be enhanced by using magneto- chemical reagents and reactions.

The application of the technique is adequately covered in the following sections of this manual. At this point, it is useful to give some basic definition and to briefly describe the electronic phenomena which are responsible for the magnetic properties of a material.

MAGNETIC SUSCEPTIBILITY IS DEFINED AS THE ***RATIO OF THE INTENSITY OF MAGNETISM INDUCED IN A*** SUBSTANCE TO THE MAGNETISING FORCE OR INTENSITY OF FIELD TO WHICH IT IS SUBJECTED.

A magnetic susceptibility value for a particular defined space within a given magnetic field has no associated units of measure, since it is a ratio of the field strength within the defined space to the applied external field strength. As a ratio, the units of force cancel out. The substances placed within this defined space will either attract and concentrate the magnetic field within this region and be paramagnetic or repel and reduce the field strength, and be diamagnetic.

1.3 Theory and Definitions continued

What is responsible for the magnetic property of a material?

One can imagine the permanent magnetic field held by some ferromagnets arising from individual spinning electron which align themselves within the substance. The field generated by each electron would extend far enough to interact with its neighbours. Heating the magnate helps to agitate the atoms and their electrons knocking them out of alignment and interrupting their interactions. The net effect is to reduce the magnets field strength. The explanations given for paramagnetic and diamagnetic compounds are more protracted.

With these compounds, the forces generated by the rotating and spinning electrons within the entire material must cancel out, when there is no external field being applied. The application of a field must however alter the orientation of the electronic circuits or spins, and /or the rate of revolution of the electrons in a manner which makes them align with the fields inductive force lines. With paramagnetic materials, there is an increase in the magnetic flux line density over and beyond that which would occur in the same external field and space if it were occupied by a vacuum. The additional flux is that which is contributed by the electronic currents, which are formed within the material only when it is placed in the external field. Without the support of an external field, the internal field forces are not strong enough to interact and align themselves throughout the substance.

The increase in flux density which occurs in paramagnetic materials also makes them move towards the stronger part of a magnetic field, when they are placed in a magnetic field gradient. If the sample or field is given the freedom to move, it may reach a point where there is no further change either in external field strength or internal material flux, due to the sample being saturated. At this point, movement would stop.

Diamagnetic materials, on the other hand, do not align with an externally applied magnetic field, usually since they have no free electrons to contribute. The magnetic fields arising from paired electrons cancel out, and, therefore, would not be affected by an external field. Instead of enhancing the flux density in the material, the external field would have to cope with non-aligned electronic movements which would effectively oppose a field. The flux density through a diamagnetic material becomes relatively smaller. The movement of the diamagnetic sample in this case would be towards the weaker external magnetic field.

The magnetic susceptibility of a material can be regarded as a measure of the ease with which the material is magnetised by a given magnetic field.

We realise that the above simplistic explanations may raise more questions than they answer. We refer to Section 7 for further reading, with particular note of Shelwood's most readable and informative earlier books.

1.3 Theory and Definitions continued

In order to measure magnetic susceptibility we need to be able to locate and define a volume of space on which we can apply an external magnetic field gradient and to which we can introduce a sample material. We also need a means of measuring the changes in magnetic flux, which arises as different sample materials are introduced to the space. The space within our Auto is formed by the sample tube internal volume as it is positioned between two permanent magnets. These magnets, and their resulting magnetic field, are balanced on a torsion wire and are able to move if the slightest force is applied. The Auto measures the force required to restore the position of movable magnets, which is directly related to the sample placed in the field. The details of the detector, based on the Evan's design are given in Section 2.2.

Several confusing situations arise in magneto-chemistry, usually arising from variations in the definition of terms in the equations dealing with magnetic field strength and magnetisation. In spite of the volume magnetic susceptibility being considered a dimensionless quantity, it has been given units in some equations, e.g. $\{\frac{emu}{\mathrm{cm3}}\}$ (i.e. electro magnetic units per cubic centimetres). Further confusion arises from some textbooks quoting magnetic susceptibility values in the Gaussian c.g.s units, whereas others, particularly the older references, use SI units. The manufacturers of magnets prefer using different units and definitions for magnetic strength than that used by magneto-chemists. These confusing elements should not prevent the user from applying the techniques of magnetic susceptibility measurement in a most simple and effective way. The three most common ways that magnetic susceptibility values are expressed are with reference to the volume of sample, the weight of sample, and the moles of sample. These terms are commonly referred to as **volume susceptibility**, **mass susceptibility**, and **molar susceptibility**. The equations relating these terms are provided below:

VOLUME SUSCEPTIBILITY

The volume susceptibility, designated $\mathbf{X}_v$, is expressed by the following formula:

$$\mathbf{X}_v = \frac{I}{H}$$

where I is the intensity of magnetism induced in the substance and
where H is the intensity of the applied external magnetic field

The volume susceptibility can be quite variable due to changes in the density of the substance, particularly when the sample is a gas or solid. The mass susceptibility, $\mathbf{X}_g$, introduces a density factor in the following manner:

1.3 Theory and Definitions continued

MASS SUSCEPTIBILITY

$$\mathbf{X}_g = \frac{X_v}{d}$$

where d is the density of the substance

Note that $\mathbf{X}_g$ has units of reciprocal density, $\{\frac{cc}{gm}\}$

MOLAR SUSCEPTIBILITY

The most common way of reporting a magnetic susceptibility value in the literature is by molar susceptibility, designated as. X_m. The relationship of molar susceptibility to mass susceptibility is shown below:

$$X_m = X_g \times MW$$

where *MW* is the molecular weight of the substance.

The X_m is the best value to use when comparing different materials qualitatively or assessing the potential of a quantitative application since the value of the term is not subjected to variations due to the method of measurement or to sample density.

The magnetic field occurs in a three-dimensional space, and although many instruments make attempts at having the field relatively homogenous at the point where the sample is being measured this is not always achieved. Solids could vary considerably in various regions of the sample, both in composition and in density. Each instrument for measuring magnetic susceptibility must in some way define the location and magnetic field distribution, as well as the sample volume and position in the field. Since the Auto, like other methods, measures Volume Susceptibility and the literature is quoted in Molar Susceptibility, the conversion between the two values is quite a common in magneto chemical studies. Examples of the conversion can be found in Section 7.6. The initial step in the conversion, that is obtaining an X_g value from X_v, is done within the Auto provided the length and weight of the sample are known and can be entered into the unit.

1.3 Theory and Definitions continued

Qualitative Analysis of Single Component Samples

Magnetic Susceptibilities provide important structural information related to the electronic configuration of the molecule. It is the classical method for examining metal complexes of the rare earth elements.

Liquid samples are easy to introduce to the small sample tubes. The tube is tared, to subtract its diamagnetic contribution, and some care is used to tap any air bubbles that may be adhering to the inside of the tube. The Auto is set at the proper amplification, and the X_v is read directly from the digital output on the hand held controller, without further calculations being required. By measuring the length of the sample and weighing the sample, the length and weight can be programmed into the Auto and the X_g can be read directly. (The Auto does the conversion to density since it knows the internal width of the standard tube and wide tube, and can sense which of these tubes are in the holder). The user must divide the Auto's X_g by the molecular weight in order to convert to molar susceptibilities, X_m.

Single component solid samples can be measured and their units converted in the same manner, with one further consideration regarding their homogeneity. The particles in the solid sample should be ground fine and dried, without agglomerates, to ensure a good packing density is achieved and air is excluded as much as possible. Repeated measurements of a solid in a tube, tapping the tube to compact the solid in between each reading is recommended, to ensure a stable representative result is obtained.

Qualitative Analysis of Mixtures Solids

A mixture of solid materials, assuming homogeneity has been achieved, would show an average mass susceptibility value, $X_{m(t)}$, which was a summation of their individual components, provided no chemical reactions altering their free electron content took place. The formula for the overall mass susceptibility of a mixture with n components is as follows:

$$X_{m(t)} = {}^{W_{(1)}}\!/_{W_t} \text{ x } X_{m(1)} + {}^{W_{(2)}}\!/_{W_t} \text{ x } X_{m(2)} + {}^{W_{(n)}}\!/_{W_t} \text{ x } X_{m(n)}$$

where
$X_{m(t)}$ is the total sample mass susceptibility
$W_{(1)}$ is the weight of sub component (1)
$W_{(2)}$ is the weight of sub component (2)
$W_{(n)}$ is the weight of sub component (n)
W_t is the total sample weight

and similarly, $X_{m(1)}$ through $X_{m(n)}$ are the mass susceptibilities of each sub component. Sample homogeneity can be achieved by grinding a representative sample in a mortar and pestle, followed by grinding between clean glass plates.

1.3 Theory and Definitions continued

Liquids

Binary solutions, consisting of two liquids or a solute and solvent, have a solution susceptibility, which can be calculated in a similar manner since it is again merely a sum of the contribution of each sub component. This means of estimating the overall mixture of components is known as the Weidemann Additivity Relationship.

For M(1) grams of solute and M(o) grams of solvent, where X_S is the solution susceptibility, we have:

$$X_s = M_{(1)} \big/ (M_{(1)} + M_{(0)}) \text{ x } X_g + M_{(0)} \big/ (M_{(1)} + M_{(0)}) + \text{ x } X_o$$

and where X_g and X_O are the mass susceptibilities of the solute and solvent.

The greater number of components in the mixture, the greater the chances for error. There is a known error that occurs when a solid is dissolved to form a solution containing paramagnetic ions. The quoted susceptibility value for solids are usually lower than the solution values which result following dissolution of the solute. Paramagnetic ion corrections are discussed along with other error compensations in Section 4.

Quantitative Analysis

Examining the mixture formulae, it is apparent that the concentrations of the components of binary mixtures can readily be determined, given their mass susceptibilities. The limit of detection of one component in the mixture is related to the magnitude of the difference between their susceptibility values. It is possible to determine the components at a % level, even when the susceptibilities are quite similar. The most sensitive quantitative analysis, down to parts per million concentrations, can be achieved when the analyte is strongly paramagnetic and the matrix is a weak diamagnetic.

As with other physico-chemical techniques, employing selective extractions, reactions, and magneto-chemical reagents expand the scope the technique.

1.3 Theory and Definitions continued

Quality Control

The Auto offers a very simple means of looking at the magnetic nature of a sample. Without much or any sample preparation, it can quickly assess whether a diamagnetic sample is contaminated with a trace of a paramagnetic component. It can also give a value which can characterise the general condition of a complex chemical mixture. Examples of measurements can be found where the following conditions are being quickly and easily appraised:

degree of complex formation
oxidation state
crystal formation
purity of a diamagnetic substance
valence state
free radical formation

Such appraisals often relate to purity of a substance, product degradation or the effectiveness of a process.

The Auto is a non-destructive test, which requires very small quantity of a sample. This makes the method attractive for high value or scarce samples. Arising as a consequence of the small size of sample, it is essential for the analyst to consider the sample homogeneity, particularly when evaluating solid samples.

1.4 Other Methods of Measuring Magnetic Susceptibility

Numerous references review the techniques available for measuring magnetic susceptibility. (The surveys done by Mulay et. al are particularly excellent in reviewing the technique. See references Nos. 48 and 49 along with the classics 1 through 7 and the most recent No. 5.) The Auto has been introduced more recently than these reviews, but is based on the principle of the Evans' detector which has been used in our Mk I unit and is described in Section 2.2.

In considering the various methods which are used to measure magnetic properties, there is a convenient distinction to be made between **static magnetic techniques** and **flux magnetic techniques**. Static techniques apply an external magnetic field to the sample which does not change in strength or direction during the course of the measurement. The properties usually measured by static techniques include magnetic susceptibility, magnetic permeability, and magnetisation. Kinetic techniques, on the other hand, measure the magnetic property in a changing or oscillating field, and the effects being monitored are partly determined by the relaxation times or frequency parameters, i.e. the time it takes for certain molecules to get into equilibrium with the fluctuating field. Kinetic techniques, which include NMR, ESR, EPR, and Mössbauer Spectroscopy can under certain conditions measure or approximate the values obtained by the static techniques. They are beyond the scope of this introduction.

1.4 Other Methods of Measuring Magnetic Susceptibility continued

Table 1: Static Methods

FARADAY's METHOD	Force on a small sample placed within a non-homogeneous field is measured. Sample is free to move.
Advantage:	Only milligrams of sample are required. Can measure ferro-magnetic samples.
Disadvantage:	Sample handling is difficult. Visual detection of force on spring is required.
QUINCKE's METHOD	Liquid sample in capillary (or gas above liquid in closed tube). As field is applied, meniscus moves.
Advantage:	Accuracy and precision is similar to Gouy's.
Disadvantage:	Not for solids. Visual detection required for measurement.
GRAYBILL's METHOD	Modified Quincke method to monitor titrations.
Advantage:	Provides for continuous monitoring.
Disadvantage:	Only for liquids.
RANKINE's Method	Method for gases which uses moving magnets.
Advantage:	High sensitivity.
Disadvantage:	Response is only relative to known standards. It applies to gases only.
BOCKRIS & PARSONS' METHOD	Modified Rankine balance for free radical measurement.
Advantage:	Very High sensitivity.
Disadvantage:	As above.
GOUY' METHOD	As Faraday Balance.
Advantage:	Can measure dia, para and ferromagnetic materials.
Disadvantage:	Large and cumbersome and expensive.

A large number of instrument configurations exist which have been developed for static magnetic measurements, normally aimed at solving the problems associated with a particularly analytical problem and referred to by the name of its inventor. The following table, compares a few of the more well know methods.

Among the more traditional methods for determining the magnetic susceptibility are the methods introduced originally by Faraday and Gouy. More recently NMR has also been used as an instrument for magnetic susceptibility determinations.

Of these approaches, only the technique introduced by the French physicist Gouy in 1889 is explored in any detail, since it still has a strong similarity with the Auto. The advantages we have with our current approach using the Evans design will be evident. The review by Woolcock and Zafar (ref. no. 53.), the most current, favourably compares our Mk I instrument with the current alternatives. Expanding this comparison to include the Auto we can show the following table.

1.4 Other Methods of Measuring Magnetic Susceptibility continued

Table 2: Comparison of the Main Techniques Used to Determine Magnetic Susceptibility

METHOD	**Gouy**	**Faraday**	**NMR**	**MSB Mk I**	**AUTO**
TYPES OF SAMPLES USED	Solids, Liquids, Solutions	Solids, Liquids	Solutions only	Solids, Liquids, Solutions	Solids, Liquids, Solutions, Gases
SOLID SAMPLE SIZE	175 - 500 mg	1 - 50 mg	0.1 - 50 mg	10 - 300 mg	10 - 500 mg
DETECTION RANGE	$\pm 2\times 10^{-5}\, to$ $\pm 5\times 10^{-8}\, cgs$	$\pm 2\times 10^{-5}\, to$ $\pm 5\times 10^{-8}\, cgs$	$1\times 10^{-3}\, to$ 5×10^{-9} ???	$\pm 2\times 10^{-5}\, to$ $\pm 5\times 10^{-8}\, cgs$	$\pm 1.999\times 10^{-4}\, to$ $\pm 5.0\times 10^{-10}\, cgs.$
SOLUTION SAMPLE SIZE	0.50 mls	0.20 mls	0.025 to 0.50 mls	0.07 to 0.30 mls	0.07 - 0.50 mls

In the original report, the cost of instruments was also compared, where the Mk I was shown to be very cost effective. As a general overview, one of the prime features of the Auto is its versatility and adaptability to a range of sample types.

The Gouy Method

The method introduced by Gouy is still the most commonly used method for the measuring of magnetic susceptibility. The basic apparatus is illustrated diagrammatically in Figure 3.

1.4 Other Methods of Measuring Magnetic Susceptibility continued

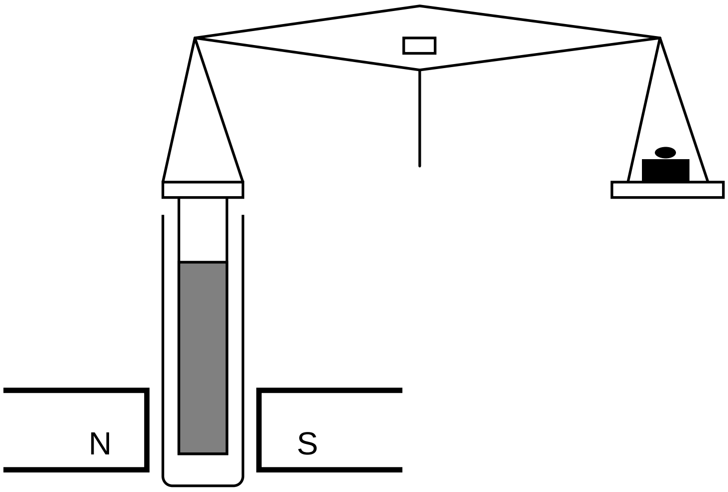


FIGURE 3 : THE TRADITIONAL GOUY BALANCE

The method can measure either paramagnetic or diamagnetic materials, solids, liquids, and solutions. For good results, the sample, which is placed in a tube, should be homogeneous and uniform in diameter. The field is only relatively homogeneous towards the centre of the magnets. (Faraday was able to extend the area of homogeneity of the field by shaping the magnets).

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 relative 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:

$$F = \frac{1}{2} X v A H^{2} \qquad \text{(i)}$$

where Xv = volume susceptibility of the sample
A = sample cross sectional area
F = δ (m) g
Xv = Xg d
A = $\frac{m}{l \times d}$
δ (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 gms cm^{-3}
l = sample length in cm.

1.4 Other Methods of Measuring Magnetic Susceptibility continued

Hence,

$$X_g = \frac{2gl\delta(m)}{H^2 m} \qquad \text{(ii)}$$

If H is kept constant, this can be written as

$$X_g = \frac{C\delta(m)l}{m} \qquad \text{(iii)}$$

where:- C = a constant.

Subtracting the reading of the empty tube from the same tube containing the sample does not correct for the susceptibly of the air displaced by the sample. The 20.9% oxygen in air is paramagnetic - Xv (air) = 0.029 $\times 10^{-6}$ cgs at 20 degrees 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: -

$$F = \frac{1}{2}\left\{X_v - 0.029 \ \ x \ \ 10^{-6}\right\}AH^2 \qquad \text{(iv)}$$

leading to the corrected expression:-

$$X_g = \frac{0.029 \times 10^{-6} V + C\delta(m)l}{m} \qquad \text{(v)}$$

where :- v = volume of the sample in cm^3 .

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

The above discussion is intended to introduce some of the details involved in making a measurement of magnetic susceptibility. The systematic error due to the presence of air is considered above, along with the attempted correction. This systematic error is one of several which occur with the Auto as well as the traditional methods. These error corrections are discussed in Section 4.

The Auto is similar to the Gouy method. It introduces a sample into a tube, which is then positioned so that the bottom of the sample is within uniform field strength while the top is in a region of negligible field. The corrections for tube error and air displacement, as well as other calculations, are automatically performed (within the reservations later given.). Both the Gouy method and the Auto measure volume susceptibility. The Auto can convert this to mass susceptibility if you input the information required for it to calculate the density, i.e. the sample weight and length.

1.5 Operating Guidelines

The Auto is a sensitive instrument which, if used in accordance to the guidelines given in Section 3.0 of this manual, should give trouble-free service. The Auto has been designed to be easy to use and transport, with the minimum of manual adjustments. This makes it significantly different than the other static techniques. The set-up procedure is critical for good results to be obtained.
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 instrument has a travel clamp which can be applied or disengaged by simply the turning of a screw that is located within the sensor housing. The clamp screw head can be found by removing the sample tube adaptor and sliding the stainless steel plate (see figure 1A ©) towards the back of the sensor housing. Turning fully clockwise applies the travel clamp and turning fully counter-clockwise releases the clamp for measurement.
   The travel clamp should always be applied before moving the instrument and released at least 15 minutes before taking measurements.
2) The instrument has been designed to have ***no user serviceable parts*** on the inside. It should not be opened by the operator under any circumstances unless under the instructions of the manufacturer. Failure to follow this guideline may result in the loss of the warranty.
3) The instrument has been designed to be portable and sensitive. The sensitivity and **performance is *dependent on the location*** of the instrument during its use. To use at the maximum sensitivity range, one needs to have a vibration free, constant temperature environment away from draughts and stray magnetic fields. If you appear to have difficulties with obtaining a reproducible result or with the zeroing, try a new location.
4) The Auto automatically zeroes itself when no sample tube is present. While doing this it allows for the influence of all magnetic materials within its immediate environment. During the measurement of a sample, however, the instrument stops correcting for environmental changes. For the **best results, keep still during the measurement**; the position of all items within 10 feet of the detector should remain unchanged during the time of measurement to ensure the zero setting remains unaffected.
5) Ferromagnetic materials should not be placed near the balance or removed during the auto-zeroing or the measurement. This includes more obvious things like metal tools and instruments, and less obvious items such as watches, jewellery, chairs, and pens. Ferro magnetic impurities introduced in sample preparation can lead to considerable errors. The use of plastic or non-magnetic spatulas and utensils is generally recommended, but becomes essential when operating at the highest sensitivities. For the best results, **keep away from ferromagnetic materials.**
6) 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 or obscure the optical tube-sensing detector near the top of the tube when cleaning.
7) The two sample tube adapters, for normal and wide bore tubes, must be securely positioned onto the top of the guide tube to ensure the sample tubes are accurately placed in the detector.
8.) When the Auto is not being used in the immediate future, apply the travel clamp. If the Auto is to be stored for a considerable time, please return it to its case, Pack the detector in its plastic bag. Store in a dust free area and a non moisture condensing environment.

Section 2.0 System Overview

The Sherwood Auto-MSB has been particularly designed for ease of application as well as convenience in use and high sensitivity. Like our MkI balance, solid, liquid or gaseous samples can be introduced to a sample tube which is in turn accurately positioned in the balance for the magnetic measurement. The tubes used are manufactured to rigorous tolerances in order to ensure that the sample is reproducibly positioned. The Auto-MSB is modular having a hand-held unit, containing the digital read-out and controls, and the main body of the instrument, comprising the detector and sample holder. This new arrangement has proven to be both convenient and practical.

Following a survey of our customer's needs, a range of special features have been incorporated into the Auto-MSB which makes this instrument ideally suited for new analytical applications in the research laboratory and many areas of industrial quality control. These features are summarised.

2.1 MSB-Auto Features

TRUE PORTABILITY

The balance weighs just under 2.2 kg and is supplied in its own robust carrying case. It contains its own rechargeable battery power supply, which gives you up to 8 hours of operations away from the laboratory. An easy to apply travel clamp (external to the instrument) and an adjustable level control makes the instrument operational within minutes and, similarly, safely prepares it for transport.

BENEFITS FROM MICROPROCESSOR CONTROL

The 8751 microprocessor has the balance calibration constant entered during manufacture. We no longer need to carry Standards to adjust each instrument. The digital display shown is in volume magnetic susceptibility units and requires no further conversion factors. By entering the sample weight and length (internal sensors detect the diameter of sample tube used), mass susceptibility units are displayed. The Auto-MSB automatically zeroes when the sample is removed, so the instrument does not require manual zero adjustment and is always ready to take an accurate measurement. An auto tare function can be implemented simply by pressing the tare key with an empty tube in the sample position.

VERSATILITY OF APPLICATION

The Auto-MSB has been design to accommodate many sample types and meet a wide range of applications.

In addition to our normal size sample tubes, which are 0.400 cm. OD x 0.324 cm. ID, we now can adapt the instrument to take wide bore tubes, 0.500 cm. OD x 0.420 cm. ID, by simply changing the tube adapter provided with the instrument. The wide bore tubes can be used to handle viscous liquid samples, like oils, since they are easier to fill and clean. They also give more signal for diamagnetic or weakly paramagnetic samples.

Special sample tubes are available for gases, and liquids, as well as reactive or volatile samples that must be sealed from the air. Flow cells are also available for use with fluids.

2.1 MSB Auto Features continued

HIGH SENSITIVITY

The Auto measures in volume susceptibility (cgs. units) directly. Under ideal conditions, the Auto can measure from 0.001×10^{-7} to 9.999×10^{-4}.in cgs units over four operating ranges. This is a total operating range of seven orders of magnitude. Under fairly poor measurement conditions, measurement can be made on the gain of 10^{-5}or even 10^{-6} ranges, provided the drift isn't too high due to a large temperature change.

FLEXIBLE INPUT AND OUTPUT

The instrument has a digital display on the hand held unit. It also has an RS232 computer interface and an analogue output for a chart recorder.

2.2 The Detector

The detector is based on the Evans' design, similar to the Mk I instrument. The design by Professor Evans has basically the same configuration as the traditional Gouy balance 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 magnet is observed. The detector used in the Auto is shown in Figure 4.

Referring to Figure 4, a sample tube adapter is positioned onto the guide tube (a). When the sample is introduced into this tube to the detector, it is accurately positioned between a pair of magnets which are on a yoke (c) and form our stationary magnetic field. There is a second pair of matching magnets (b) at the opposite end of a beam which are positioned around wound wire coils (d). The beam assembly is clamped on a torsion wire (e) and mechanically balanced. The position of the beam assembly is monitored by two optical detectors (f) situated at opposite ends of the beam which observe the optical flags (g) which are extended from the beam.

There are two wire coils (d) wound around a central core. The first is used to auto zero the detector by passing a current through the coil in order to obtain the correct position of the beam as determined by the optical detectors. The second is used to measure the sample. The sample, on introduction to the detector, interacts with the field and attempts to move the field and beam, either towards or away from it. The restorative current that is required in the second coil to return the beam to its zero position is directly proportional to the volume susceptibility of the sample.

A sample tube detector is situated at the top of the guide tube (h), and can optically detect the presence of a sample tube and differentiate between a normal tube (0.400 cm O.D. x 0.324 I.D.) and a wide bore tube (0.500 cm O.D. x 0.420 I.D.). When no tube is detected, the balance automatically zeroes, correcting for drift and environmental changes, but as soon a tube is detected, it automatically starts measuring the sample.

2.2 The Detector continued

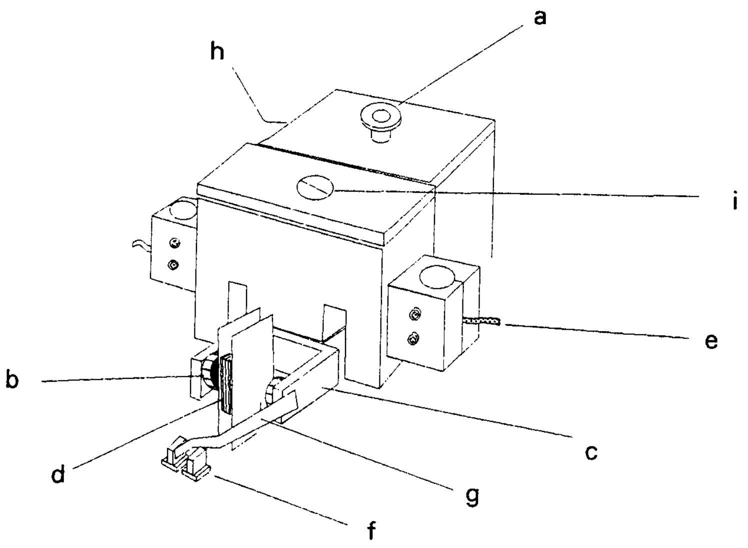


Figure 4 The Evans' Detector

At position (i), you can see the screw head which applies the travel clamp. Turning the screw clockwise pulls the balance mechanism up locking it against the solid housing, and making it secure and ready for travel. Turning the screw counter-clockwise, releases the balance mechanism, allowing it to balance on the torsion wire, ready for a measurement. The clamp design is such that it is impossible to overtighten or over-loosen the clamp.

Both the Gouy method and the Mk I MSB require calculations to obtain the volume susceptibility value, but the Auto reads in Xv directly, in cgs units. The Auto has been calibrated at the time of manufacture. Should recalibration be required see Section 5.2.

2.3 Specification

Most of the MSB instruments are considerably larger than our Auto. The comparison of specification with the Mk I instrument is provided below, since it is the only other instrument on the market that has a similar size and specification.

Table 3 SPECIFICATIONS

	Mk I MSB	**AUTO**
Size in cm (unpacked)	22w x 28d x 14h	18 w x 24d x 9h
(packed)	34 x 26 x 45	25.5 x 51 x 41
Weight (unpacked)	3 kg (7 lbs)	1.2 kg (2.5 lbs)
(packed)	4.5 kg (9.9 lbs)	9 kg (19.8 lbs.)
Power Requirements	110V or 220V	6 to 9 V dc (110/220 V ac adapter) provided with 4xAA rechargeable batteries)
Measurement Range	in the range of ±2 x 10^{-5} to ± 5 x 10^{-8} arbitrary units	±1.999 x 10^{-4} to ± 5 x 10^{-10} cgs
Sample Tubes Available	normal tubes (0.324 cm I.D.) narrow tube (0.200 cm I.D.) v. narrow tube (0.100 cm I.D.) gas sample tubes of above stopcock tube of above flow cell (normal tube) flow cell (narrow bore)	all Mk I tubes plus: wide tube (0.420 cm I.D.) wide bore gas sample tube wide bore stopcock tube wider bore flow cell
Amount of Sample Required for Accurate Determination of Magnetic Susceptibility	Volume 70 µl - (min.) 300 µl - (max) Weight (as solid) 40 milligrams (min) Weight (in solution) 2.5 milligrams (min.)	Volume 70 µl - (min.) 400 µl - (max) Weight (as solid) 40 milligrams (min) Weight (in solution) 2.5 milligrams (min.)
Detection Limits	approx. 10 x lower than above weight limits depending on M.Sus.Value	approx. 2000 lower than above weight limits depending on M.Sus. Value
Outputs	digital display	digital display RS232 Analogue Output
Magnetic Field Strength	3.5 kGauss	4.5 kGauss

2.3 Specifications continued

Drift and **Noise** are important analytical criteria that are difficult to describe simply in the above table. As described, the Auto corrects for drift continually by zeroing the instrument when no sample is detected. The drift can be observed by introducing and leaving a sample tube in position. The rate of drift is more evident at higher sensitivities and is usually directly related to the temperature change that the detector is experiencing.

If a constant temperature and humidity room is not available and readings at high sensitivities are required, there are several measures that can be taken to help. Let the Auto fully zero the balance before introducing a tube. If you must measure using the digital read out, read the values as quickly as possible after they appear. Preferably, use the tabular or graphical software package or a chart recorder. If possible, check the calibration by using known standards.

The noise in the system is related to vibration and possible changes in the magnetic environment. The relative significance of the noise on a reading, again depends on the sensitivity range that is being used. The closer to a zero magnetic susceptibility you go, the more interference you would have from a constant noise level. (Recall that the susceptibility value actually goes through zero, since it can be positive or negative).

In practice, it has been found that some solid samples appear to be noisier than others, possibly due to their packing and anisotropic nature, or due to local ferromagnetic zones which may move.

Section 3.0 Installation and Shipping

3.1 Installation Requirements

Careful consideration should be given to siting of the balance, before it arrives, since the final performance will be related to the operating conditions. In terms of the facilities required, the instrument only requires a power source. The instrument is equipped with 4 AA rechargeable batteries contained within the hand held unit. It automatically recharges, when the unit is plugged into the source electricity, whether or not the units is in operation. Once charged. the unit can operate without a power source for up to 8 hours.

The units can be set to operate at lower sensitivities within minutes. A ten minute rest period following the removal of the travel clamp is advised in order to reduce the drift before the unit can be easily use on the 10^{-6} range.

Consideration should be made as to the accessories that will be used with the unit. It would be best to have the computers, reactors, etc. operating on a different bench in order to isolate their effects from the detector. Similarly, samples should be prepared and tubes should be filled away from the instrument. Sample tubes should be wipes cleaned on the outside, prior to being introduced to the detector.

If it is intended to operate the unit, at the highest sensitivity 10^{-7}, one should consider providing a constant temperature and humidity environment. The rule of thumb is the greater the performance required, the more stringent is the requirement for providing the right environment and more carefully the samples have to be prepared.

3.2 Environmental Requirements

The ideal environment is as follows:

1. Free of mechanical vibrations.
2. Free of magnetic field fluctuations.
3. Away from ferromagnetic materials.
4. At a Constant temperature.
5. Free of draughts
6. Clean and dust free.
7. Away from direct bright light and sunlight.
8. In a relatively dry, non-condensing atmosphere.

Each of the conditions is discussed in turn to help the operator appreciate the criticality.

Mechanical Vibration:

The detector is best positioned on a vibration free bench built for a microbalance. We have never seen a poor performance when it is situated in a balance room. The balance does contain a seismic filter and is designed to be tolerant of vibrations in general. With all such devices, however, certain frequencies and amplitudes of vibrations seem to effect the balance more than others. The main negative effect that vibration has is to increase the noise. Because of the increase in noise, it takes a lot longer to zero and the samples readings seem to be much more jumpy. Higher standard deviations result.

We have seen a computer and a copying machine, result is excess noise, when positioned on the same bench about twenty feet away from the detector, but the same detector tolerated a fluid bed dryer unit on the same bench. We have notice of an increase in noise on several occasions, when operating at elevated floors of various hotels. The balance never operates that well when placed on a flimsy table of an exhibition hall, for a variety of reasons.

3.2 Environmental Requirements continued

Magnetic Field Fluctuation:

Unfortunately, used largely by inorganic chemist, the instrument has found that operating on the other side of a wall from an ESR or NMR instrument is not very conducive to a good performance. Even small motors and other such devices, which generated a magnetic field, have been found to generate noise even if not in physical contact with the bench. Often the noise generated here is periodic or is found to oscillate with the use of equipment.

Ferromagnetic Materials:

Probably enough has been said about this potential interference. The problems caused by this interference can be quite subtle. They occur as either noise or drift, and appear as a fairly large deviation from the expected result occurring at a random interval. When they occur, the first very natural reaction is to blame the instrument.

Some of our actual experiences include,

- a watch on the wrist of an operator was held over the detector only when a sample tube was placed in the detector for measurement. This systematically biased the results.
- the back of a swivel chair which contained mild steel. was occasionally swung near to and then away from the detector, resulting in a reproducible step change from the anticipated result.
- a balance was sited by a wall which was the other side of an elevator shaft and showed a large deviation as the elevator passed by.

Temperature

The change in temperature which the balance is experiencing is directly related to drift. This becomes particularly important at higher sensitivities. At the highest gain, of 10^{-7}, the balance should be operated in a constant temperature environment having less than 1 degree Centigrade change per hour. It takes the instrument several hours to come to a temperature equilibrium.

Direct sunlight falling on the balance should be avoided as well as a location that is near a heating duct or heat of any sort.

Free from Draughts

In addition to being able to heat/cool by convection, the movement of air can effect the noise of the balance, particularly if it occurs as a directed stream. The problem can result in an increased noise, when the air stream is more or less constant, a randomly occurring deviation (e.g. when a door is open or closed next to the balance) or a drift, if the air is heating or cooling.

3.2 Environmental Requirements continued

In a Dust Free Area

Dust can arise from general atmospheric conditions, for example when construction is occurring in the vicinity, or when the instrument is too close to the sample preparation area, where pulverising, grinding, sieving etc. is going on.

The problem arises when paramagnetic dust collects in the sample tube (or even worst in very severe conditions, on the magnets inside the balance). This may very well result in excessive noise in the detector until the sample tube is cleaned out with a swab. (If the magnets get covered, the balance has to be returned to the agent).

Away from a Direct Strong Light Source

Although we have made the instrument fairly light tight, there are a few directions where a strong light can interfere with the optical sensor inside the balance. This is evident when it happens.

In a Non-condensing Atmosphere

Moisture build up inside the instrument can result in optical sensors not operating properly and short circuits occurring.

All the above faults could be avoided by selecting a good site in the first place. If you are not satisfied with the performance of your balance, the first thing to do is try a different location.

3.3 Unpacking

Please follow the directions in sequence:

1 Choose the correct location for the balance.

2 Remove the carrying case from the shipping box by grasping the handle firmly and pulling. Bring the instrument in the carrying case to the location where it will be used in the laboratory or balance room. Please note that the side of the case opposite the handle has four plastic feet. There are also four similar feet located on one of the sides of the case. Rest the case on the side having the feet and release the two catches near the handle, which should be facing you.

3 Open the case slowly. You should have the instrument with its accessories on the bottom part of the case facing you. (If not close and lock the case, and gently flip it over).

4 Remove the box marked "**SAMPLE CELLS FRAGILE!"** Place this box in a safe place.

5 Making sure you have a clear bench space, remove the following items from the case and rest on the bench.

- the detector
- the strap and keys for the case
- the hand-held unit with its cable
- the AC/DC converter

and - the sample holder

6 Check for the following items in the box marked "**FRAGILE**!".

- two metal adapters
- two normal sample tubes
- one large sample tube
- one liquid flow cell
- one stoppered gas tight cell, with stopper

7 Pick up the instrument and remove it from the plastic bag. Turn it upside down, observing the push button which releases the travel clamp. (Do not touch it, but observe its position. It should be protruding out of the base plate). Turn the instrument right side up and return it to the bench.

8 Connect the cable of the hand-held unit to the back panel of the detector. Connect the AC adapter to the main power supply and then connect the DC cable to the detector. At this time, connect any other peripherals, such as a chart recorder or computer interface.

9 Choose a normal or wide tube adapter from the contents of the box and gently but firmly press into the guide tube visible in the centre of the top of the detector housing.

10 Level the instrument by observing the bubble position on the indicator located on the back panel and screwing in/out the rear feet of the detector to centre the bubble.

3.3 Unpacking continued

11 Release the travel clamp by picking up the detector and feeling for the button underneath. Keep the instrument upright; push the button up into a position closest to the base of the detector. (The clamp is released or applied by the same action of pushing this button. This should only be done when the instrument is in an upright position). The instrument should be left with the clamp off for about 30 minutes before being switched on after removing the travel clamp to reduce the drift associated with the relaxation of the torsion wire.

3.4 Start Up Procedure

Following the above set up procedure, the instrument is now ready for starting operations.

3.4.1 Turning the instrument on

The instruments on/off button is a small switch located on the hand-held unit. Turn it on and observe the display. It should come to life and indicate the version of software that is being used in the instrument (for about 3 seconds) before beginning to auto zero.

V1.6.1

This code plus you serial number on the back panel will identify your unit in future correspondences.

3.4 Start Up Procedure continued

3.4.2 Autozeroing

Following the brief software prompt, the instrument starts to auto zero, as indicated by the "**Z**" in the last position of the display. The display changes every three seconds, indicating the position of the beam as it searches for the zero position. Typically, the display shows the following output every three seconds:

1	***V1.6.1***
2	**+0.011 E-5Z**
3	**-0.017 E-5Z**
4	***-0.064 E-5Z***
5	***-0.022 E-5Z***
6	***-0.008 E-5Z***
7	***-0.002 E-5Z***
8	***-0.001 E-5Z***
9	***-0.001 E-5Z***
10	***-0.000 E-5Z***

The display gives a status report which is refreshed every three seconds. An explanation of the display is as follows:

The four digit number, "**x.xxx**", can read either plus or minus. It is followed by "**E-5**" which indicates the gain setting. The four possible gain settings are 10^{-4}, 10^{-5}, 10^{-6} and 10^{-7}. Auto zeroing always occurs on the gain of 10^{-5}.

With each display, we are given the beam position, the gain setting and the mode, i.e. auto zeroing or some other function. Following the final display, the reading remains unchanged. It normally takes from 30 to 45 seconds to obtain the zero value. The values displayed in the zeroing process could be very different from those shown above, but they will always approach zero. Once the zero is indicated, the balance is ready for the introduction of the sample tube.

3.4 Start Up Procedure continued

3.4.3 Measuring a Sample

Introducing a sample tube automatically changes the mode of operating from auto-zeroing to "**Processing**" and this is indicated on the display. After a few seconds, the display changes to a numerical value, followed by a "**V**" indicating that the measurement that is in progress is being reported in volume susceptibility units. The gain setting is also indicated. Following the introduction of an empty tube, the display can be seen to change as shown:

1		**-0.000 E-5Z**	
2	Introduce tube	**Processing**	here
3		**-0.006 E-4V**	
4		**-0.005 E-4V**	
5		**-0.004 E-4V**	
6		**-0.005 E-4V**	

The "**E-4**" indication on the display shows that the lowest gain is being measured on the empty tube. When converting to a measurement, the Auto goes to the lowest gain first and depends on the operator to increase the amplification as required. The minus value on the display shows the diamagnetic nature of the glass.

If allowed to remain in this position, the reading and display would eventually drift off the correct value initially shown. With removal of the sample tube, the instrument goes back into auto zero mode, and after a few steps, it finds zero and is ready for the next introduction of the sample.

3.5 Shutting Down

The shutting down procedure is simply the reverse of starting up. The only question that arises is for how long you are intending to leave the instrument. For a short shut down period, when you are attending to the instrument, you can merely switch the instrument off. It is advised that if you are leaving the instrument unattended or shutting down for several hours, that you apply the transit clamp.

We recommend that if the instrument will not be used for several days, it should be repacked in its case, which can then be stored in a secure area.

Simply turn the instrument off and apply the clamp. Leave the power to the instrument on if you wish to charge the batteries.

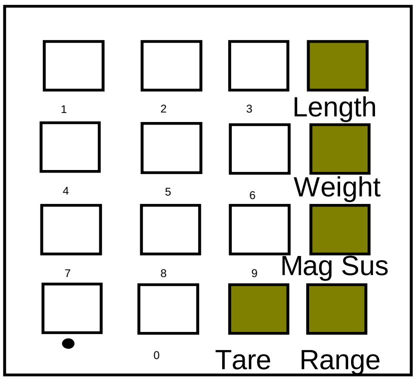
3.6 Repackaging for Transport

This process simply reverses the unpacking procedure. It would be good to make note of the original layout of the accessories so that you can achieve the optimal packing.

Section 4.0 Operating the Auto

4.1 General Operations

The hand held unit has the front panel shown in Figure 4. The green functions keys, and the numerical keys, white, are indicated below the key. All control operations are performed from this keypad.



:

Figure 5

Going back to the start up procedure, we have introduced a tube and obtained a reading, with the following display.

1 Introduce tube **Processing** here

2 **-0.006 E-4V**

3 **-0.005 E-4V**

4 **-0.004 E-4V**

5 **-0.005 E-4V**

To amplify the measurement, press the "**Range**" button on the keypad and observe the display.

Range X 10

Processing

-0.042 E-5V

The display now shows the reading at the next higher gain, 10^{-5}. Repeating the pressing of the range button puts you into the next higher gain. 10^{-6}

Range X 100

Processing

-0.428 E-6V

This is normally the highest gain that is used without taking special precautions for the siting of the balance and the sample preparation. Blank tubes usually have a value of -0.410 to -0.430 E-6

4.1 General Operations continued

If you press the range button again, you will notice that a lot of time is spent in the "**Processing**" stage of the cycle. That is because there is a running average of a dozen readings being taken at this gain in order to remove some of the noise. It should be very evident if you are experiencing drift at this high gain setting, since your display will degrade quite rapidly.

The auto zero operates at gain E-5. If operating at higher gains, one has to use the "**TARE**" function located next to the range amplification. The tare function essentially subtracts the value of the tube. Since tubes vary quite a bit at these higher gains, you will have to use either the same tube for the blank reading as the sample, or use a matching tube for the blank. In this case, two empty tubes must be found that give close readings before you load one with the sample.

An example of taring a tube, after it has been introduced and amplified to E-6.

Range X 100

Processing

-0.423 E-6V

press tare button **+0.001 E-6V TARE**

+0.000 E-6V TARE

When the tube is removed, the display changes to show the negative value of the tube, which is held in memory. This value is the correction that will be used when the next tube is introduced. The reading that will be displayed is from the sample only.

remove empty tube **+0.422 E-6V TARE**

Introducing a matched tube at this stage, but one containing distilled water, you get the following reading:

-0.720 E-6V TARE

The reading you see is that of the water alone. This is essentially the literature value for the volume susceptibility of water. The lower the gain, the longer you can hold a TARE function without experiencing drift. The best correction for drift is to remove the tare function and return to auto zero.

Overflow

The display reads "**Overflow**" if the sample value exceeds the selected measurement range of the Auto. (The operating range includes the sample reading plus any tare value that may be retained in the memory). The only thing that can be done when you see this display is to go to a lower gain setting and try again.

4.1 General Operations continued

Operating at Gain E-7

The signal processing at 10^{-7} is slow since the numbers displayed are a running average. Functions, such as TARE, have a delay before they are implemented. It takes 20 seconds to accumulate enough data to establish a running average and several seconds for the tare to be engaged or disengaged.

4.2 Volume Susceptibility

The above displayed readings are all in volume susceptibility, therefore, they all have a "**V**" in the display following the gain indicating. Once the tube contribution is subtracted manually or removed by the tare function, the value indicated is the volume susceptibility of the sample. As in the case for water above.

4.3 Mass Susceptibility

The volume susceptibility can be converted to the mass susceptibility by introducing a sample density factor. This can be done, relatively easily using the functions on the hand held unit. The following procedure should be followed:

Prior to running a sample the following information must be acquired.

- The length of the sample, liquid or solid should be measure in millimetres and recorded.
- The weight of the empty tube should be noted along with the same tube containing the sample. The difference is the weight of the sample, which should be recorded in grams (up to four decimal places can be introduced, i.e. x.xxxx.)
- Wait until a zero value is obtained during the auto zero mode.
- Introduce a matched blank tube to the detector and amplify to the desired gain.
- Press the tare function, and observed the display to ensure that the numerical value of zero is displayed. (If not, disengaged the tare and try again).
- Remove the sample tube and introduce the sample.
- Press the "**Length**" function button and observe the display:

Length = --.- mm

- Enter the length by pressing the appropriate three number keys. in order. (There is no need to press the decimal point key because the Auto is expecting the format shown).

(The length must be at or between 15.0 and 75.0 mm or the number will be rejected and the display will show "**Out of Range**" and return to the request to input the length. The maximum length on our sample tubes is about 45 mm. Pressing the Length function again at this time will return to the Xv-Tare display).

4.3 Mass Susceptibility continued

Following the input of the appropriate length, the display indicates "**Processing**" and then returns to the Xv- Tare display. Press the "**Weight**" function button, and observe the display.

Weight= 0.---- gm

(Values between 0.0010 grams and 0.9999 grams are expected by the Auto or the "**Out of Range**" indication will appear).

a. Following the input of the appropriate sample weight, the display will return to the Xv-Tare display. You can now press the "**Mag Sus**" button and observe the display.

Mass Suscept.

#.### E-5M TARE

- You have now converted the display to read Mass Susceptibility, as indicated by the "**M**" that now replaces the "**V**". By pressing the "**Mag Sus**" button again, you return to the volume susceptibility.

The Auto detects whether a normal (0.324 cm ID) or a wide (0.420 cm ID) bore tube is being used and converts the length and weight values to a density in order to convert the mass susceptibility. For narrow or very narrow tubes, you cannot use this conversion function but have to do the calculations manual.

4.4 Error Corrections

The factors are retained in memory provide the power is not turned off. This allows repeat measurements to be performed quickly, provided the density can be assumed to be constant since the original length and weight data can be used.

There are several sources of systematic error in the analysis which can be avoided when they are understood. A more thorough explanation is given in this section which should lead to an improvement in the results that can be obtained by the balance.

4.4 Error Corrections continued

Sample Tube Errors:

The contribution of the diamagnetic glass on the reading has already been discussed. This has led to several methods being mentioned in passing, such as matching of tubes and taring of blanks. The price of the expensive sample tubes that are provided with the balance is due to the very tight specification on the ID dimension, having the bore lie central to the tube. The critical lengthways dimension, from the position of the black tube stop placed on each tube. If the specification of any of these dimensions are reduced, it leads to a higher standard deviation of measurement.

On going to a tube of lesser specification, two provisions can be made, namely:

1. use the same tube for the blank reading and the sample (by recording the reading from the empty tube, it can be subtracted manually at a later time).
2. The variation of results on rotating the sample should be checked. If the bore in the tube does not lie truly central, a large deviation may occur. This can be partially corrected by marking the tubes so that its rotational position in the balance can be reproduced. (This type of problem occurs with our narrow bore tubes).

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)

4.3 Mass Susceptibility continued

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, i.e. do not fill to the part which begins to widen towards the neck of the tube, Figure 6.

Measure the length of the water in the tube from the top of the base to the bottom of the meniscus, Figure 6.

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

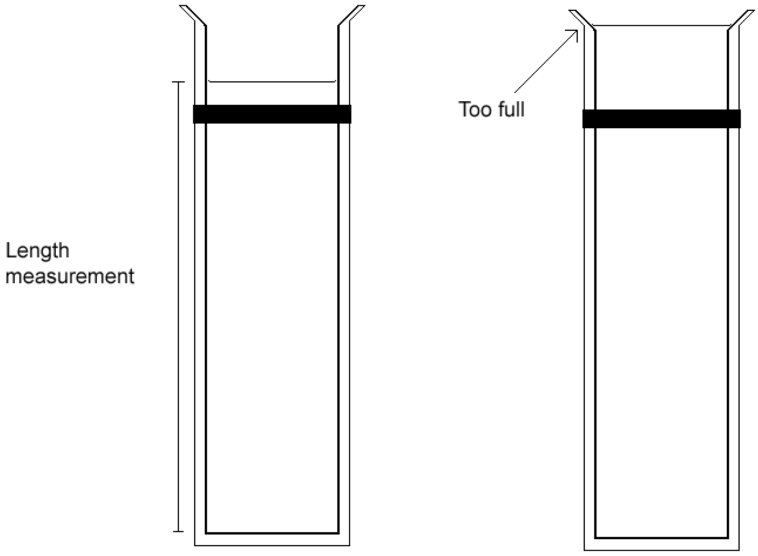


Figure 6

4.4 Error Corrections continued

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: -

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

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

4.4 Error Corrections continued

Errors in the Measurement of Length (Meniscus Errors)

The measurement of sample length in a tube containing liquid, involves an estimate of the position of the meniscus. With solid samples, the surface of the solid can be equally difficult to have flat and even, and therefore, can be equally difficult to measure accurately. Also the sealed bottom end of the sample tube is not always perfectly formed, i.e. non-constricted and ending in sharp right-angled walls. These facts tend to make the measurement of length far less accurate than the measurement of weight and also the possible Auto measurement. Since the length value is converted into density, which is used in the calculation to mass susceptibility, the errors associated with it are directly transferable to the Xm and Xg values.

Several steps can be taken to reduce the inaccuracies. Since "length errors" are constant, use largest amounts of sample possible. This will reduce the errors proportionately.

1. Estimate the meniscus on liquids by using the average value between the minimum and maximum readings.
2. Attempt to make solid surfaces flat, before measuring the length. This can more easily be accomplished with finer samples.
3. Select tubes that are well formed tubes at their sealed ends, since that portion of the tube has the greater effect on the measurement.

4.4 Error Corrections continued

Air Displacement Errors (packing of samples.)

A discussion on the error due to the presence of air has already been given in the section on the Gouy Balance, Section 1.4. The error that comes from air is primarily due to the most paramagnetic component gas, oxygen (Xv air = 0.029 x 10^{-6}). At 20 degrees C and 760 mm Hg pressure, the Xg of air in the tube is 0.051 x 10^{-6}. This is the maximum error that can be introduced into a reading due to air displacement.

When we tare the tube, the value of the silica glass tube plus the air inside is recorded for later subtraction. However, when we add the sample to the tube, we are effectively displacing the air. We then measure the sample and subtract the recorded tare value. Having already excluded the air with the displacement of the sample, there is no need to further correct for it by subtracting its value which is included with the tare. This in effect creates an air displacement error.

Amount of Sample Required

In the calibration of the balance, we correct for this air displacement error by adjusting the balance to read the value of water in volume susceptibility units. The calibration is further checked with a highly paramagnetic standard, like $Hg[Co(SCN)_4]$. A solution or liquid is usually excellent at excluding air from the sample tube. All our solid standards are intentionally very fine particulate, which also pack reasonable well, and so do exclude air as well as most solids. Basically, our calibration method corrects for the air displacement error quite well, only if the sample excludes air from the tube as efficiently as the standards.

Solid samples always have some air trapped in with them. It is not uncommon for the density of a powder to be less than half of the true solid density, in which case we are again introducing air into the tube and reintroducing an air displacement error. The few options that we have to avoid this error are:

- dry the sample.
- grind the sample to a fine uniform powder, load small amounts of sample into the tube at one time and tap frequently when loading the sample into the tube.
- check for homogeneity by taking readings while rotating the sample.
- .if a rotational inhomogeneity is found, empty the tube and reload.
- make solutions where possible to reduce this error.

For quantitative or qualitative assessment, you would like to have enough sample in the tube so that a change in the amount of sample would not effect the results of the measurement. There is a minimum amount of sample that is required in order to accomplish this. By increasing the amount of sample, and hence length, the portion of the sample near the open end of the tube is moving out of the magnetic field and, therefore, becoming less and less significant to the measurement. If one considers plotting the Auto measurement response versus the length of sample, we find that the sample should be a minimum of 1cm in length for it not to have any further effect on the reading.

We refer to the excellent Woolcock references for thorough studies on minimum sample amounts required on the Mk I MSB. These results would also apply to the Auto.

4.4 Error Corrections continued

Ion Correction for Dissolution of Solids

As a solid, atoms are effected by each others presence and all the paramagnetic and diamagnetic moments are additive. In dissolving in an aqueous solvent, the ions go into solution and separate from their interatomic influences. Molecules which ionise to form a paramagnetic ion, usually demonstrate a higher mass susceptibility than would be expected from their unionised solid value. This arrives from the diamagnetic contribution inherent in the solid from other chemical groups or ligands present. Since magnetic moments are additive, the mass susceptibility for the ion (Xa) can be calculated from the solid value (Xm) by the addition of the appropriate positive corrections for a variety of ions and ligands. A partial list of these corrections is provided below in Table 4.

Table 4 Diamagnetic Corrections for Ions and Molecules
10^6 Xm (c.g.s.)

CATIONS		ANIONS		MOLECULES	
Li^+	1	F^-	9	H_2O	13
Na^+	7	Cl^-	23	NH_3	16
K^+	15	Br^-	34	ethylenediamine	47
Rb^+	22	I^-	50	pyridine	49
Cs^+	33	CH_3COO^-	29	$(C_6H_5)_3P$	167
NH_4^+	13	$C_6H_5COO^-$	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	$C_2O_4^{2-}$	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		
		SO_4^{2-}	38		
first row transitions metals	13	$S_2O_3^{2-}$	46		
		[acetylacetonate] $acac^-$	55		

4.5 Outputs from the Auto

Chart Recorder Output

The analogue chart recorder output produces a ±1V signal which can be delivered to a chart recorder. This signal is obtained prior to digitisation, and does not show the effect of the tare or auto calculations.

Computer Interface

The Auto has an RS232 interface to communicate with a computer. The signal obtained is following digitisation, and is able to show the same numerical values as seen on the keypad display. One can see the effect of tare or read the values in mass susceptibility units.

4.6 Software Options

Two software packages are currently provided, for use with an IBM compatible computer and connected to the Auto through its RS232 port. The first is a Tabulation pack which puts all readings into a Table, labels each sample, and provides a basic standard deviation and averaging routine. This is intended to make data gathering and analysis evaluation easier, and provide a hard copy of the result.

The second software package is a Graphical pack which following a set up procedure, is able to graph each output from the auto and show the trend. It helps particular in a quality control regime or when using a flow cell. Both routines are self explanatory.

Section 5.0 Maintenance

5.1 Use of Spot Check Standards

Several standards can be used for checking the calibration of the balance. Perhaps one of the most reliable trouble free standards is water (10^6 Xg = -0.720), since it is readily available in pure form. A singly distilled water sample should suffice.

$HgCo(SCN)_4$ (10^5 Xg = +1.644) is considered one of the best solid standards (Reference 75) and is available from Johnson Matthey, Aesar and Alfa Catalogue Company (Ward Hill Massachusetts tel. 508-521-6305).

The ethylenediamine salts of nickel, $Ni\ (en)_3S_2O_3$ (10^5 Xg = +1.104) is a useful secondary standard (see Reference 76). Many solid samples of paramagnetic substances, if properly dried and ground and packed, usually give within 2 % of their literature value.

5.2 Calibration Adjustment

A special tool is provided for turning the pot to adjust the calibration which is located at the rear of the detector. Prior to doing this, it is important to consider whether the balance has drifted or whether the purity of your sample is in question. More often than not, it is the later which is the cause for a recalibration.

The adjustment screw can be turned either way to lower or raise the value on the display. The best procedure is to put your standard into the detector, tare and amplify up to the highest gain possible. Adjust the calibration dial to approximately the correct reading. Now repeat the cycle. An adjustment on the sample, is also adjusting the value of the blank tube which now must be re-tared. A few attempts at this should get your value reading with 1% of the literature value.

5.3 Battery Charging, Storage and Replacement

Four AA rechargeable Ni/Cd batteries, 1.2V, are provided in the hand held unit of the Auto. The recharger is built into the Auto. When the Auto is supplied with the 9 V DC supply from the AC adapter that comes with it, it automatically recharges the batteries. They should be fully recharged after about 3 to 4 hours and should provide for approximately 8 hours of use. If being stored for a long time, the batteries should be removed from the unit and stored in a refrigerator, at about 4 degrees C.

5.4 Cleaning and Storage of Sample Tubes

The good maintenance of the sample tubes is essential for good result. Sample tubes can be cleaned with the conventional detergents and organic solvents for reuse, as appropriate, depending on the nature of their sample. Pipe cleaners have proven to be a useful tool. Similarly an aerosol can of ethylenedichloride, supplied with a plastic capillary spout (available for degreasing electronic components) has proven invaluable in removing oil from the tubes when analysing for wear metals.

The tubes should be stored in a dust free environment and their susceptibilities should be measured before introducing a sample in every case. As a final precaution, the outside of the tube should be wiped with a dust free wiper just prior to introducing it to the balance, particularly if it has been allowed to lay on the bench.

5.5 Cleaning the Instrument

The only part of the instrument which may require cleaning is the guide tube on the detector, where some solids particles may collect from the samples being introduced. If paramagnetic dust particle collect on the inside of the guide tube, this can significantly increase noise.

Cleaning the tube with a cotton swab, available on long sticks (~8 cm) is most convenient. In any case, please use a soft material so the inside of the thin guide tube will not be damaged.

5.6 Storing the Instrument

Store the instrument in its plastic bag inside the metal case in which it has arrived.

Section 6.0 Applications

The measurement and of magnetic susceptibility is most frequently used in inorganic chemistry, particularly in the study and characterisation of the rare earth elements. In this area it is recognised as offering an important qualitative tool which describes the electronic state of the molecular structure. Its use outside this area is far less common. When compared with analytical techniques based on spectroscopy or electrochemistry, techniques based on magnetic susceptibility hardly rates at all.

Survey of the literature (References 54 through 66) show that a majority of the applications using magnetic susceptibility analyse solid samples for inorganic chemical studies. The more limited application to solutions is primarily on investigations of biological systems containing iron species. Certainly part of the reason that this technique has not been more generally applied is due to the lack of sensitivity of the classic methods along with the cumbersome and expensive instruments required to do the work. Hopefully, the Auto will partly remove these restrictions.

The diversified applications which exist are based on the detection of the electronic state of sample, or more specifically the analyte within the sample. Examples of the areas which can be explored by the MSB follow.

1) Assignment of the oxidation state of the metal in complexes of the transition, lanthanide and actinide elements.
2) Stereochemical information - for example, square planar Ni II complexes are diamagnetic while octahedral ones are paramagnetic with two unpaired electron.
3) Information concerning ligand field strength - some transition metal complexes can be high spin or low-spin depending, in part, on the field strength of the ligands. Therefore the calculation of the magnetic moments and hence the number of unpaired can be used to assess ligand field strength.
4) Antiferromagnetism interactions in dimers and polymers. Occurring between neighbouring metal atoms or ions in dimeric or polymeric complexes (e.g. cupric acetate), these lead to magnetic moments which are smaller than expected.
5) Complexation by ligands - for a number of transition metal complexes, complexation by ligands alters the magnetic behaviour. Thus, a number of square planar Ni complexes are diamagnetic when dissolved in non-coordinating solvents, such as benzene and chloroform, but paramagnetic in coordination solvents like pyridine. This is due to axial coordination of two ligand molecules to give a 6-coordinate Ni II complex.
6) Criterion of purity - pure Y_2O_3 is diamagnetic but contamination with the lanthanides such as Erbium or Dysprosium can cause the samples to be paramagnetic.
7) Measurement on air-unstable compounds - the balance is ideally suited for compounds which decompose when exposed to air. Either operate the detector of the balance in a glove box or seal the sample in one of the special tubes available. Similarly the effects of other more noxious gases, such as halides or nitrogen oxides or ozone on various solids or liquids can be studied.
8) Measurement of reactions occurring in a relatively homogeneous mixed reaction chamber, redox reactions for example, can be followed by recirculating a small sample stream through a flow cell situated in the detector.

9) The absorption and desorption of metals can be followed by flowing a stream of pH controlled liquid through an ion exchange resin which is position in the bottom of a special tube positioning the resin in the detector. Resins can be characterised this way as well as certain metal species and certain solid phase reactions. (See reference No. 54).

10) The quantitative analysis of a trace paramagnetic element in a diamagnetic matrix, such as metal wear particles (mainly iron) in lubricating oils, trace iron in diamonds, contamination of copper.

11) Archaeological studies of soil samples to indicate the prior occupation by humans. Higher oxidation states of the metal components are indicative of activities using high temperature, e.g. kilns, fires for cooking, and many early industrial processes associated with metalworking, crockery manufacture, etc.

12) Measurement of the oxidation state of haemoglobin.

13) Measurement of magnetic titration, this can again be accomplished using a back mix reactor vessel into which the titrant is added and a small side stream is sent to the Auto's detector. (See references Nos. 67, 68 and 69).

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Section 8.0 Ordering Parts

The name of your local representative is located on the back panel of the detector. If you unable to contact them for whatever reason, you can call or fax us for the latest information on price and your nearest area to purchase accessories. Our contact numbers are on the Cover. Our address is:

Sherwood Scientific Ltd.
1 The Paddocks
Cherry Hinton Road
Cambridge
CB1 8DH

The part numbers are given below:

Part Number	**ITEM**	**DESCRIPTION**
710 86 002	SAMPLE TUBE	NORMAL (0.324 cm ID)
710 86 007	SAMPLE TUBE	NARROW BORE (0.200 cm ID)
710 86 006	SAMPLE TUBE	V. NARROW BORE (0.100 cm ID)
700 86 004	SAMPLE TUBE	WIDE BORE (0.500 cm ID)
700 86 003	SAMPLE TUBE	FLOW CELL, NORMAL BORE
710 86 005	SAMPLE TUBE	GAS TIGHT TOP
700 86 010	ADAPTER	NORMAL TUBE
700 86 005	ADAPTER	WIDE TUBE
700 86 501	HAND HELD UNIT	(ALL AUTO UNITS)
700 04 126	HOLDER	SAMPLE TUBES
700 66 701	SOFTWARE	TABLE & GRAPH
001 43 901	TOOL	ADJUSTING CALIBRATION
	STANDARD	SPECIFY RANGE

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.

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.

Product Warranty Statement (continued)

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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