Johnson Matthey Magnetic Susceptibility Balance Operating Instructions Last Update: 12/19/12

The balance is located in MG 1064 (the NMR room) while the sample tubes and instruction manual are kept in MG 1026.



Instrument Set-up

1. Be sure that there are no ferromagnetic or metallic materials near the balance and that the balance is level (check the bubble is in the center of the spirit bulb on the balance's top; adjust as needed using the knobs on the rear legs).

2. Turn the **Range** knob to the **x1** setting and allow the balance to warm up for at least 10 minutes before it is used.

Be sure that the balance reads close to 000 when the **Zero** knob is set to the center of its range. The **Zero** knob is a high-precision ten-turn potentiometer and can be set to the middle of its range by <u>gently</u> turning it either clockwise or counterclockwise until it stops and then turning it in the *opposite* direction for five turns. Each turn clockwise increases the reading by about 85 (and a counterclockwise rotation has the opposite effect). The balance is useable if it gives a reading of no more than -420 at this point, but it is probably need of adjustment (place an out-of-order sign on the balance and notify one of the inorganic faculty members as this adjustment is **not** to be done by students).

3. Adjust the **Zero** knob so that the display reads 000. When this cannot be done, check that there are no metallic objects nearby, that there are no air currents blowing on the balance and that it is level. If these steps do not fix the problem, the balance will need to be re-zeroed per the instructions found in the balance's user's manual (<u>not</u> to be done by students). In the event that the balance cannot be zeroed, notify an inorganic faculty member and place an out-of-order sign on the balance.

4. Determine the calibration constant for the balance, $C_{balance}$, by inserting the calibration tube into the balance's tube guide (located on the top of balance) and recording the instrument reading, *R*. The calibration constant can then be calculated from the following equation where C_{st} and R_0 are printed in the label attached to the calibration tube (the current calibration tube has $C_{st} = 1151$ and $R_0 = -33$). Record this reading in the log book.

$$C_{balance} = \frac{C_{st}}{(R - R_0)}$$

Making a Measurement on a Solid Sample

1. Place an empty sample tube of known weight (weigh using an analytical balance) into the tube guide on the top of the balance. Record the reading, R_0 .

2. Remove the tube from the balance and pack it with the sample. It is best if the sample is a crystalline powder, which can be achieved with a mortar and pestle (but too fine of a powder will not give a good measurement) or simply crushing with a <u>plastic</u> spatula. Introduce a small amount of the solid into the tube's top and <u>gently</u> tap it on a wooden surface to force the material to the bottom of the tube. Repeat until there is 2.5-3.5 cm of sample in the tube (1.5 cm is the minimum depth that can be measured). Even packing of the material is essential! Determine the sample depth, ℓ , using a ruler to the nearest 0.01 cm, and the sample's mass, *m* (by difference of the mass of the packed and of the empty tube).

3. Place the packed sample tube into the tube guide and record the reading, R. Note that a negative reading indicates that the tube plus the sample have a net diamagnetism (the glass tube is diamagnetic and therefore the sample must be diamagnetic also).

If the display goes off scale, turn the **Range** knob to the x10 setting and multiply the reading by ten.

Be sure to also record the room's temperature as susceptibility is temperature dependent.

4. The mass susceptibility (in units of cm³/g), χ_g , at the measured temperature is then given by substitution into the equation

$$\chi_g = \frac{C_{balance} \cdot \ell \cdot (R - R_0)}{m \cdot 10^9}$$

The molar susceptibility can then be easily obtained by multiplying χ_g by the molar mass.

Instrument Shutdown

1. When no additional measurements will be made, remove the sample and turn the **Range** knob to **Off**. If additional measurements will be made, you can leave the balance on with the **Range** knob set to the x1 scale.

2. Carefully remove the sample from the tube. You may need to gently tap it on a wooden surface (protected by a weigh boat or weigh paper to catch the dislodged material). Do not damage the sample tube's rim and do not use solvents or acids to remove any recalcitrant material, except under faculty supervision. Do NOT immerse a sample tube in any solvent, acid bath or base bath as this will damage the seal near the tube's top. If a fine coating of sample remains on the inside of the tube, it may be <u>carefully</u> washed out with <u>small</u> rinses of methanol.

3. The solid sample may be kept for additional measurements or other uses. The methanol rinses should be collected and properly disposed of.

4. Invert the sample tube in a beaker with a Kim-Wipe layering the bottom. Allow the tube to air dry, then return it to the storage box and return the box and its contents to its storage location in MG 1026.