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Appendix I: Magnetic Susceptibility Measurements using the Johnson-Matthey MSB-1 Balance.

The instrument should be turned on about 10 minutes before use. Turn on the scale to x1. There is no need to zero before use, because all readings are measured as differences between the sample tube + sample and the empty tube. Check the position of the rubber collar on the sample tube. It tends to swell if it absorbs solvent and will slide along the tube. For correct use, the bottom of the collar should be 5 cm from the bottom of the tube. Weigh a clean empty sample tube (m_o) on an analytical balance and then record the reading (R_o) on the magnetic balance. Pack the tube with the sample to a length of 2.5 - 3.5 cm. Tap the tube carefully to pack the sample tightly. Record the weight (m) and the new reading on the magnetic balance (R). Measure the length of the sample (l) in cm.

The mass susceptibility, χ_g, is calculated from the equation :

$$\chi_g = \{C_{Bal} .l.(R-R_o)\}/\{(m - m_o) x10^9\}$$

where l = sample length (cm); $m - m_o$ = sample mass (g); R = reading for tube + sample; R_o = reading for empty tube; C_{Bal} = the balance calibration constant written on the back of the instrument (C_{bal} = 1.083). This incorporates the area cross-section of the sample tube.

Now χ_g is the magnetic susceptibility per gram and must be a positive number. If $R \approx R_o$ then diamagnetism is indicated. To convert this to the molar susceptibility, χ_m, multiply χ_g x molecular weight. Because ligands and the central metal will contribute a negative effect as a result of their diamagnetism, the magnetic susceptibility for the complex can be found by <u>adding</u> the diamagnetic contributions of the ligands and the metal to χ_m. This gives χ_A.

The effective magnetic moment, μ_{eff} is given by

$$\mu_{eff} = [\{3.k.T.\chi_A\}/\{N.B^2\}]^{1/2}$$

where N = Avogadro's number; B = the Bohr magneton; k = Boltzmann's constant; T = absolute temperature.

Combining the constants reduces the formula to

$$\mu_{eff} = 2.828(\chi_A T)^{1/2}$$

This is equivalent to $[n(n + 2)]^{1/2}$ (n = # unpaired electrons) when the spin-only formula can be used. This is usual for first row transition elements when the orbital contribution is largely quenched.

Hence, if μ_{eff} = 1.73, then n = 1; μ_{eff} = 2.83, then n = 2; μ_{eff} = 3.87, then n = 3; μ_{eff} = 4.90, then n = 4; μ_{eff} = 5.92, then n = 5; μ_{eff} = 6.93, then n = 6. Match your μ_{eff} to the nearest value to find n and compare this value with the theoretical number predicted knowing the element and its oxidation state.