



THREE FACTORS AFFECTING MAGNETIC SUSCEPTIBILITY MEASUREMENT USING SHERWOOD MAGNETIC SUSCEPTIBILITY BALANCE

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ABSTRACT

The magnetic susceptibility is a measured of magnetic interaction of a sample with the external magnetic fields. This method called Gouy Method and often applied to solid samples. On this research, the magnetic susceptibility of inter-related iron-oxalate compounds has been measured using Sherwood Magnetic Susceptibility Balance (MSB) at room temperature. Three factors have been found affecting on measurement of magnetic susceptibility. There are rubber ring position attached on the MSB tube, sample length and sample packing. The rubber ring position and sample length are associated with position sample in the external magnetic field. Sample packing is associated with homogeneity of the sample in MSB tube. The optimum rubber ring position is 4,7 cm and the optimum sample length is 2,5 cm, both measured from the bottom of the MSB tube and measured at well packed sample.

Keywords : *Magnetic susceptibility, Magnetic susceptibility Balance, Gouy method.*

INTRODUCTION

The magnetic susceptibility is used to identify paramagnetism of a substance which is arise from unpaired electron. Theoretically, the effective magnetic moment, μ_{eff} , can be calculated from total spin quantum number (S) and orbital contribution (L), which is expressed as $\mu_{\text{eff}} = \sqrt{4S(S+1) + L(L+1)}$. If the orbital contribution is relatively small, the equation can be reduced to $\mu_{\text{eff}} = \sqrt{4S(S+1)} \mu_B$ which is

known as spin only formula. Experimentally, the μ_{eff} can be obtained from molar magnetic susceptibility, χ_M , measurement. The relationship between μ_{eff} and χ_M is give as $\mu_{\text{eff}} = 2,828\sqrt{\chi_M T}$ in Gaussian unit [1].

The magnetic susceptibility is normally measured using Gouy Method and often applied to solid samples. For measuring magnetic susceptibility, the portable equipment namely Sherwood Magnetic Susceptibility Balance (MSB) was used. The operational this equipment is very simple, fast and the accuracy is high. Due to this reasons, this equipment is suitable for senior high school students in learning magnetic properties of inorganic compounds [2,3]. The Sherwood Magnetic Susceptibility Balance and it's schematic diagram are shown in Figure 1.

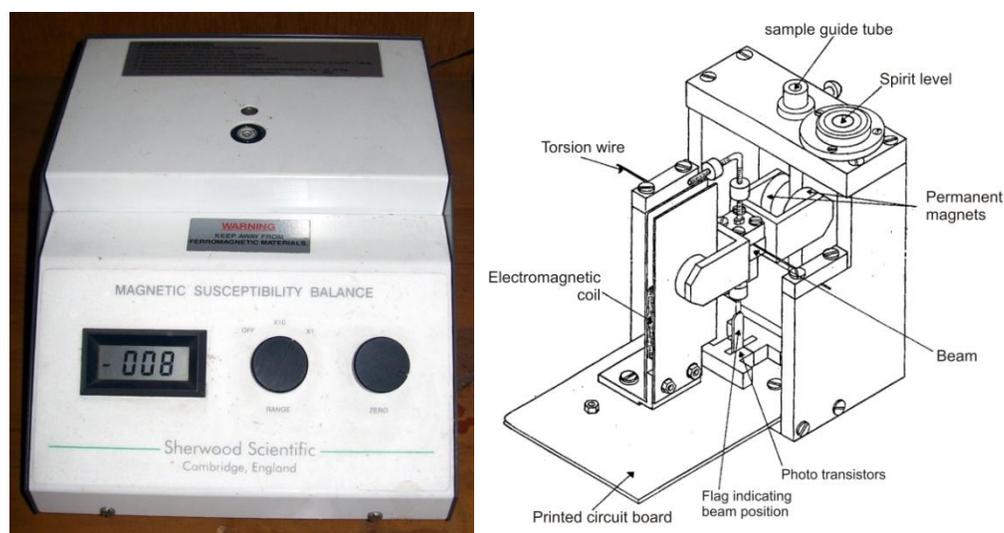


Figure 1. Sherwood Magnetic Susceptibility Balance and its schematic diagram

For complexes containing 3d metallic ions the observed value of magnetic moments are higher than the spin only value. The compounds containing hexaaquo iron(II) in high spin states allow μ_{eff} 5,0 – 5,6 μ_B higher than the $\mu_{\text{so}} = 4,9 \mu_B$, while for hexaaquo iron(III), the μ_{eff} value is range from 5,7 μ_B to 6,0 μ_B , closely related the $\mu_{\text{so}} = 5,9 \mu_B$.

In this research, three factors affected the magnetic susceptibility ($\chi_M T$) measurement of iron-oxalate compounds $\text{Fe}(\text{ox}) \cdot 2\text{H}_2\text{O}$, $\text{K}_3[\text{Fe}(\text{ox})_3] \cdot 3\text{H}_2\text{O}$ and $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$ have been studied to obtain the high accuracy of the measurement and to obtained the close relationship between μ_{eff} and μ_{so} .

EXPERIMENT

The three iron-oxalate compounds were related each other : the simple salt, $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ was a raw material for the formation of $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ and this complex was generated to produce more complicated tri metallic $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$ compound [4]. The detailed of sample preparations have been mentioned in published paper [5].

The magnetic susceptibility of three iron-oxalate compounds has been measured in different sample lengths and rubber ring positions. Rubber ring position is varied from 3,5 cm to 5,5 cm, measured from the bottom of MSB tube with sample length of 0,5 cm and 2,5 cm respectively. The sample length is also measured from the bottom of MSB tube varied from 0,5 cm to 3,5 cm with rubber ring position of 4,3 cm and 4,7 cm. Two different of packing modes have been apply to $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ and $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ samples.

RESULTS AND DISCUSSIONS

All solid samples are relatively stable in an open air at room temperature. Physically, the appearance of the three compounds can be distinguished from their physical appearance. The $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ is yellow powder, the $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ is green crystal, and the $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$ is crimson crystal. The sample performance can be seen in Figure 2.

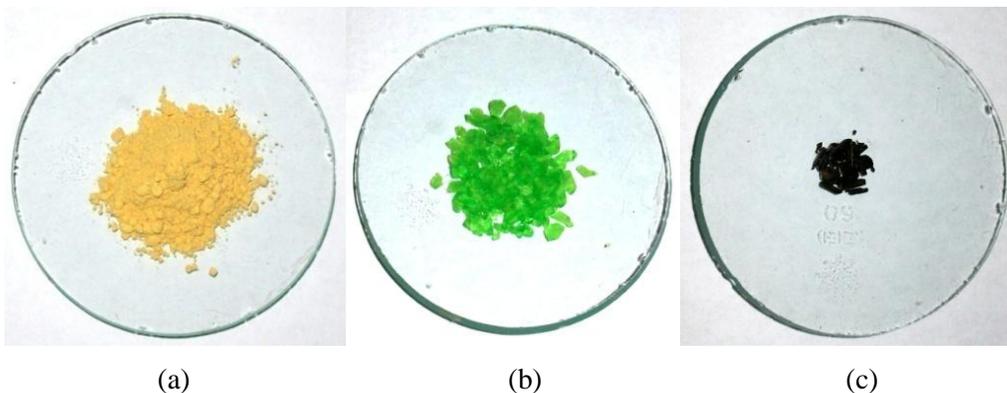


Figure 2. Sample performance of (a) $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$, (b) $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ and (c) $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$

The magnetic susceptibility of the three iron-oxalate compound have been studied with focused on the three factors, there are sample length, rubber ring position, and sample packing. The sample length is associated with the quantity of samples and the accuracy of the sample in the centre of the external magnetic fields, the rubber ring position is associated to the location of the sample in magnetic fields, and

sample packing is associated with the sample homogeneity in MSB tube. The morphology of MSB tube consist of a sample is shown in Figure 3.

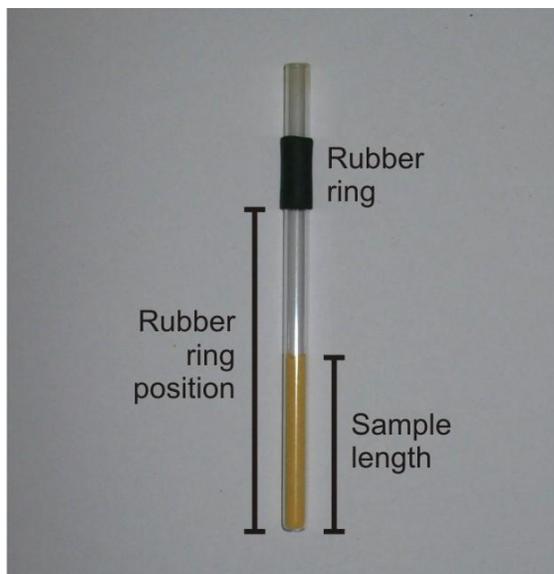


Figure 3. MSB tube morphology

When the MSB tube is put on the sample holder, the sample should be located correctly in the centre of the external magnetic field to provide the optimal result. The correct position of sample in the external magnetic field is shown in Figure 4.

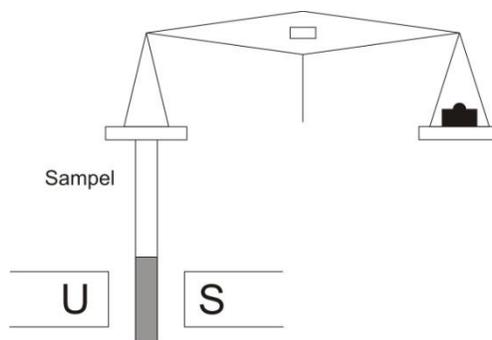


Figure 4. The position of sample in the external magnetic field.

The $\chi_M T$ of three sample $\text{Fe(ox)} \cdot 2\text{H}_2\text{O}$, $\text{K}_3[\text{Fe(ox)}_3] \cdot 3\text{H}_2\text{O}$ and $[\text{Fe(bipy)}_3][\text{FeFe(ox)}_3]$ have been measured and converted to magnetic moment value at sample length between 0,5 cm to 3,5 cm. The plot of $\chi_M T$ versus sample length has been shown in Figure 5.

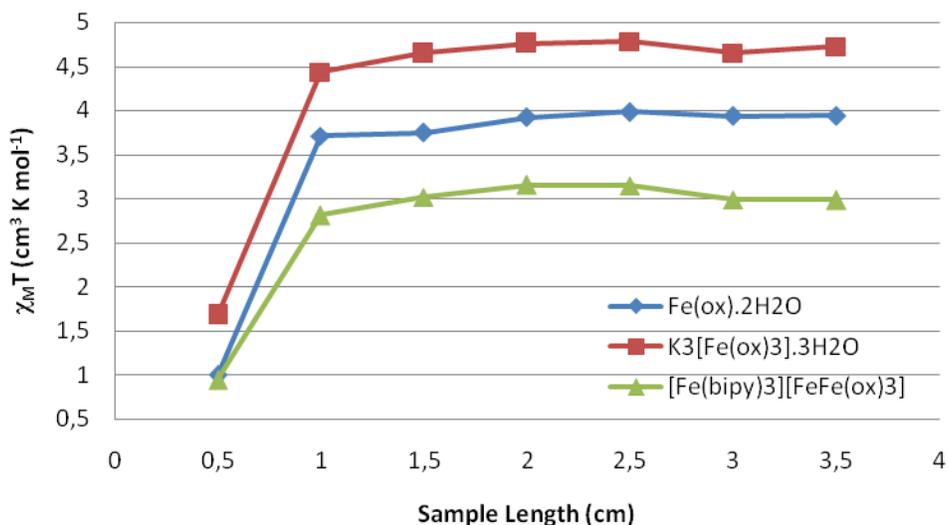


Figure 5. The plot of magnetic susceptibilities of three samples with sample length variation

At sample length of 2,5 cm, the $\chi_M T$ of Fe(ox).2H₂O was found 3,99 cm³ K mol⁻¹ or its μ_{eff} is 5,64 μ_B . This result is higher from the expected value (2,99 cm³ K mol⁻¹ or 4,89 μ_B for iron(II) in high spin state). While for K₃[Fe(ox)₃].3H₂O the $\chi_M T$ was found 4,55 cm³ K mol⁻¹ or the μ_{eff} is 6,15 μ_B , that is slightly higher than the expected value (4,36 cm³ K mol⁻¹ or 5,91 μ_B for iron(III) in high spin state). Unfortunately the μ_{eff} for multi-nuclear [Fe(bipy)₃][FeFe(ox)₃] the maximum value is 3,16 cm³ K mol⁻¹ or 4,91 μ_B . The result is smaller than the expected value. However, this result in agree with the previous data which is indicated the existence of four unpaired electrons in both iron (II) in [FeFe(ox)₃]²⁻ [4].

At sample length between 1,0 cm to 3,5 cm the μ_{eff} value remains fluctuate slightly and optimum at 2,5 cm. However if the sample length of 0,5 cm, the μ_{eff} for all sample dropped very low and the magnetic moment value was decreased down to 65%. This is due to the position of the sample which is too far from the centre of the external magnetic field.

To improve the accuracy of the results, the rubber ring position is adjusted to 4,3 cm. The μ_{eff} of all samples are increased markedly although still less than the expected values. The μ_{eff} accuracy of Fe(ox).2H₂O was improved up to 93,7 %, the K₃[Fe(ox)₃].3H₂O was improved up to 86,4 % and for [Fe(bipy)₃][FeFe(ox)₃] was improved to 29,3 %. The magnetic moment of Fe(ox).2H₂O, K₃[Fe(ox)₃].3H₂O and [Fe(bipy)₃][FeFe(ox)₃] and their accuracy are shown in Table 1.

Table 1. The μ data for iron-oxalate compounds

No	Sampel	μ (μ_B)			Accuracy to expected value at 4,3 of sample length
		Expexted value	4,7 cm of sample length	4,3 cm of sample length	
1	Fe(ox).2H ₂ O	4,89	2,80	4,58	93,7 %
2	K ₃ [Fe(ox) ₃].3H ₂ O	5,91	3,60	5,00	84,6 %
3	[Fe(bipy) ₃][FeFe(ox) ₃]	6,93	0,94	2,03	29,3 %

When the rubber ring position is varied, the μ_{eff} maximum for all iron-oxalate samples were found at rubber ring position of 4,7 cm with 2,5 cm sample length. The accuracy of measurement are still higher than 98% when the rubber ring position between 4,6 cm to 4,8 cm. The magnetic moment diagram of all samples versus rubber ring position is shown in Figure 6.

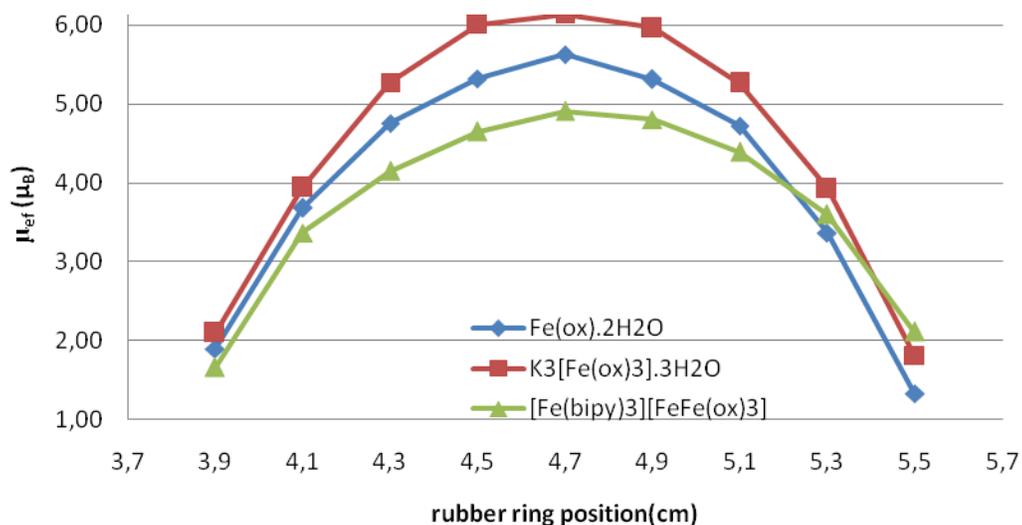


Figure 6. The plot of magnetic susceptibility with variation of rubber ring position

The third factor that influencing magnetic susceptibility measurement is sample packing. For homogeneous sample powder, the packing is relatively easy. However, for crystalline compound, normally the sample needs to be grinded to get well packed sample. For Fe(ox).2H₂O the magnetic moment was found 5,64 μ_B . However when the sample was not well packed, the μ_{eff} found 5,16 μ_B or

reduce 8,5%. This value is slightly higher than the expected value for iron(II) in high spin about 5,52%. The Figure 7 shown about well packed and unwell packed for $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ compound.

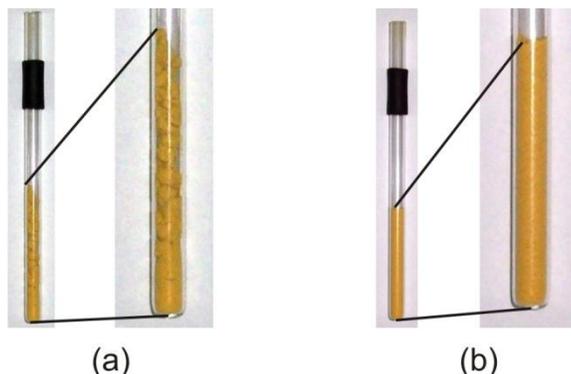


Figure 7. (a) Unwell packed and (b) Well packed for $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ compound.

The influenced of sample packing was also obtained in the magnetic susceptibility measurement for $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ sample. In this case slightly higher of magnetic moment was found for $6,14 \mu_B$. This due to the partial damage of grinding $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ crystals.

CONCLUSIONS

Three factors affecting the magnetic susceptibility measurement of three iron-oxalate compounds $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$, $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$, and $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$ have been explored, they are sample length, rubber ring position and sample packing. The high accuracy of magnetic moment measurement were found at 2,5 sample length, with rubber ring position at 4,7 cm and well packed sample. The μ_{eff} of $\text{Fe}(\text{ox})\cdot 2\text{H}_2\text{O}$ and $\text{K}_3[\text{Fe}(\text{ox})_3]\cdot 3\text{H}_2\text{O}$ was found $5,64 \mu_B$ and $6,15 \mu_B$. This result are indicated the iron(II) and iron(III) in high spin state. For multi nuclear $[\text{Fe}(\text{bipy})_3][\text{FeFe}(\text{ox})_3]$, the μ_{eff} is found $4,91 \mu_B$. Although this result was lower for iron(II) in high spin state. However, it was similar with the previous published report [4].

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