

❖ Application of Magneto-Chemistry in Structure Determination

The paramagnetic susceptibility of transition metal complexes can be used to obtain information about the electronic structure, stereochemistry and the nature of metal-ligand bond. The primary applications of magneto-chemistry in the structure determination of coordination compounds are given below.

➤ *Determination of the Oxidation State of Transition Metal Centre*

The most widely used application of paramagnetic susceptibility measurement is the determination formal oxidation number of the transition metal center. The aforementioned use relies on the finding of the number of unpaired electrons present on the central metal atom or ion. The diamagnetic allowance due to ligands attached is usually ignored; however, corrections must be made for the large orbital contribution to the magnetic moment. For instance, some of the low-spin Co^{2+} complexes have a μ_{eff} around 2.9 B.M., though they have only one unpaired electron ($t_{2g}^6 e_g^1$). Likewise, some of the high-spin Co^{2+} complexes have a μ_{eff} around 4.9 B.M.; suggesting four unpaired electrons, though they have only three unpaired electrons ($t_{2g}^5 e_g^2$). Therefore, in order to find the spin only magnetic moment; and thus the correct number of unpaired electrons; the temperature-dependent study of magnetic susceptibility is pretty much essential. Furthermore, the larger magnitude of spin-orbital interaction can also affect the spin-only magnetic moment to a significant extent in some cases like the low spin complexes of Os^{4+} ion. For example, the effective magnetic moments of low-spin complexes Fe^{4+} and Ru^{4+} complexes are about 2.83 B.M.; which is pretty much close to their spin-only value, suggesting two unpaired electrons, which is obviously correct. However, the μ_{eff} for $[\text{OsCl}_6]^{2-}$ complex is only 1.4 B.M.; suggesting only one unpaired electron, which is pretty far from reality. Therefore, spin-orbital coupling corrections are also necessary in some cases.

In addition to the orbital magnetism, or the spin-orbital coupling effect; there are two more difficulties in the precise measurement of the formal oxidation state of the transition metal centers. The first one is the electron delocalization and use of non-innocent ligands; for instance, the neutral $[\text{Be}(\text{bipyridyl})_2]$ complex is expected to be diamagnetic with neutral Be as the central atom. However, the compound is actually paramagnetic due to the delocalization of two electrons from the metal to two bipyridyl chelate ligands. Therefore, if the ligand attached can accommodate one or more unpaired electrons in the appropriate molecular orbitals, then a paramagnetic complex with bivalent beryllium can also be obtained. In such cases, the assignment of formal oxidation state is carried out only after X-ray and EPR (electronic paramagnetic resonance) studies. The second anomaly can arise from metal-metal interactions leading to unexpected electron pairing that depends upon the nature of the ligand used. For example, vanadium hexa-carbonyl, $[\text{V}(\text{CO})_6]$, is paramagnetic with one unpaired electron. However, one of the neutral dinuclear clusters of vanadium, $[(\text{Diarsine})(\text{CO})_4\text{V}-\text{V}(\text{CO})_4(\text{Diarsine})]$, is diamagnetic in nature; even though the oxidation number of the vanadium is same in both. Therefore, a correct molecular weight measurement is pretty much necessary in such cases for fruitful interpretation of experimental results. Moreover, in magnetically concentrated materials, the ferromagnetic or antiferromagnetic interaction must be resolved by magnetic susceptibility measurements as a function of temperature or the field-strength.

➤ **Determination of the Electronic Configuration of Transition Metal Centre**

The energy difference between high-spin and low-spin configurations of a transition metal center in most of the complexes is pretty larger than the available thermal energy. Therefore, most of the complexes are either high-spin like $[\text{FeF}_6]^{3-}$, or low-spin like $[\text{Fe}(\text{CN})_6]^{3-}$, over a wide range temperature; which makes it quite easy to distinguish and designate their electronic configurations. Nevertheless, there are complexes in which the energy gap between high-spin and low-spin configurations is quite comparable to thermal energy, leading to a rearrangement of electron filling as a function of temperature or a slight variation of coordinative environment. The magnetic susceptibility measurements can be used to detect such phenomena as the number of unpaired electrons varies during the course of spin-equilibria under consideration. For instance, some square-planar complexes of Ni^{2+} (d^8) are diamagnetic but can be converted into paramagnetic tetrahedral or octahedral complexes with two unpaired electrons. Similarly, penta-coordinated Schiff's-base complexes of bivalent cobalt (d^7) also show high-low spin equilibria with three and one unpaired electrons, respectively.

Moreover, the magnetic susceptibility measurements can also be used to identify the exact orbital occupied by the unpaired electron, which depends upon the formal charge and the ligand field strength. For example, the orbital occupied by the unpaired electron in potassium (K) is $4s$, while the isoelectronic Ti^{3+} ion bears the unpaired electron in its $3d$ orbital; which is obviously due to the difference in Z_{eff} arising from changes in the formal charge. The surprisingly large magnetic moment of magnetically dilute $[\text{FeCl}_4]^{2-}$ complex may also be explained in terms of mixing of $3d^6$ configuration (4 unpaired electrons) with $3d^5 4s^1$ configuration due to change of Z_{eff} . The theoretical calculations suggest that the spin-orbital coupling can only increase the μ_{eff} of $[\text{FeCl}_4]^{2-}$ from 4.9 B.M. ($\mu_{\text{S.O}}$) to 5.1 B.M. at maximum, which is not even close to the experimentally observed magnetic moment (5.5 B.M.). The only rational explanation for such an exceptionally high magnetic moment relies on the σ -bond covalency between Fe^{2+} and Cl^- ligand which decreases the charge on the metal center to an extent where a considerable contribution from the $3d^5 4s^1$ configuration (6.92 B.M.) to the $3d^6$ configuration is possible. Furthermore, spin pairing due to covalent bonding, $[(\text{Diarsine})(\text{CO})_4\text{V}-\text{V}(\text{CO})_4(\text{Diarsine})]$, can also cause μ_{eff} to deviate significantly. Finally, magnetic exchange coupling (ferromagnetism and antiferromagnetism) in magnetically concentrated systems have the capacity to make everything a mess in the analysis; and therefore, requires a special treatment in such cases.

➤ **Determination of the Stereochemistry of Various Transition Metal Complexes**

One of the most valuable applications of paramagnetic susceptibility measurements is to find the stereochemical arrangements of different ligands attached to the transition metal center. The paramagnetic susceptibility measurements can be exploited in two ways to do so; the first one relies upon the determination of unpaired electrons, while the second one depends upon the determination of the exact magnitude of orbital magnetism. Both of the approaches with suitable examples are given below.

1. Stereochemistry from unpaired electrons: The possibility of stereochemistry-prediction of transition metal complexes from unpaired electrons arises from the fact the ligand coordination prefers to adapt the most symmetrical geometry unless the metal's d -electron cloud is not spherically symmetrical. For instance, d^0 and d^{10} configurations are spherically symmetrical; and therefore, results in linear, triangular, perfect tetrahedral,

trigonal bipyramidal, octahedral and pentagonal bipyramidal geometries for 2, 3, 4, 5, 6 and 7 coordination number, respectively. Furthermore, if the cloud is not spherically symmetric, slightly distorted octahedral, tetragonal, or square-planar geometries can be obtained. The slightly distorted octahedron refers to an octahedron with two of the bond lengths differing by less than 0.05 Å from the other four. On the other hand, the tetragonal geometry here refers to an octahedral structure with two coaxial bonds considerably longer in comparison to the other four.

Table 8. Expected stereochemistry of transition metal complexes using the number of unpaired d -electrons from crystal field theory.

Weak-field		Strong-field	
Unpaired electrons / Stereochemistry		Unpaired electrons / Stereochemistry	
0 (d^0)	Symmetrical	0 (d^0)	Symmetrical
1 (d^1, t_{2g}^1)	Slightly distorted octahedron	1 (d^1, t_{2g}^1)	Slightly distorted octahedron
2 (d^2, e^2 or t_{2g}^2)	Tetrahedron or slightly distorted octahedron	2 (d^2, e^2 or t_{2g}^2)	Tetrahedron or slightly distorted octahedron
3 (d^3, t_{2g}^3)	Perfect octahedron	3 (d^3, t_{2g}^3)	Perfect octahedron
4 ($d^4, t_{2g}^3 e_g^1$)	Square-planar or tetragonal	2 or 0 (d^4, t_{2g}^4 or e^4)	Slightly distorted octahedron or tetrahedral
5 ($d^5, e^2 t_{2g}^3$ or $t_{2g}^5 e_g^2$)	Tetrahedron or perfect octahedron	1 (d^5, t_{2g}^5)	Slightly distorted octahedron
4 ($d^6, t_{2g}^4 e_g^2$)	Slightly distorted octahedron	0 (d^6, t_{2g}^6)	Perfect octahedron
3 ($d^7, e^4 t_{2g}^3$ or $t_{2g}^5 e_g^2$)	Tetrahedron or slightly distorted octahedron	1 ($d^7, t_{2g}^6 e_g^1$)	Square planar or tetragonal
2 ($d^8, t_{2g}^6 e_g^2$)	Perfect octahedron	0 ($d^8, t_{2g}^6 e_g^2$)	Square planar or tetragonal
1 ($d^9, t_{2g}^6 e_g^3$)	Square planar or tetragonal	1 ($d^9, t_{2g}^6 e_g^3$)	Square planar or tetragonal
0 (d^{10})	Symmetrical	0 (d^{10})	Symmetrical

Electronic configuration from d^1 to d^9 may or may not have a spherically symmetrical cloud after complexation; therefore, a distortion is expected if any asymmetry is present. The distortion of an octahedron can occur in one of three ways; the first one involves an unsymmetrical filling of t_{2g} level (t_{2g}^1 , t_{2g}^2 , t_{2g}^4 and t_{2g}^5) which will have little effect on the octahedron is because of the indirect encounter metal between orbitals and the ligands, leading slightly distorted perfect octahedral geometry. On the other hand, if the asymmetry occurs in e_g level, then a tetragonal or square planar geometry may be produced depending upon whether the two ligands are repelled or lost along the z -axis, respectively. However, it is also worthy to mention that the diamagnetism in d^8 complexes does not confirm a square-planar geometry as is also possible in a compatible tetragonal geometry. Metal complexes with d^4 configuration can have zero, two, or four unpaired electrons yielding tetrahedral, tetragonal or square-planar geometries. Hence, we can say that the stereochemistry-chemistry prediction on the basis of the number of unpaired electrons is not very much reliable. Furthermore, consider the example of $[\text{Co}(\text{NO}_2)_6]^{4-}$ complex in which cobalt is present in +2 oxidation state. The experimental magnetic moment for this complex is about 1.9 B.M., which unexpectedly low for an octahedral complex; because the octahedral geometries of Co(II) usually have three unpaired electrons ($\mu_{\text{eff}} = 4.85\text{--}5.2$ B.M.), while the presence of one unpaired electron in Co(II) is generally related to a square pyramidal or square planar structure ($\mu_{\text{eff}} = 2.2\text{--}2.9$ B.M.). The low magnetic moment and octahedral geometry in such cases are generally ascribed to the π -backbonding from the metal to the attached ligand.

2. Stereochemistry from orbital magnet: The unpaired electron approach is somewhat more or less capable of shortlisting a metal complex into one of four classes. classify not sufficient to predict the exact stereochemistry of transition metal complexes. for instance, Ni(II) complexes which contain two unpaired electrons may be perfect octahedron, square bipyramidal, or tetrahedral; while the diamagnetic ones may be square-planar, square pyramidal, trigonal bipyramidal, or six-coordinate tetragonal. Nevertheless, this information can be used in conjugation with orbital magnetism approach for more precise and accurate results. For instance, consider the spin-free complexes of Co(II), in which the magnitude of $\mu_{\text{S.O}}$ is expected to be 3.88 B.M. Now because there is no orbital degeneracy present in Co(II) tetrahedral complexes; and of course, ignoring any spin-orbital coupling; its μ_{eff} is expected to be comparable to $\mu_{\text{S.O}}$ value. However, even after considering spin-orbital coupling, the effective magnetic moment would still be lower than 4.5 B.M. On the other hand, the presence of orbital degeneracy in spin-free octahedral complexes of Co^{2+} ($t_{2g}^5 e_g^2$) guarantees a huge orbital magnetic component, making μ_{eff} to touch a mark of 5.1 B.M. Therefore, the contribution of orbital magnetic moment can be used as a criterion for distinguishing between tetrahedral and octahedral complexes of bivalent cobalt. It should also be noted that other analytical methods must be used for final conclusion if magnetic moment is on the borderline (4.5–4.7 B.M.), or there is large distortion of the octahedron leading to splitting of the t_{2g} orbital set, or the value of crystal field splitting in tetrahedral complex is very small as it would lead to a large orbital magnetism due to spin-orbit coupling.

It must be clear again that the structure prediction discussed in this section is totally based upon the simple ligand field theory, which only considers the metals d -electron cloud-only and ignores the lattice effects or effect of ions surrounding the complex ion. The stereochemical arrangements in both spin-free and spin-paired complexes with various electron configurations are given below.

Table 9. The correlation between orbital magnetism and the stereochemistry of weak field transition metal complexes.

Stereochemistry	Supporting configuration	Unpaired electron	Examples	Expected magnetic profile considering orbital magnetism
Perfect octahedral	t_{2g}^3	3	$[\text{Cr}(\text{H}_2\text{O})_6]^{3+}$	Spin only moment of 3.88 B.M due to absence of orbital magnet.
	$t_{2g}^3 e_g^2$	5	$[\text{FeF}_6]^{3-}$	Spin only moment of 5.9 B.M due to absence of orbital magnet.
	$t_{2g}^6 e_g^2$	2	$[\text{Ni}(\text{H}_2\text{O})_6]^{2+}$	Spin only moment of 2.83 B.M is expected but the orbital contribution from $t_{2g}^5 e_g^3$ increases μ_{eff} due to spin-orbital coupling.
Slightly distorted octahedron	t_{2g}^1	1	$[\text{Ti}(\text{H}_2\text{O})_6]^{3+}$	Orbital contribution is expected yielding an effective magnetic moment exceeding 1.73 B.M.
	t_{2g}^2	2	$[\text{V}(\text{H}_2\text{O})_6]^{3+}$	Orbital contribution is expected yielding an effective magnetic moment exceeding 2.83 B.M.
	$t_{2g}^5 e_g^2$	3	$[\text{Co}(\text{H}_2\text{O})_6]^{2+}$	The orbital contribution is expected yielding an effective magnetic moment exceeding 3.88 B.M.
Square-planar or tetragonal	$t_{2g}^3 e_g^1$	4	MnF_3	Spin only magnetic moment of 4.9 B.M., and significant orbital magnetism is expected from $t_{2g}^2 e_g^2$ states.
	$t_{2g}^6 e_g^3$	1	$[\text{Cu}(\text{H}_2\text{O})_6]^{2+}$	Spin only magnetic moment of 1.73 B.M., and significant orbital contribution expected from $t_{2g}^5 e_g^4$.
			$[\text{Cu}(\text{NH}_3)_6]^{2+}$	Spin only magnetic moment of 1.73 B.M., and less orbital contribution expected from $t_{2g}^5 e_g^4$ states.

Table 9. Continued on the next page...

Regular tetrahedral geometry	e^2	2	$[\text{FeO}_4]^{2-}$	Spin only magnetic moment of 2.83 B.M. is expected with no orbital contribution
	$e^2 t_2^3$	5	$[\text{FeCl}_4]^-$	Spin only magnetic moment of 5.9 B.M. with no orbital magnet.
	$e^4 t_2^3$	3	$[\text{CoCl}_4]^{2-}$	Spin only magnetic moment of 3.88 B.M. with considerable orbital magnetism possibility from $e^3 t_2^4$

Though the orbital magnetism profiling is pretty useful in the determination of the stereochemistry of various transition metal complexes, the conclusive remarks with full confidence are put after using magnetic susceptibility measurement in conjunction with other physical data like electronic absorption spectra or X-ray diffraction profile.

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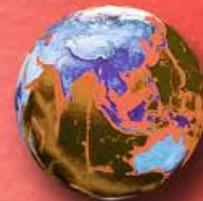
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ISBN: 978-81-938720-0-0



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