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Chemical applications of topology and group theory[☆] Part 35. Non-octahedral six-coordinate tris(dithiolene) and related complexes of the early transition metals

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Abstract

Group theory forbids either O_h octahedral or D_{3h} trigonal prismatic geometry for a six-coordinate early transition metal complex using a six-orbital sd^5 manifold thereby indicating that the observation of trigonal prismatic rather than octahedral metal coordination geometry is not a simple indication of the lack of *p* orbital participation in the chemical bonding. However, an experimentally observed C_3 geometry intermediate between octahedral and trigonal prismatic geometry is allowed by group theory for such an sd^5 manifold. Bicapped tetrahedral geometry, which is related to octahedral or trigonal prismatic geometry through combinations of various diamond-square-diamond processes, is also found in a few metal tris(dithiolenes) having saturated or benzenoid bridges between the donor sulfur atoms. The distortion of an octahedron to a trigonal prism in six-coordinate complexes of $d^{<4}$ early transition metals can result from a second-order Jahn–Teller effect involving splitting of the t_{1u} HOMO and the t_{2g} LUMO in order to allow mixing of the resulting e' orbitals in the trigonal prismatic geometry. This effect is favored when the ligands are strong σ-donors but weak π-donors and the metal is not too electropositive such as is the case with many metal tris(dithiolenes). The MS_2C_2 chelate rings in metal tris(dithiolene) complexes may be regarded as resonance hybrids of ethylenedithiolate and dithiodiketone canonical forms having different metal oxidation states. The stereochemical non-rigidity of trigonal prismatic metal tris(dithiolenes) observed experimentally by NMR requires interchange of the ligands on the top and bottom rings of the trigonal prism so that a simple trigonal twist through an octahedral intermediate is not adequate to account for this observation. A 'rotary electric switch' mechanism has been proposed for this process but rearrangement mechanisms through bicapped tetrahedral intermediates also appear reasonable. © 2001 Elsevier Science B.V. All rights reserved.

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1. Introduction

For several decades since the recognition of the nature of coordination compounds by Werner in the late 19th century, all six-coordinate metal complexes were assumed to be octahedral including the first tris(dithiolene) metal complex to be prepared, namely $Mo[S_2C_2(CF_3)_2]_3$ obtained in 1963 from $Mo(CO)_6$ and bis(trifluoromethyldithietene) [1]. However, when structural information on such metal tris(dithiolenes) became available through X-ray diffraction, this assumption was shown to be false. The first definitive demonstration of a non-octahedral structure for a metal tris(dithiolene) was the discovery of a trigonal prismatic structure for the rhenium complex $Re(S_2C_2Ph_2)_3$ by X-ray diffraction in 1965 [2,3]. This result was followed by similar structural studies on related metal tris(dithiolenes) and their selenium analogs [4]. In these early studies some compounds of the type $M[S_2C_2(CN)_2]_3^{2-}$ (M = V, Mo, W) were found to exhibit solid state structures intermediate between the trigonal prism and octahedron [5,6].

This originally unexpected observation of trigonal prismatic rather than octahedral geometry for the metal tris(dithiolenes), $M(S_2C_2R_2)_3^{z-}$, soon led to some theoretical studies in order to rationalize this unusual geometry for six-coordination. Thus molecular orbital

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calculations on the metal tris(dithiolenes) were performed as early as 1966 [7,8]. A more general theoretical study on trigonal prismatic as well as bicapped tetrahedral alternatives to octahedral coordination in six-coordinate metal complexes by Hoffmann et al. was reported about a decade later [9].

A major development during the past decade has been the discovery of non-octahedral structures for sulfur-free d^0 six-coordinate transition metal alkyls such as MMe_6 (M = W [10] and Re [11]) and $ZrMe_6^{2-}$ (Ref. [12]). This has led to renewed theoretical interest in non-octahedral six-coordinate metal complexes including ab initio computations on six-coordinate early transition metal methyls and hydrides [13–16], development of the theory of the second order Jahn–Teller effect to rationalize non-octahedral geometries [13,15], and a group-theoretical analysis of possible geometries for six-orbital sd^5 manifolds in early transition metal hydride and alkyl chemistry [17]. This paper examines various aspects of the non-octahedral geometries of metal tris(dithiolenes) in light of these recent theoretical developments as well as recent experimental information on six-coordinate complexes of early transition metals with dithiolate and related ligands.

Table 1
The irreducible representations for the hybrid orbitals corresponding to configurations for coordination number six based on an sd^5 six-orbital manifold (irreducible representations requiring p orbitals are enclosed in boxes)

Configuration	G	Γ_σ^*
Intermediate between a trigonal prism and an octahedron	C_3	$2A(s, z^2)+2E(x^2-y^2, xy; xz, yz)$
Trigonal prism	D_{3h}	$A_1(s, z^2)+E'(x^2-y^2, xy)+\boxed{A_2''(z)}+E''(xz, yz)$
Octahedron	O_h	$A_1(s)+E_g(z^2, x^2-y^2)+\boxed{T_{1u}(x, y, z)}$
Bicapped tetrahedron	C_{2v}	$3A_1(s, x^2-y^2, z^2)+B_1(xz)+\boxed{2B_2(yz, y)}$

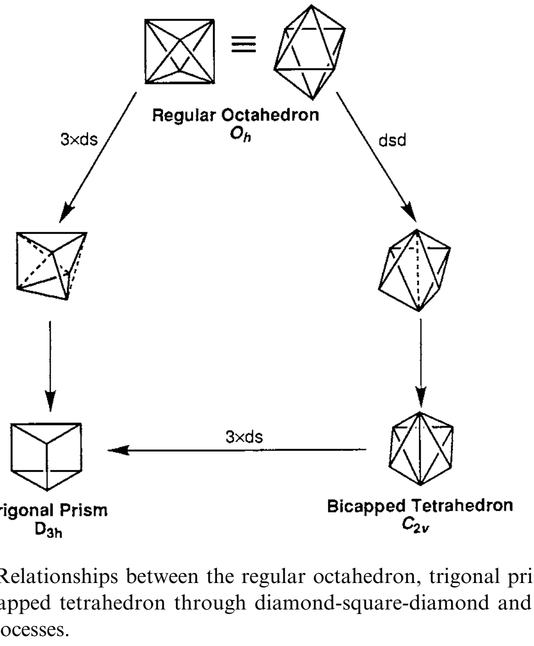

Fig. 1. Relationships between the regular octahedron, trigonal prism, and bicapped tetrahedron through diamond-square-diamond and related processes.

2. Avoiding the use of *p* orbitals: six-coordinate complexes with the six-orbital *sd*⁵ manifold

The six-orbital sd^5 manifold may be found in simple homoleptic hydrides and alkyls of the early transition metals in which the np orbitals are of significantly higher energy than the ns and $(n-1)d$ orbitals. The following bonding concepts have been proposed by Landis et al. [17] for such compounds: (a) only s and d orbitals are used to form hybrid bond orbitals; (b) the hybrid orbitals have maximal s character (or sd^{n-1} hybridization when making n bonds); (c) lone pairs are placed in pure d orbitals; (d) three-center four-electron (3c-4e) bonds are used when the central metal atom has more than 12 valence electrons.

The absence of gerade atomic orbitals in the sd^5 manifold severely limits the geometries for six-coordination (Table 1). Thus an inversion center is not possible for six-coordinate polyhedra using an sd^5 manifold and only two-center two-electron (2c-2e) bonds. This immediately rules out the O_h octahedron for such six-coordinate complexes and thus suggests non-octahedral geometries for early transition metal complexes without p orbitals in the valence orbital manifold. However, a more detailed study of the relevant group theory (Table 1) indicates that the D_{3h} trigonal prism is also not possible with an sd^5 manifold without using p orbitals. Thus more severe distortion of an octahedron with more drastic symmetry reduction is necessary to form a six-coordinate polyhedron using the six-orbital sd^5 manifold.

Fig. 1 depicts the distortions of an octahedron that are relevant to the discussion in this paper. Conversion of an octahedron to a trigonal prism involves a triple diamond-square (ds) process known as the Bailar twist in the case of tris(bidentate) chelate complexes of D_3 overall symmetry [18]. The progress of the Bailar twist from the trigonal prism to the octahedron can be measured by the angle θ where $\theta = 0$ and $\theta = 60°$ correspond to the trigonal prism and octahedron, respectively (Fig. 2). Intermediate stages of the Bailar twist (e.g. $\theta = 30°$) have only C_3 symmetry rather than the higher D_{3h} and O_h symmetries of the trigonal prism and octahedron, respectively, and thus can be formed by the sd^5 manifold (Table 1). This may explain why such twisted intermediate geometries have been found experimentally for the *saturated* dithiolate complexes $Nb(S_2C_2H_4)_3^-$ ($\theta = 30$ to $33°$) [19,20], $Nb(S_2C_3H_6)_3^-$

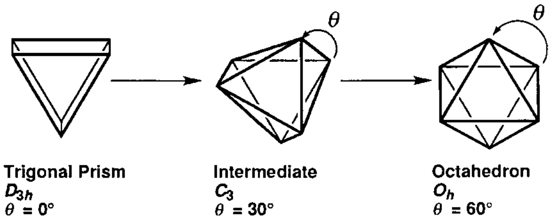

Fig. 2. The 'Bailar twist' interconverting the octahedron and trigonal prism through a C_3 intermediate showing the definition of the angle θ.

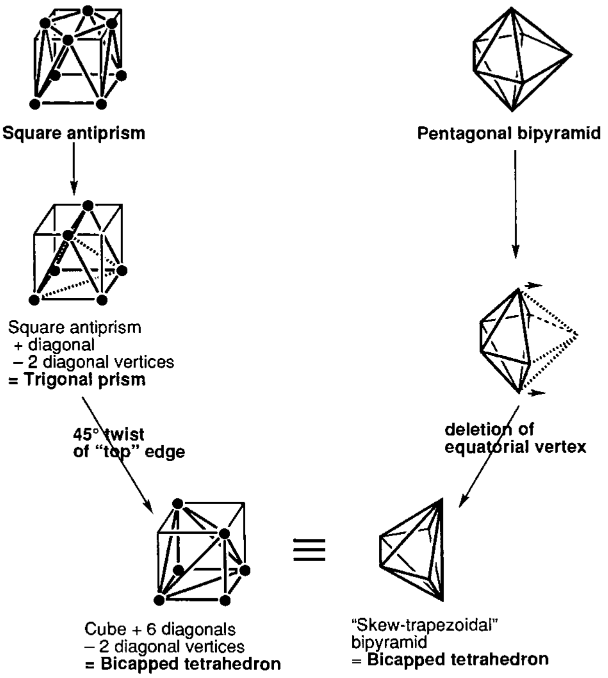

Fig. 3. Some relationships between the square antiprism, trigonal prism, bicapped tetrahedron, 'skew-trapezoidal' bipyramid, and pentagonal bipyramid.

(θ = 18°) [21], and $Ta(S_2C_7H_{10})_3^-$ (θ = 39°) [22]. The recently reported [23] monodentate d^0 hexakis(arylthiolato) zirconates $[Li(tmeda)]_2[Zr(SC_6H_4\text{-}4\text{-}R)_6]$ were found to have twist angles (θ) of 30° for $R = OCH_3$ but only 9° for $R = CH_3$.

Another possible distortion of an octahedron is the single dsd process converting the O_h octahedron to the C_{2v} bicapped tetrahedron (Fig. 1). This polyhedron rather than an octahedron, trigonal prism, or intermediate C_3 polyhedron is found in the anionic d^0 tris(-dithiolate) complexes $Nb(S_2C_7H_{10})_3^-$ (Ref. [22]) and $Ti(S_2C_6H_4)_3^{2-}$ (Ref. [24]). Furthermore, the 'distorted trigonal prismatic coordination' of $Ta(S_2C_6H_4)_3^-$ (Ref. [25]) appears to be close to a bicapped tetrahedron.

The bicapped tetrahedron has been described [24] as a 'skew-trapezoidal bipyramid' derived from a pentagonal bipyramid by deleting an equatorial vertex (Fig. 3). Alternatively, the bicapped tetrahedron can be obtained by taking a cube, adding all six diagonals to give four vertices each of degrees 4 and 5, followed by removing two diagonally situated degree 4 vertices along with all of the associated edges, where the degree of a vertex is the number of edges meeting at that vertex. If the two vertices remaining in the face of the original cube losing two vertices are allowed to rotate by 45° relative to the remaining four vertices, this bicapped tetrahedron is then converted to a trigonal prism and vice versa. This completes the following cycle of idealized six-vertex polyhedra:

$$\text{octahedron } (O_h) \xrightarrow[\text{3 dsd}]{\text{Bailar twist}} \text{trigonal prism } (D_{3h}) \xrightarrow{\text{45° edge rotation}} \text{bicapped tetrahedron } (C_{2v}) \xrightarrow{\text{dsd}} \text{octahedron}$$

Fig. 3 depicts the relationships between the pentagonal bipyramid, 'skew-trapezoidal bipyramid', bicapped tetrahedron, and trigonal prism. In this figure the bicapped tetrahedron can thus be seen to be related to the trigonal prism by a 45° twist of a suitably chosen edge. Experimentally, the approximate 45° twist of an edge in an ideal trigonal prism is seen in the observed structure of $Ta(S_2C_6H_4)_3^-$ (Ref. [25]), whose geometry can thus best be modeled by a bicapped tetrahedron.

3. Second order Jahn–Teller distortion of the octahedron to the trigonal prism

Since neither the octahedron nor the trigonal prism can be formed by the sd^5 manifold, the use of this manifold free from p orbitals cannot rationalize the occurrence of trigonal prismatic coordination in d^0 metal complexes such as the metal tris(dithiolenes). However, Albright et al. [13,15] have shown how the second-order Jahn–Teller effect can rationalize the occurrence of trigonal prismatic rather than octahedral coordination for certain types of early transition metal complexes including the metal tris(dithiolenes).

Consider the octahedron and trigonal prismatic molecular orbital diagrams for d^0 ML_6 complexes (Fig. 4). In octahedral (O_h) symmetry the highest occupied molecular orbital (HOMO) and lowest unoccupied molecular orbital (LUMO) are of different irreducible representations (t_{1u} and t_{2g}, respectively) and thus cannot mix. However, conversion of an octahedron to a trigonal prism results in splitting the t_{1u} HOMO to $a_2'' + e'$ and the t_{2g} LUMO to $a_1' + e'$. The two doubly degenerate orbitals of e' symmetry can mix thereby lowering the energy of the filled e' orbitals derived from the HOMO by a parameter Δ. The stabilization of a d^n $(0 \leq n \leq 4)$ trigonal prismatic complex over an octahedral complex by this mechanism is 4Δ for a d^0, d^1, or d^2 complex. Note that for the d^1 and d^2 complexes the 'extra' electron or two, respectively, go into the non-degenerate a_1' orbital.

This type of Jahn–Teller effect is called a second order Jahn–Teller effect since the t_{2g} LUMO is not directly involved in the M–L σ-bonding in contrast to the e_g orbital so that such Jahn–Teller effects do not affect the M–L distance to a significant extent. This second-order Jahn–Teller distortion will be favored by a small t_{1u}–t_{2g} energy gap since the stabilization provided to orbitals originating from the t_{1u} set is inversely dependent upon this energy difference [26]. A small t_{1u}–t_{2g} energy gap is favored when the ligands are strong σ-donors but weak π-donors and the metal is not too electropositive [27]. These conditions are met with the d^0 metal tris(dithiolenes) discussed in this paper but not with d^0 metal complexes of strong π-donor ligands such as halides (e.g., MoF_6 and WF_6) [28,29] or alkoxides (e.g., $W(O_2C_2H_4)_3$ or $W(O_2C_2Me_4)_3$) [30,31], which exhibit octahedral rather than trigonal prismatic structures.

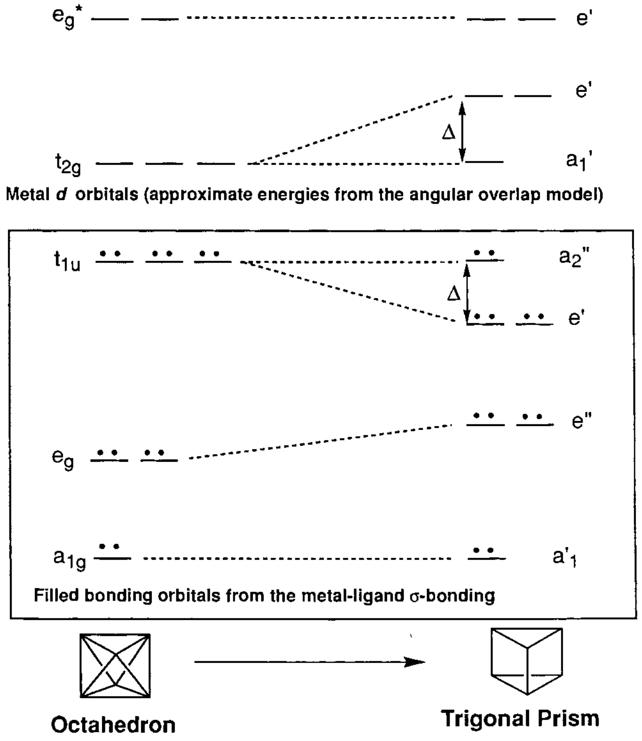

Fig. 4. Changes in the molecular orbitals in going from the octahedron to a trigonal prism for a d^0 metal complex showing the definition of Δ as the splitting of the triply degenerate t_{2g} and t_{1u} orbitals by the second-order Jahn–Teller effect.

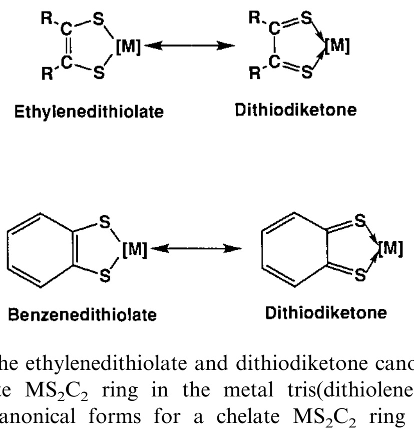

Fig. 5. (a) The ethylenedithiolate and dithiodiketone canonical forms for a chelate MS_2C_2 ring in the metal tris(dithiolenes). (b) The analogous canonical forms for a chelate MS_2C_2 ring in 1,2-benzenedithiolate complexes showing how the dithiodiketone canonical form destroys the six π-electron system of the benzenoid ring.

4. The role of the unsaturated ethylene bridges in the metal tris(dithiolenes): ethylenedithiolate versus dithiodiketone ligands

The nature of the unsaturated metal chelate ring in the metal tris(dithiolenes) is of interest since the MS_2C_2 chelate rings may be regarded as a resonance hybrids between ethylenedithiolate and dithiodiketone canonical forms (Fig. 5(a)). The ethylenedithiolate structure is derived from a dianionic dithiolene ligand whereas the dithiodiketone structure is derived from a neutral dithiolene ligand so that each canonical form necessarily has different partitions of electrons between the ligand and metal atom thus corresponding to different metal formal oxidation states. Thus in the extreme cases molybdenum tris(dithiolenes) of the type $Mo(S_2C_2R_2)_3$ can be formulated with the central molybdenum atom in the + 6 or 0 formal oxidation states depending on whether all of the three dithiolate ligands are in the ethylenedithiolate or dithiodiketone canonical forms. In this connection recent ^{95}Mo- and ^{183}W-NMR studies [32] on the tris(dithiolenes) $M(S_2C_2R_2)_3$ (M = Mo, W; R = various combinations of hydrogen and aryl groups) indicate that the central metal atoms are highly deshielded suggesting the + 6 formal oxidation state with ethylenedithiolate rather than dithiodiketone ligands. However, the dithiodiketone canonical form clearly must make some contribution to the resonance hybrid since the known [33] trigonal prismatic $V(S_2C_2Ph_2)_3$ would clearly have the impossible formal vanadium oxidation state of + 6 if all three dithiolene rings were dianionic ethylenedithiolate ligands. The opposite extreme formulation of the metal tris(dithiolenes) as tris(dithiodiketone)metal(0) complexes would make them sulfur analogs of known tris(butadiene)metal(0) complexes of the general type $M(C_4H_6)_3$ (M = Mo, W). In this connection the structures of $Mo(C_4H_6)_3$ (Refs. [34,35]) and the tris(2,3-dimethylbutadiene)metal(0) analogs $M(C_4H_4Me_2)_3$ (Ref. [36]) have all been shown by X-ray diffraction to have trigonal prismatic coordination of the six butadiene C=C bonds about the central metal atom.

A variety of six-coordinate geometries are found in tris(1,2-benzenedithiolato) complexes of the d^0 transition metals, in which the dithiodiketone canonical form is unfavorable since it destroys the π-electron sextet of the benzenoid ring (Fig. 5(b)). Trigonal prismatic ge-

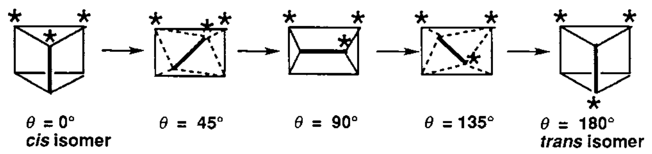

Fig. 6. The ‘rotary electric switch’ mechanism first proposed by Argyropoulos et al. [41]. The bold edge (ligand) is the one undergoing the rotation and the angle θ represents the amount of the rotation. Note that θ values of 90° and 180° correspond to trigonal prism isomers where the ligands on the top and bottom triangular faces have been interchanged.

ometries are found for $Mo(S_2C_6H_4)_3$ (Ref. [37]) and $Nb(S_2C_6H_4)_3]^-$ (Ref. [38]) whereas the isoelectronic complexes $[Ti(S_2C_6H_4)_3]^{2-}$ (Ref. [24]) and $[Ta(S_2C_6H_4)_3]^-$ (Ref. [25]) exhibit bicapped tetrahedral geometry.

5. Stereochemical non-rigidity in tris(dithiolene)metal complexes

Octahedral six-coordinate complexes are stereochemically rigid at ambient conditions. because of the high activation energy of interconversion of octahedral isomers through processes such as the Bailar twist (Fig. 2) [39]. About 30 years ago Muetterties [40] predicted trigonal prismatic coordination complexes to be stereochemically non-rigid. This prediction was verified in 1996 by Katakis et al. [41] using temperature dependent proton and metal NMR studies on unsymmetrical $M(S_2C_2RR')_3$ derivatives ($M = Mo$ and W; $R \neq R' = H$ or p-XC_6H_4 where $X = MeO$, Me, Cl, Br, H). The various $M(S_2C_2RR')_3$ isomers were found to interconvert at room temperature at a rate rapid compared with the NMR time scale so that only single dithiolenic proton resonances were observed at room temperature. However, at −40°C distinct resonances for the *cis* and *trans* isomers could be observed.

Interconversion of the *cis* and *trans* isomers of a trigonal prismatic tris(dithiolene)metal chelate is not possible by a Bailar twist mechanism (Fig. 2), which does not interchange the ‘top’ and ‘bottom’ triangles of the trigonal prism. Katakis et al.[41] have suggested a ‘rotary electric switch’ mechanism (Fig. 6) to account for the easy interconversion of the *cis* and *trans* isomers of the trigonal prismatic tris(dithiolene)metal chelates at ambient conditions. Also interconversion of trigonal prismatic isomers by square-diamond-square processes involving a bicapped tetrahedral intermediate (see Fig. 1) could interchange ligands on the top and bottom triangular faces of the trigonal prism.

6. Summary

Group theory forbids either O_h octahedral or D_{3h} trigonal prismatic geometry for a six-coordinate early transition metal complex using a six-orbital sd^5 manifold. This indicates that the observation of trigonal prismatic rather than octahedral metal coordination geometry is not an indication of the lack of p orbital participation in the chemical bonding. However, a C_3 geometry intermediate between octahedral and trigonal prismatic geometry is allowed by group theory for such an sd^5 manifold. Such metal coordination geometries are observed for some d^0 transition metal tris(dithiolates), particularly those with a saturated carbon chain between the donor sulfur atoms. In addition, there are a few examples of bicapped tetrahedral geometry in tris(dithiolates) having a saturated carbon chain or benzene ring between the donor sulfur atoms.

Distortion of an octahedron to a trigonal prism in six-coordinate complexes of $d^{<4}$ early transition metals can result from a second-order Jahn–Teller effect involving splitting of the t_{1u} HOMO and the t_{2g} LUMO. This effect is favored when the ligands are strong σ-donors but weak π-donors and the metal is not too electropositive such as is the case with many metal tris(dithiolenes).

The MS_2C_2 chelate rings in metal tris(dithiolene) complexes may be regarded as resonance hybrids of ethylenedithiolate and dithiodiketone canonical forms having different metal oxidation states. This indicates an important but indirect role of the ligand C=C double bonds in the metal-ligand bonding in such complexes.

Experimental NMR work indicates trigonal prismatic metal tris(dithiolene) complexes to be stereochemically non-rigid with interchange of the donor atoms on the ‘top’ and ‘bottom’ triangles of the trigonal prism occurring on the NMR time scale at room temperature. A Bailar twist (triple square-diamond-square process) is not sufficient to account for such observations. A more complicated mechanism such as a ‘rotary electric switch’ mechanism involving twisting of a single edge or a mechanism involving a bicapped tetrahedral intermediate is required.

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