Another plausible transition state is a gauche form of the chelate ring with a cyclohexane ring in a half-chair conformation. The $\Delta H^{\ddagger}$ values for *cis*-1,2-, *trans*-1,3-, and *cis*-1,4-dimethylcyclohexane have been reported to be 42.30, 43.85, and 44.10 kJ mol^{-1} respectively.[56] These values are close to those for cyclohexane: 43.1–48.1 kJ mol^{-1}.[57–59] Eclipsing of vicinal methyl groups, which must take place at some stage in the conformational change of cyclohexane, can occur during pseudorotation of the twist-boat form via the boat form, which is about 19 kJ mol^{-1} lower[55] in energy than the half-chair form, in a manner similar to that for 1,2- and 1,2,4,5-substituted cyclohexanes reported by Wolfe and Campbell.[60] An organic counterpart of the *cis*-chxn chelate is *cis*-bicyclo[4,3,0]nonane (*cis*-bcn), and its $\Delta H^{\ddagger}$ for the conformational change has been reported to be 37.0 kJ mol^{-1}.[61] The molecular force field calculation has shown that the cyclohexane ring takes a half-chair form at the transition stage, where the dihedral angle of the C^9–C^1–C^2–C^3 moiety is 125°.[61] The decrease in $\Delta H^{\ddagger}$ observed for *cis*-bcn compared with cyclohexane is attributable to the strain energy of the cyclopentane ring, which is estimated as 5.4 kJ mol^{-1} on the basis of the strain energies of *cis*-bcn (41.1 kJ mol^{-1}),[62] cyclohexane (5.65 kJ mol^{-1}),[62] and cyclopentane (30.1 kJ mol^{-1}),[62] assuming that the strain energy of the cyclopentane ring is maintained constant during the conformational change. Since the $\Delta H^{\ddagger}$ observed for the *cis*-chxn chelate is similar in magnitude to the values for cyclohexane and dimethylcyclohexanes, the half-chair form of the cyclohexane ring is another plausible transition state.

We have reported here some basic data with respect to the conformational interconversion between δ and λ forms of five-membered chelate ring. Since the $\Delta G^{\ddagger}$ values so far reported for the $\delta \rightleftarrows \lambda$ interconversion[33] are almost independent of the variation of the central metal ion, the results in this work are expected to be valid for other metal chelates. This study also demonstrated that the application of paramagnetic shift to NMR line-shape analysis is useful for fast dynamic process, e.g., the conformational interconversion.

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Energetics of Metal Complex Isomerization Reactions

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A stereochemical approach to transition-metal complex geometry is coupled to a recently developed model for the energetics of such systems to enable rearrangement mechanisms to be compared and the most favorable one to be determined. A useful notation for discussing such reactions is also developed.

A recently developed model, which enables a semiquantitative determination of the most stable geometry of an ML$_n$ system,[1] can equally well be applied to proposed transition states in rearrangement processes to determine the one of lowest energy. In conjunction with a classical symmetry selection rule procedure (CSRP)[2,3] for determining reaction mechanisms, we should be able to understand why some transition-metal complexes are fluxional (i.e. isomerize on an NMR time scale), others rearrange on longer time scales (isomerization), and others are effectively stable. In addition, it should be possible to determine the relative probabilities of proposed mechanisms. It has already been possible[4] to determine the conditions under which the Ray–Dutt tris chelate metal complex isomerization mechanism is favored over the Bailar twist mechanism.

The model for the energetics of coordination compounds of the type ML$_n$ resulted from considering the following expression for the energy of the molecule

$$E(\mathrm{ML}_n) = \sum \text{M–L} + \sum \text{L–L} + \sum \text{el} \qquad (1)$$

where $\sum$M–L includes all the M–L bonding energy, $\sum$L–L includes the ligand–ligand interactions and $\sum$el includes the energetic contribution made by the nonbonding electron density. For our current purposes, we shall simply report the conclusions appropriate for the systems we are studying and illustrate them by application. Further details can be found in ref 1. (i) In general if the M–L bond length remains constant at its "optimal length" then $\sum$M–L is approximately independent of the orientation of the ligands, L. (ii) $\sum$el (or its change as a function of different L orientations) is not the dominant contribution to the energy for bond angles greater than 90°. (The conclusions of our study contrast with the valence-shell electron-pair repulsion model of molecular geometry which assumes that a very significant stabilizing factor is the formation of localized electron pairs from any nonbonding valence electrons.) (iii) $\sum$L–L can be expressed as the sum of a short-range L–L repulsive potential energy (most conveniently approximated by a hard-sphere potential), plus an L–L attractive r^{-6} dispersion energy (where r is the L–L distance), plus (if appropriate) a r^{-1} charge–charge interaction energy (which is usually repulsive). In the present work, we shall exclude from our explicit consideration systems where charge–charge interactions are significant (in conjunction with ref 1 the extension to include such systems is straightforward). Thus, we shall consider systems where the ligand orientation is such that it maximizes the dispersive ligand attraction subject to hard-sphere radii that define the smallest L–L distance. Further details of how to estimate dispersion energies are given in ref 1. Two points should be noted about this approach to energetics. First, it was developed by considering all the interactions within a molecule and is not merely an empirical fit to a postulated form. Second, the *attractive* dispersion energies proved to play a large role in the determination

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of the geometries that ML_n systems adopt. This is in contrast to, for example, the work of Keppert,[5] where repulsive forces are assumed to be the dominant determiners of geometry.

It follows that ligands which are large compared with M–L bond lengths tend toward close packed (closo) ligand geometries, and may even have M–L bond lengths longer than their optimal length due to L–L hard-sphere repulsion. Many ML_5 trigonal-bipyramidal systems, where the axial bond lengths are slightly longer that the equatorial bond lengths (since equatorial ligands are less closely packed), illustrate this point. Ligands that are small compared with M–L bond lengths will favor geometries with short interligand distances and "empty spaces". The geometries with "empty spaces" may be described as nido (one hole), arachno (two holes), or hypho (three holes) forms of higher order closo polyhedra. It should be noted that the positions of the "holes" in the ligand polyhedra of transition-metal complexes do not necessarily correspond to the geometries observed in cluster chemistry. In this work they always correspond to the removal of a vertex with connectivity of four. We shall use the notation $\{n,0\}$ to denote a closo form of ML_n, $\{n + 1, -1\}$ for its nido form, etc. For example an AL_5 square-based pyramid can be viewed as a nido octahedron, $\{6,-1\}$, and an AL_4 square-planar system can be denoted an arachno octahedron, $\{6,-2\}$. It is, however, not always possible to give a unique label to a particular structure. In such a case this method of visualizing the structure becomes less helpful.

Some general comments about isomerization mechanisms for ML_n systems follow directly from the above discussion. Rearrangement reactions proceed by mechanisms that, in order of importance, retain L–L distances of at least twice the L hard-sphere radius, increase M–L bond lengths as little as possible, and involve the least loss of dispersion stabilization in the transition state (i.e., have L–L distances as close to the hard-sphere value as possible).

Closo geometries cannot rearrange without lengthening some or all of the M–L bonds. We can therefore imagine a closo reactant rearranging by stretching M–L bonds, then relaxing to a ligand geometry ($\{n + 1,-1\}$ or $\{n + 2,-2\}$ or $\{n + 3,-3\}$ depending on the amount of M–L stretch) that is more favorable for the longer M–L bond lengths, and then proceeding to the product. (It is important to recognize however, that a rearrangement would not proceed in this disjointed fashion, but rather with the stretching and relaxing processes happening in concert.) Clearly the bonds will stretch only enough to allow a rearrangement to take place. Similarly, $\{n + 1,-1\}$ ML_n molecules will rearrange by using mechanisms via $\{n + 1 + i,-1 - i\}$, $i = -1$ or $1 > 0$, favoring the smallest i value that leads to a rearrangement. If $\{n,0\}$ (i.e. $i = 1$) is a transition state on a rearrangement pathway (rather than simply a small distortion from the reactant), then it will be the favored mechanism as it requires loss only of some dispersion stabilization and no bond stretching. If, as in trigonal-bipyramidal systems, some bonds can lengthen and some shorten, then the rearrangement is not as energetically expensive as in, e.g., an octahedron where all M–L bonds must stretch. The remainder of this paper serves to illustrate these general comments.

One of the most difficult parts of studying isomerization reactions is determining the mechanisms that are worth examining. For systems with a reasonable amount of symmetry, such as the ones being studied here, the classical (or geometrical) symmetry selection rule procedure (CSRP)[2-4] is a useful tool in the determination of symmetry-allowed concerted reaction mechanisms. Its advantages compared with previous symmetry analyses is that it can be systematically followed from a reactant to generate all symmetry-allowed (in a strict geometric sense) one-step mechanisms. It can also be used to determine the allowed steps in multistep reactions. Although the development of the CSRP is somewhat formalistic, the application is straightforward. One proceeds as follows. First, one draws a reactant molecule, R, and the product molecule(s) of interest, P, labeling their atoms. (In practice one enumerates all distinct products.) For each R,P pair one then determines their common symmetry operations in a labeled atom sense. These and only these symmetry operations, which form the group G_{RP}, are retained by the molecule along the reaction path. If R and P have different template shapes, then the transition state, T, has symmetry G_{RP}. If R and P have the same template shape, then one redraws R and P so as to align their *common* symmetry elements and then determines the product-generating operation $\mathcal{L} = \mathcal{R}\boldsymbol{p}$, where $\mathcal{R}$ is a point symmetry operation and $\boldsymbol{p}$ is a permutation symmetry operation, such that $\mathcal{L}R = P$ and $\mathcal{L}P = R$. $\mathcal{L}$ then determines T as $\mathcal{L}T = T$; i.e., when $\mathcal{L}$ operates on T, all atoms remain in the same position. We now have a concerted symmetry-allowed rearrangement path if the following three criteria are satisfied: (1) if the above procedure can be followed, i.e., if a T can be constructed; (ii) if there is a *normal mode* of R that reduces its symmetry to G_{RP}; (iii) if there is a *normal mode* of P that reduces its symmetry to G_{RP}. Otherwise no concerted mechanism exists between R and P.

The CSRP is immediately useful for high-symmetry systems and is particularly suited to the closo geometries. For the lower symmetry nido etc. systems the CSRP, coupled to the concept of template symmetry restrictions,[3] is one source of potential rearrangement mechanisms. (Template symmetry restrictions are where the mechanisms deduced for a high-symmetry molecule by using the CSRP can be transferred to lower symmetry systems with the same template subject to two provisos. (i) The symmetry restrictions are less severe, so a mechanism that was not possible in one step for the high-symmetry system because T could not be constructed may be possible for the lower symmetry case. (ii) A mechanism that was concerted for the high-symmetry case and proceeded by a degenerate vibration may become strictly multistep, though looking much the same, for the lower symmetry molecule since the degenerate vibration is split into nondegenerate components.)

We shall limit our case studies to non-bond-breaking rearrangements of ML_6, ML_4, ML_5, and $M(LL)_3$, where LL denotes a bis chelate. The principles developed for these systems apply equally well to other systems and to bond-breaking mechamisms where intermediates of lower stoichiometry must be considered. Symmetry-allowed (in a strict geometric sense, rather than a state sense) mechanisms for the isomerizations of ML_6, ML_4, ML_5, and $M(LL)_3$ have already been determined[2,3] by using the classical (geometrical) symmetry selection rule procedure that was outlined above. Throughout this work, we are assuming that enthalpic rather than entropic factors dominate the relative changes in Gibbs free energy. This is unlikely to be a problem for mechanisms where no bonds are broken.

ML_6

We shall begin the case studies by looking at the isomerization of octahedral ML_6. Although more complicated than some of the smaller closo systems, it is best to begin with octahedron, since systems such as the square plane are $\{6,-n\}$ systems and so may react by lower symmetry analogues of the octahedral mechanisms. Most ML_6 systems are octahedral (or only slightly distorted from it) so we consider only $\{6,0\}$.

Application of the CSRP suggests that there are four types of mechanisms for a $\{6,0\}$ octahedral system. However, transition states cannot be constructed for three of these (Figure 1), mechanisms 1.a, 1.b, and 1.d) because the symmetry requirements imposed by the conserved symmetry elements in the reactants and products require the superposition of atoms in T. All have, however, been illustrated in Figure 1 as they may become operative for lower symmetry systems. The fourth mechanism, mechanism 1.c, which is potentially concerted, is a higher symmetry analogue of the Bailar and Ray–Dutt twists. Its transition state is a trigonal prism $\{9,-3\}$ structure. This mechanism requires some stretching of M–L bonds (8%) and a consequent loss of dispersion stabilization (19%). It is therefore probably of relatively high energy and not necessarily fluxional, though possibly feasible on a longer time scale. If a mechanism could be found proceeding via either $\{7,-1\}$ or $\{8,-2\}$ transition states, it would be energetically less

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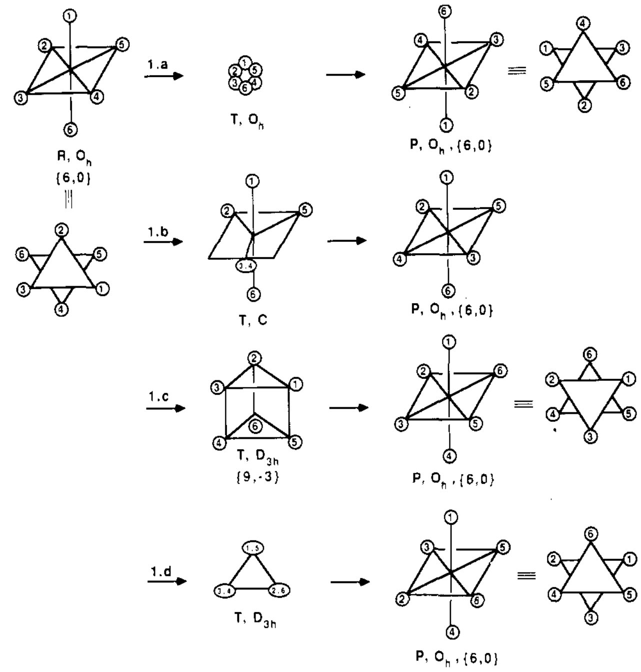

Figure 1. Concerted rearrangement mechanisms for closo {6,0} octahedral complexes.

expensive. However, these structures are not on a direct route for isomerization, so multistep processes must be considered. For example, one might consider the transition-metal complex analogues of the symmetry-allowed mechanisms proposed in ref 6 for the rearrangement of M_6 clusters. The cluster mechanisms proceed via {7,−1} transition states and intermediates for the rearrangement of ML_6. Of the two in ref 6 that lead to products distinct from R, the first would, in a transition-metal complex, involve breaking a bond. The second is, however, a possible mechanism that would probably be somewhat lower in energy than the concerted twists since M–L and L–L distances are slightly less stretched. It is interesting to note, however, that it is simply a distortion of the twist mechanism into a three-step process.

Due to the equivalence of ligating sites in an octahedron, NMR cannot be used to determine whether a ML_6 compound is fluxional. However, the fact that more than one isomer can be isolated[5] for octahedral complexes with different ligands indicates that many octahedral complexes are not fluxional and in fact rearrange only on slow or very slow time scales.

ML_4

The simplest examples we shall consider are the isomerizations of ML_4 molecules. A tetrahedron is the least crowded or closo geometry that four ligands can adopt around a central atom. As it has less dispersion stabilization than any other four coordination structure, if a tetrahedral structure is adopted, the L–L distance will be approximately twice the L hard-sphere radius, viz. $2\rho_R[\sin(\tau/2)]$ where ρ_R is the reactant M–L distance (it may be larger than optimal) and τ is the tetrahedral angle. Proceeding as suggested above, we shall consider mechanisms via {5,−1} (cf. Figure 3 with atom 1 omitted) and {6,−2} (square-plane) geometries.

Although distortion to {5,−1} would require less M–L bond stretching than to {6,−2}, it is not a reaction path as it is not a direct route to a different tetrahedral structure. The energetically most favorable reaction path therefore proceeds via {6,−2}, which is in fact the only mechanisms that the CSRP indicates may be concerted. Due to the bond stretching required to go from R to T (~15%), as well as the 33% loss of dispersion stabilization, T of Figure 2, mechanism 2.a, is a transition state not an intermediate, so the mechanism is concerted. A bond-breaking mechanism may be faster than this concerted intramolecular mechanism. In any case, it is reasonable to conclude that tetrahedral reactants are not fluxional.

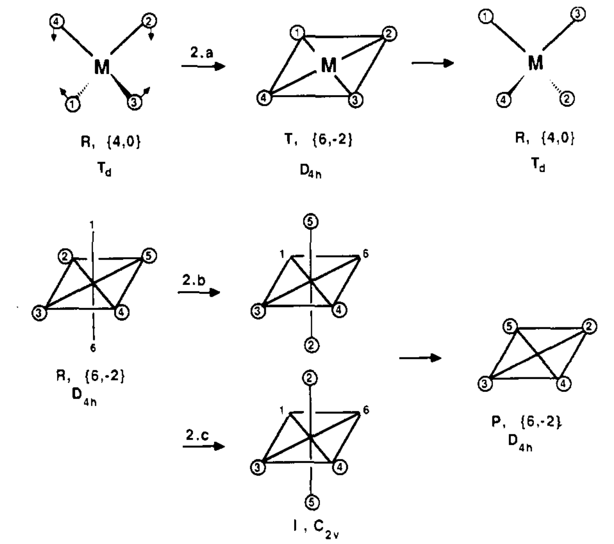

Figure 2. Rearrangement mechanisms for {4,0} and {6,−2} complexes. Atoms in dotted circles are those not present.

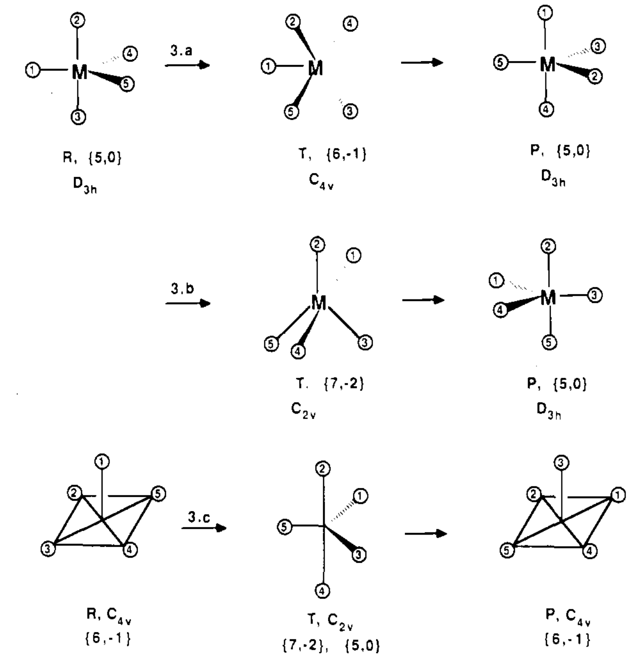

Figure 3. Rearrangement mechanisms for five-coordinated complexes.

The other ML_4 limiting structure is the square plane. By the CSRP no concerted square-planar to square-planar mechanism is possible. Our search for mechanisms for this arachno–6 structure can therefore proceed in two directions, the first to look for mechanisms via {5,−1}, or {4,0} geometries and the second to find low-symmetry analogues of the octahedral rearrangements of Figure 1.

If we begin by considering low-symmetry analogues of octahedral rearrangement mechanisms,[4] there proves to be essentially one viable two-step mechanism where each step is a low-symmetry analogue of either mechanism 1.c or mechanism 1.d. That mechanism 1.d is now potentially operative is due to the lowering of the symmetry restrictions for the {6,−2} system from those for the {6,0} system. These mechanisms are illustrated in Figure 2, mechanisms 2.b and 2.c. Since a square-planar molecule is 4-coordinate because it does not have sufficient bonding electron density to be 6-coordinate, the intermediates as illustrated are of higher energy than the reactants because their M–L bonds are somewhat weaker than those in the reactant due to smaller electron density. In practice, therefore, the intermediate structures will

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relax from the one illustrated, but be of the same symmetry. They could then be described as distorted {4,0} or {5,–1} geometries. Thus such a mechanism is more likely to be the path for a rearrangement than the analogous ML_6 rearrangement, as it involves less M–L stretching. This is supported by the fact that many, but not all, cis and trans isomers can be isolated.[7-9] It may, however, be that a bond-cleavage mechanism is even more favorable (cf., e.g., ref 10).

AL_5

The closo AL_5 geometry is a trigonal bipyramid. Unlike the tetrahedron the five M–L bonds need not be the same length since the axial L–M–L equatorial bond angle is 90° and the equatorial L–M–L equatorial angle is 120°. If the ligand geometry is determined by placing L hard spheres in contact at distance λ, then the axial M–L distance is $(2/3)^{1/2}\lambda$ and the equatorial M–L distance is $(1/3)^{1/2}\lambda$. In practice, it is unlikely that such a variation in bond length is energetically favorable and values closer to $(1/2^{1/2})\lambda$ might be expected.

The two mechanisms generated by the CSRP are illustrated in Figure 3, mechanisms 3.a and 3.b. They are known respectively as the Berry pseudorotation mechanism[10] and the piano-stool or turnstile mechanism. Inspection of their transition states indicates that they proceed via {6,–1} and {7,–2} transition-state structures. (It should be noted that the {6,–1} transition state is drawn as a square-based pyramid with the base of the pyramid coplanar with the metal and all M–L bond lengths of $(1/2^{1/2})\lambda$, in contrast to the usual practice that puts the metal above the base of the pyramid. If the metal were above the base of the pyramid, then in order to maintain L–L distances additional energetically expensive M–L bond stretching is required, as well as loss of dispersion stabilization.) M–L bond lengths suggest the C_{4v} {6,–1} T might be expected to be more stable than the C_{2v} {7,–2} T. However, if the {7,–2} T relaxes from the pentagonal-bipyramid bond angles of 72° to angles of at least 90°, inspection of the requirements of the geometry show that the M–L bond lengths in the {7,–2} structure need be no longer than those of the axial ones in R. The next most significant energy term is dispersion stabilization: that of the {7,–2} geometry is about 7% less than the {6,–1} geometry so the Berry pseudorotation is slightly favored. In any case one would expect such a system to be fluxional as there is at most a 5% increase in *average* M–L bond lengths in going from R to T, and the T M–L bonds are shorter than the R axial bond lengths. (The 5% increase is for bond lengths of $(2/3)^{1/2}\lambda$ and $(1/3^{1/2})\lambda$; smaller average increases are required if the R bond lengths are closer in magnitude.)

The main conclusions to be drawn from this discussion are that trigonal-bipyramidal complexes can be expected to be fluxional in accord with experiment.[7-9,11] However, it cannot be assumed that only the Berry pseudorotation is operative. The energy difference between the mechanisms is sufficiently small that both are expected to play a role.

The square-based pyramid is another common AL_5 geometry. As one can find molecules representing all stages from trigonal bipyramid to distorted trigonal bipyramid to distorted square-based pyramid to square-based pyramid, one might expect at least one of the trigonal-prism mechanisms to have an analogue for the square-based pyramid. The mechanism via a C_{2v} {7,–2} structure is an obvious candidate for the rearrangement of a {6,–1} structure. In fact, the only concerted mechanism can be described in this way. Inspection of Figure 3, mechanism 3.b, indicates that it is somewhat academic whether one describes the T in Figure 3, mechanism 3.b, as a distorted {7,–2} or as a {5,0} structure. In any case, such a mechanism is expected to be fluxional.

$M(LL)_3$

The energetics of the symmetry-allowed nondissociative tris chelate mechanisms have already been considered. The conclusions from this study were that a tris chelate complex will rearrange via the Bailar twist mechanism if chelate bite, *b*, is much smaller than the L–L hard-sphere distance, via the Ray–Dutt twist if *b* is much greater than the L–L hard-sphere distance, and via both if *b* is of the order of the L–L distance. We shall therefore limit our current discussion to interpreting the previous results in the notation of this paper.

$M(LL)_3$ is a closo geometry, so we are looking for mechanisms proceeding via {6 + *i*,–*i*} structures. As for the octahedron, *i* = 1 and *i* = 2 are simply small distortions from the reactant geometry that do not lead to rearrangement. Mechanisms proceeding via a {9,–3} geometry are therefore likely to be the lowest energy type of pathway. As the Bailar twist and the Ray–Dutt twist transition states are {9,–3} geometries (they are both lower symmetry analogues of the {6,0} to {9,–3} to {6,0} octahedral mechanisms), our previous results are in accord with this.

Rearrangements of tris chelate complexes have been widely studied (see for example references 7–9, 11, and 12). It appears that bond-breaking mechanisms are usually operative. Dithiocarbamate complexes, however, do proceed via an intramolecular mechanism.[8] Complexes whose chelates have sulfur ligating atoms adopt ground-state geometries with comparatively small twist angles (sometimes being trigonal prismatic),[12] which suggests that the change from D_3 reactant to D_{3h} transition state is less energetically expensive for these complexes than for others that appear to favor a bond breaking mechanism.

A related point is that tris chelate complexes are thought to usually be comparatively more stable than their octahedral counterparts despite the fact that their rearrangement mechanisms are so similar.[8] Most tris chelate complexes have chelate bite sizes that are smaller than the other interligand distances. As a result less M–L bond stretching is required to go from the reactant to trigonal-prismatic transition state for a tris chelate than for an octahedral complex. One might therefore expect tris chelates to be more reactive than their octahedral counterparts along intramolecular rearrangement pathways. We therefore conclude that bond-breaking mechanisms are likely to be favored when the tris chelate complex is more stable than its octahedral analogue. This is in accord with available experimental data and provides another indirect probe of the mechanisms of tris chelate rearrangements.

The other two symmetry-allowed mechanisms are lower symmetry analogues of the octahedral mechanisms for which a transition state of sufficiently high symmetry requires coincidence of atoms. The lower symmetry of a tris chelate complex reduces those restrictions, and transition states can be constructed. As noted previously, the "push through" mechanism, whose planar T can best be viewed as an {11,–5} geometry, has a completely unfeasible transition state as it requires a M–L bond length stretch of the order of 40%. Similarly, the "crossover" mechanism, whose T can be viewed as a {10,–4} geometry, is also unlikely to be observed as it requires approximately an average 32% increase in M–L bond length.

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