butene on the basis that $Z$-crotyl anion (I) is more stable than $E$-crotyl anion (II). ${ }^{2}$ Although considerable spectral evidence has been accumulated that $Z$ is more stable than $E$ in related anions, ${ }^{3}$ the only study in which a relative stability assignment was made for crotyl anion itself led to the conclusion that the $E$ form (II) predominated. ${ }^{4}$ We wish to report that crotyllithium, previously prepared by a laborious route, ${ }^{5}$ is readily available by metalating l-butene or $Z$ - or $E$-2-butene, that at $35^{\circ}$ in tetramethylethylenediamine (TMEDA) it is ionic, with the $Z$ form (I) predominating over the $E$

form (II) by $\sim 85: 15$, and that on alkylation with $n$-butyl bromide, it gives a $6: 1$ ratio of $Z$ - to $E$-2-octene.

Crotyllithium is readily prepared by slowly adding TMEDA ( 5 ml ) at $-10^{\circ}$ to a solution of 1 -butene or $Z$ or $E$-2-butene ( 0.02 mol ), tert-butyllithium ( 0.02 mol ), and hexane $(10 \mathrm{ml}) .{ }^{6}$ After 0.5 hr at room temperature (or 24 hr if $n$-butyllithium is used instead), the reaction is essentially complete. If desired, the bulk of the hexane can be removed by vacuum.

The most obvious pmr spectral feature of crotyllithium prepared in this way is the $1: 3: 3: 1$ quartet $(J=$ 10.2 Hz ) for the $\beta$-proton at $\delta 6.0$, also observed in earlier preparations in diethyl ${ }^{5}$ and dimethyl ether. ${ }^{4,5}$ The other peaks for crotyllithium, visible though partially obscured by TMEDA or residual hexane, were shown by spin decoupling experiments to be consistent with the assignments on formula I and give essentially the same parameters observed in ether solvents. ${ }^{4,5}$ Indication that these parameters are due to an ionic species comes from the upfield location of the $\gamma$ proton ( $\delta 3.0$; ionic 3-neopentylallyllithium (NAL), $\delta 3.3 ;{ }^{3 f}$ covalent $Z-\mathrm{NAL}^{3 f}$ and crotylmagnesium bromide, $\delta 4.5^{7}$ ), downfield location of the $\alpha$-proton ( $\delta 1.2$; ionic NAL, $\delta$ $1.2^{3 \mathrm{f}}$; covalent $Z-\mathrm{NAL}^{3 \mathrm{f}}$ and crotylmagnesium bromide, ${ }^{7} \delta 0.8$ ), and especially from the nonequivalence of the $\alpha$-protons at $-90^{\circ}$ when rotation about the $\mathrm{C}_{\alpha}-\mathrm{C}_{\beta}$
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bond has been frozen out. ${ }^{4,8}$ The $Z$ nature of the predominant isomer is shown by the relatively small magnitude of $J_{\beta \gamma}(10.2 \mathrm{~Hz} ; c f .8 .7-10.2 \mathrm{~Hz}$ in related ionic $Z$ anions and $13.4-14.3 \mathrm{~Hz}$ in related ionic $E$ anions ${ }^{3}$ ). The amount of $E$ isomer is difficult to estimate accurately from the $\beta$-proton absorption due to partial overlap, but in dimethyl ether the $\alpha$ and $\delta$ absorptions of the minor isomer are well resolved and show $\sim 15 \%$ of the minor isomer to be present. ${ }^{4}$

Alkylation results correlate well with these pmr findings. When a solution of crotyllithium in TMEDAhexane prepared as described above was added at $-78^{\circ}$ to excess $n$-butyl bromide, the resulting $\mathrm{C}_{8}$ alkene mixture was $46 \%$ Z-2-octene, $8 \% E$-2-octene, and $46 \% 3$ -methyl-1-heptene. ${ }^{9}$

These findings contrast with the report that in dimethyl ether the equilibrium mixture contains mostly $E$ crotyllithium (II), ${ }^{4}$ with stereochemical assignments based on an interpretation of small chemical shift differences. These chemical shift arguments are evidently unreliable, since they give the answer opposite to that derived from the generally employed ${ }^{3 a-f}$ method of coupling constant differences used above.

The $85 \%$ value of $Z$-crotyllithium (I) at equilibrium is higher than that found for NAL ( $57 \%{ }^{3 f}$ ), presumably because in the latter the attractive interaction in the $Z$ isomer ${ }^{10}$ is partially offset by a larger steric interaction. It is close to most of the values found for 3 -substituted 1 -methylallyl anions ${ }^{3 a-e}$ and is sufficiently high that it can indeed serve as the major cause for the rapid basecatalyzed isomerization of 1-butene to Z-2-butene. ${ }^{2}$

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## Stereochemical Control of Valence. II. <br> The Behavior of the $\{\mathbf{M N O}\}^{n}$ Group in Ligand Fields ${ }^{1}$

Sir:
It is well-known that $\mathrm{M}-\mathrm{N}-\mathrm{O}$ angles ranging from 120 to $180^{\circ}$ occur in mononitrosyl complexes. ${ }^{2}$ Both valence-bond concepts ${ }^{3}$ and molecular orbital con-
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Figure 1. Four possible molecular orbital diagrams for the metal d and $\pi^{*}(\mathrm{NO})$ orbitals of the covalent $\{\mathrm{MNO}\}^{n}$ group with $C_{\infty v}$ symmetry. Diagrams a and b represent cases where the metal $d$ orbitals of the free ion are of higher energy than the $\pi^{*}(\mathrm{NO})$ orbitals; diagrams cand d represent cases where the metal d orbitals of the free ion are lower in energy than the $\pi^{*}(\mathrm{NO})$ orbitals. The dependence of the $\mathrm{M}-\mathrm{N}-\mathrm{O}$ angle upon $n$ is shown for each diagram.
cepts ${ }^{4}$ have been utilized to rationalize these angles for various complexes or groups of complexes. Recently we demonstrated ${ }^{1}$ that the simple reaction [ $\mathrm{Co}(\mathrm{NO})$ (das) $)^{2+}+\mathrm{NCS}^{-} \rightarrow\left[\mathrm{Co}(\mathrm{NO})(\text { das })_{2}(\mathrm{NCS})\right]^{+}$(where das is o-phenylenebis(dimethylarsine)) and the concomitant change in stereochemistry about the Co atom from five- to six-coordination resulted in the conversion of a linear CoNO group to strongly bent CoNO group. We have chosen to call the process "stereochemical control of valence" because the distribution of valence electrons and the geometry of the $[\mathrm{CoNO}]^{2+}$ moiety are dictated by the overall stereochemistry of the complex ion. Here we show that such transformations can be understood by considering the $\{\mathrm{MNO}\}^{n}$ moiety ${ }^{5}$ as an "inorganic functional group" that is perturbed by the coordination of other ligands to the metal.
Four possible molecular orbital diagrams for the covalent triatomic MNO group possessing $C_{\infty}$ symmetry are shown in Figure 1. These diagrams have been constructed from the metal d orbitals and the $\pi^{*}(\mathrm{NO})$ orbitals, which detailed molecular orbital calculations have shown to be similar in energy and strongly interacting. ${ }^{6}$ Experimental evidence also indicates that the NO ligand is one of the strongest "back-accepting" ligands. ${ }^{7.8}$ Hence, in all of the diagrams of Figure 1 the orbital $2 \pi\left(x z, y z, \pi^{*}(\mathrm{NO})\right.$ ), which is antibonding with respect to N and O but bonding with respect to M and N , is shown as the lowest energy molecular orbital. The $\delta\left(x y, x^{2}-y^{2}\right)$ orbital is nonbonding in $C_{\omega \nu}$ symmetry, and the $\sigma^{+}\left(z^{2}\right)$ orbital is $\sigma$ antibonding with respect to M and N . Finally, the $3 \pi\left(\pi^{*}(\mathrm{NO}), x z, y z\right)$ orbital is antibonding with respect to $\mathrm{M}, \mathrm{N}$, and O . For triatomic molecules of the nontransition elements, the occupation of this latter type of orbital leads to a nonlinear triatomic species. ${ }^{9}$ If
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(5) $n$ is the number of electrons associated with the metal d orbitals and the $\pi^{*}(\mathrm{NO})$ orbitals of the MNO group; $n$ corresponds to the familiar number of $d$ electrons on the metal when the nitrosyl ligand is formally considered to be $\mathrm{NO}^{+}$.
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0
$i$
$i$
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| $2 \pi\left(x z, y z, \pi^{*}(N O)\right)$ |
| :--- | :--- |
| 11near $n \leqslant 8$ |
| bent $n>8$ |$\quad \xlongequal{2 e\left(x z, y z, \pi^{*}(N O)\right)}$

Figure 2. An orbital correlation diagram showing the splitting of the molecular orbitals of the $\{\mathrm{MNO}\}^{n}$ group from Figure 1 la in a ligand field of $C_{4 r}$ symmetry. The dependence of the $\mathrm{M}-\mathrm{N}-\mathrm{O}$ angle upon $n$ is shown for each symmetry.

Walsh's concepts ${ }^{9}$ can also be applied ${ }^{\text {4a }}$ to the $\{\text { MNO }\}^{n}$ group then only the energy of the $3 \pi$ orbital relative to the other orbitals need be considered in order to predict whether a given value of $n$ will produce a linear or a bent nitrosyl group. Figure la and lb has $3 \pi$ higher in energy than $\delta$, the nonbonding metal orbital. This ordering is reasonable if the metal $d$ orbitals of the free ion are of lower energy than the $\pi^{*}(\mathrm{NO})$ orbitals. Figure 1 c and 1 d has $3 \pi$ of lower energy than $\delta$, an ordering expected only for strongly reducing metals. The dependence of the M-N-O angle upon $n$ for each of the orderings is also shown in Figure 1.

The coordination of other ligands to the metal of the MNO triatomic species will lower the symmetry from $C_{\infty} r$ and can have important effects on the geometry of the MNO group. For example, Figure 2 shows that a large $C_{4 v}$ perturbation of Figure la raises the $b_{1}\left(x^{2}-\right.$ $y^{2}$ ) orbital higher in energy than $3 \mathrm{e}\left(\pi^{*}(\mathrm{NO}), x z, y z\right)$ and can thereby convert a linear $\{\mathrm{MNO}\}^{8}$ group into a bent $\{\mathrm{MNO}\}^{8}$ group. ${ }^{10}$ Figure 3 depicts the splitting of the MNO orbitals of Figure 1c in a field of $C_{i \tau} \mathrm{sym}-$ metry. For this example, a large $C_{i v}$ perturbation results in $\mathrm{b}_{2}(x y)$ becoming lower in energy than $3 \mathrm{e}\left(\pi^{*}-\right.$ (NO), $x z, y z$ ). Here decreasing the magnitude of the $C_{4 v}$ perturbation will convert a linear $\{\mathrm{MNO})^{6}$ group into a bent $\{\mathrm{MNO}\}^{6}$ group. Thus, Figure 2 describes the conditions for the formal reduction of a coordinated NO ligand by an increase in the tetragonal ligand field splitting, and Figure 3 describes the conditions for the formal reduction of a coordinated NO ligand by a decrease in the tetragonal ligand field splitting. ${ }^{11}$

Common to the $\{\mathrm{MNO}\}^{8}$ case of Figure 2 and the $\{\mathrm{MNO}\}^{6}$ case of Figure 3 is a crossing point at intermediate perturbing fields where the highest occupied molecular orbital and the lowest unoccupied molecular orbital become degenerate. This crossing point repre-

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Figure 3. An orbital correlation diagram showing the splitting of the molecular orbitals of the $\{\mathrm{MNO}\}^{n}$ group from Figure 1 c in a field of $C_{4 v}$ symmetry. The dependence of the $\mathrm{M}-\mathrm{N}-\mathrm{O}$ angle upon $n$ is shown for each symmetry.
sents the transition state for the conversion of the MNO group from linear geometry to bent geometry. Several electronic states arise from such a degenerate crossing point, and a simple electrostatic calculation has been carried out for a representative example in $C_{40}$ symmetry. ${ }^{12}$ The results of the calculation and relationship of the state symmetry diagram to the reactions of mononitrosyl complexes are discussed in the succeeding paper. ${ }^{13}$

Finally, we suggest that the concept of inorganic functional groups has general utility for understanding covalently bound transition metal complexes. The concept should be especially applicable to metal complexes of other small molecules (such as $\mathrm{O}_{2}$ and $\mathrm{N}_{2}$ ) and to metal cluster compounds. ${ }^{14.15}$

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Stereochemical Control of Valence. III. The $\{\mathrm{CoNO}\}^{8}$ Group in Ligand Fields of $C_{4 v}, C_{2 v}$, and $C_{s}$ Symmetry
Sir:
The role of symmetry in chemical reactions has received much attention. ${ }^{1}$ Consideration of molecular orbital symmetry alone provides significant insight into

[^1]the chemical reactivity of strongly bound compounds with no low-lying excited states. ${ }^{\text {a.d }}$ However, molecular orbital symmetry by itself may be insufficient for understanding the reactions of transition metal complexes. ${ }^{10}$ In the preceding communication ${ }^{2}$ we introduced the concept of an inorganic functional group and showed that the properties of mononitrosyl complexes could be understood by examining the behavior of the $\{\mathrm{MNO}\}^{n}$ group ${ }^{3}$ in ligand fields. In this communication, the concept is applied to the specific problem of understanding the properties of the $\{\mathrm{CoNO}\}^{8}$ group in ligand fields of various symmetries.

Five-coordinate complexes containing the $\{\mathrm{CoNO}\}^{8}$ group exhibit $\mathrm{Co}-\mathrm{N}-\mathrm{O}$ angles ranging from 120 to $180^{\circ}$. Moreover, to date all linear $\{\mathrm{CoNO}\}^{8}$ groups occur in trigonal bipyramidal (TBP) geometry and strongly bent $\{\mathrm{CoNO}\}^{8}$ groups occur in tetragonal pyramidal (TP) geometry. ${ }^{4}$ The molecular orbital correlation diagram appropriate for these complexes is shown in Figure 1. The maximum symmetry possible for $\mathrm{CoNOL}_{4}$ is $C_{4 v}$, and two possible molecular orbital orderings for it are shown in Figures 1 b and 1 c . If the molecular ordering is that depicted in Figure 1c, then the electron configuration will be $(2 \mathrm{e})^{4}\left(1 \mathrm{~b}_{2}\right)^{2}(3 \mathrm{e})^{2}$. With the totally antibonding 3 e orbital doubly occupied, the $\{\mathrm{CoNO}\}^{8}$ group will bend ${ }^{2}$ producing $\mathrm{CoNOL}_{4}$ with $C_{s}$ symmetry (Figure 1 d ). If the $4 \mathrm{a}_{1}$ orbital is lowest (Figure lb) then the electron configuration is (2e) ${ }^{4}$ $\left(1 b_{2}\right)^{2}\left(4 a_{1}\right)^{2}$. With two electrons in $4 a_{1}$, the $\{\mathrm{CoNO}\}^{8}$ group will not bend, but the $\mathrm{CoNOL}_{4}$ complex will distort to TBP geometry with $C_{2 v}$ symmetry. This distortion to TBP geometry makes $4 \mathrm{a}_{1}\left(\mathrm{~d}_{2^{2}}\right)$ less antibonding thereby lowering the energy of the complex. A TBP distortion also allows the $y z$ component of the e orbitals to interact with the $\sigma$ orbitals of the other two ligands in the equatorial plane of the TBP complex, thereby facilitating delocalization of electron density from the $\sigma$ orbitals of the equatorial ligands into the $\pi^{*}$ orbitals of the NO group. Thus, for five-coordinate complexes of the $\{\mathrm{CoNO}\}^{8}$ group, the choice between a $T B P$ structure of $C_{2}$. symmetry (Figure la) and a $T P$ structure with $C_{s}$ symmetry (Figure 1d) is dictated by the relative energies of the $4 a_{1}$ and $3 e$ orbitals of the hypothetical $C_{4 v}$ species. ${ }^{5}$

The presence of two electrons in the $4 a_{1}$ and $3 e$ molecular orbitals gives rise to several electronic states. ${ }^{9}$ Figure 2 shows the relative energies of the manifold of singlet states arising from the ( $\left.4 \mathrm{a}_{1}, 3 \mathrm{e}\right)^{2}$ electronic configurations in $C_{4 t}$ symmetry and the behavior of these states in $C_{2 v}$ and $C_{s}$ symmetry. The far left-hand

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[^0]:    (10) In symmetries lower than $C_{v}$ the degeneracies of the $2 e$ and $3 e$ orbitals will also be lifted, but the discussion remains unchanged.
    (11) Similar correlations can be drawn for diagrams 1 b and 1 d . The perturbation of 16 by a field of $C_{4 v}$ symmetry can result in a linear $\{\mathrm{MNO}\}{ }^{10}$ group going to a bent $\{\mathrm{MNO}\}^{10}$ group. The perturbation of Id by field of $\mathrm{C}_{4 v}$ symmetry can result in a bent $\{\mathrm{MNO}\}^{s}$ group going to a linear $\{\mathrm{MNO}\}^{8}$ group.

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