Table IV. Mo-C and C-O Bond Lengths in Selected Molybdenum Carbonyl Complexes[a]

complex	av Mo-C (carbonyl) bond length, Å	av C-O bond length, Å	ref
$(MeCp)Mo(CO)_2I_3$	2.043 (2)	1.128 (5)	this work
$(\eta^5\text{-}C_9H_7)Mo(CO)_3I$	1.93 (4)	1.187 (13)	17
$(\eta^5\text{-}C_5H_5)Mo(CO)_3Cl$	1.990 (7)	1.163 (22)	18
$(\eta^5\text{-}C_5H_5)Mo(CO)_3(C_2H_5)$	1.96 (1)	1.16 (1)	19
$(\eta^5\text{-}C_5H_5)Mo(CO)_3(C_3F_7)$	2.012 (6)	1.167 (11)	20
$(\eta^5\text{-}C_5H_5)_2Mo_2(CO)_6$	1.960 (17)	1.16 (13)	21
$(\eta^5\text{-}C_5H_5)Mo(CO)_2(PPh_3)I$	1.983 (1)	1.124 (14)	22
$[(C_4H_9)_4N][(\eta^5\text{-}C_5H_5)Mo(CO)_3]$	1.909 (6)	1.176 (5)	23

[a] Standard deviations (in parentheses) on mean values ($\bar{p}$) of n equivalent distances (p_i) were calculated by the formula $[\Sigma_i(p_i - \bar{p})^2/n(n-1)]^{1/2}$.

been proposed generally have bond lengths of about 1.388 Å for the "ene" bond in the Cp ring.[16]

The methyl substituent on the Cp ring is directed between the two CO ligands. This orientation minimizes steric repulsions between the "equatorial" iodide ligands and the methyl group. Noteworthy is the fact that the methyl carbon atom C(8) is displaced by 0.214 (7) Å out of the mean plane through the C_5 ring away from the coordination sphere. Similar results have been found for other methyl-substituted cyclopentadienyl rings.[16]

The structure of the $(MeCp)Mo(CO)_2I_3$ complex is consistent with spectroscopic data previously reported for the $CpM(CO)_2X_3$ complexes.[2,3] In particular, the high $\nu(C{\equiv}O)$ frequencies reported reflect the short C-O bond lengths [C(1)-O(1) = 1.123 (7), C(2)-O(2) = 1.133 (7) Å]. These short distances indicate that there is little π back-bonding from the metal to the carbonyl groups, as expected in a Mo(IV) complex. That the C-O bonds are short can be seen by comparison to the C-O bond lengths reported for other molybdenum carbonyl complexes (Table IV). The low degree of π back-bonding is also reflected in the long Mo-CO bond distances [Mo-C(1) = 2.045 (6) Å, Mo-C(2) = 2.041 (6) Å]. Table IV shows that the Mo-CO bonds in $(MeCp)Mo(CO)_2I_3$ are longer than the Mo-CO bonds in other molybdenum carbonyl complexes. The high coordination number of the $(MeCp)Mo(CO)_2I_3$ complex is also expected to lengthen the M-CO bonds in comparison to less sterically crowded molecules.[24]

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Registry No. $(MeCp)Mo(CO)_2I_3$, 77590-15-9; $[(MeCp)Mo(CO)_3]_2$, 33056-03-0.

Supplementary Material Available: Table S1, listing observed and calculated structure factor amplitudes, and Table S2, listing least-squares planes (14 pages). Ordering information is given on any current masthead page.

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Bonding Mode and Trans Influence of the Nitromethyl Ligand. Structure of *trans*-Bis(dimethylglyoximato)(nitromethyl)(pyridine)cobalt(III)

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We are interested in developing new methods of forming Co-C bonds as part of a program aimed at elucidating the effects of alkyl groups with differing electronic properties on the structure,[1-5] reactions,[5-8] and spectral properties[1,2,5-7,9-12] of cobalt complexes. We describe here a synthetic procedure for preparing cobaloxime complexes which contain the nitromethyl substituent, $LCo(DH)_2CH_2NO_2$, where DH = monoanion of dimethylglyoximme. A study of the interrelationship between the Co-C bond and systematic variations in the L ligand and the substituent on carbon may provide insight into the factors which promote cleavage of the Co-C bond in vitamin B_{12} coenzyme.[1,2]

We have previously investigated in detail cobaloximes containing good electron-donor alkyl ligands. The nitromethyl substituent is a relatively poor electron-donating ligand. Since there was a small possibility that bonding was via O rather than C,[13] we have elucidated the structure of the complex with L = pyridine. This complex serves as a "bridge" between organocobaloximes and acidocobaloximes. In addition, it can be argued that the chemistry of cobaloximes and of Pt(II) compounds has many analogies,[9,10] and we wanted to explore the application to cobaloximes of preparative procedures used in Pt(II) chemistry.[14]

Experimental Section

(a) Preparation of $pyCo(DH)_2CH_2NO_2$. A solution of 3.0 g (7.43 mmol) of $pyCo(DH)_2Cl$[9] in 40 mL of nitromethane was treated with 1.73 g (7.46 mmol) of silver oxide and heated with stirring at 60-65 °C for 1.5 h. At the end of this time, the hot reaction mixture was filtered through Celite to remove precipitated silver chloride. The filtrate was allowed to evaporate to dryness overnight in a fume hood. The residue, either a dry solid or an oil, was taken up in about 50 mL of warm acetone. Then, 20 mL of water was added and the acetone was allowed to evaporate slowly, giving product which is ~95% pure, as evidenced by ^1H NMR spectra. Two recrystallizations from acetone/H_2O yielded pure product in about 40-50% yield. The contaminant which was removed in the recrystallizations was $pyCo(DH)_2NO_2$ (determined by ^{13}C NMR spectral comparison with the authentic complex), although no study was made to determine the mechanism of formation of the NO_2 complex. This procedure has proved useful in preparing analogous complexes with substituted pyridines.

(b) Crystal Data. Crystals of $pyCo(DH)_2CH_2NO_2$ were obtained by slow crystallization from acetone/H_2O. Cell dimensions were determined from Weissenberg and precession photographs and refined on a Siemens AED single-crystal diffractometer. The results are given in Table I. One check reflection intensity was measured every 100 reflections. There was no systematic variation throughout the data collection. The intensities for which $I > 3\sigma(I)$ were corrected for Lorentz and polarization effects but not for absorption.

(c) Solution and Refinement of Structure. The structure was solved by conventional Patterson and Fourier methods and refined by the block-diagonal anisotropic least-squares method to a final R value of 0.035. This approach probably underestimates the esd's. Only the oxime bridge hydrogen atoms were refined anisotropically; the remainder were held constant at $B = 5$ Å^2.

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Table I. Crystallographic Data for $pyCo(DH)_2CH_2NO_2$

$CoO_6N_6C_{14}H_{21}$	$\gamma = 135.2\ (1)°$
$M_r = 428.3$	$D_m = 1.54$ g cm^{-3}
$a = 10.904\ (5)$ Å	$D_c = 1.53$ g cm^{-3}
$b = 16.589\ (7)$ Å	$Z = 2$
$c = 9.079\ (3)$ Å	space group = $P\bar{1}$
$\alpha = 64.5\ (1)°$	$\mu = 10.0$ cm
$\beta = 126.6\ (1)°$	λ(Mo K$\bar{\alpha}$) 0.7107 Å

cryst dimens	$0.03 \times 0.08 \times 0.08$ cm^3
no. of reflctns measd ($I > 3\sigma(I)$)	4086
no. of independent reflctns ($I \geqslant 3\sigma(I)$)	2562
max 2θ	56°

Table II. Nonhydrogen Atom Positional Parameters ($\times 10^4$) for $pyCo(DH)_2CH_2NO_2$

atom	x	y	z
Co	95 (0)	2253 (0)	1367 (0)
O(1)	1168 (3)	4364 (2)	−598 (3)
O(2)	582 (2)	1714 (1)	5019 (2)
O(3)	−1197 (3)	98 (1)	3224 (3)
O(4)	−487 (2)	2788 (2)	−2338 (2)
O(5)	−3821 (3)	800 (2)	2266 (3)
O(6)	−2448 (3)	2603 (2)	1931 (4)
N(1)	1218 (3)	3817 (2)	1034 (3)
N(2)	883 (2)	2533 (2)	3703 (3)
N(3)	−1099 (2)	683 (2)	1661 (3)
N(4)	−722 (2)	1975 (2)	−993 (3)
N(5)	−2965 (3)	1749 (2)	1557 (3)
N(6)	2676 (2)	2627 (2)	2346 (3)
C(1)	2715 (4)	5526 (2)	2314 (6)
C(2)	1922 (3)	4321 (2)	2396 (4)
C(3)	1764 (3)	3566 (2)	4007 (4)
C(4)	2467 (4)	3924 (3)	5771 (4)
C(5)	−2558 (4)	−1022 (2)	385 (5)
C(6)	−1742 (3)	187 (2)	313 (4)
C(7)	−1559 (3)	957 (2)	−1293 (3)
C(8)	−2330 (4)	580 (3)	−3082 (4)
C(9)	−2514 (3)	1852 (2)	236 (3)
C(10)	3359 (3)	2142 (2)	3924 (4)
C(11)	5065 (4)	2346 (2)	4587 (5)
C(12)	6145 (4)	3072 (3)	3641 (5)
C(13)	5480 (4)	3573 (3)	2046 (5)
C(14)	3742 (3)	3337 (2)	1425 (4)

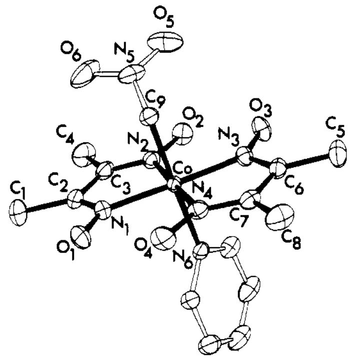

Figure 1. Molecular structure of the title complex.

Table III. Bond Lengths (Å) and Selected Bond Angles (Deg) for $pyCo(DH)_2CH_2NO_2$

(a) Bond Lengths

Co-N(1)	1.892 (2)	N(4)-C(7)	1.294 (4)
Co-N(2)	1.887 (3)	C(1)-C(2)	1.499 (5)
Co-N(3)	1.886 (2)	C(2)-C(3)	1.467 (4)
Co-N(4)	1.894 (3)	C(3)-C(4)	1.488 (6)
Co-N(6)	2.028 (3)	C(5)-C(6)	1.498 (5)
Co-C(9)	2.002 (3)	C(6)-C(7)	1.476 (4)
N(1)-O(1)	1.355 (3)	C(7)-C(8)	1.495 (5)
N(2)-O(2)	1.348 (3)	N(6)-C(10)	1.355 (3)
N(3)-O(3)	1.346 (3)	N(6)-C(14)	1.344 (4)
N(4)-O(4)	1.356 (3)	C(10)-C(11)	1.373 (6)
N(5)-O(5)	1.234 (3)	C(11)-C(12)	1.371 (6)
N(5)-O(6)	1.222 (5)	C(12)-C(13)	1.369 (5)
C(9)-N(5)	1.455 (6)	C(13)-C(14)	1.388 (6)
N(1)-C(2)	1.291 (5)	O(1)···O(4)	2.460 (4)
N(2)-C(3)	1.301 (4)	O(2)···O(3)	2.495 (4)
N(3)-C(6)	1.302 (2)		

(b) Bond Angles

N(1)-Co-N(2)	81.2 (1)	N(3)-Co-N(6)	89.4 (1)
N(1)-Co-N(3)	178.3 (1)	N(3)-Co-C(9)	89.6 (1)
N(1)-Co-N(4)	98.6 (1)	N(4)-Co-N(6)	91.0 (1)
N(1)-Co-N(6)	92.3 (1)	N(4)-Co-C(9)	85.1 (1)
N(1)-Co-C(9)	88.7 (1)	N(6)-Co-C(9)	176.0 (1)
N(2)-Co-N(3)	99.1 (1)	Co-C(9)-N(5)	113.7 (2)
N(2)-Co-N(4)	179.2 (1)	C(9)-N(5)-O(5)	117.7 (4)
N(2)-Co-N(6)	89.8 (1)	C(9)-N(5)-O(6)	118.7 (3)
N(2)-Co-C(9)	94.2 (1)	O(5)-N(5)-O(6)	123.6 (4)
N(3)-Co-N(4)	81.2 (1)		

The weighting scheme in the final calculation was $w = 1/(A + |F_o| + C|F_o|^2)$ where $A = 2.9$ and $C = 0.091$ were chosen so as to maintain $w(|F_o| - |F_c|)^2$ essentially constant over all ranges of $|F_o|$ and $(\sin \theta)/\lambda$. Atomic scattering factors were those given in ref 15.

Final nonhydrogen positional parameters are collected in Table II. Hydrogen positional parameters, anisotropic thermal parameters, and a list of final calculated and observed structure factors have been deposited. All calculations were done with a standard set of computer programs.[16]

Results and Discussion

Synthesis. Recently it was reported that treatment of *cis*-$[PtCl_2(PEt_3)_2]$, dissolved in CH_3NO_2, with Ag_2O yields *cis*-$[PtCl(CH_2NO_2)(PEt_3)_2]$.[14] We now report that this same reaction is applicable to cobaloximes, especially those with nitrogen donor ligands.[17] Although silver-ion-promoted syntheses have previously been employed in the formation of (solvato)-[18,19] and (purinato)cobaloximes,[20] this note describes the first time an alkylcobaloxime has been prepared utilizing such a method.

In the case of L = substituted pyridine, the desired $LCo(DH)_2CH_2NO_2$ complex forms in comparable yields (40–50%) to Beck's preparation (vide infra).[21] In the case of L =

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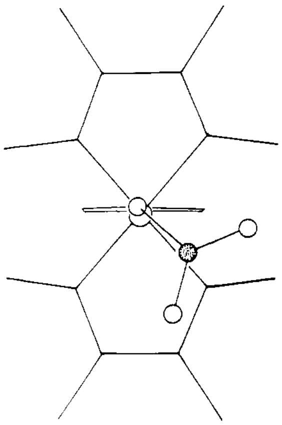

Figure 2. Perspective view along the Co–C bond.

$P(OCH_3)_3$, a small quantity (<10% yield) of $(CH_3O)_3PCo(DH)_2CH_2NO_2$ formed which was heavily contaminated with $(CH_3O)_3PCo(DH)_2NO_2$. ($LCo(DH)_2NO_2$ also formed in the case of L = substituted pyridine but to a negligible extent.) Later fractions yielded $H_2OCo(DH)_2P(O)(OCH_3)_2$.[6,22] It is known that this complex can be produced from nucleophilic attack at the phosphite ester carbon of cobaloximes containing trimethyl phosphite as a ligand.[6,22] For L = $P(C_6H_5)_3$, no useful product was obtained.

While this work was in progress, three preparations of $pyCo(DH)_2CH_2NO_2$ were reported. Widdowson and Roussi obtained this complex in a yield of 19% by alkylation of the cobalt(I) anion with $BrCH_2NO_2$.[23] These workers found alternatively that a yield of 24% could be obtained by alkylating $(py)_2Co^{II}(DH)_2$ with $BrCH_2NO_2$ in the presence of zinc dust. Beck and co-workers realized a yield of 58% of $pyCo(DH)_2CH_2NO_2$ by treating $pyCo(DH)_2Cl$ with $NaCH_2NO_2$ in THF.[21] This latter synthesis gives comparable results to that reported here but is not as convenient.[24]

Structure. An ORTEP drawing of a crystallographically independent molecule of $pyCo(DH)_2CH_2NO_2$ with the atom numbering scheme is shown in Figure 1. Bond lengths and selected bond angles are listed in Table III. The CH_2NO_2 group is bonded via C to the cobalt.

The cobalt atom has a distorted octahedral stereochemistry and is displaced (toward py) 0.02 Å above the plane of the four oxime N donors, which are coplanar within ±0.008 Å. The two oxime ligands make an angle of 0.2°. In addition, both make angles of 3.0° with the four-nitrogen-donor equatorial plane. These values suggest a slightly stepped conformation of the essentially planar equatorial ligands. The coordinated pyridine (planar within ±0.002 Å) makes an angle of 87.5° with the equatorial plane. The orientation of the py ligand is that always found in $pyCo(DH)_2R$ compounds, i.e., nearly bisecting the oxime bridges.[1,3,4,25] The C(9)–NO_2 group is planar within ±0.007 Å and makes an angle of 28.0° with the equatorial plane. Its orientation is shown in Figure 2.

The bond lengths and angles in the oxime ligands are quite normal.[1,3,4] The Co–C bond length of 2.002 (3) Å is not significantly different from the value of 1.998 (5) Å reported for the methyl complex,[3] whereas the Co–N(py) distance is only 2.028 (3) Å. The C(9)–Co–N(6) angle is 176.01 (8)°, the bending being away from the NO_2 group. The Co–C(9)–N(5) angle is 113.7 (2)° and does not differ significantly from those for Co–C–C of 114.3 (2) and 114.0 (2)° found in $pyCo(DH)_2$-i-C_3H_7.[1]

Table IV. Structural Data for $pyCo(DH)_2R$ Complexes

R	Co–N(py), Å	Co–C, Å	d,[a] Å	α,[b] deg
i-C_3H_7[c]	2.099 (2)	2.085 (3)	0.02	4.0
CH_3[d]	2.068 (3)	1.998 (5)	0.04	3.2
CH_2COOCH_3[e]	2.040 (6)	2.040 (6)		
CH_2NO_2[f]	2.028 (3)	2.002 (3)	0.02	0.2

[a] Displacement of Co out of the four-nitrogen-donor atom equatorial plane. [b] Angle between the planes passing through the oxime ligands. [c] Reference 1. [d] Reference 3. [e] Reference 25. [f] This work.

The most important structural result is that the Co–N(py) bond length is significantly shorter than those so far reported for any other alkylcobaloximes, which vary from 2.040 (6) to 2.099 (2) Å (see Table IV). This is the first structural evaluation of a complex containing the CH_2NO_2 ligand. The result indicates that the CH_2NO_2 group exerts a trans influence weaker than that of CH_3 and slightly weaker than that of CH_2COOCH_3. For a typical acido ligand, N_3, the Co–N(py) bond length is 1.973 (5) Å.[26] On the other hand, i-C_3H_7 lengthens the trans Co–N(py) bond more than CH_3.[1] Thus, as expected, acceptor groups such as NO_2 and $COOCH_3$ attract electron density from the carbon atom bound to cobalt, decreasing its trans-influencing ability. On the contrary, methyl groups which increase electron density on the σ-bonded carbon atom increase its trans-influencing ability. Work in progress in these laboratories suggests that the effects of the CH_2NO_2 group on structure, reactivity, and spectra is intermediate between those of the more trans-directing alkyl groups and the less trans-directing acido groups. The results of these comparative studies will be reported once a larger number of compounds has been studied.[27]

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Registry No. $pyCo(DH)_2CH_2NO_2$, 73202-88-7; $pyCo(DH)_2Cl$, 23295-32-1.

Supplementary Material Available: Tables of hydrogen atom parameters, anisotropic temperature factors, and structure factor amplitudes (11 pages). Ordering information is given on any current masthead page.

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Triborane(7) Adducts of Phosphine and Methylphosphines

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The base strength of phosphine (PH_3) toward borane(3) is considerably weaker than the base strength of methyl-substituted phosphines. Thus, phosphine–borane(3) $PH_3{\cdot}BH_3$[1]

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