Inorganica Chimica Acta, 27 (1978) 47–52

Structural Study of Cobaloxime Derivatives. The Molecular Structure of the Sodium Salt of *trans*-Dinitro(dimethylglyoximato)-(dimethylglyoxime)cobaltate(III) Dihydrate and *trans*-Nitrobis(dimethylglyoximato)-(triphenylphosphine)cobalt(III)

NEVENKA BRESCIANI-PAHOR, MARIO CALLIGARIS and LUCIO RANDACCIO*
Istituto di Chimica, Università di Trieste, 34127 Trieste, Italy

Received June 21, 1977

The crystal structure analysis of two title cobaloximes: [trans-$(NO_2)_2Co(dmg)(dmgH_2)$]$Na\cdot 2H_2O$ *(I),* trans-$(NO_2)(PPh_3)Co(dmgH)_2$*, (II), has revealed that crystals of (I) are triclinic, space group* $P\bar{1}$*, with* $a = 16.23$, $b = 6.621$, $c = 8.018$ Å, $\alpha = 91.59$, $\beta = 104.4$, $\gamma = 92.61°$; $Z = 2$*, while crystals of (II) are orthorhombic, space group,* $P2_12_12_1$*, with* $a = 17.357$, $b = 14.360$, $c = 10.783$ Å; $Z = 4$*. The structures have been solved by three-dimensional Patterson and Fourier methods and refined by the least-squares technique. The final* R *values were 0.044 for (I) and 0.049 for (II), calculated on the basis of 3157 and 1349 independent reflections respectively.*

The Co–N and N–O bond lengths of (I) are significantly different in the two equatorial moieties and are in agreement with the above formulation [$dmgH_2$*: Co–N 1.918(2), N–O 1.369(3)* Å*; dmg: Co–N 1.903(2), N–O 1.352(3)* Å]*. The sodium ion is surrounded by five oxygen atoms with a co-ordination polyhedron intermediate between a distorted trigonal bipyramid and a square-planar pyramid.*

The lengthening of the $Co–NO_2$ *bond distance (1.980(9)* Å*) in (II) with respect to the mean value found in complex (I) (1.946(3)* Å*) could be due to the* trans-*influence of the phosphine ligand. On the other hand the Co–P bond length (2.392(3)* Å*) is longer than those usually found.*

The structure of both compounds confirms the marked rigidity of the equatorial ligand.

Introduction

Recently the interest in the structural characterization of cobaloximes has been considerably increased. In fact it has been understood that many features of such compounds, which have considerable chemical interest, may be rationalized on the basis of structural correlations.

Thus, structural results have allowed an assessment of the *trans*-influence [1–4] in the octahedral compounds, although a more accurate analysis has yet to be performed. On the contrary, the existence in these complexes of the *cis*-influence is much more questionable [1, 2, 4, 5]. Furthermore one of the effects of the axial ligands on the $Co(dmgH)_2$ skeleton has been recently suggested [6] to produce a proton shift (LIPS) from one dmgH to another, producing a new skeleton $Co(dmg)(dmgH_2)$.*

Structural data [3] have also shown a noticeable rigidity of the $(dmgH)_2$ ligand, which may be distorted towards an umbrella-shape with interplanar angles not exceeding a few degrees, depending upon the steric hindrance of the axial ligands.

Finally, examples have been reported [7] showing the ability of dimethylglyoximato copper(II) to coordinate other metal atoms through its oxime oxygens, giving polynuclear complexes in a way similar to that of metal complexes containing Schiff base dianions like salen [8]. The latter "metal complex ligands" have been shown to co-ordinate alkali metal ions [9]. It seems likely that a similar coordination to alkali ions should be possible also for dimethylglyoximato complexes. The determination of the crystal and molecular structure of the sodium salt of [*trans*-$(NO_2)_2(dmg)(dmgH_2)Co^{III}$]$^-$ dihydrate (I) and of *trans*-$NO_2(PPh_3)(dmgH)_2Co^{III}$ (II) should give useful information about some of the above points.

Experimental

Crystal Data

$CoC_8H_{14}O_8N_6Na\cdot 2H_2O$ (I), $M = 440.2$, Triclinic, $a = 16.23$, $b = 6.621$, $c = 8.018$ Å, $\alpha = 91.59$, $\beta = 104.40$, $\gamma = 92.61°$, $U = 833.0$ Å^3, D_m (by flotation) $= 1.74$, $Z = 2$, $D_c = 1.75$, Mo–Kα radiation, $\lambda = 0.7107$ Å, μ(Mo–Kα) $= 11.69$ cm^{-1}. Space group $P\bar{1}$.

---

*To whom correspondence should be addressed.

*dmgH is the monoprotonated form of the dianion dmg of the dimethylglyoxime.

TABLE I. Atomic Positional Parameters ($\times 10^4$) of Non-Hydrogen Atoms for (I) with Estimated Standard Deviations in Parentheses.

	X	Y	Z
Co	2606(0)	7(1)	1543(0)
Na	2151(1)	4674(3)	4835(2)
O(1)	2999(1)	−654(4)	5229(3)
O(2)	3602(1)	−1110(4)	−848(3)
O(3)	2273(2)	645(4)	−2075(3)
O(4)	1638(1)	1108(4)	3940(3)
O(5)	3627(2)	3270(4)	1010(4)
O(6)	3203(2)	3661(4)	3325(4)
O(7)	1704(2)	−3396(4)	2237(4)
O(8)	1877(2)	−3502(4)	−334(4)
O(9)	1257(2)	6954(5)	5733(4)
O(10)	3152(2)	4783(5)	7423(4)
N(1)	3251(2)	−853(4)	3725(3)
N(2)	3544(1)	−1077(4)	823(3)
N(3)	1987(2)	859(4)	−640(3)
N(4)	1675(1)	1055(4)	2272(3)
N(5)	3214(2)	2650(4)	2012(3)
N(6)	1993(2)	−2619(4)	1082(3)
C(1)	4518(2)	−2456(5)	5358(5)
C(2)	3960(2)	−1688(4)	3750(4)
C(3)	4141(2)	−1798(4)	2012(4)
C(4)	4925(2)	−2640(5)	1667(5)
C(5)	682(2)	2316(5)	−2297(5)
C(6)	1263(2)	1627(4)	−666(4)
C(7)	1082(2)	1756(4)	1055(4)
C(8)	308(2)	2645(5)	1409(5)

Atomic Positional Parameters ($\times 10^3$) of Hydrogen Atoms for (I) with Their Estimated Standard Deviations in Parentheses. The hydrogen atoms are numbered according to the atom to which they are bonded.

	X	Y	Z
H1(C1)	430	−380	560
H2(C1)	510	−265	520
H3(C1)	450	−140	625
H1(C4)	545	−220	260
H2(C4)	486	−415	170
H3(C4)	495	−215	50
H1(C5)	5	220	−240
H2(C5)	83	378	−248
H3(C5)	75	143	−323
H1(C8)	43	406	162
H2(C8)	−20	241	43
H3(C8)	20	220	253
H1(O9)	124	805	493
H2(O9)	163	752	681
H1(O10)	325	602	815
H(1)	240	−35	480
H(2)	310	−25	−145

$CoC_{26}H_{29}O_6N_5P$ (II), $M = 597.5$, Orthorhombic, $a = 17.357$, $b = 14.360$, $c = 10.783$ Å, $U = 2687.6$ Å^3, D_m (by flotation) = 1.51, $Z = 4$, $D_c = 1.48$. μ(Mo–Kα) = 7.78 cm^{-1}. Space group $P2_12_12_1$.

Cell parameters were determined from Weissenberg and precession photographs taken with Co–Kα radiation and refined with an on-line automated single-crystal Siemens diffractometer (Mo–Kα).

Intensity Measurements

Three-dimensional intensity data were collected on a Siemens diffractometer by the θ–2θ scan technique with Mo–Kα radiation for a maximum 2θ angle of 56° for (I) and 50° for (II). Reflections with $I_o < 3\sigma(I_o)$ were rejected and the remainder corrected for Lorentz-polarization factors. A total of 3157 independent reflections for (I) and 1349 for (II) were used in the subsequent calculations. No absorption correction was applied (μR 0.17 for (I) and 0.05 for (II)).

Structure Determination and Refinement

Both structures were solved by conventional Patterson and Fourier methods. The final anisotropic refinement, including the contribution of hydrogen atoms (set at B = 3.5 Å^2) held constant and of two water molecules detected in the three-dimensional difference-Fourier map, gave R 0.044 for (I). For (II) the carbon atoms were refined isotropically while the others had anisotropic thermal factors. The final least-squares refinement, including the contribution of hydrogen atoms (with B = 4.0 Å^2) held constant, led to a value of the conventional R factor of 0.049. The final weighting scheme was $w = 1/(A + B\,|F_o| + C\,|F_o|^2)$ where $A = 3.0$, $B = 1.0$, $C = 0.0052$ for (I) and $A = 13.0$, $B = 1.0$, $C = 0.0046$ for (II) were chosen so as to maintain $w(|F_o| - |F_c|)^2$ essentially constant over all ranges of $|F_o|$ and $(\sin\theta/\lambda)$.

Final atomic parameters are listed in Tables I and II together with their estimated standard deviations calculated from the inverse matrix of the final least-squares cycle. A list of structure factors and anisotropic temperature factors is available from the Editor. Atomic scattering factors were calculated according to ref. 10.

Calculations

All calculations were carried out on a CDC 6200 computer with programs described in ref. 11.

Results and Discussion

Description and Discussion of the Structure (I)

The crystal structure of (I) is built up by [*trans*-$(NO_2)_2$(dmg)(dmgH$_2$)Co(III)]$^-$ macro-anions and Na$^+$ cations, alternating along the *b* axis. Two water molecules for each Na$^+$ ion are present. The structure of the anion and location of the cation are shown in Figure 1. The sodium ion is surrounded by five oxygen atoms. Two water molecules, O(9) and O(10), the O(4) and O(6) atoms of the macro-anion

TABLE II. Atomic Co-ordinates ($\times 10^4$) and Isotropic Temperature Factors for (II) together with Their Estimated Standard Deviations in Parentheses.

	X	Y	Z	$B(\text{Å}^2)$
Co	816(1)	1522(1)	887(1)	*
P	489(1)	446(2)	−723(3)	*
O(1)	2407(4)	1381(7)	291(8)	*
O(2)	−305(5)	2734(6)	−172(8)	*
O(3)	−777(4)	1661(6)	1533(8)	*
O(4)	1956(4)	367(6)	2009(9)	*
O(5)	1690(5)	2703(7)	2391(9)	*
O(6)	497(5)	2878(6)	2716(9)	*
N(1)	1742(5)	1835(7)	63(9)	*
N(2)	423(5)	2482(6)	−185(9)	*
N(3)	−106(5)	1238(6)	1765(8)	*
N(4)	1218(5)	615(6)	2015(9)	*
N(5)	1035(5)	2483(6)	2156(9)	*
C(1)	2402(9)	2898(11)	−1408(15)	5.41(34)
C(2)	1711(7)	2532(8)	−733(13)	3.64(24)
C(3)	934(6)	2901(8)	−828(12)	3.35(23)
C(4)	709(9)	3722(11)	−1685(15)	6.09(37)
C(5)	−738(9)	304(11)	3385(15)	5.46(33)
C(6)	−58(7)	592(8)	2650(11)	2.67(21)
C(7)	731(7)	256(8)	2817(11)	2.88(21)
C(8)	976(8)	−451(10)	3768(14)	4.96(32)
C(9)	794(6)	883(7)	−2278(10)	2.18(18)
C(10)	273(7)	1260(8)	−3188(12)	3.37(26)
C(11)	557(7)	1641(9)	−4305(13)	4.28(27)
C(12)	1349(7)	1620(10)	−4572(13)	4.26(28)
C(13)	1844(7)	1245(8)	−3684(12)	3.42(25)
C(14)	1566(7)	893(8)	−2531(11)	2.68(22)
C(15)	919(6)	−712(7)	−662(10)	2.29(19)
C(16)	931(6)	−1229(8)	474(11)	3.24(24)
C(17)	1219(7)	−2130(9)	487(12)	3.76(26)
C(18)	1557(8)	−2506(10)	−557(13)	4.38(29)
C(19)	1548(8)	−2022(10)	−1665(14)	4.61(31)
C(20)	1243(7)	−1116(9)	−1754(12)	3.56(26)
C(21)	−549(6)	233(7)	−708(11)	2.23(19)
C(22)	−1075(6)	892(7)	−1173(11)	2.59(21)
C(23)	−1855(7)	726(9)	−1107(12)	3.80(26)
C(24)	−2118(8)	−84(9)	−579(14)	4.08(28)
C(25)	−1634(7)	−731(9)	−101(12)	3.62(27)
C(26)	−844(6)	−584(8)	−169(11)	3.18(22)

Atomic Parameters ($\times 10^3$) of Hydrogen Atoms for (II) with Estimated Standard Deviations in Parentheses. The hydrogen atoms are numbered according to the atoms to which they are bonded.

TABLE II *(Continued)*

	X	Y	Z
H1(C1)	226	343	−198
H2(C1)	265	240	−193
H3(C1)	280	314	−81
H1(C4)	13	383	−164
H2(C4)	84	357	−258
H3(C4)	98	430	−144
H1(C5)	−58	−20	399
H2(C5)	−114	0	280
H3(C5)	−977	82	383
H1(C8)	154	−58	372
H2(C8)	70	−107	362
H3(C8)	85	−24	463
H(C10)	−29	124	−304
H(C11)	18	192	−490
H(C12)	155	186	−537
H(C13)	240	122	−387
H(C14)	194	67	−189
H(C16)	72	−94	125
H(C17)	119	−251	125
H(C18)	182	−312	−50
H(C19)	178	−233	−242
H(C20)	123	−76	−255
H(C22)	−88	148	−156
H(C23)	−222	120	−144
H(C24)	−268	−19	−54
H(C25)	−183	−131	30
H(C26)	−48	−105	18

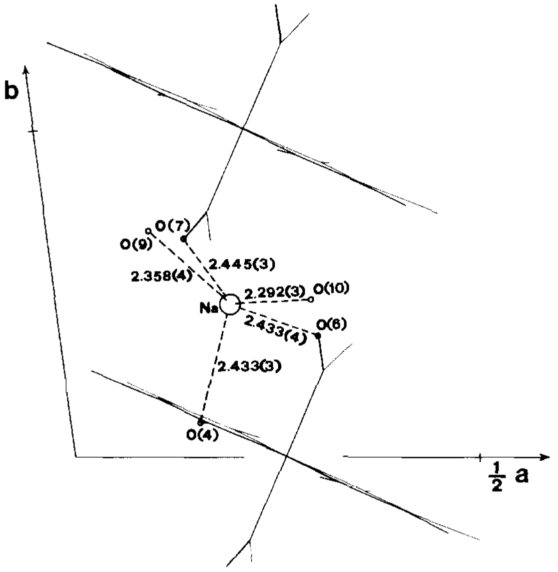


Figure 1. A view of the structure of (I) along the *c* axis. Co-ordination bond lengths of Na^+ are also reported.

immediately below and the O(7)′ atom of the macroanion immediately above the cation, make up the co-ordination polyhedron of the cation. The co-ordination polyhedron may be described as intermediate between a distorted trigonal bipyramid with O(9) and O(6) atoms in axial positions and a square planar pyramid with O(4) in apical position. An environment of five oxygen atoms around an alkali metal has been already found for K^+ in [*trans*-diglycinato(bae)Co^{III}]$K \cdot 6H_2O$ [12] although the

TABLE III. Bond Lengths in Some Octahedral Cobalt(III) Dimethylglyoximato Complexes.

XCo(dmg)(dmgH$_2$)Y	Co–N$_1$ Co–N$_2$	Co–N$_3$ Co–N$_4$	N$_1$–O$_1$ N$_2$–O$_2$	N$_3$–O$_3$ N$_4$–O$_4$	O$_1$–H O$_2$–H
(NO$_2$)$_2$(dmg)(dmgH$_2$)Co[a]	1.918(2)	1.902(2)	1.371(4)	1.350(4)	0.98
	1.918(2)	1.903(2)	1.366(3)	1.353(3)	0.82
Cl(dmg)(dmgH$_2$)Co(NH$_2$C$_6$H$_4$-SO$_2$NH$_2$)[b]	1.905(8)	1.870(8)	1.323(11)	1.326(10)	0.90
	1.896(8)	1.884(8)	1.344(11)	1.338(11)	1.02
Cl(dmg)(dmgH$_2$)Co(NH$_2$C$_6$H$_4$Cl)[b]	1.908(5)	1.874(5)	1.348(6)	1.337(6)	1.13
	1.906(5)	1.884(5)	1.359(6)	1.329(6)	1.16
CH$_3$(dmg)(dmgH$_2$)CoH$_2$O[c]	1.896(3)	1.884(3)	1.358(4)	1.347(3)	1.11
Cl(dmg)(dmgH$_2$)CoNH$_3$[d]	1.883(5)	1.887(5)	1.343(6)	1.335(6)	0.98
	1.883(5)	1.909(5)	1.345(6)	1.361(5)	0.96

[a] Present work. [b] Ref. 6. [c] Ref. 5. [d] Ref. 1.

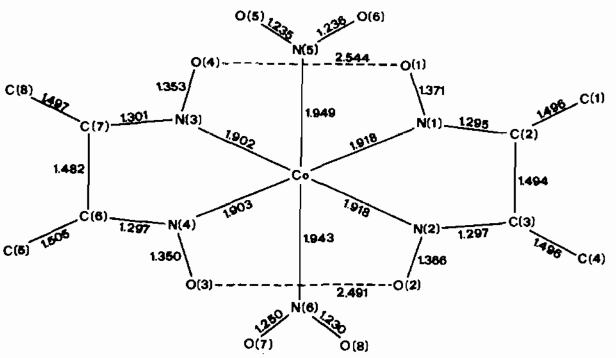


Figure 2. Numbering scheme for the atoms of the macro-anion of (I) together with bond lengths of interest. E.s.d.'s are 0.002–0.003 Å for Co–N, 0.003–0.004 Å for N–O, 0.004–0.005 Å for C–C.

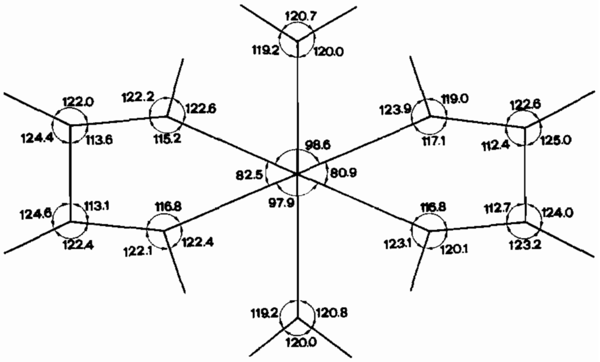
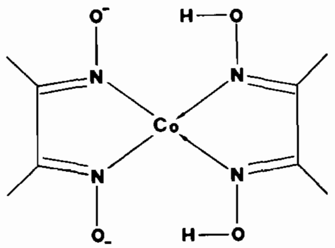


Figure 3. Bond angles of interest for the macro-anion of (I). E.s.d.'s are 0.1–0.2° for angles involving Co and 0.2–0.3° for other angles.

co-ordination polyhedron is better described as a distorted square-planar pyramid. The Na$^+$·····O distances, shown in Figure 1, are in the range of the values already reported for similar ionic compounds [9]. The ability of metaldimethylglyoximato complexes to co-ordinate alkali ions through their oxime oxygen atoms has been established in the solid state.

The numbering scheme for the atoms is shown in Figure 2 together with bond lengths. Bond angles of interest are given in Figure 3. As expected, the nitro-groups are nitrogen-bonded to cobalt and occupy the axial positions. Their planes are nearly parallel, the dihedral angle between the mean planes passing through Co, N(5), O(5), O(6) and Co, N(6), O(7), O(8) atoms being 12.3°. The latter planes are nearly perpendicular to the plane containing Co and N equatorial donor atoms (90.5 and 89.4° respectively) and both contain the line separating the two oximes. The Co–NO$_2$ bond lengths are equal within twice their e.s.d.'s, being 1.943(3) and 1.949(3) Å. On the contrary in the analogous compound *trans*-dinitro-2,2′-(1,3-diaminopropane)bis(2-methyl-3-butanone)-dioximatocobalt(III) [13], in which the equatorial ligand differs from (dmgH)$_2$ by having one –CH$_2$–CH$_2$–CH$_2$ bridge instead of an O···H···O bridge, the two nitro-group planes are nearly perpendicular, owing to the steric interactions of the propylene bridge with one nitro group. Furthermore the Co–NO$_2$ bond lengths in the latter compound are not equal (1.937(3) and 1.984(3)) the shortest value having been assumed [13] as the "normal" one, on the basis of the Co–NO$_2$ distances in other cobalt complexes. The mean N–O bond length of 1.238(4) Å and the mean bond angle ONO of 120.4(3)° in compound (I) are comparable to those reported [13] and those of ionic nitrites [14]. The atoms of the equatorial ligands are all coplanar within ±0.06 Å. The four oxime nitrogens are coplanar within ±0.007 Å with a slight tetrahedral distortion. The cobalt atom is in this plane being displaced by 0.001 Å. The bond length trend of the two equatorial ligands is in agreement with their formulation as dmgH$_2$ and dmg respectively, as illustrated in Scheme I.



As already noticed, the same formulation may be assumed in Cl(dmg)(dmgH$_2$)CoNH$_3$ [2], CH$_3$(dmg)(dmgH$_2$)CoH$_2$O [5] and in the more recently determined structures [6] of Cl(dmg)(dmgH$_2$)Co(NH$_2$C$_6$H$_4$-Cl) and Cl(dmg)(dmgH$_2$)Co(NH$_2$-C$_6$H$_4$SO$_2$NH$_2$). Analysis of the data relative to these structures reported in Table III shows: i) the oxime bridge hydrogens are both localized on the same dmg unit, which may be formulated as neutral dmgH$_2$, ii) the Co–N and N–O bond lengths of the dmgH$_2$ ligand are slightly, but significantly, longer than the corresponding lengths of the dmg unit. Only for ClCo(dmg)(dmgH$_2$)NH$_3$ [1], (ii) is trend reversed. Palenik *et al.* [6] have suggested that in the 4-chloroaniline and the sulfanilamide derivatives of Table III the proton transfer from one to the other dmgH anion is induced by the axial ligand (LIPS) to favour a π interaction between the aniline ring and the dianionic dmg group. Results summarized in Table III, however, do not entirely support this hypothesis. LIPS may be one effect, but it is certainly not the only one.

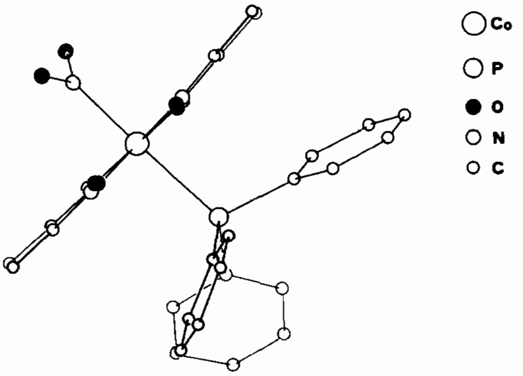


Figure 4. A side view of the molecule (II).

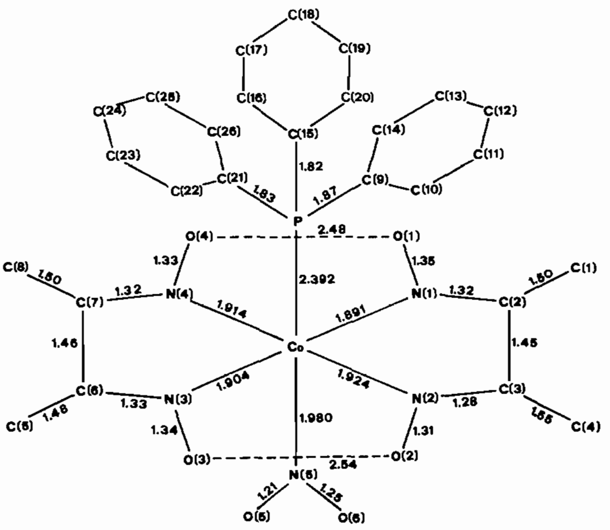


Figure 5. Numbering scheme for the atoms of (II) and bond lengths of interest. E.s.d.'s are 0.003 Å for Co–P, 0.009 Å for Co–N, 0.01 Å for N–O and 0.02 Å for C–N and C–C.

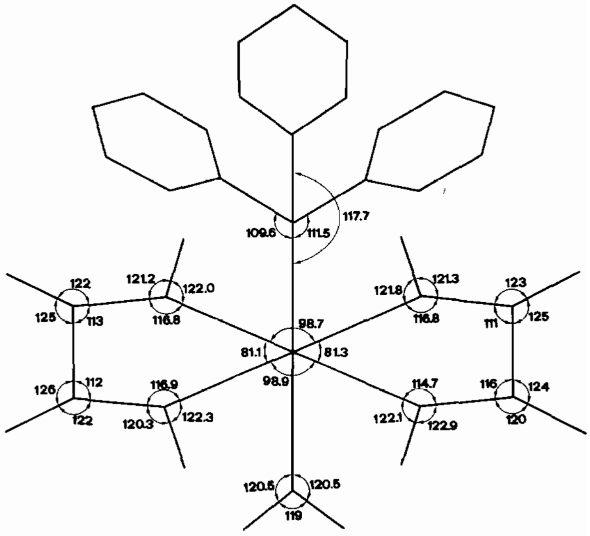


Figure 6. Bond angles of interest for (II). E.s.d.'s are 0.4° for angles involving Co and 0.8–1.0° for other angles.

Description and Discussion of Structure II

The crystal of (II) consists of discrete molecules NO$_2$(dmgH)$_2$Co(PPh$_3$) held together by Van der Waals forces. A side view of the molecule is shown in Figure 4. The dmgH units are both nearly planar with ±0.002 Å, their planes making an angle of 7.7°. The four equatorial N atoms are coplanar within ±0.007 Å, the cobalt atom being displaced by 0.042 Å from this plane towards the P atom. The coordination plane makes angles of 5.7 and 2.3° with the two planes passing through the two dmgH units, the equatorial ligands assuming an umbrella shape. The amount of such distortion, very similar to that found in Cl(dmgH)$_2$CoPPh$_3$ [1] (6.0 and 4.3°, respectively), confirms the noticeable rigidity of the equatorial ligand, which can be only slightly distorted by axial ligands. The NO$_2$ group plane is perpendicular to the equatorial plane (90.1°) and it has an orientation close to that of the NO$_2$ groups of (I). The orientation of PPh$_3$ with respect to the equatorial ligand is the same as that of PPh$_3$ in the isomorphous Cl(dmgH)$_2$CoPPh$_3$ [1]. Bond lengths are shown in Figure 5 together with the numbering scheme for the atoms. Bond angles of interest are shown in Figure 6. The accuracy of the data did not allow us to locate hydrogen atoms in the O···O bridge, so that we have assumed the ligands to be present as mono-anions dmgH on the basis of the Co–N bond lengths. The bond lengths and angles of Figure 5 and 6 are in the ranges already reported [1–6].

The Co–NO_2 distance of 1.980(9) Å appears significantly longer than those found in (I) which average to 1.946(9). The lengthening of this Co–N bond could be attributed to the *trans*-influence of the PPh_3 group. On the other hand the Co–P bond length of 2.392(3) Å is unexpected, since it is markedly longer than that of 2.327(4) Å found in $Cl(dmgH)_2$-$CoPPh_3$ [1], unless a strong *trans*-influence of the nitro-group is assumed.

Structural data confirm that the compound (II) is the nitro-isomer, as already suggested by Marzilli *et et al.* [15] in contrast with Hill's formulation as a nitrito isomer [16]. In fact the n.m.r. spectrum [17] of the sample, from which crystals have been chosen for the X-ray analysis, is that attributed to the nitrito isomer by the latter authors.

Acknowledgements

The authors thank Dr. G. Tauzher for supplying the compounds and C.N.R. (Rome) for financial support. Professor L. G. Marzilli is acknowledged for having discussed the manuscript.

References

1 S. Brückner and L. Randaccio, *J. Chem. Soc. Dalton,* 1017 (1974).
2 L. Randaccio and E. Zangrando, *Cryst. Struct. Comm., 3,* 565 (1974).
3 L. G. Marzilli, L. A. Epps, T. Sorrell, and T. J. Kistenmaker, *J. Am. Chem. Soc., 97,* 3351 (1975).
4 A. Bigotto, L. Randaccio, and E. Zangrando, *J. Chem. Soc. Dalton,* 96 (1976).
5 D. L. McFadden and A. T. McPhail, *J. Chem. Soc. Dalton,* 363 (1974).
6 G. J. Palenik, D. A. Sullivan, and D. V. Naik, *J. Am. Chem. Soc., 98,* 1177 (1976).
7 C. B. Singh and B. Sahoo, *J. Inorg. Nucl. Chem., 36,* 1259 (1974).
8 S. J. Gruber, C. M. Harris, and E. Sinn, *Inorg. Chem., 7,* 268 (1968).
9 G. H. W. Milburn, M. R. Truter, and B. L. Vickery, *Chem. Comm.,* 1188 (1968); F. Calderazzo, C. Floriani, and L. Randaccio, *Chem. Comm.,* 384 (1973); L. Randaccio, *Gazzetta, 104,* 991 (1974); N. Bresciani-Pahor, M. Calligaris, P. Delise, G. Nardin, L. Randaccio, E. Zotti, G. Fachinetti, and C. Floriani, *J. Chem. Soc. Dalton,* 2310 (1976).
10 F. H. Moore, *Acta Cryst., 16,* 1169 (1963).
11 V. Albano, A. Domenicano, and A. Vaciago, *Gazzetta, 3,* 565 (1966).
12 S. R. Holbrook and D. Van der Helm, *Acta Cryst., B31,* 1658 (1975).
13 B. K. Nurmann and E. O. S. Schlenger, *Inorg. Chem., 12,* 2625 (1973).
14 M. Ito, F. Marumo, and Y. Saito, *Acta Cryst., B28,* 463 (1972).
15 W. C. Trogler, R. C. Stewart, L. A. Epps, and L. G. Marzilli, *Inorg. Chem., 13,* 1564 (1974).
16 H. A. O. Hill and K. G. Morallee, *J. Chem. Soc. A,* 554 (1969).
17 L. G. Marzilli, personal communication.