Refinement

Refinement on *F*
$R = 0.022$
$wR = 0.038$
$S = 1.301$
1502 reflections
52 parameters
No H atoms located
$w = 1/[\sigma^2(F) + 0.0003F^2]$
$(\Delta/\sigma)_{\max} = 0.003$
$\Delta\rho_{\max} = 2.00$ e Å^{-3}
$\Delta\rho_{\min} = -1.24$ e Å^{-3}
Extinction correction: *SHELXTL-Plus* (Sheldrick, 1989)
Extinction coefficient: 0.0030 (1)
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.3.1)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Nd	0	0.32466 (1)	1/4	0.0106 (1)
Co	0	0	0	0.0103 (2)
C1	0.3195 (2)	0.4548 (1)	0.0870 (1)	0.0169 (6)
C2	0	0.1337 (2)	0.0579 (2)	0.017 (1)
N1	0.2064 (3)	0.4266 (2)	0.1382 (1)	0.0267 (8)
N2	0	0.2148 (2)	0.0938 (2)	0.027 (1)
O1	0.2649 (4)	0.2132 (3)	1/4	0.036 (2)
O2	0	0.6544 (2)	0.0997 (2)	0.035 (2)

Table 2. *Selected geometric parameters (Å, °)*

Co—C1$^{\rm i}$	1.886 (2)	Nd—O1	2.429 (3)
Co—C2	1.885 (3)	C1—N1	1.153 (3)
Nd—N1	2.531 (2)	C2—N2	1.149 (4)
Nd—N2	2.563 (3)		
N1—Nd—N1$^{\rm ii}$	74.7 (1)	C1$^{\rm i}$—Co—C1$^{\rm vii}$	90.6 (1)
N1—Nd—N1$^{\rm iii}$	117.9 (1)	C1$^{\rm i}$—Co—C1$^{\rm viii}$	180.0 (1)
N1—Nd—N1$^{\rm iv}$	74.5 (1)	C1$^{\rm i}$—Co—C2$^{\rm ix}$	89.4 (1)
N1—Nd—N2	77.0 (1)	C1$^{\rm i}$—Co—C2	90.6 (1)
N1—Nd—N2$^{\rm v}$	142.2 (1)	C2—Co—C2$^{\rm ix}$	180.0 (1)
N1—Nd—O1	79.2 (1)	Co$^{\rm x}$—C1—N1	178.3 (2)
N1—Nd—O1$^{\rm iii}$	142.4 (1)	Co—C2—N2	179.6 (3)
N2—Nd—N2$^{\rm v}$	113.5 (1)	Nd—N1—C1	166.4 (2)
N2—Nd—O1	71.2 (1)	Nd—N2—C2	148.6 (2)
O1—Nd—O1$^{\rm iii}$	108.2 (1)	O2$^{\rm xi}$—O1—O2$^{\rm xii}$	94.7 (1)
C1$^{\rm i}$—Co—C1$^{\rm vi}$	89.4 (1)	O1$^{\rm xiii}$—O2—O1$^{\rm xiv}$	77.0 (1)

Symmetry codes: (i) $x-\frac{1}{2}, y-\frac{1}{2}, z$; (ii) $x, y, \frac{1}{2}-z$; (iii) $-x, y, \frac{1}{2}-z$; (iv) $-x, y, z$; (v) $x, y, \frac{3}{2}-z$; (vi) $x-\frac{1}{2}, \frac{1}{2}-y, -z$; (vii) $\frac{1}{2}-x, y-\frac{1}{2}, z$; (viii) $\frac{1}{2}-x, \frac{1}{2}-y, -z$; (ix) $-x, -y, -z$; (x) $\frac{1}{2}+x, \frac{1}{2}+y, z$; (xi) $\frac{1}{2}+x, y-\frac{1}{2}, z$; (xii) $\frac{1}{2}+x, y-\frac{1}{2}, \frac{1}{2}-z$; (xiii) $x-\frac{1}{2}, \frac{1}{2}+y, z$; (xiv) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

A conoscopic rotational examination using two crossed polarizers on a Zeiss Photomicroscope II confirmed the optical quality and biaxial nature of the crystal. A Mattson FTIR spectrometer using the KBr pressed pellet method was employed to obtain pertinent spectral data. The water molecules of hydration [3.9 (1) water molecules per formula unit] were verified by a thermal gravimetric analysis (Perkin-Elmer TGS-1). Excess maximum and minimum density in the difference Fourier is in the area of the heavy lanthanide atom. The structure was checked for additional symmetry by the *MISSYM* program (Le Page, 1987, 1988; Gabe, Le Page, Charland, Lee & White, 1989).

Data collection: *CAD*-4 *Software* (Enraf-Nonius, 1988). Cell refinement: *CAD*-4 *Software*. Data reduction: *SDP* (Enraf-Nonius, 1985). Program(s) used to solve structure: *SHELXTL-Plus* (Sheldrick, 1989). Program(s) used to refine structure: *SHELXTL-Plus*. Molecular graphics: *SHELXTL-Plus XP*. Software used to prepare material for publication: *CIFGEN* (local program).

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Lists of structure factors, anisotropic displacement parameters and complete geometry have been deposited with the IUCr (Reference: BR1115). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Anhydrous *trans*-(Aniline)chlorobis(dimethylglyoximato)cobalt(III)

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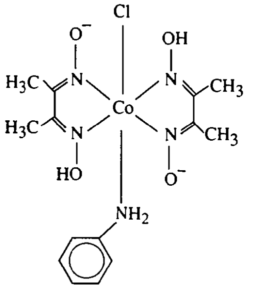
Abstract

The crystal structure of anhydrous *trans*-(aniline)-chlorobis[dimethylglyoximato(1−)]cobalt(III), $[\mathrm{CoCl(C_4H_7N_2O_2)_2(C_6H_7N)}]$, has been determined. The Co ion shows distorted octahedral coordination with the N atoms of the dimethylglyoximato ligands in the equatorial sites and the N atom of aniline and the Cl atom in the apical sites. From the values of the Co—N distances and the N—Co—N angles it is deduced that both equa-

torial ligands are dimethylglyoximato(1−), while in the previously determined dihydrated compound the protonation sites are such as to give a (dimethylglyoxime)[dimethylglyoximato(2−)]cobalt complex.

Comment

The chemistry, spectral and structural properties of *trans*-bis(ligand)bis(dimethylglyoximato)cobalt(III) complexes of the type $[Co(dmgH)_2X(L)]$ have been explored extensively in recent years (Bresciani-Pahor *et al.*, 1985; Toscano & Marzilli, 1984; Samus & Ablov, 1979; Chakravorty, 1974) because of interest in their use as models for important biochemical complexes (Schrauzer & Kohnle, 1964; Schrauzer, 1968; Dolphin, 1982) as well as their usefulness as templates in many organic syntheses (Langer, 1984) or as catalysts in chemical processes (Rockenbauer, Eyor, Kwiecinski & Tyrlik, 1982; Noguchi & Kambara, 1963; Nemeth & Simandi, 1982). In the course of our work on cobaloxime compounds (López, Alvarez, Aguiló, Solans & Font-Altaba, 1987; López, Alvarez, Solans & Aguiló, 1987, and references therein) we isolated the title compound, (I), with a different state of hydration to that reported previously. The crystal structure of the dihydrate was reported by Botoshanski, Simonov, Malinovskii, Ablov & Bologa (1975). We now compare the results of the crystal structure determination of the anhydrous compound with those obtained for the dihydrate compound.



(I)

Fig. 1 shows an *ORTEP* (Johnson, 1976; Brueggemann & Schmid, 1990) drawing of the molecule. The Co ion has distorted octahedral coordination from two dimethylglyoximato ligands in the equatorial positions, and the Cl atom and N atom of aniline in the apical sites. The CoN_4(glyoximato) system is planar with the largest deviations from the mean plane being −0.006 (9) for molecule *A* and 0.022 (9) Å for molecule *B*. The N(glyoximato)—Co—N(aniline) bond angle varies from 87.8 (4) to 93.9 (4)° in the two molecules of (I) compound, and from 88.9 to 90.8° in the dihydrate compound (Botoshanski *et al.*, 1975).

In the dihydrate compound the two H atoms linked to the O atoms in the dimethylglyoximato moiety were found to be bound to the O atoms of the same ligand, so that the dihydrate compound must be formulated as $[Cl(aniline)(dmgH_2)(dmg)Co]$, analogous to $[(H_2O)(CN)(dmgH_2)(dmg)Co]$ (Solans, Font-Altaba & Briansó, 1983) and $[Cl(dimethylpyrazine)(gH_2)(g)Co]$ (g = glyoximato) (López, Alvarez, Solans & Font-Altaba, 1986). A comparative study of bond distances and angles has been carried out for 27 structures of type $[(X)(L)(Y_2gH)_2Co]$ (Y = H, CH_3 or C_6H_5) retrieved from the Cambridge Structural Database (Allen *et al.*, 1979). The Co—N(glyoximato) bond length shows two mean values: 1.879 (9) for Co—NO and 1.903 (10) Å for Co—NOH. The N(glyoximato)—Co—N(glyoximato) angle for the N atoms of the same ligand shows three mean values: 80.0 (8) for Co(Yg); 80.9 (7) for Co(YgH) and 81.8 (6)° for Co(YgH_2). The differences between these values are not statistically significant, but they do suggest the formulation of the title compound.

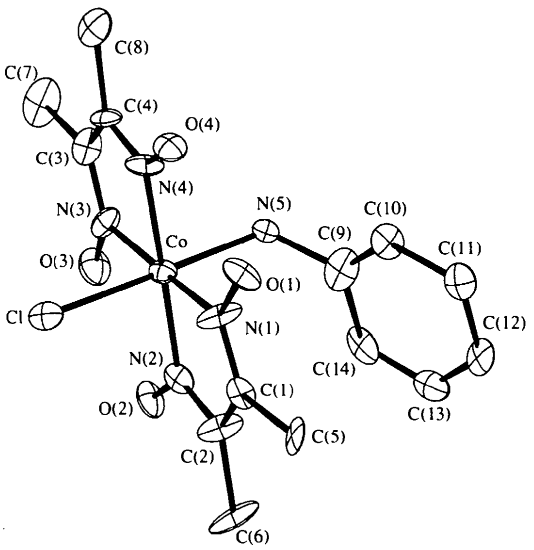


Fig. 1. Molecular structure showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

1.81 g (5 mmol) of *trans*-$[Co(dmgH)(dmgH_2)Cl_2]$ (Reigl & Rubistein, 1923; Trogler, Stewart, Epps & Marzilli, 1974) were added to an ethanolic solution (75 ml) of KOH (0.28 g, 5 mmol). When most of the starting material had dissolved, 0.5 ml (5.2 mmol) of aniline were added, and the resulting mixture was stirred at room temperature for 1 h. The brown precipitate formed was collected by filtration, washed with three (10 ml) portions of water and air-dried. The crystals were grown by slow evaporation at 277 K of an acetone/water (10:1) solution of the complex.

Crystal data

$[CoCl(C_4H_7N_2O_2)_2(C_6H_7N)]$
M_r = 417.72

Mo $K\alpha$ radiation
λ = 0.71069 Å

Monoclinic

$P2_1/c$

$a = 19.929$ (4) Å

$b = 10.855$ (3) Å

$c = 18.334$ (4) Å

$\beta = 117.28$ (3)°

$V = 3525.1$ (14) Å^3

$Z = 8$

$D_x = 1.567$ Mg m^{-3}

Cell parameters from 25 reflections

$\theta = 8$–12°

$\mu = 1.155$ mm^{-1}

$T = 293$ (2) K

Prismatic

0.08 × 0.05 × 0.05 mm

Dark brown

Data collection

Philips PW1100 four-circle diffractometer

ω scans

Absorption correction: none

2432 measured reflections

2129 independent reflections

1906 observed reflections $[I > 2\sigma(I)]$

$R_{\rm int} = 0.037$

$\theta_{\rm max} = 25°$

$h = -22 \rightarrow 20$

$k = 0 \rightarrow 12$

$l = 0 \rightarrow 18$

3 standard reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F^2

$R(F) = 0.041$

$wR(F^2) = 0.095$

$S = 0.936$

1906 reflections

447 parameters

38 H atoms computed using riding model and overall isotropic displacement coefficient (OH H atoms not localized nor considered in calculations)

$w = 1/[\sigma^2(I) + (0.1016P)^2 + 110.91P]$ where $P = (|F_o|^2 + 2|F_c|^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.8$

$\Delta\rho_{\rm max} = 0.52$ e Å^{-3}

$\Delta\rho_{\rm min} = -0.35$ e Å^{-3}

Extinction correction: *SHELXL*93 (Sheldrick, 1993)

Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
Co(*A*)	1.0799 (1)	1.1866 (1)	0.2915 (1)	0.031 (1)
Cl(*A*)	1.0899 (2)	0.9964 (3)	0.2501 (2)	0.044 (1)
O(1*A*)	1.0306 (5)	1.0967 (7)	0.4034 (5)	0.052 (2)
O(2*A*)	1.2290 (4)	1.2256 (8)	0.3172 (5)	0.049 (2)
O(3*A*)	1.1282 (4)	1.2920 (7)	0.1826 (5)	0.053 (2)
O(4*A*)	0.9300 (4)	1.1321 (8)	0.2650 (5)	0.045 (2)
N(1*A*)	1.0902 (6)	1.1270 (9)	0.3896 (6)	0.046 (3)
N(2*A*)	1.1875 (5)	1.1899 (8)	0.3524 (7)	0.036 (3)
N(3*A*)	1.0664 (5)	1.2462 (9)	0.1915 (5)	0.042 (3)
N(4*A*)	0.9721 (4)	1.1800 (9)	0.2325 (7)	0.040 (3)
N(5*A*)	1.0734 (5)	1.3599 (8)	0.3263 (6)	0.034 (3)
C(1*A*)	1.1576 (8)	1.1152 (9)	0.4479 (8)	0.038 (3)
C(2*A*)	1.2163 (7)	1.1537 (12)	0.4258 (8)	0.052 (4)
C(3*A*)	1.0010 (7)	1.2687 (11)	0.1341 (7)	0.037 (3)
C(4*A*)	0.9465 (6)	1.2182 (10)	0.1585 (7)	0.032 (3)
C(5*A*)	1.1786 (7)	1.0782 (11)	0.5377 (7)	0.054 (4)
C(6*A*)	1.2994 (7)	1.1488 (14)	0.4790 (8)	0.077 (5)
C(7*A*)	0.9818 (8)	1.3115 (12)	0.0542 (8)	0.067 (4)
C(8*A*)	0.8600 (4)	1.2297 (6)	0.1045 (4)	0.059 (4)
C(9*A*)	1.1051 (4)	1.3913 (6)	0.4098 (4)	0.036 (3)
C(10*A*)	1.1833 (4)	1.4210 (6)	0.4531 (4)	0.052 (4)
C(11*A*)	1.2145 (7)	1.4379 (11)	0.5365 (10)	0.062 (5)
C(12*A*)	1.1721 (9)	1.4406 (10)	0.5770 (9)	0.062 (5)
C(13*A*)	1.0934 (8)	1.4182 (10)	0.5317 (8)	0.044 (3)
C(14*A*)	1.0629 (7)	1.3951 (11)	0.4494 (7)	0.042 (3)
Co(*B*)	0.4177 (1)	0.7700 (1)	0.2036 (1)	0.026 (1)
Cl(*B*)	0.4089 (2)	0.9601 (3)	0.1501 (2)	0.041 (1)
O(1*B*)	0.2779 (4)	0.7006 (7)	0.0799 (5)	0.039 (2)
O(2*B*)	0.4566 (4)	0.8855 (7)	0.3565 (5)	0.043 (2)
O(3*B*)	0.5651 (4)	0.8285 (8)	0.3322 (5)	0.049 (2)
O(4*B*)	0.3780 (5)	0.6704 (8)	0.0472 (5)	0.055 (2)
N(1*B*)	0.3107 (4)	0.7536 (8)	0.1535 (6)	0.028 (2)
N(2*B*)	0.4005 (5)	0.8422 (7)	0.2868 (5)	0.027 (2)
N(3*B*)	0.5256 (5)	0.7854 (9)	0.2548 (7)	0.040 (3)
N(4*B*)	0.4339 (5)	0.7036 (8)	0.1195 (5)	0.033 (2)
N(5*B*)	0.4245 (5)	0.5980 (8)	0.2473 (5)	0.041 (3)
C(1*B*)	0.2795 (7)	0.7878 (10)	0.1964 (7)	0.035 (3)
C(2*B*)	0.3303 (7)	0.8446 (11)	0.2769 (8)	0.047 (4)
C(3*B*)	0.5568 (6)	0.7378 (12)	0.2115 (8)	0.045 (3)
C(4*B*)	0.5046 (6)	0.7028 (9)	0.1309 (7)	0.025 (3)
C(5*B*)	0.1969 (6)	0.7758 (12)	0.1686 (8)	0.064 (4)
C(6*B*)	0.3080 (7)	0.8971 (12)	0.3347 (8)	0.052 (4)
C(7*B*)	0.6409 (6)	0.7276 (12)	0.2458 (8)	0.063 (4)
C(8*B*)	0.5303 (7)	0.6600 (12)	0.0724 (7)	0.053 (4)
C(9*B*)	0.3970 (7)	0.5740 (10)	0.3058 (7)	0.033 (3)
C(10*B*)	0.3203 (7)	0.5379 (11)	0.2737 (8)	0.045 (3)
C(11*B*)	0.2945 (8)	0.5266 (12)	0.3309 (9)	0.055 (4)
C(12*B*)	0.3350 (7)	0.5448 (13)	0.4102 (9)	0.054 (4)
C(13*B*)	0.4100 (7)	0.5827 (11)	0.4386 (8)	0.052 (4)
C(14*B*)	0.4408 (8)	0.5952 (11)	0.3859 (8)	0.049 (4)

Table 2. *Selected geometric parameters (Å, °)*

Co(*A*)—N(1*A*)	1.831 (10)	Co(*B*)—N(1*B*)	1.904 (8)
Co(*A*)—N(2*A*)	1.911 (8)	Co(*B*)—N(2*B*)	1.880 (8)
Co(*A*)—N(3*A*)	1.845 (9)	Co(*B*)—N(3*B*)	1.920 (9)
Co(*A*)—N(4*A*)	1.913 (8)	Co(*B*)—N(4*B*)	1.859 (8)
Co(*A*)—N(5*A*)	2.010 (9)	Co(*B*)—N(5*B*)	2.012 (9)
Co(*A*)—Cl(*A*)	2.240 (3)	Co(*B*)—Cl(*B*)	2.257 (3)
O(1*A*)—N(1*A*)	1.364 (12)	O(1*B*)—N(1*B*)	1.338 (10)
O(2*A*)—N(2*A*)	1.318 (11)	O(2*B*)—N(2*B*)	1.339 (10)
O(3*A*)—N(3*A*)	1.403 (11)	O(3*B*)—N(3*B*)	1.353 (12)
O(4*A*)—N(4*A*)	1.337 (12)	O(4*B*)—N(4*B*)	1.333 (10)
N(1*A*)—C(1*A*)	1.283 (13)	N(1*B*)—C(1*B*)	1.260 (14)
N(2*A*)—C(2*A*)	1.259 (14)	N(2*B*)—C(2*B*)	1.326 (13)
N(3*A*)—C(3*A*)	1.268 (13)	N(3*B*)—C(3*B*)	1.317 (14)
N(4*A*)—C(4*A*)	1.280 (14)	N(4*B*)—C(4*B*)	1.327 (11)
N(5*A*)—C(9*A*)	1.405 (11)	N(5*B*)—C(9*B*)	1.434 (13)
C(1*A*)—C(2*A*)	1.46 (2)	C(1*B*)—C(2*B*)	1.49 (2)
C(1*A*)—C(5*A*)	1.55 (2)	C(1*B*)—C(5*B*)	1.49 (2)
C(2*A*)—C(6*A*)	1.49 (2)	C(2*B*)—C(6*B*)	1.44 (2)
C(3*A*)—C(7*A*)	1.41 (2)	C(3*B*)—C(7*B*)	1.50 (2)
C(3*A*)—C(4*A*)	1.46 (2)	C(3*B*)—C(4*B*)	1.415 (14)
C(4*A*)—C(8*A*)	1.549 (12)	C(4*B*)—C(8*B*)	1.460 (14)
C(9*A*)—C(10*A*)	1.42 (2)	C(9*B*)—C(10*B*)	1.42 (2)
C(9*A*)—C(14*A*)	1.339 (13)	C(9*B*)—C(14*B*)	1.34 (2)
C(11*A*)—C(10*A*)	1.37 (2)	C(11*B*)—C(10*B*)	1.37 (2)
C(12*A*)—C(11*A*)	1.36 (2)	C(12*B*)—C(11*B*)	1.32 (2)
C(13*A*)—C(12*A*)	1.42 (2)	C(13*B*)—C(12*B*)	1.40 (2)
C(14*A*)—C(13*A*)	1.366 (14)	C(14*B*)—C(13*B*)	1.37 (2)
N(2*A*)—Co(*A*)—N(1*A*)	80.9 (5)	N(2*B*)—Co(*B*)—N(1*B*)	81.4 (4)
N(1*A*)—Co(*A*)—N(3*A*)	178.3 (4)	N(1*B*)—Co(*B*)—N(3*B*)	179.5 (4)
N(2*A*)—Co(*A*)—N(3*A*)	100.7 (5)	N(2*B*)—Co(*B*)—N(3*B*)	98.4 (4)
N(1*A*)—Co(*A*)—N(4*A*)	97.7 (5)	N(1*B*)—Co(*B*)—N(4*B*)	98.4 (4)
N(2*A*)—Co(*A*)—N(4*A*)	178.5 (5)	N(2*B*)—Co(*B*)—N(4*B*)	178.1 (4)
N(3*A*)—Co(*A*)—N(4*A*)	80.6 (5)	N(3*B*)—Co(*B*)—N(4*B*)	81.8 (4)
N(1*A*)—Co(*A*)—N(5*A*)	90.8 (4)	N(1*B*)—Co(*B*)—N(5*B*)	87.8 (4)
N(2*A*)—Co(*A*)—N(5*A*)	90.9 (4)	N(2*B*)—Co(*B*)—N(5*B*)	93.9 (4)
N(3*A*)—Co(*A*)—N(5*A*)	89.2 (4)	N(3*B*)—Co(*B*)—N(5*B*)	91.7 (4)
N(4*A*)—Co(*A*)—N(5*A*)	89.7 (4)	N(4*B*)—Co(*B*)—N(5*B*)	88.0 (4)
N(1*A*)—Co(*A*)—Cl(*A*)	91.1 (3)	N(1*B*)—Co(*B*)—Cl(*B*)	91.7 (3)
N(2*A*)—Co(*A*)—Cl(*A*)	88.0 (3)	N(2*B*)—Co(*B*)—Cl(*B*)	88.0 (3)
N(3*A*)—Co(*A*)—Cl(*A*)	88.9 (3)	N(3*B*)—Co(*B*)—Cl(*B*)	88.8 (3)
N(4*A*)—Co(*A*)—Cl(*A*)	91.5 (3)	N(4*B*)—Co(*B*)—Cl(*B*)	90.1 (3)
N(5*A*)—Co(*A*)—Cl(*A*)	177.5 (3)	N(5*B*)—Co(*B*)—Cl(*B*)	178.0 (3)
C(1*A*)—N(1*A*)—O(1*A*)	119.3 (10)	C(1*B*)—N(1*B*)—O(1*B*)	127.3 (10)
C(1*A*)—N(1*A*)—Co(*A*)	117.2 (9)	C(1*B*)—N(1*B*)—Co(*B*)	115.8 (9)
O(1*A*)—N(1*A*)—Co(*A*)	123.5 (7)	O(1*B*)—N(1*B*)—Co(*B*)	116.8 (7)
C(2*A*)—N(2*A*)—O(2*A*)	122.3 (11)	C(2*B*)—N(2*B*)—O(2*B*)	119.4 (9)
C(2*A*)—N(2*A*)—Co(*A*)	117.2 (9)	C(2*B*)—N(2*B*)—Co(*B*)	118.0 (8)
O(2*A*)—N(2*A*)—Co(*A*)	120.5 (8)	O(2*B*)—N(2*B*)—Co(*B*)	122.5 (6)
C(3*A*)—N(3*A*)—O(3*A*)	117.3 (10)	C(3*B*)—N(3*B*)—O(3*B*)	124.1 (10)
C(3*A*)—N(3*A*)—Co(*A*)	121.1 (9)	C(3*B*)—N(3*B*)—Co(*B*)	113.8 (9)
O(3*A*)—N(3*A*)—Co(*A*)	120.2 (7)	O(3*B*)—N(3*B*)—Co(*B*)	121.7 (8)

C(4*A*)—N(4*A*)—O(4*A*)	124.8 (9)	C(4*B*)—N(4*B*)—O(4*B*)	119.8 (9)
C(4*A*)—N(4*A*)—Co(*A*)	112.7 (9)	C(4*B*)—N(4*B*)—Co(*B*)	116.7 (7)
O(4*A*)—N(4*A*)—Co(*A*)	122.3 (8)	O(4*B*)—N(4*B*)—Co(*B*)	123.1 (7)
C(9*A*)—N(5*A*)—Co(*A*)	120.7 (6)	C(9*B*)—N(5*B*)—Co(*B*)	119.2 (7)
N(1*A*)—C(1*A*)—C(2*A*)	113.8 (11)	N(1*B*)—C(1*B*)—C(2*B*)	115.8 (11)
N(1*A*)—C(1*A*)—C(5*A*)	125.4 (12)	N(1*B*)—C(1*B*)—C(5*B*)	123.0 (11)
C(2*A*)—C(1*A*)—C(5*A*)	120.4 (11)	C(2*B*)—C(1*B*)—C(5*B*)	121.2 (12)
N(2*A*)—C(2*A*)—C(1*A*)	110.8 (11)	N(2*B*)—C(2*B*)—C(1*B*)	108.8 (11)
N(2*A*)—C(2*A*)—C(6*A*)	122 (2)	N(2*B*)—C(2*B*)—C(6*B*)	124.7 (11)
C(1*A*)—C(2*A*)—C(6*A*)	126.7 (13)	C(1*B*)—C(2*B*)—C(6*B*)	126.5 (12)
N(3*A*)—C(3*A*)—C(7*A*)	127.5 (12)	N(3*B*)—C(3*B*)—C(7*B*)	121.4 (11)
N(3*A*)—C(3*A*)—C(4*A*)	107.5 (10)	N(3*B*)—C(3*B*)—C(4*B*)	114.3 (10)
C(7*A*)—C(3*A*)—C(4*A*)	123.8 (12)	C(7*B*)—C(3*B*)—C(4*B*)	124.2 (11)
N(4*A*)—C(4*A*)—C(8*A*)	119.4 (10)	N(4*B*)—C(4*B*)—C(8*B*)	126.3 (10)
N(4*A*)—C(4*A*)—C(3*A*)	117.1 (10)	N(4*B*)—C(4*B*)—C(3*B*)	112.4 (9)
C(8*A*)—C(4*A*)—C(3*A*)	122.7 (10)	C(8*B*)—C(4*B*)—C(3*B*)	121.1 (10)
C(10*A*)—C(9*A*)—C(14*A*)	119.3 (6)	C(10*B*)—C(9*B*)—C(14*B*)	122.6 (12)
C(10*A*)—C(9*A*)—N(5*A*)	119.5 (5)	C(10*B*)—C(9*B*)—N(5*B*)	116.6 (11)
C(14*A*)—C(9*A*)—N(5*A*)	121.3 (9)	C(14*B*)—C(9*B*)—N(5*B*)	120.6 (11)
C(11*A*)—C(10*A*)—C(9*A*)	118.2 (7)	C(11*B*)—C(10*B*)—C(9*B*)	114.7 (12)
C(10*A*)—C(11*A*)—C(12*A*)	122.3 (12)	C(10*B*)—C(11*B*)—C(12*B*)	125.3 (13)
C(13*A*)—C(12*A*)—C(11*A*)	118.3 (13)	C(13*B*)—C(12*B*)—C(11*B*)	117.6 (13)
C(12*A*)—C(13*A*)—C(14*A*)	119.3 (12)	C(12*B*)—C(13*B*)—C(14*B*)	121.0 (13)
C(13*A*)—C(14*A*)—C(9*A*)	122.2 (11)	C(13*B*)—C(14*B*)—C(9*B*)	118.6 (13)

The structure was solved by direct methods and refined by full-matrix least squares. A pseudo-orthorhombic cell exists with $a_o = 18.334$, $b_o = 35.425$, $c_o = 10.855$ Å [transformation matrix: (001/201/010)]; space group *Cmca*, but the Laue class was monoclinic and $R_{\rm int}$ for the orthorhombic cell was 0.317, so the determination and refinement were carried out using the monoclinic cell. Owing to the small size of the crystal, a low number of non-negative intensities were collected and the resolution was of low accuracy.

Data collection: Philips diffractometer software. Cell refinement: Philips diffractometer software. Data reduction: *CFEO* (Solans, 1978). Structure solution: *SHELXS*86 (Sheldrick, 1985). Structure refinement: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP* (Johnson, 1976; Brueggemann & Schmid, 1990). Preparation of material for publication: *CIFTAB* (Sheldrick, 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: NA1144). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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trans-Tetracarbonylbis(tri-*tert*-butyl phosphite)molybdenum(0)

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Abstract

The Mo atom in the title compound, $[Mo(CO)_4(C_{12}H_{27}O_3P)_2]$, lies on an inversion centre and has slightly distorted octahedral geometry, with principal dimensions Mo—P 2.4699 (7), Mo—C 2.027 (4), 2.031 (3) Å, P—Mo—P 180, *cis*-C—Mo—C 88.7 (2), C—Mo—P 86.12 (10) and 87.27 (9)°. The geometry about the P atom is distorted from tetrahedral, with Mo—P—O angles in the range 108.04 (9)–126.03 (10)° and reduced O—P—O angles in the range 96.71 (13)–106.09 (14)°. The average cone angle for the *tert*-butyl phosphite ligand is 156°.

Comment

The title compound, $[Mo(CO)_4\{P(O^tBu)_3\}_2]$, (1), was synthesized during our investigations of numerous $[Mo(CO)_{6-n}(PY_3)_n]$ (n = 1–3) complexes by ^{95}Mo