C2	0.2299 (3)	0.4120 (3)	0.2985 (3)	4.01 (5)
C3	−0.0706 (3)	0.6384 (3)	0.1959 (3)	3.70 (5)
C4	−0.0107 (4)	0.2173 (4)	0.3827 (3)	4.29 (6)
C5	−0.1688 (3)	0.3039 (4)	0.2852 (4)	4.58 (6)
C6	−0.1042 (3)	0.2252 (3)	0.1417 (3)	4.52 (5)
C7	0.0929 (4)	0.0940 (3)	0.1498 (3)	4.20 (5)
C8	0.1540 (3)	0.0846 (3)	0.3000 (3)	3.97 (5)
C9	0.3466 (4)	−0.0494 (5)	0.3638 (4)	5.72 (8)

Table 2. *Bond lengths* (Å) *and angles* (°)

Cr—Cr	3.247 (1)	O3—C3	1.148 (3)
Cr—C1	1.863 (2)	Cr—C2	1.833 (2)
Cr—C3	1.859 (3)	Cr—C4	2.179 (3)
Cr—C5	2.197 (3)	Cr—C6	2.214 (3)
Cr—C7	2.193 (3)	Cr—C8	2.198 (3)
O1—C1	1.146 (3)	O2—C2	1.151 (3)
C4—C8	1.420 (4)	C5—C6	1.407 (4)
C7—C8	1.412 (4)	C8—C9	1.484 (4)
C6—C7	1.395 (4)	C4—C5	1.401 (4)
Cr—Cr—C1	72.27 (8)	C7—C8—C9	126.7 (3)
Cr—Cr—C3	71.65 (7)	Cr—Cr—C2	128.21 (9)
Cr—Cr—C4	141.71 (8)	Cr—Cr—C5	104.53 (8)
Cr—Cr—C6	84.00 (8)	Cr—Cr—C7	101.31 (7)
Cr—Cr—C8	138.80 (7)	Cr—Cr—C9	145.17 (8)
C2—Cr—C3	79.3 (1)	C1—Cr—C2	79.7 (1)
C1—Cr—C3	110.8 (1)	Cr—C1—O1	172.2 (2)
Cr—C2—O2	179.4 (3)	Cr—C3—O3	172.4 (2)
C5—C4—C8	108.8 (3)	C4—C5—C6	107.6 (3)
C5—C6—C7	108.3 (3)	C6—C7—C8	108.9 (2)
C4—C8—C7	106.4 (2)	C4—C8—C9	126.7 (3)

Table 3. *A comparison of geometric parameters* (Å, °) *for* $[Cr(MeC_5H_4)(CO_3)]_2$ *and its* C_5H_5 *and* C_5Me_5 *analogues*

	$(Cp)_2Cr_2(CO)_6$	$(MeCp)_2Cr_2(CO)_6$	$(Me_5Cp)_2Cr_2(CO)_6$
Cr—Cr	3.281 (1)	3.247 (1)	3.310 (1)
Cr—C	1.846–1.870 (3)	1.833–1.863 (2)	1.818–1.859 (3)
Cr—C(Cp)	2.174–2.227 (3)	2.179–2.214 (3)	2.179–2.264 (3)
C—O	1.141–1.146 (3)	1.146–1.151 (3)	1.152–1.157 (4)
Cr—C—O	172.0–178.7 (2)	172.2–179.4 (2)	167.8–178.6 (3)
Cr—Cr—C	79.2 (1)	79.3 (1)	77.9 (1)
	80.8 (1)	79.7 (1)	78.9 (1)
	110.4 (1)	110.8 (1)	117.3 (1)

The structure was solved by Patterson synthesis and refined by full-matrix least-squares methods using *MolEN* (Fair, 1990) on a DEC MicroVAX II minicomputer. The H atoms were located from difference Fourier maps and were refined isotropically.

The authors thank the University of Malaya for support. LYG also acknowledges with thanks an R & D grant (04-07-04-127) from the Malaysian Government.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: MU1098). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). C**50**, 1875–1878

Structural Analogues of Ferroverdin. Tris(methyl isonitrosoacetoacetato)cobalt(III)

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(*Received* 9 *June* 1994; *accepted* 28 *June* 1994)

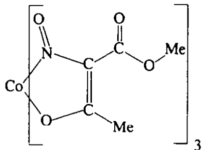
Abstract

The title compound (*OC*-6-22)-tris[methyl 2-(nitroso-*N*)acetoacetato-*O*]cobalt(III), $[Co(C_5H_6NO_4)_3]$, has been synthesized and its structure solved by X-ray diffraction. Each of the three bidentate ligands binds through the N atom of the nitroso group and the ketonic O atom to the Co atom, which displays trigonally distorted *fac*-octahedral coordination. The three ligands display different conformations around the C—C(O)OMe bond.

Comment

Isonitrosoketones and related ligands such as nitrosophenols are used as analytical reagents (Welcher, 1947). Complexes of transition metals with these ligands (Wilkinson, Gillard & McCleverty, 1987; Basu & Chakravorty, 1992) are potential models for metal binding sites in ferroverdin (Ballio, Barcellona, Chain, Tonolo & Vero-Barcellona, 1964; Candeloro *et al.*, 1969) and have industrial applications as dyes and light-absorbing agents for accelerating the solar evaporation of water (Venkataraman, 1952). This has prompted investigation of the structure and overall coordination modes of these complexes.

In view of the importance of metal complexes of isonitrosoketones and nitrosophenols and as a continuation of our work in this field (Raston, Sharma, Skeleton & White, 1978; Figgis, Raston, Sharma & White, 1978*a*,*b*), we have synthesized a large number of tris(isonitrosoketonato)–metal(III) complexes; we report here the synthesis, spectroscopic analysis and X-ray structure determination of tris(methyl isonitrosoacetoacetato)cobalt(III), (I). This is the first reported crystal structure of a metal complex containing the anion of the ligand $CH_3C(O)C(NOH)C(O)OCH_3$.



(I)

Early works by Ponzio (1922) and Taylor & Ewbank (1926) report the synthesis of the bis and tris Co^{III} complexes of isonitrosoacetylacetone $[CH_3C(O)C(NO)C(O)CH_3]$, and the latter work assigned a structural formula with the ligand coordinated *via* the nitrosyl O atom and one of the carbonyl O atoms. Contrary to this, Pfeiffer & Richarz (1928) proposed the involvement of the N atom instead of the oximic O atom for the Co^{III} complex with the related α-benzyl monoxime ligand.

In the case of tris(methyl isonitrosoacetoacetato)-cobalt(III), four possibilities may be considered, involving either the N or O atom of the oxime group and either the ketonic or ester carbonyl O atom. The equivalence of the three coordinated ligands in solution is revealed by ^1H NMR since the possibility of geometrical changes within the timescale of the NMR measurements may be ruled out (Basolo & Pearson, 1967). The inertness of low-spin Co^{III} complexes is not favourable to a rapid *fac–mer* equilibrium at room temperature (Eaton & Eaton, 1973).

The title structure consists of discrete well separated molecules. A view of the molecule showing the numbering scheme appears in Fig. 1. Each ligand is bidentate, binding to the metal *via* the N atom of the nitroso group and the ketonic O atom, thereby forming a five-membered chelate ring. The disposition of the three ligands around the Co atom is *fac*, the overall geometry being very similar to that found in analogous compounds, not only with cobalt(III) (Figgis, Raston, Sharma & White, 1978*b*), but also with other d^6 metal ions, such as iron(II) (Raston, White & Golding, 1977) and ruthenium(II) (Romero, Salas, Simard, Quirós & Beauchamp, 1990).

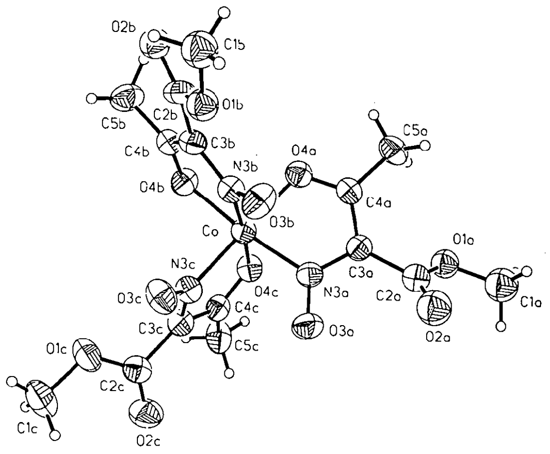


Fig. 1. View of the title molecule. Only one of the two disordered positions of the ester groups of ligands *A* and *B* is shown for clarity. Atoms are represented as 50% probability ellipsoids except for H atoms which are shown as spheres of arbitrary radii.

The coordination octahedron is trigonally distorted, as would be expected for a tris-chelate complex. The individual chelate rings are planar, with the Co atom displaced by less than 0.2 Å and atoms C2, O3 and C5 displaced by less than 0.1 Å from their respective planes defined by atoms N3, C3, C4 and O4.

The methyl ester group (atoms C1, O1 and O2) is disordered over two equally populated positions for ligands *A* and *B* (though not for ligand *C*). Corresponding distances and angles are virtually the same within the three ligands but the conformations around the exocyclic C2—C3 bonds, probably affected by the crystal packing, are quite different, as is shown by the torsion angles in Table 2. The ester carbonyl atom O2 is near N3 for the two disordered sites of ligand *A*, whereas it is near C4 for ligands *B* and *C*. Ligands *B* and *C* differ in the sign of the torsion angle O2—C2—C3—C4 which is negative for the two ligand *B* positions but positive for ligand *C*.

Experimental

Methyl isonitrosoacetoacetate was prepared by an improved version of the method of Adkins & Reeves (1938). Analytical grade hydrated cobaltous acetate was used. When an ethanolic solution of methyl isonitrosoacetoacetate was mixed with an aqueous solution of cobaltous acetate (2:1) and stirred for a few hours, an orange–red solid separated which was filtered, washed with water and dried. The yield was almost quantitative. Ruby-red crystals of the title compound were obtained on recrystallization from an acetone–ethanol solution. Elemental analysis: found, C 36.6, H 3.7, N 8.6%; calculated (for $C_{15}H_{18}CoN_3O_{12}$), C 36.66, H 3.67, N 8.55%. M.p. 461–463 K. Main IR bands: 1715 [ν(C═O)], 1540 [ν(C═N) and ν(C═O)], 1305 [ν(N—O)] and 625 cm^{-1} [ν(Co—N)]. ^1H NMR: 3.80 (*s*, OCH_3), 2.75 p.p.m. (*s*, CH_3). ^{13}C NMR: 27.67 (C5), 52.15 (C1), 146.38 (C3), 158.30 (C4), 210.12 p.p.m. (C2). $M^+ = 491$.

Crystal data

$[Co(C_5H_6NO_4)_3]$
$M_r = 491.25$
Monoclinic
$P2_1/n$
$a = 10.650\ (1)$ Å
$b = 13.895\ (1)$ Å
$c = 13.892\ (1)$ Å
$\beta = 99.55\ (1)°$
$V = 2027\ (1)$ Å^3
$Z = 4$
$D_x = 1.61$ Mg m^{-3}
$D_m = 1.60$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 15–25°$
$\mu = 0.909$ mm^{-1}
$T = 293$ K
Prisms
$0.40 \times 0.25 \times 0.15$ mm
Ruby red

Data collection

Stoe Siemens AED-2 diffractometer
$\omega/2\theta$ scans (learnt profile method)
Absorption correction: none
5085 measured reflections
4641 independent reflections
3145 observed reflections [$F > 4\sigma(F)$]

$R_{\rm int} = 0.038$
$\theta_{\rm max} = 27.51°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 18$
$l = -18 \rightarrow 17$
3 standard reflections
frequency: 90 min
intensity variation: none

Refinement

Refinement on F
$R = 0.056$
$wR = 0.084$
$S = 0.92$
3145 reflections
293 parameters
H atoms riding with C—H = 0.96 Å and a common $U_{\rm iso}$ = 0.098 (6) Å^2

$w = 1/[\sigma^2(F) + 0.00550F^2]$
$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.52$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.49$ e Å^{-3}
Atomic scattering factors from *SHELXTL-Plus* (Sheldrick, 1987)

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

A prime (′) indicates one half of a disordered atom pair, each site having half occupancy and being refined isotropically. For other atoms $U_{\rm eq} = (1/3)\Sigma_i\Sigma_jU_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
Co	0.05856 (5)	0.16735 (4)	0.06819 (4)	0.0336 (2)
C1*A*	−0.209 (1)	0.025 (1)	0.4146 (9)	0.066 (3)
C1*A*′	−0.163 (1)	0.040 (1)	0.4482 (9)	0.077 (4)
O1*A*	−0.1444 (7)	0.0393 (4)	0.3325 (5)	0.047 (2)
O1*A*′	−0.1063 (9)	0.0364 (5)	0.3614 (7)	0.062 (2)
C2*A*	−0.0783 (5)	0.1226 (4)	0.3276 (4)	0.048 (2)
O2*A*	−0.067 (1)	0.1863 (6)	0.3859 (7)	0.064 (3)
O2*A*′	−0.103 (1)	0.1967 (5)	0.3646 (7)	0.060 (3)
C3*A*	−0.0159 (4)	0.1174 (3)	0.2398 (3)	0.037 (1)
N3*A*	−0.0096 (3)	0.1953 (3)	0.1827 (3)	0.035 (1)
O3*A*	−0.0524 (3)	0.2756 (2)	0.1997 (2)	0.051 (1)
C4*A*	0.0419 (4)	0.0341 (3)	0.2047 (3)	0.036 (1)
O4*A*	0.0871 (3)	0.0429 (2)	0.1271 (2)	0.041 (1)
C5*A*	0.0585 (5)	−0.0609 (3)	0.2547 (4)	0.049 (2)
C1*B*	0.6267 (8)	0.2160 (9)	0.2123 (8)	0.058 (3)
C1*B*′	0.6364 (9)	0.2453 (9)	0.1706 (9)	0.061 (3)
O1*B*	0.4915 (6)	0.2210 (5)	0.1709 (5)	0.047 (2)
O1*B*′	0.4996 (6)	0.2493 (4)	0.1336 (5)	0.047 (2)
C2*B*	0.4526 (4)	0.1684 (3)	0.0897 (4)	0.044 (1)
O2*B*	0.5209 (7)	0.1210 (5)	0.0462 (6)	0.046 (2)
O2*B*′	0.5190 (8)	0.1014 (6)	0.0751 (7)	0.064 (3)
C3*B*	0.3124 (4)	0.1665 (3)	0.0614 (3)	0.038 (1)
N3*B*	0.2296 (3)	0.1983 (2)	0.1184 (2)	0.035 (1)
O3*B*	0.2624 (3)	0.2379 (3)	0.1977 (2)	0.053 (1)
C4*B*	0.2486 (4)	0.1260 (3)	−0.0285 (3)	0.039 (1)
O4*B*	0.1290 (3)	0.1251 (2)	−0.0427 (2)	0.041 (1)
C5*B*	0.3165 (5)	0.0856 (4)	−0.1053 (4)	0.055 (2)
C1*C*	−0.0874 (7)	0.4996 (4)	−0.2117 (4)	0.071 (2)
O1*C*	−0.0497 (3)	0.4156 (3)	−0.1514 (3)	0.058 (1)
C2*C*	−0.1408 (4)	0.3764 (3)	−0.1092 (3)	0.043 (1)
O2*C*	−0.2485 (4)	0.4048 (3)	−0.1185 (3)	0.062 (1)
C3*C*	−0.0939 (4)	0.2908 (3)	−0.0490 (3)	0.038 (1)
N3*C*	0.0218 (3)	0.2874 (2)	0.0078 (3)	0.037 (1)
O3*C*	0.0970 (3)	0.3558 (2)	0.0185 (2)	0.052 (1)
C4*C*	−0.1654 (4)	0.2045 (4)	−0.0430 (3)	0.038 (1)
O4*C*	−0.1130 (3)	0.1365 (2)	0.0075 (2)	0.040 (1)
C5*C*	−0.2981 (5)	0.1901 (5)	−0.0950 (4)	0.054 (2)

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

Co—N3*A*	1.895 (4)	Co—O4*B*	1.914 (3)
Co—O4*A*	1.916 (3)	Co—N3*C*	1.879 (3)
Co—N3*B*	1.889 (3)	Co—O4*C*	1.929 (3)
N3*A*—Co—O4*A*	83.2 (1)	N3*B*—Co—N3*C*	94.6 (1)
N3*A*—Co—N3*B*	97.1 (1)	O4*B*—Co—N3*C*	89.6 (2)
O4*A*—Co—N3*B*	88.5 (1)	N3*A*—Co—O4*C*	86.9 (1)
N3*A*—Co—O4*B*	173.8 (1)	O4*A*—Co—O4*C*	93.3 (1)
O4*A*—Co—O4*B*	90.7 (1)	N3*B*—Co—O4*C*	175.8 (1)
N3*B*—Co—O4*B*	83.4 (1)	O4*B*—Co—O4*C*	92.8 (1)
N3*A*—Co—N3*C*	96.5 (2)	N3*C*—Co—O4*C*	83.7 (1)
O4*A*—Co—N3*C*	177.0 (1)		
O1*A*—C2*A*—C3*A*—C4*A*	−38.9 (6)	O1*B*′—C2*B*—C3*B*—C4*B*	140.4 (5)
O1*A*′—C2*A*—C3*A*—C4*A*	−15.9 (7)	O2*B*—C2*B*—C3*B*—C4*B*	−13.0 (8)
O2*A*—C2*A*—C3*A*—C4*A*	137.5 (7)	O2*B*′—C2*B*—C3*B*—C4*B*	−40.4 (9)
O2*A*′—C2*A*—C3*A*—C4*A*	164.9 (7)	O1*C*—C2*C*—C3*C*—C4*C*	−140.8 (4)
O1*B*—C2*B*—C3*B*—C4*B*	171.8 (5)	O2*C*—C2*C*—C3*C*—C4*C*	38.5 (7)

Atoms C1*A*, O1*A*, O2*A*, C1*B*, O1*B* and O2*B* are disordered over two positions, each with half occupancy, and were refined isotropically, restraining the distances C1—O1, C2—O1 and C2—O2 to be equal for the two sites of each ligand.

Data collection: *DIF*4 (Stoe & Cie, 1992*a*). Cell refinement: *DIF*4. Data reduction: *REDU*4 (Stoe & Cie, 1992*b*). Program(s) used to solve structure: *SHELXTL-Plus XS* (Sheldrick, 1987). Program(s) used to refine structure: *SHELXTL-Plus XLS*. Molecular graphics: *SHELXTL-Plus XP*. Software used to prepare material for publication: *PASACIF* (Quirós, 1994).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry, including H-atom geometry, and torsion angles have been deposited with the IUCr (Reference: MU1135). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1994). **C50**, 1878–1880

Allomorphs of a Complex of [Mo(O)$_2$(dmso)] with a Derivative of SADH

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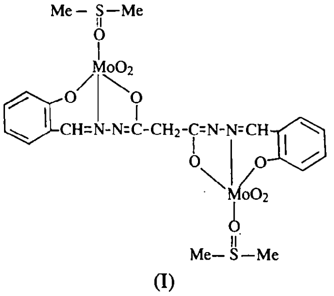
(*Received* 21 *October* 1993; *accepted* 13 *April* 1994)

Abstract

In the title complex, [μ-2,2′-(4,6-dihydroxy-2,3,7,8-tetraaza-1,3,6,8-nonatetraene-1,9-diyl)diphenolato(4−)-N^2,O,O^4:N^8,O',O^6]bis[(dimethyl sulfoxide)dioxomolybdenum], [{Mo(O)$_2$(C$_2$H$_6$OS)}$_2$(μ-C$_{17}$H$_{12}$N$_4$O$_4$)], (I), a binuclear *cis*-dioxomolybdenum(VI) complex of salicylaldehyde malonyl dihydrazone, the interatomic distances Mo(1)···Mo(2) 7.370 (2) and S(1)···S(2) 5.097 (3) Å are different from those of 6.370 (6) and 9.636 (3) Å, respectively, found in the allomorphic complex (II) [Wang, Zhang & Liu (1993). Submitted to *Polyhedron*]. By comparing some of the bond lengths and torsion angles in (I) and (II) it is shown that the two complexes are allomorphs.

Comment

Molybdenum chemistry has aroused considerable interest in recent years in view of its importance in catalytic, materials and biochemical science (Bustos *et al.*, 1990). Here we report the synthesis and crystal structure of a binuclear molybdenum–Schiff base complex, (I).



(I)

The structure of (I), shown in Fig. 1, may be compared with that of the complex (II) (Wang, Zhang & Liu, 1993), which crystallizes in space group *P*2/*c* with $a = 9.856$ (2), $b = 7.646$ (1), $c = 18.373$ (2) Å, $\beta = 95.60$ (1)°, $V = 1377.95$ Å^3 and $Z = 2$. The difference between complexes (I) and (II) is only a difference of conformation. As shown in Fig. 1, complex (I) has a *trans* conformation in which the two Mo atoms lie on different sides of the bridging ligand. Complex (II) has a *cis* conformation, in which the two Mo atoms lie on the same side of the ligand. Some corresponding interatomic distances in (I) and (II) are listed in Table 3.

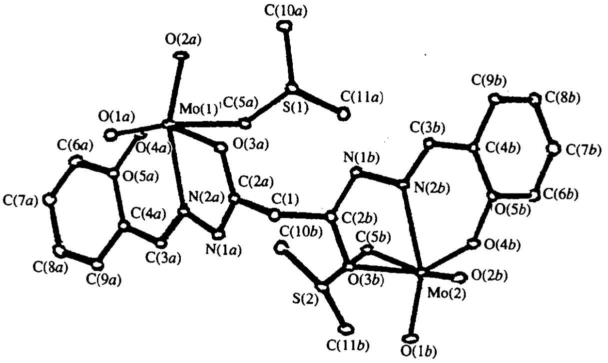


Fig. 1. The molecular structure of (I), showing the atomic labelling scheme.

The average length of the four Mo—O terminal bonds, 1.699 (4) Å, and the average of the O═Mo═O bond angles, 104.6 (8)°, in (I) are comparable to other values reported for the MoO$_2^{2+}$ group (Bustos *et al.*, 1990; Zhai, Xu & Wang, 1992). The N(1*a*)—C(2*a*) [1.267 (8) Å], N(1*b*)—C(2*b*) [1.275 (8) Å], N(2*a*)—C(3*a*) [1.275 (7) Å] and N(2*b*—C(3*b*) [1.281 (7) Å] distances indicate that these correspond to double bonds (Bustos *et al.*, 1990). The Mo(1)—O(5*a*) [2.341 (4) Å]

Acta Crystallographica Section C
ISSN 0108-2701 ©1994