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Structure of Tris(2-mercaptopyridine 1-oxido)cobalt(III) Acetonitrile Solvate

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Abstract. $[Co(C_{15}H_{12}N_3O_3S_3)].C_2H_3N$, $M_r = 478{\cdot}46$, triclinic, $P\bar{1}$, $a = 9{\cdot}332\ (2)$, $b = 12{\cdot}726\ (3)$, $c = 8{\cdot}721\ (2)$ Å, $\alpha = 94{\cdot}01\ (2)$, $\beta = 94{\cdot}44\ (2)$, $\gamma = 75{\cdot}94\ (2)°$, $V = 1000{\cdot}2$ Å^3, $Z = 2$, $D_x = 1{\cdot}59$ g cm^{-3}, Mo $K\alpha$ (graphite monochromated), $\lambda = 0{\cdot}71069$ Å, $\mu = 11{\cdot}8$ cm^{-1}, $F(000) = 488$, $T = 296$ K, final $R = 0{\cdot}046$ for 3731 reflections with $I > 3\sigma(I)$. The three 2-mercaptopyridine 1-oxido ligands with an average bite angle of 88·0° chelate to the cobalt atom *via* oxygen and sulfur atoms forming a distorted octahedral coordination unit CoO_3S_3. The average bond distance of Co—S is 2·194 and that of Co—O is 1·933 Å.

Experimental. Crystals of the title compound $Co(mpo)_3.CH_3CN$ are obtained from the anaerobic reaction of $CoCl_2.6H_2O$ with dtpo and CH_3ONa in a 1:2:2 ratio in mixed solvent CH_3OH/CH_3CN (1:1) at room temperature. Large quantities (64% yield) of rectangular crystals up to 0·1 cm in length are obtained as an MeCN solvate. Density not measured. Rectangular fragment: 0·80 × 0·60 × 0·50 mm. MSC/Rigaku AFC5R diffractometer, Mo $K\alpha$ radiation with graphite monochromator ($\lambda = 0{\cdot}71069$ Å). No systematic absences. $\omega/2\theta$ mode, 2θ range: 2–53·9° ($0 \le h \le 11$, $-15 \le k \le 15$, $-10 \le l \le 10$). 4183 unique reflections measured of which 3731 with $F \ge 3\sigma(F)$. Intensity data corrected for the fluctuation of the monitored reflections, Lorentz–polarization factors and empirical absorption, no extinction correction was made. Maximum and minimum transmission factors were 1·319 and 0·898, respectively. Program *DIFABS* used. The structure was solved by direct methods and refined by the full-matrix least-squares method for 238 parameters based on F. Hydrogen atoms not included. Function minimized was $\sum w(|F_o| - |F_c|)^2$, w defined as 1·0 for all observed reflections. $R = 0{\cdot}046$, $wR = 0{\cdot}048$, $S = 0{\cdot}95$, $(\Delta/\sigma)_{max} = 0{\cdot}50$, $\Delta\rho_{max} = 0{\cdot}67$, $\Delta\rho_{min} = -0{\cdot}44$ e Å^{-3}. Scattering factors and f', f'' values were taken from Cromer & Waber (1974). Calculations carried out on a VAX 11/785 computer with the *SDP* program package (Frenz, 1978). Table 1

Table 1. *Positional parameters and their e.s.d.'s for* $[Co(mpo)_3].MeCN$

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2\beta(1,1) + b^2\beta(2,2) + c^2\beta(3,3) + ab(\cos\gamma)\beta(1,2) + ac(\cos\beta)\beta(1,3) + bc(\cos\alpha)\beta(2,3)]$.

	x	y	z	B(Å^2)
Co	0·27625 (6)	0·23505 (4)	0·12270 (6)	3·35 (1)
S(1)	0·4149 (1)	0·27465 (8)	0·3235 (1)	4·24 (2)
S(2)	0·1625 (1)	0·40578 (9)	0·0997 (1)	3·36 (2)
S(3)	0·1092 (1)	0·21413 (9)	0·2737 (1)	4·17 (2)
O(1)	0·3888 (3)	0·0865 (2)	0·1301 (3)	3·84 (6)
O(2)	0·4104 (3)	0·2536 (2)	−0·0258 (3)	3·90 (6)
O(3)	0·1604 (3)	0·1854 (2)	−0·0487 (3)	3·79 (6)
N(1)	0·4942 (3)	0·0653 (2)	0·2461 (3)	3·38 (6)
N(2)	0·3872 (4)	0·3544 (3)	−0·0782 (4)	3·85 (7)
N(3)	0·0332 (3)	0·1638 (2)	−0·0144 (3)	3·32 (6)
C(11)	0·5201 (4)	0·1452 (3)	0·3466 (4)	3·52 (8)
C(12)	0·6329 (5)	0·1187 (4)	0·4639 (5)	4·6 (1)
C(13)	0·7129 (6)	0·1113 (4)	0·4734 (5)	5·3 (1)
C(14)	0·6822 (5)	−0·0683 (4)	0·3649 (5)	5·2 (1)
C(15)	0·5729 (5)	−0·0410 (3)	0·2506 (5)	4·18 (9)
C(21)	0·2755 (4)	0·4344 (3)	−0·0291 (5)	3·93 (8)
C(22)	0·2552 (6)	0·5373 (4)	−0·0892 (6)	5·6 (1)
C(23)	0·3501 (6)	0·5527 (4)	−0·1951 (7)	6·9 (1)
C(24)	0·4644 (6)	0·4679 (4)	−0·2434 (7)	6·5 (1)
C(25)	0·4841 (5)	0·3664 (4)	−0·1823 (5)	5·2 (1)
C(31)	−0·0085 (4)	0·1758 (3)	0·1314 (4)	3·47 (8)
C(32)	−0·1449 (5)	0·1542 (4)	0·1588 (5)	4·59 (9)
C(33)	−0·2301 (5)	0·1178 (4)	0·0398 (6)	5·4 (1)
C(34)	−0·1801 (5)	0·1038 (4)	−0·1091 (6)	5·1 (1)
C(35)	−0·0482 (5)	0·1273 (3)	−0·1348 (5)	4·21 (9)
N(4)	0·1937 (8)	−0·3227 (5)	−0·5961 (8)	10·9 (2)*
C(41)	0·0857 (7)	−0·3481 (5)	−0·6149 (7)	7·7 (1)*
C(42)	−0·0519 (8)	−0·3796 (5)	−0·6459 (8)	8·0 (2)*

* Atoms were refined isotropically.

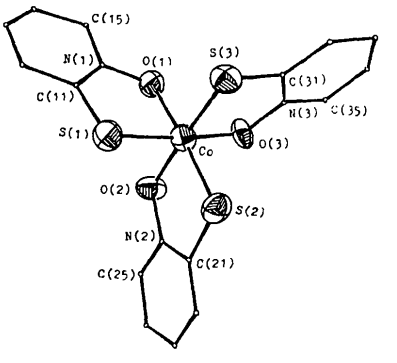

Fig. 1. Molecular structure and atomic labelling scheme for $Co(mpo)_3.CH_3CN$.

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Table 2. *Selected bond distances* (Å) *and bond angles* (°) *of the title compound with e.s.d.'s in parentheses*

Co—S(1)	2·199 (1)	S(1)—C(11)	1·717 (4)
Co—S(2)	2·190 (1)	S(2)—C(21)	1·713 (5)
Co—S(3)	2·192 (1)	S(3)—C(31)	1·718 (4)
Co—O(1)	1·929 (3)	O(1)—N(1)	1·349 (5)
Co—O(2)	1·933 (3)	O(2)—N(2)	1·353 (5)
Co—O(3)	1·937 (3)	O(3)—N(3)	1·341 (4)
S(1)—Co—S(2)	92·04 (5)	S(3)—Co—O(2)	174·8 (1)
S(1)—Co—S(3)	90·66 (5)	S(3)—Co—O(3)	87·92 (9)
S(1)—Co—O(1)	88·20 (9)	O(1)—Co—O(2)	87·0 (1)
S(1)—Co—O(2)	94·3 (1)	O(1)—Co—O(3)	86·5 (1)
S(1)—Co—O(3)	174·4 (1)	O(2)—Co—O(3)	87·2 (1)
S(2)—Co—S(3)	90·74 (5)	Co—S(1)—C(11)	96·6 (1)
S(2)—Co—O(1)	174·8 (1)	Co—S(2)—C(21)	97·0 (1)
S(2)—Co—O(2)	87·88 (9)	Co—S(3)—C(31)	96·8 (1)
S(2)—Co—O(3)	93·4 (1)	Co—O(1)—N(1)	115·9 (2)
S(3)—Co—O(1)	94·42 (9)	Co—O(2)—N(2)	116·0 (2)
		Co—O(3)—N(3)	116·0 (2)

contains a listing of atomic positional parameters and their e.s.d.'s, and Table 2 selected interatomic distances and bond angles. Fig. 1 shows the *ORTEP* drawing (Johnson, 1976) of the molecule.*

Related literature. The title compound was synthesized as part of our investigation of transition-metal complexes with bidentate sulfur:oxygen ligands (Kang, Weng, Liu, Wu, Huang, Lu, Cai, Chen & Lu, 1990; Kang, Weng, Wu, Wang, Guo, Huang, Huang & Liu, 1988; Weng, Huang & Kang, 1989), whose precursor Co^{II} was found to be oxidized to Co^{III} compared to that reported (Robinson, 1964). Its structure is similar to that of $Co(mtb)_3$ (mtb = *N*-methylthiobenzohydroxamate; Freyberg, Abu-Dari & Raymond, 1979). The arrangement of the chelate ligands is such that all three sulfur atoms are *cis* to each other, giving a facial complex. Owing to the bite-distance limit (2·874 Å) of the ligands, the coordination about the cobalt atom is distorted from a regular octahedron with bite angles averaged to 88·0°.

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* Tables of anisotropic thermal parameters, structure factors and a complete list of bond distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54378 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure Determination of a Copper–Histamine Croconate Complex $[Cu(C_5O_5)(C_5H_9N_3)(OH_2)].H_2O$

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Abstract. Aquacroconatohistaminecopper(II) monohydrate, $C_{10}H_{11}CuN_3O_6.H_2O$, $M_r = 350·8$, monoclinic, $P2_1/n$, $a = 7·418$ (1), $b = 17·070$ (2), $c = 10·851$ (1) Å, $\beta = 108·48$ (1)°, $V = 1303·2$ (6) Å^3, $Z = 4$, $D_x = 1·788$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 1·72$ mm^{-1}, $F(000) = 716$, $T = 293$ K, full-matrix least-squares refinement based on 1751 reflections led to R and wR values of 0·031 and 0·032 respectively.

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