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Tris(2-sulfidopyridine *N*-oxide-$\kappa^2 O,S$)cobalt(III)

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Key indicators

Single-crystal X-ray study
T = 296 K
Mean σ(C–C) = 0.007 Å
R factor = 0.040
wR factor = 0.059
Data-to-parameter ratio = 13.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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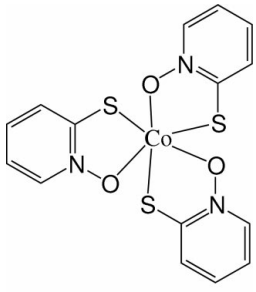
The title compound, [tri(2-sulfidopyridine *N*-oxide-*O,S*)-cobalt(III)], [$Co(C_5H_4NSO)_3$], was synthesized and its structure characterized by X-ray crystallographic techniques. Three mpo^- (Hmpo = 2-mercaptopyridine-*N*-oxide) ligands chelate the Co atom, forming a distorted octahedral O_3S_3 coordination environment around Co. The asymmetric unit contains two molecules of $Co(C_5H_4NOS)_3$.

Comment

Metal thiolate complexes are important analogues of metal enzymes and are known as biological electron-transfer mediators (Dance, 1986). We are interested in investigating cobalt complexes with thiolate because of the multifunctional nature of thiolates and their intriguing structural variations, and also the electron-transfer properties of cobalt complexes (Kang *et al.*, 1996; Chen *et al.*, 1998). The single-crystal structures of numerous cobalt complexes with Hmpo (Hmpo = 2-mercaptopyridine–*N*-oxide) have been reported (Hu *et al.*, 1991; Kang *et al.*, 1993; Xu *et al.*, 1995); the title compound, (I), is described here.



(I)

The title compound contains a distorted octahedral Co(III) ion, chelated by three bidentate mpo^- ligands through their S and O atoms. Its structure is similar to those reported earlier (Hu *et al.*, 1991; Xu *et al.*, 1995). The average Co—S and Co—O bond distances and the average O—Co—S bite angle (2.199 and 1.947 Å, and 87.85°, respectively) agree well with those of $Co(mpo)_3{\cdot}CH_3CN$ (2.194 and 1.933 Å, and 88.00°, respectively; Hu *et al.*, 1991).

The X-ray structure shows that the asymmetric unit contains two molecules of $Co(mpo)_3$. Between the two molecules, there is a π–π stack of pyridine rings; the distance between the centres of the rings is 3.577 Å. The three dimensional arrangement of molecules within the crystal lattice is determined by the interactions between the stacks.

Experimental

To a methanol solution (20 ml) containing $CoCl_2(PPh_3)_2$ (0.654 g, 1 mmol) was added Na(mpo) (0.447 g, 3 mmol), with stirring, at room

temperature. The resulting dark solution, after stirring for 2 h, was reduced to dryness *in vacuo* and then extracted with 20 ml CH_2Cl_2. The resulting solution evaporated at room temperature to give black crystalline blocks of the title compound, in 5% yield. Analysis calculated for $C_{15}H_{12}N_3CoO_3S_3$: C, 41.19; H, 2.77; N, 9.61; S, 21.99%. Found: C, 41.45; H, 2.92; N, 9.40; S, 22.42%.

Crystal data

$[Co(C_5H_4NSO)_3]$	D_x = 1.656 Mg m^{-3}
M_r = 437.39	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 28 reflections
a = 9.480 (2) Å	θ = 2.7–13.7°
b = 26.303 (5) Å	μ = 1.36 mm^{-1}
c = 14.437 (4) Å	T = 296 (2) K
β = 102.90 (2)°	Block, black
V = 3509.0 (14) Å^3	0.50 × 0.30 × 0.10 mm
Z = 8	

Data collection

Siemens *P*4 diffractometer	R_{int} = 0.028
ω scans	θ_{max} = 25.0°
Absorption correction: empirical (North *et al.*, 1968)	$h = 0 \rightarrow 11$
T_{min} = 0.800, T_{max} = 0.990	$k = -1 \rightarrow 31$
7223 measured reflections	$l = -17 \rightarrow 16$
6188 independent reflections	3 standard reflections
3363 reflections with I>2sigma(I)	every 97 reflections
	intensity decay: 1.9%

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.040	$w = 1/[\sigma^2(F_o^2) + (0.0130P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.059	$(\Delta/\sigma)_{max}$ = 0.001
S = 0.80	$\Delta\rho_{max}$ = 0.29 e Å^{-3}
6188 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
451 parameters	

H atoms were placed in calculated positions and refined using a riding model.

Data collection: *XSCANS* (Siemens, 1996); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1990); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1994); software used to prepare material for publication: *SHELXTL*.

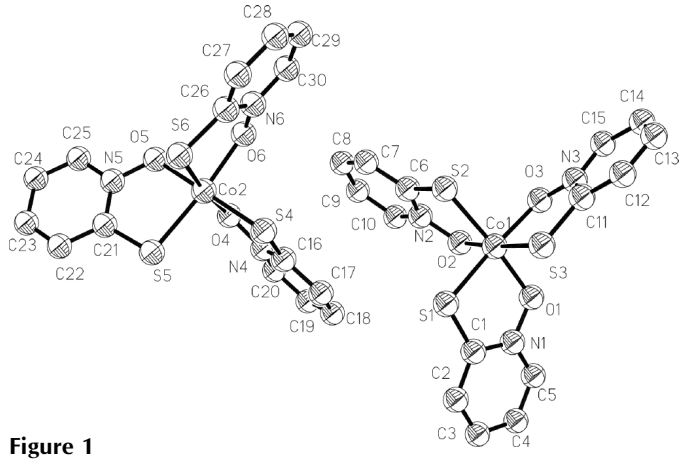


Figure 1
A view of the two independent molecules of the title compound, showing 50% probability displacement ellipsoids. H atoms are omitted for clarity.

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References

Chen, Z. N., Zhang, H. X., Kang, B. S. & Sun, J. (1998). *Synth. React. Inorg. Met. Org. Chem.* **28**, 245–26.
Dance, I. G. (1986). *Polyhedron*, **5**, 1037.
Hu, Y. H., Weng, L. H., Huang, L. R., Chen, X. T., Wu, D. X. & Kang, B. S. (1991). *Acta Cryst.* C**47**, 2655–2656.
Kang, B. S., Xu, Y. J., Peng, J. H., Wu, D. X., Chen, X. T., Hu, Y. H., Hong, M. C. & Lu, J. X. (1993). *Polyhedron*, **12**, 871–878.
Kang, B. S., Chen, Z. N., Zhou, Z. Y., Liu, H. Q., Gao, H. R., Wu, B. M., Mak, T. C., Cai, Y. B., Xu, Y. J. & Xu, Z. T. (1996). *J. Cluster Sci.* **7**, 317–329.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Sheldrick, G. M. (1990). *Acta Cryst.* A**46**, 467–473.
Sheldrick, G. M. (1997). *SHELXL*97. University of Göttingen, Germany.
Siemens (1994). *SHELXTL*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Siemens (1996). *XSCANS*. Version 2.2. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
Xu, Y. J., Kang, B. S., Chen, X. T., & Huang, L. R. (1995). *Acta Cryst.* C**51**, 370–374.