

Hexakis(dimethyl sulfoxide- κ O)-cobalt(III) trinitrate

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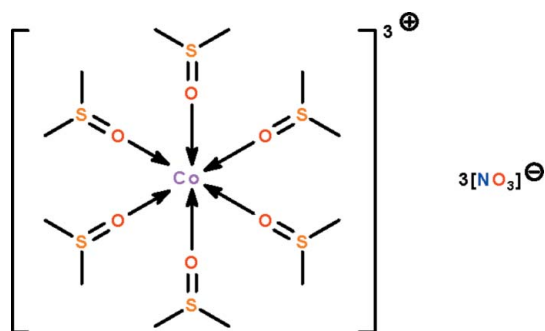
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Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{S}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.046; wR factor = 0.136; data-to-parameter ratio = 17.3.

The metal atom of the title salt, $[\text{Co}(\text{C}_2\text{H}_6\text{OS})_6](\text{NO}_3)_3$, is coordinated by six dimethyl sulfoxide molecules in an octahedral geometry. The metal atom lies on a special position of $\bar{3}$ site symmetry. One of the nitrate ions lies on a special position of 3 site symmetry and the other independent ion is disordered about a special position of $\bar{3}$ site symmetry.

Related literature

For the isostructural chromium(III) and iron(III) analogs, see: Öhrström & Svensson (2000); Tzou *et al.* (1995).



Experimental

Crystal data

$[\text{Co}(\text{C}_2\text{H}_6\text{OS})_6](\text{NO}_3)_3$
 $M_r = 713.73$
 Trigonal, $R\bar{3}$
 $a = 11.526$ (3) Å
 $c = 19.998$ (5) Å
 $V = 2300.8$ (10) Å³

$Z = 3$
 Mo $K\alpha$ radiation
 $\mu = 1.03$ mm⁻¹
 $T = 298$ K
 $0.49 \times 0.41 \times 0.38$ mm

Data collection

Bruker SMART 1000 area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.632$, $T_{\max} = 0.695$

3840 measured reflections
 1158 independent reflections
 879 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.062$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.136$
 $S = 1.07$
 1158 reflections
 67 parameters

7 restraints
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.59$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.62$ e Å⁻³

Data collection: *SMART* (Bruker, 1996); cell refinement: *SAINTE* (Bruker, 1996); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *pubCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CI2949).

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supplementary materials

Acta Cryst. (2010). E66, m21 [doi:10.1107/S1600536809051423]

Hexakis(dimethyl sulfoxide- κ O)cobalt(III) trinitrate

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Experimental

To a solution of cobalt(II) nitrate hexahydrate (0.10 g, 0.4 mmol) in methanol (10 ml) was added a solution of 1,2-disali-cyloxyhydrazine (0.05 g, 0.2 mmol) in DMSO (10 ml). The red solution was allowed to stand for one week, whereupon red block-shaped crystals were obtained in 60% yield (m.p. > 573 K). CH&N elemental analysis for $C_{12}H_{36}CoN_3O_{15}S_6$: Calculated: C 20.19, H 5.08, N 5.89%; found: C 20.10, H 5.17, N 5.81%.

Refinement

The nitrate anion lying on the Wyckoff $3b$ position is disordered. This was refined off the special position as a planar four-atom species, with N–O distances restrained to 1.24 (1) Å and O···O distances restrained to 2.15 (1) Å. The isotropic displacement parameters of the three O atoms were restrained to be identical; the O and N atoms were refined isotropically. The methyl H-atoms were placed in idealized positions and treated as riding on their parent atoms with a C–H distance of 0.96 Å [$U_{iso}(H) = 1.5U_{eq}(C)$].

Figures

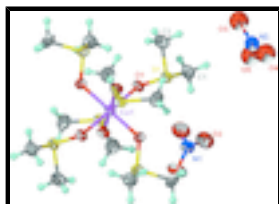


Fig. 1. Displacement ellipsoid plot (Barbour, 2001) of hexakis(dimethylsulfoxide)cobalt(III) trinitrate at the 50% probability level.

Hexakis(dimethyl sulfoxide- κ O)cobalt(III) trinitrate

Crystal data

$[Co(C_2H_6OS)_6](NO_3)_3$

$M_r = 713.73$

Trigonal, $R\bar{3}$

Hall symbol: $-R\ 3$

$a = 11.526$ (3) Å

$c = 19.998$ (5) Å

$V = 2300.8$ (10) Å³

$Z = 3$

$F(000) = 1116$

$D_x = 1.545$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1446 reflections

$\theta = 2.3$ – 27.2°

$\mu = 1.03$ mm⁻¹

$T = 298$ K

Block, red

$0.49 \times 0.41 \times 0.38$ mm

supplementary materials

Data collection

Bruker SMART 1000 area-detector diffractometer	1158 independent reflections
Radiation source: fine-focus sealed tube graphite	879 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{\text{int}} = 0.062$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$\theta_{\text{max}} = 27.4^\circ$, $\theta_{\text{min}} = 2.3^\circ$
$T_{\text{min}} = 0.632$, $T_{\text{max}} = 0.695$	$h = -14 \rightarrow 5$
3840 measured reflections	$k = -11 \rightarrow 14$
	$l = -25 \rightarrow 25$

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.136$	H-atom parameters constrained
$S = 1.07$	$w = 1/[\sigma^2(F_o^2) + (0.0617P)^2 + 5.5038P]$
1158 reflections	where $P = (F_o^2 + 2F_c^2)/3$
67 parameters	$(\Delta/\sigma)_{\text{max}} = 0.001$
7 restraints	$\Delta\rho_{\text{max}} = 0.59 \text{ e } \text{\AA}^{-3}$
	$\Delta\rho_{\text{min}} = -0.62 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Co1	0.6667	0.3333	0.3333	0.0336 (3)	
S1	0.48481 (8)	0.41879 (8)	0.25413 (4)	0.0388 (3)	
C1	0.3940 (4)	0.3538 (5)	0.17922 (18)	0.0567 (10)	
H1A	0.4494	0.3427	0.1470	0.085*	
H1B	0.3673	0.4147	0.1620	0.085*	
H1C	0.3158	0.2686	0.1880	0.085*	
C2	0.3544 (4)	0.4062 (5)	0.3061 (2)	0.0591 (10)	
H2A	0.2805	0.3160	0.3051	0.089*	
H2B	0.3254	0.4662	0.2903	0.089*	
H2C	0.3867	0.4296	0.3511	0.089*	
O1	0.5100 (2)	0.3062 (2)	0.27796 (11)	0.0379 (5)	

O2	0.6649 (4)	0.4393 (3)	0.0814 (2)	0.0838 (11)	
N1	0.6667	0.3333	0.0819 (3)	0.0516 (13)	
N2	0.331 (2)	0.657 (2)	0.1524 (6)	0.060 (4)*	0.1667
O3	0.246 (2)	0.612 (3)	0.1984 (10)	0.104 (5)*	0.1667
O4	0.407 (3)	0.7797 (19)	0.1464 (11)	0.104 (5)*	0.1667
O5	0.338 (2)	0.578 (2)	0.1132 (11)	0.104 (5)*	0.1667

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0312 (4)	0.0312 (4)	0.0385 (5)	0.01558 (19)	0.000	0.000
S1	0.0334 (5)	0.0343 (5)	0.0474 (5)	0.0159 (4)	-0.0051 (3)	0.0031 (3)
C1	0.057 (2)	0.077 (3)	0.0426 (19)	0.038 (2)	-0.0106 (17)	0.0001 (18)
C2	0.063 (3)	0.069 (3)	0.062 (2)	0.046 (2)	0.000 (2)	-0.009 (2)
O1	0.0327 (12)	0.0352 (12)	0.0462 (12)	0.0173 (10)	-0.0078 (9)	-0.0010 (10)
O2	0.078 (2)	0.0523 (19)	0.130 (3)	0.0389 (18)	0.007 (2)	0.0059 (19)
N1	0.047 (2)	0.047 (2)	0.061 (3)	0.0234 (10)	0.000	0.000

Geometric parameters (\AA , $^\circ$)

Co1—O1 ⁱ	2.005 (2)	C1—H1C	0.96
Co1—O1 ⁱⁱ	2.005 (2)	C2—H2A	0.96
Co1—O1 ⁱⁱⁱ	2.005 (2)	C2—H2B	0.96
Co1—O1 ^{iv}	2.005 (2)	C2—H2C	0.96
Co1—O1	2.005 (2)	O2—N1	1.232 (3)
Co1—O1 ^v	2.005 (2)	N1—O2 ^{iv}	1.232 (3)
S1—O1	1.540 (2)	N1—O2 ⁱⁱⁱ	1.232 (3)
S1—C1	1.765 (4)	N2—O4	1.238 (9)
S1—C2	1.773 (4)	N2—O5	1.238 (9)
C1—H1A	0.96	N2—O3	1.253 (9)
C1—H1B	0.96		
O1 ⁱ —Co1—O1 ⁱⁱ	92.45 (9)	S1—C1—H1B	109.5
O1 ⁱ —Co1—O1 ⁱⁱⁱ	180.0	H1A—C1—H1B	109.5
O1 ⁱⁱ —Co1—O1 ⁱⁱⁱ	87.55 (9)	S1—C1—H1C	109.5
O1 ⁱ —Co1—O1 ^{iv}	87.56 (9)	H1A—C1—H1C	109.5
O1 ⁱⁱ —Co1—O1 ^{iv}	180.0	H1B—C1—H1C	109.5
O1 ⁱⁱⁱ —Co1—O1 ^{iv}	92.44 (9)	S1—C2—H2A	109.5
O1 ⁱ —Co1—O1	87.56 (9)	S1—C2—H2B	109.5
O1 ⁱⁱ —Co1—O1	87.56 (9)	H2A—C2—H2B	109.5
O1 ⁱⁱⁱ —Co1—O1	92.44 (9)	S1—C2—H2C	109.5
O1 ^{iv} —Co1—O1	92.44 (9)	H2A—C2—H2C	109.5
O1 ⁱ —Co1—O1 ^v	92.45 (9)	H2B—C2—H2C	109.5
O1 ⁱⁱ —Co1—O1 ^v	92.44 (9)	S1—O1—Co1	125.03 (13)
O1 ⁱⁱⁱ —Co1—O1 ^v	87.55 (9)	O2 ^{iv} —N1—O2 ⁱⁱⁱ	119.996 (15)
O1 ^{iv} —Co1—O1 ^v	87.56 (9)	O2 ^{iv} —N1—O2	119.995 (10)

supplementary materials

O1—Co1—O1 ^v	179.998 (1)	O2 ⁱⁱⁱ —N1—O2	119.996 (15)
O1—S1—C1	103.10 (17)	O4—N2—O5	120.6 (10)
O1—S1—C2	104.99 (17)	O4—N2—O3	120.3 (10)
C1—S1—C2	99.5 (2)	O5—N2—O3	119.0 (10)
S1—C1—H1A	109.5		
C1—S1—O1—Co1	151.6 (2)	O1 ⁱⁱ —Co1—O1—S1	135.7 (2)
C2—S1—O1—Co1	-104.7 (2)	O1 ⁱⁱⁱ —Co1—O1—S1	-136.90 (12)
O1 ⁱ —Co1—O1—S1	43.10 (12)	O1 ^{iv} —Co1—O1—S1	-44.3 (2)

Symmetry codes: (i) $y+1/3, -x+y+2/3, -z+2/3$; (ii) $x-y+1/3, x-1/3, -z+2/3$; (iii) $-y+1, x-y, z$; (iv) $-x+y+1, -x+1, z$; (v) $-x+4/3, -y+2/3, -z+2/3$.

Fig. 1

