The structure was solved by Patterson methods. The H atom was located from a difference map and refined isotropically. Non-H atoms were treated anisotropically by full-matrix least-squares techniques using all data except for four reflections (100, 110, $\bar{1}02$, 102) which were suspected of strong extinction.

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1989). Cell refinement: *CAD*-4 *Software*. Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1971) in the *NRCVAX* package (Gabe, Le Page, Charland, Lee & White, 1989).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1080). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tris(*O*,*O*′-dicyclohexyl dithiophosphato-*S*,*S*′)chromium(III)

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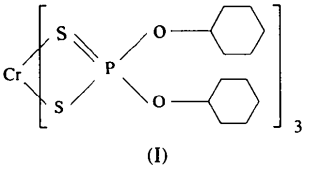
Abstract

The title complex, $[Cr\{S_2P(OC_6H_{11})_2\}_3]$, contains a CrS_6 distorted octahedral core, in which the Cr—S bond lengths range from 2.423 (2) to 2.439 (2) Å and the S—Cr—S bidentate angles range from 81.39 (6) to 81.66 (7)°.

Comment

Chromium complexes such as those with dialkyl dithiophosphates and alkylphosphonic acid monoalkyl ester were found to have practical applications in the petroleum and plastics industries (Xiong & Dong, 1994*a*,*b*; Mikhailov, Kokhanov, Kazaryan, Matveeva & Kozodoi, 1972). We report here the crystal structure of the title complex, (I), which is very similar to that of $[Cr\{S_2P(OC_2H_5)_2\}_3]$ (Schousboe-Jensen & Hazell, 1972).



(I)

The crystal structure of $[Cr\{S_2P(OC_6H_{11})_2\}_3]$ is composed of four neutral molecules per unit cell in the $P2_1/n$ space group. The central Cr atom has distorted octahedral coordination with six S atoms from three dithiophosphate bidentate ligands. The Cr—S bond lengths [2.423 (2)–2.439 (2) Å] and S—Cr—S bidentate bond angles [81.39 (6)–81.66 (7)°] do not differ significantly from those of $[Cr\{Sr_2P(OC_2H_5)_2\}_3]$ [2.421 (3)–2.430 (3) Å and 82.5 (1)°, respectively (Schousboe-Jensen & Hazell, 1972)].

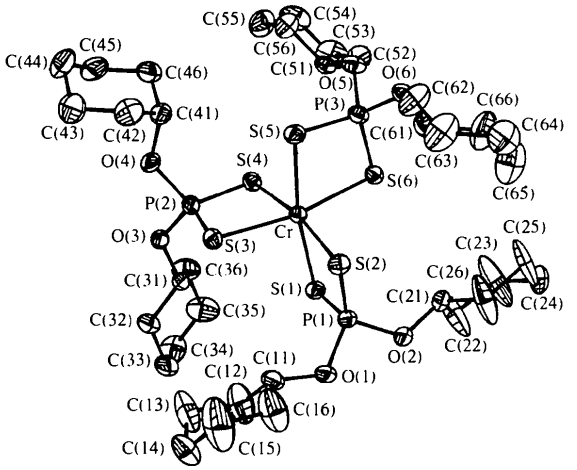


Fig 1. Molecular structure showing 30% probability displacement ellipsoids. H atoms are omitted for clarity.

Experimental

The title complex was obtained by the reaction of $NaS_2P(OC_6H_{11})_2$ and $CrCl_3$ (molar ratio 3:1) in ethanol solution for 0.5 h at room temperature. Recrystallization was from $EtOH/CHCl_3$.

Crystal data

$[Cr(C_{12}H_{22}O_2PS_2)_3]$
$M_r = 932.19$
Monoclinic
$P2_1/n$
$a = 16.578\ (3)$ Å
$b = 10.193\ (8)$ Å
$c = 31.574\ (4)$ Å
$\beta = 100.92\ (1)°$
$V = 5239\ (4)$ Å^3
$Z = 4$
$D_x = 1.18$ Mg m^{-3}
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 25 reflections
$\theta = 13.95$–$14.80°$
$\mu = 0.567$ mm^{-1}
$T = 296$ K
Rod
0.80 × 0.25 × 0.20 mm
Purple

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: ψ scan (Molecular Structure Corporation, 1985)
$T_{min} = 0.948$, $T_{max} = 1.000$
10 125 measured reflections
9766 independent reflections
5626 observed reflections $[I > 3\sigma(I)]$
$R_{int} = 0.019$
$\theta_{max} = 25°$
$h = 0 \rightarrow 19$
$k = 0 \rightarrow 12$
$l = -37 \rightarrow 37$
3 standard reflections monitored every 250 reflections
intensity decay: 5.2%

Refinement

Refinement on F
$R = 0.061$
$wR = 0.072$
$S = 1.49$
5626 reflections
469 parameters
H-atom parameters not refined
$w = 1/\sigma^2(F)$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.37$ e Å^{-3}
$\Delta\rho_{min} = -0.41$ e Å^{-3}
Extinction correction: none
Atomic scattering factors from Cromer & Waber (1974)

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

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	B_{eq}
Cr	0.53839 (4)	0.15881 (8)	0.84288 (2)	2.77 (3)
S(1)	0.58075 (8)	0.3137 (1)	0.79299 (4)	3.72 (6)
S(2)	0.57232 (8)	0.0022 (1)	0.79069 (4)	3.75 (6)
S(3)	0.39313 (8)	0.1547 (2)	0.81028 (5)	3.84 (6)
S(4)	0.49203 (8)	0.3368 (1)	0.88366 (4)	3.56 (6)
S(5)	0.51823 (8)	−0.0142 (1)	0.89246 (4)	3.80 (6)
S(6)	0.67497 (8)	0.1604 (1)	0.88819 (4)	3.63 (6)
P(1)	0.60347 (8)	0.1574 (2)	0.75905 (4)	3.52 (6)
P(2)	0.37940 (8)	0.3005 (1)	0.85050 (5)	3.40 (6)
P(3)	0.63290 (9)	0.0219 (1)	0.92313 (4)	3.64 (6)
O(1)	0.5591 (2)	0.1621 (4)	0.7101 (1)	5.0 (2)
O(2)	0.6935 (2)	0.1534 (4)	0.7506 (1)	4.4 (2)
O(3)	0.3353 (2)	0.4254 (4)	0.8275 (1)	4.1 (2)
O(4)	0.3139 (2)	0.2722 (4)	0.8795 (1)	4.4 (2)
O(5)	0.6407 (2)	0.0627 (4)	0.9721 (1)	4.6 (2)
O(6)	0.6873 (2)	−0.1044 (4)	0.9314 (1)	4.6 (2)
C(11)	0.4699 (4)	0.1479 (7)	0.6979 (2)	4.7 (3)
C(12)	0.4320 (5)	0.2731 (9)	0.6830 (4)	9.4 (5)
C(13)	0.3405 (5)	0.254 (1)	0.6690 (5)	11.2 (7)
C(14)	0.3235 (6)	0.156 (2)	0.6326 (3)	11.6 (7)
C(15)	0.3626 (7)	0.030 (1)	0.6495 (5)	14.5 (9)
C(16)	0.4509 (5)	0.038 (1)	0.6666 (3)	10.3 (6)
C(21)	0.7645 (3)	0.1522 (6)	0.7858 (2)	3.7 (2)
C(22)	0.8104 (7)	0.270 (1)	0.7860 (6)	17.3 (9)
C(23)	0.8891 (8)	0.265 (1)	0.8207 (7)	21 (1)
C(24)	0.9398 (5)	0.150 (2)	0.8175 (3)	10.9 (7)
C(25)	0.8900 (7)	0.035 (1)	0.8193 (5)	15.4 (8)
C(26)	0.8138 (6)	0.038 (1)	0.7827 (5)	16.1 (8)
C(31)	0.3751 (3)	0.5084 (6)	0.7995 (2)	3.9 (2)
C(32)	0.3161 (4)	0.5283 (7)	0.7585 (2)	5.8 (3)
C(33)	0.3510 (4)	0.624 (1)	0.7296 (2)	7.4 (4)
C(34)	0.3770 (6)	0.751 (1)	0.7533 (4)	9.0 (6)
C(35)	0.4353 (6)	0.7265 (8)	0.7938 (3)	8.3 (5)
C(36)	0.3998 (5)	0.6355 (8)	0.8219 (2)	6.8 (4)
C(41)	0.3256 (4)	0.1635 (6)	0.9118 (2)	4.7 (3)
C(42)	0.2637 (5)	0.0611 (7)	0.8969 (2)	6.4 (4)
C(43)	0.1776 (5)	0.1075 (9)	0.8976 (3)	7.6 (5)
C(44)	0.1708 (5)	0.159 (1)	0.9416 (3)	8.2 (5)
C(45)	0.2301 (5)	0.2650 (9)	0.9545 (2)	7.2 (4)
C(46)	0.3173 (4)	0.2222 (7)	0.9547 (2)	6.0 (3)
C(51)	0.6157 (3)	0.1919 (5)	0.9845 (2)	3.7 (2)
C(52)	0.6896 (4)	0.2613 (8)	1.0063 (2)	6.2 (4)
C(53)	0.6654 (6)	0.3965 (8)	1.0227 (3)	8.2 (5)
C(54)	0.6024 (6)	0.3825 (8)	1.0501 (2)	7.6 (4)
C(55)	0.5303 (5)	0.3074 (8)	1.0289 (3)	7.2 (4)
C(56)	0.5528 (4)	0.1744 (6)	1.0123 (2)	5.5 (3)
C(61)	0.6980 (4)	−0.1915 (6)	0.8966 (2)	4.9 (3)
C(62)	0.6584 (6)	−0.318 (1)	0.9005 (3)	9.4 (6)
C(63)	0.6741 (9)	−0.412 (1)	0.8662 (5)	13.4 (8)
C(64)	0.7647 (9)	−0.429 (1)	0.8681 (4)	11.5 (7)
C(65)	0.8019 (7)	−0.301 (1)	0.8647 (5)	13.5 (9)
C(66)	0.7877 (5)	−0.208 (1)	0.8983 (3)	9.5 (6)

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

Cr—S(1)	2.426 (2)	Cr—S(2)	2.435 (2)
Cr—S(3)	2.433 (2)	Cr—S(4)	2.432 (2)
Cr—S(5)	2.423 (2)	Cr—S(6)	2.439 (2)
S(1)—P(1)	1.996 (2)	S(2)—P(1)	1.990 (2)
S(3)—P(2)	1.996 (2)	S(4)—P(2)	1.995 (2)
S(5)—P(3)	1.998 (2)	S(6)—P(3)	1.997 (2)
P(1)—O(1)	1.583 (4)	P(1)—O(2)	1.566 (4)
P(2)—O(3)	1.575 (4)	P(2)—O(4)	1.573 (4)
P(3)—O(5)	1.583 (4)	P(3)—O(6)	1.565 (4)
O(1)—C(11)	1.463 (7)	O(2)—C(21)	1.458 (6)
O(3)—C(31)	1.468 (6)	O(4)—C(41)	1.496 (6)
O(5)—C(51)	1.456 (6)	O(6)—C(61)	1.450 (7)

S(1)—Cr—S(2)	81.66 (7)	S(3)—Cr—S(4)	81.39 (6)
S(5)—Cr—S(6)	81.50 (6)	S(1)—Cr—S(3)	96.98 (6)
S(2)—Cr—S(6)	95.37 (6)	S(4)—Cr—S(5)	96.01 (7)
S(1)—P(1)—S(2)	105.77 (9)	S(3)—P(2)—S(4)	105.30 (9)
S(5)—P(3)—S(6)	105.22 (9)	O(1)—P(1)—O(2)	96.6 (2)
O(3)—P(2)—O(4)	96.0 (2)	O(5)—P(3)—O(6)	96.6 (2)

Data were collected with *CONTROL* (Molecular Structure Corporation, 1986) software. The structure was solved by direct methods using *MITHRIL* (Gilmore, 1983); the Cr heavy atom was located in an *E* map and the remaining non-H atoms were located using *DIRDIF* (Beurskens, 1984). H atoms were placed in geometrically calculated positions with C—H = 0.95 Å, but were not included in the refinement. The structure was refined by full-matrix least-squares techniques with anisotropic displacement parameters for all non-H atoms. Calculations were performed on a VAX 3100 computer using the *TEXSAN* (Molecular Structure Corporation, 1985) program package.

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KH1031). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A *cis*-Oxo[diphenylhydrazido(2–)]-molybdenum Complex: $(Et_3NH)_2[MoO(NNPh_2)(SC_6H_4CO_2)_2]$

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Abstract

The structure of bis(triethylammonium) (diphenylhydrazido)bis(2-mercaptobenzoato-*O*,*S*)oxomolybdate(VI) consists of two triethylammonium cations and a dianionic molybdenum complex. The structure exhibits the anticipated *cis*-oxo[diphenylhydrazido(2−)] geometry with two dianionic mercaptobenzoate ligands whose S donor atoms are mutually *trans*.

Comment

Transition metal complexes containing the organohydrazido(2−) ligand have been studied extensively because the NN*RR'* ligand (*RR'* = alkyl and/or aryl) is of interest as a potential model of the NNH_2 intermediate detected in the chemical and enzymatic conversion of dinitrogen into ammonia (Henderson, Leigh & Pickett, 1992; Leigh, 1992). As part of our current research (Bustos *et al.*, 1991; Bustos, Manzur, Carrillo, Robert & Gouzerh, 1994), the study of the title compound (1) was undertaken.