

I thank SERC for a research grant towards crystallographic equipment.

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Structure of Bis(tetramethylammonium) Oxopentathiocyanatomolybdate(V)

BY WILLIAM CLEGG

Department of Inorganic Chemistry, The University, Newcastle upon Tyne NE1 7RU, England

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Abstract. $[\text{N}(\text{CH}_3)_4]_2[\text{Mo}(\text{NCS})_5\text{O}]$, $M_r = 550.6$, orthorhombic, $Pnma$, $a = 18.5152(10)$, $b = 14.3794(8)$, $c = 9.3321(5)$ Å, $V = 2484.6$ Å³, $Z = 4$, $D_x = 1.472$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71073$ Å, $\mu = 0.94$ mm⁻¹, $F(000) = 1124$, $T = 298$ K, $R = 0.048$ for 1711 unique reflections with $F > 4\sigma(F)$. The entire anion, except for two mutually *trans* NCS⁻ ligands, lies on a crystallographic mirror plane; the approximate symmetry is $4mm$ (C_{4v}). The NCS⁻ ligands are coordinated to Mo through N. The axial Mo–N bond *trans* to Mo=O is considerably longer than the equatorial ones [2.228 (5) Å compared with 2.050 (6)–2.063 (6) Å; Mo=O = 1.646 (5) Å]. The four equatorial ligands are bent towards the axial NCS⁻ ligand and away from the oxo ligand, both in terms of O–Mo–N angles [in the range 96.4 (1)–98.4 (1)°] and by significant bending at N [in the range 162.3 (6)–169.4 (6)°]. By contrast the Mo–N–CS angle *trans* to O is 179.4 (5)°. The shortest NCS⁻⋯C(cation) distance is 3.43 Å, and shortest interionic NCS⁻⋯NCS contact is 3.97 Å, but these both involve the same NCS ligand (and its mirror-related counterpart) of the anion; in view of this marked difference in the packing surroundings of the various NCS ligands and the consistent degree and direction of their bending, this distortion is probably an intrinsic property of the anion rather than of its lattice environment.

Experimental. Crystal size 0.8 × 0.3 × 0.08 mm, Siemens AED2 diffractometer, cell parameters from 2 θ values of 32 reflections with $20 < 2\theta < 25^\circ$. Intensity measurements in ω/θ scan mode, scan width = $1.02^\circ + \alpha$ -doublet separation, scan time = 14–56 s, $2\theta_{\text{max}} = 50^\circ$, h 0→21, k -17→13, l 0→11, no significant variation for three standard reflections, semi-empirical

absorption correction, transmission 0.524–0.609, extinction negligible. 4146 reflections, 2270 unique ($R_{\text{int}} = 0.027$), 1711 with $F > 4\sigma(F)$. Structure solved by multiresolution direct methods and difference syntheses, blocked-cascade refinement on F , $w = 1/\sigma^2(F)$, anisotropic thermal parameters for non-H atoms, H atoms constrained [C–H = 0.96 Å, H–C–H = 109.5° in rigid methyl groups, $U(\text{H}) = 1.2U_{\text{eq}}(\text{C})$]. 139 parameters, $R = 0.048$, $wR = 0.039$, slope of normal probability plot = 1.69; max. $\Delta/\sigma = 0.002$, mean = 0.001, max. $\Delta\rho = 0.51$, min. = -0.43 e Å⁻³. Scattering factors from *International Tables for X-ray Crystallography* (1974), *SHELXTL* (Sheldrick, 1985) programs. The structure and atom-numbering scheme are shown in Fig. 1. Atomic coordinates are listed in Table 1,* and bond lengths and angles in Table 2.

Related literature. The oxo chemistry of Mo^V is dominated by binuclear species (Cotton & Wilkinson, 1980). Known structures of mononuclear anions are mainly of the type MoOX_3^{2-} ($X^- = \text{halide}$) (e.g. Bino & Cotton, 1979). Bonding of thiocyanate to Mo *via* N has been demonstrated in various oxidation states (Knox & Eriks, 1968; Viossat, Rodier & Khodadad, 1979; Bino, Cotton & Fanwick, 1979); bonding *via* S is unknown.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43613 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

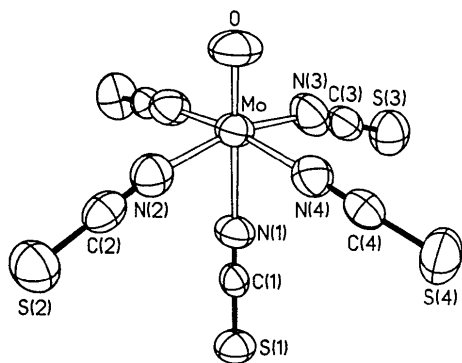


Fig. 1. Structure of the anion, with 50% probability ellipsoids and the labelling of the independent atoms.

Table 1. Atomic coordinates ($\times 10^4$) and equivalent thermal parameters ($\text{\AA}^2 \times 10^4$)

$$U_{eq} = \frac{1}{3}(\text{trace of orthogonalized } U_{ij} \text{ matrix}).$$

	x	y	z	U_{eq}
Mo	5575 (1)	7500	5933 (1)	546 (2)
O	4778 (3)	7500	6715 (5)	837 (20)
N(1)	6658 (3)	7500	4892 (5)	588 (20)
C(1)	7203 (3)	7500	4355 (6)	452 (20)
S(1)	8002 (1)	7500	3607 (2)	680 (7)
N(2)	5234 (3)	7500	3843 (6)	640 (21)
C(2)	5154 (4)	7500	2614 (9)	583 (25)
S(2)	5050 (1)	7500	898 (2)	862 (8)
N(3)	6194 (3)	7500	7771 (6)	781 (24)
C(3)	6681 (4)	7500	8539 (7)	644 (25)
S(3)	7345 (1)	7500	9629 (2)	892 (9)
N(4)	5701 (2)	6082 (2)	5890 (4)	656 (15)
C(4)	5919 (2)	5328 (3)	5968 (5)	528 (15)
S(4)	6204 (1)	4282 (1)	6089 (1)	851 (6)
N(5)	6405 (2)	4865 (2)	1479 (3)	526 (12)
C(51)	6628 (3)	5690 (3)	2323 (5)	965 (25)
C(52)	6446 (4)	5054 (4)	-62 (5)	1182 (31)
C(53)	5656 (3)	4653 (5)	1875 (8)	1516 (40)
C(54)	6845 (4)	4064 (4)	1884 (7)	1642 (43)

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fac-(2,2'-Bipyridyl)tricarbonylchloromanganese(I)

BY ERNST HORN, MICHAEL R. SNOW AND EDWARD R. T. TIEKINK

Jordan Laboratories, Department of Physical and Inorganic Chemistry, University of Adelaide, South Australia 5001, Australia

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Abstract. $[\text{MnCl}(\text{CO})_3(\text{C}_{10}\text{H}_8\text{N}_2)]$, $M_r = 330.6$, triclinic, $P\bar{1}$, $a = 11.040$ (1), $b = 11.491$ (1), $c = 10.9649$ (7) \AA , $\alpha = 94.03$ (1), $\beta = 102.45$ (1), $\gamma = 84.80$ (1) $^\circ$, $V = 1351$ (2) \AA^3 , $Z = 4$, $D_x = 1.627$ Mg m^{-3} , $\lambda(\text{Mo K}\alpha) = 0.7107$ \AA , $\mu = 1.01$ mm^{-1} , $F(000) = 664$, $T = 295$ (2) K, $R = 0.037$ for 3245

0108-2701/87/040792-03\$01.50

Table 2. Bond lengths (\AA) and angles ($^\circ$)

Mo—O	1.646 (5)	Mo—N(1)	2.228 (5)
Mo—N(2)	2.050 (6)	Mo—N(3)	2.063 (6)
Mo—N(4)	2.053 (4)	N(1)—C(1)	1.127 (8)
C(1)—S(1)	1.636 (6)	N(2)—C(2)	1.157 (10)
C(2)—S(2)	1.612 (8)	N(3)—C(3)	1.151 (9)
C(3)—S(3)	1.596 (7)	N(4)—C(4)	1.158 (6)
C(4)—S(4)	1.599 (5)	N(5)—C(51)	1.482 (6)
N(5)—C(52)	1.465 (6)	N(5)—C(53)	1.465 (7)
N(5)—C(54)	1.461 (8)		
O—Mo—N(1)	179.5 (2)	O—Mo—N(2)	98.4 (2)
N(1)—Mo—N(2)	82.1 (2)	O—Mo—N(3)	97.4 (2)
N(1)—Mo—N(3)	82.1 (2)	N(2)—Mo—N(3)	164.2 (2)
O—Mo—N(4)	96.4 (1)	N(1)—Mo—N(4)	83.6 (1)
N(2)—Mo—N(4)	91.0 (1)	N(3)—Mo—N(4)	87.3 (1)
N(4)—Mo—N(4')	166.7 (2)	Mo—N(1)—C(1)	179.4 (5)
N(1)—C(1)—S(1)	178.8 (5)	Mo—N(2)—C(2)	169.4 (6)
N(2)—C(2)—S(2)	179.5 (6)	Mo—N(3)—C(3)	162.3 (6)
N(3)—C(3)—S(3)	178.9 (7)	Mo—N(4)—C(4)	165.4 (4)
N(4)—C(4)—S(4)	178.9 (4)	C(51)—N(5)—C(52)	111.0 (4)
C(51)—N(5)—C(53)	107.2 (4)	C(52)—N(5)—C(53)	109.6 (4)
C(51)—N(5)—C(54)	109.8 (4)	C(52)—N(5)—C(54)	111.7 (4)
C(53)—N(5)—C(54)	107.3 (5)		

N(4') is related to N(4) by the mirror plane (symmetry operator $x, \frac{1}{2}-y, z$).

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observed reflections. The distorted octahedral Mn environment is comprised of three CO ligands, defining one octahedral face, two N atoms and the Cl atom. The greatest distortion from the ideal geometry arises as a result of the restricted bite distance of the chelate; N(1)—Mn—N(2) 78.5 (1), 78.8 (1) $^\circ$. There are no

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