

occupying the axial sites. The geometry is similar to that reported for the complexes of the ligand with triphenyltin chloride and nitrate (Nardelli *et al.*, 1979; Pelizzi & Pelizzi, 1980a). All three complexes are dinuclear with respect to the Sn atoms they contain: the {Ph₂P(O)CH₂}₂ ligand bridges the Sn atoms through the O atoms and the molecule has a crystallographic centre of symmetry lying in the middle of the CH₂—CH₂ bond. The Sn—O—P angle in the title compound [147.2 (3)°] is close to that in the nitrate analogue [148.3 (8)°] (Nardelli *et al.*, 1979), but significantly less than the value of 161.8 (2)° observed in the chloro complex (Pelizzi & Pelizzi, 1980a). The odd result for the latter complex has been ascribed to steric effects of the phenyl rings which although located in the equatorial plane are bent towards the O—P vector (Sn is 0.18 Å from the equatorial plane towards the Cl atom). In the title compound, the metal atom is only 0.001 Å from the equatorial plane towards the Br atom, and the longer Sn—O and P=O bonds together with larger O—P—CH₂ bond angle minimize the steric hindrance. The dihedral angles that the phenyl rings make with the equatorial plane are 40.6 (5)° for ring C(11)—C(16), 70.1 (4)° for ring C(21)—C(26) and 11.5 (9)° for ring C(31)—C(36), respectively. The coordination around P is not regularly tetrahedral with O=P—C angles significantly larger than the

C—P—C angles, in agreement with the increased electron density associated with the P=O double bond.

References

- CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
- DONDI, S., NARDELLI, M., PELIZZI, C., PELIZZI, G. & PREDIERI, G. (1985). *J. Chem. Soc. Dalton Trans.* pp. 487–491.
- DONDI, S., NARDELLI, M., PELIZZI, C., PELIZZI, G. & PREDIERI, G. (1986). *J. Organomet. Chem.* **308**, 195–206.
- Enraf-Nonius (1979). *Structure Determination Package*. Enraf-Nonius, Delft, The Netherlands.
- HARRISON, P. G., SHARPE, N. W., PELIZZI, C., PELIZZI, G. & TARASCONI, P. (1983a). *J. Chem. Soc. Dalton Trans.* pp. 921–926.
- HARRISON, P. G., SHARPE, N. W., PELIZZI, C., PELIZZI, G. & TARASCONI, P. (1983b). *J. Chem. Soc. Dalton Trans.* pp. 1687–1693.
- Molecular Structure Corporation (1985). *TEXRAY Structure Analysis Package*. MSC, 3200A Research Forest Drive, The Woodlands, TX77381, USA.
- NARDELLI, M., PELIZZI, C. & PELIZZI, G. (1979). *Inorg. Chim. Acta*, **33**, 181–187.
- PELIZZI, C. & PELIZZI, G. (1980a). *J. Organomet. Chem.* **202**, 411–419.
- PELIZZI, C. & PELIZZI, G. (1980b). *Inorg. Nucl. Chem. Lett.* **16**, 451–454.

Acta Cryst. (1990). **C46**, 2036–2039

Structure of Tris(diethyldithiophosphato-*S,S'*)- μ -(diethylthiophosphate-*O,S*)-(oxazole)- μ_3 -sulfido-tri- μ_2 -sulfido-cyclo-trimolybdenum(3 *Mo-Mo*)

BY LU SHAO-FANG, HUANG MING-DONG, HUANG JIAN-QUAN,* HUANG ZI-XIANG AND HUANG JIN-LING*

Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, Fuzhou, Fujian 350002, People's Republic of China

(Received 5 July 1989; accepted 22 January 1990)

Abstract. [Mo₃(C₄H₁₀O₃PS)(C₄H₁₀O₂PS₂)₃(S)₄(C₃H₃NO)], *M_r* = 1209.694, triclinic, *P* $\bar{1}$, *a* = 11.931 (3), *b* = 13.203 (4), *c* = 15.014 (3) Å, α = 78.17 (2), β = 91.34 (2), γ = 109.63 (2)°, *V* = 2178 (2) Å³, *Z* = 2, *D_x* = 1.845 g cm⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 15.038 cm⁻¹, *F*(000) = 1212, *T* = 296 K. The final *R* = 0.048 for 6548 reflections. The crystal structure consists of discrete neutral molecules [Mo₃S₄{ μ -SOP(OEt)₂}{S₂P(OEt)₂}(C₃H₃ON)] which contain three Mo—Mo bonds. One of these is bridged

by an unsymmetrical bidentate ligand obtained by the oxidation of μ -{S₂P(OEt)₂}. The three Mo—Mo bond lengths are 2.7328 (7), 2.7605 (7) and 2.7687 (7) Å. A comparison of the structure with those of [Mo₃S₄{S₂P(OEt)₂}(C₃H₃ON)] and [Mo₃OS₃{S₂P(OEt)₂}(C₃H₃ON)] is given.

Introduction. As part of a systematic study of the reactivity of the molybdenum cluster [Mo₃S₄{S₂P(OEt)₂}(H₂O)] (1) (Huang *et al.*, 1987), we treated Fe(ClO₄)₃ with (1) in CH₃CN solution containing 0.3% oxazole as impurity. The reaction led unexpectedly to the title compound.

* Invited Professor at Fuzhou University.

Experimental. 0.4 g of the cluster (1) was dissolved in 10 ml oxazole containing CH₃CN, 0.3 g Fe(ClO₄)₃ was added and the mixture heated under reflux. On cooling, brownish-black block-like crystals were obtained.

A crystal of approximate dimensions 0.5 × 0.2 × 0.7 mm was used. All X-ray measurements were made with a CAD-4 diffractometer. The unit-cell dimensions were obtained by a least-squares treatment of the setting angles of 25 automatically centered reflections with 14° ≤ θ(Mo Kα) ≤ 15°. The intensities of 8530 independent reflections with 1 < θ ≤ 26° were measured using the ω-scan technique (0 ≤ h ≤ 14, -16 ≤ k ≤ 16, -18 ≤ l ≤ 18). Scan range (0.6 + 0.35tanθ)°, variable scan speed between 0.9 and 5° min⁻¹. There was no evidence of crystal decay from three standard reflections monitored every 1.5 h of exposure. The crystal orientation, checked after every 200 measurements throughout the experiment, showed no change in setting angle larger than 0.12°. 6548 reflections were considered observed [I ≥ 3σ(I)] and used for the structure determination. Corrections for Lorentz, polarization and empirical ψ-scan absorption applied (max. transmission 99.9%, min. 71.4%).

The structure was solved by a combination of Patterson and direct methods (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982) which gave the positions of the three Mo atoms. The locations of all non-H atoms were obtained from successive difference syntheses. H atoms were not located. Refinements on F by full-matrix least-squares methods with anisotropic thermal parameters for all atoms led to the final values of R and wR of 0.048 and 0.068, S = 1.80 for 433 refined parameters, w = 1/σ²(F), (Δ/σ)_{max} = 0.13. Final difference syntheses showed residual electron density ranging from 2.43 [ca 0.90 Å from P(4)] to -1.54 e Å⁻³. Atomic scattering factors and dispersion corrections were taken from *International Tables for X-ray Crystallography* (1974). All calculations were performed on a VAX-785 computer with the SDP program package (Frenz, 1985).

Discussion. Final atomic coordinates and equivalent isotropic thermal parameters are listed in Table 1. Important bond lengths and angles are given in Table 2.* A perspective view of the molecule is shown in Fig. 1.

The results suggest that Fe(ClO₄)₃ only catalyzes the replacement of one S atom in the μ-[S₂P(OEt)₂]

* Lists of structure factors, anisotropic thermal parameters bond angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52989 (38 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Fractional atomic coordinates and equivalent isotropic temperature factors

$$B_{eq} = (4/3)[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)].$$

	x	y	z	B _{eq} (Å ²)
Mo(1)	0.19505 (5)	0.26177 (4)	0.30358 (4)	2.73 (1)
Mo(2)	0.25175 (5)	0.12409 (4)	0.21350 (4)	2.79 (1)
Mo(3)	0.18939 (5)	0.29765 (4)	0.11604 (4)	2.70 (1)
S	0.3586 (1)	0.3106 (1)	0.2016 (1)	2.82 (3)
S(12)	0.1134 (2)	0.0734 (1)	0.3316 (1)	3.33 (4)
S(13)	0.0433 (1)	0.2892 (1)	0.2163 (1)	3.28 (4)
S(23)	0.1177 (2)	0.1189 (1)	0.1018 (1)	3.32 (4)
S(01)	0.2753 (2)	0.4582 (1)	0.3273 (1)	3.54 (4)
S(02)	0.0681 (2)	0.2523 (2)	0.4431 (1)	4.03 (4)
S(03)	0.4181 (2)	0.1226 (1)	0.1117 (1)	3.88 (4)
S(04)	0.2052 (2)	-0.0823 (1)	0.2195 (1)	4.18 (4)
S(05)	0.3272 (2)	0.3577 (2)	-0.0259 (1)	3.91 (4)
S(06)	0.0533 (2)	0.3372 (2)	-0.0128 (1)	4.19 (4)
S(07)	0.4017 (2)	0.0873 (2)	0.3380 (2)	5.03 (5)
O(01)	0.3279 (4)	0.2458 (3)	0.4125 (3)	3.5 (1)
N(31)	0.2499 (5)	0.4858 (4)	0.1011 (4)	3.3 (1)
C(32)	0.1785 (7)	0.5433 (5)	0.1008 (6)	4.6 (2)
O(33)	0.2405 (6)	0.6517 (4)	0.0918 (5)	6.8 (2)
C(34)	0.3600 (7)	0.6601 (6)	0.0848 (6)	4.8 (2)
C(35)	0.3632 (7)	0.5593 (6)	0.0901 (5)	4.4 (2)
P(1)	0.1625 (2)	0.4103 (1)	0.4357 (1)	3.52 (4)
P(2)	0.3427 (2)	-0.0402 (1)	0.1315 (1)	3.84 (4)
P(3)	0.1909 (2)	0.3742 (1)	-0.0992 (1)	3.77 (4)
P(4)	0.3896 (2)	0.1667 (2)	0.4325 (1)	4.36 (5)
O(1)	0.2264 (4)	0.4361 (4)	0.5252 (3)	4.8 (1)
O(2)	0.0842 (4)	0.4848 (4)	0.4342 (3)	4.7 (1)
O(3)	0.4327 (5)	-0.1043 (4)	0.1628 (4)	5.2 (1)
O(4)	0.3137 (5)	-0.0811 (4)	0.0403 (4)	5.6 (1)
O(5)	0.1651 (5)	0.3062 (4)	-0.1758 (3)	5.0 (1)
O(6)	0.2200 (6)	0.4881 (4)	-0.1668 (4)	6.4 (2)
O(03)	0.5165 (5)	0.2388 (4)	0.4589 (4)	5.4 (1)
O(04)	0.3427 (6)	0.0860 (5)	0.5290 (4)	6.9 (2)
C(1)	0.3219 (8)	0.3967 (8)	0.5585 (6)	7.1 (3)
C(2)	0.3777 (8)	0.4544 (8)	0.6314 (6)	6.6 (3)
C(3)	0.0086 (7)	0.5011 (6)	0.3606 (6)	5.3 (2)
C(4)	0.048 (1)	0.6216 (8)	0.3193 (8)	8.5 (3)
C(5)	0.4770 (8)	-0.1139 (7)	0.2531 (6)	6.4 (2)
C(6)	0.5228 (9)	-0.2075 (8)	0.2697 (8)	8.5 (3)
C(7)	0.2334 (9)	-0.0487 (8)	-0.0220 (7)	7.7 (3)
C(8)	0.2241 (9)	-0.1060 (8)	-0.0999 (6)	6.6 (3)
C(9)	0.273 (1)	0.5919 (8)	-0.1417 (8)	10.3 (4)
C(10)	0.298 (1)	0.6801 (7)	-0.2246 (8)	7.5 (3)
C(11)	0.1215 (8)	0.1841 (6)	-0.1530 (6)	5.8 (2)
C(12)	0.1196 (8)	0.1476 (7)	-0.2409 (6)	6.3 (2)
C(01)	0.6010 (7)	0.1848 (7)	0.4898 (7)	6.2 (2)
C(02)	0.7122 (8)	0.2709 (9)	0.5066 (9)	8.3 (3)
C(03)	0.2256 (8)	0.0087 (8)	0.5448 (7)	6.9 (3)
C(04)	0.2208 (8)	-0.0538 (7)	0.6413 (6)	5.9 (2)

group in (1) by an O atom. In addition, an oxazole ligand substitutes for H₂O and occupies the loose coordination site on Mo(3) to form the title compound. The failure of Fe to add to (1) could be interpreted in terms of either an inappropriate oxidation state or steric factors.

The molecular structure of the title compound (4) is similar to those of [Mo₃S₄{S₂P(OEt)₂}]₄-(C₃H₅ON) (2) and [Mo₃OS₃{S₂P(OEt)₂}]₄-(C₃H₅ON) (3) (Lu, Shang, Huang, Huang & Lu,

Table 2. Bond lengths (Å) and bond angles (°)

Mo(1)	Mo(2)	2.7328 (7)	N(31)	C(32)	1.317 (7)
Mo(1)	Mo(3)	2.7605 (7)	N(31)	C(35)	1.364 (7)
Mo(2)	Mo(3)	2.7687 (7)	C(32)	O(33)	1.352 (7)
Mo(1)	S	2.336 (1)	O(33)	C(34)	1.397 (8)
Mo(1)	S(12)	2.295 (1)	O(34)	C(35)	1.330 (8)
Mo(1)	S(13)	2.295 (1)	P(1)	O(1)	1.572 (5)
Mo(1)	S(01)	2.542 (1)	P(1)	O(2)	1.565 (4)
Mo(1)	S(02)	2.570 (1)	P(2)	O(3)	1.586 (4)
Mo(1)	O(01)	2.279 (4)	P(2)	O(4)	1.563 (4)
Mo(2)	S	2.332 (1)	P(3)	O(5)	1.565 (4)
Mo(2)	S(12)	2.285 (1)	P(3)	O(6)	1.565 (4)
Mo(2)	S(23)	2.286 (1)	P(4)	O(03)	1.585 (4)
Mo(2)	S(03)	2.541 (1)	P(4)	O(04)	1.596 (5)
Mo(2)	S(04)	2.574 (1)	O(1)	C(1)	1.443 (8)
Mo(2)	S(07)	2.644 (2)	O(2)	C(3)	1.438 (7)
Mo(3)	S	2.345 (1)	O(3)	C(5)	1.441 (8)
Mo(3)	S(13)	2.289 (1)	O(4)	C(7)	1.431 (8)
Mo(3)	S(23)	2.278 (1)	O(5)	C(11)	1.484 (7)
Mo(3)	S(05)	2.543 (2)	O(6)	C(9)	1.428 (9)
Mo(3)	S(06)	2.582 (2)	O(03)	C(01)	1.438 (7)
Mo(3)	N(31)	2.305 (4)	O(04)	C(03)	1.417 (8)
S(01)	P(1)	1.990 (3)	C(1)	C(2)	1.48 (1)
S(02)	P(1)	1.992 (3)	C(3)	C(4)	1.50 (2)
S(03)	P(2)	1.992 (2)	C(5)	C(6)	1.49 (1)
S(04)	P(2)	1.982 (2)	C(7)	C(8)	1.50 (1)
S(05)	P(3)	1.990 (2)	C(9)	C(10)	1.48 (2)
S(06)	P(3)	1.978 (2)	C(11)	C(12)	1.493 (9)
S(07)	P(4)	1.964 (2)	C(01)	C(02)	1.48 (1)
O(01)	P(4)	1.448 (3)	C(03)	C(04)	1.50 (1)

Mo(2)	Mo(1)	Mo(3)	60.54 (2)	Mo(3)	N(31)	C(32)	125.4 (4)
Mo(1)	Mo(2)	Mo(3)	60.24 (2)	Mo(3)	N(31)	C(35)	128.1 (4)
Mo(1)	Mo(3)	Mo(2)	59.24 (2)	C(32)	N(31)	C(35)	106.5 (5)
Mo(2)	Mo(1)	O(01)	92.43 (8)	N(31)	C(32)	O(33)	111.6 (5)
Mo(3)	Mo(1)	O(01)	138.06 (9)	C(32)	O(33)	C(34)	105.0 (5)
S	Mo(1)	S(02)	160.65 (5)	O(33)	C(34)	C(35)	107.6 (6)
S(12)	Mo(1)	S(01)	161.70 (6)	N(31)	C(35)	C(34)	109.5 (6)
S(13)	Mo(1)	O(01)	168.11 (9)	S(01)	P(1)	S(02)	107.11 (8)
S(01)	Mo(1)	S(02)	77.61 (5)	S(03)	P(2)	S(04)	106.68 (9)
Mo(1)	Mo(2)	S(07)	91.85 (4)	S(05)	P(3)	S(06)	107.24 (9)
Mo(3)	Mo(2)	S(07)	139.45 (4)	S(07)	P(4)	O(01)	118.2 (2)
S	Mo(2)	S(04)	160.67 (5)	S(01)	P(1)	O(1)	112.9 (2)
S(12)	Mo(2)	S(03)	160.57 (6)	S(01)	P(1)	O(2)	113.0 (2)
S(23)	Mo(2)	S(07)	167.71 (5)	S(02)	P(1)	O(1)	114.3 (2)
S(03)	Mo(2)	S(04)	77.10 (5)	S(02)	P(1)	O(2)	113.7 (2)
S	Mo(3)	S(06)	161.20 (6)	O(1)	P(1)	O(2)	95.9 (2)
S(13)	Mo(3)	S(05)	160.48 (5)	S(03)	P(2)	O(3)	113.4 (2)
S(23)	Mo(3)	N(31)	168.5 (1)	S(03)	P(2)	O(4)	112.7 (2)
S(05)	Mo(3)	S(06)	77.15 (6)	S(04)	P(2)	O(3)	113.1 (2)
Mo(1)	S	Mo(2)	71.67 (4)	S(04)	P(2)	O(4)	115.5 (3)
Mo(1)	S	Mo(3)	72.29 (4)	O(3)	P(2)	O(4)	95.2 (2)
Mo(2)	S	Mo(3)	72.61 (4)	S(05)	P(3)	O(5)	112.7 (2)
Mo(1)	S(12)	Mo(2)	73.26 (5)	S(05)	P(3)	O(6)	113.9 (3)
Mo(1)	S(13)	Mo(3)	74.07 (5)	S(06)	P(3)	O(5)	113.8 (2)
Mo(2)	S(23)	Mo(3)	74.71 (5)	S(06)	P(3)	O(6)	114.4 (2)
Mo(1)	S(01)	P(1)	88.01 (6)	O(5)	P(3)	O(6)	94.5 (2)
Mo(1)	S(02)	P(1)	87.20 (6)	S(07)	P(4)	O(03)	112.0 (2)
Mo(2)	S(03)	P(2)	88.38 (7)	S(07)	P(4)	O(04)	112.3 (2)
Mo(2)	S(04)	P(2)	87.64 (6)	O(01)	P(4)	O(03)	102.4 (2)
Mo(3)	S(05)	P(3)	88.23 (7)	O(01)	P(4)	O(04)	110.4 (3)
Mo(3)	S(06)	P(3)	87.37 (7)	O(03)	P(4)	O(04)	99.5 (3)
Mo(2)	S(07)	P(4)	106.10 (8)				
Mo(1)	O(01)	P(4)	129.2 (3)				

Numbers in parentheses are estimated standard deviations in the least significant digits.

1987). The main difference is that μ -{SOP(OEt)₂} acts as the bridging chelating ligand in (4) instead of μ -{S₂P(OEt)₂} as in (2) and (3). In order to show the influence of this bridging ligand on the bond parameters some representative bond lengths and angles for (2)–(4) are given in Table 3.

Table 3. Some distances (Å) and angles (°) in [Mo₃(μ_3 -X)(μ -S)₃L₄(C₃H₃ON)] complexes where L = SXP(OEt)₂, X = O or S

	(2)	(3)	(4)
Mo—Mo	2.752 (1)	2.644 (1)	2.733 (1)
	2.760 (1)	2.628 (1)	2.761 (1)
	2.767 (1)	2.642 (1)	2.769 (1)
Mo—(μ_3 -X)	2.337 ± 0.002 (S)	2.027 ± 0.012 (O)	2.338 ± 0.005 (S)
Mo—(μ_2 -S)	2.282 ± 0.012	2.282 ± 0.007	2.288 ± 0.006
Mo—S(<i>t</i>)*	2.572 ± 0.014	2.590 ± 0.040	2.559 ± 0.017
Mo—S(<i>b</i>)†	2.617 ± 0.023	2.584 ± 0.003	2.644 (2)
Mo—O(<i>b</i>)‡			2.279 (4)
Mo—N	2.316 (6)	2.281 (7)	2.305 (5)
Mo—S—P(<i>t</i>)	87.66 ± 0.93	87.43 ± 1.51	87.81 ± 0.44
Mo—S—P(<i>b</i>)	107.84 ± 0.31	103.85 ± 1.10	106.09 (08)
Mo—O—P			129.21 (20)
S—P—S(<i>t</i>)	107.27 ± 0.99	108.17 ± 0.83	107.01 ± 0.24
S—P—S(<i>b</i>)	117.05 (14)	115.06 (15)	
O—P—S			118.22 (17)

(2) [Mo₃(μ_3 -S)(μ -S)₃(dtp)₄(C₃H₃ON)], dtp = S₂P(OEt)₂.

(3) [Mo₃(μ_3 -O)(μ -S)₃(dtp)₄(C₃H₃ON)].

(4) [Mo₃(μ_3 -S)(μ -S)₃(dtp)₃{SOP(OEt)₂}(C₃H₃ON)].

(*t*) = terminal, (*b*) = bridging.

Wherever possible means, *m*, and population standard deviations, $\sigma = [(\sum x^2 - n\bar{x}^2)/n]^{1/2}$, are given in the form *m* ± σ . Individual bond lengths or angles are given with standard deviations in parentheses.

* S atom in terminal chelating dtp ligand.

† S atom in bridging chelating dtp or SOP(OEt)₂ ligand.

‡ O atom in bridging chelating SOP(OEt)₂ ligand.

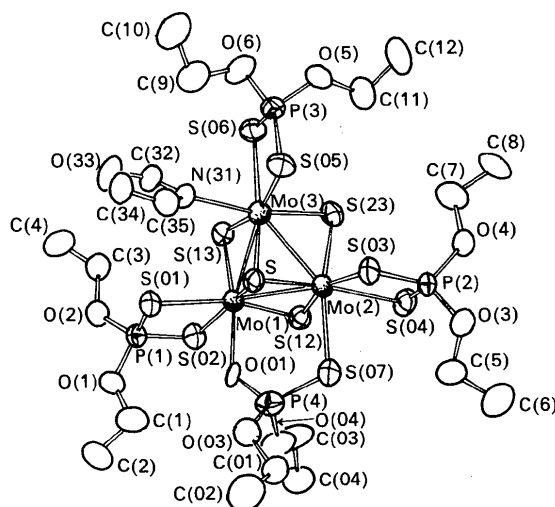


Fig. 1. The molecular structure of [Mo₃S₄{ μ -SOP(OEt)₂}₃{S₂P(OEt)₂}₃(C₃H₃ON)].

There is a substantial shortening of the Mo—Mo distances between (2) and (3) which can be ascribed to the change from μ_3 -S to μ_3 -O. Two of the Mo—Mo distances in (4) are comparable to those in (2) but the μ_2 -OS-bridged bond in (4) is shorter than the corresponding μ_2 -S₂-bridged bond in (2), possibly reflecting the lower steric demand of O compared with S. The Mo—S(*b*) bridging chelate bond in (4) is longer than corresponding values in (2) and (3), possibly as a result of the differing electronic properties of O compared with S.

Finally, it is interesting to note that the {SOP(OEt)₂} ligand always adopts a bridging position to form an Mo₂OSP five-membered ring (Lu, Huang, Huang & Lu, 1984; Lu, Huang, Lin & Huang, 1987). We are unaware of any examples of {SOP(OEt)₂} chelating to an Mo atom to form an MoSOP four-membered ring. This leads us to believe that the [Mo₃S₄{μ-SOP(OEt)₂}₃.L₃] cluster compound may be stable.

References

FRENZ, B. A. (1985). Enraf-Nonius *SDP-Plus Structure Determination Package*. Version 3.0. Enraf-Nonius, Delft, The Netherlands.

HUANG, J.-Q., LU, S.-F., SHANG, M.-Y., LIN, X.-T., HUANG, M.-D., LIN, Y.-H., WU, D.-M., ZHUANG, H.-H., HUANG, J.-L. & LU, J.-L. (1987). *J. Struct. Chem.* **6**, 219–233.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

LU, S.-F., HUANG, J.-Q., HUANG, J.-L. & LU, J.-X. (1984). *Jiegou Huaxue, Engl. Ed.* **3**, 151–154.

LU, S.-F., HUANG, J.-Q., LIN, Y.-H. & HUANG, J.-L. (1987). *Acta Chim. Sin.* **3**, 199–209.

LU, S.-F., SHANG, M.-Y., HUANG, J.-Q., HUANG, J.-L. & LU, J.-X. (1987). *Sci. Sin.* **B30**, 147–160.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN11/82. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

Acta Cryst. (1990). **C46**, 2039–2041

Structure of *catena*-Bis[μ-chloro-chloro-μ-quinoxaline-*N,N'*-copper(II)]

BY SEPPO LINDROOS* AND PAAVO LUMME

Division of Inorganic Chemistry, Department of Chemistry, University of Helsinki, Vuorik. 20, SF-00100 Helsinki, Finland

(Received 20 December 1989; accepted 22 February 1990)

Abstract. [Cu(C₈H₆N₂)Cl₂]_n, *M_r* = 264.60, monoclinic, *C*2/*m*, *a* = 13.237 (5), *b* = 6.935 (3), *c* = 9.775 (3) Å, β = 107.88 (2)°, *V* = 853.9 (5) Å³, *Z* = 4, *D_m* = 2.01 (2), *D_x* = 2.06 g cm⁻³, λ(Mo *Kα*) = 0.71069 Å, μ(Mo *Kα*) = 30.0 cm⁻¹, *F*(000) = 524, *T* = 295 K, final *R* = 0.025 for 979 unique reflections. The polymeric structure is composed of double chains in which quinoxaline molecules form bridges between Cu atoms along the *b* axis and the two chains are bound together by two Cl atoms. The coordination polyhedron about the Cu atom is a trigonal bipyramid with three Cl atoms in the equatorial plane with the quinoxaline N atoms occupying the apical positions.

Introduction. Quinoxaline, like other nitrogen heterocycles with two N atoms in the molecule, has a tendency to form polymeric structures in which quinoxaline forms a bridge between two metal atoms (Lumme, Lindroos & Lindell, 1987; Richardson, Hatfield, Stoklosa & Wasson, 1973).

The metal-bridging capabilities of pyrazine, which quinoxaline chemically resembles, have been well studied (Santoro, Mighell & Reimann, 1970; Darriet, Haddad, Duesler & Hendrickson, 1979; Havnes, Rettig, Sams, Thompson & Trotter, 1987). Unlike

pyrazine only a few structures of quinoxaline metal complexes have been determined. To learn more about the coordination behaviour of polymeric quinoxaline (= Q) compounds, we have determined the structure of Cu(Q)Cl₂, the results of which we present here.

Experimental. Quinoxaline (0.5 mmol) in ethanol (100 cm³) was added to CuCl₂.2H₂O (0.5 mmol) in ethanol (100 cm³). The mixture was carefully heated (at ~ 313 K) for 6 h and allowed to stand for several weeks. The green crystals that formed were filtered and washed successively with ethanol and ether. The crystal selected for data collection had dimensions 0.2 × 0.2 × 0.4 mm. The density was determined by flotation. The unit-cell parameters were determined by a least-squares fit of the setting angles of 25 reflections with 13.2 ≤ 2θ ≤ 27.7°, measured on a Nicolet *P3F* diffractometer. The intensity measurements were carried out at room temperature (295 K) with graphite-monochromatized Mo *Kα* radiation and the ω–2θ scan technique. The scan rate varied from 2.0 to 29.3° min⁻¹.

A set of 1056 unique reflections was obtained from 1113 reflections measured in the range 3 ≤ 2θ ≤ 55° (*h* 0→19, *k* 0→11, *l* –14→14). 979 reflections with *F_o* > 4σ(*F_o*) were considered as observed and used in refinement. Three strong reflections (133, 204 and

* Author to whom correspondence should be addressed.