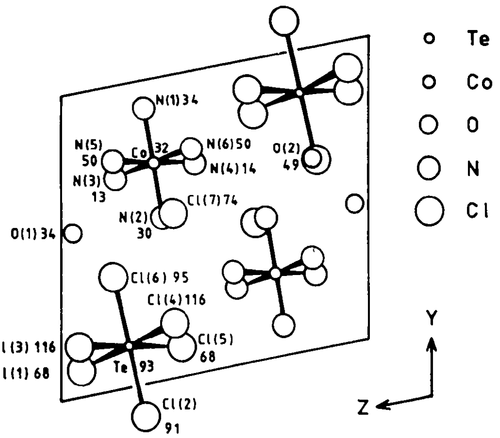

Fig. 1. Projection of the crystal structure of $[Co(NH_3)_6]Cl(TeCl_6).(2-x)H_2O$ along [100]. Heights of atoms are indicated as percentages of the *a* length. The unit cell is outlined.

Zehnder (1987) and Abriel (1987). The last paper gives basic structural data for all compounds containing AX_6^{2-} ions (A = Se, Te; X = Cl, Br, I) known to date including Δ values for octahedral distortion. Extended symmetry rules considering SbX_6^{3-} and BiX_6^{3-} species are given by du Bois & Abriel (1988). Relevant interatomic distances for hydrogen-bonding contacts [here O(1)···N(5) and O(1)···O(2)] are listed by Wells (1986).

Financial support by the Deutsche Forschungsgemeinschaft and the Fonds der Chemischen Industrie is gratefully acknowledged.

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Structure of 2,3,4-Tris(diethyl dithiophosphato-*S*,*S*′)-1-iodo-2,3-μ_2-methanoato-4-(pyridine)-tetrakis(μ_3-sulfido)-copper(I)trimolybdenum(IV)

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(*Received* 26 *April* 1989; *accepted* 20 *June* 1989)

Abstract. $[CuMo_3S_4(I)(CHO_2)(C_4H_{10}O_2PS_2)_3(C_5H_5N)]$, M_r = 1286, triclinic, $P\overline{1}$, a = 11·460 (8), b = 14·237 (8), c = 15·046 (8) Å, α = 112·35 (5), β = 90·09 (5), γ = 111·77 (5)°, V = 2079·3 Å^3, Z = 2, D_x = 2·05 g cm^{-3}, $\lambda(\text{Mo } K\alpha)$ = 0·71073 Å, μ_m = 27·4 cm^{-1}, $F(000)$ = 1256, R = 0·058 for 3372 observed unique reflections $I \geq 5\sigma(I)$. The structure contains discrete molecules with a central $CuMo_3S_4$ distorted cubic cluster (mean Mo—S 2·329, Cu—S 2·280, Mo···Mo 2·744, Mo···Cu 2·856 Å). Octahedral coordination at each Mo is completed by two S atoms of bidentate chelating diethyl dithiophosphate ligands (mean Mo—S 2·531 Å), and by a terminal pyridine ligand for one Mo (Mo—N 2·368 Å) and an $HCOO^-$ bridging ligand for the other two Mo (mean Mo—O 2·187 Å); an I^- ligand completes tetrahedral geometry at the Cu atom.

Experimental. The title compound was prepared by the method described by Wu, Lu, Zhu, Wu & Lu (1987). The crystal measured 0·50 × 0·25 × 1·00 mm. Data were collected using a CAD-4 κ-geometry diffractometer using Mo $K\alpha$ radiation at *ca* 296 K. $\omega/2\theta$ scan, scan speed varied from 2 to 7° min^{-1} (in ω), the scan width was $(0{\cdot}50 + 0{\cdot}35\tan\theta)°$. Cell constants were obtained by least-squares analysis on 25 diffraction maxima ($26 < 2\theta < 27°$). The intensities were corrected for absorption using empirical scan data (maximum and minimum transmission factors 1·06 and 0·86, respectively), and Lorentz and polarization factors to give a total of 7703 intensities, to a maximum 2θ of 50° ($0 \leq h \leq 13$, $-16 \leq k \leq 15$, $-17 \leq l \leq 17$). Max. $(\sin\theta)/\lambda$ = 0·59 Å^{-1}, 4331 reflections with $I < 5\sigma(I)$ are considered unobserved. 3372 reflections with $I \geq 5\sigma(I)$ used in the refinement. Three standard reflections were measured periodically, no random deviations.

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0108-2701/89/121988-03$03.00

Table 1. *Atomic coordinates and thermal parmeters* (Å^2)

	x	y	z	B_{eq}*
Mo1	0·7391 (2)	0·0670 (1)	0·8464 (1)	3·52 (4)
Mo2	0·8010 (2)	0·1201 (1)	0·6883 (1)	3·99 (4)
Mo3	0·5880 (2)	−0·0585 (1)	0·6650 (1)	3·99 (4)
Cu	0·5960 (2)	0·1592 (2)	0·7823 (2)	4·96 (7)
I	0·4817 (2)	0·2830 (1)	0·8256 (1)	6·50 (4)
S	0·7944 (4)	−0·0378 (4)	0·7059 (3)	3·9 (1)
S1	0·5984 (5)	0·0845 (4)	0·6197 (3)	4·4 (1)
S2	0·5181 (4)	0·0045 (4)	0·8129 (4)	4·2 (1)
S3	0·8087 (5)	0·2415 (4)	0·8466 (4)	4·5 (1)
S11	0·9554 (5)	0·1154 (4)	0·9352 (4)	4·3 (1)
S12	0·7217 (5)	0·1623 (4)	1·0259 (4)	4·3 (1)
S21	1·0342 (5)	0·1647 (4)	0·6980 (4)	5·6 (2)
S22	0·8774 (6)	0·2856 (4)	0·6428 (4)	5·6 (2)
S31	0·3579 (5)	−0·1517 (5)	0·5795 (4)	5·8 (2)
S32	0·5290 (6)	−0·2533 (4)	0·6462 (4)	6·1 (2)
N	0·695 (1)	−0·090 (1)	0·8825 (9)	4·0 (3)
O21	0·803 (1)	0·0231 (9)	0·5362 (9)	5·0 (4)
O31	0·628 (1)	−0·1263 (9)	0·5172 (9)	5·3 (4)
P1	0·8986 (5)	0·1814 (4)	1·0590 (4)	4·0 (1)
P2	1·0513 (6)	0·2985 (5)	0·6763 (4)	5·4 (2)
P3	0·3580 (7)	−0·2922 (5)	0·5790 (5)	6·9 (2)
O1	0·913 (1)	0·1360 (9)	1·1358 (9)	4·8 (4)
O2	0·993 (1)	0·305 (1)	1·1219 (9)	5·4 (4)
O3	1·136 (1)	0·312 (1)	0·596 (1)	6·9 (4)
O4	1·124 (1)	0·407 (1)	0·769 (1)	7·2 (5)
O5	0·325 (2)	−0·387 (1)	0·475 (1)	9·2 (6)
O6	0·251 (2)	−0·352 (1)	0·628 (1)	15·2 (7)
C1	0·718 (2)	−0·070 (2)	0·482 (1)	5·8 (6)
C2	0·767 (2)	−0·149 (1)	0·857 (1)	4·0 (5)
C3	0·744 (2)	−0·240 (1)	0·877 (1)	4·9 (5)
C4	0·642 (2)	−0·271 (2)	0·924 (2)	5·8 (6)
C5	0·566 (2)	−0·209 (2)	0·950 (1)	5·7 (6)
C6	0·595 (2)	−0·121 (1)	0·926 (1)	4·2 (5)
C11	0·828 (2)	0·033 (1)	1·134 (1)	6·6 (6)
C12	0·853 (2)	0·025 (1)	1·231 (1)	5·8 (6)
C21	1·017 (3)	0·391 (2)	1·082 (2)	8·0 (8)
C22	1·145 (3)	0·470 (2)	1·122 (2)	12 (1)
C31	1·098 (3)	0·234 (2)	0·495 (1)	9·7 (9)
C32	1·161 (3)	0·298 (2)	0·437 (2)	11 (1)
C41	1·250 (2)	0·433 (2)	0·809 (2)	11·0 (8)
C42	1·305 (3)	0·525 (3)	0·882 (3)	17 (1)
C51	0·400 (3)	−0·372 (2)	0·401 (2)	8·8 (9)
C52	0·413 (4)	−0·475 (2)	0·346 (3)	17 (1)
C61	0·180 (4)	−0·346 (4)	0·676)4)	20 (2)
C62	0·054 (3)	−0·424 (4)	0·646 (3)	16 (2)

* Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as: $\frac{4}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

The structure was solved by direct methods using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), five heavy atoms (3 Mo, Cu, I) were located in the *E* map. The remaining non-H atoms were located in the succeeding difference Fourier syntheses (H atoms were placed in geometrically calculated positions with C—H 0·95 Å, but not included in the refinement). The structure was refined by full-matrix least-squares techniques with anisotropic thermal parameters for all non-H atoms (406 variables). Final $R = 0{\cdot}058$, $wR = 0{\cdot}068$ and $S = 4{\cdot}85$; the function minimized was $\sum w(|F_o| - |F_c|)^2$, $w = 4F_o^2/\sigma^2(F_o^2)$, $\sigma^2(F_o^2) = [\sigma_o^2(F_o^2{}_{\max}) + (0{\cdot}04F_o^2)^2]$ where $\sigma_o^2(F_o^2)$ is the standard deviation based on counting statistics. $(\Delta/\sigma)_{\max} = 0{\cdot}48$, in final difference electron density synthesis max. height less than 1·12 e Å^{-3}. All calculations were performed on a VAX 785 computer using *SDP* (Frenz, 1978), the scattering factors were taken from Cromer & Waber (1974). *ORTEP*II (Johnson, 1976)

Table 2. *Selected bond distances* (Å) *and bond angles* (°)

Mo1—Mo2	2·777 (1)	Cu—S1	2·271 (3)
Mo1—Mo3	2·761 (1)	Cu—S2	2·274 (3)
Mo1—Cu	2·811 (1)	Cu—S3	2·294 (3)
Mo1—S	2·325 (2)	S11—P1	1·976 (3)
Mo1—S2	2·332 (2)	S12—P1	1·980 (3)
Mo1—S3	2·310 (3)	S21—P2	1·993 (4)
Mo1—S11	2·535 (3)	S22—P2	1·978 (4)
Mo1—S12	2·567 (3)	S31—P3	1·998 (5)
Mo1—N	2·368 (7)	S32—P3	1·983 (5)
Mo2—Mo3	2·694 (1)	N—C2	1·35 (2)
Mo2—Cu	2·865 (2)	N—C6	1·33 (2)
Mo2—S	2·336 (2)	O21—C1	1·26 (1)
Mo2—S1	2·332 (3)	O31—C1	1·31 (1)
Mo2—S3	2·329 (3)	P1—O1	1·559 (6)
Mo2—S21	2·498 (3)	P1—O2	1·579 (6)
Mo2—S22	2·547 (3)	P2—O3	1·581 (7)
Mo2—O21	2·183 (7)	P2—O4	1·556 (7)
Mo3—Cu	2·891 (1)	P3—O5	1·551 (9)
Mo3—S	2·324 (3)	P3—O6	1·57 (1)
Mo3—S1	2·345 (3)	C2—C3	1·38 (1)
Mo3—S2	2·327 (3)	C3—C4	1·39 (1)
Mo3—S31	2·543 (3)	C4—C5	1·42 (1)
Mo3—S32	2·498 (3)	C5—C6	1·36 (1)
Mo3—O31	2·190 (6)	O—C(av.)(in OEt)	1·39 ± 0·14*
Cu—I	2·467 (1)	C—C(av.)(in OEt)	1·43 ± 0·09*

Mo2—Mo1—Mo3	58·24 (3)	Mo3—S2—Cu	77·85 (8)
Mo2—Mo1—Cu	61·70 (3)	Mo1—S3—Mo2	73·55 (8)
S—Mo1—S2	105·10 (9)	Mo1—S3—Cu	75·25 (8)
S—Mo1—S3	104·53 (9)	Mo2—S3—Cu	76·61 (8)
S11—Mo1—S12	77·16 (8)	S11—P1—S12	107·2 (1)
Mo1—Mo2—Mo3	60·57 (3)	S11—P1—O1	114·3 (3)
Mo1—Mo2—Cu	59·73 (3)	S11—P1—O2	112·1 (3)
S—Mo2—S1	108·6 (1)	S12—P1—O1	113·1 (3)
S—Mo2—S3	103·59 (9)	S12—P1—O2	114·2 (3)
S1—Mo2—S3	99·41 (9)	O1—P1—O2	96·0 (4)
S21—Mo2—S22	78·82 (9)	S21—P2—S22	107·5 (2)
Mo1—Mo3—Mo2	61·20 (3)	S21—P2—O3	111·7 (3)
Mo1—Mo3—Cu	59·60 (3)	S21—P2—O4	111·2 (3)
S—Mo3—S1	108·6 (1)	S22—P2—O3	112·7 (3)
S—Mo3—S2	105·33 (9)	S22—P2—O4	109·9 (4)
S1—Mo3—S2	97·49 (9)	O3—P2—O4	103·8 (4)
S31—Mo3—S32	78·4 (2)	S31—P3—S32	106·3 (2)
Mo1—Cu—Mo2	58·57 (3)	S31—P3—O5	113·3 (4)
Mo1—Cu—Mo3	57·89 (3)	S31—P3—O6	115·4 (6)
Mo2—Cu—Mo3	55·82 (3)	S32—P3—O5	112·1 (4)
S1—Cu—S2	101·2 (1)	S32—P3—O6	110·3 (5)
S1—Cu—S3	102·3 (2)	O5—P3—O6	99·6 (8)
S2—Cu—S3	104·7 (1)	O21—C1—O31	122 (1)
Mo1—S—Mo2	73·14 (7)	C2—N—C6	120·4 (8)
Mo1—S—Mo3	72·84 (7)	N—C2—C3	122·0 (9)
Mo2—S—Mo3	70·66 (7)	C2—C3—C4	118 (1)
Mo2—S1—Mo3	70·36 (7)	C3—C4—C5	119 (1)
Mo2—S1—Cu	76·99 (8)	C4—C5—C6	119 (1)
Mo3—S1—Cu	77·52 (8)	N—C6—C5	122 (1)
Mo1—S2—Mo3	72·66 (7)	P—O—C(av.)(in dtp)	126·0 ± 9·9*
Mo1—S2—Cu	75·19 (8)	O—C—C(av.)(in OEt)	111·6 ± 5·5*

* Standard e.s.d. $\sigma = [(\sum x^2 - n\bar{x}^2)/n]$.

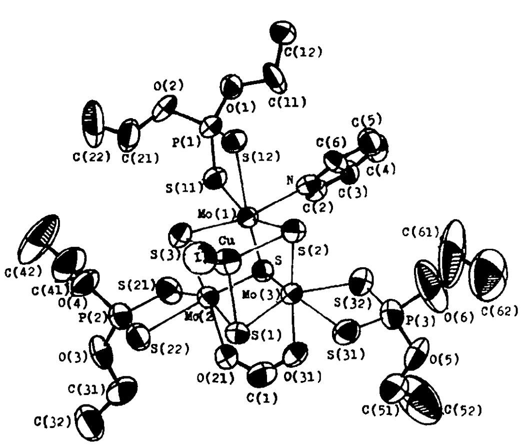

Fig. 1. Drawing of the title compound with thermal ellipsoids.

was used to produce the view of the molecule (Fig. 1). The atom coordinates and thermal parameters are listed in Table 1; the important bond lengths and bond angles are given in Table 2.*

Related literature. A related derivative of the title compound [$CuMo_3S_4\{S_2P(OC_2H_5)_3\}(I)(\mu_2\text{-}CH_3COO)\{HCON(CH_3)_2\}$] has been reported (Wu *et al.*, 1987).

This research has been supported by grants from the Fuzhou Laboratory of Structural Chemistry, Fujian Institute of Research on the Structure of Matter, Chinese Academy of Sciences, and the National Science Foundation of China.

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* Fuller lists of bond lengths and angles, and lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52065 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1989). C45, 1990–1992

Structure of 2-(Acetonitrile)-2,3,4-tris(diethyl dithiophosphato-*S*,*S′*)-1-iodo-tetrakis(μ_3-sulfido)-3,4-μ_2-trichloroacetato-copper(I)tritungsten(IV)

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(*Received* 17 *March* 1989; *accepted* 21 *June* 1989)

Abstract. [$CuW_3S_4(I)(C_2Cl_3O_2)(C_4H_{10}O_2PS_2)_3(C_2H_3N)$], $M_r = 1629$, triclinic, $P\bar{1}$, $a = 11{\cdot}684\,(6)$, $b = 14{\cdot}243\,(5)$, $c = 15{\cdot}455\,(4)$ Å, $\alpha = 103{\cdot}88\,(3)$, $\beta = 109{\cdot}01\,(3)$, $\gamma = 65{\cdot}64\,(4)°$, $V = 2198{\cdot}4$ Å^3, $Z = 2$, $D_x = 2{\cdot}46$ g cm^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71073$ Å, $\mu = 99{\cdot}5$ cm^{-1}, $F(000) = 1453$, $T = 296$ K, $R = 0{\cdot}033$ for 3659 unique observed reflections with $I \geq 10\sigma(I)$. Each W atom is octahedrally coordinated by three μ_3-S atoms (W—μ_3-S av. 2·334 Å) and an $S_2P(OEt)_2$(dtp) chelating ligand [W—S_t(dtp) 2·517 Å]. The octahedra surrounding the W(2) and W(3) atoms are completed by a CCl_3COO bridging ligand (W—O_b 2·200 Å), and that surrounding W(1) by a CH_3CN molecule (W—N 2·211 Å). The Cu atom is tetrahedrally coordinated by three μ_3-S atoms (Cu—μ_3-S av. 2·300 Å) and one I atom. There are some distortions in the cubane-like $(W_3CuS_4)^{5+}$ core, with three W—W bonds and three weak W—Cu bonds: W—W (av.) 2·728, W—Cu(av.) 2·874 Å. The molecule can alternatively be described in terms of a W_3Cu tetrahedral cluster with a μ-S atom bridging each triangular face.

Experimental. Crystals of the title compound were prepared by the method described by Zhan (1989). Crystal dimensions 0·25 × 0·20 × 0·25 mm. Data were collected using a CAD-4 κ-geometry diffractometer, $\omega/2\theta$ scans, scan speed varied from 1 to 7° min^{-1} (in ω), the scan width was $(0{\cdot}40 + 0{\cdot}35\tan\theta)°$. Cell constants were obtained by least-squares analy-

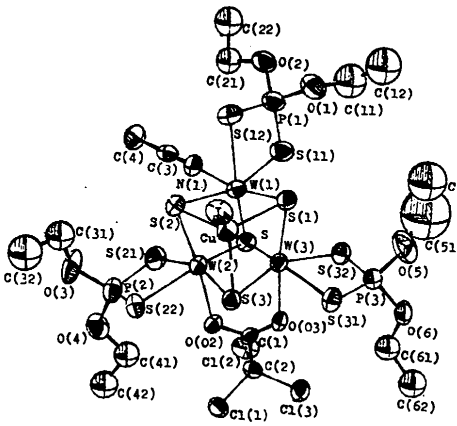

Fig. 1. Drawing of the title compound with thermal ellipsoids.

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