

difference Fourier map was 1.002 e Å<sup>-3</sup>. An *ORTEP* (Johnson, 1965) drawing of the molecule is shown in Fig. 1. Final positional and equivalent isotropic thermal parameters are listed in Table 1;\* some selected distances and angles are listed in Table 2.

**Related literature.** A previous report (Cotton & Shang, 1988) described the same tetranuclear cluster in the compound Nb<sub>4</sub>Cl<sub>10</sub>(PMe<sub>3</sub>)<sub>6</sub>·2C<sub>4</sub>H<sub>8</sub>O, the incorporated thf having been present in the reaction solution. In this work no thf was present at any time

\* Lists of structure factors, anisotropic thermal parameters and complete bond lengths and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 53973 (17 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

and a different crystalline form containing no solvent of crystallization was obtained.

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## Structure of Pentakis(3,3',4,4'-tetramethyl-2,2',5,5'-tetrathiafulvalenium) Dodeca-μ-chloro-octahedro-hexakis(chlorotantalate) Dichloromethane Solvate (2/1): (TMTTF)<sub>5</sub>[Ta<sub>6</sub>Cl<sub>18</sub>].0.5CH<sub>2</sub>Cl<sub>2</sub>

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**Abstract.** [C<sub>10</sub>H<sub>12</sub>S<sub>4</sub>]<sub>5</sub>[Ta<sub>6</sub>Cl<sub>18</sub>].0.5CH<sub>2</sub>Cl<sub>2</sub>, *M*<sub>r</sub> = 3068.62, triclinic, *P* $\bar{1}$ , *a* = 12.931 (5), *b* = 13.712 (5), *c* = 14.302 (6) Å, α = 114.31 (3), β = 97.01 (4), γ = 99.10 (5)°, *V* = 2232 Å<sup>3</sup>, *Z* = 1, *D*<sub>x</sub> = 2.283 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71073 Å, μ = 82.99 cm<sup>-1</sup>, *F*(000) = 1495, *T* = 293 K, *R* = 0.058 based on 3221 observed reflections with *I* ≥ 3σ(*I*). Four partially oxidized TMTTF molecules form stacks parallel to the [001] direction and one additional neutral TMTTF molecule, lying on the (010) plane, is perpendicular to this stack. The stacking mode of the TMTTF molecules is not regular. The separation between adjacent molecules ranges from 3.49 to 3.60 Å. The bond distances within the anion [average Ta—Ta: 2.959 (1); Ta—Cl<sup>l</sup>: 2.437 (6); Ta—Cl<sup>a</sup>: 2.502 (6) Å, where Cl<sup>l</sup> and Cl<sup>a</sup> are bridging and non-bridging Cl atoms, respectively] are in the range expected for a [Ta<sub>6</sub>Cl<sub>18</sub>]<sup>3-</sup> unit. The disordered dichloromethane

solvent molecule is located near the (0,0, $\frac{1}{2}$ ) position with a statistical occupancy.

**Experimental.** The compound was grown on a platinum wire electrode by anodic oxidation of the organic donor (2 × 10<sup>-3</sup> M) in a mixture of DMF (85%) (DMF = *N,N*-dimethylformamide) and dichloromethane (15%) under low constant current (*I* = 0.95 μA) in the presence of tetraethylammonium [(Et<sub>4</sub>N)<sub>3</sub>Ta<sub>6</sub>Cl<sub>18</sub>] salts of the anion (10<sup>-2</sup> M) as supporting electrolyte.

Black crystal 0.09 × 0.07 × 0.07 mm. Enraf-Nonius CAD-4 diffractometer, graphite-crystal-monochromatized Mo *K*α radiation. Cell dimensions: least-squares refinement from setting angles of 25 centered reflections (θ ≤ 15°). Intensities collected by θ–2θ scans. The crystals of the title compound are unstable. In fact, after several days the intensities of the three standard reflections decreased slightly and the data collection procedure was stopped. Three standard reflections measured every hour: no fluctuation in intensities in the set of reflections used in

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Table 1. Atomic coordinates and equivalent isotropic temperature factors ( $\text{\AA}^2$ )

$B_{\text{eq}} = (4/3)\sum_i \beta_{ij} \mathbf{a}_i \cdot \mathbf{a}_j$				
	x	y	z	$B_{\text{eq}}$
Ta1	0.02585 (6)	0.09272 (6)	-0.08412 (9)	3.84 (3)
Ta2	-0.16127 (6)	-0.05732 (6)	-0.07628 (9)	3.90 (3)
Ta3	-0.03732 (6)	0.13336 (6)	0.11815 (9)	3.89 (3)
Cl1	0.0736 (4)	-0.0469 (4)	-0.2330 (5)	5.0 (2)
Cl2	-0.1561 (4)	0.0401 (4)	-0.1861 (5)	4.7 (2)
Cl3	-0.2290 (4)	0.0868 (4)	0.0497 (5)	4.4 (2)
Cl4	0.2157 (4)	0.1752 (4)	-0.0079 (5)	4.7 (2)
Cl5	-0.0128 (4)	0.2617 (3)	0.0401 (5)	4.4 (2)
Cl6	-0.1429 (4)	-0.2199 (4)	-0.2236 (5)	4.8 (2)
Cl7	0.0537 (5)	0.2054 (4)	-0.1846 (6)	5.3 (2)
Cl8	-0.3532 (4)	-0.1220 (4)	-0.1661 (6)	5.9 (2)
Cl9	-0.0814 (4)	0.2948 (4)	0.2577 (6)	5.4 (2)
S1	0.6714 (4)	0.5643 (4)	0.0271 (6)	5.1 (2)
S2	0.5388 (4)	0.3405 (4)	-0.0741 (6)	5.0 (2)
C1	0.546 (2)	0.481 (1)	-0.010 (2)	3.8 (6)
C2	0.739 (1)	0.459 (2)	-0.020 (2)	4.6 (7)
C3	0.677 (2)	0.354 (2)	-0.062 (2)	4.5 (7)
C4	0.857 (2)	0.491 (2)	0.003 (2)	6.1 (8)
C5	0.715 (2)	0.250 (2)	-0.105 (2)	5.2 (7)
S3	0.5871 (4)	0.6032 (4)	0.3001 (6)	5.5 (2)
S4	0.4448 (4)	0.3850 (4)	0.2042 (6)	5.1 (2)
S5	0.2411 (4)	0.4901 (4)	0.2410 (6)	5.0 (2)
S6	0.3794 (4)	0.7107 (4)	0.3399 (6)	4.9 (2)
C6	0.456 (2)	0.527 (1)	0.267 (2)	4.0 (7)
C7	0.369 (2)	0.571 (2)	0.276 (2)	4.6 (7)
C8	0.648 (2)	0.493 (2)	0.252 (2)	5.1 (8)
C9	0.582 (2)	0.391 (2)	0.204 (2)	5.0 (7)
Cl10	0.179 (2)	0.600 (2)	0.280 (2)	5.0 (7)
Cl11	0.242 (2)	0.701 (2)	0.326 (2)	6.6 (9)
Cl12	0.762 (2)	0.518 (2)	0.265 (2)	6.4 (9)
Cl13	0.610 (2)	0.282 (2)	0.158 (2)	5.0 (7)
Cl14	0.059 (2)	0.571 (2)	0.260 (2)	5.3 (8)
Cl15	0.207 (2)	0.808 (2)	0.365 (2)	5.6 (8)
S7	0.5418 (6)	0.3403 (7)	0.4344 (7)	8.0 (3)
S8	0.6724 (6)	0.5612 (6)	0.5353 (7)	8.0 (3)
Cl16	0.547 (2)	0.481 (2)	0.494 (2)	6.2 (9)
Cl17	0.675 (2)	0.356 (2)	0.443 (2)	8 (1)
Cl18	0.738 (2)	0.455 (2)	0.493 (2)	7.0 (9)
Cl19	0.714 (2)	0.249 (3)	0.400 (2)	8 (1)
C20	0.859 (2)	0.488 (2)	0.510 (3)	8 (1)
S9	0.3780 (4)	0.0846 (5)	0.5673 (6)	5.6 (2)
S10	0.5266 (4)	-0.0174 (5)	0.6450 (6)	5.6 (2)
C21	0.482 (2)	0.015 (2)	0.547 (2)	5.0 (8)
C22	0.369 (2)	0.089 (2)	0.686 (2)	3.4 (6)
C23	0.437 (2)	0.043 (2)	0.727 (2)	6.7 (9)
C24	0.287 (2)	0.142 (2)	0.738 (2)	6.5 (9)
C25	0.445 (2)	0.034 (2)	0.829 (3)	8 (1)
C26	0.135 (7)	0.189 (6)	0.491 (7)	6 (2)*
Cl10	0.053 (8)	0.054 (7)	0.486 (7)	26 (4)*
Cl11	0.055 (4)	0.230 (4)	0.512 (4)	12 (1)*

\* Atoms refined isotropically.

the solution and refinement of the structure. One set of reflections collected up to  $2\theta = 44^\circ$ . 4521 independent reflections measured ( $-13 \leq h \leq 13$ ,  $-14 \leq k \leq 14$ ,  $0 \leq l \leq 15$ ), 3221 with  $I \geq 3\sigma(I)$ . Lorentz and polarization corrections, no absorption correction. Structure solved by analogy with the isomorphous niobium salt (Penicaud, Batail, Perrin, Coulon, Parkin & Torrance, 1987). H atoms placed at computed positions [ $d(\text{C}-\text{H}) = 1 \text{\AA}$ ;  $B_{\text{eq}} = 4 \text{\AA}^2$ ]. Full-matrix least-squares anisotropic ( $\beta_{ij}$ ) refinement (H atoms isotropic, not refined); the C and Cl atoms of the solvent molecule are included in fixed observed positions with occupancy factors of 0.25.  $R = 0.058$ ,  $wR = 0.076$ ,  $\sum w(|F_o| - |F_c|)^2$  minimized,  $w = 4F_o^2 / [\sigma^2(F_o^2) - (0.07F_o^2)^2]$ ,  $S = 1.69$ ,  $\Delta/\sigma_{\text{max}} = 1.9$ ,  $\Delta\rho_{\text{max}} = 3.05$ ,  $\Delta\rho_{\text{min}} = -3.12 \text{ e \AA}^{-3}$ . Scattering factors from *International Tables for X-ray Crystallography*

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with e.s.d.'s in parentheses

Ta1—Ta2	2.963 (1)	S3—C8	1.73 (2)
Ta1—Ta2	2.961 (3)	S4—S4	1.75 (2)
Ta1—Ta3	2.956 (2)	S4—C9	1.77 (2)
Ta1—Ta3	2.965 (1)	S5—C7	1.72 (2)
Ta1—Cl1	2.433 (6)	S5—C10	1.74 (2)
Ta1—Cl2	2.437 (5)	S6—C7	1.73 (2)
Ta1—Cl4	2.439 (4)	S6—Cl1	1.74 (2)
Ta1—Cl5	2.443 (5)	C6—C7	1.35 (3)
Ta1—Cl7	2.522 (8)	C8—C9	1.35 (3)
Ta2—Ta3	2.956 (1)	C8—C12	1.43 (3)
Ta2—Ta3	2.958 (1)	C9—C13	1.49 (3)
Ta2—Cl2	2.445 (8)	C10—C11	1.33 (3)
Ta2—Cl3	2.433 (5)	C10—C14	1.50 (3)
Ta2—Cl4	2.451 (8)	C11—C15	1.51 (3)
Ta2—Cl6	2.426 (5)	S7—Cl6	1.75 (3)
Ta2—Cl8	2.486 (5)	S7—Cl7	1.69 (3)
Ta3—Cl1	2.437 (8)	S8—Cl6	1.69 (3)
Ta3—Cl3	2.429 (5)	S8—Cl18	1.74 (3)
Ta3—Cl5	2.444 (7)	C16—C16	1.40 (4)
Ta3—Cl6	2.430 (5)	C17—C18	1.31 (3)
Ta3—Cl9	2.500 (6)	C17—C19	1.54 (5)
S1—C1	1.71 (2)	C18—C20	1.52 (4)
S1—C2	1.75 (2)	S9—C21	1.75 (2)
S2—C1	1.74 (2)	S9—C22	1.69 (3)
S2—C3	1.74 (2)	S10—C21	1.70 (3)
Cl1—C1	1.39 (3)	S10—C23	1.81 (3)
C2—C3	1.37 (3)	C21—C21	1.40 (4)
C2—C4	1.47 (3)	C22—C23	1.38 (4)
C3—C5	1.50 (3)	C22—C24	1.48 (3)
S3—C6	1.74 (2)	C23—C25	1.51 (5)
Ta2—Ta1—Ta2	89.85 (4)	Cl3—Ta3—Cl9	82.3 (2)
Ta2—Ta1—Ta3	59.91 (3)	Cl5—Ta3—Cl6	88.6 (2)
Ta2—Ta1—Cl1	95.4 (1)	Cl5—Ta3—Cl9	81.0 (2)
Ta2—Ta1—Cl2	52.7 (2)	Cl6—Ta3—Cl9	82.6 (2)
Ta2—Ta1—Cl4	142.8 (2)	Ta1—Cl1—Ta3	75.0 (2)
Ta2—Ta1—Cl5	95.6 (1)	Ta1—Cl2—Ta2	74.7 (2)
Ta2—Ta1—Cl7	134.3 (1)	Ta2—Cl3—Ta3	74.9 (1)
Ta3—Ta1—Ta3	89.79 (6)	Ta1—Cl4—Ta2	74.6 (2)
Ta3—Ta1—Cl1	142.3 (2)	Ta1—Cl5—Ta3	74.4 (2)
Ta3—Ta1—Cl2	95.6 (2)	Ta2—Cl6—Ta3	75.0 (1)
Ta3—Ta1—Cl4	95.1 (2)	C1—S1—C2	96.3 (9)
Ta3—Ta1—Cl5	52.8 (2)	C1—S1—C3	95.1 (9)
Ta3—Ta1—Cl7	134.3 (1)	S1—C1—S2	116. (1)
Cl1—Ta1—Cl2	89.0 (2)	S1—C1—C1	124. (1)
Cl1—Ta1—Cl4	89.3 (2)	S2—C1—C1	120. (1)
Cl1—Ta1—Cl5	164.9 (3)	S1—C2—C3	115. (2)
Cl1—Ta1—Cl7	83.4 (2)	S1—C2—C4	117. (1)
Cl2—Ta1—Cl4	164.5 (3)	C3—C2—C4	127. (2)
Cl2—Ta1—Cl5	89.4 (2)	S2—C3—C4	117. (2)
Cl2—Ta1—Cl7	81.6 (2)	S2—C3—C5	117. (1)
Cl4—Ta1—Cl5	88.2 (2)	C2—C3—C5	126. (2)
Cl4—Ta1—Cl7	82.9 (2)	C6—S3—C8	97. (1)
Cl5—Ta1—Cl7	81.5 (2)	C6—S4—C9	96.5 (9)
Ta1—Ta2—Ta1	90.15 (3)	C7—S5—C10	95. (1)
Ta1—Ta2—Ta3	59.93 (3)	C7—S6—C11	95. (1)
Ta1—Ta2—Cl2	52.5 (1)	S3—C6—S4	113. (1)
Ta1—Ta2—Cl3	95.6 (1)	S3—C6—C7	124. (2)
Ta1—Ta2—Cl4	142.7 (1)	S4—C6—C7	122. (1)
Ta1—Ta2—Cl6	95.5 (2)	S5—C7—S6	115. (1)
Ta1—Ta2—Cl8	134.3 (2)	S5—C7—C6	122. (2)
Ta3—Ta2—Ta3	89.93 (3)	S6—C7—C6	122. (1)
Ta3—Ta2—Cl2	95.5 (1)	S3—C8—C9	117. (2)
Ta3—Ta2—Cl3	52.5 (1)	S3—C8—C12	118. (2)
Ta3—Ta2—Cl4	95.9 (1)	C9—C8—C12	126. (2)
Ta3—Ta2—Cl6	142.5 (1)	S4—C9—C8	116. (2)
Ta3—Ta2—Cl8	134.2 (1)	S4—C9—C13	114. (1)
Cl2—Ta3—Cl3	89.8 (2)	C8—C9—C13	129. (2)
Cl2—Ta2—Cl4	164.8 (2)	S5—C10—C11	117. (2)
Cl2—Ta2—Cl6	89.0 (2)	S5—C10—C14	116. (2)
Cl2—Ta2—Cl8	81.8 (2)	C10—C11—C10	127. (2)
Cl3—Ta2—Cl4	89.1 (2)	S6—C11—C10	117. (2)
Cl3—Ta3—Cl6	165.0 (2)	S6—C11—C15	116. (1)
Cl3—Ta2—Cl8	81.7 (2)	C10—C11—C15	127. (2)
Cl4—Ta2—Cl6	88.1 (2)	Cl6—S7—C17	95. (1)
Cl4—Ta2—Cl8	83.0 (2)	Cl6—S8—C18	96. (1)
Cl6—Ta2—Cl8	83.3 (2)	S7—Cl6—S8	114. (2)
Ta1—Ta3—Ta1	90.21 (4)	S7—Cl6—Cl6	120. (2)
Ta1—Ta3—Ta2	60.16 (3)	S8—Cl6—Cl6	126. (2)
Ta1—Ta3—Cl1	142.7 (1)	S7—Cl7—C18	119. (2)
Ta1—Ta3—Cl3	95.9 (2)	S7—Cl7—C19	116. (2)
Ta1—Ta3—Cl5	52.8 (1)	Cl8—C17—C19	125. (3)
Ta1—Ta3—Cl6	95.1 (2)	S8—C18—C17	115. (2)
Ta1—Ta3—Cl9	133.8 (2)	S8—C18—C20	116. (2)
Ta2—Ta3—Ta2	90.07 (3)	C17—C18—C20	129. (3)

Table 2 (cont.)

Ta2—Ta3—C11	95.1 (1)	C21—S9—C22	97. (1)
Ta2—Ta3—C13	52.6 (1)	C21—S10—C23	96. (1)
Ta2—Ta3—C15	95.8 (1)	S9—C21—S10	115. (2)
Ta2—Ta3—C16	142.5 (1)	S9—C21—C21	120. (2)
Ta2—Ta3—C19	134.9 (1)	S10—C21—C21	125. (2)
Cl1—Ta3—C13	88.1 (2)	S9—C22—C23	118. (2)
Cl1—Ta3—C15	164.6 (2)	S9—C22—C24	118. (2)
Cl1—Ta3—C16	89.5 (2)	C23—C22—C24	124. (3)
Cl1—Ta3—C19	83.5 (2)	S10—C23—C22	114. (3)
Cl3—Ta3—C15	89.8 (2)	S10—C23—C25	114. (2)
Cl3—Ta3—C16	164.9 (2)	C22—C23—C25	130. (3)

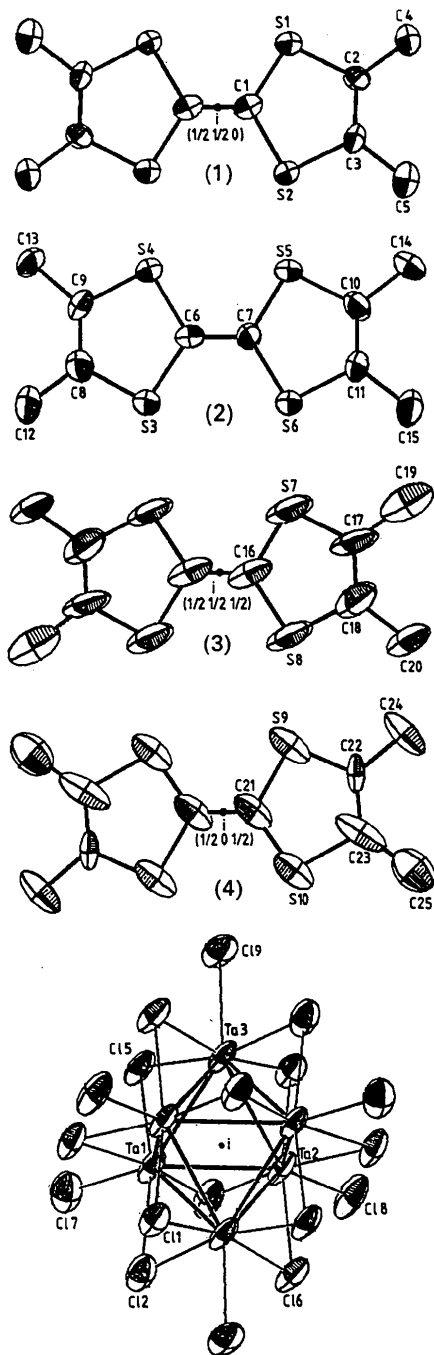


Fig. 1. Atomic numbering.

(1974, Vol. IV). All computer programs from Enraf-Nonius *SDP* described by Frenz (1978). Final atomic parameters are in Table 1,\* bond distances and angles in Table 2. The atomic numbering is shown in Fig. 1 and the crystal structure is presented in Fig. 2.

**Related literature.** The title compound was studied as part of our investigations concerning 'organo-mineral' radical cation based salts, prepared by the electrocrystallization technique. The general formula of these compounds is  $D_xC$ , where  $D$  is an organic TTF derivative (TTF = tetrathiafulvalene) and  $C$  an inorganic hexanuclear metal cluster anion. Previous works have included  $[Mo_6Cl_{14}]^{2-}$  (Ouahab, 1985; Batail & Ouahab, 1985; Ouahab, Batail, Perrin & Garrigou-Lagrange, 1986),  $[Re_6Se_5Cl_9]^-$  (Ouahab, 1985; Batail, Ouahab, Penicaud, Lenoir & Perrin, 1987; Penicaud, 1988),  $[Nb_6Cl_{18}]^{n-}$  (Penicaud *et al.*, 1987; Penicaud, Batail, Coulon, Canadell & Perrin, 1990) and the diamagnetic  $[Ta_6Cl_{18}]^{2-}$  (Slougui, Ouahab, Perrin, Grandjean & Batail, 1989). The present work investigates the crystal structure of  $(TMTTF)_5[Ta_6Cl_{18}] \cdot 0.5CH_2Cl_2$  for which the inorganic anion is paramagnetic (3-) as in the isomorphous niobium salts  $(TMTCF)_5[Nb_6Cl_{18}] \cdot 0.5CH_2Cl_2$ ,  $C = S, Se$  (Penicaud *et al.*, 1987; Penicaud *et al.*, 1990) for which a trivalent  $Nb_6Cl_{18}$  anion was observed and the charges of the TMTTF molecules

\* 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 53950 (27 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

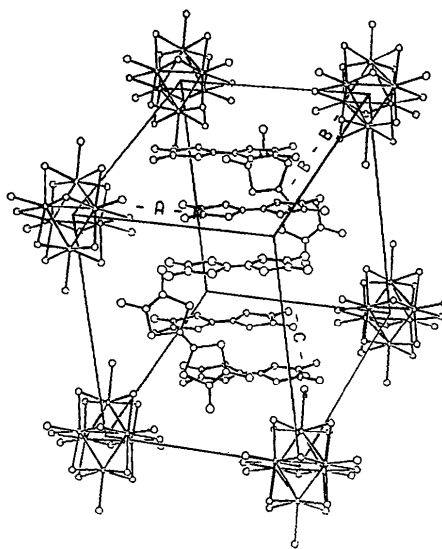


Fig. 2. Projection of the unit-cell contents.

were determined. Moreover, interatomic distances in the  $\text{Ta}_6\text{Cl}_{18}$  unit are in good agreement with the corresponding distances reported for such a unit (Brnićević, Ružić-Toroš, Kojić-Prodić, 1985; Slougui *et al.*, 1989).

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## Lewis-Base Adducts of Group 11 Metal(I) Compounds. 60. Binuclear Adducts of Copper(I) Halides with 2-Hindered Pyridine Bases

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**Abstract.** (I): Di- $\mu$ -bromo-bis[bis(2-bromopyridine)-copper(I)],  $[\text{Cu}_2\text{Br}_2(\text{C}_5\text{H}_4\text{BrN})_4]$ ,  $M_r = 918.9$ , triclinic,  $P\bar{1}$ ,  $a = 10.224(4)$ ,  $b = 8.935(2)$ ,  $c = 7.892(2)$  Å,  $\alpha = 68.84(2)^\circ$ ,  $\beta = 71.96(3)^\circ$ ,  $\gamma = 83.23(3)^\circ$ ,  $V = 639(1)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 2.39$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 109$  cm<sup>-1</sup>,  $F(000) = 864$ ,  $T = 293$  K, final  $R = 0.052$ . (II): Di- $\mu$ -chloro-bis[bis(2-benzylpyridine)copper(I)],  $[\text{Cu}_2\text{Cl}_2(\text{C}_{12}\text{H}_{11}\text{N})_4]$ ,  $M_r = 874.9$ , triclinic,  $P\bar{1}$ ,  $a = 16.441(5)$ ,  $b = 9.183(5)$ ,  $c = 7.661(2)$  Å,  $\alpha = 76.87(4)^\circ$ ,  $\beta = 81.65(3)^\circ$ ,  $\gamma = 73.17(4)^\circ$ ,  $V = 1074(1)$  Å<sup>3</sup>,  $Z = 2$ ,  $D_x = 1.35$  g cm<sup>-3</sup>, Mo  $K\alpha$  radiation,  $\lambda = 0.71069$  Å,  $\mu = 8.1$  cm<sup>-1</sup>,  $F(000) = 452$ ,  $T = 293$  K, final  $R = 0.047$ .

**Experimental.** (I): Prism, dark red–brown, crystal size  $0.44 \times 0.35 \times 0.20$  mm, scintillation detector. Diffraction measurement method:  $2\theta/\theta$ , diffraction temperature 293 K. Absorption correction type: analytical.  $T_{\min}$ ,  $T_{\max}$  not recorded.  $\theta_{\min} = 2.09^\circ$ ,  $\theta_{\max} = 27.47^\circ$ ;  $h$  0→13,  $k$  -11→11,  $l$  -9→10. Six standard reflections, measured every 100 reflections, intensity variation 0%. Criterion for observed reflections:  $I > 3\sigma(I)$ . Full-matrix least-squares refinement, 1909 reflections 'observed' out of 2934 independent measured; number of parameters in least-squares refinement 145,  $wR(\text{all reflections}) = 0.061$ ,

$wR(\text{observed reflections}) = 0.059$ ,  $S(\text{all reflections}) = 2.29$ ,  $S(\text{observed reflections}) = 2.77$ , weights based on measured  $\sigma$ 's;  $(\Delta/\sigma)_{\max} = 0.088$ ,  $(\Delta/\sigma)_{\text{mean}} = 0.004$ ,  $\Delta\rho_{\max} = 0.966$ ,  $\Delta\rho_{\min} = -0.946$  e Å<sup>-3</sup>, no correction for secondary extinction.

(II): Plate, colourless, crystal size  $0.5 \times 0.2 \times 0.03$  mm, scintillation counter. Diffraction measurement method:  $2\theta/\theta$ , diffraction temperature 293 K. Absorption correction type: Gaussian.  $A_{\min}^* = 1.03$ ,  $A_{\max}^* = 1.17$ .  $\theta_{\min} = 1.30^\circ$ ,  $\theta_{\max} = 24.99^\circ$ ;  $h$  0→19,  $k$  -10→10,  $l$  -8→9. Eight standard reflections, measured every 100 reflections, intensity variation 0%. Criterion for observed reflections:  $I > 3\sigma(I)$ . Full-matrix least-squares refinement, 2189 reflections 'observed' out of 3778 independent measured; number of parameters in least-squares refinement 254,  $R(\text{all reflections}) = 0.098$ ,  $wR(\text{all reflections}) = 0.057$ ,  $wR(\text{observed reflections}) = 0.050$ ,  $S(\text{all reflections}) = 1.26$ ,  $S(\text{observed reflections}) = 1.48$ , weights based on measured  $\sigma$ 's;  $(\Delta/\sigma)_{\max} = 0.101$ ,  $(\Delta/\sigma)_{\text{mean}} = 0.012$ ,  $\Delta\rho_{\max} = 0.370$ ,  $\Delta\rho_{\min} = -0.368$  e Å<sup>-3</sup>, Gaussian extinction correction (Zachariasen, 1963), secondary-extinction coefficient = 0.131.

Data collection: Enraf–Nonius CAD-4 software. All calculations and diagrams were performed with the XTAL3.0 package (Hall & Stewart, 1990), as was the