Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) plot showing the anion–cation relationships, the Ni coordination and the atomic numbering scheme.

Díaz & Gómez-Beltrán, 1990*a,b*). The Ni—N distances, 2·106 (6) and 2·099 (6) Å, are typical for pseudo-octahedral coordination and the N—Ni—N bite angle is 83·3 (2)° which is close to the value 82·5 (2)° found for the Ni–2-methyl-1,2-propanediamine pseudo-octahedral complex (García-Granda & Gómez-Beltrán, 1984). It should be pointed out that the differences found in the N—Ni distances is the same effect as observed in the case of the 2-methyl-1,2-propanediamine complex. No unusual geometric features are present in the molecule. Weak hydrogen bonds are observed between the coordinated and uncoordinated water molecules and the bromide anions. The amino group N(2) is hydrogen bonded to the uncoordinated water molecule O(2) though

H(4). Water molecules, coordinated O(1) and uncoordinated O(2), are hydrogen bonded through H(5).

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Structure of the 1:4 Complex of Bis[1,2-oxalato(2 −)]copper(II) and Bis(ethylenedithio)tetrathiafulvalene (BEDT-TTF)

By Minxie Qian, Rainer Rudert and Peter Luger

Institut für Kristallographie, Freie Universität Berlin, Takustrasse 6, D-1000 Berlin 33, Germany

and Chuanjie Ge and Xiaohong Wang

Institute of Chemistry, Academia Sinica Beijing, People's Republic of China

Abstract. 4C₁₀H₈S₈.[Cu(C₂O₄)₂], $M_r = 1778·20$, triclinic, $P\bar{1}$, $a = 11·781$ (2), $b = 15·841$ (3), $c = 8·710$ (2) Å, $\alpha = 91·15$ (2), $\beta = 94·00$ (2), $\gamma = 104·81$ (1)°, $V = 1567$ (1) Å³, $Z = 1$, $D_x = 1·884$ Mg m⁻³, λ(Cu $K\alpha$) = 1·5406 Å, $\mu = 10·45$ mm⁻¹, $F(000) = 901$, $T = 295$ K, $R = 0·069$ ($wR = 0·066$) for 4743 observed reflections. BEDT-TTF molecules form stacks along the c axis. The stacks are dimerized with the interplanar spacings being 3·674 (5) [(I)⋯(II)] and 3·926 (6) Å [(I)⋯(II′)]. The interstack side-by-side S⋯S contact distances are in the range of 3·30 (1) to 3·593 (3) Å.

Introduction. In recent years a family of organic conductors based on BEDT-TTF has been synthesized. It includes the superconductor (BEDT-TTF)₂ReO₄ ($T_c = 2$ K at 0·4 GPa pressure) (Parkin, Engler, Schumaker, Lagier, Lee, Scott & Greene, 1983) and (BEDT-TTF)I₃ ($T_c = 1·5$ K at ambient pressure) (Yagubskii, Shchegolev, Laukhin, Kononovich, Kartsovnik, Zvarykina & Buravov, 1984).

In many known BEDT-TTF salts the acceptor X is an anion with linear, planar, tetrahedral or octahedral geometry, such as I₃ (Leung, Emge, Beno, Wang

& Williams, 1984; Shibaeva, Kaminskii & Yagubskii, 1985; Qian, Wang, Zhu, Zhu, Li, Ma, Duan & Zhang, 1988), IBr_2 (Zhu, Wang, Wan, Yu & Zhu, 1986), AuI_2 (Geiser, Wang, Webb, Firestone, Beno & Williams, 1987), $CuCl_2$ (Kawamoto, Tanaka & Tanaka, 1987), $Ni(dmit)_2$ (dmit = 4,5-dimercapto-1,3-dithiole-2-thione) (Kobayashi, Kato, Kobayashi & Sasaki, 1985), $Pd(dmit)_2$ (Qian, Fu & Zhu, 1984), HSO_4 (Porter, Wang, Miller & Williams, 1987), $InBr_4$ (Beno, Cox, Williams & Kwak, 1984), PF_6 (Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983), AsF_6 (Leung, Beno, Blackman, Coughlin, Miderski, Joss, Crabtree & Williams, 1984).

In this kind of donor–acceptor compound the electrical properties are related to the nature of the constituent species. For example, (BEDT-TTF)$_2$ReO$_4$ and (BEDT-TTF)$_2$ClO$_4$(TCE)$_{0.5}$ (TCE = 1,1,2-trichloroethane) (Kobayashi, Kobayashi, Sasaki, Saito, Enoki & Inokuchi, 1983) are metallic. Both have tetrahedral anions and contain segregated stacks of donor and acceptor molecules and have significant intermolecular S$\cdots$S interactions in two directions, i.e. parallel to and normal to the stacking direction. As a result, they have been characterized as two-dimensional electrical conductors. However, the salts containing octahedral anions, e.g. a- and b-(BEDT-TTF)$_2$PF$_6$ and (BEDT-TTF)$_2$AsF$_6$, are either semiconductors or undergo metal–insulator transitions near ambient temperature. They have a one-dimensional structure generated by short S$\cdots$S contacts normal to the stacking direction. The salts with planar anions, e.g. (BEDT-TTF)[Ni(dmit)$_2$] and (BEDT-TTF)[Pd(dmit)$_2$], contain mixed stacks and are only semiconductors.

As part of an on-going project aimed at exploring structure–conductivity relationships in BEDT-TTF salts, this paper presents a salt containing a planar bivalent anion Cu(C$_2$O$_4$)$_2$.

Experimental. The title compound was prepared in dichloromethane using oxidative electrocrystallization at constant current (5 μA). A black crystal, size 0·4 × 0·3 × 0·6 mm, was mounted on a DEC Micro PDP computer-controlled Stoe four-circle diffractometer. Lattice constants obtained from least-squares refinement of 60 reflections ($35 < 2\theta < 45°$). Intensity data in the hemisphere $h - 17$ to 17, $k - 23$ to 23, l 0 to 13 in the range $5 < 2\theta < 128°$ were measured in the ω–2θ scan mode (Ni-filtered Cu $K\alpha$ radiation). A variable scan range, $\Delta\theta = (1·42 + 0·26\tan\theta)°$, was used with minimum and maximum scan speeds of 0·01 and 0·07° s^{-1}. Three standard reflections measured every 50 reflections showed no significant variation. 5537 reflections, 5157 unique reflections, 794 unobserved reflections [$F < 3\sigma(F)$], $R_{int} = 0·030$. Data corrected for Lorentz–polarization

effects. A numerical correction for absorption was applied; T_{max}/T_{min}: 0·056/0·003.

The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1985). Structure completed by the Fourier difference method and refined subsequently with *XTAL* (Hall & Stewart, 1987). The Cu atom lies on an inversion center. All non-H atoms refined with anisotropic displacement parameters, the methylene groups restrained to tetrahedral symmetry with a C—H bond length of 1·08 Å. All H-atom positions were calculated. The H atoms were refined with isotropic displacement parameters, constrained to one value for each BEDT-TTF unit. Because of large atomic displacement parameters of some atoms in the six-membered rings a disorder was assumed. But treatment of the atoms C(1), C(2), C(9) and C(10) in each BEDT-TTF unit as pairs of separate atomic sites with site populations of 0·5 did not lead to significantly better agreement factors. Therefore, only S(22) and S(28), which were near the largest difference density peaks, were refined as disordered pairs. This resulted in a reduction of the agreement factor wR from 0·073 to 0·066. Atomic scattering factors and dispersion parameters from *International Tables for X-ray Crystallography* (1974, Vol. IV). After convergence, $R = 0·069$ for 4743 observed reflections, $w = 1/\sigma(F)$, 405 refined parameters, $S = 6·6$, $(\Delta/\sigma)_{max} = 0·21$, $(\Delta/\sigma)_{av} = 0·01$, largest peak in final difference map = 1·2, largest hole = $-0·9$ e Å^{-3}.

Discussion. Fractional coordinates are listed in Table 1.* Bond lengths and angles are listed in Tables 2 and 3. The intermolecular S$\cdots$S and S$\cdots$O contact distances that are smaller than the sums of the van der Waals radii (3·6 and 3·3 Å, respectively) are summarized in Table 4. The atomic numbering scheme is given in Fig. 1; the leading digit gives the molecule number. The crystal structure of the title complex consists of one Cu(C$_2$O$_4$)$_2$ and two crystallographically independent BEDT-TTF molecules (I) and (II) (see Figs. 1 and 2).

Complex conformations. The centrosymmetric acceptor molecule Cu(C$_2$O$_4$)$_2$ is slightly nonplanar; a chair conformation is present with an angle of 6·9 (2)° between the CuO$_4$ central plane and the C$_2$O$_4$ planes of the two ligands. The CuO$_4$ chromophore is strictly planar. The Cu—O bond lengths [1·919 (5) and 1·924 (5) Å] are in the same ranges in Na$_2$Cu(C$_2$O$_4$)$_2$.2H$_2$O [Cu—O 1·929 (1) and 1·930 (1) Å] (Gleizes, Maury & Galy, 1980).

Table 1. Atomic parameters (U_{eq} and U_{iso} in $Å^2 \times 10^2$)

	x	y	z	U_{eq}/U_{iso}
BEDT-TTF (I)				
S(11)	0·4964 (2)	0·7468 (2)	0·0596 (4)	9·6 (1)
S(12)	0·2032 (2)	0·7371 (2)	0·1081 (4)	9·8 (1)
S(13)	0·4006 (2)	0·5734 (1)	−0·0925 (3)	6·68 (9)
S(14)	0·1553 (2)	0·5660 (1)	−0·0538 (3)	6·76 (9)
S(15)	0·3213 (2)	0·3846 (1)	−0·2746 (3)	5·88 (8)
S(16)	0·0755 (2)	0·3789 (1)	−0·2361 (3)	6·13 (8)
S(17)	0·2765 (2)	0·2024 (2)	−0·3857 (4)	8·1 (1)
S(18)	−0·0180 (2)	0·1941 (1)	−0·3362 (3)	6·44 (8)
C(11)	0·4379 (8)	0·8294 (6)	0·138 (2)	8·3 (4)
C(12)	0·3247 (9)	0·8136 (7)	0·182 (2)	12·3 (6)
C(13)	0·3736 (7)	0·6641 (5)	0·000 (1)	5·2 (3)
C(14)	0·2597 (7)	0·6595 (5)	0·021 (1)	5·4 (3)
C(15)	0·2548 (6)	0·5153 (4)	−0·126 (1)	4·7 (3)
C(16)	0·2223 (6)	0·4359 (4)	−0·201 (1)	4·7 (3)
C(17)	0·2185 (6)	0·2853 (4)	−0·321 (1)	4·7 (3)
C(18)	0·1044 (7)	0·2822 (4)	−0·302 (1)	4·7 (3)
C(19)	0·1524 (7)	0·1086 (5)	−0·406 (1)	5·5 (3)
C(110)	0·0385 (7)	0·1263 (5)	−0·463 (1)	5·2 (3)
BEDT-TTF (II)				
S(21)	0·2318 (2)	−0·1571 (1)	0·6897 (3)	6·06 (8)
S(22A)	0·525 (2)	−0·150 (1)	0·660 (2)	8·2 (6)
S(22B)	0·526 (2)	−0·142 (1)	0·616 (2)	4·8 (2)
S(23)	0·1883 (2)	−0·3368 (1)	0·5537 (3)	5·35 (7)
S(24)	0·4362 (2)	−0·3265 (1)	0·5196 (3)	5·92 (8)
S(25)	0·1160 (2)	−0·5277 (1)	0·3778 (3)	6·14 (8)
S(26)	0·3640 (2)	−0·5144 (1)	0·3397 (3)	6·21 (8)
S(27)	0·0271 (2)	−0·6984 (1)	0·2174 (4)	7·3 (1)
S(28A)	0·322 (1)	−0·6919 (8)	0·200 (1)	11·0 (5)
S(28B)	0·3171 (7)	−0·6752 (5)	0·141 (1)	5·5 (2)
C(21)	0·3444 (7)	−0·0617 (5)	0·654 (1)	4·9 (3)
C(22)	0·4648 (7)	−0·0638 (5)	0·717 (1)	5·6 (3)
C(23)	0·2891 (6)	−0·2386 (4)	0·6160 (9)	4·0 (2)
C(24)	0·4039 (6)	−0·2329 (4)	0·596 (1)	4·7 (3)
C(25)	0·2901 (6)	−0·3869 (4)	0·4852 (9)	4·2 (2)
C(26)	0·2612 (6)	−0·4661 (4)	0·4115 (9)	4·2 (2)
C(27)	0·1483 (6)	−0·6144 (4)	0·2798 (9)	4·1 (2)
C(28)	0·2621 (6)	−0·6075 (5)	0·260 (1)	5·0 (3)
C(29)	0·0864 (7)	−0·7733 (6)	0·113 (1)	6·6 (3)
C(210)	0·2093 (8)	−0·7754 (6)	0·154 (2)	9·1 (5)
Copper complex				
Cu	0·0	0·0	0·0	3·98 (5)
O(1)	0·3329 (5)	0·0098 (5)	0·0141 (8)	7·9 (3)
O(2)	0·4490 (5)	−0·0600 (4)	0·3086 (6)	5·6 (2)
O(3)	0·1481 (4)	−0·0014 (4)	−0·0775 (6)	5·5 (2)
O(4)	0·0804 (4)	0·0116 (3)	0·2023 (6)	4·9 (2)
C(31)	0·2281 (6)	0·0032 (5)	0·036 (1)	4·7 (3)
C(32)	0·1871 (6)	0·0025 (4)	0·1965 (9)	4·1 (2)

Table 2. Bond distances (Å) with e.s.d.'s in parentheses

(a) BEDT-TTF

	(I)	(II)		(I)	(II)
S(1)—C(1)	1·77 (1)	1·786 (7)	S(6)—C(8)	1·745 (8)	1·743 (7)
S(1)—C(3)	1·725 (7)	1·734 (8)	S(7)—C(7)	1·730 (8)	1·731 (6)
S(2A)—C(2)	1·70 (1)	1·76 (2)	S(7)—C(9)	1·794 (7)	1·79 (1)
S(2B)—C(2)	–	1·83 (2)	S(8A)—C(8)	1·736 (7)	1·75 (2)
S(2A)—C(4)	1·726 (9)	1·73 (2)	S(8B)—C(8)	–	1·74 (1)
S(2B)—C(4)	–	1·76 (2)	S(8A)—C(10)	1·798 (9)	1·64 (1)
S(3)—C(3)	1·743 (9)	1·749 (6)	S(8B)—C(10)	–	1·77 (1)
S(3)—C(5)	1·730 (7)	1·730 (8)	C(1)—C(2)	1·38 (2)	1·49 (1)
S(4)—C(4)	1·747 (7)	1·750 (8)	C(3)—C(4)	1·35 (1)	1·36 (1)
S(4)—C(5)	1·726 (9)	1·744 (6)	C(5)—C(6)	1·36 (1)	1·35 (1)
S(5)—C(6)	1·733 (8)	1·741 (6)	C(7)—C(8)	1·35 (1)	1·34 (1)
S(5)—C(7)	1·743 (6)	1·740 (8)	C(9)—C(10)	1·49 (1)	1·47 (1)
S(6)—C(6)	1·738 (7)	1·733 (8)			

(b) $Cu(C_2O_4)_2$

Cu—O(3)	1·919 (5)		Cu—O(4)	1·924 (5)
O(1)—C(31)	1·239 (9)		O(2)—C(32)	1·208 (9)
O(3)—C(31)	1·304 (9)		O(4)—C(32)	1·306 (9)
C(31)—C(32)	1·51 (1)			

Table 3. Valence angles (°) with e.s.d.'s in parentheses

(a) BEDT-TTF

	(I)	(II)		(I)	(II)
C(1)—S(1)—C(3)	104·0 (4)	101·1 (4)	S(2B)—C(4)—C(3)		129·7 (9)
C(2)—S(2A)—C(4)	103·8 (5)	105 (1)	S(4)—C(4)—C(3)	116·9 (6)	116·7 (5)
C(2)—S(2B)—C(4)		101 (1)	S(3)—C(5)—S(4)	114·4 (4)	114·4 (4)
C(3)—S(3)—C(5)	96·5 (4)	96·4 (3)	S(3)—C(5)—C(6)	122·4 (6)	123·8 (5)
C(4)—S(4)—C(5)	96·1 (4)	95·9 (3)	S(4)—C(5)—C(6)	123·1 (6)	121·8 (6)
C(6)—S(5)—C(7)	95·6 (3)	96·0 (3)	S(5)—C(6)—S(6)	114·4 (4)	114·3 (4)
C(6)—S(6)—C(8)	95·5 (4)	95·8 (3)	S(5)—C(6)—C(5)	123·6 (5)	122·4 (6)
C(7)—S(7)—C(9)	103·9 (4)	104·5 (4)	S(6)—C(6)—C(5)	122·0 (6)	123·3 (5)
C(8)—S(8A)—C(10)	100·7 (4)	105·9 (8)	S(5)—C(7)—S(7)	115·1 (4)	114·9 (6)
C(8)—S(8B)—C(10)		100·3 (6)	S(5)—C(7)—C(8)	116·7 (6)	116·7 (5)
S(1)—C(1)—C(2)	123·0 (7)	114·1 (6)	S(7)—C(7)—C(8)	128·2 (5)	128·4 (6)
S(2A)—C(2)—C(1)	128 (1)	119·8 (8)	S(6)—C(8)—S(8A)	115·4 (5)	115·3 (5)
S(2B)—C(2)—C(1)		114·1 (8)	S(6)—C(8)—S(8B)		116·0 (5)
S(1)—C(3)—S(3)	115·8 (5)	116·9 (4)	S(6)—C(8)—C(7)	116·5 (5)	117·1 (6)
S(1)—C(3)—C(4)	128·1 (7)	126·7 (5)	S(8A)—C(8)—C(7)	128·1 (6)	126·0 (6)
S(3)—C(3)—C(4)	116·0 (5)	116·3 (6)	S(8B)—C(8)—C(7)		126·2 (6)
S(2A)—C(4)—S(4)	115·3 (5)	115·3 (9)	S(7)—C(9)—C(10)	115·3 (6)	118·5 (7)
S(2B)—C(4)—S(4)		113·3 (8)	S(8A)—C(10)—C(9)	115·2 (6)	127·1 (9)
S(2A)—C(4)—C(3)	127·8 (6)	127 (1)	S(8B)—C(10)—C(9)		115·8 (7)

(b) $Cu(C_2O_4)_2$

O(3)—Cu—O(4)	86·6 (2)		O(3)—Cu—O(4')	93·4 (2)
Cu—O(3)—C(31)	110·5 (5)		Cu—O(4)—C(32)	111·1 (5)
O(1)—C(31)—O(3)	122·2 (8)		O(1)—C(31)—C(32)	121·3 (7)
O(3)—C(31)—C(32)	116·4 (6)		O(2)—C(32)—O(4)	123·7 (7)
O(2)—C(32)—C(31)	112·0 (7)		O(4)—C(32)—C(31)	114·3 (6)

Table 4. Selected intermolecular contacts (Å)

(a) BEDT-TTF and BEDT-TTF

S(28B)···S(11)ⁱ	3·30 (1)
S(28B)···S(13)ⁱ	3·366 (8)
S(12)···S(18)ⁱⁱ	3·410 (4)
S(28A)···S(11)ⁱ	3·47 (1)
S(22B)···S(17)ⁱ	3·47 (2)
S(27)···S(21)ⁱⁱⁱ	3·471 (3)
S(27)···S(23)ⁱⁱⁱ	3·480 (3)
S(28A)···S(13)ⁱ	3·53 (1)
S(12)···S(16)ⁱⁱ	3·593 (3)

(b) BEDT-TTF and $Cu(C_2O_4)_2$

O(2)···S(22B)ⁱᵛ	3·07 (2)
O(2)···S(22A)ⁱᵛ	3·13 (2)

(i) $1 - x, -y, -z$; (ii) $-x, 1 - y, -z$; (iii) $-x, -1 - y, 1 - z$; (iv) $1 - x, -y, 1 - z$.

In the two donor BEDT-TTF molecules [(I) and (II)], the central TTF groups are planar with an average deviation of 0·007 (5) Å for the contributing atoms S(3), S(4), S(5), S(6), C(5) and C(6). The outer parts of the BEDT-TTF molecules are bent away from the short interplanar spacings [Fig. 2(b)]. The maximum deviations of the S atoms in the six-membered rings from the TTF least-squares plane are 0·576 (5) Å for S(18) of (I) and 0·55 (2) Å for S(22B) of (II). The C atoms comprising the outer six-membered rings of the BEDT-TTF donors are displaced slightly above and below the plane defined by the atoms of the central TTF fragment. The unusually large thermal ellipsoids of the C atoms and S atoms in the six-membered rings indicate disordered atomic sites which are common to BEDT-TTF salts (Leung, Beno, Blackman, Coughlin, Miderski, Joss, Crabtree & Williams, 1984; Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983). The disorder of the two outer C atoms in each of the four six-membered rings can be approximated by two pairs of C atoms being 0·38 (3) [C(21)] to 0·78 (3) Å [C(12)] apart. S(22) and S(28) form disordered pairs with atoms being 0·41 (3) and 0·59 (2) Å apart. The S—C bond lengths of the outer
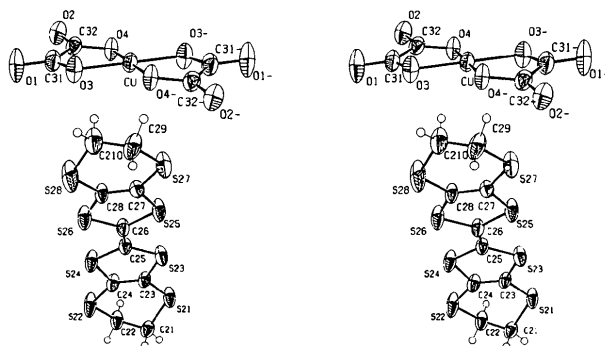
Fig. 1. An *ORTEP* (Johnson, 1976) stereo drawing of BEDT-TTF (II) and Cu(C₂O₄)₂.

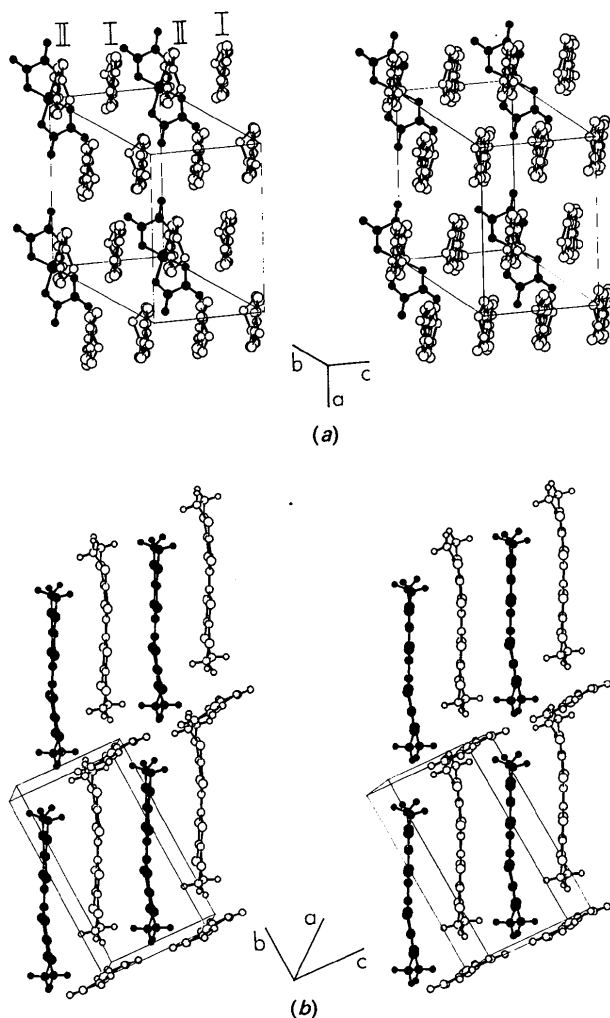six-membered rings are in a large range from 1·64 (1) to 1·83 (2) Å, which has also been observed in (BEDT-TTF)₂InBr₄ (S—C bonds in the range 1·74 to 1·90 Å).

Complex packing. The BEDT-TTF molecules form a face-to-face stack along the c axis [see Fig. 2(a)]. The stack is dimerized, *i.e.* the molecules form pairs with short interplanar distances with respect to the TTF planes [3·674 (5) Å] which are separated from the neighbouring pairs by a larger distance [3·926 (6) Å]. Within the BEDT-TTF stack the molecules are shifted along the long molecular axes. The shift between the molecules in the dimer is 0·24 Å; the shift between the adjacent molecules of different dimers is 3·74 Å. There are short interstack side-by-side S···S contact distances along [20$\bar{1}$] between all stacks, the shortest being 3·30 (1) Å (see Fig. 3 and Table 4). No short contact distances between S atoms are observed along the molecular stacking direction (out-of-plane contacts).

The sheets of planar Cu anions separate the BEDT-TTF stacks, as shown in Fig. 2(b). The shortest non-bonding contact between Cu(C₂O₄)₂ and

(a)

(b)

Fig. 2. (a) *SCHAKAL* (Keller, 1986) stereoview of the BEDT-TTF network (white spheres) and adjacent anion sheet (black spheres) down the molecular axes of the BEDT-TTF cations. (b) Stereoview perpendicular to the BEDT-TTF molecular axes. The black spheres represent molecule (I), the white spheres represent molecule (II) and the anions.
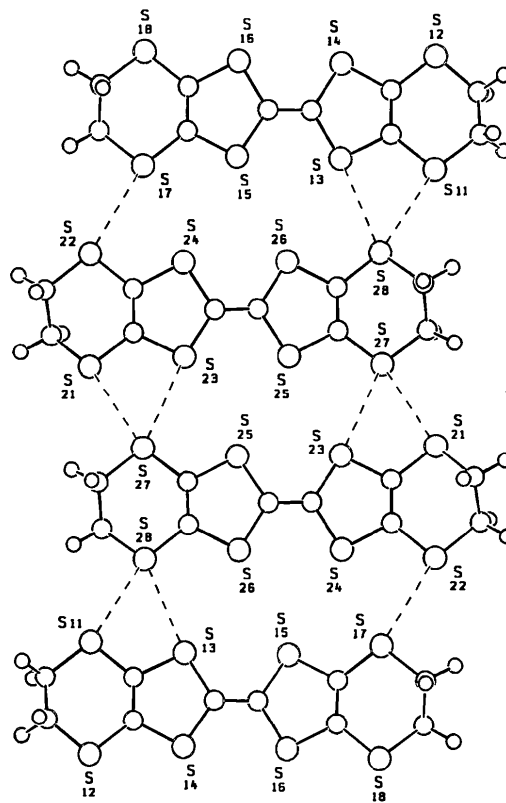

Fig. 3. A *SCHAKAL* drawing of the BEDT-TTF packing in the *ab* plane. The dashed lines show S···S contacts which are less than the sum of the van der Waals radii (3·60 Å).

BEDT-TTF is 3·07 (2) Å for O(2)···S(22B) (see also Table 4).

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Structure of Dicarbonyl(η5-cyclopentadienyl)[(diethyldithiocarbamato)mercurio](trimethyl phosphite)molybdenum(II)(Mo–Hg)

By MAREK M. KUBICKI* AND PHILIPPE RICHARD

Laboratoire de Synthèse et d'Electrosynthèse Organométalliques associé au CNRS (URA 33), Faculté des Sciences, 6 bd Gabriel 21000 Dijon, France

AND MICHEL JANNIN

Laboratoire de Physique du Solide associé au CNRS (URA 785), Faculté des Sciences Mirande, 21000 Dijon, France

Abstract. $[(C_{10}H_{14}O_5P)MoHg(C_5H_{10}NS_2)]$, $M_r = 689.99$, monoclinic, $P2_1/n$, $a = 8.853 (1)$, $b = 9.873 (2)$, $c = 25.231 (4)$ Å, $\beta = 97.09 (1)°$, $V = 2188.2$ Å^3, $Z = 4$, $D_x = 2.094$ Mg m^{-3}, $\lambda(Mo\ K\alpha) = 0.71073$ Å, $\mu = 7.277$ mm^{-1}, $F(000) = 1320$, $T = 296 (1)$ K, final $R = 0.029$ for 2676 observed reflections and 235 parameters. The structure is built of discrete molecules without the intermolecular Hg···S interactions which are generally observed in the crystal structures of compounds containing Hg and S atoms. The Mo—Hg bond length is 2·680 (1) Å and the Mo—Hg—S unit deviates slightly from linearity [170·47 (6)°]. A large Hg—Mo—P angle [131·50 (5)°] is observed.

Introduction. For some years we have been interested in systematic qualitative NMR studies of metallic nuclei in carbonyl cyclopentadienyl complexes involving the molybdenum—mercury bond (Kubicki, Kergoat, Le Gall, Guerchais, Douglade & Mercier,

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* To whom correspondence should be addressed.