None of the benzene ring distances and angles show anomalous values; the latter lie within 1 and 5°, respectively, of the 120° expected for trigonal planar coordinated atoms. The relatively small angle O—C1—C2 (115·7°) in Me_3NTPH, if compared to its homologue in $[H_4NTPH]^+$ (117·4°), can be explained in terms of the enhanced steric requirements of a methyl group (C7) compared to those of a proton. The small angle C1—C2—N (115·3°) in H_4NTPH^+ could be due to an attractive interaction of the proton H4 with the phenolic O atoms (O1, O2, O3; mean contact distance H4···O of 2·27 Å). Such an interaction is absent in Me_3NTPH (angle C1—C2—N of 119·8°). Finally we mention the hydrogen bridges between the phenolic O atoms of H_4NTPH^+ and the SO_3 group of the benzenesulfonate anion, mediated by H1, H2 and H3.

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First Unsymmetrical Donor Which Gives Organic Superconductors: DMET

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Abstract. 2-(4,5-Dimethyldiselenol-2-ylidene)dithiolo[4,5-*b*]dihydrodithiin, $C_{10}H_{10}S_4Se_2$, $M_r = 416{\cdot}34$, monoclinic, $P2_1/c$, $a = 6{\cdot}724\,(1)$, $b = 12{\cdot}144\,(3)$, $c = 18{\cdot}228\,(3)$ Å, $\beta = 111{\cdot}21\,(1)°$, $V = 1387{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}99$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71068$ Å, $\mu = 5{\cdot}84$ mm^{-1}, $F(000) = 808$, $T = 297$ K, $R = 0{\cdot}058$ for 1816 reflections. The molecule is bent at the inner and the outer S atoms in contrast to the planar conformation in the partially oxidized states in organic conductors $(DMET)_2X$ (X = monovalent anion).

Introduction. The title compound DMET is an unsymmetrical hybrid donor between TMTSF and BEDT–TTF, and forms radical salts of the type $(DMET)_2X$ with various monovalent anions X. The electrical properties of $(DMET)_2X$ have been investigated for a few years by the present authors (Kikuchi, Ikemoto & Kobayashi, 1987; Murata *et al.*, 1988; Kikuchi, Saito, Ikemoto, Murata, Ishiguro & Kobayashi, 1988). In the course of the study, some superconductors were discovered in 1987 (Kikuchi, Kikuchi *et al.*, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Kobayashi, Ishiguro & Ikemoto, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Ishiguro, Kobayashi & Ikemoto, 1987; Kikuchi, Murata, Honda, Namiki, Saito, Anzai, Kobayashi, Ishiguro & Ikemoto, 1987; Kikuchi, Honda *et al.*, 1988). Thus, $(DMET)_2X$ is the first group of organic superconductors based on an unsymmetrical donor. From

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Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å^2)

$$B_{eq} = \tfrac{4}{3}(B_{11}\mathbf{a}.\mathbf{a} + B_{12}\mathbf{a}.\mathbf{b} + B_{13}\mathbf{a}.\mathbf{c} + \ldots).$$

	x	*y*	*z*	B_{eq}
Se(1)	7715 (1)	4685 (1)	7049 (1)	3·47 (3)
Se(2)	2659 (1)	4561 (1)	6436 (1)	3·42 (3)
S(1)	7243 (4)	6435 (2)	5593 (1)	3·1 (1)
S(2)	2564 (4)	6382 (2)	5038 (1)	3·2 (1)
S(3)	7476 (5)	8521 (3)	4801 (2)	5·6 (1)
S(4)	1989 (5)	8444 (2)	4097 (2)	5·0 (1)
C(1)	5090 (12)	5186 (7)	6315 (5)	2·6 (2)
C(2)	4991 (12)	5914 (7)	5746 (5)	2·5 (2)
C(3)	6402 (15)	3829 (8)	7616 (5)	3·3 (3)
C(4)	4244 (14)	3764 (7)	7358 (5)	3·1 (3)
C(5)	5873 (14)	7560 (7)	5017 (5)	3·1 (3)
C(6)	3726 (15)	7529 (7)	4768 (5)	3·4 (3)
C(7)	7958 (17)	3195 (10)	8318 (6)	5·7 (4)
C(8)	2945 (16)	3061 (9)	7707 (6)	5·0 (4)
C(9)	5655 (22)	9166 (15)	3921 (9)	10·8 (7)
C(10)	3671 (22)	8840 (15)	3546 (8)	9·2 (6)

the point of view of developing new organic superconductors, it is desirable to clarify the relation between oxidized and neutral states of the donor molecule. The molecule of BEDT–TTF, for example, is of boat form in the neutral state (Kobayashi, Kobayashi, Sasaki, Saito & Inokuchi, 1986) and nearly planar in the partially oxidized states (Kobayashi, Kato, Mori, Kobayashi, Sasaki, Saito & Inokuchi, 1983; Shibaeva, Kaminskii & Yagubskii, 1985). On the other hand, that of TMTSF is planar in both the neutral and the oxidized states (Kistenmacher, Emge, Shu & Cowan, 1979; Thorup, Rindorf, Soling & Bechgaard, 1981; Rindorf, Soling & Thorup, 1982). In this paper, the crystal structure of DMET is reported. The molecular geometry of neutral DMET is compared with those of neutral TMTSF and BEDT–TTF, and those of partially oxidized DMET.

Experimental. Crystals of DMET prepared by recrystallization from chloroform solution. D_m not determined. A red needle-like crystal 0·1 × 0·1 × 0·3 mm. Rigaku AFC-M automated four-circle diffractometer. Unit-cell dimensions determined from 20 selected reflections ($27 < 2\theta < 31°$). Intensity data collected using the ω–2θ scan technique ($\Delta\omega = 1{\cdot}00° + 0{\cdot}14° \tan\theta$) with a scan rate 4° min^{-1} in ω to $(\sin\theta)/\lambda = 0{\cdot}65$ Å^{-1} ($-8 \leq h \leq 8$, $0 \leq k \leq 15$, $0 \leq l \leq 23$). Three standard reflections measured at an interval of 100 reflections, small (<2%) random variations. No absorption correction. 3439 reflections collected; 1816 reflections [$|F_o| > 3\sigma(|F_o|)$] used in the structure refinement. Structure solved by direct methods with *SHELXS86* (Sheldrick, 1986) and refined by the block-diagonal least-squares method. Atomic and anomalous-scattering factors from *International Tables for X-ray Crystallography* (1974). Computations carried out using the *UNICSIII* program (Sakurai & Kobayashi, 1979) except for structure solution. H atoms not located. $\sum w(|F_o| - |F_c|)^2$ minimized, where $w = [\sigma^2(|F_o|) + 0{\cdot}0002|F_o|^2]^{-1}$, with $\sigma(|F_o|)$ based on counting statistics. $\Delta/\sigma = 0{\cdot}06$ in final least-squares cycle which resulted in the agreement factors $R = 0{\cdot}058$, $wR = 0{\cdot}054$ and $S = 2{\cdot}13$. No correction for secondary extinction. A difference synthesis based on the structure factors derived from the final parameter values showed some peaks of density ($-0{\cdot}8$ to $0{\cdot}9$ e Å^{-3}). *ORTEPII* (Johnson, 1971) was used to produce crystal structure illustrations.

Discussion. Final positional and thermal parameters are tabulated in Table 1.* The space group of the crystal, $P2_1/c$, is widely observed for crystals of plane-like molecules. The asymmetry of the molecule, however, requires 'pairing' of molecules for the fulfillment of the symmetry as shown in Fig. 1. The paired molecules are related by an inversion center, resulting in cancellation of the molecular electric-dipole moments. The herringbone packing of 'pairs' may be attributable to the interaction between the electric-quadrupole moments. The distance between two paired molecules is nearly equal to the sum of van der Waals radii of S and Se atoms. The molecules shown in Fig. 1 lie at almost the same height (*x* coordinate): The crystal can be described as consisting of sheets of molecules as clearly seen in Fig. 2. The intersheet distance is also determined by van der Waals radii of S and Se atoms. In most $(\mathrm{DMET})_2X$

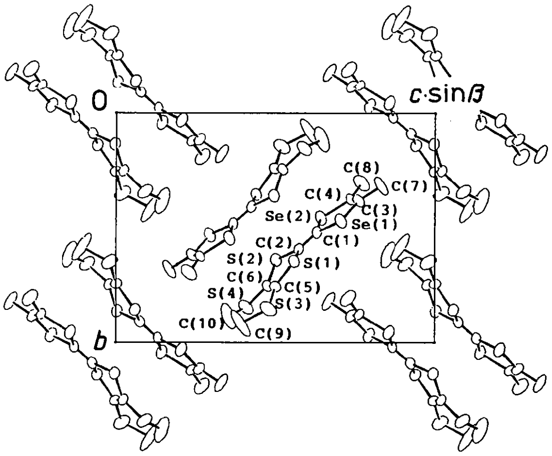

Fig. 1. Crystal structure viewed along the *a* axis. The thermal ellipsoids indicate the region of 50% probability. The numbering scheme is also given.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51913 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

crystals, the molecules of DMET form a column which is a conduction path in the crystal (Kikuchi, Ikemoto & Kobayashi, 1987; Kikuchi, Saito *et al.*, 1988; Kikuchi, Ishikawa, Saito, Ikemoto & Kobayashi, 1988*a*,*b*; Ishikawa, Kikuchi, Saito, Ikemoto & Kobayashi, 1989). This clearly shows that the stability of columnar structures results mainly from the energy gain of conduction electrons through formation of the partially filled band (delocalization).

The bond lengths and angles are given in Table 2. They are in reasonable agreement with the corresponding values of neutral TMTSF (Kistenmacher, Emge, Shu & Cowan, 1979) and BEDT–TTF (Kobayashi, Kobayashi, Sasaki, Saito & Inokuchi, 1986) molecules except for the ethylenedithio group. In the $(DMET)_2X$ crystals so far studied crystallographically [$X = PF_6$, BF_4, $Au(CN)_2$, AuI_2, $AuBr_2$, $AuCl_2$, IBr_2 (Kikuchi, Ikemoto & Kobayashi, 1987; Kikuchi, Saito *et al.*, 1988; Kikuchi, Ishikawa *et al.*, 1988*a*,*b*; Ishikawa *et al.*, 1989) and I_3 (Aldoshina, Atovmyan, Gol'denberg, Krasochka, Lyubovskaya, Lyubovskii & Khidekel', 1986)], $DMET^{+1/2}$ is almost planar (within 4°), irrespective of the crystal structure. The neutral molecule is bent at the inner and the outer S atoms. The dihedral angle between the planes determined by two Se, two inner S, C(1) and C(2) atoms and by four S, C(5) and C(6) atoms is 18·0°. The angle between the latter and that formed by S(3), S(4) and C(9) is 24·0°. These are the most marked differences in the molecular structure between the neutral and the partially oxidized states. The same kind of difference was reported for BEDT–TTF (Kobayashi, Kobayashi *et al.*, 1986; Kobayashi, Kato *et al.*, 1983; Shibaeva, Kaminskii & Yagubskii, 1985). Although the bond lengths and angles in principle reflect the change in the electronic state, the rather large e.s.d.'s prevent decisive comparison of them. However, it seems that the geometry of one part of the half of TMTSF is essentially the same in both states and that of the other part different. A little change in TMTSF (Kistenmacher, Emge, Shu & Cowan, 1979; Thorup, Rindorf, Soling & Bechgaard, 1981; Rindorf, Soling & Thorup, 1982) may be responsible for that in the TMTSF part of DMET.

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

Se(1)—C(1)	1·888 (9)	S(3)—C(9)	1·806 (19)
Se(1)—C(3)	1·893 (10)	S(4)—C(6)	1·750 (11)
Se(2)—C(1)	1·885 (9)	S(4)—C(10)	1·829 (18)
Se(2)—C(4)	1·897 (10)	C(1)—C(2)	1·348 (12)
S(1)—C(2)	1·754 (9)	C(3)—C(4)	1·356 (14)
S(1)—C(5)	1·766 (10)	C(3)—C(7)	1·535 (16)
S(2)—C(2)	1·768 (9)	C(4)—C(8)	1·516 (15)
S(2)—C(6)	1·752 (11)	C(5)—C(6)	1·348 (14)
S(3)—C(5)	1·728 (10)	C(9)—C(10)	1·321 (26)

C(1)—Se(1)—C(3)	93·6 (4)	Se(1)—C(3)—C(7)	114·8 (7)
C(1)—Se(2)—C(4)	94·4 (4)	C(4)—C(3)—C(7)	125·5 (9)
C(2)—S(1)—C(5)	94·5 (4)	Se(2)—C(4)—C(3)	117·7 (7)
C(2)—S(2)—C(6)	94·2 (5)	Se(2)—C(4)—C(8)	115·8 (7)
C(5)—S(3)—C(9)	102·5 (7)	C(3)—C(4)—C(8)	126·4 (9)
C(6)—S(4)—C(10)	99·3 (7)	S(1)—C(5)—S(3)	115·1 (6)
Se(1)—C(1)—Se(2)	114·5 (5)	S(1)—C(5)—C(6)	116·3 (8)
Se(1)—C(1)—C(2)	122·0 (7)	S(3)—C(5)—C(6)	128·5 (8)
Se(2)—C(1)—C(2)	123·5 (7)	S(2)—C(6)—S(4)	116·6 (6)
S(1)—C(2)—S(2)	113·0 (5)	S(2)—C(6)—C(5)	117·4 (8)
S(1)—C(2)—C(1)	123·7 (7)	S(4)—C(6)—C(5)	125·5 (8)
S(2)—C(2)—C(1)	123·3 (7)	S(3)—C(9)—C(10)	124·3 (15)
Se(1)—C(3)—C(4)	119·6 (7)	S(4)—C(10)—C(9)	120·2 (14)

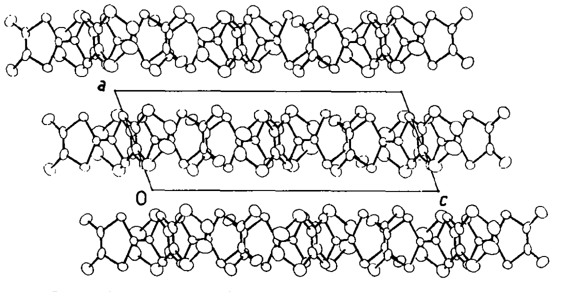

Fig. 2. Crystal structure viewed along the *b* axis. The thermal ellipsoids indicate the region of 50% probability.

The large thermal parameters of C(9) and C(10) suggest some disorder. The Fourier synthesis, however, gave only one peak for each atom. An activation energy of 15 kJ mol^{-1} was reported for the reorientational motion of the ethylenedithio group in BEDT–TTF salts (Tokiwa *et al.*, 1988). The large thermal parameters are, therefore, truly due to large thermal oscillation.

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Structure of (−)-Malic Acid

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Abstract. $C_4H_6O_5$, $M_r = 134{\cdot}09$, monoclinic, $P2_1$, $a = 5{\cdot}041$ (1), $b = 9{\cdot}188$ (3), $c = 11{\cdot}792$ (4) Å, $\beta = 94{\cdot}06$ (2)°, $V = 544{\cdot}8$ (3) Å^3, $Z = 4$, $D_x = 1{\cdot}635$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, μ(Mo $K\alpha$) = 0·15 mm^{-1}, $F(000) = 280$, $T = 294$ K, $R = 0{\cdot}064$ for 667 unique observed diffractometer data [$I > \sigma(I)$], $wR = 0{\cdot}071$. The carbon chains of the two independent molecules are in the extended form. The molecules form infinite chains being coupled *via* cyclic hydrogen-bonded carboxyl dimers. The chains are linked together in a sheet by hydrogen-bond interactions between aliphatic hydroxyl groups.

Introduction. "Die gewöhnliche links drehende Apfelsäure = COOH.CH(OH)CH$_2$.COOH krystallisiert nur schwierig in zerfließlichen Nadeln, von derem Form bisher keine brauchbare Bestimmung vorliegt" (Groth, 1910). Van Loock, van Havere & Lenstra (1981) confirm this problem in a paper in which the structure of α-(±)-malic acid is presented. We, however, were able by careful evaporation of an aqueous solution at room temperature to grow crystals of (−)-malic acid by means of which it was possible to elucidate the heavy-atom structure, the results of which are presented here.

Experimental. Crystal 0·2 × 0·2 × 0·2 mm, Enraf–Nonius CAD-4F diffractometer. Zr-filtered Mo $K\alpha$ radiation, unit cell determined using 23 reflexions ($7 < \theta < 10$°). In one half of the reflexion sphere (h −5,5; k 0,10; l −16,16) 1757 reflexions measured, $\theta_{\max} = 23{\cdot}5$°, $R_{\rm int} = 0{\cdot}063$ for 864 independent reflexions, 667 unique observed reflexions [$I > \sigma(I)$, $\sigma^2(I) = \sigma^2(I)_{\rm cs} + (pI)^2$ with $p = 0{\cdot}0063$ (McCandlish, Stout & Andrews, 1975)]. Intensities obtained by the $\omega/2\theta$-scan method with a scan width of $(1{\cdot}40 + 0{\cdot}35\tan\theta)$°. For each reflection the scan speed was adjusted in order to obtain a minimum $I/\sigma(I)$ ratio of 100, provided that the scan time did not exceed 60 s. The 020 reflexion was used as an intensity and orientation check; decay during data collection 2·4%. Instability and Lp correction applied; no correction for absorption. Systematic absences $0k0$ with $k = 2n + 1$, indicating $P2_1$.

Applying direct methods using *MULTAN*80 (Main, Woolfson, Lessinger, Germain & Declercq, 1980) with a statistically weighted tangent refinement revealed, amongst several ghost peaks, the position of one cyclic carboxyl dimer. Using these six atoms, four subsequent difference Fourier syntheses (*SHELX*76, Sheldrick, 1976) resulted in the positions of all non-H atoms. After full-matrix isotropic and full-matrix anisotropic refinement a difference Fourier was calculated and unambiguous positions were found for the aliphatic H atoms, but no accurate positions could be assigned to the hydroxyl

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