for 292 variables. Maximum shift/e.s.d. in final cycle was 0.63 and no residual electron density $>|0.385|$ e Å^{-3} was found. Calculations were carried out on a PDP11/34 computer with *SDP* (B. A. Frenz & Associates, Inc., 1982). Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B).

The fractional coordinates and thermal parameters for non-H atoms are listed in Table 1* and selected bond lengths and angles in Table 2. A perspective drawing of the molecule is shown in Fig. 1 and the packing diagram in Fig. 2.

Related literature. This is the first crystal structure of a 1,4,2-diazaphospholidine derivative to be reported. The planarity of the five-membered ring is in good agreement with results reported by Hutton, Modro, Niven & Scaillet (1986) for 1,3,2-diazaphospholidine-2,4,5-trione. Similar five-membered rings with envelope and half-chair conformations have been reported by Prange, Pascard, Devillers & Navech (1977) and Marre, Sanchez, Wolf, Jaud & Galy (1984). P=O, P—N and P—C bond lengths compare well with values reported by Perales & Garcia-Blanco (1977) and Marre, Sanchez, Wolf, Jaud & Galy (1984).

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* Lists of structure factor amplitudes, anisotropic displacement parameters for non-H atoms, H-atom parameters, C—H and N—H bond lengths, and calculations of least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54714 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0242]

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Structure of a Dibenzo[*d*,*d*′]benzo[1,2-*b*:4,3-*b*′]dithiophene (DBBDT) Complex with 7,7,8,8-Tetracyano-*p*-quinodimethane (TCNQ)*

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Abstract. $C_{18}H_{10}S_2.C_{12}H_4N_4$, $M_r = 494.59$, monoclinic, $P2_1/c$, $a = 16.159$ (7), $b = 17.958$ (2), $c = 16.159$ (7) Å, $\beta = 96.41$ (2)°, $V = 4660$ (3) Å^3, $Z = 8$, $D_x = 1.410$ g cm^{-3}, λ(Mo $K\alpha$) = 0.71073 Å, $\mu = 2.45$ cm^{-1}, $F(000) = 2032$, $T = 294$ K, $R = 0.067$ for 2241 observed reflections. The asymmetric unit contains two non-planar DBBDT molecules and two planar TCNQ molecules. The crystal structure consists of stacks along the *a* axis of alternating DBBDT and TCNQ molecules.

Experimental. Crystals of DBBDT–TCNQ were produced by dissolving equal amounts of DBBDT and TCNQ (gradient sublimed) in hot acetonitrile followed by slow cooling. A single crystal having the form of a prism elongated along [100] with dimensions 0.33 × 0.10 × 0.13 mm was selected for data collection using an Enraf–Nonius CAD-4 diffractometer with graphite monochromator. Unit-cell

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* IUPAC name for TCNQ is 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile.

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dimensions were derived from 25 reflections (four settings of each) having $8 < \theta < 13°$. X-ray intensity data were measured in ω-scan mode with $\Delta\omega = (1.00 + 0.35\tan\theta)°$ within the range $0 < \theta < 25°$, $0 \leq h \leq 19$, $0 \leq k \leq 21$, $-19 \leq l \leq 19$. No significant decay was observed for the three intensity control reflections. The intensities were corrected for Lorentz, polarization and absorption effects (transmission in the range 0.97–0.98). A total of 8815 reflections were measured, and after removal of systematic absences they were merged to a set of 8199 unique reflections, $R_{\rm int}(F) = 0.028$, and 2241 of these having $I > 3\sigma(I)$ were later used in the structure refinement. The systematic absences were consistent with space group $P2_1/c$ (see below). The structure was solved by direct methods using *SHELXS*86 (Sheldrick, 1990), and for refinement *SHELX*76 (Sheldrick, 1976) was used. The final model with 309 parameters had anisotropic temperature factors for S and isotropic temperature factors for C and N atoms. Assigning anisotropic thermal parameters to all non-H atoms was not justifiable considering the number of observations. H atoms were included in calculated positions with a C—H distance of 1.08 Å. They were kept riding on the parent C atoms with fixed $U_{\rm iso} = 0.08$ Å^2. The function minimized was $\sum w(|F_o| - |F_c|)^2$ with $w = 1/[\sigma^2_{\rm count}(F_o) + 0.0004|F_o|^2]$. The final agreement factors were $R(F) = 0.067$, $wR(F) = 0.069$ and $S = 1.85$. Complex atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV). Maximum (Δ/σ) in final cycle was 0.02 and maximum and minimum residual electron densities were 0.44 and -0.30 e Å^{-3}. The resultant positional and thermal parameters are given in Table 1. For molecular geometry calculations *PLATON* (Spek, 1990) was used, and *PLUTO* (Motherwell & Clegg, 1978) was used for crystal structure illustrations.

The unit-cell parameters actually suggest an orthorhombic *C*-centered lattice. Axial photographs as well as merging of possibly equivalent reflections, however, confirmed the absence of symmetry higher than monoclinic. Furthermore, examination of the refined structure model with the program *MISSYM* (Le Page, 1988) indicated no additional symmetry. We are thus dealing with a case where the metric symmetry fortuitously exceeds the crystal symmetry.

Table 1. *Fractional coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* (Å$^2 \times 10^3$) *with e.s.d.'s in parentheses*

$$U_{\rm eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
S(1)	5574 (2)	979 (2)	403 (2)	50 (1)
S(2)	4634 (2)	277 (2)	3927 (2)	53 (1)
C(1)	5662 (7)	1898 (6)	723 (6)	43 (3)
C(2)	5943 (8)	2480 (6)	240 (7)	55 (3)
C(3)	6078 (7)	3157 (6)	617 (7)	56 (3)
C(4)	5955 (7)	3266 (6)	1441 (6)	50 (3)
C(5)	5664 (7)	2704 (5)	1915 (6)	43 (3)
C(6)	5470 (6)	2001 (5)	1540 (6)	33 (3)
C(7)	5216 (6)	1312 (5)	1900 (5)	27 (2)
C(8)	4930 (6)	1159 (6)	2670 (6)	34 (3)
C(9)	4600 (6)	1629 (5)	3300 (6)	39 (3)
C(10)	4344 (7)	2372 (5)	3261 (6)	46 (3)
C(11)	4010 (7)	2696 (6)	3917 (6)	52 (3)
C(12)	3928 (7)	2278 (6)	4629 (7)	62 (3)
C(13)	4100 (7)	1552 (6)	4685 (7)	55 (3)
C(14)	4443 (7)	1222 (6)	4008 (6)	45 (3)
C(15)	4918 (6)	422 (5)	2923 (6)	38 (3)
C(16)	5074 (6)	−178 (6)	2395 (6)	39 (3)
C(17)	5237 (6)	−30 (5)	1610 (6)	41 (3)
C(18)	5315 (6)	709 (5)	1380 (5)	31 (2)
S(3)	742 (2)	1629 (2)	151 (2)	75 (1)
S(4)	−366 (2)	2166 (2)	3623 (3)	88 (2)
C(19)	832 (7)	698 (6)	426 (7)	51 (3)
C(20)	1121 (7)	161 (6)	−61 (7)	58 (3)
C(21)	1224 (7)	−547 (6)	247 (7)	61 (3)
C(22)	1071 (7)	−705 (6)	1069 (6)	49 (3)
C(23)	785 (7)	−154 (6)	1562 (6)	46 (3)
C(24)	616 (6)	568 (6)	1227 (6)	41 (3)
C(25)	311 (7)	1222 (6)	1627 (6)	46 (3)
C(26)	−13 (7)	1336 (7)	2384 (7)	53 (3)
C(27)	−296 (7)	838 (6)	2978 (7)	53 (3)
C(28)	−515 (6)	80 (5)	2930 (6)	47 (3)
C(29)	−806 (8)	−285 (7)	3587 (7)	67 (4)
C(30)	−896 (8)	94 (7)	4340 (8)	72 (4)
C(31)	−737 (8)	844 (7)	4349 (8)	70 (4)
C(32)	−477 (8)	1217 (7)	3697 (8)	71 (4)
C(33)	−70 (7)	2105 (7)	2632 (7)	62 (4)
C(34)	61 (7)	2683 (7)	2105 (7)	68 (4)
C(35)	259 (8)	2572 (7)	1352 (8)	74 (4)
C(36)	407 (8)	1844 (7)	1075 (7)	65 (4)
N(1)	8076 (6)	2326 (5)	−110 (6)	72 (3)
N(2)	8182 (7)	3758 (6)	1976 (6)	76 (3)
N(3)	7065 (6)	242 (5)	5009 (6)	67 (3)
N(4)	6839 (7)	−1262 (6)	2955 (6)	76 (3)
C(37)	7997 (7)	2371 (6)	587 (7)	49 (3)
C(38)	8047 (7)	3163 (6)	1739 (6)	53 (3)
C(39)	7891 (7)	2431 (6)	1431 (6)	41 (3)
C(40)	7659 (7)	1862 (5)	1917 (6)	37 (3)
C(41)	7555 (7)	1977 (6)	2777 (6)	46 (3)
C(42)	7371 (6)	1413 (5)	3265 (6)	42 (3)
C(43)	7261 (7)	673 (5)	2940 (6)	42 (3)
C(44)	7108 (7)	85 (6)	3455 (6)	47 (3)
C(45)	7062 (7)	175 (6)	4306 (7)	52 (3)
C(46)	6982 (7)	−659 (6)	3173 (7)	57 (3)
C(47)	7342 (6)	565 (6)	2069 (6)	44 (3)
C(48)	7528 (6)	1131 (5)	1573 (6)	42 (3)
N(5)	1749 (7)	1630 (6)	5017 (7)	90 (4)
N(6)	1696 (7)	3505 (6)	3324 (6)	79 (3)
N(7)	3284 (6)	671 (5)	−277 (6)	73 (3)
N(8)	3324 (6)	−1181 (5)	1459 (5)	64 (3)
C(49)	1838 (7)	1847 (6)	4370 (7)	61 (3)
C(50)	1832 (7)	2888 (7)	3442 (6)	55 (3)
C(51)	1976 (7)	2113 (6)	3567 (6)	45 (3)
C(52)	2249 (7)	1645 (6)	2983 (6)	46 (3)
C(53)	2396 (6)	1922 (6)	2184 (6)	45 (3)
C(54)	2649 (6)	1472 (5)	1603 (6)	41 (3)
C(55)	2794 (6)	682 (5)	1780 (6)	36 (3)
C(56)	3051 (7)	219 (6)	1185 (6)	43 (3)
C(57)	3176 (7)	465 (6)	362 (7)	52 (3)
C(58)	3180 (7)	−566 (6)	1337 (6)	45 (3)
C(59)	2645 (6)	415 (6)	2583 (6)	45 (3)
C(60)	2396 (7)	871 (6)	3166 (6)	49 (3)

The atomic numbering of the four unique molecules is shown in Fig. 1. Selected bond lengths and bond angles are given in Table 2 whereas full lists have been deposited.* Bond lengths in the two DBBDT molecules are very similar and close to the expected C—C and C—S distances. The DBBDT molecules are non-planar since planarity would imply unreasonably short contacts between the two

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* Lists of anisotropic thermal parameters, H-atom parameters, bond distances, bond angles and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54785 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AB0246]

H atoms attached to C(5) and C(10) as well as the two H atoms attached to C(23) and C(28). For each DBBDT molecule the two outer benzene rings are tilted in opposite directions from the central plane of the remaining atoms with tilt angles in the range 12.4 to 16.9°. All DBBDT and TCNQ molecules are stacked alternately along the *a* axis of the structure as shown in Fig. 2, which also illustrates the conformations of the constituent molecules.

Related literature. DBBDT was prepared as part of our program to develop new organic materials with non-linear optical properties. TCNQ is probably the most widely used acceptor for preparation of charge-transfer complexes. Structural aspects of a large number of such complexes are thoroughly discussed in the classic review by Herbstein (1971). The exact molecular geometry of TCNQ in a particular complex is related to the formal charge of TCNQ (Flandrois & Chasseau, 1977). Bond lengths in the present study are not accurate enough for a detailed analysis. The title structure belongs to the majority of TCNQ complexes, which have a mixed stack crystal structure and exhibit poor electrical conductivity.

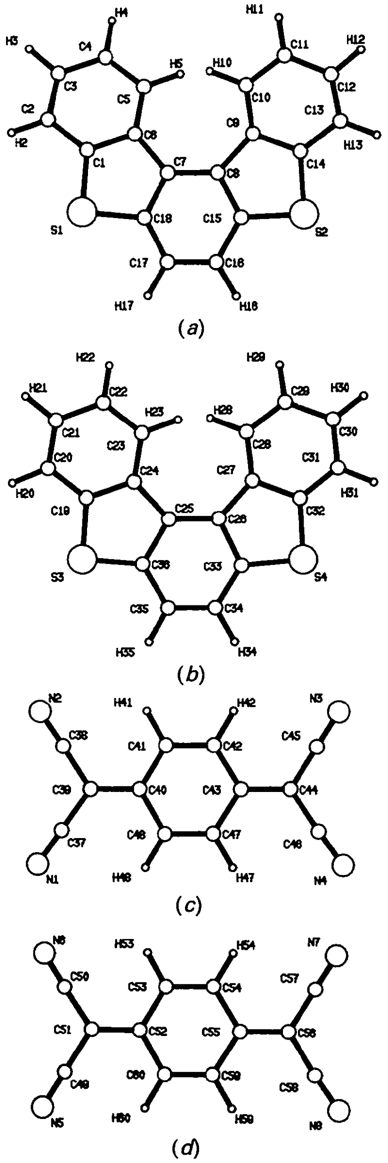

Fig. 1. View perpendicular to (central) molecular planes with atomic numbering scheme.

Table 2. *Selected bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

S(1)—C(1)	1.731 (11)	S(3)—C(19)	1.732 (12)
C(1)—C(6)	1.403 (13)	C(19)—C(24)	1.398 (13)
C(6)—C(7)	1.447 (12)	C(24)—C(25)	1.453 (14)
C(7)—C(18)	1.391 (12)	C(25)—C(36)	1.449 (14)
S(1)—C(18)	1.746 (10)	S(3)—C(36)	1.688 (12)
S(2)—C(14)	1.733 (11)	S(4)—C(32)	1.720 (14)
C(14)—C(9)	1.404 (13)	C(32)—C(27)	1.404 (15)
C(8)—C(9)	1.469 (13)	C(26)—C(27)	1.423 (14)
C(8)—C(15)	1.385 (14)	C(26)—C(33)	1.444 (16)
S(2)—C(15)	1.754 (10)	S(4)—C(33)	1.726 (12)
C(1)—S(1)—C(18)	90.9 (5)	C(19)—S(3)—C(36)	91.1 (6)
S(1)—C(1)—C(6)	112.8 (7)	S(3)—C(19)—C(24)	112.1 (8)
C(1)—C(6)—C(7)	111.6 (8)	C(19)—C(24)—C(25)	114.2 (9)
C(6)—C(7)—C(18)	111.5 (7)	C(24)—C(25)—C(36)	106.5 (9)
S(1)—C(18)—C(7)	112.7 (6)	S(3)—C(36)—C(25)	115.9 (8)
C(14)—S(2)—C(15)	89.5 (5)	C(32)—S(4)—C(33)	92.5 (6)
S(2)—C(14)—C(9)	113.5 (7)	S(4)—C(32)—C(27)	112.9 (9)
C(14)—C(9)—C(8)	112.2 (9)	C(26)—C(27)—C(32)	111.5 (10)
C(9)—C(8)—C(15)	109.1 (8)	C(27)—C(26)—C(33)	112.3 (10)
S(2)—C(15)—C(8)	115.4 (7)	S(4)—C(33)—C(26)	110.5 (9)

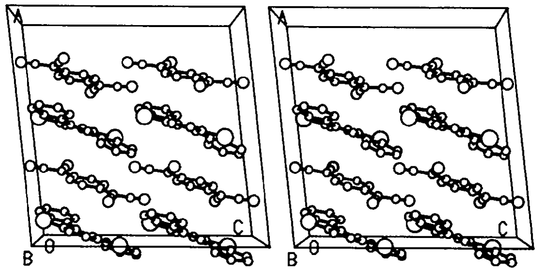

Fig. 2. Stereoview of molecular stacks. H atoms are omitted for clarity.

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