ment forts entre atomes d'hydrogène et d'oxygène des deux molécules centrosymétriquement liées. Un dessin d'un des dimères ainsi formés est porté sur la Fig. 3. Les distances H(171)···O(18) sont toutes deux égales à 2,11 Å. Les distances N(17)···O(18) valent 2,929 Å et les angles N(17)—H(171)···O(18) sont de 174°. La conséquence de la formation de dimères par liaisons hydrogène est de rendre plan deux à deux les groupements carboxamides en vis-à-vis et centrosymétriquement liés. Le plan moyen *C'* des deux groupements carboxamides d'un des dimères est porté dans le Tableau 4.

La cohésion cristalline est assurée par des contacts de van der Waals entre dimères, du type carbone–hydrogène et oxygène–hydrogène dont les plus courts sont respectivement 2,80 et 2,63 Å.

L'empilement moléculaire s'effectue perpendiculairement à l'axe *b* (Fig. 2).

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The Structure of the 1:1 Molecular Complex of Dibenzothiophene and 7,7,8,8-Tetracyanoquinodimethane

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Abstract

$C_{12}H_8S.C_{12}H_4N_4$, $M_r = 388{\cdot}4$, is triclinic, space group $P\bar{1}$, with $a = 16{\cdot}316\,(4)$, $b = 8{\cdot}910\,(2)$, $c = 6{\cdot}839\,(3)$ Å, $\alpha = 98{\cdot}35\,(3)$, $\beta = 98{\cdot}66\,(4)$, $\gamma = 100{\cdot}13\,(2)°$, $Z = 2$, $D_m = 1{\cdot}357$ (by flotation), $D_c = 1{\cdot}355$ Mg m^{-3}. The structure was refined to $R = 0{\cdot}082$ for 2112 counter reflections. Alternate planar dibenzothiophene and tetracyanoquinodimethane (TCNQ) molecules, with dimensions similar to those from previous determinations, stack along **c** with the molecular planes nearly parallel to each other and perpendicular to **c**. The mean perpendicular spacings and relative orientations of dibenzothiophene and TCNQ molecules above and below are very similar. The relative orientations are not those maximizing charge-transfer interactions, and the separations (3·43 and 3·40 Å) are the largest reported for triclinic TCNQ complexes. All peripheral atoms of TCNQ but not all those of dibenzothiophene are in van der Waals contact with atoms of other molecules, and in-plane motion is larger for dibenzothiophene than TCNQ. Dibenzofuran/TCNQ has a closely related unit cell with the *a* axis halved, requiring disordered $P\bar{1}$ or ordered $P1$ structures. For dibenzothiophene/TCNQ, but not for dibenzofuran/TCNQ, such structures involve abnormally short contacts between the donor hetero atom and an N atom of TCNQ. These compounds form a continuous series of mixed-molecular complexes.

Introduction

As part of a study of crystalline mixed-molecular complexes we have prepared the crystalline TCNQ complexes of dibenzofuran and dibenzothiophene and a series of mixed complexes (dibenzofuran)$_x$(dibenzothiophene)$_{1-x}$TCNQ ($0 < x < 1$). We have determined the crystal structure of the dibenzothiophene/TCNQ complex to aid interpretation of spectroscopic and electrical-conductivity data, and to investigate the origin of the different cell dimensions of these complexes involving donor molecules of similar shapes, which are as follows at 293 K: Dibenzofuran/TCNQ ($Z = 1$): $a = 8{\cdot}010\,(2)$, $b = 8{\cdot}997\,(2)$, $c = 6{\cdot}780\,(1)$ Å, $\alpha = 97{\cdot}75\,(2)$, $\beta = 100{\cdot}68\,(2)$, $\gamma = 99{\cdot}58\,(3)°$, $U = 466{\cdot}5$ Å^3. Dibenzothiophene/TCNQ ($Z = 2$): $a = 16{\cdot}316\,(4)$, $b = 8{\cdot}910\,(2)$, $c = 6{\cdot}839\,(3)$ Å, $\alpha =$

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98·35 (3), β = 98·66 (4), γ = 100·13 (2)°, U = 952·4 Å^3.

Although the crystal structures of dibenzofuran (Dideberg, Dupont & André, 1972; Banerjee, 1973) and dibenzothiophene (Schaffrin & Trotter, 1970) have been reported, no structural data are available for their molecular complexes.

Experimental

The complex, which was in the form of black crystals elongated along **c**, was obtained by slow evaporation of a solution of equimolar quantities of the components in acetonitrile. A General Electric XRD-6 manual diffractometer with Cu $K\alpha$ radiation, was used to obtain cell parameters, and the intensities of 2385 independent reflections ($2\theta < 110°$) were measured by a stationary-crystal–stationary-counter technique, converted to equivalent integrated values and corrected for background and polarization. Those with $|F_o| < 3\sigma(F_o)$ were regarded as unobserved, and the remaining 2112 reflections were used in the structure determination. No absorption correction was applied [μ(Cu $K\alpha$) = 1·61 mm^{-1}].

Structure solution and refinement

The structure was solved by Patterson and Fourier methods analogous to those used by Tickle & Prout (1973) for the triclinic acenaphthene/TCNQ complex. The plane and orientation of the TCNQ molecule were deduced from the extensive isometric array of peaks with 1·4 Å spacing around the origin of the three-dimensional sharpened Patterson synthesis, and its centroid was deduced from the large 16-fold vector at (0·520, −0·011, 0·048) included in the intermolecular vectors between two TCNQ molecules related by a centre of symmetry and defining the centroid-to-centroid separation. An F_o synthesis with phases calculated from the TCNQ coordinates revealed the dibenzothiophene molecule. Structure factors calculated from this model, with the overall scale and temperature factors calculated from a Wilson plot, produced an initial R of 0·341. Full-matrix least-squares refinement of coordinates and individual isotropic temperature factors, with unit weights, converged with R = 0·161. A difference Fourier synthesis revealed peaks in the positions expected for H atoms, and these were placed geometrically, with C—H 1·00 Å, to lie in the relevant molecular plane along the bisector of the appropriate C—C—C angle, and assigned isotropic temperature factors of 0·05 Å^2. Further refinement used a large-block approximation to the normal matrix, with five blocks as follows: (1) scale and dummy overall temperature factors, (2) TCNQ coordinates, (3) dibenzothiophene coordinates, (4) TCNQ temperature factors, (5) dibenzothiophene temperature factors. Refinement of all C, N and S coordinates and anisotropic temperature factors converged with R = 0·090. Following an agreement analysis by ranges of F_o, the two most intense reflections were considered to be suffering from secondary extinction and were given zero weight, while the 70 reflections with $F_o > 250$ were given weights $w = 250/F_o$. Refinement of all positional and thermal parameters (C, N, S anisotropic, H isotropic) converged with R = 0·080 but led to unreasonable H-atom positions and temperature factors. H atoms were therefore repositioned geometrically, with isotropic temperature factors equal to those of the C atoms to which they were attached. Final refinement of coordinates and anisotropic temperature factors of C, N and S atoms converged with R = 0·082. The average and maximum parameter shifts in the final cycle of refinement were 0·03 and 0·60σ respectively. All calculations used the Oxford *CRYSTALS* program (Carruthers, 1975) on the University of London CDC 7600 computer, with scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974).

Results and discussion

Atomic coordinates and isotropic temperature factors are given in Table 1, and bond distances and angles in Table 2.† There are no significant differences between chemically equivalent bond lengths or angles in the two molecules or between these parameters and those reported for dibenzothiophene (Schaffrin & Trotter, 1970) and TCNQ (Long, Sparks & Trueblood, 1965).

The structure is shown in Fig. 1 projected onto the least-squares best plane through the dibenzothiophene molecule. The crystal is built up of stacks along **c** of alternating donor and acceptor molecules characteristic of π–π^* electron donor–acceptor complexes. The dibenzothiophene molecule as a whole is close to planar (maximum deviation 0·017 Å). However, better planes (within 0·006 Å) can be fitted to the three individual rings of the molecule, and the angles between the normals to the five-membered ring and the six-membered rings are 0·4 and 0·7°, similar to those found in the structure of pure dibenzothiophene. Similarly, the maximum deviation from the least-squares best plane through the whole TCNQ molecule is 0·034 Å, but the central ring is very accurately planar (within 0·002 Å), and planes through the two —C(CN)$_2$ groups (±0·007 Å) make angles of 0·4 and

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† Lists of structure factors and thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36160 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^3$; $U_{iso} \times 10^4$ *for* H)

$$U_{eq} = (U_{11}\,U_{22}\,U_{33})^{1/3}.$$

	x	y	z	U_{eq} (Å^2)
C(1)	3230 (3)	6327 (6)	822 (8)	39 (3)
C(2)	3431 (3)	4805 (6)	570 (9)	42 (3)
C(3)	2801 (4)	3510 (6)	67 (9)	44 (4)
C(4)	1933 (4)	3659 (6)	−206 (8)	41 (3)
C(5)	1736 (4)	5180 (7)	57 (9)	45 (4)
C(6)	2354 (4)	6453 (6)	545 (9)	42 (4)
C(7)	3867 (4)	7608 (6)	1349 (9)	45 (4)
C(8)	1288 (4)	2347 (7)	−738 (9)	44 (4)
C(9)	3706 (4)	9160 (7)	1623 (10)	50 (4)
C(10)	4750 (4)	7523 (7)	1619 (10)	50 (4)
C(11)	1464 (4)	823 (7)	−1046 (10)	48 (4)
C(12)	417 (4)	2457 (7)	−1005 (9)	51 (4)
N(1)	3609 (4)	10415 (6)	1888 (11)	68 (4)
N(2)	5447 (4)	7476 (6)	1863 (10)	66 (4)
N(3)	1598 (4)	−377 (6)	−1316 (10)	69 (4)
N(4)	−279 (4)	2546 (7)	−1199 (10)	74 (4)
C(13)	4024 (5)	8267 (8)	6548 (11)	63 (5)
C(14)	3184 (5)	8218 (7)	6243 (10)	62 (5)
C(15)	2640 (4)	6750 (7)	5714 (9)	48 (4)
C(16)	2963 (4)	5385 (7)	5532 (9)	45 (4)
C(17)	3838 (4)	5484 (8)	5857 (9)	53 (4)
C(18)	4363 (4)	6950 (9)	6375 (11)	62 (5)
C(19)	859 (5)	1719 (11)	3883 (11)	74 (6)
C(20)	758 (4)	3205 (10)	4182 (11)	66 (5)
C(21)	1498 (4)	4366 (8)	4754 (9)	52 (4)
C(22)	2300 (4)	3999 (7)	4964 (9)	47 (4)
C(23)	2381 (4)	2475 (7)	4655 (10)	58 (4)
C(24)	1648 (5)	1324 (9)	4098 (11)	69 (5)
S(1)	1538 (1)	6370 (2)	5241 (3)	66 (1)
H(1)	4034	4688	755	348
H(2)	2947	2463	−105	392
H(3)	1133	5291	−125	396
H(4)	2207	7488	740	399
H(5)	4416	9289	6910	602
H(6)	2962	9187	6396	618
H(7)	4083	4539	5714	504
H(8)	4988	7048	6640	592
H(9)	346	883	3499	743
H(10)	184	3457	3996	636
H(11)	2947	2190	4830	546
H(12)	1691	217	3850	698

1·5° with the central-ring plane. These small twists of the ends of the molecule may arise from lattice-packing effects due to the slight asymmetry in the environment of the TCNQ molecule. The normal to the least-squares best plane through the whole dibenzothiophene molecule is only 1·9° away from the *c* axis, and the best planes through the donor and acceptor molecules are only 0·7° away from parallel.

In $P\bar{1}$ with two molecules per cell in general positions, the orientation and separation of TCNQ molecules above and below a given dibenzothiophene molecule need not be the same. In this case the mean interplanar spacings are nearly equal (3·40 and 3·43 Å) and the relative orientations of donor and acceptor are almost the same (symmetrical in one case and slightly displaced along the long axis of the

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

(*a*) TCNQ

C(1)–C(2)	1·443 (7)	C(7)–C(9)	1·443 (8)
C(2)–C(3)	1·367 (8)	C(7)–C(10)	1·442 (8)
C(3)–C(4)	1·432 (8)	C(8)–C(11)	1·429 (8)
C(4)–C(5)	1·439 (8)	C(8)–C(12)	1·427 (8)
C(5)–C(6)	1·343 (8)	C(9)–N(1)	1·149 (7)
C(6)–C(1)	1·440 (8)	C(10)–N(2)	1·133 (7)
C(1)–C(7)	1·365 (8)	C(11)–N(3)	1·124 (7)
C(4)–C(8)	1·391 (8)	C(12)–N(4)	1·143 (8)

C(2)–C(1)–C(6)	118·7 (5)	C(1)–C(7)–C(9)	122·4 (5)
C(1)–C(2)–C(3)	120·5 (5)	C(1)–C(7)–C(10)	122·9 (5)
C(2)–C(3)–C(4)	119·9 (5)	C(4)–C(8)–C(11)	121·7 (5)
C(3)–C(4)–C(5)	119·3 (5)	C(4)–C(8)–C(12)	121·6 (5)
C(4)–C(5)–C(6)	120·8 (5)	C(9)–C(7)–C(10)	114·7 (5)
C(5)–C(6)–C(1)	120·6 (5)	C(11)–C(8)–C(12)	116·7 (5)
C(6)–C(1)–C(7)	121·4 (5)	C(7)–C(9)–N(1)	177·3 (7)
C(2)–C(1)–C(7)	119·8 (5)	C(7)–C(10)–N(2)	178·8 (7)
C(3)–C(4)–C(8)	120·3 (5)	C(8)–C(11)–N(3)	179·0 (8)
C(5)–C(4)–C(8)	120·4 (5)	C(8)–C(12)–N(4)	179·3 (7)

(*b*) Dibenzothiophene

C(13)–C(14)	1·346 (10)	C(19)–C(20)	1·353 (11)
C(13)–C(18)	1·381 (10)	C(19)–C(24)	1·386 (11)
C(14)–C(15)	1·409 (9)	C(20)–C(21)	1·408 (9)
C(15)–C(16)	1·406 (8)	C(21)–C(22)	1·395 (8)
C(15)–S(1)	1·741 (6)	C(21)–S(1)	1·756 (7)
C(16)–C(17)	1·396 (8)	C(22)–C(23)	1·377 (9)
C(16)–C(22)	1·452 (8)	C(23)–C(24)	1·395 (9)
C(17)–C(18)	1·395 (9)		

C(14)–C(13)–C(18)	122·7 (6)	C(19)–C(20)–C(21)	117·0 (7)
C(13)–C(14)–C(15)	117·7 (6)	C(20)–C(21)–C(22)	121·4 (7)
C(14)–C(15)–C(16)	121·1 (6)	C(20)–C(21)–S(1)	125·9 (5)
C(14)–C(15)–S(1)	126·8 (5)	C(22)–C(21)–S(1)	112·7 (5)
C(16)–C(15)–S(1)	112·1 (5)	C(15)–S(1)–C(21)	91·2 (6)
C(15)–C(16)–C(17)	119·4 (6)	C(21)–C(22)–C(23)	120·1 (6)
C(15)–C(16)–C(22)	112·5 (5)	C(21)–C(22)–C(16)	111·4 (6)
C(17)–C(16)–C(22)	128·0 (6)	C(16)–C(22)–C(23)	128·5 (6)
C(16)–C(17)–C(18)	118·4 (6)	C(22)–C(23)–C(24)	118·5 (7)
C(17)–C(18)–C(13)	120·6 (6)	C(23)–C(24)–C(19)	120·3 (7)
C(20)–C(19)–C(24)	122·6 (7)		

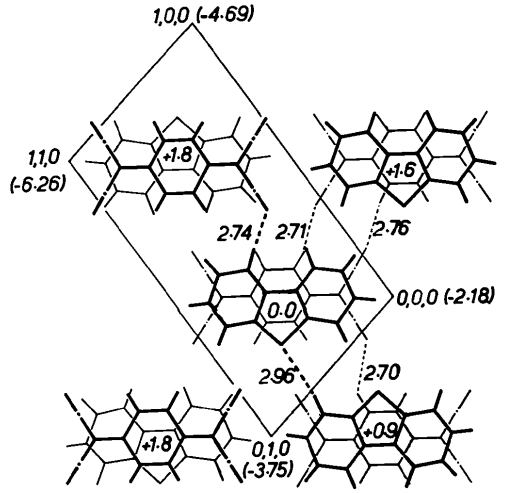

Fig. 1. Projection of the structure on the plane of the dibenzothiophene molecule at *x,y,z*. The figures inside the more heavily drawn molecules and at the corners of the projected cell denote heights in Å above and below the plane of this dibenzothiophene molecule.

molecules in the other). The longer spacing corresponds to the displaced orientation, and may reflect poorer charge-transfer overlap in this orientation, although the effect is very small and could equally be ascribed to differences in intermolecular repulsive forces in the two slightly different orientations. These mean separations of donor and acceptor planes are the longest reported for TCNQ complexes having triclinic lattices, and fit the previously noted correlation between *D–A* separation and donor ionization potential (Munnoch & Wright, 1974), consistent with only weak charge-transfer interaction [the ionization potential of dibenzothiophene is 8·14 eV (Mukherjee, 1969)]. The relative orientation of donor and acceptor is very similar to that observed in the carbazole/TCNQ complex (Kobayashi, 1973) but different from that observed in the *o*-phenanthroline/TCNQ complex (Goldberg & Shmueli, 1977), in which the long axis of the TCNQ molecule is rotated approximately 22° with respect to that of the donor. Semi-empirical calculations (Goldberg, 1975) suggest that the latter orientation is close to the one most favourable to charge-transfer interactions. Although no similar calculations have been carried out for dibenzothiophene/TCNQ, the symmetries of the seven occupied orbitals of the donor only permit significant overlap between the second and sixth highest occupied orbitals and the lowest vacant orbital of TCNQ in the observed orientation. The fact that this orientation places the polar quinoid double bonds of TCNQ over the centres of polarizable rings of the donor molecule, together with favourable lattice packing, is thus more likely than charge-transfer interactions to be the orientation-determining factor in this complex.

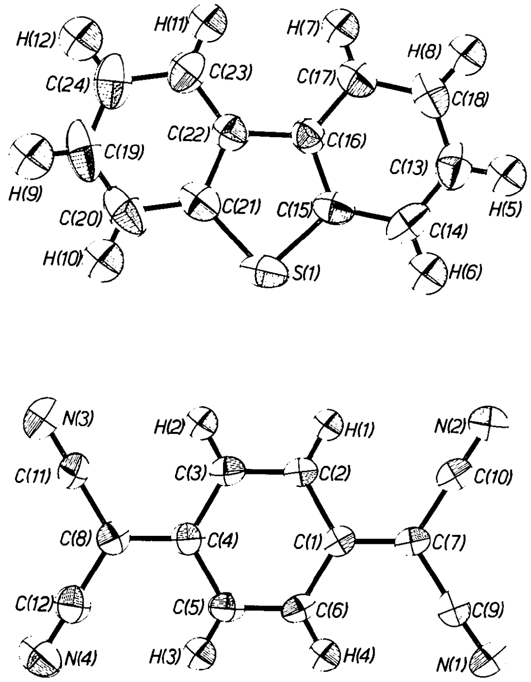

Fig. 2. Thermal ellipsoids for dibenzothiophene and TCNQ, at the 50% probability level, projected onto the molecular planes (Johnson, 1965).

All the peripheral atoms of the TCNQ molecule are in contact with N or H atoms of neighbouring TCNQ molecules. These N···H contacts range from 2·70–2·77 Å, close to the sum of the van der Waals radii of N and H (2·70 Å). In contrast, only two of the peripheral atoms of dibenzothiophene are in van der Waals contact with atoms of neighbouring molecules [S(1)···H(10) at $(-x, 1-y, 1-z)$, 2·96 Å, *cf.* van der Waals radius sum 3·05 Å; and H(7)···N(2) at $(1-x, 1-y, 1-z)$, 2·74 Å], and this lack of close contacts accounts for the larger in-plane motion of dibenzothiophene compared to TCNQ, apparent from the thermal ellipsoid plots of Fig. 2. Many other molecular complexes show larger in-plane motion of the donor than of the acceptor (Fyfe, 1973), suggesting that lattice packing may often be determined by interactions between electron-rich and electron-deficient regions of neighbouring acceptor molecules, as well as by the desire to attain a relative orientation of donor and acceptor within the stacks which optimizes or nearly optimizes charge-transfer interactions. Apart from the above mentioned S(1)···H(10) contact, there are no intermolecular contacts shorter than the sum of the van der Waals radii of the atoms involved. However, twofold rotation of the dibenzothiophene molecule about its long axis would lead to an abnormally short contact (2·76 Å) between S(1) and N(2) at $(1-x, 1-y, 1-z)$ (*cf.* sum of van der Waals radii of N and S, 3·35 Å). This accounts for the absence of disorder at the donor site and the fact that the space group is $P\bar{1}$ with two asymmetric donor molecules related by a centre of symmetry, rather than $P1$ with one molecule of the complex in a cell with the *a* axis halved. In contrast, a dibenzofuran molecule may be fitted in either orientation at the dibenzothiophene site without leading to abnormally short N···O contacts. Hence the observed halving of the *a* axis in the dibenzofuran/TCNQ complex corresponds to either an ordered structure in $P1$ with every dibenzofuran molecule in the same orientation, or a disordered $P\bar{1}$ structure with 50% site occupancy for each of the two possible orientations. The crystal structure does not provide appreciable interstack donor–donor or acceptor–acceptor π-orbital overlap. There are thus no structural features facilitating separation of charge-transfer excited-state ion pairs to produce charge carriers (Vincent & Wright, 1974), and this is consistent with our observation that these two complexes are poor photoconductors.

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Etude Structurale par Diffraction des Rayons X d'un Dianhydride Glycofurannique: Bis(*O*-acétyl-3 désoxy-5 α-D-xylofurannose) Dianhydride-1,2′ : 2,1′

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Abstract

The title compound, $C_{14}H_{20}O_8$, crystallizes in the monoclinic space group $P2_1$, with $a = 12{\cdot}543$ (6), $b = 11{\cdot}117$ (6), $c = 11{\cdot}293$ (6) Å, $\beta = 95{\cdot}7$ (3)°, $Z = 4$, $V = 1566{\cdot}8$ Å^3, $d_c = 1{\cdot}331$, $d_m = 1{\cdot}34$ Mg m^{-3}. The refinement of the three-dimensional structure obtained by direct methods was stopped at an R value of 0·054 for 2923 reflexions. Two molecules which are related by a pseudo binary screw axis define the asymmetric unit. Each molecule adopts the symmetry of a non-crystallographic binary axis. The perturbation caused by the anomeric effect is studied. The geometry of the *O*-acetyl group is compared with that of previously reported compounds, as are the dihedral angles and the ring conformation.

Introduction

Dans une étude récente (Amvam Zollo, Pougny & Sinaÿ, 1979), il a été montré que le traitement de différents *O*-isopropylidène-1,2 α-D-glycofurannoses avec l'éthérate de trifluorure de bore dans le benzène anhydre à température ordinaire conduisait avec un excellent rendement à des 1,2-*cis* diglycofurannose dianhydrides-1,2′ : 2,1′.

Avant que ces résultats soient connus, il était intéressant d'entreprendre une analyse cristallographique de l'un des représentants pour élucider la structure de ces molécules.

Le système tétraoxa-2,4,8,10 tricyclo[7.3.0.0^{3,7}]dodécane, auquel est affilié le composé envisagé, avait été antérieurement signalé par Bock & Pedersen (1972),

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