It is noteworthy that in contrast to the single crystals of bis(tetramethylethylenediamine)lithium(I) naphthalenide (Brooks, Wendell & Stucky, 1972) in which we detected an e.p.r. signal probably due to the presence of a small amount of the mononegative ion of naphthalene, we were unable to detect an e.p.r. signal in the alkali COT single crystals, even after ultraviolet irradiation at low temperature: a common technique for removing electrons from radicals in solution or in a glass.

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The Crystal Structures of the Molecular Complexes between Benzidine and 7,7,8,8-Tetracyano-*p*-quinodimethane. II. The Benzidine–TCNQ Solvent-Free Complex

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Abstract. Monoclinic, $C2/m$, $a=12{\cdot}231$ (2), $b=12{\cdot}679$ (4), $c=6{\cdot}477$ (8) Å, $\beta=94{\cdot}84$ (1)°, $C_{12}H_{12}N_2 . C_{12}H_4N_4$, $Z=2$, $D_x=1{\cdot}33$, $D_m=1{\cdot}32$ g cm^{-3}. Crystals were grown from a chloroform solution. BD and TCNQ are alternately stacked along the c axis to form mixed molecular columns. The structure within these columns is the same as that found in crystals of the BD–TCNQ complex containing dichloromethane [Ikemoto, Chikaishi, Yakushi & Kuroda (1972). *Acta Cryst.* B**28**, 3502–3506], but, instead of forming a channelled structure as observed in the latter complex, the molecular columns are closely packed.

Introduction. Preliminary cell constants and space group were determined from Weissenberg and precession photographs. Since the systematic absence is $h+k=2n+1$ for general reflexions, the space group is either $C2/m$, $C2$ or Cm. The precise cell constants and intensity data were measured on a Rigaku four-circle automatic diffractometer using monochromated Mo $K\alpha$ radiation. 1163 independent reflexions in the range $2\theta \le 55°$ were measured. Of these reflexions 425 were three times as large as their standard deviations. No absorption correction was made, since the absorption effect is small ($R\mu \le 0{\cdot}05$).

The structure was solved by the Patterson method. It was refined by the block-diagonal least-squares method assuming the space group $C2/m$. The final R value was 0·082. The weighting scheme was as follows: $w=1/[\sigma^2(F_o)+(cF_o)^2]^{1/2}$, where σ is the standard deviation estimated from the counting statistics and c is the parameter which stands for the other types of error (Grant, Killean & Lawrence, 1969). The value of c was determined to be 0·06 after a few least-squares calculations. The least-squares refinements in the space group

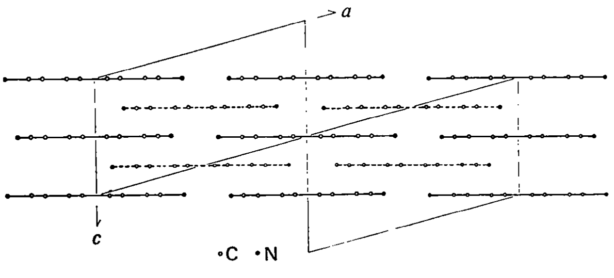

Fig. 1. Projection of the molecular arrangement onto the (010) plane. Molecules with centres at $y=0$ are shown by solid lines, those lying at $y=\frac{1}{2}$ are indicated by broken lines.

Table 1. *Atomic parameters*

(*a*) Atomic coordinates in fractions of cell edges and their standard deviations (in 10^{-3} Å)

	x	y	z
BD			
C(1)	0·3016 (10)	0·0 (0)	0·1497 (10)
C(2)	0·2425 (6)	0·0935 (8)	0·1159 (7)
C(3)	0·1254 (7)	0·0930 (7)	0·0596 (7)
C(4)	0·0632 (9)	0·0 (0)	0·0300 (9)
N(1)	0·4182 (10)	0·0 (0)	0·2048 (12)
H(1)	0·449 (78)	0·059 (68)	0·183 (79)
H(2)	0·289 (68)	0·154 (68)	0·153 (64)
H(3)	0·089 (62)	0·155 (61)	0·055 (60)
TCNQ			
C(5)	0·3020 (6)	0·0950 (7)	0·6469 (6)
C(6)	0·2357 (9)	0·0 (0)	0·6090 (9)
C(7)	0·1197 (9)	0·0 (0)	0·5558 (9)
C(8)	0·0569 (6)	0·0971 (6)	0·5270 (6)
N(2)	0·3607 (6)	0·1662 (6)	0·6739 (7)
H(8)	0·101 (60)	0·158 (60)	0·528 (58)

Table 1 (*cont.*)

(*b*) Thermal parameters. The β's are used in the expression $\exp\left[-(h^2\beta_{11}+k^2\beta_{22}+l^2\beta_{33}+hk\beta_{12}+hl\beta_{13}+kl\beta_{23})\right]$.

BD	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·00551	0·00702	0·01657	0·0	0·00585	0·0
C(2)	0·00366	0·00560	0·01794	−0·00316	−0·00272	−0·00353
C(3)	0·00549	0·00515	0·01515	0·00124	0·00377	−0·00169
C(4)	0·00384	0·00346	0·01295	0·0	0·00252	0·0
N(1)	0·00383	0·01066	0·05022	0·0	0·00760	0·0
H(1)	3·83					
H(2)	2·14					
H(3)	0·71					
TCNQ						
C(5)	0·00314	0·00372	0·01856	−0·00119	0·00315	0·00115
C(6)	0·00375	0·00347	0·01344	0·0	0·00758	0·0
C(7)	0·00309	0·00376	0·01599	0·0	0·00361	0·0
N(2)	0·00470	0·00415	0·03547	−0·00309	0·00839	−0·00353
H(8)	0·32					

*C*2 and *Cm* were also carried out. The *R* values for *C*2 and *Cm* were 0·10 and 0·14, respectively. In these two cases molecular geometries were appreciably distorted. The (F_o-F_c) synthesis in *C*2/*m* showed that the residual electron densities fell within the range +0·35 to −0·35 e Å^{-3}. There was no significant difference, within experimental error, between the minimum value of the structure factors used in the least-squares calculation and the maximum among the structure factors calculated from the final model in *C*2/*m* on the unobservable reflexions. On the basis of these results, we adopted the space group *C*2/*m*.

Results. Final parameters with their standard deviations are listed in Table 1.* Figs. 1 and 2 show the arrangements of the molecules in the crystal. Molecular dimensions and interatomic distances are also shown. The equations of the molecular planes of BD and TCNQ were calculated by the method of least-squares.

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* A table of observed and calculated structure factors has been deposited with the British Library Lending Division as Supplementary Publication No. SUP 30289 (4 pp.). Copies may be obtained through the Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

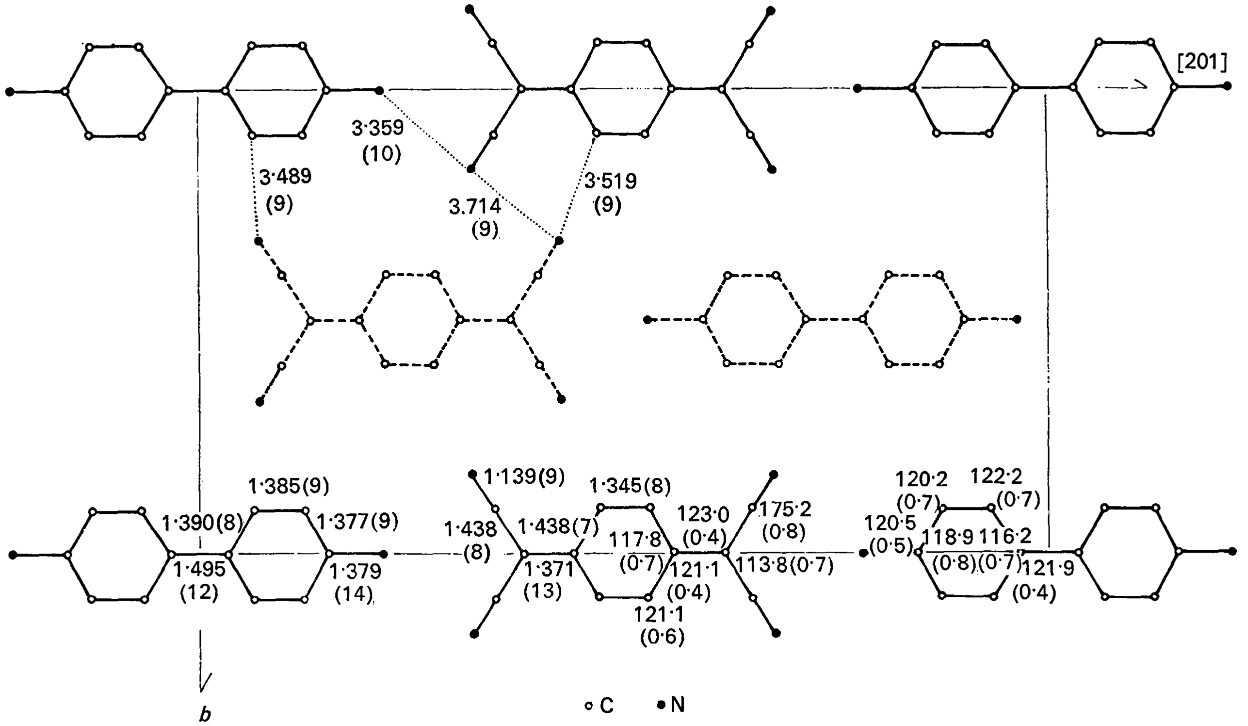

Fig. 2. Projection of the molecular arrangement onto the $(10\bar{2})$ plane. The positions of the molecules shown by broken lines are higher by *c*/4 than those shown by solid lines.

The equations are

$$-0{\cdot}2577X+0{\cdot}0Y+1{\cdot}0000Z=0{\cdot}0$$

for BD and

$$-0{\cdot}2547X+0{\cdot}0Y+1{\cdot}0000Z=3{\cdot}238$$

for TCNQ, where X, Y and Z are coordinates in Å with respect to the crystal axes a, b and c. The planes of BD and TCNQ make an angle of 0·2°.

Discussion. BD and TCNQ form solid molecular complexes which contain solvent molecules in the crystal lattice as well as the solvent-free complex. The structure of the BD–TCNQ complex containing dichloromethane has already been reported (Ikemoto, Chikaishi, Yakushi & Kuroda, 1972). In the solvent-free crystal, the relative orientation of the donor and acceptor molecules within a BD–TCNQ column is the same as that in the dichloromethane-containing crystal, but these molecular columns are closely packed without leaving space to accommodate solvent molecules.

In the solvent-containing crystals, there exist two kinds of specific interaction between BD and TCNQ: the charge-transfer interaction and the hydrogen bonding. The former is reflected in the formation of the BD–TCNQ column, and the latter gives the infinite hydrogen-bonded sheet parallel to the (001) plane. In the solvent-free crystal, however, there is no indication of the hydrogen bonding between BD and TCNQ. Although all molecular planes are parallel to the $(10\bar{2})$ plane, which is perpendicular to the c axis, the molecular columns lying at $y=\frac{1}{2}$ are shifted by $c/4$ along the c axis, so that the molecules shown by broken lines in Fig. 2 are not on the same plane as those shown by solid lines.

The bond lengths of TCNQ in the solvent-free complex are in good agreement with those in the TCNQ crystal (Long, Sparks & Trueblood, 1965). This implies that TCNQ is in the non-ionic state in this complex.

The computer used in this work was the HITAC 5020E at the Computer Centre, University of Tokyo. The program used were the *UNICS* program (1967).

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SHORT COMMUNICATIONS

Contributions intended for publication under this heading should be expressly so marked; they should not exceed about 1000 *words; they should be forwarded in the usual way to the appropriate Co-editor; they will be published as speedily as possible. Publication will be quicker if the contributions are without illustrations.*

Acta Cryst. (1974). **B30**, 837

The crystal and molecular structure of 1-kestose: a correction.

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(*Received* 14 *December* 1973)

In Table 1 of Jeffrey & Park [*Acta Cryst.* (1972). **B28**, 257–267], the C′(3) x coordinate should read −779 instead of −9.

The correction of a typographical error: In the paper on 1-kestose by Jeffrey & Park (1972), Table 1, the C′(3) x coordinate should read −779, instead of −9. All numbers derived using this parameter are correct.

Reference

Jeffrey, G. A. & Park, Y. J. (1972). *Acta Cryst.* **B28**, 257–267.