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Molecular Structures of [2](2,5)(7,7,8,8-Tetracyanoquinodimethano)[2]-paracyclophane and 14,17-Dimethoxy[2](2,5)(7,7,8,8-tetracyanoquinodimethano)[2]paracyclophane†

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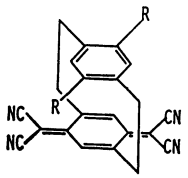
The molecular structures of two [2.2]paracyclophanes containing a 7,7,8,8-tetracyanoquinodimethan (TCNQ) moiety as an electron acceptor have been determined by means of X-ray diffraction. The electron-donating moieties in these cyclopahnes are benzene (TCNQ-X) and *p*-dimethoxybenzene (TCNQ-X($OMe)_2$) rings. TCNQ-X crystallizes in a triclinic space group $P\bar{1}$ with two molecules per unit cell; $a=9.772(2)$, $b=18.477(4)$, $c=6.569(1)$ Å, $\alpha=90.38(2)$, $\beta=107.92(2)$, $\gamma=100.32(2)°$. The crystals of TCNQ-X($OMe)_2$ belong to the orthorhombic system; space group *Fdd2*; $a=25.618(3)$, $b=31.895(5)$, $c=9.808(1)$ Å, $Z=16$. Both structures were solved by the direct method and refined by the block-diagonal least-squares procedure to $R=0.088$ (TCNQ-X) and 0.109 (TCNQ-X($OMe)_2$) for 3141 and 1695 non-zero rehections. In the two structures determined, the [2.2] paracyclophane skeleton has the usual structure; the benzene ring and six-membered ring of the TCNQ moiety have a boat form, and two $C(CN)_2$ portions in the TCNQ moiety are bent down from the mean plane defined by the C(12), C(13), C(15), and C(16) atoms.

As part of a series of structural studies of layered cyclophanes, the X-ray structure determination of [2,2]paracyclophanes containing a 7,7,8,8-tetracyanoquinodimethan (TCNQ) moiety as an electron acceptor has been carried out. This paper will deal with the molecular structures of **1**, [2](2,5)(7,7,8,8-tetracyanoquinodimethano)[2]paracyclophane (abbreviated as TCNQ-X, hereafter) and **2**, 14,17-dimethoxy[2](2,5)-(7,7,8,8-tetracyanoquinodimethano)[2]paracyclophane (TCNQ-X($OMe)_2$).



1, R=H
2, R=OCH_3

Experimental

For the X-ray experiment, a Rigaku automated four-circle diffractometer was used.

TCNQ-X. Graphite-monochromatized Mo $K\alpha$ radiation was used. A reddish violet crystal with approximate dimensions of 0.30 mm×0.25 mm×0.17 mm was mounted on a goniometer of the diffractometer. By searching for the locations of strong diffractions, the setting parameters of the crystal with a primitive lattice were first determined. Based on these parameters, a set of 206 diffraction intensities were measured by the θ-2θ scan technique up to $2\theta=14.3°$ as a preliminary work. By statistically treating these data, the point group symmetry of the crystal was determined as $\bar{1}$, and then the space group as $P\bar{1}$. The *SPAGD* program[1] was used. At this stage, the tentative unit-cell was transformed into the reduced cell, the parameters of which were then refined by the least-squares fit.

Crystal Data. $C_{22}H_{14}N_4\cdot C_6H_6$, *F.W.* 412.5, triclinic, space group $P\bar{1}$, $a=9.772(2)$, $b=18.477(4)$, $c=6.569(1)$ Å, $\alpha=90.38(2)$, $\beta=107.92(2)$, $\gamma=100.32(2)°$, $V=1107.8(4)$ Å^3, $D_m=1.240$ (by flotation), $D_c=1.236$ g cm^{-3} for $Z=2$.

The integrated intensities were measured by the θ-2θ scan method at a rate of 4° min^{-1}. The backgrounds were counted for 7.5 s before and after the scan of each peak. A total of 4412 (3141 non-zero) reflections was collected up to $\sin\theta/\lambda=0.62$ Å^{-1}. The usual Lp corrections were made, but absorption corrections were ignored [μ(Mo $K\alpha$)=0.81 cm^{-1}].

TCNQ-X($OMe)_2$. The space group was determined by means of photographic work. The unit-cell parameters were determined by the least-squares fit of 2θ values of higher-order reflections determined on the Rigaku diffractometer.

Crystal Data. $C_{24}H_{18}N_4O_2$, *F.W.* 394.4, orthorhombic, space group *Fdd2*, $a=25.618(3)$, $b=31.895(5)$, $c=9.808(4)$ Å, $V=8014(3)$ Å^3, $D_m=1.30$, $D_c=1.307$ g cm^{-3} for $Z=16$.

The intensity data were collected on the diffractometer with Zr-filtered Mo $K\alpha$ radiation. The θ-2θ scan technique was employed at a rate of 4° min^{-1}. A bluish violet crystal with approximate dimensions of 0.15 mm×0.18 mm×0.30 mm was used. A total of 2336 reflections was collected up to $2\theta=54°$, of which 1695 $|F|$'s larger than $3\sigma(F)$ were classed as observed. The usual Lp corrections were made, but no absorption correction was applied [μ(Mo $K\alpha$)=0.92 cm^{-1}].

Structure Solution and Refinement

TCNQ-X. The structure was solved by the direct method (*MULTAN* 74)[2] and refined by the block-diagonal least-squares procedure (*HBLS* V).[3] Of the two crystalline benzene molecules centered on (0,0,0) and (1/2,0,1/2), the latter was supposed to be disordered by the feature of peaks on the Fourier map, but the isotropic refinement in which the disorder (two locations with a 3:2 occupancy ratio) was considered was unsuccessful ($R=0.102$). Isotropic refinement was, therefore, again carried out from the beginning, the

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TABLE 1. FRACTIONAL ATOMIC COORDINATES OF TCNQ-X, C_6H_6, WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

(a) C and N atoms with equivalent temperature factors.

Atom	x	y	z	B/Å^2
C(1)	0.7784(4)	0.1871(2)	0.6254(7)	4.52
C(2)	0.8127(5)	0.1746(3)	0.4127(8)	5.94
C(3)	0.7317(4)	0.2174(3)	0.2381(6)	4.39
C(4)	0.5800(4)	0.19937(19)	0.1453(6)	4.22
C(5)	0.4999(4)	0.2535(2)	0.0661(6)	4.11
C(6)	0.5678(5)	0.3260(2)	0.0649(6)	4.26
C(7)	0.7205(5)	0.3398(3)	0.1137(6)	4.91
C(8)	0.8003(5)	0.2865(3)	0.2003(6)	4.82
C(9)	0.4861(6)	0.3879(3)	0.0630(7)	5.83
C(10)	0.5337(5)	0.43102(19)	0.2900(6)	4.46
C(11)	0.5923(4)	0.38191(18)	0.4676(6)	3.61
C(12)	0.7386(4)	0.39009(17)	0.5618(6)	3.49
C(13)	0.8100(4)	0.33154(18)	0.6656(5)	3.23
C(14)	0.7208(4)	0.25729(17)	0.6331(5)	3.25
C(15)	0.5751(4)	0.25388(17)	0.5776(5)	3.25
C(16)	0.5039(4)	0.31536(18)	0.5129(5)	3.29
C(17)	0.9573(4)	0.34794(19)	0.7774(6)	3.64
C(18)	1.0401(5)	0.4211(3)	0.8009(7)	5.22
C(19)	1.0428(4)	0.2977(3)	0.8909(6)	4.42
C(20)	0.3565(4)	0.3075(3)	0.4876(6)	3.93
C(21)	0.2748(4)	0.2395(3)	0.5267(6)	4.89
C(22)	0.2714(5)	0.3641(3)	0.4308(7)	5.43
N(1)	1.1101(5)	0.4785(3)	0.8222(9)	8.90
N(2)	1.1182(4)	0.2609(3)	0.9901(7)	6.60
N(3)	0.2097(4)	0.1858(3)	0.5602(7)	6.89
N(4)	0.1984(5)	0.4069(3)	0.3914(9)	8.46
CB(11)	0.0451(6)	−0.0099(4)	−0.1722(8)	7.3
CB(12)	0.0512(6)	0.0594(3)	−0.0953(9)	6.83
CB(13)	0.0073(7)	0.0703(3)	0.0757(9)	7.28
CB(21)	0.4314(9)	−0.0067(4)	0.2818(12)	11.4
CB(22)	0.3742(7)	0.0257(4)	0.4176(14)	11.4
CB(23)	0.4398(9)	0.0335(4)	0.6328(15)	11.3

(b) H atoms with isotropic temperature factors.

Atom	x	y	z	B/Å^2
H(1A)	0.699(5)	0.151(3)	0.649(7)	6.4(10)
H(1B)	0.865(5)	0.185(2)	0.751(6)	5.6(9)
H(2A)	0.778(5)	0.120(3)	0.374(7)	7.0(11)
H(2B)	0.920(5)	0.188(3)	0.438(7)	7.1(11)
H(4)	0.528(5)	0.148(3)	0.163(7)	6.1(10)
H(5)	0.389(5)	0.240(2)	0.027(6)	5.5(9)
H(7)	0.767(5)	0.391(3)	0.099(6)	5.8(9)
H(8)	0.912(4)	0.301(2)	0.270(6)	5.6(9)
H(9A)	0.369(6)	0.363(3)	0.018(9)	9.5(14)
H(9B)	0.499(5)	0.422(3)	−0.037(7)	6.1(10)
H(10A)	0.619(4)	0.4743(19)	0.299(6)	4.8(8)
H(10B)	0.451(5)	0.453(3)	0.300(7)	6.6(10)
H(12)	0.803(4)	0.4370(18)	0.548(6)	4.4(8)
H(15)	0.512(4)	0.2059(17)	0.571(6)	4.1(8)
HB(11)	0.070(5)	−0.022(3)	−0.298(8)	7.9(12)
HB(12)	0.086(6)	0.103(3)	−0.160(8)	8.7(13)
HB(13)	0.012(7)	0.121(4)	0.143(10)	11.0(16)

TABLE 2. FRACTIONAL ATOMIC COORDINATES OF TCNQ-X(OMe)$_2$, WITH ESTIMATED STANDARD DEVIATIONS IN PARENTHESES

(a) C, N, and O atoms with equivalent temperature factors.

Atom	x	y	z	B_{eq}/Å^2
C(1)	0.0535(4)	0.2173(3)	0.6624(9)	2.4
C(2)	0.1017(5)	0.1961(4)	0.7312(11)	3.2
C(3)	0.1246(4)	0.1608(4)	0.6485(10)	2.8
C(4)	0.1463(4)	0.1687(4)	0.5206(12)	3.0
C(5)	0.1458(4)	0.1374(4)	0.4249(12)	3.4
C(6)	0.1242(4)	0.0977(4)	0.4460(12)	3.0
C(7)	0.1127(4)	0.0874(4)	0.5822(13)	3.6
C(8)	0.1108(4)	0.1198(3)	0.6847(11)	2.8
C(9)	0.1019(5)	0.0720(4)	0.3341(12)	3.8
C(10)	0.0423(4)	0.0736(3)	0.3285(12)	3.2
C(11)	0.0216(4)	0.1147(3)	0.3902(11)	2.4
C(12)	−0.0002(4)	0.1139(3)	0.5169(11)	2.5
C(13)	−0.0027(4)	0.1501(3)	0.6038(9)	1.68
C(14)	0.0277(3)	0.1871(3)	0.5627(10)	1.65
C(15)	0.0381(4)	0.1896(3)	0.4287(10)	2.2
C(16)	0.0319(3)	0.1560(3)	0.3342(10)	2.0
C(17)	−0.0301(4)	0.1478(3)	0.7253(10)	2.4
C(18)	−0.0602(4)	0.1106(4)	0.7571(12)	3.2
C(19)	−0.0362(5)	0.1809(3)	0.8236(13)	3.5
C(20)	0.0391(4)	0.1635(4)	0.1956(12)	3.1
C(21)	0.0497(4)	0.2049(4)	0.1487(11)	3 2
C(22)	0.0367(5)	0.1329(4)	0.0875(12)	3.9
C(23)	0.1778(5)	0.2188(5)	0.3613(16)	5.1
C(24)	0.0794(7)	0.0380(4)	0.7425(16)	6.2
N(1)	−0.0830(5)	0.0820(4)	0.7904(11)	5.4
N(2)	−0.0437(5)	0.2048(3)	0.9065(12)	5.4
N(3)	0.0607(4)	0.2381(4)	0.1097(12)	4.6
N(4)	0.0342(5)	0.1101(4)	0.	5.7
O(1)	0.1623(3)	0.2092(3)	0.4976(9)	4.1
O(2)	0.0935(4)	0.0479(3)	0.6057(10)	4.9

(b) H atoms with isotropic temperature factors.

Atom	x	y	z	B/Å
H(1A)	0.025(4)	0.222(3)	0.720(11)	3.(3)
H(1B)	0.062(4)	0.234(3)	0.609(12)	4.(3)
H(2A)	0.101(5)	0.177(4)	0.794(12)	5.(3)
H(2B)	0.112(5)	0.214(4)	0.788(14)	6.(4)
H(5)	0.146(4)	0.139(4)	0.336(13)	4.(3)
H(8)	0.090(4)	0.109(4)	0.767(13)	5.(3)
H(9A)	0.105(5)	0.043(4)	0.318(13)	5.(3)
H(9B)	0.104(5)	0.087(4)	0.271(14)	7.(4)
H(10A)	0.021(5)	0.081(4)	0.292(14)	7.(4)
H(10B)	0.031(4)	0.054(3)	0.367(12)	4.(3)
H(12)	−0.016(5)	0.084(4)	0.570(13)	6.(3)
H(15)	0.056(4)	0.218(3)	0.391(12)	4.(3)
H(23A)	0.181(5)	0.240(4)	0.357(13)	5.(3)
H(23B)	0.141(5)	0.218(4)	0.299(12)	5.(3)
H(23C)	0.204(5)	0.206(4)	0.371(13)	5.(3)
H(24A)	0.072(4)	0.012(3)	0.738(10)	3.(2)
H(24B)	0.093(5)	0.037(4)	0.849(15)	7.(4)
H(24C)	0.110(5)	0.063(4)	0.806(13)	6.(3)

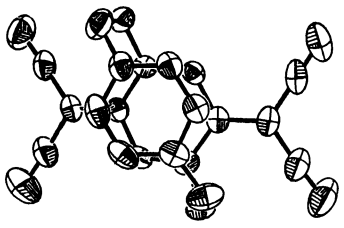
atoms of the benzene molecules being not included. Six carbon atoms of the benzene molecules were located on the difference Fourier map. The possibility of disorder of the benzene molecule at (1/2,0,1/2) was ignored in the subsequent refinement. All the hydrogen atoms except those belonging to the benzene centered on (1/2,0,1/2) could be located on the difference Fourier map computed during the course of anisotropic refinement; they were included in the subsequent cycles with isotropic temperature factors. The final R was 0.088 for non-zero reflections (R_w=0.124). The weighting schemes used at this stage were $w=\{\sigma^2(F)+0.0204|F_o|+0.0015|F_o|^2\}^{-1}$ for $|F_o|>0$ and $w=0.6236$ for $|F_o|=0$. The final atomic parameters are given in Table 1.**

TCNQ-X(OMe)$_2$. The structure was solved by using the *MULTAN* 74 program.[2] On the E map, 25 atoms of the 30 non-hydrogen atoms could be located. The successive Fourier synthesis revealed the remaining 5 atoms.

The structure was refined, isotropically with the unit weight at the earlier stage and anisotropically at the later stage, by means of the *HBLS* V program.[3] The hydrogen atoms were located on the difference Fourier map and then included in the refinement with isotropic temperature factors. R converged to a rather large value of 0.109 for 1615 observed reflections (R_w=0.113). The weighting schemes used were $w=\{\sigma^2(F)+0.0483|F_o|-0.0002|F_o|^2\}^{-1}$ and $w=0$ for $|F_o|=0$ at the final cycle of the refinement. The final atomic parameters are listed in Table 2.** In the analyses of the two compounds, the atomic scattering factors for C, N, and O were taken from "International Tables for X-Ray Crystallography," Vol. IV,[4] and those for H, from Stewart, Davidson, and Simpson.[5]

Results and Discussion

Molecular Structure. TCNQ-X: A perspective view of the molecule (*ORTEP* II)[6] is shown in Fig. 1, while selected bond distances and bond angles are given in Fig. 2. The most remarkable feature of the structure is that the [2.2]paracyclophane skeleton has the usual structure. The upper benzene ring takes a boat form, as is observed in [2.2]paracyclophane;[7,8] the planes defined by the C(3), C(4), and C(8) atoms and by the C(5), C(6), and C(7) atoms form angles of 12.1(5) and 9.7(5)° respectively with the mean plane made by the C(4), C(5), C(7), and C(8) atoms. The C(3)–C(2) and C(6)–C(9) bonds are further bent down by 12.8(5) and 11.9(5)° from the planes formed by the C(3), C(4), and C(8) atoms and by the C(5), C(6), and C(7) atoms respectively. The TCNQ moiety has a twisted structure. The six-membered ring is in a twisted boat form, the planes formed by the C(13), C(14), and C(15) atoms and by the C(11), C(12), and C(16) atoms are bent up from the mean plane made by the C(12), C(13), C(15), and C(16) atoms by 15.2(4) and 15.7(4)° respectively. On the other hand, both of the two C(CN)$_2$ portions are bent down from the mean plane. The two vectors,



(1) TCNQ-X

Fig. 1. Perspective view of the TCNQ-X molecule.
Non-hydrogen atoms are drawn as thermal ellipsoids at 50% probability level.

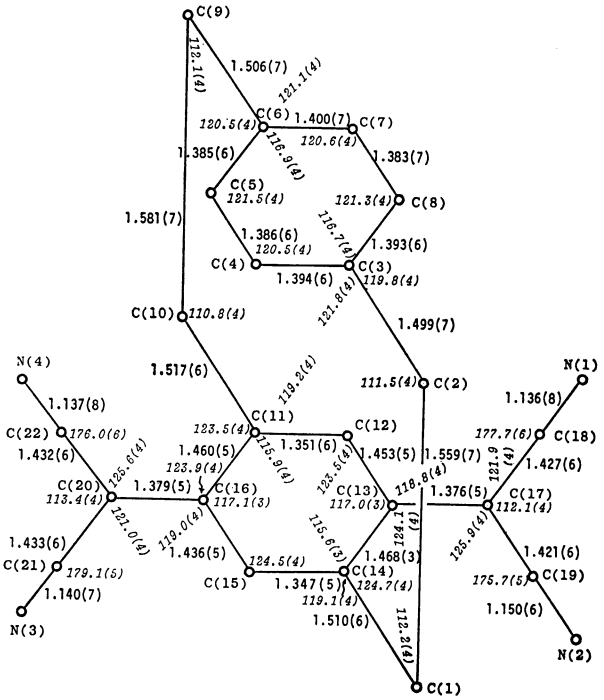
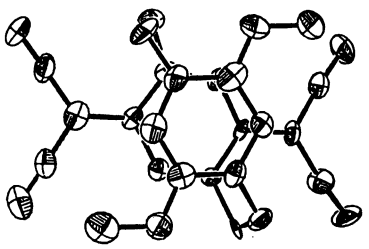


Fig. 2. Bond distances and bond angles in the TCNQ-X molecule (Estimated standard deviations in parentheses).



(2) TCNQ-X(OMe)$_2$

Fig. 3. Perspective view of the TCNQ-X(OMe)$_2$ molecule.
Non-hydrogen atoms are drawn as thermal ellipsoids at 50% probability level.

C(13)–C(17) and C(16)–C(20), make angles of 11.9(3) and 12.8(3)° respectively, with the plane defined by the C(12), C(13), C(15), and C(16) atoms. The dihedral angles between two –C(CN)$_2$ planes and the plane made by the C(12), C(13), C(15), and C(16) atoms are 15.0(3) and 15.9(3)°. The C(10)–C(11)–C(16), C(11)–

** Tables of observed and calculated structure factors and anisotropic thermal parameters are kept at the Chemical Society of Japan; Document No. 8207.

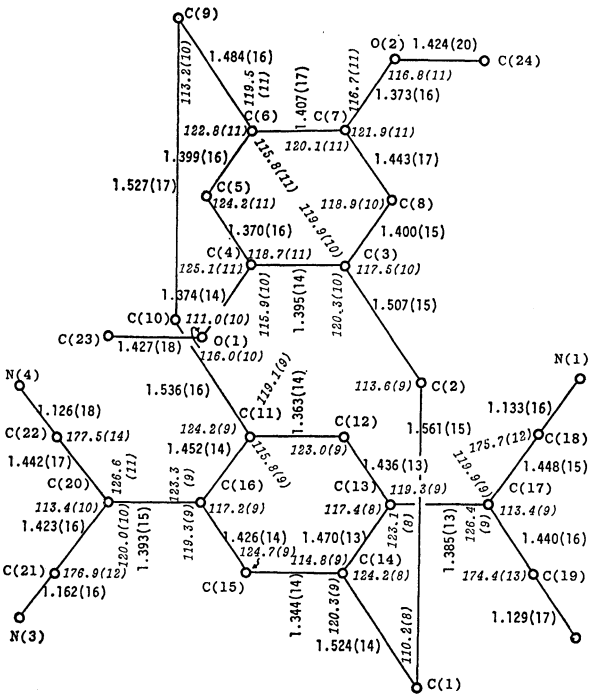


Fig. 4. Bond distances and bond angles in the TCNQ-X-$(OMe)_2$ molecule (Estimated standard deviations in parentheses).

C(16)–C(20), C(16)–C(20)–C(22), C(1)–C(14)–C(13), C(14)–C(13)–C(17), and C(13)–C(17)–C(19) angles ((av. 124.6°) are larger than 120°; they are considered to be due to the repulsions between two pairs of C–C≡N portions and $-(CH_2)_2-$ bridges in the molecule. The C(14)–C(1) and C(11)–C(10) bonds bent up by 7.7(4) and 11.3(4)° from the lower ring. In the $-(CH_2)_2-$ bridges, the torsion angles around the C(1)–C(2) and C(9)–C(10) bonds are −21.0(6) and 5.1(5)° respectively.

TCNQ-X(OMe)$_2$: The molecular structure (*ORTEP* II)[6] is shown in Fig. 3, while the distances and bond angles are given in Fig. 4. The present molecule has a structure similar to that of TCNQ-X except for the OMe portions. As is observed in TCNQ-X, both the upper and lower six-membered rings take boat forms. In the upper ring, the planes formed by the C(3), C(4), and C(8) atoms and by the C(5), C(6), and C(7) atoms are bent down by 8.2(12) and 12.4(12)° respectively from the plane formed by the C(4), C(5), C(7), and C(8) atoms. The OMe groups lie within 0.08 Å on the

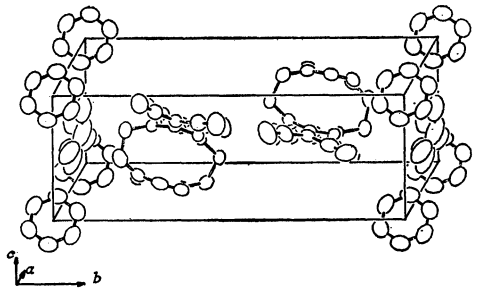


Fig. 5. Perspective view of the crystal structure of TCNQ-X, C_6H_6.

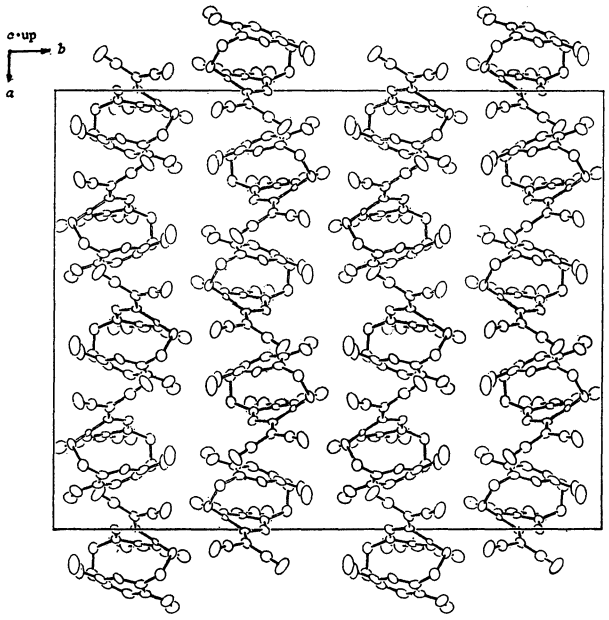


Fig. 6. Crystal structure of TCNQ-X$(OMe)_2$ projected along the c axis.

plane made by the C(4), C(5), C(7), and C(8) atoms. The C(3)–C(2) bond is further bent down by 15.3(12)° from the plane made by the C(3), C(4), and C(8) atoms, and the C(6)–C(9) bond, by 13.0(13)° from the C(5), C(6), and C(7) plane. The TCNQ moiety has a structure similar to that in TCNQ-X. The six-membered ring is also in a boat form; the plane defined by the

Table 3. Short intermolecular atomic contacts less than 3.5 Å
(Estimated standard deviations in parentheses)

TCNQ-X, C_6H_6	
C(20)·····N(2)[a]	3.378(6)
C(21)·····N(2)[a]	3.441(7)
C(22)·····N(2)[a]	3.240(7)
C(6)·····C(15)[b]	3.485(6)
C(6)·····C(16)[b]	3.483(6)
C(7)·····C(13)[b]	3.328(6)
C(7)·····N(1)[c]	3.428(8)
key: a, $-1+x, y, -1+z$;	
b, $x, y, -1+z$;	
c, $2-x, 1-y, 1-z$.	
TCNQ-X$(OMe)_2$	
N(4)·····C(17)[a]	3.379(16)
N(4)·····C(18)[a]	3.394(17)
C(21)·····N(2)[a]	3.371(16)
N(3)·····C(24)[b]	3.43(2)
C(9)·····N(2)[c]	3.305(17)
C(21)·····N(1)[c]	3.397(16)
C(15)·····N(2)[d]	3.378(15)
N(3)·····C(1)[d]	3.293(15)
N(3)·····C(14)[d]	3.320(15)
N(3)·····C(19)[d]	3.386(17)
key: a, $x, y, -1+z$;	
b, $0.25-x, 0.25+y, -0.75+z$;	
c, $0.25+x, 0.25-y, -0.75+z$;	
d, $-x, 0.5-y, -0.5+z$.	

C(13), C(14), and C(15) atoms and the plane defined by the C(11), C(12), and C(16) atoms form angles of 16.9(10) and 16.4(10)° respectively with the mean plane formed by the C(12), C(13), C(15), and C(16) atoms. On the other hand, both of the $C(CN)_2$ portions are bent down from the mean plane, and the C(10)–C(11)–C(16), C(11)–C(16)–C(20), C(16)–C(20)–C(22), C(1)–C(14)–C(13), C(14)–C(13)–C(17), and C(13)–C(17)–C(19) angles (av. 124.6°) are slightly distorted from 120°. The torsion angles around the C(1)–C(2) and C(9)–C(10) bonds in the [2.2] bridges are 23.8(12) and −4.0(12)° respectively.

Crystal Structure. *TCNQ*: Figure 5 shows the crystal structure. The crystalline benzene molecule centered on (1/2,0,1/2) does not give any good shape. Short intermolecular atomic distances less than 3.5 Å are listed in Table 3, the shortest distance being 3.240(7) Å (C(22)$(x,y,z)\cdots$N(2)$(-1+x, y, -1+z)$).

TCNQ-X(OMe)$_2$. The molecular packing in the TCNQ-$X(OMe)_2$ crystal is drawn in Fig. 6. The closest intermolecular atomic contact is 3.293(15) Å (N(3)$(x,y,z)\cdots$C(1)$(-x, 0.5-y, -0.5+z)$), which is listed in Table 3 along with other close contacts.

The calculations were done on an ACOS 700S computer at the Crystallographic Reserach Center, Institute for Protein Research, Osaka University.

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