Electrical properties

The electrical conductivity at 300 K and activation energy of single crystals (short axis) are $2 \times 10^{-3}\ \Omega^{-1}\ cm^{-1}$ and 0·3 eV respectively. This corresponds to the TCNQ stacking direction (*b* axis) of the structure. The data are consistent with values reported previously for *N*,*N*-diethyl-4,4′-bipyridylium(TCNQ)$_4$ (Ashwell, Eley, Wallwork & Willis, 1975) and 1,2-di(*N*-ethyl-4-pyridinium)ethylene(TCNQ)$_4$ (Ashwell, Eley, Fleming, Wallwork & Willis, 1976) which are also homosoric.

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The Crystal Structures of Free Radical Salts and Complexes. XII. The Crystal Structure and Electrical Properties of [1,3-Di(*N*-pyridinium)propane]$^{2+}$(7,7,8,8-Tetracyanoquinodimethane)$_4^{2-}$

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The structure and electrical properties of [1,3-di(*N*-pyridinium)propane]$^{2+}$(7,7,8,8-tetracyanoquinodimethane)$_4^{2-}$, (DPP)$^{2+}$(TCNQ)$_4^{2-}$, are reported. The complex is monoclinic, space group $P2_1$, with a = 7·737, b = 25·289, c = 13·059 Å, β = 92·771°, Z = 2. The TCNQ's are stacked plane-to-plane in isolated groups of four in the (001) plane separated along **c** by layers of cations. Within the tetrads the mean intermolecular separations are 3·20, 3·25 and 3·18 Å. The electrical properties are discussed in terms of the crystal structure.

Introduction

The crystal structures of TCNQ complexes fall into three distinct groups (Dahm, Horn, Johnson, Miles & Wilson, 1975), *i.e.* homosoric, heterosoric and nonsoric with characteristic high, intermediate and low conductivities respectively. The complex salts of bipyridinium cations generally have homosoric structures in which the TCNQ's are stacked plane-to-plane in isolated triads (Ashwell & Wallwork, 1975) or tetrads (Ashwell, Eley, Wallwork & Willis, 1975; Ashwell, Eley, Fleming, Wallwork & Willis, 1976; Ashwell, Eley, Drew, Wallwork & Willis, 1977). In this paper we report the electrical conductivity and crystal structure of [1,3-di(*N*-pyridinium)propane]$^{2+}$(TCNQ)$_4^{2-}$, which is also homosoric.

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Experimental

Crystal data

$(C_{13}H_{16}N_2)(C_{12}H_4N_4)_4$, M_r = 1017·1, monoclinic, a = 7·737 (2), b = 25·289 (9), c = 13·059 (3) Å, β = 92·771 (6)°, U = 2552·1 Å^3, D_m = 1·31 (1), Z = 2, D_c = 1·324 g cm^{-3}; $F(000)$ = 524. Mo $K\alpha$ (λ = 0·71069 Å), μ = 0·91 cm^{-1}. Space group $P2_1$.

Black crystals of the complex were deposited when a warm acetonitrile solution (200 ml) of TCNQ (0·2 g) and LiTCNQ (0·2 g) was added to an aqueous solution

(10 ml) of 1,3-di(*N*-pyridinium)propane dibromide (0·2 g) and allowed to cool slowly. The space group and cell constants were obtained initially from oscillation and Weissenberg photographs. The cell dimensions were subsequently refined on a Hilger & Watts computer-controlled, four-circle diffractometer. Intensities were collected with a $\theta/2\theta$ scan, a scintillation counter and Mo $K\alpha$ radiation. Because the quality of the crystals was poorer than average for this type of complex, only 1400 reflexions were measured of which significant counts $|I > 3\sigma(I)|$ were recorded for 1200. The intensities were corrected for Lorentz and polarization factors but not for absorption.

The structure was solved from a Patterson synthesis and refined by block-diagonal least squares. Because of the paucity of data, only isotropic temperature factors were refined. Positional parameters of the H atoms were calculated from the coordinates of the C atoms and confirmed by a difference synthesis. The H atoms were given isotropic temperature factors of 0·06 Å^2 and were included in fixed calculated positions. Block diagonal, least-squares refinement with the weighting scheme $w = 1/(1 + [(|F_o| - A)/B]^2)$, where $|F_o|$ is on the absolute scale, $A = 10{\cdot}5$ and $B = 20{\cdot}5$, gave a final $R = 0{\cdot}066$. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974). The final positional parameters are listed in Table 1. Least-squares planes were calculated for each of the TCNQ moieties and the pyridinium rings of the cation; the results are summarized in Table 2.*

* Lists of structure factors and isotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32532 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Table 1. *Final positional parameters* ($\times 10^4$)

The figures in parentheses indicate standard deviations.

	x	*y*	*z*		*x*	*y*	*z*
C(1)	−1792 (21)	1864 (7)	1279 (13)	C(33)	−2222 (23)	−535 (8)	−4394 (14)
C(2)	39 (20)	1831 (6)	1255 (12)	C(34)	−4399 (19)	−668 (7)	329 (11)
C(3)	924 (17)	1817 (6)	360 (10)	C(35)	−6240 (21)	−648 (7)	402 (12)
C(4)	73 (19)	1849 (6)	−611 (11)	C(36)	−3471 (21)	−672 (7)	1268 (13)
C(5)	−1905 (19)	1880 (6)	−577 (12)	N(9)	2050 (18)	−631 (6)	−3622 (10)
C(6)	2684 (19)	1868 (6)	274 (12)	N(10)	−3060 (18)	−465 (6)	−5142 (11)
C(7)	−2563 (21)	1884 (7)	2207 (13)	N(11)	−7664 (19)	−607 (6)	484 (11)
C(8)	−4395 (23)	1880 (7)	2254 (13)	N(12)	−2711 (19)	−665 (7)	2048 (12)
C(9)	−1614 (21)	1932 (7)	3143 (13)	C(37)	−3215 (18)	−1886 (7)	−1260 (12)
C(10)	826 (20)	1867 (6)	−1513 (12)	C(38)	−5080 (19)	−1861 (7)	−1280 (12)
C(11)	2655 (24)	1817 (7)	−1576 (13)	C(39)	−5858 (17)	−1868 (6)	−358 (11)
C(12)	125 (21)	1922 (7)	−2502 (13)	C(40)	−4926 (17)	−1906 (6)	599 (11)
N(1)	−5862 (19)	1888 (6)	2281 (11)	C(41)	−3060 (18)	−1907 (6)	555 (11)
N(2)	−774 (19)	2001 (7)	3875 (12)	C(42)	−2247 (18)	−1904 (6)	−325 (11)
N(3)	4079 (21)	1793 (6)	−1566 (12)	C(43)	−2364 (19)	−1870 (7)	−2167 (12)
N(4)	−895 (19)	2014 (6)	−3263 (12)	C(44)	−572 (18)	−1880 (7)	−2197 (11)
C(13)	−956 (18)	614 (7)	−170 (12)	C(45)	−3256 (22)	−1875 (8)	−3146 (14)
C(14)	877 (17)	575 (6)	−234 (10)	C(46)	−5783 (19)	−1923 (7)	1525 (12)
C(15)	1673 (18)	583 (6)	−1105 (11)	C(47)	−7576 (23)	−1893 (8)	1516 (13)
C(16)	637 (18)	629 (6)	−2051 (12)	C(48)	−4852 (21)	−1990 (7)	2434 (13)
C(17)	−1170 (17)	682 (6)	−2015 (11)	N(13)	920 (17)	−1871 (6)	−2206 (10)
C(18)	−1943 (17)	673 (6)	−1140 (11)	N(14)	−3948 (1)	−1841 (7)	−3954 (12)
C(19)	−1789 (18)	614 (6)	761 (12)	N(15)	−9076 (19)	−1846 (6)	1553 (11)
C(20)	−3618 (19)	640 (7)	829 (11)	N(16)	−4138 (19)	−2042 (6)	3223 (12)
C(21)	−830 (21)	554 (7)	1700 (14)	C(49)	2932 (20)	1180 (7)	4231 (12)
C(22)	1420 (19)	633 (7)	−2986 (12)	C(50)	3002 (24)	1616 (8)	4815 (14)
C(23)	3226 (21)	591 (7)	−3073 (13)	C(51)	4619 (28)	1772 (9)	5326 (15)
C(24)	441 (19)	653 (7)	−3927 (12)	C(52)	5993 (23)	1440 (8)	5134 (14)
N(5)	−5052 (17)	689 (6)	878 (10)	C(53)	5882 (23)	1006 (7)	4567 (14)
N(6)	−23 (20)	527 (7)	2481 (12)	C(54)	3072 (19)	−1332 (7)	4400 (12)
N(7)	4695 (18)	579 (6)	−3147 (11)	C(55)	2190 (22)	−1739 (7)	4804 (14)
N(8)	303 (18)	674 (6)	−4719 (11)	C(56)	466 (25)	−1793 (8)	4588 (15)
C(25)	−2006 (18)	−634 (6)	−2515 (11)	C(57)	−352 (24)	−1420 (8)	3989 (15)
C(26)	−3839 (19)	−603 (7)	−2489 (12)	C(58)	503 (22)	−1025 (7)	3584 (13)
C(27)	−4597 (18)	−622 (6)	−1566 (11)	C(59)	3201 (21)	−548 (7)	3344 (13)
C(28)	−3610 (18)	−645 (6)	−588 (11)	C(60)	3130 (21)	−52 (7)	3988 (12)
C(29)	−1740 (17)	−665 (6)	−676 (11)	C(61)	4132 (22)	382 (7)	3490 (13)
C(30)	−982 (16)	−667 (6)	−1585 (10)	N(17)	4306 (17)	866 (5)	4096 (10)
C(31)	1223 (19)	−597 (7)	−3428 (12)	N(18)	2226 (17)	−968 (5)	3824 (10)
C(32)	591 (21)	−640 (7)	−3528 (12)				

Table 2. *Details of molecular planes (x, y, z are fractional atomic coordinates; asterisks indicate atoms not defining the planes)*

TCNQ(*A*)
Equations to the planes

Molecule	$0{\cdot}275x + 25{\cdot}273y + 0{\cdot}072z - 4{\cdot}734 = 0$
Quinonoid group	$0{\cdot}371x + 25{\cdot}256y + 0{\cdot}213z - 4{\cdot}665 = 0$

Distances from the planes (Å)

	Molecule	Quinonoid group
C(1)	−0·06	0·00
C(2)	−0·10	−0·01
C(3)	−0·11	−0·03
C(4)	−0·07	−0·01
C(5)	−0·04	0·00
C(6)	−0·09	−0·04
C(7)	−0·03	0·05
C(8)	−0·09	−0·03*
C(9)	0·13	0·22*
C(10)	−0·00	0·05
C(11)	−0·08	−0·01*
C(12)	0·10	0·13*
N(1)	−0·11	−0·07*
N(2)	0·33	0·44*
N(3)	−0·10	−0·02*
N(4)	0·31	0·32*

TCNQ(*B*)
Equations to the planes

Molecule	$0{\cdot}523x + 25{\cdot}205y + 0{\cdot}555z - 1{\cdot}490 = 0$
Quinonoid group	$0{\cdot}651x + 25{\cdot}156y + 0{\cdot}711z - 1{\cdot}483 = 0$

Distances from the planes (Å)

	Molecule	Quinonoid group
C(13)	−0·00	−0·01
C(14)	−0·01	0·00
C(15)	0·00	0·01
C(16)	0·02	−0·01
C(17)	0·06	0·01
C(18)	0·04	0·00
C(19)	0·01	−0·00
C(20)	−0·02	−0·05*
C(21)	−0·04	−0·02*
C(22)	0·01	−0·01
C(23)	−0·00	−0·01*
C(24)	−0·04	−0·09*
N(5)	0·03	−0·02*
N(6)	−0·03	0·02*
N(7)	0·04	0·05*
N(8)	−0·07	−0·14*

TCNQ(*C*)
Equations to the planes

Molecule	$0{\cdot}345x + 25{\cdot}227y + 0{\cdot}674z + 1{\cdot}773 = 0$
Quinonoid group	$0{\cdot}269x + 25{\cdot}243y + 0{\cdot}622z + 1{\cdot}782 = 0$

Distances from the planes (Å)

	Molecule	Quinonoid group
C(25)	−0·06	−0·03
C(26)	−0·05	0·00
C(27)	−0·06	−0·01
C(28)	−0·02	0·02
C(29)	−0·01	0·02
C(30)	−0·05	−0·03
C(31)	−0·01	0·03
C(32)	−0·06	−0·04*
C(33)	0·05	0·10*
C(34)	−0·04	−0·00
C(35)	−0·05	0·00*
C(36)	0·04	0·07*
N(9)	0·01	0·02*
N(10)	0·15	0·21*
N(11)	0·01	0·07*
N(12)	0·14	0·16*

TCNQ(*D*)
Equations to the planes

Molecule	$0{\cdot}297x + 25{\cdot}237y + 0{\cdot}647z + 4{\cdot}932 = 0$
Quinonoid group	$0{\cdot}199x + 25{\cdot}260y + 0{\cdot}509z + 4{\cdot}874 = 0$

Distances from the planes (Å)

	Molecule	Quinonoid group
C(37)	−0·00	−0·02
C(38)	0·00	0·01
C(39)	0·02	0·02
C(40)	0·01	−0·01
C(41)	0·06	0·02
C(42)	0·04	0·00
C(43)	0·00	−0·01
C(44)	0·03	0·00*
C(45)	−0·01	−0·09*
C(46)	0·01	−0·02
C(47)	0·03	0·02*
C(48)	−0·08	−0·13*
N(13)	0·09	0·05*
N(14)	−0·09	−0·06*
N(15)	0·10	0·11*
N(16)	−0·13	−0·20*

Pyridine(*A*)
Equation to the plane

$1{\cdot}977x + 13{\cdot}693y - 10{\cdot}609z + 2{\cdot}302 = 0$

	Distance from the plane (Å)
C(49)	0·01
C(50)	−0·00
C(51)	−0·01
C(52)	0·01
C(53)	−0·00
N(17)	−0·01

Pyridine(*B*)
Equation to the plane

$-1{\cdot}711x + 13{\cdot}837y + 10{\cdot}669z - 2{\cdot}339 = 0$

	Distance from the plane (Å)
C(54)	−0·01
C(55)	0·01
C(56)	−0·01
C(57)	0·01
C(58)	−0·02
N(18)	0·02

Discussion

Description of the structure

Fig. 1 shows a general view of the structure in which the TCNQ molecules are stacked plane-to-plane in groups of four in the (001) plane with **b** as the general direction of stacking. There is no direct overlap between adjacent tetrads and between molecules *B* and *C* within each tetrad there is rather poor overlap so the tetrad is better described as a pair of partially interacting dimers. Between tetrads the closest contacts are C(38)···N(13) = 3·27 along *x* and C(9)···N(13) = 3·32 Å along *y*. Several other short intermolecular contacts are listed in Table 3.

Within the tetrads two types of overlap (Fig. 2) are observed, *AB* and *CD* both having the 'ideal' overlap found in highly conducting complexes. The mean interplanar separations are 3·20 ± 0·14 (3·13 ± 0·06), 3·25 ± 0·07 (3·29 ± 0·05) and 3·18 ± 0·07 (3·12 ± 0·02) Å between molecules *AB*, *BC* and *CD* respectively and corresponding dihedral angles are 2·9 (3·1), 1·4 (2·9) and 0·4 (0·7)°. Between tetrads, *i.e.* between

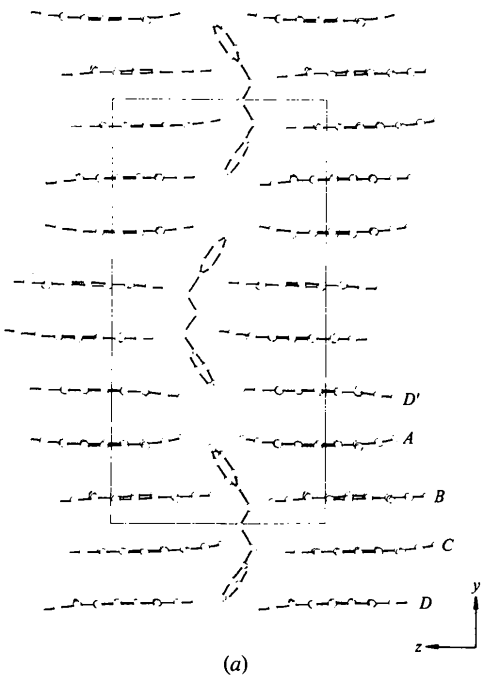

Fig. 1. The crystal structure of [1,3-di(*N*-pyridinium)propane]$^{2+}$-(TCNQ)$_4$ projected (*a*) along **a** and (*b*) along **c**.

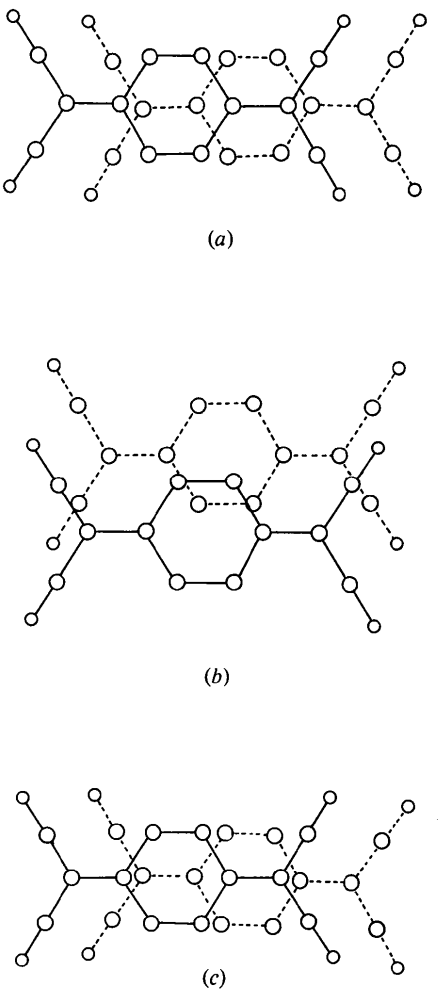

Fig. 2. TCNQ molecular overlaps. (*a*) TCNQ(*A*)–TCNQ(*B*), (*b*) TCNQ(*B*)–TCNQ(*C*) and (*c*) TCNQ(*C*)–TCNQ(*D*).

Table 3. *Short intermolecular contacts* (Å)

The standard deviations are 0·02 Å.

TCNQ(*A*)–TCNQ(*B*)		TCNQ(*A*)–TCNQ(*D*)		Inter-tetrad	
C(1^{i})–C(19^{i})	3·23	C(3^{i})–C(42^{ii})	3·39	N(1^{i})–C(2^{iv})	3·39
C(2^{i})–C(21^{i})	3·36	C(5^{i})–C(47^{iii})	3·36	N(3^{i})–C(5^{v})	3·31
C(3^{i})–C(14^{i})	3·24	C(6^{i})–C(39^{iii})	3·39	N(3^{i})–C(6^{v})	3·39
C(4^{i})–C(13^{i})	3·26	C(7^{i})–N(13^{ii})	3·40	N(7^{i})–C(26^{v})	3·30
C(4^{i})–C(14^{i})	3·34	C(9^{i})–N(13^{ii})	3·32	N(11^{i})–C(14^{iv})	3·31
C(4^{i})–C(18^{i})	3·37	N(3^{i})–C(48^{ii})	3·34	N(13^{i})–C(38^{v})	3·27
C(5^{i})–C(13^{i})	3·32			N(13^{i})–C(39^{v})	3·39
C(5^{i})–C(18^{i})	3·14	TCNQ(*C*)–TCNQ(*D*)		N(15^{i})–C(41^{iv})	3·29
C(6^{i})–C(19^{i})	3·30	C(25^{i})–C(43^{i})	3·17	N(15^{i})–C(42^{iv})	3·39
C(6^{i})–C(20^{i})	3·28	C(25^{i})–C(44^{i})	3·36		
C(10^{i})–C(15^{i})	3·35	C(25^{i})–C(45^{i})	3·38	TCNQ–cation	
C(10^{i})–C(16^{i})	3·21	C(26^{i})–C(45^{i})	3·36	N(1^{i})–C(49^{iv})	3·29
C(11^{i})–C(15^{i})	3·28	C(27^{i})–C(37^{i})	3·39	N(2^{i})–C(50^{i})	3·26
C(12^{i})–C(16^{i})	3·37	C(27^{i})–C(38^{i})	3·18	N(2^{i})–C(52^{iv})	3·37
C(12^{i})–C(17^{i})	3·31	C(28^{i})–C(37^{i})	3·28	N(6^{i})–C(60^{i})	3·39
		C(28^{i})–C(38^{i})	3·39	N(7^{i})–C(52^{vi})	3·32
TCNQ(*B*)–TCNQ(*C*)		C(28^{i})–C(42^{i})	3·37	N(7^{i})–C(53^{vi})	3·34
C(13^{i})–C(29^{i})	3·35	C(29^{i})–C(37^{i})	3·37	N(8^{i})–C(49^{vi})	3·18
C(20^{i})–C(36^{i})	3·37	C(29^{i})–C(42^{i})	3·19	N(8^{i})–C(53^{vii})	3·17
C(22^{i})–C(32^{i})	3·35	C(30^{i})–C(43^{i})	3·30	N(9^{i})–C(54^{vi})	3·26
C(22^{i})–N(9^{i})	3·34	C(30^{i})–C(44^{i})	3·19	N(10^{i})–C(60^{vii})	3·28
C(23^{i})–N(9^{i})	3·29	C(34^{i})–C(39^{i})	3·34	N(12^{i})–C(58^{i})	3·25
C(24^{i})–C(32^{i})	3·32	C(34^{i})–C(40^{i})	3·18	N(14^{i})–C(54^{vii})	3·33
		C(34^{i})–C(41^{i})	3·31	N(14^{i})–C(55^{vii})	3·34
		C(35^{i})–C(39^{i})	3·26	N(15^{i})–C(58^{iv})	3·40
		C(35^{i})–C(40^{i})	3·35	N(16^{i})–C(54^{iv})	3·25
		C(36^{i})–C(41^{i})	3·28		

Superscripts indicate equivalent positions as follows:

(i)	x, y, z	(iv)	$x-1, y, z$	(vi)	$x, y, z-1$
(ii)	$\bar{x}, y+\frac{1}{2}, \bar{z}$	(v)	$x+1, y, z$	(vii)	$x-1, y, z-1$
(iii)	$\bar{x}-1, y+\frac{1}{2}, \bar{z}$				

TCNQ(*A*)–TCNQ(*D'*), the dihedral angle is 5·4 (5·4)°. The figures in parentheses indicate the distances and angles between quinonoid groups. The discrepancies between the whole molecule and quinonoid group values are caused by C–$(CN)_2$ out-of-plane distortions.

The dimensions of the four crystallographically independent TCNQ moieties and the cation (Fig. 3) are in agreement with values reported previously for similar complexes (Ashwell, Eley, Wallwork & Willis, 1975; Ashwell, Eley, Fleming, Wallwork & Willis, 1976; Ashwell, Eley, Drew, Wallwork & Willis, 1977). However, in view of the rather large standard deviations, no indication of the degree of delocalization of negative charge within the TCNQ tetrads is obtained. The angle between the mean planes through the two pyridinium rings is 66·0°. The angles that pyridine(*A*) makes with TCNQ(*A*) and TCNQ(*B*) are 57·0 and 58·7°, and the angle that pyridine(*B*) makes with TCNQ(*C*) and TCNQ(*D*) is 54·6°.

Electrical properties

In $(DPP)^{2+}(TCNQ)_4^{2-}$ anisotropic resistivities of 700, 300 and 5200 Ω cm at 300 K were observed along the long, intermediate and short crystal directions respectively, which correspond to the *a*, *b* and *c* axes of the unit cell. The TCNQ's are stacked plane-to-plane in isolated tetrads approximately along **b** and in the (001) plane each consisting of a pair of only partially interacting dimers. This results in nearly isotropic conductivities of $\sim 2 \times 10^{-3}$ Ω^{-1} cm^{-1} along **a** and **b**. The lower conductivity along **c** reflects the alternate layers of cations and TCNQ's in this direction.

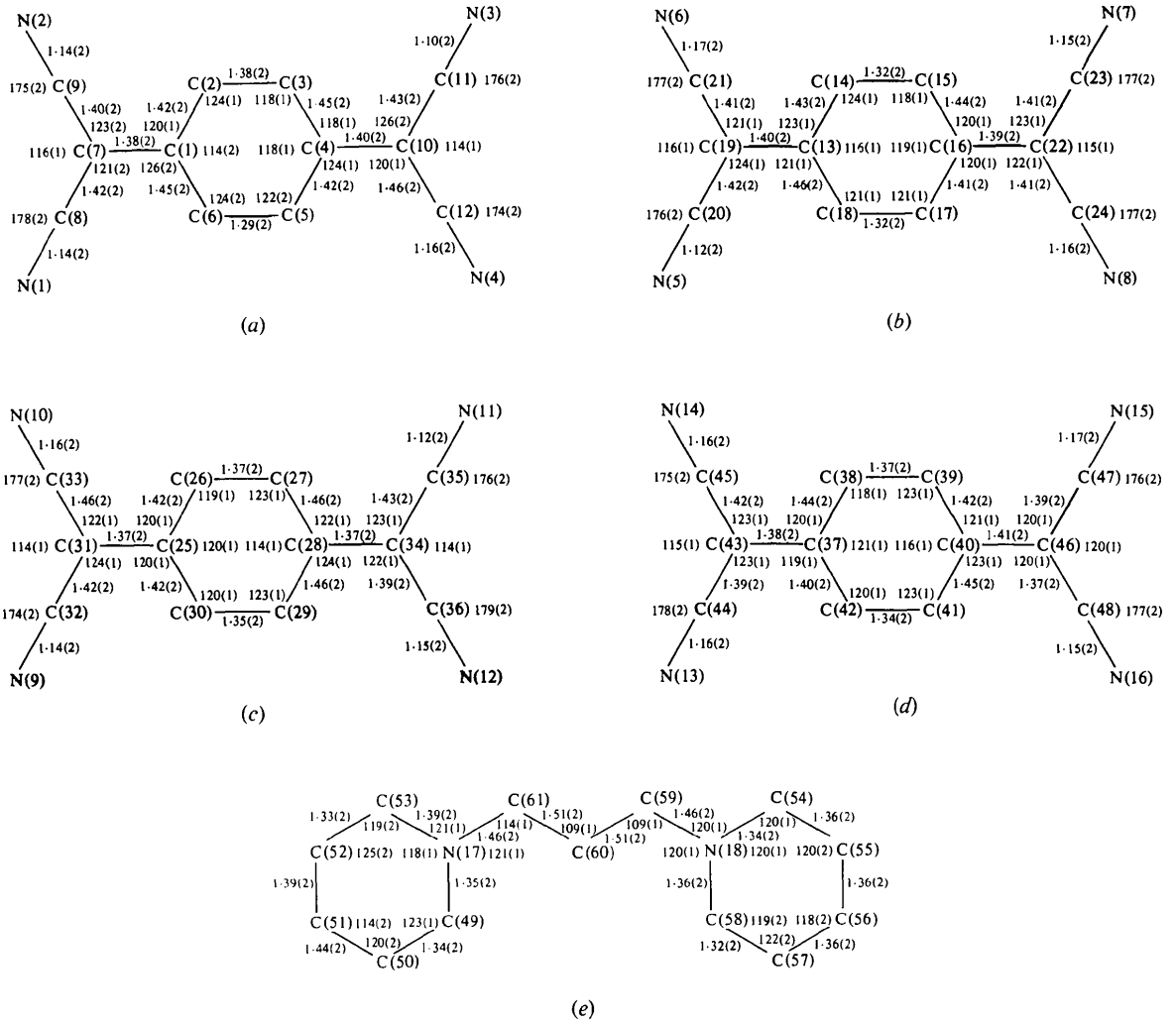

Fig. 3. Molecular dimensions of (*a*) TCNQ(*A*), (*b*) TCNQ(*B*), (*c*) TCNQ(*C*), (*d*) TCNQ(*D*) and (*e*) the cation.

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