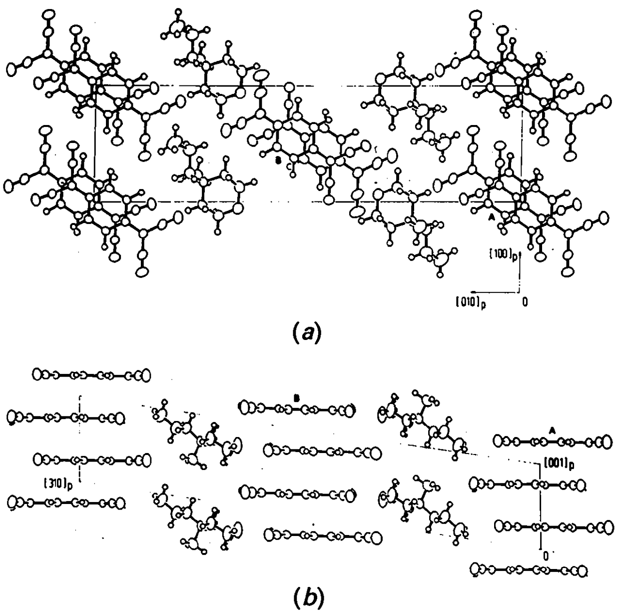

Fig. 3. MPM(TCNQ)$_2$: (*a*) projection along [001]; (*b*) projection along **M** (see Table 1 in Part I) of one layer of cations and anions.

replacement of MPM by MBM makes the diagonal $3a + b$ longer by an increase of a and b and a decrease of γ. The structures contain zigzag stacks along c in which all overlaps have a type I character. The largest deviation from an idealized type I occurs for B^3–B^2 (this is from TCNQ group B at position 3 $= -x+1, -y+1, -z$ to TCNQ group B at position $2 = x,y,z+1$; see Table 3) in MPM(TCNQ)$_2$. Successive stacks along a are connected to form sheets parallel to the ac plane by electrostatically favourable H···N contacts down to 2·65 (1) Å for MPM(TCNQ)$_2$ and 2·71 (1) Å for MBM(TCNQ)$_2$.

Molecules within a sheet are related by inversion centres, but sheets $A(y=0)$ and $B(y=\frac{1}{2})$ are not equivalent. The TCNQ molecules in successive sheets are approximately parallel with stack inclinations between 1·8 and 5·9°. In both compounds the charges ρ_A and ρ_B are equal within experimental error.

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1:2 Complexes of (Thio)Morpholinium Derivatives and the Electron Acceptor 7,7,8,8-Tetracyano-*p*-quinodimethane. Part III.* *N*-Ethyl-*N*-butylmorpholinium Di-7,7,8,8-tetracyano-*p*-quinodimethanide EBM(TCNQ)$_2$ and *N*-Ethyl-*N*-butylthiomorpholinium Di-7,7,8,8-tetracyano-*p*-quinodimethanide EBTM(TCNQ)$_2$†

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(*Received* 1 *September* 1988; *accepted* 1 *June* 1989)

Abstract. EBM(TCNQ)$_2$, $C_{10}H_{22}NO^+.2C_{12}H_4N_4^{1/2-}$, $M_r = 580{\cdot}7$, monoclinic, $P2_1/c$, $a = 7{\cdot}965\,(1)$, $b = 28{\cdot}523\,(2)$, $c = 15{\cdot}886\,(2)$ Å, $\beta = 123{\cdot}82\,(1)°$, $V = 2998\,(1)$ Å^3, $Z = 4$, $D_x = 1{\cdot}286$ Mg m^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}089$ mm^{-1}, $F(000) = 1220$, $T = 294$ K, $R = 0{\cdot}089$ for 5150 observed reflections. Transformation to conventional cell by (1,0,0/0,1,0/1,0,1). EBTM(TCNQ)$_2$, $C_{10}H_{22}NS^+.2C_{12}H_4N_4^{1/2-}$, $M_r = 596{\cdot}7$ triclinic, $P\bar{1}$, $a = 7{\cdot}725\,(2)$, $b = 15{\cdot}179\,(3)$, $c = 14{\cdot}333\,(3)$ Å, $\alpha = 107{\cdot}79\,(2)$, $\beta = 96{\cdot}19\,(2)$, $\gamma = 101{\cdot}79\,(2)°$, $V = 1540\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}287$ Mg m^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu =$

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* Part II: Visser, de Boer & Vos (1990).

† 1:2 *N*-Butyl-*N*-ethylmorpholinium and *N*-butyl-*N*-ethylthiomorpholinium salts with the radical anion of 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile.

0108-2701/90/050860-05$03.00

Table 1. *Final fractional coordinates* ($\times 10^{-4}$) *and* U_{eq} *values* (Å$^2 \times 10^{-3}$) *for the non*-H *atoms of EBM(TCNQ)*$_2$*, with e.s.d.'s in parentheses*

$U_{eq} = \sum_i U_{ii}/3$ after diagonalization of the **U** tensor.

	x	y	z	U_{eq}
N(1)	−1032 (5)	−1908 (1)	821 (2)	76 (2)
C(2)	−1580 (4)	−1535 (1)	779 (2)	47 (2)
C(3)	−2281 (4)	−1072 (1)	739 (2)	39 (1)
C(4)	−4257 (5)	−1031 (1)	540 (2)	44 (2)
N(5)	−5833 (4)	−1004 (1)	391 (2)	62 (2)
C(6)	−1086 (4)	−677 (1)	918 (2)	33 (1)
C(7)	−1809 (4)	−217 (1)	913 (2)	36 (1)
C(8)	−659 (4)	166 (1)	1085 (2)	35 (1)
C(9)	1318 (4)	124 (1)	1285 (2)	34 (1)
C(10)	2048 (4)	−336 (1)	1297 (2)	36 (1)
C(11)	884 (4)	−719 (1)	1112 (2)	36 (1)
N(12)	6044 (4)	462 (1)	1788 (2)	63 (2)
C(13)	4461 (4)	482 (1)	1639 (2)	42 (1)
C(14)	2494 (4)	517 (1)	1443 (2)	37 (1)
C(15)	1816 (4)	986 (1)	1402 (2)	43 (1)
N(16)	1339 (4)	1367 (1)	1361 (2)	65 (2)
N(17)	2337 (4)	−1429 (1)	3270 (2)	54 (1)
C(18)	1795 (4)	−1069 (1)	3327 (2)	39 (1)
C(19)	1082 (4)	−617 (1)	3378 (2)	37 (1)
C(20)	−879 (4)	−620 (1)	3202 (2)	44 (1)
N(21)	−2454 (4)	−634 (1)	3053 (2)	68 (2)
C(22)	2226 (4)	−214 (1)	3570 (2)	35 (1)
C(23)	1471 (4)	239 (1)	3593 (2)	39 (1)
C(24)	2590 (4)	630 (1)	3777 (2)	40 (1)
C(25)	4548 (4)	607 (1)	3947 (2)	36 (1)
C(26)	5290 (4)	157 (1)	3920 (2)	35 (1)
C(27)	4178 (4)	−236 (1)	3737 (2)	35 (1)
N(28)	9133 (5)	951 (1)	4348 (2)	68 (2)
C(29)	7616 (5)	988 (1)	4262 (2)	46 (2)
C(30)	5695 (4)	1012 (1)	4127 (2)	38 (1)
C(31)	4975 (5)	1462 (1)	4170 (2)	47 (2)
N(32)	4345 (4)	1819 (1)	4198 (2)	69 (2)
N(33)	3600 (3)	2894 (1)	2024 (2)	37 (1)
C(34)	3053 (4)	3171 (1)	1082 (2)	41 (1)
C(35)	1116 (4)	3004 (1)	152 (2)	51 (2)
O(36)	−514 (3)	3033 (1)	282 (2)	61 (1)
C(37)	−129 (4)	2747 (1)	1096 (3)	58 (2)
C(38)	1777 (4)	2900 (1)	2087 (2)	49 (2)
C(39)	5355 (4)	3154 (1)	2929 (2)	46 (1)
C(40)	6176 (5)	2918 (1)	3943 (2)	65 (2)
C(41)	4160 (4)	2390 (1)	1985 (2)	43 (1)
C(42)	5994 (4)	2322 (1)	1952 (2)	56 (2)
C(43)	6498 (6)	1798 (1)	2072 (3)	70 (2)
C(44)	8341 (6)	1682 (1)	2081 (3)	82 (3)

Table 2. *Final fractional coordinates* ($\times 10^{-4}$) *and* U_{eq} *values* (Å$^2 \times 10^{-3}$) *for the non*-H *atoms of EBTM(TCNQ)*$_2$*, with e.s.d.'s in parentheses*

$U_{eq} = \sum_i U_{ii}/3$ after diagonalization of the **U** tensor.

	x	y	z	U_{eq}
N(1)	109 (4)	2797 (2)	3223 (2)	76 (1)
C(2)	389 (4)	2094 (2)	2780 (2)	50 (1)
C(3)	752 (3)	1218 (2)	2229 (2)	41 (1)
C(4)	2603 (4)	1194 (2)	2319 (2)	49 (1)
N(5)	4071 (3)	1168 (2)	2383 (2)	72 (1)
C(6)	−592 (3)	439 (2)	1612 (2)	36 (1)
C(7)	−173 (3)	−412 (2)	1018 (2)	40 (1)
C(8)	−1477 (3)	−1159 (2)	390 (2)	40 (1)
C(9)	−3339 (3)	−1141 (2)	321 (2)	37 (1)
C(10)	−3765 (3)	−304 (2)	934 (2)	37 (1)
C(11)	−2454 (3)	456 (2)	1544 (2)	38 (1)
N(12)	−8060 (3)	−2074 (2)	−431 (2)	65 (1)
C(13)	−6548 (4)	−1987 (2)	−377 (2)	44 (1)
C(14)	−4660 (3)	−1913 (2)	−331 (2)	40 (1)
C(15)	−4200 (3)	−2698 (2)	−1008 (2)	46 (1)
N(16)	−3819 (4)	−3308 (2)	−1569 (2)	68 (1)
N(17)	1773 (4)	3093 (2)	6035 (2)	77 (1)
C(18)	1970 (3)	2378 (2)	5567 (2)	50 (1)
C(19)	2277 (3)	1493 (2)	5002 (2)	42 (1)
C(20)	4088 (4)	1436 (2)	5159 (2)	46 (1)
N(21)	5568 (3)	1417 (2)	5312 (2)	67 (1)
C(22)	893 (3)	744 (2)	4339 (2)	38 (1)
C(23)	1267 (3)	−125 (2)	3769 (2)	39 (1)
C(24)	−69 (3)	−859 (2)	3140 (2)	39 (1)
C(25)	−1894 (3)	−797 (2)	3030 (2)	35 (1)
C(26)	−2264 (3)	73 (2)	3605 (2)	39 (1)
C(27)	−926 (3)	811 (2)	4232 (2)	40 (1)
N(28)	−6547 (3)	−1437 (2)	2161 (2)	54 (1)
C(29)	−5090 (3)	−1499 (2)	2256 (2)	40 (1)
C(30)	−3270 (3)	−1558 (2)	2374 (2)	37 (1)
C(31)	−2900 (3)	−2425 (2)	1809 (2)	44 (1)
N(32)	−2596 (3)	−3125 (2)	1352 (2)	63 (1)
N(33)	1847 (2)	5629 (1)	2491 (1)	34 (1)
C(34)	1258 (3)	6408 (2)	2190 (2)	40 (1)
C(35)	936 (3)	6220 (2)	1079 (2)	43 (1)
S(36)	2913 (1)	6090 (1)	538 (1)	45 (1)
C(37)	3581 (3)	5246 (2)	1066 (2)	43 (1)
C(38)	3627 (3)	5529 (2)	2176 (2)	40 (1)
C(39)	459 (3)	4673 (2)	2035 (2)	43 (1)
C(40)	−1346 (4)	4669 (2)	2344 (2)	61 (1)
C(41)	2123 (3)	5966 (2)	3627 (2)	43 (1)
C(42)	2692 (4)	5279 (2)	4102 (2)	59 (1)
C(43)	2734 (5)	5636 (2)	5225 (2)	69 (1)
C(44)	4145 (6)	6523 (3)	5791 (3)	102 (2)

0·149 mm^{-1}, $F(000) = 626$, $T = 294$ K, $R = 0{\cdot}080$ for 6189 observed reflections. Transformation to conventional cell by (-1,0,0/0,0,-1/0,-1,0). Both compounds contain tetrameric stacks which are connected to form sheets. In contrast to EBM(TCNQ)$_2$ where the stacks are rather regular, in EBTM(TCNQ)$_2$ the stacks are composed of almost isolated tetramers consisting of two distinct equivalent dimers.

Introduction. A general introduction to the series is given by Visser, Bouwmeester, de Boer & Vos (1990).

Experimental. Dark violet, for the thio compound almost black, crystals obtained by slowly cooling a hot solution of EBM or EBTM iodide, and neutral TCNQ in acetonitrile. All data collected with Mo $K\alpha$ radiation on a CAD-4F diffractometer equipped with a graphite monochromator and beam flattener (Helmholdt & Vos, 1977). Cell constants of EBM(TCNQ)$_2$ from 22 optimized reflections with $0{\cdot}39 < \sin\theta/\lambda < 0{\cdot}47$ Å^{-1}, EBTM(TCNQ)$_2$ cell from 18 reflections with $0{\cdot}42 < \sin\theta/\lambda < 0{\cdot}50$ Å^{-1}. Intensities measured with ω–2θ scan up to $\theta = 30°$. For EBM(TCNQ)$_2$ 8558 intensities ($h\ 0 \rightarrow 12$, $k\ 0 \rightarrow 41$, $l\ -23 \rightarrow 23$) were collected from a crystal of 0·20 × 0·35 × 0·40 mm; equivalent $h0l$ intensities averaged, final data set of 8038 independent reflections, including 1421 reflections with $I < 0$. Corrections for intensity control within ±0·5%. For EBTM(TCNQ)$_2$ two series of reflections (data collection was interrupted for prolonged periods due to apparatus failure) with 521 common intensities were collected from a crystal measuring 0·07 × 0·14 × 0·38 mm. Scaling according to Hamilton, Rollett & Sparks (1965) gives $R_{\rm int} = [\sum w(I_{\rm obs} - \bar{I})^2/\sum wI^2]^{1/2} = 0{\cdot}040$ and a final data set of 8941 intensities ($h\ 0 \rightarrow 10$, $k\ -21 \rightarrow 21$, $l\ -20 \rightarrow 20$), 1278 of which have $I < 0$. Correction for intensity control was within ±2·6%. Corrections were made for Lorentz and polarization effects, but not for absorption.

The major part of the EBM(TCNQ)$_2$ structure could be determined by the direct methods program *MULTAN*78 (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978). For EBTM(TCNQ)$_2$, where this procedure did not work, the TCNQ part of the structure has been deduced from the Patterson synthesis by trial and error. After isotropic refinement of the known parts of the structures, difference Fourier maps revealed the positions of the remaining non-H atoms. Least-squares blocked-matrix refinement on $|F|$, with blocks for each independent molecule. Scattering factors for non-H atoms from Cromer & Mann (1968) and for H atoms from Stewart, Davidson & Simpson (1965). Anisotropic thermal parameters for non-H atoms and isotropic for H atoms (constrained at 1·08 Å from C and at 1·00 Å from N). No correction for extinction. Details for EBM(TCNQ)$_2$: weight $w = 0$ for $|F_o| < 2\sigma(\text{count};F_o)$, $w = [\sigma^2(\text{count};F_o) + 0{\cdot}0009|F_o|^2]^{-1}$ for remaining reflections. Max. $|\Delta|/\sigma = 0{\cdot}53 \times 10^{-2}$, mean $= 0{\cdot}31 \times 10^{-3}$, $R = 0{\cdot}089$, $wR = 0{\cdot}080$, $S = 1{\cdot}42$, $N_o = 5150$, $N_v = 428$. Residual density $\Delta\rho$ close to molecules: $\Delta\rho_{\max} = 0{\cdot}31$, $\Delta\rho_{\min} = -0{\cdot}31$ e Å^{-3}. Remote from molecules $|\Delta\rho_{\max}| = 0{\cdot}31$, $\sigma(\Delta\rho) = 0{\cdot}07$ e Å^{-3}. Details for EBTM(TCNQ)$_2$: weight $w = 0$ for $|F_o| < 2\sigma(\text{count};F_o)$, $w = [\sigma^2(\text{count};F_o) + 0{\cdot}0007|F_o|^2]^{-1}$ for remaining reflections. Max. $|\Delta|/\sigma = 0{\cdot}50 \times 10^{-3}$, mean $= 0{\cdot}47 \times 10^{-4}$, $R = 0{\cdot}080$, $wR = 0{\cdot}073$, $S = 1{\cdot}39$, $N_o = 6189$, $N_v = 428$. Residual density $\Delta\rho$ close to molecules: $\Delta\rho_{\max} = 0{\cdot}29$, $\Delta\rho_{\min} = -0{\cdot}32$ e Å^{-3}. Remote from molecules $|\Delta_{\max}| = 0{\cdot}25$, $\sigma(\Delta\rho) = 0{\cdot}07$ e Å^{-3}. All computations were carried out on a Cyber 170/760 with the *XRAY* system (1976) and local crystallographic programs. Final coordinates of the non-H atoms and U_{eq} values for EBM(TCNQ)$_2$ and EBTM(TCNQ)$_2$ are given in Tables 1 and 2 respectively. Bond lengths and atomic numbering for the heavy-atom skeletons are shown in Figs. 1 and 2.*

For comparison of R values in similar cases in this series, see remark in the *Introduction* of Part I.

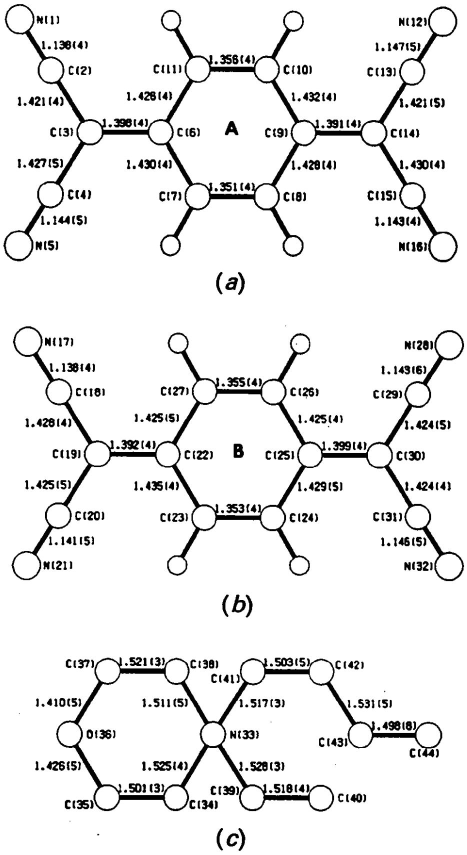

Fig. 1. EBM(TCNQ)$_2$: bond lengths (Å) and atomic numbering for the non-H atoms.

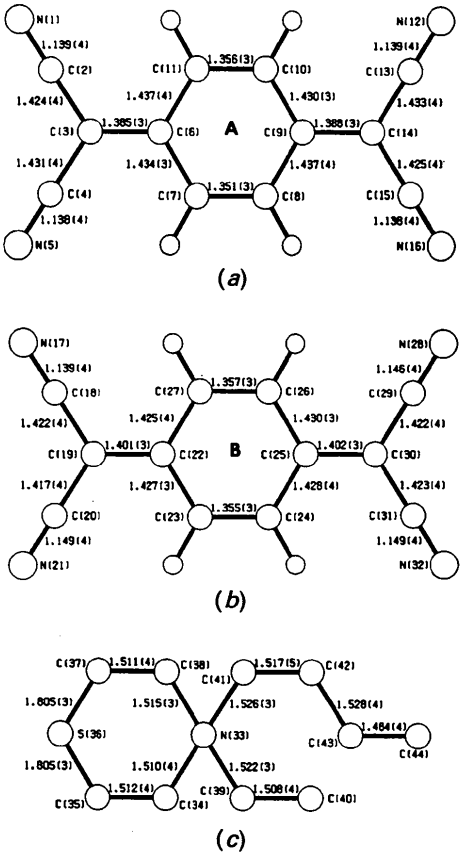

Fig. 2. EBTM(TCNQ)$_2$: bond lengths (Å) and atomic numbering for the non-H atoms.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters and figures giving bond angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52318 (56 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Crystal structure data block for EBM(TCNQ)$_2$*

	TCNQ *A*			TCNQ *B*		
G	0·01138 (38)	−0·02769 (9)	0·10991 (18)	0·33850 (39)	0·01972 (9)	0·37561 (18)
L	0·085148	0·028299	0·012682	0·082039	0·028958	0·013331
M	−0·110069	0·020541	−0·008351	−0·110577	0·019422	−0·005860
N	0·058924	−0·002524	0·074232	0·062286	−0·003661	0·074357
U	0·0357 (12)			0·0369 (13)		
Charge (e)	$\rho_A = 0{\cdot}51\ (6)$	1·354 (4)	1·429 (4)	1·395 (5)	1·425 (5)	1·143 (5)
	$\rho_B = 0{\cdot}53\ (6)$	1·354 (4)	1·429 (4)	1·396 (4)	1·425 (5)	1·142 (5)
Stack	$t^c(A^{\text{i}}\text{–}A) = 0{\cdot}122$ eV	$\nu(A^{\text{i}}\text{–}A) = -1{\cdot}787\mathbf{L} + 0{\cdot}170\mathbf{M} + 3{\cdot}286\mathbf{N}$				
	$t^c(A-B) = 0{\cdot}126$ eV	$\nu(A\text{–}B) = 1{\cdot}847\bar{\mathbf{L}} + 0{\cdot}233\bar{\mathbf{M}} + 3{\cdot}265\bar{\mathbf{N}}$				
	$t^c(B\text{–}B^{\text{ii}}) = 0{\cdot}138$ eV	$\nu(B\text{–}B^{\text{ii}}) = -0{\cdot}152\mathbf{L} - 1{\cdot}186\mathbf{M} + 3{\cdot}279\mathbf{N}$				

Symmetry code: (i) $\bar{x}, \bar{y}, \bar{z}$; (ii) $\bar{x} + 1, \bar{y}, \bar{z} + 1$.

Table 4. *Crystal structure data block for EBTM(TCNQ)$_2$*

	TCNQ *A*			TCNQ *B*		
G	0·19636 (32)	−0·03518 (16)	0·09646 (17)	−0·04981 (31)	−0·00275 (17)	0·36862 (17)
L	0·096560	0·055818	0·045638	0·098511	0·054191	0·046525
M	−0·093088	0·035016	0·021739	−0·089586	0·038317	0·019059
N	0·010084	−0·027681	0·054761	−0·018993	−0·026517	0·055012
U	0·0386 (9)			0·0387 (9)		
Charge (e)	$\rho_A = 0{\cdot}32\ (4)$	1·354 (3)	1·435 (3)	1·386 (3)	1·428 (4)	1·139 (4)
	$\rho_B = 0{\cdot}65\ (4)$	1·356 (3)	1·427 (3)	1·401 (3)	1·421 (4)	1·146 (4)
Stack	$t^c(A^{\text{i}}\text{–}A) = 0{\cdot}017$ eV	$\nu(A^{\text{i}}\text{–}A) = -1{\cdot}182\mathbf{L} + 2{\cdot}617\mathbf{M} + 3{\cdot}469\mathbf{N}$				
	$t^c(A - B^{\text{ii}}) = 0{\cdot}178$ eV	$\nu(A\text{–}B) = 2{\cdot}080\bar{\mathbf{L}} + 0{\cdot}112\bar{\mathbf{M}} + 3{\cdot}169\bar{\mathbf{N}}$				
	$t^c(B\text{–}B) = 0{\cdot}101$ eV	$\nu(B\text{–}B^{\text{ii}}) = 1{\cdot}695\mathbf{L} + 0{\cdot}047\mathbf{M} + 3{\cdot}326\mathbf{N}$				

Symmetry code: (i) $\bar{x}, \bar{y}, \bar{z}$; (ii) $\bar{x}, \bar{y}, \bar{z} + 1$.

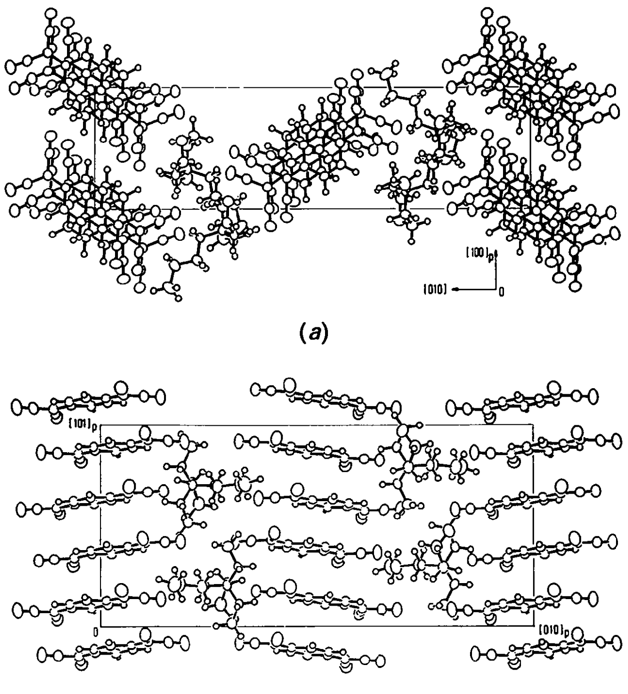

Fig. 3. EBM(TCNQ)$_2$: (*a*) projection along [101]; (*b*) projection along [100]. Note that the stacks are along [101].

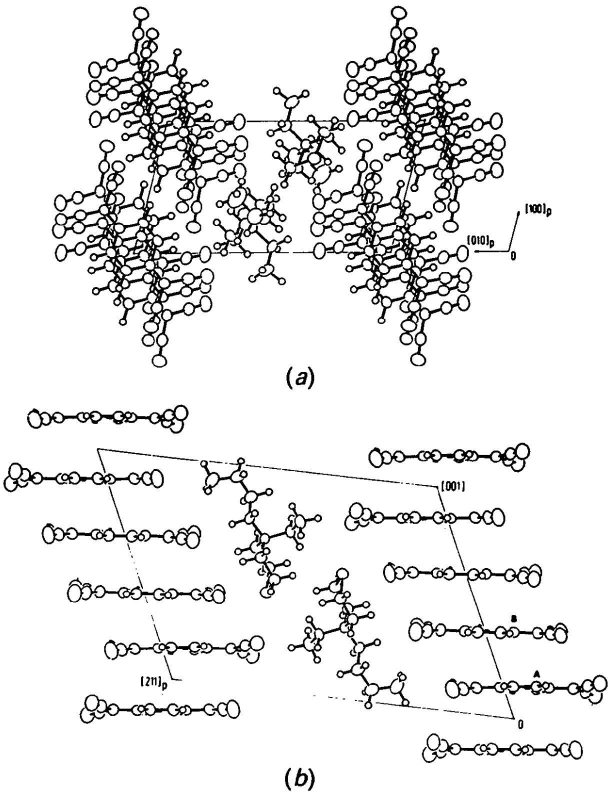

Fig. 4. EBTM(TCNQ)$_2$: (*a*) projection along [001]; (*b*) projection along the molecular axis **M** (see Table 1 in Part I) of one layer of cations and anions.

Discussion. Projections of the EBM(TCNQ)$_2$ structure are shown in Fig. 3 and projections of EBTM(TCNQ)$_2$ in Fig. 4. The structure data blocks are presented in Tables 3 and 4. Both structures contain tetrameric TCNQ stacks connected to form sheets parallel to the *ac* plane by electrostatically favourable H···N contacts ranging from 2·55 to 2·67 Å in EBTM(TCNQ)$_2$ and from 2·61 to 2·69 Å in EBM(TCNQ)$_2$. The cations are extended along the stacks. The packing of the sheets in the two structures is different. In EBM(TCNQ)$_2$ successive sheets are related by the *c* glide plane at $y = \pm\frac{1}{4}$, whereas in EBTM(TCNQ)$_2$ successive sheets along *y* are translationally equivalent. In both cases the stack contains two, parallel within 1°, independent molecules *A* and *B* in the sequence $[ABB^2A^1][ABB^2A^1]$... *etc.* with inversion centres between molecules of the same kind. The structure data blocks show that the character of the stacks in the two compounds is very different. In EBM(TCNQ)$_2$ the stacks are almost regular with no significant charge difference between *A* and *B*. Three overlaps of type I with practically equal t^c values alternate with an overlap of type II with slightly larger t^c. The stack inclination is 8·1°. In EBTM(TCNQ)$_2$ the stacks are composed of almost isolated tetramers consisting of two distinct equivalent dimers in which most of the (negative) charge is accumulated at the *B* molecules [$\rho^s_B - \rho^s_A = 0{\cdot}34\,(6)$ e for $\rho^s_A + \rho^s_B$ normalized to 1]. The stack inclination, 22·4°, is larger than for EBM(TCNQ)$_2$. The observed charge accumulation on *B* in EBTM(TCNQ)$_2$ agrees with the average $C{\equiv}N\cdots N^+$ distances being shorter for *B* than for *A*.

The present crystal structures reveal that replacement of O by S can have a profound influence on the crystal packing and on the character of the stacks.

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1:2 Complexes of (Thio)Morpholinium Derivatives and the Electron Acceptor 7,7,8,8-Tetracyano-*p*-quinodimethane. Part IV.[*] *N,N*-Dimethylthiomorpholinium Di-7,7,8,8-tetracyano-*p*-quinodimethanide DMTM(TCNQ)$_2$[†] at Room Temperature and its Phase Transition at 272 K

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(*Received* 1 *September* 1988; *accepted* 1 *June* 1989)

Abstract. DMTM(TCNQ)$_2$, $C_6H_{14}NS^+.2C_{12}H_4N_4^{1/2-}$, $M_r = 540{\cdot}6$, monoclinic, $P2_1/m$, $a = 7{\cdot}846\,(3)$, $b = 27{\cdot}051\,(14)$, $c = 7{\cdot}914\,(3)$ Å, $\beta = 55{\cdot}56\,(3)°$, $V = 1385\,(1)$ Å^3, $Z = 2$, $D_x = 1{\cdot}30$ Mg m^{-3}, Mo $K\bar{\alpha}$, $\lambda = 0{\cdot}71069$ Å, $\mu = 0{\cdot}157$ mm^{-1}, $F(000) = 562$, $T = 294$ K, $R = 0{\cdot}094$ for 2535 observed reflections. Transformation to conventional cell by $(1,0,-1/0,-1,0/-1,0,0)$. The room-temperature structure, which is isomorphous with DMM(TCNQ)$_2$ (I) [Kamminga & van Bodegom (1981). *Acta Cryst.* **B37**, 114–119], contains dimeric stacks connected to form sheets parallel to the *ac* plane. The DMTM cations are disordered around the mirror planes. Disappearance of the mirror symmetry at the phase transition suggests that the cations are ordered in the low-temperature structure.

Introduction. A general introduction to the subject is given in Part I (Visser, Bouwmeester, de Boer & Vos, 1990*a*) of this series. The special interest of

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[*] Part III: Visser, Bouwmeester, de Boer & Vos (1990*b*).

[†] 1:2 *N,N*-Dimethylthiomorpholinium salt with the radical anion of 2,2′-(2,5-cyclohexadiene-1,4-diylidene)bispropanedinitrile.

0108-2701/90/050864-03$03.00