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A Refinement of the Crystal Structure of Cesium Tetracyanoquinodimethanide*

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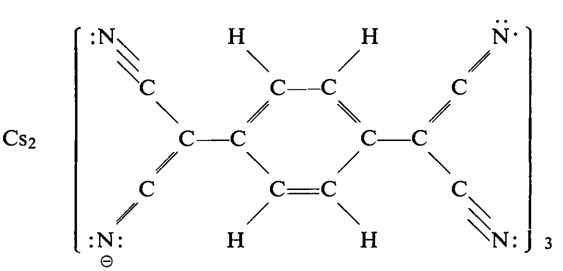
The previously reported structure of cesium tetracyanoquinodimethanide, $Cs_2(TCNQ)_3$, has been refined with new three-dimensional data; the final R is 3·9 %. The refined structure shows that all interplanar separations within the TCNQ columns are short charge-resonance contacts of 3·22 and 3·26 Å. The steric relationship of two-thirds of the vicinal pairs is strikingly similar to that in *N*-methylphenazinium TCNQ.

Introduction

A two-dimensional investigation (Arthur, 1964) of the crystal structure of the organic semiconductor, cesium 7,7,8,8-tetracyanoquinodimethanide, $Cs_2(TCNQ)_3$, (I), seemed to show that the planar TCNQ molecules, which pack face-to-face to form somewhat irregular columns parallel to **b**, were grouped into charge-resonance-bonded triads; interplanar spacings of 3·21 Å within a triad and 3·44 Å between triads were found. Recently, study of a second electrically conductive salt, *N*-methylphenazinium TCNQ (Fritchie, 1966), showed that it contains regular columns of TCNQ anions whose intracolumnar steric configuration is virtually identical with that in the reported triads in $Cs_2(TCNQ)_3$. The implication that this charge-resonance configuration persisted despite the obvious difference between the two salts made further refinement of the structure of the cesium salt desirable.



(I)

Experimental data

As reported by Arthur, the $Cs_2(TCNQ)_3$ structure has the symmetry of space group $P2_1/c$ (C_{2h}^5), with two formula units per cell. The lattice constants, redetermined with a Picker diffractometer, are $a = 7{\cdot}34 \pm 0{\cdot}01$, $b = 10{\cdot}40 \pm 0{\cdot}02$, $c = 21{\cdot}98 \pm 0{\cdot}04$ Å, $\beta = 97{\cdot}18 \pm 0{\cdot}05°$, and agree very well with those given previously. A multifaced crystal of maximum and minimum thicknesses approximately 0·24 mm and 0·15 mm was used for data collection on a Picker diffractometer. Zr-filtered Mo $K\alpha$ radiation was used, with a scintillation detector and a pulse-height analyser set to collect about 90% of the $K\alpha$ radiation. Data were collected in the θ–2θ scanning technique (Furnas, 1956). A range of 1·67° in 2θ was scanned at a rate of 1° per minute. A 20-second background count was collected at each end of the scan range. Standard deviations were assigned to each reflection on the basis of statistical errors of counting, except that no $\sigma(|F_o|)$ was allowed to be less than $0{\cdot}025\ |F_o|$. If the similarly assigned $\sigma(I_o)$ exceeded $\frac{1}{2}I_o$, the reflection was considered unobserved and given $2\ \sigma(I_o)$ as an observational threshold. A total of 3845 reflections was measured, of which 2749 are above their respective observational thresholds. The average standard deviation in $|F_o|$ is about 4%.

The usual Lorentz and polarization factors were applied to the intensity data, and an absorption factor was also applied to each reflection with the aid of a program written by C. T. Prewitt. This program evaluates the absorption integral by summation over a specified number of points within the crystal; eight were used in this case. The transmission factors for $|F_o|$ ranged from 0·78 to 0·86.

Refinement

The parameters of Arthur (1964) gave an initial R value of 22%. Ten cycles of least-squares refinement of the non-hydrogen atoms, performed with the aid of a modified Gantzel–Sparks–Trueblood block-diagonal program (Gantzel, Sparks & Trueblood, 1962), reduced R to 5·5%. Thermal parameters were converted from isotropic to ellipsoidal when R dropped below 15%; no difficulty was experienced in the refinement. An $(F_o - F_c)$ synthesis computed with the parameters from the most recent cycle then revealed the six hydrogen atoms. These were the largest peaks in the map, except for some perturbations very close to the cesium atom. The hydrogen peaks were well-defined and were 0·5 to 0·8 e.Å^{-3} in height. The peaks near

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Table 1. *Positional parameters*

All figures have been multiplied by 10^5. Standard deviations are in parentheses. Hydrogen positions were not refined.

	x	y	z
Cs(1)	51750 (3)	23071 (3)	26651 (1)
N(2)	−38808 (49)	00335 (37)	16494 (16)
N(3)	20271 (51)	00096 (39)	23681 (15)
N(4)	47584 (49)	30093 (42)	−08661 (17)
N(5)	−08822 (51)	29856 (36)	−16944 (16)
N(6)	−41626 (53)	34015 (46)	12713 (17)
N(7)	16208 (55)	36427 (41)	20226 (16)
C(8)	−23755 (52)	01926 (36)	15706 (16)
C(9)	−05195 (50)	04119 (33)	14743 (16)
C(10)	08598 (53)	01881 (36)	19739 (16)
C(11)	−14012 (43)	11155 (32)	03929 (16)
C(12)	−00554 (46)	09049 (31)	09115 (15)
C(13)	18004 (48)	12070 (33)	08434 (16)
C(14)	22664 (45)	16927 (34)	03107 (16)
C(15)	09230 (45)	19037 (31)	−02082 (15)
C(16)	−09342 (45)	15877 (32)	−01400 (15)
C(17)	32597 (54)	27421 (38)	−08142 (17)
C(18)	14142 (47)	24091 (31)	−07562 (15)
C(19)	01321 (51)	27053 (35)	−12741 (16)
C(20)	−26902 (56)	36950 (39)	12017 (17)
C(21)	−08562 (50)	40318 (33)	11133 (15)
C(22)	05244 (54)	38327 (38)	16243 (16)
C(23)	−18286 (48)	46627 (32)	00467 (15)
C(24)	−04397 (48)	45104 (31)	05659 (15)
C(25)	14255 (48)	48715 (33)	04894 (15)
H(26)	−26700	08800	04600
H(27)	27400	11200	12100
H(28)	35500	18800	02600
H(29)	−19200	17200	−04900
H(30)	−31100	44200	01000
H(31)	23800	47700	08500

the cesium atom, shown to be the result of a program error causing the imaginary part of the scattering factor to be improperly treated, were ±2·4 e.Å^{-3} in height. General background variation did not exceed ±0·2 e.Å^{-3}.

Hydrogen atoms were added to the model at positions 1·0 Å from the appropriate carbon atoms; following Jensen's (1965) suggestion that hydrogen atoms be given temperature factors about 2 Å^2 smaller than attached carbon atoms, a value of 0·5 Å^2 was assigned to each. Introduction of the hydrogen atoms lowered R by 0·4%. Correction of the above-mentioned program error permitted reduction of R to 3·9% in four further least-squares cycles. As before, only non-hydrogen parameters were refined.

The final positional and thermal parameters and their standard deviations are listed in Tables 1 and 2 respectively. The average standard deviation in a carbon or nitrogen position, calculated with the formula

$$\sigma_i = \{[\sum_H w_H|\Delta F_H|^2]/[A_{ii}(n-s)]\}^{\frac{1}{2}} ,$$

is about 0·0027 Å and implies a bond-length standard deviation of 0·004 Å. This latter value must be increased to 0·005 Å, however, because of uncertainty in the cell dimensions. In the formula above, A_{ii} is the diagonal element of the normal equation matrix corresponding to the ith parameter; n is the number of observations (2828) contributing to the normal equations after removal of 14 reflections suffering from extinction and 1003 experimentally unobserved reflections having calculated values below their observational thresholds, and s is the number of varied parameters (226). The final value of the goodness-of-fit function, $[\sum_H w_H|\Delta F_H|^2]^{\frac{1}{2}}/[n-s]^{\frac{1}{2}}$, is 1·24.

The experimental and the final calculated structure factors are given in Table 3. Scattering factors were

Table 2. *Thermal parameters*

The temperature factor is of the form $\exp[-(B_{11}h^2+B_{22}k^2+B_{33}l^2+B_{12}hk+B_{13}hl+B_{23}kl)]$. All figures are multiplied by 10^5, and standard deviations are given in parentheses.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Cs(1)	1470 (4)	1126 (2)	161 (0·4)	194 (7)	23 (2)	−54 (2)
N(2)	1595 (66)	1180 (41)	234 (8)	104 (85)	484 (37)	147 (32)
N(3)	1711 (73)	1312 (43)	196 (7)	−315 (90)	73 (39)	300 (31)
N(4)	1675 (67)	1662 (49)	200 (8)	−877 (98)	267 (40)	65 (36)
N(5)	2025 (75)	1039 (40)	186 (7)	−112 (91)	−173 (41)	56 (29)
N(6)	2081 (76)	1608 (50)	224 (8)	−438 (111)	404 (43)	194 (37)
N(7)	2307 (79)	1576 (48)	158 (7)	1465 (102)	−133 (42)	1 (33)
C(8)	1455 (68)	657 (33)	161 (7)	181 (82)	256 (37)	51 (28)
C(9)	1329 (65)	586 (32)	152 (7)	40 (77)	171 (37)	82 (26)
C(10)	1607 (68)	681 (34)	165 (7)	−15 (84)	448 (37)	92 (28)
C(11)	837 (53)	570 (30)	158 (7)	−60 (70)	100 (32)	0 (26)
C(12)	1236 (59)	474 (28)	124 (6)	−119 (70)	200 (32)	−57 (23)
C(13)	1208 (60)	591 (31)	142 (6)	−11 (75)	46 (35)	−13 (26)
C(14)	1066 (57)	578 (29)	149 (6)	−226 (75)	112 (34)	13 (26)
C(15)	1149 (56)	435 (27)	133 (6)	−91 (68)	129 (33)	−38 (23)
C(16)	1014 (55)	516 (29)	140 (6)	153 (71)	17 (32)	−28 (24)
C(17)	1572 (67)	872 (35)	140 (6)	−337 (92)	148 (37)	41 (30)
C(18)	1204 (56)	578 (32)	133 (6)	−132 (73)	129 (32)	26 (24)
C(19)	1461 (62)	643 (30)	139 (6)	−374 (82)	184 (34)	−14 (27)
C(20)	1888 (77)	796 (36)	138 (7)	−44 (95)	148 (41)	28 (28)
C(21)	1402 (64)	569 (31)	134 (6)	−46 (78)	143 (36)	−26 (25)
C(22)	1793 (75)	766 (36)	137 (7)	452 (87)	217 (40)	−57 (28)
C(23)	1129 (60)	607 (32)	145 (6)	−117 (75)	78 (35)	−63 (26)
C(24)	1271 (61)	476 (29)	122 (6)	49 (72)	99 (34)	−26 (23)
C(25)	1157 (60)	604 (30)	126 (6)	85 (74)	−40 (34)	−82 (25)

Table 3. *Observed and calculated structure factors*

Each group of four columns contains k, l, $10|F_o|$, and $10F_c$ respectively, and is headed by the value of h common to the group. A negative figure in the $|F_o|$ column gives the observational threshold of an unobserved reflection (omitted from the refinement if $|F_c| < |F_o|$); an asterisk following $|F_o|$ marks a reflection omitted from the final refinement because of extinction error or for some other reason.

		0	
0	6	1177	-1262
0	8	1013	1058
0	10	745	-752
0	12	625	627
0	14	93	-117
0	16	194	-190
0	18	158	159
0	20	352	-348
0	22	295	285
0	24	154	-145
0	26	377	384
0	28	442	-430
0	30	204	186
1	6	1063	1089
1	7	189	170
1	8	1236	-1249
1	9	358	-335
1	10	813	830
1	11	175	166
1	12	1156	-1190
1	13	133	126
1	14	1299	1360
1	15	183	-184
1	16	678	-710
1	17	423	-424
1	18	907	949
1	19	-88	-105
1	20	533	-552
1	21	-82	3
1	22	358	362
1	23	-78	-46
1	24	185	-183
1	25	-76	-15
1	26	137	151
1	27	-78	18
1	28	-78	-52
1	29	-80	-2
1	30	-82	32
2	4	1301	-1297
2	5	758	-706
2	6	1585	1664
2	7	251	233
2	8	1107*	-1036
2	9	84	72
2	10	299	311
2	11	520	484
2	12	381	-361
2	13	263	-253
2	14	76*	104
2	15	185	-183
2	16	107	80
2	17	160	157
2	18	-61	41
2	19	232	226
2	20	648	654
2	21	354	344
2	22	282	-279
2	23	-78	-74
2	24	246	252
2	25	91	90
2	26	255	-248
2	27	-82	-43
2	28	234	227
2	29	-82	46
3	0	-61	-0
3	1	120	-114
3	2	1954*	-2068
3	3	2040*	-2189
3	4	128	130
3	5	91	84
3	6	810	-797
3	7	272	-261
3	8	1042	1031
3	9	375	354
3	10	827	-821
3	11	398	-370
3	12	939	929
3	13	501	482
3	14	954	-966
3	15	326	-324
3	16	653	672
3	17	291	289
3	18	577	-591
3	19	166	-190
3	20	469	478
3	21	-78	73
3	22	251	-256
3	23	145	-141
3	24	257	254
3	25	187	190
3	26	236	-251
3	27	-80	-56
3	28	-80	17
3	29	-80	16
4	0	1522	1544
4	1	705	674
4	2	1480	-1509
4	3	472	-442
4	4	1219	1253
4	5	745	714
4	6	914	-910
4	7	442	-421
4	8	522	540
4	9	152	146
4	10	800	-792
4	11	337	-328
4	12	577	567
4	13	72	78
4	14	93	78
4	15	122	-121
4	16	-74	44
4	17	177	-182
4	18	162	174
4	19	185	200
4	20	173	-171
4	21	185	-197
4	22	295	288
4	23	122	123
4	24	259	-265
4	25	93	-116
4	26	234	241
4	27	95	106
4	28	183	-195
4	29	86	-95
5	0	-80	0
5	1	208	-198
5	2	457	447
5	3	86	-81
5	4	331	-330
5	5	103	116
5	6	335	352
5	7	749	718
5	8	886	-874
5	9	373	-383
5	10	695	703
5	11	349	346
5	12	602	-610
5	13	465	-472
5	14	501	507
5	15	333	334
5	16	490	-494
5	17	288	-284
5	18	478	493
5	19	293	299
5	20	448	-455
5	21	309	-319
5	22	-91	-23
5	23	135	-148
5	24	274	-274
5	25	97	-77
5	26	109	111
5	27	-91	37
5	28	-82	-56
6	0	427	-430
6	1	1147	-1123
6	2	665	666
6	3	743	728
6	4	312	-338
6	6	1008	990
6	7	533	509
6	8	364	-369
6	9	318	-304
6	10	196	203
6	11	194	206
6	12	234	-232
6	13	-69	-90
6	14	-67	22
6	15	-69	-31
6	16	-69	9
6	17	-76	86
6	18	-76	-74
6	19	139	-140
6	20	145	156
6	21	112	117
6	22	183	-193
6	23	-80	-35
6	24	86	120
6	25	175	163
6	26	168	-159
6	27	145	-145
7	0	-65	-0
7	0	-80	-0
7	1	206	208
7	1	215	208
7	2	122	-120
7	3	291	-285
7	4	253	247
7	5	362	356
7	6	318	-323
7	7	362	-375
7	8	244	246
7	9	362	362
7	10	324	-328
7	11	349	-350
7	12	552	567
7	13	520	527
7	14	177	-184
7	15	425	-426
7	16	229	226
7	17	335	331
7	18	267	-269
7	19	288	-285
7	20	215	216
7	21	166	161
7	22	194	-193
7	23	137	-131
7	24	116	117
7	25	-84	88
7	26	-84	-41
8	0	608	619
8	1	575	573
8	2	512	-509
8	3	299	-311
8	4	177	185
8	6	389	-384
8	7	392	-393
8	8	171	166
8	9	211	214
8	10	183	-191
8	12	82	107
8	13	-84	61
8	14	-78	-37
8	15	-78	6
8	16	-76	25
8	17	-91	-7
8	18	-76	33
8	19	164	144
8	20	-78	-56
8	21	179	-194
8	22	-84	73
8	23	187	205
8	24	-80	40
8	25	-84	21
9	0	-76	0
9	1	80	-101
9	2	-69	-97
9	3	149	146
9	4	-69	46
9	5	223	-230
9	6	-69	30
9	7	114	114
9	8	305	-301
9	9	356	-365
9	10	154	151
9	11	322	337
9	12	149	-137
9	13	244	-247
9	14	164	159
9	15	278	291
9	16	179	-152
9	17	217	-218
9	18	147	136
9	19	185	188
9	20	141	-135
9	21	99	-73
9	22	-80	41
9	23	109	107
10	0	-76	-47
10	1	425	-431
10	2	107	80
10	3	318	322
10	4	166	-164
10	5	240	-245
10	6	122	91
10	7	253	249
10	8	126	-125
10	9	189	-189
10	10	-76	76
10	11	105	105
10	12	-74	-13
10	12	-74	-13
10	13	-74	-59
10	14	107	-118
10	15	-74	-56
10	16	103	-119
10	17	-78	-24
10	18	-78	2
10	19	-80	-49
10	20	-80	15
10	21	-84	90
10	22	-80	-13
11	0	-74	0
11	1	162	165
11	2	128	-145
11	3	-74	-48
11	4	-74	3
11	5	194	188
11	6	-76	-52
11	7	162	-161
11	8	-76	19
11	9	219	225
11	10	-76	-11
11	11	185	-191
11	12	84	59
11	13	189	180
11	14	-80	-49
11	15	187	-173
11	16	-80	18
11	17	171	160
11	18	-82	-19
11	19	162	-156
11	20	-82	16
12	0	-76	5
12	1	175	167
12	2	-76	-8
12	3	181	-189
12	4	-76	61
12	5	164	155
12	6	-76	-65
12	7	198	-185
12	8	-76	36
12	9	143	156
12	11	80	-28
12	12	-80	5
12	13	-80	30
12	14	-80	-61
12	15	-80	-19
12	16	-80	-10
12	17	-80	-24
13	0	-76	0
13	1	-78	-19
13	2	-76	-58
13	3	-78	51
13	4	-78	14
13	5	-76	-30
13	6	-78	-12
13	7	101	90
13	8	-80	-8
13	9	116	-102
13	10	-80	-14
13	11	171	169
13	12	-80	-40
13	13	-82	-56
13	14	-82	-56
14	0	-80	-78
14	1	145	-142
14	2	-82	-79
14	3	-82	93
14	4	-78	-3
14	5	101	-70
14	6	-78	-27
14	7	-80	52
		1	
0	6	838	911
0	8	819	-867
0	10	448	474
0	12	392	-388
0	14	-95	-5
0	16	211	205
0	18	181	-180
0	20	554	566
0	22	505	-496
0	24	335	321
0	26	373	-368
0	-6	2126*	2714
0	-8	1438	-1563
0	-10	823	844
0	-12	518	-515
0	-14	196	203
0	-16	-78	-55
0	-18	173	-172
0	-20	238	245
0	-22	282	-289
0	-24	324	315
0	-26	286	-291
0	-28	118	111
1	5	396	-376
1	6	888	-921
1	7	472	439
1	8	1882*	2000
1	9	484	452
1	10	1010	-1047
1	11	368	-346
1	12	1010	1041
1	13	126	111
1	14	1187	-1220
1	15	-82	-40
1	16	981	1014
1	17	360	-369
1	18	196	-185
1	19	341	-358
1	20	625	632
1	21	175	169
1	22	337	-349
1	23	-91	-58
1	24	234	225
1	25	-91	27
1	26	143	-137
1	27	-93	-2
1	-5	352	336
1	-6	1324	1408
1	-7	872	-853
1	-8	1042	-1074
1	-9	528	510
1	-10	581	596
1	-11	200	194
1	-12	1278	-1288
1	-13	253	251
1	-14	975	988
1	-15	109	-121
1	-16	773	-787
1	-17	-86	56
1	-18	701	706
1	-19	168	-210
1	-20	610	-616
1	-21	-97	104
1	-22	501	505
1	-23	-95	-15
1	-24	352	-346
1	-25	-91	6
1	-26	177	175
1	-27	-93	18
1	-28	-91	-64
2	3	499	-467
2	4	964	993
2	4	964	993
2	5	958	924
2	6	1661	-1770
2	7	276	-279
2	8	1250	1264
2	9	149	-127
2	10	488	-493
2	11	-69	-56
2	12	244	262
2	13	152	149
2	14	147	136
2	15	95	94
2	16	223	-220
2	17	158	-148
2	18	152	142
2	19	303	311
2	20	-86	14
2	21	137	150
2	22	444	451
2	23	-97	87
2	24	324	-336
2	25	-97	-97
2	26	297	301
2	27	-97	65
3	1	688	-650
3	2	141	141
3	3	312	305
3	4	853	-842
3	5	335	-312
3	6	821	825
3	7	400	379
3	8	968	-977
3	9	274	-267
3	10	973	973
3	11	312	306
3	12	937	-938
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-1	-12	217	202
-1	-13	-95	91
-1	-14	240	-247
-1	-15	-93	-60
-1	-16	280	278
-1	-17	-93	5
-1	-18	261	-269
-1	-19	-99	30

-1	-20	208	230
-1	-21	-99	15
-2	14	-93	73
-2	-1	-86	94
-2	-2	257	-249
-2	-3	-86	-67
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-2	-6	552	-549
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-2	-8	427	437
-2	-9	-91	88
-2	-10	493	-492
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-2	-17	-97	112
-2	-18	147	-138
-2	-19	-95	5
-2	-20	105	57
-2	-21	-95	28
-3	-1	156	150
-3	-2	322	-325
-3	-3	-88	10
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-3	-5	-88	-9
-3	-6	-91	-87
-3	-7	154	154
-3	-8	-88	30
-3	-9	187	171
-3	-10	-91	79
-3	-11	-93	65
-3	-12	152	-173
-3	-13	137	-133
-3	-14	189	180
-3	-15	112	112
-3	-16	278	-280
-3	-17	147	-150
-3	-18	238	224
-3	-19	122	116
-3	-20	-103	-164
-4	-1	-91	-106
-4	-2	333	312
-4	-3	189	194
-4	-4	423	-430
-4	-5	242	-248
-4	-6	406	402
-4	-7	234	216
-4	-8	293	-289
-4	-9	156	-160
-4	-10	335	332
-4	-11	179	163
-4	-12	295	-297
-4	-13	131	-127
-4	-14	339	324
-4	-15	103	103
-4	-16	200	-189
-4	-17	-97	14
-4	-18	158	148
-4	-19	-99	13
-5	-1	192	-186
-5	-2	253	245

-5	-3	-93	106
-5	-4	198	-172
-5	-5	131	-114
-5	-6	-91	5
-5	-7	-91	-17
-5	-8	-91	3
-5	-9	103	83
-5	-10	-95	-56
-5	-11	-95	-67
-5	-12	-95	59
-5	-13	253	260
-5	-14	246	-250
-5	-15	-95	-20
-5	-16	173	166
-5	-17	133	118
-5	-18	173	-143
-6	-1	257	241
-6	-2	232	-227
-6	-3	181	-178
-6	-4	173	153
-6	-5	267	283
-6	-6	307	-296
-6	-7	-97	-124
-6	-8	261	261
-6	-9	154	176
-6	-10	269	-259
-6	-11	251	-252
-6	-12	-97	81
-6	-13	120	146
-6	-14	147	-143
-6	-15	-97	-62
-6	-16	160	142
-6	-17	126	99
-7	-1	-93	22
-7	-2	183	-182
-7	-3	-97	-92
-7	-4	-93	82
-7	-5	-93	0
-7	-6	-95	-29
-7	-7	101	43
-7	-8	-97	39
-7	-9	-97	-57
-7	-10	-97	17
-7	-11	-97	2
-7	-12	-97	-15
-7	-13	-99	-98
-7	-14	-99	47
-8	-1	-99	-11
-8	-2	291	286
-8	-3	282	276
-8	-4	103	-103
-8	-5	192	-213
-8	-6	147	129
-8	-7	177	219
-8	-8	147	-130
-8	-9	-99	-105
-8	-10	-99	18
-8	-11	244	249
-9	-1	-99	-7
-9	-2	120	103
-9	-3	-103	119
-9	-4	-99	-63
-9	-5	-101	-33
-9	-6	114	49

		8	
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0	2	-88	-106
0	4	-86	71
0	6	-86	-10
0	8	-88	-8
0	10	101	118
0	12	221	-233
0	-2	360	-364
0	-4	343	351
0	-6	427	-460
0	-8	265	268
0	-10	465	-475
0	-12	269	276
0	-14	263	-274
0	-16	105	131
1	0	291	-300
1	1	-93	101
1	2	486	468
1	3	-93	66
1	4	368	-370
1	5	-93	33
1	6	316	315
1	7	152	122
1	8	381	-367
1	9	-93	-36
1	10	280	265
1	11	103	109
1	12	328	-312
2	0	303	-298
2	1	-93	-69
2	2	175	162
2	3	-93	6
2	4	164	-147
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2	9	-95	-53
2	10	152	-139
2	11	-95	-85
2	12	105	146
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3	1	133	139
3	2	314	-308
3	3	168	-153
3	4	305	281
3	5	-95	66
3	6	318	-310
3	7	107	-109
3	8	297	282
3	9	137	142
3	10	326	-321
3	11	-99	-63
4	0	213	209
4	1	-95	92
4	2	120	-129
4	3	124	-127
4	4	95	-83
4	5	-97	-46
4	6	-97	-44
4	7	-97	-26

4	8	137	-143
4	9	-99	55
5	0	213	-200
5	1	149	-155
5	2	240	235
5	3	114	147
5	4	173	-182
5	5	154	-111
5	6	269	261
5	7	179	163
5	8	213	-198
6	0	149	-122
6	1	147	-100
6	2	-99	70
6	3	-99	58
6	4	-99	-75
6	5	-99	-20
6	6	-99	29
6	7	112	87
7	0	189	196
7	1	135	138
-1	-1	-91	38
-1	-2	284	-298
-1	-3	-91	-5
-1	-4	149	140
-1	-5	-93	6
-1	-6	124	-128
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-1	-8	-95	-8
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-1	-14	208	185
-1	-15	-97	-19
-1	-16	105	-109
-1	-17	-99	16
-1	-18	291	290
-2	-1	120	-132
-2	-2	322	300
-2	-3	97	60
-2	-4	366	-359
-2	-5	181	-175
-2	-6	343	332
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-2	-8	385	-380
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-2	-11	-97	74
-2	-12	286	-276
-2	-13	-97	-7
-2	-14	143	148
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-2	-16	309	-286
-2	-17	-101	66
-3	-1	133	-117
-3	-2	221	215
-3	-3	116	96
-3	-4	198	-208
-3	-5	-91	30
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-3	-8	-95	19

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-3	-13	133	147
-3	-14	152	-134
-3	-15	-101	2
-3	-16	139	128
-3	-17	-101	83
-4	-1	116	119
-4	-2	303	-292
-4	-3	131	-136
-4	-4	272	259
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-4	-6	263	-251
-4	-7	122	-132
-4	-8	301	304
-4	-9	101	145
-4	-10	240	-235
-4	-11	126	-138
-4	-12	312	285
-4	-13	-99	53
-4	-14	183	-160
-4	-15	-101	-110
-5	-1	-97	69
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-5	-3	107	-68
-5	-4	114	108
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-5	-7	-93	35
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-5	-10	-95	26
-5	-11	135	143
-5	-12	-99	-117
-5	-13	-99	2
-5	-14	-133	9
-6	-1	-97	-113
-6	-2	-97	128
-6	-3	166	158
-6	-4	107	-137
-6	-5	206	-204
-6	-6	248	242
-6	-7	158	143
-6	-8	200	-207
-6	-9	158	-177
-6	-10	120	84
-6	-11	124	127
-6	-12	208	-205
-6	-13	-101	-89
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-7	-2	116	70
-7	-3	-99	76
-7	-4	-101	-52
-7	-5	-99	-38
-7	-6	105	74
-7	-7	-101	-8
		9	
0	0	154	-178

0	2	215	216
0	4	-91	-6
0	-2	152	175
0	-4	229	-225
0	-6	202	208
0	-8	187	-218
0	-10	373	373
0	-12	198	-218
1	0	204	207
1	1	166	154
1	2	223	-226
1	3	-97	44
1	4	324	309
1	5	122	97
3	0	158	-153
3	1	-99	-69
3	2	267	256
3	3	-103	72
4	0	-103	-109
-1	-1	-95	-45
-1	-2	265	260
-1	-3	-93	34
-1	-4	208	-215
-1	-5	-97	-5
-1	-6	-101	85
-1	-7	-99	-32
-1	-8	-99	-14
-1	-9	-99	12
-1	-10	-101	17
-1	-11	-99	-74
-1	-12	-101	69
-2	0	-97	70
-2	1	-97	-76
-2	2	145	-166
-2	3	-97	63
-2	4	-95	-11
-2	-1	-97	-54
-2	-2	251	-238
-2	-3	-97	-64
-2	-4	291	289
-2	-5	128	92
-2	-6	324	-333
-2	-7	109	-71
-2	-8	236	233
-2	-9	-101	65
-2	-10	225	-233
-2	-11	-101	-61
-3	-1	-97	5
-3	-2	206	-183
-3	-3	-99	-23
-3	-4	-105	161
-3	-5	-99	-10
-3	-6	-99	-30
-3	-7	105	-91
-3	-8	156	142
-3	-9	-99	-14
-3	-10	-99	36
-4	-1	-99	-101
-4	-2	-99	80
-4	-3	-99	74
-4	-4	128	-142
-4	-5	154	-151
-4	-6	221	217
-4	-7	122	97

taken from *International Tables for X-ray Crystallography*, except that for Cs^+, which was taken from the tabulation of Thomas & Umeda (1957) and changed to account for anomalous dispersion (*International Tables for X-ray Crystallography*, 1962, p.216).

Discussion

This refinement has confirmed the general similarity between the packing of neighboring $TCNQ^-$ ions in *N*-methylphenazinium TCNQ and that of centric-noncentric vicinal pairs in the $Cs_2(TCNQ)_3$ structure. (For convenience in description, molecules lying on centers of symmetry are called centric and those not on centers, noncentric. Molecules of both kinds have nearly *mmm* symmetry). Fig. 1, the [100] projection, provides a general view of the columnar packing; Fig. 2 shows the configuration of a neighboring centric-noncentric pair. Although the mixed pairs in $Cs_2(TCNQ)_3$ and pairs in *N*-methylphenazinium TCNQ share a longitudinal translation just sufficient to center one ring over a quinomethan double bond of the next ring, refinement has shown that lateral translation of about 0·35 Å is also present in the cesium salt. The separation of adjacent molecules of mixed type in $Cs_2(TCNQ)_3$ is about 3·22 Å in the region of overlap. The separation of these nonparallel and even imperfectly planar molecules is ill-defined. The value adopted is the dot product of the vector between molecular centers with the average unit normal vector. The mutual inclination of centric and noncentric molecules within a column is 2·7°.

An important change from the earlier two-dimensional study is the revelation that adjacent noncentric molecules are separated by 3·26 Å rather than 3·44 Å. The smaller spacing implies charge-resonance interaction and it is therefore of interest to examine the steric relationship of the pair. Fig. 3 is a view of a noncentric pair in a direction perpendicular to the molecular planes. This packing relationship is quite unlike that of a mixed pair, and can be considered a distortion from direct superposition. A complete discussion of the two probable energy minima represented by these packing modes and of calculated distortion barriers will be deferred until later (Fritchie, Chesnut & Simmons, 1966). It suffices here to call attention to the remarkable similarity of packing in the two very dissimilar salts, $Cs_2(TCNQ)_3$ and *N*-methylphenazinium TCNQ.

The accuracy of this structure determination permits meaningful discussion of bond lengths within the TCNQ molecules and leads to a rather unexpected result. Examination of the bond lengths, shown in Fig. 4, reveals that each TCNQ molecule possesses D_{2h} (*mmm*) symmetry within experimental error, but that the two molecules differ. (As noted below, D_{2h} symmetry applies only to the bond lengths; the molecules are somewhat non-planar.) Bond lengths, averaged under D_{2h} symmetry, are compared in Table 4 with average values for $TCNQ^\circ$ and for $TCNQ^-$ obtained from TCNQ, *N*-methylphenazinium TCNQ, and potassium TCNQ. Although the differences between $TCNQ^\circ$ and $TCNQ^-$ are small, there can be little doubt that they are real. Comparison of the noncentric molecule in $Cs_2(TCNQ)_3$ with $TCNQ^-$ and the centric molecule with $TCNQ^\circ$ reveals a remarkable coincidence, better in fact than could be expected on the basis of the estimated standard deviations. It thus seems quite likely that the negative charges within $Cs_2(TCNQ)_3$ are localized predominantly on the noncentric molecules. This apparent charge localization is unexpected, but the vicinal packing can be rationalized on this basis (Fritchie, Chesnut & Simmons, 1966).

Least-squares planes through the two TCNQ molecules were calculated (Blow, 1960) and are described in Table 5. As in *N*-methylphenazinium TCNQ and to a much smaller extent in TCNQ the molecules are not quite planar. The major distortions in the cesium salt seem to be twisting of the central ring with respect to the dicyanomethylene groups, but out-of-plane folding is also present. Each dicyanomethylene group (including the attached ring carbon) is itself non-planar, although the rings and attached methylene carbons are planar. These distortions must be ascribed primarily to the very rigid and close packing within TCNQ columns.

The coordination polyhedron about the cesium ion, just as that about potassium in potassium TCNQ, is approximately a cube composed of nitrogen atoms from four adjacent TCNQ columns. The eight Cs–N contacts are 3·38, 3·33, 3·36, 3·13, 3·30, 3·07, 3·40 and 3·49 Å. In the potassium salt the coordination cubes lie flat above one another in a column; however, as Fig. 1 shows, the cubes in $Cs_2(TCNQ)_3$ are 'hinged' on opposite sides of the column and the column is opened so that wedge-shaped spaces appear between adjoining cubes.

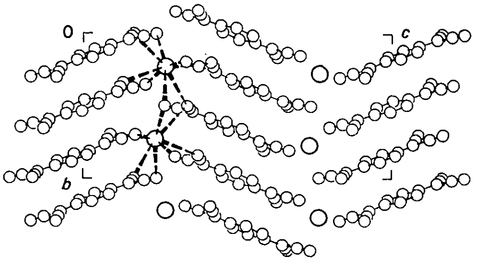


Fig. 1. [100] projection. All TCNQ molecules are centered near $x=0$, all cesium ions near $x=\frac{1}{2}$. The cesium coordination is roughly cubic, involving TCNQ molecules at both $x=0$ and $x=1$.

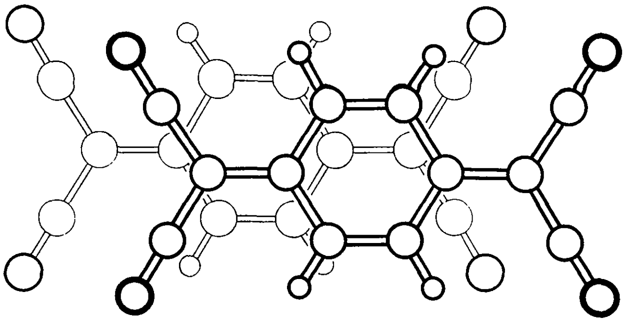


Fig. 2. Nearest-neighbor packing of centric-noncentric pairs of TCNQ molecules, viewed in a direction perpendicular to the mean molecular planes.

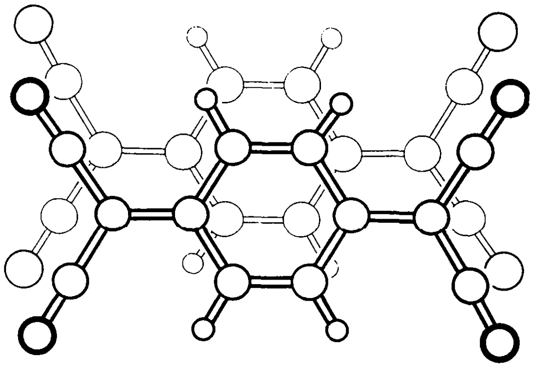


Fig. 3. Nearest-neighbor packing of pairs of noncentric TCNQ molecules, viewed in a direction perpendicular to the mean molecular plane.

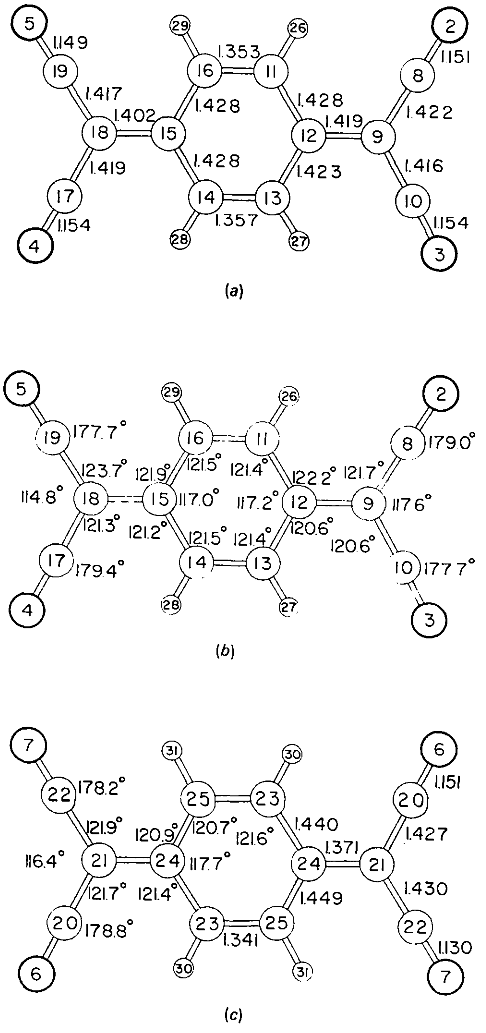
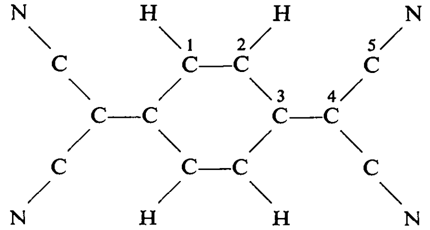


Fig. 4. Bond lengths and angles. (*a*) Bonds in a noncentric molecule; (*b*) angles in a noncentric molecule; (*c*) bonds and angles in a centric molecule.

Table 4. *Comparison of mean bond lengths*

The numbering system is that used in *N*-methylphenazinium TCNQ and is illustrated below. Figures in parentheses are standard deviations of the means. No librational corrections have been applied.



	TCNQ⁻			TCNQ°	'Noncentric'	'Centric'
Bond	$(C_{13}H_{11}N_2)^+$ TCNQ*	K(TCNQ)†	Average	TCNQ‡	TCNQ in $Cs_2(TCNQ)_3$	TCNQ in $Cs_2(TCNQ)_3$
C(1)–C(2)	1·341 (7)	1·37 (1)	1·351 (6)	1·344 (3)	1·355 (4)	1·341 (5)
C(2)–C(3)	1·434 (5)	1·42 (1)	1·431 (5)	1·442 (3)	1·427 (3)	1·444 (4)
C(3)–C(4)	1·388 (7)	1·40 (1)	1·392 (6)	1·373 (3)	1·410 (4)	1·371 (5)
C(4)–C(5)	1·420 (5)	1·41 (1)	1·418 (5)	1·436 (3)	1·419 (3)	1·428 (4)
C(5)–N	1·156 (5)	1·13 (1)	1·151 (5)	1·138 (2)	1·152 (3)	1·140 (4)

* Fritchie (1966) (*N*-methylphenazinium TCNQ).
† Anderson & Fritchie (1963).
‡ Long, Sparks & Trueblood (1965).

Table 5. *Least-squares planes*

The planes are described using an orthonormal vector basis $\{\mathbf{m},\mathbf{n},\mathbf{p}\}$ having $\mathbf{n} \parallel \mathbf{b}$ and $\mathbf{p} \parallel \mathbf{c}^*$.

Plane 1 $0{\cdot}15153m-0{\cdot}92945n-0{\cdot}33638p=-1{\cdot}6253$
Plane 2 $0{\cdot}18402m-0{\cdot}92938n-0{\cdot}31997p=-1{\cdot}5632$
Plane 3 $0{\cdot}19389m-0{\cdot}92813n-0{\cdot}31779p=-4{\cdot}8284$
Plane 4 $0{\cdot}21735m-0{\cdot}92844n-0{\cdot}30126p=-4{\cdot}8297$

	Plane 1		Plane 2			Plane 3		Plane 4	
	Weight	Deviation*	Weight	Deviation*		Weight	Deviation*	Weight	Deviation*
N(2)	7	−117		227	N(6)	7	3		−31
N(3)	7	6		56	N(7)	7	32		120
N(4)	7	−84		−56	C(20)	6	−19		−30
N(5)	7	−47		−175	C(21)	6	−17	6	1
C(8)	6	−42		−119	C(22)	6	−10		47
C(9)	6	27	6	−8	C(23)	6	31	6	1
C(10)	6	9		21	C(24)	6	−12	6	−3
C(11)	6	86	6	1	C(25)	6	−38	6	1
C(12)	6	37	6	−1					
C(13)	6	5	6	8					
C(14)	6	0	6	0					
C(15)	6	48	6	3					
C(16)	6	95	6	7					
C(17)	6	−33		39					
C(18)	6	39	6	−10					
C(19)	6	11		−81					

* Deviations are given in 10^{-3} Å.

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