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Crystal and Molecular Structure of 1-Bromotriptycene $BrC_{20}H_{13}$

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The crystal structure of 1-bromotriptycene, $BrC(C_6H_4)_3CH$, has been determined by X-ray diffraction. The space group is $R\bar{3}$. The hexagonal unit cell contains six molecules and has the dimensions $a=11{\cdot}852$ Å, $c=17{\cdot}574$ Å. The final value for R is 12%. Lack of better agreement is believed to be due to molecular disorder in the crystal. The bridging C–C bonds have lengths 1·53 Å at the bromine end and 1·51 Å at the hydrogen end. These distances and the bond angles at the bridges indicate that there is no conjugation between benzene rings.

Electric dipole moments of isomeric triptycene derivatives (Ogura, Toshiyasu, Kimura, Fujishiro & Nakagawa, 1964) and the infrared spectrum of triptycene (Bartlett & Greene, 1954; Theilacker, Albrecht & Uffmann, 1965) support the conclusion that triptycene is a highly symmetrical and rigid molecule. The ultraviolet spectra of triptycene and a number of its derivatives led Bartlett & Lewis (1950) to conclude that inter-ring resonance occurs. On the other hand, Wilcox (Wilcox, 1960; Wilcox & Craig, 1961) concluded on the basis of quantum mechanical calculations that interaction between benzene rings is negligible. The present investigation was undertaken to determine the length of the bridge bonds and thereby evaluate their bond character. In addition, 1-bromotriptycene, shown in Fig. 1, cannot form without considerable deviation from normal values of one or more of the angles made by the bridge bonds. This study shows which bonds are strained and by what amount.

Experimental

The 1-bromotriptycene used in this investigation was obtained from Prof. A. Streitwieser, who had received it from Prof. L. Friedman. It was synthesized by the method of Friedman & Logullo (1963). Dr E. Ziegler checked the purity of this material by passing it through a gas–liquid chromatographic column. He found only one peak.

Crystals of 1-bromotriptycene suitable for X-ray analysis were prepared by sublimation at approximately 250°. The crystals were rhombohedral in shape. In polarized light the extinction is symmetrical and the birefringence is high. c-Axis views showed a uniaxial interference figure of positive character.

Refractive indices obtained by immersion methods with white light are only approximate because the crystals dissolved in the immersion liquids. A crystal lying on a rhombohedral face shows ω and ε'; an edge view shows ω and ε (Fig. 2). Approximate values for these indices are: $\omega=1{\cdot}71$, $\varepsilon'=1{\cdot}74$ and $\varepsilon=1{\cdot}81$.

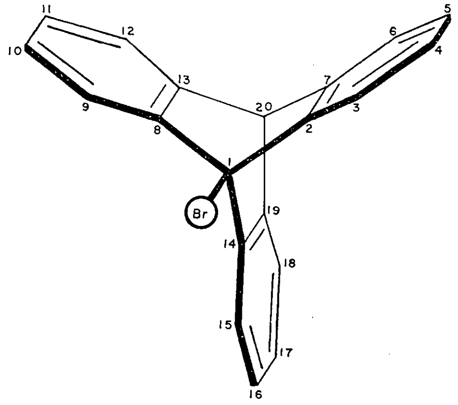


Fig. 1. 1-Bromotriptycene molecule with numbering system used in this investigation. The asymmetric unit is $(BrC_2)_{1/3}C_6$.

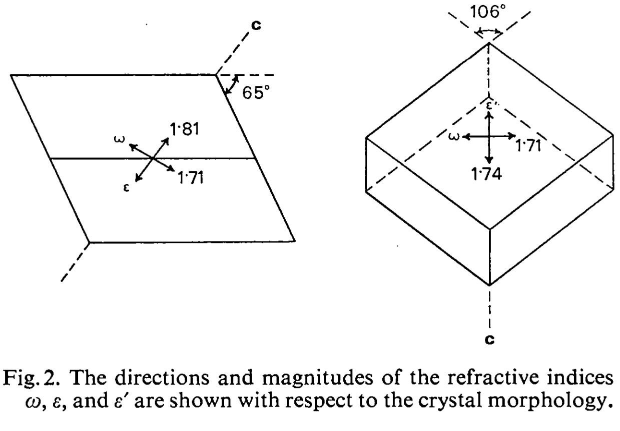


Fig. 2. The directions and magnitudes of the refractive indices ω, ε, and ε' are shown with respect to the crystal morphology.

Multiple level Weissenberg and precession photographs were taken. The precession photographs are compatible with a Laue symmetry of $\bar{3}$. Reflections were indexed on a hexagonal lattice and were found to obey the rule $(-h+k+l)=3n$. The primitive lattice is therefore rhombohedral and the orientation of the rhombohedral lattice in the hexagonal lattice is obverse.

The observed density of 1·52 g.cm^{-3} requires six molecules per hexagonal cell or two per rhombohedral cell. The most probable space group, therefore, is $R\bar{3}$, and the asymmetric unit is $C_6H_4(C_2HBr)_{1/3}$.

Unit-cell parameters and intensity data were obtained with a diffractometer equipped with a quarter circle goniostat, solid state radiation analyzer and scintillation counter.

2θ scans with a 1° take-off angle for all observable orders of $h0.0$ and $00.l$ led to the following hexagonal unit cell data: $a=11{\cdot}852$ (5) Å, $c=17{\cdot}574$ (9) Å, $V=$ 2,138·0 Å^3, $Z=6$, ϱ calc. $=1{\cdot}54$ g.cm^{-3}, ϱ obs. $=1{\cdot}52$ g.cm^{-3}. Estimated standard deviations ($\times 10^3$) are given in parenthesis.

Intensity data were collected by counting for 10 sec with a stationary crystal and stationary counter at a take-off angle of 4°. The background was measured by counting for 10 sec at the minimum between the $K\beta$ and $K\alpha$ peaks. A total of 1050 independent reflections were measured (maximum $2\theta=150°$), 35 of which were assigned zero intensity. Cu $K\alpha$ X-rays ($\lambda=$ 1·5418 Å) filtered with Ni foil were used. Lorentz and polarization corrections were applied by means of a program developed by A. Zalkin.

The crystal used for intensity measurements had dimensions of 0·088 mm × 0·088 mm × 0·118 mm. The absorption factor for this crystal is 35·0 cm^{-1} and μR is about 0·21 or less, making the absorption quite low. No corrections were applied to the data for either absorption or extinction.

Determination of the structure

The three-dimensional Patterson function showed many well-resolved peaks. Since the 1-bromotriptycene molecule has threefold symmetry and there are six molecules in the hexagonal cell, the bromine and the two aliphatic carbon atoms were assumed to occupy the special positions (c) with parameters $(0,0,\pm z)$, $(\frac{2}{3},\frac{1}{3},\frac{1}{3}\pm z)$, and $(\frac{1}{3},\frac{2}{3},\frac{2}{3}\pm z)$. A straightforward interpretation of the Patterson function led to the z coordinates of the bromine atom and the carbon atom bonded to it. These two parameters and the isotropic temperature factor B for each atom, of the form $\exp(-B\sin^2\theta.\lambda^{-2})$, were refined by least squares. The function minimized in the least squares calculation was $\Sigma\, w(|F_o|-|F_c|)^2/\,\Sigma\, w|F_o|^2$, where w is the weighting factor and F_o and F_c are the observed and calculated structure factors. Atomic scattering factors for neutral bromine and carbon were taken from *International Tables for X-ray Crystallography* (Ibers, 1962). The values for bromine were corrected for dispersion by subtracting 1·0 electron (Templeton, 1962). The imaginary part of the dispersion correction was neglected.

After four cycles the residual R value, defined as $R=\Sigma\,||F_o|-|F_c||/\,\Sigma\,|F_o|$, was 0·443. A three-dimensional electron density map was then calculated with use of signs determined by the carbon and bromine atoms. The resulting map permitted the evaluation of the parameters of the remaining seven carbon atoms in the asymmetric unit. The parameters of these nine atoms were refined by four cycles of least squares. This reduced R to 0·161. Bromine was then assigned an anisotropic temperature factor; four more cycles of least-squares refinement reduced R to 0·156.

A detailed comparison was then made between calculated and observed $|F|$ values. Four reflections had large discrepancies, and many more had discrepancies which seemed outside experimental error. As a result the intensities of 125 reflections were remeasured on the same crystal. Outside of the four obvious errors, no significant discrepancies between measured intensities were found. After correction of the intensities of the four reflections the bromine and eight carbon atoms were given anisotropic temperature factors and their parameters refined with four cycles of least squares. This reduced R to 0·139. The temperature factors for all atoms were satisfactorily small except for the B_{11} and B_{22} terms of bromine, which were 5·3, and the B_{33} term of C(4), which was 6·65.

The positions of the five hydrogen atoms were then calculated and included in the next refinement. The hydrogen coordinates were held fixed but the isotropic temperature factors were allowed to vary. This reduced R to 0·128.

Since R still seemed high, three least-squares refinements were calculated in which a portion of the experimental data was eliminated. In the first calculation zero weight was given to those reflections which had an appreciable discrepancy between calculated and observed structure factors. Thirty-two reflections were selected, and after two cycles of least-squares refinement R was 0·113. In the second and third calculations all low angle data below $\sin\theta/\lambda=0{\cdot}30$ and all high angle data above $\sin\theta/\lambda=0{\cdot}531$ were eliminated, respectively. After two cycles of least-squares refinement R became 0·116 when the low angle data were not used and 0·124 when the high angle data were not used. Although the first two procedures led to a small improvement in R the improvement did not seem sufficiently dramatic to warrant eliminating a portion of the experimental data.

Fourier calculations of the electron density showed a peak at 0,0,0·46 about half as high as a carbon atom. Because of space limitations this position is impossible for any plausible chemical impurity which we have been able to invent. Attempts to identify an error in the data which would produce the peak also failed. We suggest that this peak is the result of disorder of some kind, but we have failed to find a completely satisfactory model of the details of the disorder.

Least-squares calculations with one-tenth of a bromine atom, or with a carbon atom at that position, improved the agreement slightly and at the same time shifted other atoms by small amounts. The differences in coordinates found in these refinements suggest that standard deviations of positions should be stated as about 0·02 Å, rather than the values given in Table 1, to include the uncertainty introduced by our ignorance of the nature of the disorder.

Since no significant improvement could be effected by the procedures described above and no appreciable discrepancies could be found in the experimental data it was decided to terminate the refinement.

The positional and thermal parameters and their calculated standard deviations are listed in Tables 1 and 2 respectively for all atoms except hydrogen. These parameters were obtained with use of all data, not refining the positional parameters of the hydrogen atoms, and holding the temperature parameters of the hydrogen atoms equal, but allowing them to be refined. The R value of this refinement was 0·123. The observed and calculated structure factors are shown in Table 3.

Table 1. *Final positional parameters and standard deviations* in parentheses* × 10^4

	x	y	z
Br	0·0	0·0	0·1052 (1)
C(1)	0·0	0·0	0·2173 (7)
C(20)	0·0	0·0	0·3595 (8)
C(2)	0·1212 (7)	0·1184 (7)	0·2490 (5)
C(3)	0·2241 (8)	0·2153 (9)	0·2099 (6)
C(4)	0·3275 (9)	0·3147 (8)	0·2525 (7)
C(5)	0·3249 (8)	0·3146 (8)	0·3291 (6)
C(6)	0·2207 (8)	0·2154 (9)	0·3679 (6)
C(7)	0·1198 (7)	0·1170 (7)	0·3282 (5)
H(20)†	0·0	0·0	0·420
H(3)†	0·225	0·215	0·150
H(4)†	0·405	0·395	0·220
H(5)†	0·400	0·390	0·360
H(6)†	0·225	0·215	0·430

* The standard deviations listed are those estimated by least squares. Because of disorder, as described in the text, the uncertainty is judged to be about twice as great as indicated by these values.

† These parameters not refined.

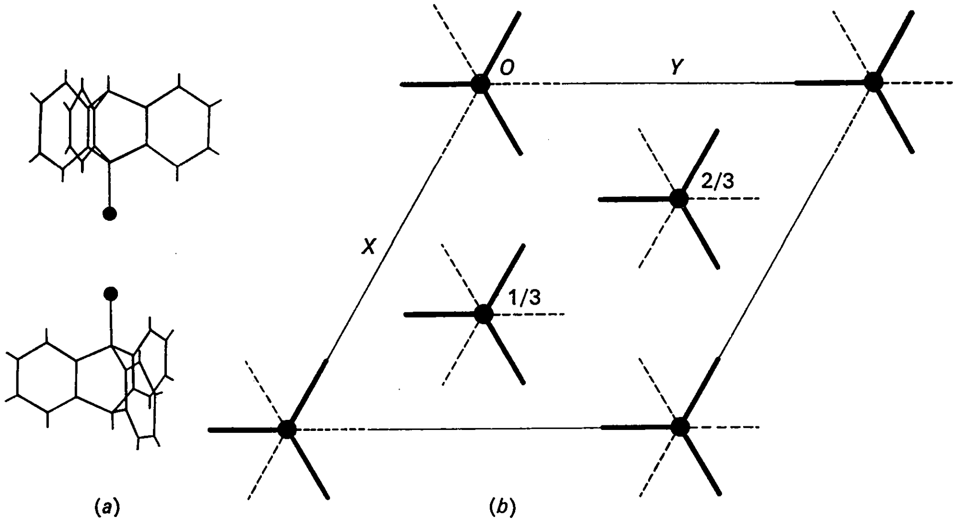
Description of the structure

The structure consists of three non-polar molecular doublets arranged on the three threefold inversion axes

Table 2. *Anisotropic thermal parameters* (Å²) *and standard deviations in parentheses* × 10^2

The temperature factors are in the form exp $(-\Sigma\, \beta_{ij}h_ih_jb_ib_j/4)$, where b_ib_j are reciprocal cell lengths.

	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br	4·78 (7)	B11	3·73 (9)	B11/2	0·0	0·0
C(1)	1·72 (31)	B11	2·38 (53)	B11/2	0·0	0·0
C(20)	1·58 (30)	B11	2·94 (58)	B11/2	0·0	0·0
C(2)	1·31 (27)	1·46 (29)	3·78 (38)	0·74 (23)	−0·16 (25)	−0·12 (26)
C(3)	2·32 (35)	2·51 (3i)	4·48 (46)	1·33 (30)	−0·08 (32)	−0·53 (32)
C(4)	2·31 (36)	1·63 (32)	6·80 (61)	1·16 (29)	0·36 (37)	−0·39 (35)
C(5)	1·95 (34)	2·09 (34)	4·97 (47)	1·07 (29)	0·42 (32)	0·91 (32)
C(6)	1·62 (22)	2·97 (25)	4·51 (33)	1·38 (20)	0·17 (21)	0·88 (23)
C(7)	1·17 (27)	1·48 (29)	3·53 (36)	0·46 (23)	−0·15 (25)	0·18 (25)



(*a*) (*b*)

Fig. 3. (*a*) The stacking arrangement of two 1-bromotriptycene molecules about a center of symmetry. (*b*) The packing arrangement as viewed down the c axes. The numbers refer to the z coordinate of the inversion center.

of the hexagonal cell. The two molecules of each doublet are related by a center of symmetry. The orientation of the benzene rings is shown schematically in Fig. 3(*a*). The heavy lines in Fig. 3(*b*) represent the benzene rings of one molecule and the dashed lines the benzene rings of the molecule related to it by the center of symmetry. The solid circle represents bromine.

The interatomic distances and bond angles are given in Tables 4 and 5, and in Fig. 4. It is interesting to compare the values of the bond angles at C(1) and C(2) because these angles cannot all have their normal value in triptycene. The angles about C(1) are within 2° of the tetrahedral value whereas the angles at C(2) are: C(1)–C(2)–C(7) 110·7°; C(1)–C(2)–C(3) 128·5°; and C(3)–C(2)–C(7) 120·7°. The two angles external to the benzene ring are distorted approximately 9° from the usual value of 120°, which can be interpreted as evidence that these bond angles are more easily distorted than are the sp^3 tetrahedral bonds at C(1).

The variation in the observed bond angles and distances in the benzene ring may be due to the strain caused by the distortion of the C(1)–C(2)–C(3) bond angle, but is more likely a result of the molecular disorder mentioned above.

The C–Br bond distance (1·97 Å) is in excellent agreement with that determined by Robertson & Sheldrick (1965) in α-methyl D-galactoside 6-bromohydrin of 1·974 Å. In both cases bromine is bonded

Table 3. *Observed and calculated structure factors* (× 10) *of* 1-*bromotriptycene*

H,K	L	FOB	FCA
-12, 12	3	30	-11
	6	19	-9
-12, 13	2	0	-15
	5	9	-15
-11, 11	2	28	9
	5	60	-52
	8	0	4
	11	20	15
-11, 12	1	44	-65
	4	73	-87
	7	0	-1
-11, 13	3	42	-38
	6	76	-86
-11, 14	2	70	84
-10, 10	1	54	61
	4	93	-89
	7	116	-128
	10	40	20
	13	26	-42
-10, 11	3	0	24
	6	74	-62
	9	71	63
	12	21	-13
-10, 12	2	121	128
	5	76	-69
	8	96	101
	11	53	70
-10, 13	1	45	-46
	4	71	-78
	7	25	-24
-10, 14	3	0	24
-9, 9	3	85	86
	6	74	-68
	9	235	227
	12	23	43
	15	18	-26
-9, 10	2	180	-182
	5	76	-75
	8	102	105
	11	33	15
	14	80	-78
-9, 11	1	71	70
	4	86	-92
	7	28	-28
	10	55	55
	13	35	-41
-9, 12	3	28	37
	6	57	-43
	9	74	76
-9, 13	2	37	-40
	5	31	-28
	8	65	66
-9, 14	1	0	-4
	4	87	-101
-8, 8	2	133	-137
	5	125	-130
	8	91	99
	11	35	17
	14	115	-93
	17	0	2
-8, 9	1	36	-9
	4	118	-130
	7	128	-135
	10	43	33
	13	63	-70
	16	0	-17
-8, 10	3	100	-95
	6	60	-60
	9	170	165
	12	0	2
	15	55	-65
-8, 11	2	0	3
	5	125	-128
	8	0	-17
	11	50	33
	14	41	-31
-8, 12	1	79	83
	4	110	-112
	7	33	-41
	10	47	48
-8, 13	3	93	-90
	6	54	-50
	9	74	71
-8, 14	2	21	-26
	5	0	7
-7, 7	1	195	212
	4	127	-145
	7	65	-70
	10	58	65
	13	95	-91
	16	29	-16
-7, 8	3	34	27
	6	176	-182
	9	179	171
	12	29	29
	15	50	-55
-7, 9	2	27	13
	5	181	-171
	8	108	116
	11	64	52
	14	106	-84
	17	0	9
-7, 10	1	255	270
	4	102	-107
	7	36	-43
	10	75	78
	13	70	-68
-7, 11	3	100	-78
	6	21	16
	9	87	84
	12	49	-40
-7, 12	2	88	81
	5	58	-44
	8	47	44
	11	47	47
-7, 13	1	114	112
	4	23	-14
	7	22	-31
	10	84	91
-7, 14	3	45	-31
	6	0	-11
-6, 6	3	57	23
	6	133	-142
	9	306	293
	12	28	55
	15	73	-71
	18	53	55
-6, 7	2	38	-27
	5	260	-265
	8	190	201
	11	40	18
	14	193	-175
	17	18	-2
-6, 8	1	35	45
	4	95	-118
	7	33	10
	10	125	134
	13	25	-16
	16	46	-47
-6, 9	3	40	-5
	6	136	-134
	9	203	198
	12	32	41
	15	57	-56
-6, 10	2	69	-74
	5	118	-116
	8	48	38
	11	49	27
	14	75	-54
-6, 11	1	204	210
	4	101	-105
	7	25	-18
	10	165	167
	13	27	31
-6, 12	3	89	-73
	6	147	-143
	9	93	86
	12	31	-22
-6, 13	2	30	-37
	5	63	-68
	8	34	32
-6, 14	1	64	70
	4	58	-54
	7	0	-18
-5, 5	2	62	-35
	5	80	-38
	8	164	182
	11	176	148
	14	46	-14
	17	24	16
	20	37	42
-5, 6	1	90	10
	4	340	-347
	7	127	-119
	10	141	161
	13	128	-137
	16	64	-70
	19	88	100
-5, 7	3	210	-194
	6	282	-276
	9	187	177
	12	74	-51
	15	132	-144
	18	76	68
-5, 8	2	99	74
	5	130	-149
	8	131	135
	11	88	64
	14	72	-54
	17	40	30
-5, 9	1	91	128
	4	198	-203
	7	129	-141
	10	146	143
	13	52	-55
	16	40	-41
-5, 10	3	241	-237
	6	118	-115
	9	148	144
	12	175	-166
	15	134	-163
-5, 11	2	52	35
	5	44	-33
	8	32	22
	11	40	-44
	14	41	-27
-5, 12	1	77	91
	7	0	3
	4	20	-21
	10	85	93
-5, 13	3	104	-95
	6	67	-67
	9	75	77
-5, 14	2	11	-25
	5	0	12
-4, 4	1	283	330
	4	451	-454
	7	305	-277
	10	257	283
	13	92	-120
	16	85	-79
	19	133	140
-4, 5	3	100	-66
	6	146	-160
	9	182	175
	12	221	-190
	15	108	-119
	18	87	79
-4, 6	2	322	266
	5	295	-295
	8	164	174
	11	301	275
	14	65	-34
	17	47	40
	20	61	71
-4, 7	1	189	231
	4	326	-326
	7	57	-50
	10	118	133
	13	170	-179
	16	84	-87
	19	61	63
-4, 8	3	86	-66
	6	82	-82
	9	130	129
	12	156	-134
	15	92	-98
	18	54	48
-4, 9	2	66	40
	5	36	4
	8	116	126
	11	145	127
	14	19	46
	17	21	22
-4, 10	1	270	282
	7	95	-101
	4	105	-112
	10	172	173
	13	23	19
	16	22	-17
-4, 11	3	115	-100
	6	43	-38
	9	95	83
	12	70	-60
-4, 12	2	62	57
	5	59	-52
	8	50	47
	11	59	71
-4, 13	1	73	72
	4	82	-84
	7	84	-98
-4, 14	3	10	9
-3, 3	3	108	31
	6	237	-247
	9	481	444
	12	40	53
	15	116	-125
	18	97	93
	21	18	3
-3, 4	2	197	145
	5	228	-273
	8	278	284
	11	219	180
	14	118	-103
	17	35	18
	20	65	51
-3, 5	1	114	192
	4	576	-554
	7	72	-58
	10	187	224
	13	237	-256
	16	123	-129
	19	94	91
-3, 6	3	122	87
	6	54	-19
	9	232	226
	12	55	80
	15	25	-20
	18	55	57
-3, 7	2	143	104
	5	308	-302
	8	128	134
	11	225	205
	14	81	-60
	17	53	41
-3, 8	1	77	100
	4	265	-270
	7	88	-94
	10	156	166
	13	92	-93
	16	69	-75
-3, 9	3	67	-38
	6	119	-129
	9	209	202
	12	28	0
	15	64	-67
-3, 10	2	65	-68
	5	170	-168
	8	21	15
	11	46	23
	14	84	-76
-3, 11	1	171	186
	7	14	-13
	4	121	-143
	10	70	69
	13	70	-86
-3, 12	3	20	21
	6	23	-18
	9	85	84
-3, 13	2	13	4
	5	21	-19
	8	57	69
-3, 14	1	27	-54
-2, 2	2	97	-59
	5	459	-510
-0, 3	0	1196	1184
2, 1	1	961	1015
-0, 1	2	568	-496
1, 1	0	682	686
-2, 2	8	193	225
	11	152	119
	14	233	-217
	17	50	41
	20	79	69
-2, 3	1	325	-108
	4	267	-340
	7	63	-20
	10	184	230
	13	96	-124
	16	96	-101
	19	92	96
-2, 4	3	463	-378
	6	419	-413
	9	254	247
	12	130	-96
	15	153	-184
	18	125	111
	21	39	-11
-2, 5	2	366	281
	5	346	-351
	8	141	157
	11	287	255
	14	89	-64
	17	53	46
	20	83	76
-2, 6	1	125	184
	4	484	-466
	7	43	-41
	10	109	135
	13	242	-265
	16	93	-101
	19	79	74
-2, 7	3	229	-203
	6	228	-230
	9	181	176
	12	85	-63
	15	119	-140
	18	77	60
-2, 8	2	75	46
	5	177	-183
	8	119	135
	11	25	-6
	14	128	-118
	17	29	13
-2, 9	1	88	111
	7	28	-31
	4	88	-93
	10	70	64
	13	63	-60
	16	30	-24
-2, 10	3	120	-111
	6	33	-27
	9	160	158
	12	23	16
	15	54	-66
-2, 11	2	21	-6
	5	116	-111
	8	31	22
	11	39	30
-2, 12	1	66	-68
	4	70	-69
	7	18	-12
	10	18	10
-2, 13	3	45	-38
	6	57	-66
-1, 1	1	164	277
	4	402	-463
	7	373	-332
	10	147	208
	13	133	-165
	16	57	-44
	19	139	149
-1, 2	3	149	83
	6	525	-499
	9	299	288
	12	51	-10
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	14	211	-189
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	20	62	46
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	19	106	108
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	9	146	150
	12	194	-169
	15	91	-109
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	11	215	196
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	17	33	24
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	6	201	-201
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	12	20	30
	15	58	-61
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	14	90	-73
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	10	64	59
	13	34	-28
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	12	23	-18
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	13	134	-160
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	19	104	107
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	5	80	-121
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	11	227	197
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	17	28	16
	20	63	50
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	18	69	67
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	8	152	158
	11	45	26
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	17	32	6
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	4	149	-156
	7	123	-123
	10	48	42
	13	75	-79
	16	33	-27
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	15	34	-26
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	5	145	-151
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	14	86	-77
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	8	0	-11
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	12	181	-155
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	11	118	104
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	17	15	-3
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	10	57	47
	13	75	-81
	16	30	-47
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	3	88	-77
	6	152	-149
	9	80	61
	12	47	-40
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	5	56	-50
	8	58	55
	11	11	8
4, 9	1	66	64
	4	65	-53
	7	69	-80
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	3	85	-102
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	5	68	-38
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	11	172	148
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	17	0	16
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	10	250	275
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	16	50	-41
	19	85	95
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	3	152	-130
	6	107	-114
	9	186	181
	12	86	-62
	15	110	-118
	18	69	55
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	5	90	-112
	8	75	87
	11	107	86
	14	45	-17
	17	47	38
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	4	236	-236
	7	145	-157
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	13	70	-64
	16	55	-55
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	3	19	10
	6	55	-55
	9	65	48
	12	130	-121
	15	43	-40
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	11	138	139
	14	0	25
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	4	113	-115
	7	27	-29
	10	74	76
5, 8	0	132	116
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	6	34	-20
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	14	128	-114
	17	47	38
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	4	88	-54
	7	47	-61
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	13	89	-91
	16	18	-16
7, 1	0	285	273
	3	130	-122
	6	46	-61
	9	224	226
	12	29	43
	15	90	-91
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	5	220	-222
	8	87	97
	11	50	25
	14	132	-123
	17	20	9
7, 3	1	46	-24
	4	100	-96
	7	25	-24
	10	51	45
	13	54	-47
7, 4	0	38	21
	3	101	-91
	6	106	-106
	9	59	47
	12	58	-51
7, 5	2	54	45
	5	131	-113
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	11	98	93
7, 6	1	36	28
	4	105	-91
	7	13	-21
	10	0	-16
7, 7	0	55	51
	3	33	-27
	6	18	-53
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	5	125	-130
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	11	35	17
	14	109	-95
	17	0	2
8, 1	1	118	138
	4	107	-104
	7	81	-88
	10	71	70
	13	47	-35
	16	14	-6
8, 2	0	178	163
	3	28	22
	6	152	-142
	9	107	92
	12	13	5
	15	39	-51
8, 3	2	124	106
	5	71	-63
	8	84	89
	11	22	10
	14	40	-28
8, 4	1	82	88
	4	99	-90
	7	23	-25
	10	90	99
8, 5	0	99	79
	3	19	-5
	6	22	-18
	9	34	29
8, 6	2	33	30
	5	22	-18
9, 0	3	83	86
	6	72	-68
	9	230	227
	12	30	43
	15	34	-26
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	5	96	-91
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	14	60	-46
	11	65	53
9, 2	1	21	13
	4	85	-91
	7	20	-17
	10	65	56
	13	30	-28
9, 3	0	188	181
	3	39	-31
	6	65	-72
	9	105	96
9, 4	2	19	14
	5	13	22
	8	52	57
9, 5	1	12	-17
	4	81	-95
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	4	90	-69
	7	119	-128
	10	40	20
	13	34	-42
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	3	92	-93
	6	34	-21
	9	108	107
	12	25	-17
10, 2	2	61	-68
	5	90	-78
	8	0	-4
	11	0	-11
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	4	48	-34
	7	68	-75
10, 4	0	90	85
	3	77	-95
11, 0	2	34	9
	5	60	-52
	8	0	4
	11	24	15
11, 1	1	229	235
	4	58	-53
	7	0	-7
11, 2	0	0	-7
	3	69	-75
	6	15	4
11, 3	2	16	15
12, 0	3	24	-11
	6	15	-9
12, 1	2	28	35
	5	29	-37

Table 4. *Interatomic distances with standard deviations* ($\times 10^2$) *for* 1-*bromotriptycene*

Br —C(1)	1·97 (1) Å
C(1)–C(2)	1·53 (1)
C(2)–C(3)	1·37 (2)
C(2)–C(7)	1·39 (2)
C(3)–C(4)	1·42 (2)
C(4)–C(5)	1·35 (2)
C(5)–C(6)	1·39 (2)
C(6)–C(7)	1·37 (2)
C(7)–C(20)	1·51 (2)

Table 5. *Interatomic bond angles with standard deviations* ($\times 10$) *for* 1-*bromotriptycene*

Br —C(1)—C(2)	111·4 (6)°
H(20)-C(20)-C(7)	111·4 (6)
C(1)—C(2)—C(3)	128·5 (9)
C(20)-C(7)—C(6)	128·0 (9)
C(2)—C(1)—C(2)	107·5 (6)
C(7)—C(20)-C(7)	107·4 (6)
C(1)—C(2)—C(7)	110·7 (8)
C(20)-C(7)—C(2)	112·2 (8)
C(2)—C(3)—C(4)	118·1 (9)
C(7)—C(6)—C(5)	120·0 (9)
C(3)—C(4)—C(5)	121·1 (9)
C(2)—C(7)—C(6)	119·9 (7)
C(4)—C(5)—C(6)	120·2 (8)
C(3)—C(2)—C(7)	120·7 (7)
C(2)—C(3)—H(3)	120·2 (8)
C(3)—C(4)—H(4)	116·7 (10)
C(4)—C(5)—H(5)	120·6 (8)
C(5)—C(6)—H(6)	118·1 (8)

to an aliphatic carbon atom. The C(1)–C(2) and C(7)–C(20) distances of 1·53 Å and 151 Å, respectively, are in good agreement with the expected value of 1·515 Å for an sp^2–sp^3 carbon bond (Dewar & Schmeising, 1960). It is concluded, therefore, that there is no interaction between benzene rings in 1-bromotriptycene. Since the bond angles and interatomic distances are the same for both bridges (within the accuracy of this investigation), it appears that the bromine atom has a negligible effect on adjacent bond angles and bond distances.

Estlin & Karle (1967) recently reported the results of a crystal structure investigation of β-chloroethyltriptycene. Their bond angles and distances are in good agreement with the results reported in this investigation.

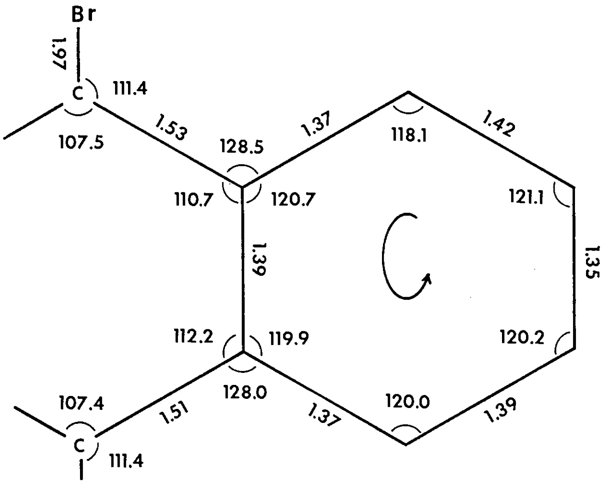

Fig. 4. Interatomic bond distances and angles for the asymmetric unit.

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