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Crystal Structure of *cis,trans*-1,2-Di[4-bromobenzylidene]-cyclohexane

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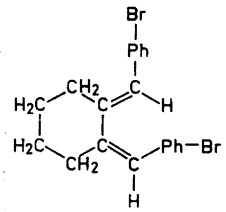
The crystals are monoclinic with space group $P2_1/c$ and cell dimensions $a=15.50_3$ Å, $b=5.53_0$ Å, $c=20.71_3$ Å, $\beta=95.7_3°$. 1374 reflections were recorded as observed by an automatic four circle diffractometer. The structure was solved by the heavy atom method and refined by full-matrix least squares technique ($R=6.0$ %, $R_w=3.8$ %). The dihedral angle C=C–C=C of 60.9° corresponds to a *gauche*-conformation for the diene system.

The preferred conformation of non-transoid conjugated dienes has been discussed by Dale *et al.*[1,2] The diene system of 1,2-dibenzylidene cycloalkanes (and 1,2-di-β-naphthylidene cycloalkanes)[2] appears to be forced into planarity by the five-membered ring, while it is allowed to assume a non-planar conformation in the corresponding six- and seven-membered rings. To obtain more precise structural information, in particular the dihedral angle C=C–C=C, single crystals of *cis,trans*-1,2,-di[4-bromobenzylidene] cyclohexane were prepared for the purpose of an X-ray investigation.*



* In the original publication[2] the minor isomer, occurring together with the *trans,trans*-isomer of 1,2-dibenzylidene (and naphthylidene) cyclohexanes and cycloheptanes, was assigned a *cis,cis*-configuration on the basis of the single NMR-line observed for the olefinic protons when CCl_4 and $CHCl_3$ were the solvents. After the present X-ray work revealed a *cis,trans*-configuration for the dibromoderivative with assumed *cis,cis*-configuration, the authors have re-examined the NMR-spectra for this and the other assumed *cis,cis*-isomers. A slight splitting can in fact be observed in benzene solution, a stronger one in dimethylsulfoxide.

Table 1. Fractional atomic coordinates for bromine and carbon atoms with estimated standard deviations (multiplied by 10^5).[a]

Atom	x	y	z
Br_1	36 117	12 364	21 970
	6	22	4
Br_2	97 542	01 626	40 041
	6	19	4
C_1	44 250	01 729	16 345
	57	185	36
C_2	42 693	−18 615	12 934
	54	177	45
C_3	48 474	−25 385	08 507
	66	153	38
C_4	55 883	−11 997	07 745
	54	177	35
C_5	57 330	08 367	11 405
	49	160	37
C_6	51 535	15 455	15 792
	67	162	37
C_7	61 683	−19 422	02 731
	50	135	30
C_8	70 315	−18 539	02 952
	55	141	33
C_9	74 973	−23 759	−02 992
	50	154	31
C_{10}	80 595	−02 167	−04 448
	54	173	33
C_{11}	86 851	04 247	01 493
	48	153	35
C_{12}	81 982	09 599	07 444
	49	151	32
C_{13}	76 019	−10 893	08 655
	45	146	30
C_{14}	76 127	−23 070	14 247
	41	129	29
C_{15}	81 535	−16 825	20 414
	51	176	31
C_{16}	87 949	−31 940	23 099
	55	153	37
C_{17}	92 762	−26 698	28 888
	47	170	40
C_{18}	90 964	−06 130	32 059
	52	191	32
C_{19}	84 666	09 624	29 676
	58	147	38
C_{20}	79 957	04 190	23 780
	48	160	39

[a] For numbering of atoms, see Fig. 1.

Table 2. Anisotropic thermal vibration parameters and their estimated standard deviations (multiplied by 10^5).

Atom	B_{11}	B_{22}	B_{33}	B_{12}	B_{13}	B_{23}
Br_1	869	9085	434	1636	450	412
	7	71	3	37	7	24
Br_2	726	9054	260	−146	−21	−578
	6	66	2	34	5	21
C_1	652	4545	345	949	6	−29
	59	568	27	333	64	204
C_2	647	4229	445	−301	229	100
	56	547	33	301	71	206
C_3	726	4317	383	−734	−22	−576
	59	496	29	312	69	186
C_4	484	3913	298	−108	−56	−384
	50	475	25	276	58	189
C_5	589	4540	350	−224	13	−905
	51	520	26	264	61	191
C_6	676	5011	335	546	−137	−923
	56	520	27	323	66	190
C_7	711	3874	234	257	−30	−271
	48	406	20	239	53	146
C_8	570	3868	263	−335	33	−166
	49	436	24	277	60	160
C_9	745	5438	218	605	77	−616
	53	494	21	279	55	165
C_{10}	820	6226	293	304	331	364
	56	514	24	312	59	201
C_{11}	705	7209	306	−466	232	710
	49	526	23	278	58	194
C_{12}	651	5333	292	863	18	417
	50	490	24	280	55	169
C_{13}	449	4001	243	98	74	−25
	42	416	21	236	48	154
C_{14}	531	4193	226	−321	87	−114
	42	406	20	219	49	141
C_{15}	518	4054	189	−215	144	133
	49	492	23	261	52	178
C_{16}	652	4391	256	619	122	−278
	52	481	25	278	58	177
C_{17}	558	4788	282	991	15	31
	49	503	26	260	58	183
C_{18}	498	5365	229	113	132	−236
	48	552	22	270	52	188
C_{19}	719	3940	305	92	136	−491
	55	462	26	288	60	178
C_{20}	691	4103	281	533	−13	−133
	51	494	24	261	588	180

Table 3. Observed and calculated structure factors on 10 times absolute scale.

h	k	l	F_o	F_c
0	0	2	604	- 567
0	0	4	41	33
0	0	6	1242	-1278
0	0	8	141	126
0	0	10	1056	1051
0	0	12	174	- 142
0	0	14	797	- 791
1	0	22	172	134
1	0	20	224	236
1	0	18	184	146
1	0	16	110	46
1	0	14	693	- 667
1	0	12	337	350
1	0	10	586	550
1	0	8	539	532
1	0	6	1241	-1257
1	0	4	338	322
1	0	2	441	396
1	0	0	237	194
1	0	-2	646	588
1	0	-4	1290	-1302
1	0	-6	113	141
1	0	-8	431	- 440
1	0	-10	1175	1183
1	0	-12	140	188
1	0	-14	645	- 615
1	0	-16	279	- 263
1	0	-18	244	- 174
2	0	-20	442	458
2	0	-22	145	122
2	0	-16	278	- 277
2	0	-14	455	- 446
2	0	-12	1273	1289
2	0	-10	98	86
2	0	-8	967	937
2	0	-6	1339	-1361
2	0	-4	667	- 664
2	0	-2	153	144
2	0	0	687	656
2	0	2	86	97
2	0	4	2402	-2471
2	0	6	192	192
2	0	8	387	- 381
2	0	10	1369	1385
2	0	12	296	- 308
2	0	14	249	- 245
2	0	16	154	- 180
2	0	18	299	313
2	0	20	292	273
3	0	19	372	376
3	0	16	431	- 426
3	0	14	422	- 420
3	0	12	170	- 176
3	0	10	706	705
3	0	8	1298	1273
3	0	6	700	- 724
3	0	4	497	488
3	0	2	1425	-1435
3	0	0	424	448
3	0	-2	953	946
3	0	-4	955	- 929
3	0	-6	737	- 745
3	0	-8	430	- 431
3	0	-10	852	881
3	0	-12	370	- 344
3	0	-14	585	- 579
3	0	-22	141	131
4	0	-20	399	385
4	0	-18	352	- 342
4	0	-16	131	- 111
4	0	-14	345	- 347
4	0	-12	735	734
4	0	-10	107	138
4	0	-8	1322	-1217
4	0	-6	341	- 331
4	0	-4	1898	-1902
4	0	-2	2380	2479
4	0	2	385	394
4	0	4	1708	-1687
4	0	6	456	- 455
4	0	8	1148	1143
4	0	10	156	182
4	0	12	331	328
4	0	14	650	- 664
4	0	16	149	- 132
4	0	20	219	239
5	0	20	206	173
5	0	18	147	157
5	0	14	293	- 294
5	0	12	454	- 458
5	0	10	587	608
5	0	6	289	- 278
5	0	4	1222	-1238
5	0	2	226	- 200
5	0	0	1193	1209
5	0	-2	275	288
5	0	-4	496	513
5	0	-6	1443	-1481
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5	0	-10	120	82
5	0	-12	448	450
5	0	-16	389	- 396
5	0	-22	202	185
6	0	-20	337	316
6	0	-16	488	- 492
6	0	-14	406	413
6	0	-10	1087	1083
6	0	-8	1020	-1049
6	0	-6	215	- 258
6	0	-4	197	176
6	0	-2	610	615
6	0	0	603	631
6	0	2	1006	-1034
6	0	4	723	- 741
6	0	8	696	706
6	0	12	151	- 143
6	0	14	308	- 322
6	0	18	305	274
7	0	18	170	179
7	0	16	157	160
7	0	14	490	- 476
7	0	12	147	- 185
7	0	10	146	173
7	0	8	493	486
7	0	6	662	667
7	0	4	1144	-1281
7	0	2	200	198
7	0	0	279	- 299
7	0	-2	1533	1534
7	0	-4	296	- 324
7	0	-6	425	- 435
7	0	-8	126	- 169
7	0	-10	131	119
7	0	-12	439	445
7	0	-14	225	- 215
7	0	-16	147	66
7	0	-18	371	- 355
8	0	-16	425	- 403
8	0	-14	195	169
8	0	-12	330	314
8	0	-10	82	- 55
8	0	-8	76	44
8	0	-6	959	- 950
8	0	-4	706	729
8	0	-2	170	183
8	0	0	595	614
8	0	2	578	- 579
8	0	4	243	- 248
8	0	6	432	441
8	0	8	486	498
8	0	10	301	297
8	0	12	261	- 243
8	0	14	148	- 159
8	0	18	207	195
9	0	18	216	189
9	0	14	136	116
9	0	12	214	- 208
9	0	10	134	- 118
9	0	8	178	- 179
9	0	6	822	821
9	0	4	232	- 209
9	0	2	353	- 355
9	0	0	561	- 557
9	0	-2	172	142
9	0	-4	549	553
9	0	-6	138	- 130
9	0	-8	180	187
9	0	-10	812	- 831
9	0	-12	375	386
9	0	-14	198	205
9	0	-16	195	206
9	0	-18	176	- 177
10	0	-18	245	- 219
10	0	-14	249	- 268
10	0	-12	531	525
10	0	-10	386	- 361
10	0	-8	356	- 350
10	0	-6	330	322
10	0	-2	607	611
10	0	0	268	- 254
10	0	4	376	- 351
10	0	6	440	437
10	0	8	100	62
10	0	12	268	- 254
10	0	14	226	- 212
11	0	12	242	- 246
11	0	10	145	185
11	0	8	218	209
11	0	6	338	342
11	0	4	149	- 166
11	0	2	614	- 599
11	0	0	312	301
11	0	-2	95	- 80
11	0	-4	730	725
11	0	-6	521	- 531
11	0	-8	454	- 485
11	0	-10	157	- 175
11	0	-14	231	235
11	0	-16	228	- 199
12	0	-18	243	- 193
12	0	-14	143	189
12	0	-10	219	190
12	0	-8	298	- 273
12	0	-6	120	119
12	0	-4	206	- 221
12	0	-2	412	393
12	0	0	156	- 124
12	0	2	308	- 324
12	0	4	177	- 196
12	0	6	209	189
12	0	8	115	5
12	0	12	144	- 144
13	0	12	216	- 216
13	0	6	306	275
13	0	4	247	- 233
13	0	0	159	- 160
13	0	-2	154	167
13	0	-4	371	378
13	0	-10	207	- 202
13	0	-12	353	335
14	0	-14	237	203
14	0	-8	162	- 165
14	0	2	324	- 297
14	0	4	193	226
14	0	8	152	148
14	0	12	155	- 116
15	0	8	149	172
15	0	2	170	- 176
15	0	0	256	- 263
15	0	-2	246	224
15	0	-6	243	171
15	0	-8	265	- 241
16	0	-10	178	- 156
16	0	-4	146	144
16	0	2	155	- 92
17	0	2	147	- 71
17	0	-4	152	85
17	1	-3	147	- 113
16	1	1	213	200
16	1	-5	146	- 94
16	1	-9	147	48
15	1	-13	144	- 166
15	1	-3	141	- 70
15	1	-2	121	76
15	1	4	126	- 73
15	1	5	148	- 101
15	1	7	155	- 38
14	1	7	133	- 120
14	1	4	132	91
14	1	1	141	171
14	1	-2	113	35
14	1	-3	268	- 278
14	1	-9	241	244
14	1	-12	147	115
13	1	-15	147	- 125
13	1	-14	133	51
13	1	-9	146	132
13	1	-7	196	201
13	1	-5	172	- 173
13	1	-2	138	- 119
13	1	-1	265	- 265
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13	1	3	153	144
13	1	6	138	117
13	1	8	174	- 154
13	1	7	152	- 103
13	1	11	130	124
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12	1	-9	148	168
12	1	-13	138	149
12	1	-13	246	- 230
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12	1	-15	132	109
11	1	-17	118	97
11	1	-16	114	- 67
11	1	-15	131	- 83
11	1	-14	176	140
11	1	-12	196	- 89
11	1	-11	304	- 304
11	1	-10	190	190
11	1	-9	247	249
11	1	-8	241	- 261
11	1	-6	123	113
11	1	-5	124	117
11	1	-4	203	- 232
11	1	-3	246	- 277
11	1	-2	124	151
11	1	-1	192	- 166
11	1	0	97	63
11	1	1	310	306
11	1	3	144	- 130
11	1	6	157	- 129
11	1	7	402	- 399
11	1	12	146	117
11	1	13	188	226
10	1	17	133	- 137
10	1	13	144	119
10	1	12	149	- 121
10	1	11	134	149
10	1	10	248	269
10	1	9	239	- 226
10	1	7	302	- 287
10	1	6	172	206
10	1	4	126	117
10	1	3	497	497
10	1	2	190	- 163
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10	1	0	198	224
10	1	-1	372	- 361
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10	1	-3	118	- 127
10	1	-4	274	250
10	1	-5	227	- 220
10	1	-6	287	- 289
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10	1	-11	138	- 209
10	1	-12	196	- 235
10	1	-13	313	- 282
10	1	-17	198	183
10	1	-18	130	120
9	1	-20	127	115
9	1	-19	203	195
9	1	-16	131	121
9	1	-15	144	142
9	1	-14	214	- 211
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9	1	-4	135	- 129
9	1	-3	854	- 844
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9	1	-1	127	119
9	1	0	189	- 209
9	1	1	100	- 68
9	1	2	270	270
9	1	3	644	665
9	1	4	132	- 166
9	1	6	101	59
9	1	7	275	- 260
9	1	8	182	- 192
9	1	9	144	- 168
9	1	12	106	- 99
9	1	13	309	309
9	1	17	193	- 134
9	1	18	125	12
8	1	17	210	- 216
8	1	15	126	142
8	1	13	200	208
8	1	12	105	- 64
8	1	11	206	226
8	1	9	209	- 223
8	1	8	102	60
8	1	7	370	- 365
8	1	6	383	- 383
8	1	5	526	520
8	1	4	197	172
8	1	3	365	361
8	1	2	316	- 313
8	1	1	302	294
8	1	0	280	295
8	1	-1	757	- 762
8	1	-2	284	- 291
8	1	-3	238	- 238
8	1	-4	211	- 214
8	1	-5	177	184
8	1	-6	175	179
8	1	-7	387	389
8	1	-9	379	394
8	1	-10	372	372
8	1	-11	543	- 553
8	1	-12	294	- 288
8	1	-14	194	196
8	1	-15	129	- 115

Table 3. Continued.

h	k	l	F_o	F_c
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8	1	-21	149	- 125
7	1	-21	131	- 117
7	1	-20	130	- 116
7	1	-19	129	- 51
7	1	-17	236	265
7	1	-15	113	116
7	1	-13	289	- 302
7	1	-12	279	265
7	1	-10	415	- 422
7	1	-9	250	- 251
7	1	-8	464	476
7	1	-7	1190	1233
7	1	-6	583	- 594
7	1	-5	309	- 302
7	1	-4	482	472
7	1	-2	319	- 323
7	1	-1	592	- 601
7	1	0	548	548
7	1	1	247	225
7	1	2	235	226
7	1	3	649	660
7	1	4	357	- 378
7	1	6	281	288
7	1	7	85	- 63
7	1	8	139	- 158
7	1	9	671	- 667
7	1	10	203	193
7	1	11	245	244
7	1	12	103	- 98
7	1	13	145	154
7	1	15	186	193
7	1	16	191	- 187
7	1	18	173	- 217
6	1	16	115	61
6	1	15	145	101
6	1	14	211	- 234
6	1	13	495	498
6	1	12	368	369
6	1	11	288	- 285
6	1	10	101	- 90
6	1	9	411	- 433
6	1	7	587	- 607
6	1	6	115	102
6	1	5	541	533
6	1	4	210	- 213
6	1	3	662	665
6	1	2	288	290
6	1	1	299	- 288
6	1	0	534	- 536
6	1	-1	427	- 425
6	1	-2	604	597
6	1	-3	646	- 668
6	1	-4	322	- 328
6	1	-5	1104	1098
6	1	-6	191	- 165
6	1	-7	395	390
6	1	-10	108	- 106
6	1	-11	325	- 323
6	1	-13	327	- 324
6	1	-14	150	- 156
6	1	-15	284	272
6	1	-16	213	197
6	1	-17	150	143
6	1	-21	210	- 196
5	1	-19	141	- 147
5	1	-18	268	267
5	1	-17	481	470
5	1	-16	193	- 205
5	1	-14	235	230
5	1	-12	393	- 371
5	1	-11	705	- 715
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5	1	-9	153	149
5	1	-8	123	- 87
5	1	-7	529	536
5	1	-5	207	219
5	1	-4	544	502
5	1	-3	385	- 387
5	1	-2	687	- 703
5	1	-1	1022	-1016
5	1	0	305	310
5	1	1	678	667
5	1	2	423	- 427
5	1	3	184	168
5	1	4	90	63
5	1	5	924	903
5	1	6	417	- 420
5	1	7	886	- 880
5	1	8	359	354
5	1	9	612	- 618
5	1	11	145	- 139
5	1	13	258	266
5	1	14	127	- 95
5	1	15	311	301
5	1	17	154	- 133
5	1	18	132	78
4	1	19	210	- 211
4	1	19	297	- 286
4	1	18	108	55
4	1	15	180	179
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3	3	4	202	215
3	3	5	200	185
3	3	6	383	- 375
3	3	8	144	145
3	3	9	410	- 420
3	3	12	130	63
3	3	15	305	304
2	3	19	149	- 109
2	3	16	127	- 108
2	3	15	174	142
2	3	14	136	173
2	3	13	255	275
2	3	12	136	- 152
2	3	11	237	- 220
2	3	10	233	266
2	3	9	153	- 138
2	3	8	108	- 107
2	3	7	124	111
2	3	6	192	- 188
2	3	5	347	356
2	3	4	167	151
2	3	3	411	412
2	3	2	291	- 302
2	3	1	406	- 418
2	3	0	448	472
2	3	-1	152	- 175
2	3	-2	427	- 423
2	3	-3	198	- 201
2	3	-4	271	271
2	3	-5	443	445
2	3	-6	235	- 216
2	3	-7	87	47
2	3	-8	131	92
2	3	-9	202	- 224
2	3	-11	137	- 118
2	3	-13	246	- 277
2	3	-14	222	241
2	3	-15	196	206
1	3	-20	136	- 56
1	3	-19	190	- 183
1	3	-17	154	110
1	3	-16	110	88
1	3	-15	159	98
1	3	-14	136	- 65
1	3	-13	159	160
1	3	-12	116	- 82
1	3	-11	379	- 394
1	3	-10	296	- 301
1	3	-8	375	388
1	3	-7	278	274
1	3	-6	126	103
1	3	-5	378	391
1	3	-3	420	430
1	3	-2	96	- 78
1	3	-1	311	- 309
1	3	2	503	501
1	3	3	202	- 193
1	3	4	146	- 135
1	3	5	516	524
1	3	6	115	111
1	3	7	216	- 242
1	3	8	176	- 164
1	3	10	154	147
1	3	11	421	- 398
1	3	13	102	140
1	3	15	140	113
1	3	16	144	- 13
1	3	18	195	- 159
1	3	20	145	100
0	3	18	195	- 81
0	3	17	194	183
0	3	15	199	160
0	3	12	204	187

Table 3. Continued.

h	k	l	F_o	F_c
0	3	11	140	- 134
0	3	10	155	- 141
0	3	9	391	- 401
0	3	7	344	325
0	3	6	195	- 187
0	3	5	215	207
0	3	4	242	253
0	3	3	346	329
0	3	2	201	- 211
0	3	1	611	- 617
0	4	1	175	- 157
0	4	2	279	287
0	4	3	160	- 168
0	4	4	204	- 191
0	4	11	167	- 181
0	4	14	137	- 156
1	4	10	283	280
1	4	7	119	120
1	4	6	132	- 123
1	4	5	122	72
1	4	4	227	- 265
1	4	2	108	- 115
1	4	0	351	326
1	4	-3	140	100
1	4	-4	178	197
1	4	-5	101	95
1	4	-6	240	- 243
1	4	-8	117	95
1	4	-12	149	98
2	4	-14	135	17
2	4	-13	127	87
2	4	-10	344	362
2	4	-8	213	- 221
2	4	-7	121	- 123
2	4	-6	106	- 52
2	4	-4	146	- 194
2	4	0	198	187
2	4	3	227	- 209
2	4	5	134	117
2	4	6	344	- 349
2	4	7	140	116
2	4	8	147	143
2	4	10	123	119
2	4	12	131	107
3	4	14	142	- 90
3	4	10	200	193
3	4	6	244	- 237
3	4	-2	285	252
3	4	-3	129	114
3	4	-4	197	- 199
3	4	-6	194	- 232
4	4	-9	164	- 132
4	4	-6	238	- 248
4	4	-3	106	- 20
4	4	-2	150	- 163
4	4	0	338	347
4	4	2	124	- 60
4	4	6	119	- 97
4	4	7	163	166
4	4	9	118	- 127
4	4	12	129	- 105
4	4	16	156	- 75
5	4	14	147	- 199
5	4	8	241	279
5	4	6	132	- 105
5	4	5	118	105
5	4	2	147	- 192
5	4	1	146	- 241
5	4	-2	309	322
5	4	-4	138	- 106
5	4	-5	115	47
5	4	-6	161	173
5	4	-8	178	- 195
6	4	-14	140	- 123
6	4	-12	243	256
6	4	-6	229	- 246
6	4	-5	189	197
6	4	-3	164	- 158
6	4	-2	133	185
6	4	1	172	- 146
6	4	2	144	170
6	4	4	241	- 274
6	4	10	168	119
6	4	16	162	9
7	4	10	151	61
7	4	2	241	- 247
7	4	-3	190	144
8	4	-11	124	- 47
8	4	-10	137	87
8	4	-8	136	- 103
8	4	-3	132	88
8	4	-2	259	263
8	4	1	143	149
8	4	4	153	- 112
8	4	8	142	106
9	4	7	155	115
9	4	6	197	184
9	4	5	145	42
9	4	4	151	- 156
9	4	3	133	105
9	4	-2	140	- 24
9	4	-4	146	151
9	4	-12	146	104
10	4	-11	155	28
10	4	-1	157	- 102
11	4	5	156	52
12	4	-9	137	- 49
12	4	-5	148	- 10
13	4	-2	157	- 80
0	5	-5	155	- 12
7	5	5	147	73
6	5	-2	156	- 160
6	5	-5	129	- 15
5	5	-6	125	93
5	5	1	149	- 90
4	5	-1	153	- 155
4	5	-11	161	- 121
3	5	-8	121	119
3	5	-7	119	58
3	5	-4	136	25
3	5	9	145	- 91
2	5	13	136	78
2	5	11	154	- 62
2	5	9	121	- 102
2	5	-5	148	127
1	5	-10	130	41
1	5	4	148	137
1	5	6	161	- 177
1	5	10	121	41
0	5	10	143	- 25
0	5	8	106	- 47
0	5	7	137	151
0	5	5	128	163
0	5	1	112	- 19
1	6	6	143	5
1	6	2	117	10

The crystals are monoclinic with space group $P2_1/c$. The cell dimensions, determined by a manual four circle diffractometer, with estimated standard deviations [3] * are:

$$a = 15.503(9)\ \text{Å},\ b = 5.530(5)\ \text{Å},\ c = 20.713(4)\ \text{Å},\ \beta = 95.73(1)^\circ.$$

The unit cell contains two molecules ($\varrho_{calc}=1.52$ g/cm^3, $\varrho_{obs}=1.51$ g/cm^3).

With 2θ max equal to 48° and Mo$K\alpha$-radiation, about 2500 reflections were measured by an automatic four circle diffractometer. 1374 were recorded as observed using an observed-unobserved cutoff at $2.0\cdot\sigma(I)$. The intensities were corrected for absorption effects (crystal size 0.32 mm $\times$0.19 mm $\times$0.06 mm).

The structure was solved by the heavy atom method and refined by full-matrix least squares technique. Hydrogen positions were calculated assuming C—H bond lengths of 1.03 Å. With a common B-value of 5.0 Å^2, hydrogen parameters were included in structure factor calculations, but not refined. Anisotropic temperature factors were introduced for bromine and carbon atoms. The weights in least squares were calculated from the standard deviations in intensities, $\sigma(I)$, taken as

$$\sigma(I) = [C_T + (0.02C_N)^2]^{\frac{1}{2}}$$

where C_T is the total number of counts and C_N the net count (peak minus background). The conventional R-value arrived at was 6.0 % (weighted value $R_W=3.8$ %) for 1374 observed reflections. The form factors used were those of Hanson *et al.*[4] Final fractional coordinates and thermal parameters with estimated standard deviations are given in Tables 1 and 2. A comparison between observed and calculated structure factors is presented in Table 3.

* All programs used are included in this reference.

Table 4. The principal axes of the thermal vibration ellipsoids given by the components of a unit vector in fractional coordinates e_x, e_y, e_z; the corresponding r.m.s. amplitudes, and the B-values.

Atom	e_x	e_y	e_z	$(\overline{u^2})^{\frac{1}{2}}$ (Å)	B (Å^2)
	.036	.143	.015	.410	13.27
Br_1	.032	−.101	.035	.316	7.89
	−.044	.044	.030	.256	5.18
	.001	−.178	.009	.379	11.33
Br_2	−.062	.006	.009	.301	7.17
	.019	.033	.047	.227	4.06
	.048	.106	−.012	.313	7.74
C_1	.012	.053	.046	.271	5.80
	.042	−.137	.009	.231	4.21
	.029	.001	.045	.315	7.84
C_2	−.049	.096	.015	.278	6.10
	.031	.153	−.010	.247	4.80
	−.056	.040	.018	.312	7.68
C_3	.017	−.108	.038	.305	7.34
	.029	.140	.024	.221	3.86
	.025	.084	−.037	.279	6.13
C_4	−.046	.128	.000	.249	4.88
	.039	.097	.032	.214	3.63
	.003	.119	−.036	.315	7.85
C_5	−0.62	.045	.003	.271	5.78
	.019	.129	.032	.213	3.57
	.034	.106	−.028	.344	9.32
C_6	.054	−.095	.013	.264	5.48
	.014	.112	.038	.218	3.75
	−.062	.021	.008	.299	7.06
C_7	.002	−.157	.024	.254	5.08
	.019	.087	.041	.210	3.49
	−0.57	.082	.001	.270	5.76
C_8	.015	.115	−.034	.249	4.90
	.027	.113	.034	.225	4.01
	.044	.125	−.010	.323	8.23
C_9	.048	−.109	.018	.279	6.16
	−.004	.073	.044	.197	3.06
	.049	.101	.021	.334	8.81
C_{10}	.037	−.147	.005	.302	7.20
	.020	.029	−.044	.228	4.09
	−.011	.169	.015	.348	9.55
C_{11}	.060	.006	.024	.299	7.04
	.023	.065	−.040	.224	3.97

Table 4. Continued.

	−.039	.131	.014	.324	8.26
C_{12}	.049	.068	.029	.254	5.11
	.016	.104	−.036	.236	4.40
	.015	.175	−.003	.250	4.94
C_{13}	.058	−.035	.023	.232	4.25
	−.024	.029	.043	.227	4.08
	.044	−.132	.008	.268	5.66
C_{14}	.048	.120	−.003	.240	4.56
	.002	.029	.048	.219	3.78
	.046	−.128	.005	.259	5.31
C_{15}	.043	.122	.018	.245	4.75
	.016	.037	−.045	.193	2.93
	.052	.106	−.002	.297	6.96
C_{16}	.034	−.109	.032	.256	5.18
	−.018	.098	.037	.218	3.74
	.042	.135	−.004	.305	7.35
C_{17}	−.007	.049	.046	.249	4.88
	.049	−.110	.016	.220	3.80
	.004	.177	−.009	.291	6.68
C_{18}	.061	.000	.020	.248	4.86
	−.021	.035	.043	.213	3.56
	.065	.006	.009	.295	6.86
C_{19}	.003	.120	−.036	.278	6.12
	−.005	.136	.031	.219	3.80
	.055	.072	−.012	.304	7.29
C_{20}	.033	−.093	.036	.243	4.65
	−.007	.137	.031	.240	4.56

The principal axes of the thermal vibration ellipsoids for bromine and carbon atoms were calculated from the thermal parameters of Table 2. Root mean square amplitudes and the corresponding B-values for the atomic anisotropic thermal vibration along the principal axes together with the components of these axes along the crystal axes are given in Table 4. The amplitudes are indeed large, and should probably be interpreted in terms of some sort of molecular oscillation with C_8, C_{13} situated close to, and the bromines far from the axes of libration. However, due to the presence of two single bonds, C_4—C_7 and C_{14}—C_{15}, with possible rotational freedom, no rigid-body motion analysis has been carried out.

Distances and angles with estimated standard deviations are shown in Figs. 1 and 2. Although the double bond distances C_7=C_8 (1.335 Å) and C_{13}=C_{14} (1.339 Å) are normal, a certain amount of delocalization is indicated by the distance C_8—C_{13} of 1.465 Å. These findings and the angles C_7=C_8—C_{13} (124.1°) and C_{14}=C_{13}—C_8 (120.4°) agree with the results obtained by gas electron diffraction investigation of butadiene (C=C = 1.341 Å, C—C = 1.463 Å,

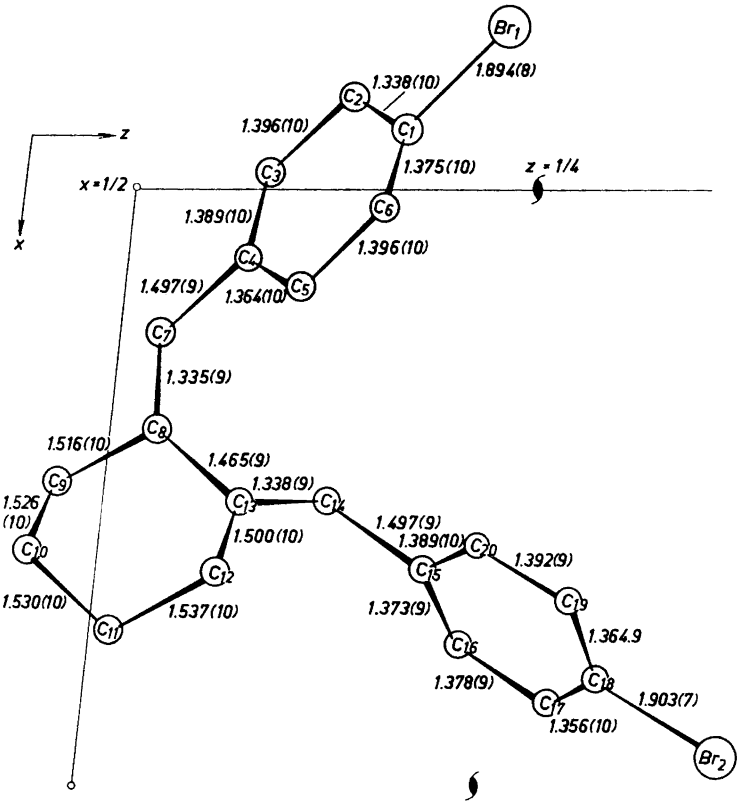

Fig. 1. Schematical drawing of the molecule (viewed along [010]) showing interatomic distances.

Table 5. Dihedral angles (estimated standard deviations are about 1°).

Angle	(°)
$C_7-C_8-C_{13}-C_{14}$	60.9
$C_3-C_4-C_7-C_8$	−143.4
$C_5-C_4-C_7-C_8$	40.2
$C_{20}-C_{15}-C_{14}-C_{13}$	67.4
$C_{16}-C_{15}-C_{14}-C_{13}$	−115.4
$C_4-C_7-C_8-C_9$	−171.6
$C_4-C_7-C_8-C_{13}$	−3.8
$C_{15}-C_{14}-C_{13}-C_{12}$	−6.6
$C_{15}-C_{14}-C_{13}-C_8$	178.9
$C_7-C_8-C_9-C_{10}$	122.8
$C_{14}-C_{13}-C_{12}-C_{11}$	124.3
$C_8-C_9-C_{10}-C_{11}$	55.3
$C_9-C_{10}-C_{11}-C_{12}$	−57.1
$C_{10}-C_{11}-C_{12}-C_{13}$	53.3
$C_{11}-C_{12}-C_{13}-C_8$	−50.5
$C_{12}-C_{13}-C_8-C_9$	51.6
$C_{13}-C_8-C_9-C_{10}$	−52.9

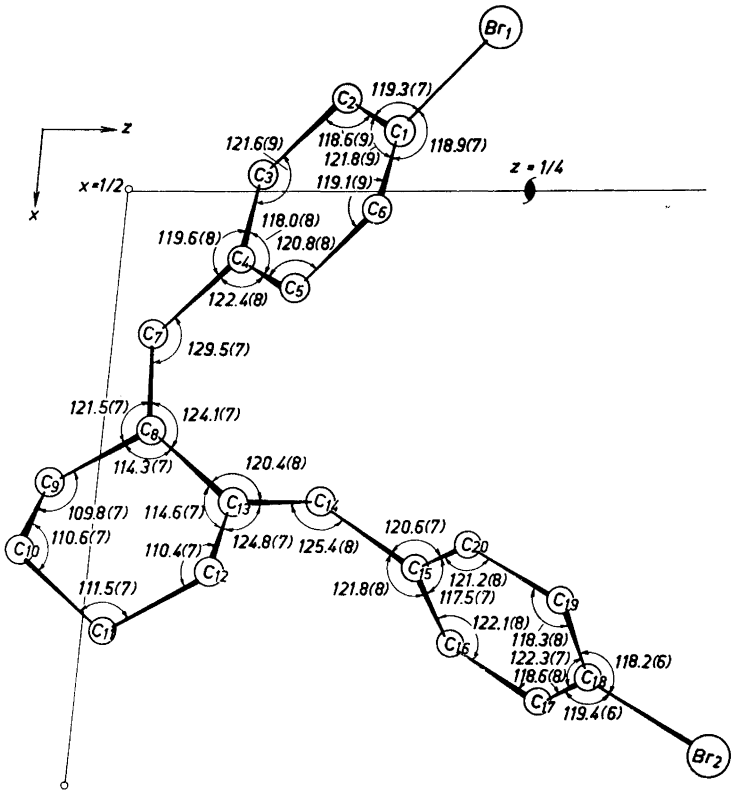

Fig. 2. Schematical drawing of the molecule (viewed along [010]) showing bond angles.

C=C—C = 123.3°).[5] The cyclohexane ring is a normal chair except for the shorter distance $C_8—C_{13}$, and a slight opening of the angles $C_9—C_8—C_{13}$ (114.3°) and $C_8—C_{13}—C_{12}$ (114.6°).

Dihedral angles are given in Table 5. The angle $C_7=C_8—C_{13}=C_{14}$ of 60.9° corresponds to a *gauche*-conformation. The angles between the essentially planar groups $C_4—C_7=C_8—C_{13}$ (−3.8°) and $C_{15}—C_{14}—C_{13}—C_8$ (178.9°) and the corresponding benzene ring planes are about 38° and 66°, respectively. Least squares planes for the two benzene rings give the following fits:

Atom	Deviation (Å)	Atom	Deviation (Å)
C_1	0.011	C_{15}	0.001
C_2	−0.012	C_{16}	0.005
C_3	0.006	C_{17}	−0.006
C_4	0.001	C_{18}	0.003
C_5	−0.002	C_{19}	0.003
C_6	−0.004	C_{20}	−0.005

No *inter*molecular hydrogen-hydrogen contacts are significantly shorter than 2.4 Å. The shortest *inter*molecular H—Br distance occur between Br_2 and H_{11e} (H_{11e} is equatorially bonded to C_{11}) in position $x,\frac{1}{2}-y,\ \frac{1}{2}+z$) and is 2.97 Å, while the sum of the van der Waals radii is 3.15 Å.

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