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The Crystal Structure of [2.2]Metaparacyclophane-1,9-diene

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(*Received* 26 *January* 1970)

[2.2]Metaparacyclophane-1,9-diene, $C_{16}H_{12}$, crystallizes in the orthorhombic system, space group *Pbca*, with $a=14\cdot91\pm1$, $b=18\cdot51\pm1$, $c=8\cdot133\pm5$ Å, $Z=8$. The intensity data were measured with a four-circle diffractometer and scintillation counter. The structure was determined by symbolic addition and refined by block-diagonal least-squares analysis of 1378 reflexions to a final R index of 0·046. The two aromatic rings are inclined to each other at 41°. Both show significant boat distortion, which is moderate for the *meta*-bridged ring, and severe for the *para*-bridged ring.

[2.2]Metaparacyclophane-1,9-diene (I; Hylton & Boekelheide, 1968) is one of a series of compounds prepared by Professor Boekelheide and his associates. Nuclear magnetic resonance studies of the material in solution fail to resolve the individual protons of the *para*-bridged ring, suggesting that the molecule has *mm*2 symmetry, with the two rings perpendicular to each other (Boekelheide, 1968). This conformation could, of course, be simulated by rapid 'flipping' of the molecule between two conformations related by the apparent symmetry. The X-ray analysis was undertaken in order to determine the conformation in the solid state. It is found that the rings are in fact by no means perpendicular, but are inclined to each other at 41°.

Experimental

Crystal data

$C_{16}H_{12}$ F.W. 204·3

Orthorhombic,
$a=14\cdot91\pm1$, $b=18\cdot51\pm1$, $c=8\cdot133\pm5$ Å

(Cu $K\alpha_1$, $\lambda=1\cdot5405$ Å), $V=2245$ Å^3, $D_m=1\cdot20$ g.cm^{-3}, $Z=8$, $D_x=1\cdot21$ g.cm^{-3}, $\mu=6\cdot3$ cm^{-1}.

Space group *Pbca* (D_{2h}^{15}) (from precession photographs. Systematic absences: $0kl$ for k odd, $h0l$ for l odd, $hk0$ for h odd).

The crystals supplied were colourless and translucent, displaying various faces of which [010] was the most prominent. The specimen used for data collection was a triangular plate, 0·3 mm thick, and 0·5 mm to the side. The material was found to decompose in conditions of moderate humidity, and the specimen was therefore enclosed in a thin-walled Lindemann glass capillary.

The intensities were measured with a Picker four-circle diffractometer and scintillation counter, using nickel-filtered Cu $K\alpha$ radiation with pulse-height discrimination. The θ–2θ scan method was used (2° for $2\theta<100°$, 3° otherwise), and background counts were measured at the beginning and end of each scan. Reflexions for which the net count was less than 10, or less than 20% of the gross count, were treated as unobserved. The yield of observed reflexions was rather low. In the range explored ($2\theta<130°$), 1380 of a possible 1904 reflexions were observed, and of these the net count exceeded four times the threshold value for only 956. Absorption corrections were not applied.

The structure was determined routinely by the symbolic addition procedure of Karle & Karle (1966). Refinement was by block-diagonal least-squares. The weighting scheme used was $w=w_1w_2$, where

$$\begin{aligned} w_1 &= F_o/10 \text{ for } F_o<10\\ &= 10/F_o \text{ for } F_o\ge 10\\ w_2 &= 2\cdot5\sin^2\theta \text{ for } \sin^2\theta<0\cdot4\\ &= 1 \qquad\qquad \text{ for } \sin^2\theta\ge 0\cdot4 \end{aligned}$$

(The nominal minimum value of F_o is 2·5).

This weighting scheme was reasonably effective in removing trends in the magnitudes of the residuals.

The scattering factor curves used were those of Hanson, Herman, Lea & Skillman (1964). The hydrogen atoms were initially assumed to lie in chemically reasonable positions, and their parameters were refined concurrently with those of the carbon atoms. During refinement it was apparent that the stronger reflexions were suffering from extinction, and they were corrected in the manner described by Pinnock, Taylor & Lipson (1956). 65 reflexions were corrected, but the correction exceeded 10% of F_o for only 12 of these. The procedure was considered to be of questionable validity for the strongest reflexion (040; $F_c=277$), which was given zero weight in subsequent refinement. (It should be noted that the weighting scheme discriminates so strongly against the very intense reflexions that the form of the extinction correction is of little consequence to the analysis). Because of unacceptably poor agreement one other reflexion was given zero weight: for 531, $F_o=5{\cdot}7$, and $F_c=0$; the counts recorded for this reflexion presumably result from some extraneous phenomenon.

In the final cycle no coordinate shift exceeded 40% of the corresponding e.s.d. The final parameters are given in Table 1. Observed and calculated structure amplitudes are compared in Table 2. The agreement is satisfactory, and there can be no doubt of the essential correctness of the structure. The agreement residual is 0·046, for observed reflexions only. A three-dimensional difference synthesis is reasonably featureless; the residual electron density is everywhere within the limits ±0·12 e.Å^{-3}. (Peak electron densities range from 4.7 to 7·2 e.Å^{-3} for carbon atoms, and from 0·55 to 0·94 e.Å^{-3} for hydrogen atoms.)

Table 1. *Final atomic parameters* (*e.s.d.'s*)

The coordinates are fractional, × 10^4. U_{ij} values are in Å^2 × 10^4. B values are in Å^2.

(*a*) Positions of the carbon atoms

The B values given are equivalent isotropic values in the directions of the principal axes of the vibration ellipsoids.

Atom	x	y	z	B_1	B_2	B_3
C(1)	5057(2)	4221(2)	3457(3)	10.1	9.1	4.4
C(2)	5754(2)	4105(2)	2467(4)	9.7	8.4	4.4
C(3)	5711(1)	3840(1)	783(3)	7.0	5.9	3.0
C(4)	6348(1)	3348(1)	130(4)	11.9	5.8	3.1
C(5)	6210(2)	3038(1)	-1376(4)	11.4	6.8	4.2
C(6)	5454(2)	3168(1)	-2263(3)	10.7	5.7	4.1
C(7)	4812(1)	3649(1)	-1686(3)	7.0	4.8	3.7
C(8)	4997(1)	4015(1)	-231(2)	5.3	4.6	2.9
C(9)	3920(2)	3274(1)	-2484(3)	9.4	6.3	4.3
C(10)	3142(2)	3831(1)	-1694(4)	10.1	6.8	4.2
C(11)	3108(1)	3877(1)	98(3)	9.0	5.8	3.0
C(12)	3075(1)	4541(1)	921(4)	10.3	5.4	3.6
C(13)	3529(2)	4646(1)	2307(4)	10.3	6.1	4.0
C(14)	4128(2)	4086(1)	2877(3)	8.7	6.8	3.6
C(15)	3901(2)	3385(1)	2383(3)	8.3	5.9	3.6
C(16)	3401(1)	3283(1)	1000(3)	8.6	5.6	3.0

(*b*) Anistropic temperature-factor coefficients

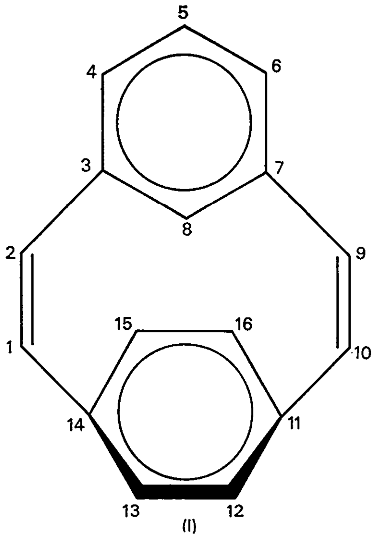
The temperature factor is defined as

$$\exp\{-2\pi^2(U_{11}a^{*2}h^2+2U_{12}a^*b^*hk+2U_{13}a^*c^*hl + U_{22}b^{*2}k^2+2U_{23}b^*c^*kl+U_{33}c^{*2}l^2)\}\,.$$

Atom	U_{11}	U_{12}	U_{13}	U_{22}	U_{23}	U_{33}
C(1)	1213(20)	-90(17)	-177(14)	1092(17)	-178(14)	685(14)
C(2)	794(14)	-182(13)	-268(13)	1050(17)	-28(16)	994(16)
C(3)	452(9)	-146(8)	10(10)	708(10)	73(10)	858(13)
C(4)	419(9)	-45(9)	93(12)	882(14)	316(16)	1340(21)
C(5)	751(14)	7(12)	390(14)	856(16)	-2(14)	1224(20)
C(6)	994(16)	-120(12)	390(13)	762(12)	-105(12)	842(15)
C(7)	734(11)	-113(9)	166(10)	613(10)	-6(10)	627(11)
C(8)	479(8)	-97(7)	95(8)	524(9)	18(8)	624(10)
C(9)	1115(17)	-81(13)	-187(13)	786(12)	-38(12)	627(11)
C(10)	752(14)	-38(11)	-328(14)	842(16)	-82(13)	1078(17)
C(11)	398(8)	-84(8)	24(10)	715(10)	-28(12)	1142(17)
C(12)	511(9)	46(9)	200(12)	674(10)	-18(13)	1257(19)
C(13)	811(14)	4(11)	342(13)	759(12)	-175(13)	1012(18)
C(14)	940(15)	-55(12)	248(11)	892(14)	-71(11)	594(12)
C(15)	796(12)	-42(10)	272(11)	708(12)	94(11)	744(12)
C(16)	533(9)	-133(8)	205(10)	632(10)	25(11)	1008(16)

(*c*) Hydrogen atom positions and isotropic temperature factors

Atom	x	y	z	B
H(1)	5146(23)	4364(19)	4514(44)	7.6(0.8)
H(2)	6346(23)	4166(17)	2919(40)	7.3(0.8)
H(4)	6850(20)	3233(16)	810(37)	6.4(0.7)
H(5)	6689(22)	2666(19)	-1856(44)	8.1(0.9)
H(6)	5354(18)	2926(15)	-3363(36)	5.9(0.6)
H(8)	4585(11)	4402(9)	90(22)	1.5(0.3)
H(9)	3912(21)	3671(17)	-3680(40)	7.0(0.7)
H(10)	2579(21)	3916(15)	-2352(36)	6.3(0.6)
H(12)	2810(15)	4946(13)	356(31)	4.5(0.5)
H(13)	3669(19)	5184(14)	2683(34)	5.4(0.6)
H(15)	4219(14)	2973(12)	2882(27)	3.7(0.5)
H(16)	3385(15)	2767(12)	508(30)	4.0(0.5)



(I)

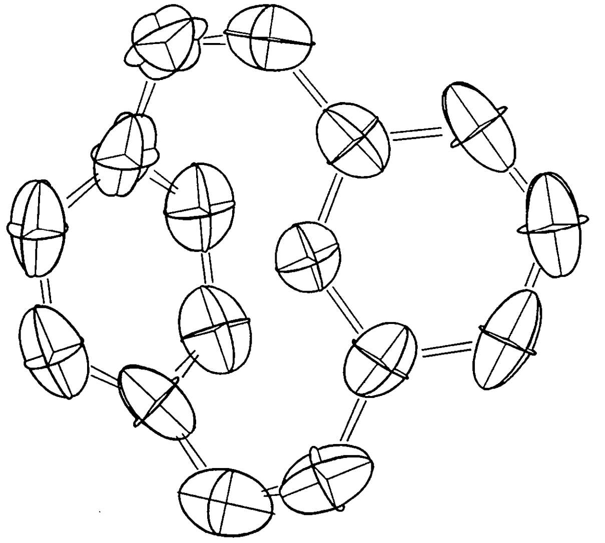


Fig. 1. The principal planes of the thermal motion ellipsoids of 50% probability.

Table 2. *Observed and calculated structure amplitudes,* × 10

An asterisk denotes the threshold value of an unobserved reflexion.

L	FO	FC
H= 0, K= 0		
2	1144	-1185
4	658	682
6	90	-90
8	101	102
H= 0, K= 2		
0	321	326
1	409	422
2	76	-62
3	282	289
4	154	152
5	279	272
6	22*	2
7	124	-127
8	24*	-8
9	34	-46
H= 0, K= 4		
0	2857	-2773
1	1246	1376
2	278	246
3	343	-340
4	364	-364
5	63	-68
6	39	-32
7	31	-25
8	37	-42
9	20*	-3
H= 0, K= 6		
0	20*	-27
1	584	596
2	83	-79
3	173	-175
4	53	-41
5	21	-19
6	126	-124
7	24	31
8	28	22
9	21	34
H= 0, K= 8		
0	91	-92
1	63	59
2	105	108
3	222	215
4	76	84
5	113	112
6	55	59
7	24*	-12
8	39	-28
H= 0, K= 10		
0	314	327
1	485	-485
2	75	63
3	145	145
4	82	80
5	95	94
6	78	70
7	23*	4
8	23*	1
H= 0, K= 12		
0	69	-70
1	40	-37
2	25	16
3	22*	-7
4	112	113
5	29	-32
6	24*	-2
7	27	-24
H= 0, K= 14		
0	373	-387
1	377	389
2	101	-96
3	206	-213
4	115	-113
5	24*	-10
6	60	59
7	40	-36
H= 0, K= 16		
0	75	-18
1	107	-110
2	26	-16
3	103	-105
4	99	-96
5	22*	-6
6	20*	11
H= 0, K= 18		
0	61	62
1	148	-151
2	147	147
3	133	128
4	28	24
5	36	-35
H= 0, K= 20		
0	23*	-18
1	29	35
2	56	54
3	29	26
H= 1, K= 0		
2	431	-454
4	205	-197
6	67	58
8	34	27
H= 1, K= 1		
1	1019	1083
2	385	-401
3	327	314
4	487	486
5	53	53
6	69	-69
7	24*	-1
8	56	-58
9	23*	8
H= 1, K= 2		
1	388	407
2	98	103
3	103	-112
4	98	-102
5	32	23
6	28	-19
7	24*	30
8	24*	-9
9	22*	-4
H= 1, K= 3		
1	974	-1049
2	305	-304
3	186	195
4	309	306
5	113	-110
6	81	-76
7	24*	-6
8	79	-84
9	23*	6
H= 1, K= 4		
1	519	543
2	442	-472
3	46	38
4	112	107
5	56	60
6	60	-64
7	47	46
8	26*	10
9	23	-9
H= 1, K= 5		
1	150	-149
2	68	-50
3	478	-497
4	180	-174
5	24*	24
6	25*	8
7	52	-53
8	25*	13
9	40	36
H= 1, K= 6		
1	27	-35
2	298	-309
3	64	68
4	105	105
5	147	-143
6	30	-29
7	76	-81
8	60	56
9	21*	9
H= 1, K= 7		
1	262	266
2	200	200
3	323	-326
4	92	-90
5	93	95
6	61	64
7	24*	-5
8	27	31
9	33	-29
H= 1, K= 8		
1	46	48
2	439	447
3	269	-275
4	32	-33
5	64	-65
6	24	-19
7	76	-83
8	26*	-21
H= 1, K= 9		
1	140	141
2	236	239
3	88	83
4	24*	-12
5	24*	-3
6	80	82
7	43	43
8	28	-19
H= 1, K= 10		
1	164	162
2	122	120
3	23*	3
4	31	-25
5	152	157
6	24*	19
7	25*	16
8	37	-38
H= 1, K= 11		
1	49	55
2	23	-14
3	222	221
4	26	24
5	24*	-1
6	24*	-20
7	25*	5
8	22*	-9
H= 1, K= 12		
1	24	35
2	24	-24
3	44	54
4	27	27
5	34	35
6	24*	17
7	26	16
H= 1, K= 13		
1	189	-196
2	63	64
3	101	104
4	68	-70
5	24*	-12
6	53	-50
7	23*	11
H= 1, K= 14		
1	52	-53
2	66	63
3	38	-39
4	31	23
5	42	-42
6	23*	12
7	22	-11
H= 1, K= 15		
1	99	-105
2	69	-75
3	172	-129
4	48	-43
5	25*	-14
6	24*	14
H= 1, K= 16		
1	108	-111
2	24*	15
3	41	36
4	28*	-14
5	24*	18
6	23*	-4
H= 1, K= 17		
1	53	61
2	98	-103
3	24*	-19
4	60	58
5	24*	-10
H= 1, K= 18		
1	24*	-1
2	28*	-3
3	27*	-14
4	28	-23
5	22*	11
H= 1, K= 19		
1	45	44
2	69	67
3	62	64
4	23*	12
H= 1, K= 20		
1	23*	13
2	25*	-9
3	23*	-11
H= 1, K= 21		
1	23*	8
2	22*	15
H= 2, K= 0		
0	1241	1277
2	784	-776
4	239	234
6	72	70
8	24*	13
H= 2, K= 1		
0	465	451
1	361	-368
2	552	-547
3	84	91
4	182	181
5	48	-46
6	99	-97
7	38	35
8	26	30
9	23*	11
H= 2, K= 2		
0	40	-48
1	170	123
2	226	217
3	160	-153
4	32	-37
5	90	-98
6	66	65
7	48	-53
8	26*	2
9	29	-25
H= 2, K= 3		
0	1120	1160
1	20*	18
2	433	-432
3	42	49
4	169	152
5	80	-87
6	39	41
7	27	-30
8	33	29
9	22*	-2
H= 2, K= 4		
0	874	-909
1	727	778
2	182	173
3	221	-231
4	64	-48
5	81	-81
6	23*	15
7	24*	-7
8	23*	6
9	20*	-5
H= 2, K= 5		
0	44	-66
1	1184	-1267
2	481	500
3	80	74
4	26	-20
5	87	-88
6	31	34
7	89	84
8	40	-35
9	32	-24
H= 2, K= 6		
0	472	-476
1	144	-251
2	245	-264
3	59	-60
4	33	24
5	64	-65
6	37	-40
7	79	83
8	24*	-6
9	23*	30
H= 2, K= 7		
0	57	-56
1	326	317
2	106	-105
3	108	109
4	126	-123
5	27	-17
6	195	-194
7	24*	19
8	24*	5
H= 2, K= 8		
0	117	-113
1	527	-537
2	190	187
3	38	34
4	142	-145
5	79	74
6	45	-51
7	36	38
8	27	-25
H= 2, K= 9		
0	571	600
1	553	572
2	360	-372
3	60	-65
4	101	96
5	113	110
6	41	-44
7	103	-105
8	52	50
H= 2, K= 10		
0	275	282
1	102	-103
2	55	50
3	66	70
4	38	46
5	76	72
6	56	-60
7	36	-39
8	23	14
H= 2, K= 11		
0	28	37
1	191	-184
2	39	37
3	21*	21
4	153	153
5	24*	3
6	77	82
7	22*	1
8	21*	-4
H= 2, K= 12		
0	195	194
1	38	37
2	51	-44
3	24	19
4	76	74
5	73	-74
6	24*	6
7	21*	-15
H= 2, K= 13		
0	40	-33
1	23*	11
2	22*	6
3	41	37
4	78	-77
5	28	-18
6	23*	-8
7	23*	20
H= 2, K= 14		
0	112	115
1	118	124
2	37	-35
3	94	-94
4	81	-80
5	25	-20
6	52	51
7	26	-19
H= 2, K= 15		
0	62	-64
1	175	173
2	32	-26
3	88	-91
4	24*	0
5	71	68
6	24*	-13
H= 2, K= 16		
0	24*	-13
1	24*	11
2	68	-65
3	24*	31
4	38	-32
5	39	43
6	23	-18
H= 2, K= 17		
0	111	-107
1	60	60
2	48	46
3	46	-43
4	26*	9
5	23*	4
H= 2, K= 18		
0	117	-118
1	24*	-20
2	30	27
3	90	71
4	25*	14
5	20*	5
H= 2, K= 19		
0	26*	22
1	36	-29
2	24*	-12
3	27	25
4	26	-23
H= 2, K= 20		
0	23*	8
1	56	53
2	41	41
3	23*	-3
H= 2, K= 21		
0	50	-42
1	27	-20
2	22*	13
H= 3, K= 0		
2	140	133
4	212	213
6	99	101
8	31	-32
H= 3, K= 1		
1	770	-767
2	468	-447
3	302	303
4	94	-98
5	25*	30
6	77	-82
7	24*	10
8	26*	-20
9	23*	-11
H= 3, K= 2		
1	803	-827
2	350	354
3	48	53
4	50	-34
5	59	-61
6	26*	34
7	65	72
8	31	-36
9	27	-33
H= 3, K= 3		
1	478	481
2	531	-537
3	178	160
4	23*	-11
5	46	-49
6	26*	-16
7	26	24
8	83	-81
9	26	29
H= 3, K= 4		
1	34	38
2	209	-209
3	35	32
4	38	-40
5	41	37
6	81	-80
7	50	59
8	29	29
9	22*	-16
H= 3, K= 5		
1	439	-471
2	25	-28
3	148	-148
4	23*	13
5	61	57
6	25*	15
7	24*	12
8	25*	4
9	23*	-14
H= 3, K= 6		
1	186	171
2	235	-235
3	152	152
4	116	113
5	25*	-10
6	85	-82
7	26	-23
8	68	73
9	23*	6
H= 3, K= 7		
1	36	29
2	311	-307
3	344	-346
4	59	58
5	61	-65
6	25*	-19
7	57	48
8	100	101
H= , K= 8		
1	80	77
2	230	226
3	198	-190
4	25*	0
5	87	86
6	43	41
7	47	-58
8	26*	-12
H= 3, K= 9		
1	319	299
2	85	-86
3	34	45
4	50	48
5	68	-71
6	24*	8
7	27*	-16
8	25*	15
H= 3, K= 10		
1	99	-98
2	89	-66
3	65	-60
4	23*	6
5	98	-98
6	46	44
7	24*	-10
8	36	-29
H= 3, K= 11		
1	23*	-8
2	222	219
3	139	141
4	47	-42
5	41	37
6	41	-49
7	42	-44
8	21*	-7
H= 3, K= 12		
1	141	-139
2	46	-50
3	66	71
4	64	-61
5	61	-61
6	25*	4
7	26*	19
H= 3, K= 13		
1	66	71
2	51	-51
3	23*	-26
4	63	-61
5	24*	5
6	37	-34
7	23*	3
H= 3, K= 14		
1	73	71
2	23*	2
3	35	-36
4	72	-69
5	44	-48
6	24*	19
7	22*	-4
H= 3, K= 15		
1	59	59
2	27	-28
3	69	-69
4	52	53
5	26*	5
6	24*	-3
H= 3, K= 16		
1	77	81
2	24*	-24
3	29	29
4	46	36
5	25*	2
6	23*	-16
H= 3, K= 17		
1	98	-99
2	37	37
3	35	-31
4	83	80
5	23	-16
H= 3, K= 18		
1	26*	9
2	31	27
3	25*	-15
4	38	36
5	22*	8
H= 3, K= 19		
1	25*	-13
2	26*	12
3	24*	24
4	24*	-12
H= 3, K= 20		
1	36	-37
2	24*	7
3	26	-23
H= 3, K= 21		
1	59	62
H= 4, K= 0		
0	1015	-1054
2	735	728
4	346	-338
6	327	327
8	75	-73
H= 4, K= 1		
0	848	-898
1	318	325
2	194	-180
3	361	343
4	75	64
5	142	144
6	35	38
7	61	-58
8	26*	1
9	23*	3
H= 4, K= 2		
0	522	536
1	555	-550
2	520	512
3	129	131
4	172	-171
5	224	-221
6	101	98
7	33	-33
8	26	11
9	21*	-7
H= 4, K= 3		
0	819	-833
1	374	369
2	289	-270
3	327	-311
4	52	22
5	25*	17
6	41	37
7	24*	5
8	26*	-16
9	22*	9
H= 4, K= 4		
0	39	29
1	92	88
2	187	-177
3	95	83
4	189	192
5	51	-50
6	206	-207
7	24*	-10
8	29	31
9	21*	-1
H= 4, K= 5		
0	94	-82
1	239	-240
2	194	188
3	114	115
4	53	-54
5	166	-166
6	37	40
7	39	44
8	24*	-13
9	36	-27
H= 4, K= 6		
0	772	-789
1	46	44
2	329	-312
3	331	337
4	51	-36
5	153	-155
6	72	-65
7	112	115
8	46	-48
H= 4, K= 7		
0	37	31
1	69	67
2	305	298
3	23*	-3
4	92	85
5	25*	-10
6	69	64
7	34	25
8	23*	18
H= 4, K= 8		
0	178	172
1	323	-316
2	169	144
3	53	53
4	107	-107
5	73	70
6	89	83
7	52	48
8	24*	-9
H= 4, K= 9		
0	71	65
1	22*	-22
2	201	-210
3	83	-88
4	78	80
5	130	135
6	24*	-12
7	25	-18
8	27	22
H= 4, K= 10		
0	152	141
1	65	69
2	130	121
3	141	-137
4	194	204
5	167	165
6	27	-27
7	80	-83
8	44	45
H= 4, K= 11		
0	323	-321
1	215	-215
2	93	-89
3	67	70
4	65	-65
5	36	-39
6	26	-28
7	24*	-6
H= 4, K= 12		
0	22*	1
1	30	-37
2	47	-50
3	34	31
4	24*	6
5	82	-80
6	24*	-16
7	24*	-1
H= 4, K= 13		
0	112	-105
1	24*	4
2	60	58
3	109	-108
4	24	-26
5	71	-68
6	25*	11
7	30	26
H= 4, K= 14		
0	121	119
1	160	-161
2	87	-88
3	61	58
4	57	-61
5	105	-102
6	23*	19
H= 4, K= 15		
0	265	266
1	24*	-2
2	42	36
3	74	-74
4	24*	-1
5	25*	5
6	23*	-19
H= 4, K= 16		
0	46	36
1	78	78
2	114	-113
3	24*	11
4	28	31
5	29	28
6	23*	-18
H= 4, K= 17		
0	90	90
1	24*	-15
2	45	43
3	76	80
4	26	-27
5	22*	15
H= 4, K= 18		
0	30	-40
1	56	57
2	24*	-8
3	55	-52
4	21*	12
H= 4, K= 19		
0	62	-59
1	24*	-8
2	34	-32
3	25	31
4	21*	15
H= 4, K= 20		
0	47	51
1	26	16
2	43	50
3	23	-21
H= 4, K= 21		
0	22*	-8
1	21*	0
H= 5, K= 0		
2	628	635
4	62	64
6	41	-41
8	28*	-33
H= 5, K= 1		
1	585	571
2	131	-125
3	105	99
4	54	59
5	148	147
6	27*	-16
7	24*	-3
8	53	-59
9	22*	18
H= 5, K= 2		
1	196	-192
2	119	-121
3	176	175
4	75	-74
5	25*	9
6	89	78
7	126	127
8	31	-26
9	46	-50
H= 5, K= 3		
1	55	0
2	409	-415
3	45	49
4	67	71
5	86	-88
6	28	30
7	44	-46
8	107	-108
9	21*	25
H= 5, K= 4		
1	153	-152
2	135	-134
3	48	-42
4	88	-83
5	31	-26
6	27*	7
7	46	-40
8	25*	4
H= 5, K= 5		
1	437	-429
2	268	266
3	30	-24
4	25	-21
5	263	-274
6	25*	26
7	37	-38
8	23*	-13
H= 5, K= 6		
1	153	160
2	104	105
3	121	113
4	24*	-21
5	43	-49
6	62	-67
7	24*	19
8	24*	1
H= 5, K= 7		
1	65	66
2	452	455
3	276	-279
4	98	94
5	25*	-6
6	32	-34
7	87	88
8	47	50
H= 5, K= 8		
1	78	70
2	39	-40
3	157	160
4	24*	-17
5	47	51
6	69	61
7	28	21
8	23*	6
H= 5, K= 9		
1	203	190
2	119	-111
3	62	65
4	122	129
5	233	237
6	24*	-13
7	49	50
8	23*	18
H= 5, K= 10		
1	112	-115
2	24*	29
3	143	-137
4	88	97
5	35	-38
6	24*	-11
7	23*	-11
8	20*	12
H= 5, K= 11		
1	47	-43
2	27	-22
3	191	200
4	63	-67
5	58	55
6	25*	-8
7	32	-33
H= 5, K= 12		
1	113	-114
2	41	-40
3	47	47
4	24*	-12
5	50	-54
6	23	-9
7	34	37
H= 5, K= 13		
1	119	-117
2	27	22
3	24*	-26
4	72	-67
5	51	-50
6	23*	-10
7	29	-26
H= 5, K= 14		
1	24*	30
2	78	-71
3	81	79
4	58	-67
5	24*	12
6	23*	10
H= 5, K= 15		
1	106	-108
2	28	-31
3	42	-49
4	26*	-23
5	24*	-11
6	23*	10
H= 5, K= 16		
1	94	92
2	24*	3
3	58	-59
4	65	56
5	34	-27
H= 5, K= 17		
1	24*	10
2	55	-49
3	26*	-9
4	24*	17
5	22*	-15
H= 5, K= 18		
1	26*	-25
2	24*	14
3	26	-7
4	37	34
H= 5, K= 19		
1	52	55
2	39	-36
3	33	32
H= 5, K= 20		
1	22	-23
2	22*	-17
H= 6, K= 0		
0	657	649
2	727	706
4	261	-261
6	270	272
8	24*	-2
H= 6, K= 1		
0	71	68
1	353	351
2	565	568
3	170	-169
4	212	-215
5	45	-54
6	222	224
7	24*	6
8	45	-42
H= 6, K= 2		
0	121	132
1	347	-338
2	61	-64
3	206	206
4	24*	9
5	113	-115
6	60	61
7	54	-57
8	25*	5
H= 6, K= 3		
0	109	89
1	114	-107
2	310	298
3	327	-324
4	87	-82
5	171	175
6	119	122
7	29	-27
8	46	-41
H= 6, K= 4		
0	30	-28
1	81	-76
2	427	-439
3	54	-45
4	114	116
5	107	-113
6	257	-256
7	24*	-16
8	23*	8
H= 6, K= 5		
0	24	26
1	48	-46
2	124	-123
3	25	18
4	24*	-4
5	26*	-8
6	137	-147
7	43	-38
8	79	91
H= 6, K= 6		
0	105	111
1	438	444
2	168	-165
3	149	144
4	35	-42
5	31	-36
6	118	-116
7	64	67
8	26	-74
H= 6, K= 7		
0	98	-94
1	56	41
2	298	296
3	25*	-17
4	121	-119
5	47	-50
6	24*	-16
7	30	22
8	23*	-1
H= 6, K= 8		
0	77	-78
1	293	287
2	94	-90
3	34	32
4	78	81
5	140	144
6	135	133
7	26*	-3
8	25	-18
H= 6, K= 9		
0	49	40
1	26	21
2	24*	18
3	163	167
4	30	-32
5	48	-46
6	29	31
7	37	33
8	26	-30
H= 6, K= 10		
0	158	-154
1	55	-48
2	216	214
3	255	-257
4	82	87
5	135	139
6	52	47
7	59	-52
H= 6, K= 11		
0	80	80
1	24*	11
2	81	-77
3	123	124
4	39	-46
5	24*	14
6	31	34
7	31	-27
H= 6, K= 12		
0	38	-31
1	44	-48
2	178	181
3	24*	-20
4	72	-71
5	60	-67
6	24*	-9
7	22*	-9
H= 6, K= 13		
0	132	-124
1	23*	15
2	101	104
3	91	-91
4	54	49
5	63	59
6	26*	14
H= 6, K= 14		
0	105	-109
1	65	-63
2	83	-85
3	131	136
4	66	-71
5	85	-84
6	23*	14
H= 6, K= 15		
0	34	-39
1	42	-42
2	31	-34
3	77	80
4	29*	22
5	59	-62
6	31	-30
H= 6, K= 16		
0	29	-30
1	27	-21
2	86	-91
3	26*	-24
4	27*	-8
5	24*	11
H= 6, K= 17		
0	28	-13
1	57	-52
2	56	-59
3	60	63
4	37	-27
H= 6, K= 18		
0	46	42
1	29	22
2	26*	24
3	76	-73
4	27	27
H= 6, K= 19		
0	25*	-16
1	24*	5
2	23*	8
3	46	-45
H= 6, K= 20		
0	22*	2
1	23*	-6
H= 7, K= 0		
2	23*	-2
4	174	-173
6	59	-66
8	86	81
H= 7, K= 1		
1	23*	-15
2	118	-112
3	24*	16
4	111	-111
5	182	180
6	44	46
7	24*	-17
8	57	-60
H= 7, K= 2		
1	619	625
2	182	-177
3	160	-168
4	62	60
5	100	99
6	25*	2
7	24*	29
8	24*	0
H= 7, K= 3		
1	373	-362
2	271	-261
3	25*	-2
4	166	-160
5	26	-11
6	24	-15
7	50	-46
8	56	-52
H= 7, K= 4		
1	23	24
2	151	-147
3	34	-34
4	109	109
5	115	116
6	25*	4
7	79	-75
8	24*	-26
H= 7, K= 5		
1	235	238
2	89	-95
3	78	-73
4	60	-59
5	163	-168
6	32	24
7	27*	78
8	22*	-3
H= 7, K= 6		
1	48	-42
2	25*	27
3	26	20
4	100	-104
5	134	-133
6	36	34
7	27*	-4
8	24*	-2
H= 7, K= 7		
1	54	46
2	184	171
3	126	-125
4	60	59
5	35	36
6	75	76
7	27	20
8	23*	-6
H= 7, K= 8		
1	45	40
2	144	148
3	35	38
4	68	-72
5	106	-105
6	31	25
7	24*	18
8	21*	-12
H= 7, K= 9		
1	149	-138
2	24*	-10
3	34	-38
4	40	45
5	55	63
6	27	-20
7	24*	1
H= 7, K= 10		
1	61	57
2	116	114
3	69	-69
4	52	61
5	64	64
6	36	-43
7	23*	1
H= 7, K= 11		
1	153	156
2	77	-76
3	124	121
4	131	-134
5	24*	-20
6	26	-18
7	22*	-2
H= 7, K= 12		
1	39	37
2	79	-78
3	24*	1
4	78	84
5	24*	10
6	22*	6
H= 7, K= 13		
1	93	96
2	111	115
3	37	39
4	75	-77
5	24*	-5
6	24*	6
H= 7, K= 14		
1	54	-58
2	43	50
3	54	52
4	27*	7
5	24*	-6
6	21*	0
H= 7, K= 15		
1	85	-85
2	24*	22
3	52	-51
4	46	53
5	57	-56
H= 7, K= 16		
1	61	-61
2	57	51
3	50	-44
4	40	-35
5	23*	23
H= 7, K= 17		
1	64	-59
2	33	-28
3	24*	9
4	52	49
H= 7, K= 18		
1	23*	0
2	35	-32
3	22*	3
H= 7, K= 19		
1	23*	-1
2	29	24
H= 8, K= 0		
0	33	35
2	124	-118
4	104	105
6	83	81
8	87	85
H= 8, K= 1		
0	60	-59
1	36	-48
2	166	148
3	25*	11
4	242	-233
5	64	-59
6	64	61
7	93	84
8	58	-53
H= 8, K= 2		
0	183	-180
1	146	-136
2	124	-119
3	245	-241
4	87	86
5	127	-126
6	24*	5
7	25*	-2
8	23*	-5
H= 8, K= 3		
0	21*	12
1	23	-12
2	23*	-24
3	172	173
4	24*	-7
5	67	59
6	24*	-1
7	85	-76
8	22*	-9
H= 8, K= 4		
0	87	88
1	67	-63
2	24*	-2
3	41	40
4	29	-38
5	162	-165
6	32	-26
7	26*	1
8	37	-36
H= 8, K= 5		
0	23*	21
1	140	-135
2	128	-131
3	24*	9
4	221	226
5	26	21
6	63	-64
7	27	-24
8	69	64
H= 8, K= 6		
0	294	291
1	90	83
2	85	-90
3	120	-112
4	81	77
5	135	128
6	24*	-30
7	25*	10
8	23*	7
H= 8, K= 7		
0	32	-3
1	23*	-12
2	25*	-14
3	33	-32
4	87	-80
5	25*	-16
6	33	26
7	25*	18
H= 8, K= 8		
0	154	-156
1	136	133
2	137	-137
3	113	-113
4	51	55
5	157	162
6	59	-69
7	23*	-1
H= 8, K= 9		
0	117	121
1	24*	23
2	89	-88
3	78	16
4	24*	13
5	59	-51
6	42	40
7	22*	9
H= 8, K= 10		
0	107	-105
1	71	-71
2	153	152
3	42	-43
4	107	-106
5	25	-16
6	23*	11
7	22*	1
H= 8, K= 11		
0	67	78
1	23*	-12
2	61	-61
3	59	-58
4	49	46
5	31	35
6	22*	-6
H= 8, K= 12		
0	171	163
1	24*	-9
2	178	183
3	79	-77
4	24*	-3
5	54	-59
6	44	43
H= 8, K= 13		
0	44	-50
1	99	95
2	34	32
3	43	-46
4	44	-50
5	62	61
6	39	-43
H= 8, K= 14		
0	122	119
1	52	54
2	24*	0
3	46	44
4	66	-65
5	23*	4
H= 8, K= 15		
0	24*	-25
1	39	40
2	26*	3
3	27*	20
4	27*	23
5	48	-48
H= 8, K= 16		
0	93	-91
1	26	-26
2	49	-49
3	89	90
4	61	-62
H= 8, K= 17		
0	33	28
1	44	-44
2	24*	20
3	39	-26
4	23*	-1
H= 8, K= 18		
0	91	-84
1	24*	16
2	28	-24
3	35	31
H= 8, K= 19		
0	23*	6
1	23*	21
H= 9, K= 0		
2	151	-145
4	120	122
6	24*	-20
8	64	61
H= 9, K= 1		
1	50	-52
2	245	-244
3	141	134
4	134	-130
5	56	55
6	62	62
7	33	40
8	51	-46
H= 9, K= 2		
1	140	-144
2	83	85
3	289	-281
4	114	110
5	27	31
6	67	-62
7	100	-103
8	22*	-2
H= 9, K= 3		
1	38	34
2	128	-124
3	49	50
4	63	-56
5	66	-73
6	25	-31
7	52	-52
8	27	-21
H= 9, K= 4		
1	24*	13
2	111	110
3	34	34
4	24*	13
5	85	86
6	28	-18
7	26*	-21
8	29	-29
H= 9, K= 5		
1	23*	-11
2	43	36
3	100	-98
4	99	103
5	45	-45
6	37	-45

Table 2 (*cont.*)

L	FO	FC
H=	9, K=	5
7	24*	11
H=	9, K=	6
1	53	-55
2	32	29
3	100	99
4	68	-66
5	88	93
6	49	48
7	47	45
H=	9, K=	7
1	34	-36
2	42	-44
3	51	-50
4	60	60
5	24*	22
6	60	64
7	24*	3
H=	9, K=	8
1	66	-64
2	24*	-5
3	41	-37
4	69	-75
5	24*	20
6	26*	17
7	29	29
H=	9, K=	9
1	57	56
2	50	44
3	24*	-7
4	48	51
5	24*	-5
6	25*	-21
7	33	21
H=	9, K=	10
1	23*	1
2	169	-166
3	24*	15
4	24*	0
5	24*	-1
6	22*	0
H=	9, K=	11
1	34	38
2	24	-14
3	71	71
4	38	-40
5	56	51
6	23*	-23
H=	9, K=	12
1	25	-32
2	40	-41
3	24*	-24
4	54	60
5	45	-47
6	23*	-13
H=	9, K=	13
1	28	32
2	25	-25
3	26*	4
4	82	-87
5	36	33
H=	9, K=	14
1	24*	15
2	62	54
3	55	-49
4	35	-34
5	23*	4
H=	9, K=	15
1	24*	1
2	25*	11
3	75	-72
4	49	50
H=	9, K=	16
1	71	76
2	24*	-16
3	23*	14
4	67	-68
H=	9, K=	17
1	67	-73
2	22*	13
3	22*	-1
H=	9, K=	18
1	38	39
2	23*	-12
H=	10, K=	0
0	327	331
2	50	31
4	59	58
6	67	68
H=	10, K=	1
0	152	-150
1	64	-71
2	164	-157
3	82	85
4	24*	18
5	79	-80
6	52	-57
7	64	61
H=	10, K=	2
0	205	202
1	24*	-18
2	156	157
3	141	-139
4	40	-37
5	28	33
6	27*	2
7	47	-43
H=	10, K=	3
0	103	109
1	44	41
2	87	-81
3	26	23
4	24*	18
5	24*	-11
6	108	-101
7	26*	-33
H=	10, K=	4
0	326	-326
1	24*	11
2	81	75
3	190	185
4	147	-145
5	97	-100
6	36	-36
7	22*	4
H=	10, K=	5
0	28	25
1	48	-48
2	108	102
3	66	59
4	92	90
5	39	40
6	24*	-5
7	23*	-19
H=	10, K=	6
0	298	-305
1	87	88
2	133	-127
3	91	99
4	36	-36
5	24*	2
6	48	-44
7	21*	8
H=	10, K=	7
0	169	-172
1	45	41
2	77	-72
3	24*	3
4	72	72
5	26	-20
6	79	75
7	22*	-9
H=	10, K=	8
0	137	190
1	41	-38
2	25	5
3	51	46
4	80	78
5	44	46
6	23*	1
7	21*	14
H=	10, K=	9
0	127	132
1	32	-27
2	73	-78
3	136	-139
4	26	-16
5	25	12
6	23*	10
H=	10, K=	10
0	76	72
1	137	-132
2	78	82
3	123	123
4	82	83
5	24*	-17
6	68	66
H=	10, K=	11
0	24*	18
1	71	-66
2	43	48
3	24*	21
4	25*	-12
5	24*	14
6	44	-46
H=	10, K=	12
0	73	-74
1	24*	-12
2	68	-68
3	68	-70
4	57	60
5	23*	2
H=	10, K=	13
0	60	-72
1	50	50
2	32	-23
3	51	54
4	25*	-7
5	22*	-2
H=	10, K=	14
0	25*	20
1	37	34
2	82	-84
3	84	-85
4	42	-35
H=	10, K=	15
0	46	39
1	35	28
2	25*	-12
3	80	-77
4	33	27
H=	10, K=	16
0	32	-33
1	23*	-4
2	25	-17
3	36	30
H=	10, K=	17
0	47	45
1	21*	-9
2	32	31
H=	11, K=	0
2	26*	-23
4	81	-82
6	25*	-13
H=	11, K=	1
1	143	140
2	30	-27
3	76	78
4	56	52
5	24*	-21
6	26	-19
7	34	40
H=	11, K=	2
1	142	144
2	60	-54
3	39	-37
4	66	-66
5	45	51
6	24*	6
7	23*	-27
H=	11, K=	3
1	29	25
2	78	-75
3	24*	-9
4	53	53
5	24*	-5
6	29	-41
7	24	-24
H=	11, K=	4
1	195	189
2	26	-25
3	24*	5
4	45	47
5	83	-89
6	36	37
7	24	25
H=	11, K=	5
1	238	-240
2	50	44
3	98	-96
4	24*	5
5	85	-81
6	24*	-27
7	33	-31
H=	11, K=	6
1	130	-130
2	99	98
3	78	81
4	24*	11
5	26*	-8
6	24*	-9
H=	11, K=	7
1	25*	10
2	111	100
3	71	-74
4	24	8
5	40	-30
6	22	9
H=	11, K=	8
1	70	-70
2	24*	20
3	24*	-12
4	87	-87
5	24*	13
6	23*	-9
H=	11, K=	9
1	163	165
2	24*	10
3	90	87
4	45	39
5	92	102
6	21*	3
H=	11, K=	10
1	110	112
2	57	-53
3	24*	-10
4	36	33
5	60	-65
6	21*	8
H=	11, K=	11
1	44	-44
2	24*	12
3	80	79
4	29	16
5	28	33
H=	11, K=	12
1	24*	11
2	49	48
3	49	46
4	90	95
5	22*	-5
H=	11, K=	13
1	88	-88
2	40	-33
3	32	-33
4	23*	-22
H=	11, K=	14
1	54	-55
2	24*	11
3	22*	-1
4	22*	13
H=	11, K=	15
1	34	-32
2	40	-40
3	32	-28
H=	11, K=	16
1	24	-20
2	21*	-6
H=	12, K=	0
0	81	84
2	25*	23
4	32	-35
6	23*	11
H=	12, K=	1
0	293	298
1	35	33
2	30	18
3	81	-86
4	92	95
5	80	-81
6	23	-14
H=	12, K=	2
0	88	90
1	154	158
2	41	-39
3	24*	2
4	24*	27
5	27*	-12
6	26	34
H=	12, K=	3
0	307	318
1	26*	-29
2	43	34
3	60	-62
4	24*	9
5	98	97
6	23*	-21
H=	12, K=	4
0	187	-184
1	43	-40
2	24*	-10
3	96	-95
4	24*	-2
5	41	-43
6	26	18
H=	12, K=	5
0	183	-179
1	24*	-10
2	65	59
3	45	45
4	92	-100
5	59	65
6	24	-25
H=	12, K=	6
0	201	-204
1	24*	12
2	40	44
3	24*	3
4	129	-130
5	39	-33
6	21*	-20
H=	12, K=	7
0	167	-163
1	80	79
2	35	30
3	30	-33
4	25*	-12
5	46	-47
6	22*	0
H=	12, K=	8
0	110	109
1	95	-90
2	24*	23
3	69	67
4	25	20
5	23*	1
6	23*	-4
H=	12, K=	9
0	81	81
1	51	52
2	24*	7
3	47	-41
4	68	-62
5	25*	-2
H=	12, K=	10
0	95	97
1	60	-66
2	39	33
3	28*	23
4	93	98
5	23*	-5
H=	12, K=	11
0	57	53
1	58	-51
2	61	59
3	112	104
4	33	-39
5	21	-13
H=	12, K=	12
0	66	55
1	80	68
2	26*	-23
3	25*	-7
4	23*	-15
H=	12, K=	13
0	27	-25
1	44	-46
2	27*	27
3	82	85
H=	12, K=	14
0	25*	-14
1	25*	-2
2	37	-38
3	22*	-16
H=	12, K=	15
0	61	-54
1	36	35
2	40	-44
H=	13, K=	0
2	36	38
4	124	-125
6	45	44
H=	13, K=	1
1	25	-30
2	85	84
3	53	53
4	28*	1
5	25*	-23
6	30	-34
H=	13, K=	2
1	172	173
2	24	-16
3	24*	5
4	27*	21
5	25*	1
6	25	24
H=	13, K=	3
1	52	-54
2	50	-44
3	44	-44
4	34	-32
5	106	109
6	22*	-10
H=	13, K=	4
1	98	-97
2	24*	-8
3	24*	-19
4	100	98
5	25*	-29
6	23	-16
H=	13, K=	5
1	24*	-4
2	107	-115
3	40	-36
4	53	-54
5	25*	22
6	26	27
H=	13, K=	6
1	39	-45
2	24*	5
3	28*	9
4	28	-36
5	23*	-14
H=	13, K=	7
1	30	33
2	24*	0
3	55	-51
4	24*	-9
5	48	-47
H=	13, K=	8
1	42	42
2	26*	-1
3	25*	8
4	37	-41
5	26	25
H=	13, K=	9
1	29*	18
2	26*	-6
3	31	31
4	25	-12
5	25	-18
H=	13, K=	10
1	34	-22
2	25*	14
3	45	-45
4	23*	4
H=	13, K=	11
1	66	63
2	51	49
3	37	44
4	39	-36
H=	13, K=	12
1	26*	17
2	24*	-26
3	24*	12
H=	13, K=	13
1	32	29
2	23*	18
H=	13, K=	14
1	22	13
H=	14, K=	0
0	89	88
2	123	123
4	24*	0
H=	14, K=	1
0	98	99
1	36	41
2	24*	-15
3	27*	15
4	33	-35
5	23*	12
H=	14, K=	2
0	63	65
1	119	124
2	77	-77
3	31	-30
4	49	52
5	24*	-6
H=	14, K=	3
0	109	-108
1	24*	-10
2	57	-60
3	57	60
4	25*	1
5	25	28
H=	14, K=	4
0	32	-29
1	41	40
2	111	-110
3	48	-54
4	49	45
5	22*	18
H=	14, K=	5
0	119	-122
1	33	29
2	29	-25
3	27*	-25
4	32	31
5	22*	-12
H=	14, K=	6
0	77	-76
1	30	-24
2	51	53
3	38	-41
4	42	-40
5	22*	8
H=	14, K=	7
0	107	103
1	25*	0
2	46	53
3	51	-50
4	23*	10
H=	14, K=	8
0	26*	-20
1	54	-47
2	25*	17
3	23	13
4	23*	-28
H=	14, K=	9
0	63	-62
1	26*	-10
2	24*	-7
3	23*	13
4	22*	2
H=	14, K=	10
0	58	60
1	39	35
2	29	26
3	22*	-8
H=	14, K=	11
0	55	-67
1	43	-42
2	52	-51
3	36	-30
H=	14, K=	12
0	54	52
1	42	45
2	23*	25
H=	14, K=	13
0	40	40
H=	15, K=	0
2	72	75
4	23*	3
H=	15, K=	1
1	144	141
2	31	-22
3	73	79
4	24*	-3
H=	15, K=	2
1	58	-64
2	26*	7
3	54	-52.
4	39	41
H=	15, K=	3
1	76	-72
2	33	32
3	45	-52
4	40	34
H=	15, K=	4
1	62	-62
2	26*	-23
3	50	50
4	24*	-4
H=	15, K=	5
1	85	-87
2	26*	23
3	50	-57
4	23*	21
H=	15, K=	6
1	70	64
2	25*	-3
3	24*	2
4	22*	-21
H=	15, K=	7
1	25*	1
2	24*	-21
3	21*	3
H=	15, K=	8
1	24*	15
2	23*	-1
3	22*	-15
H=	15, K=	9
1	53	53
2	47	44
H=	15, K=	10
1	52	-58
2	23*	-10
H=	15, K=	11
1	22*	18
H=	16, K=	0
0	182	188
2	125	126
H=	16, K=	1
0	57	-56
1	26	27
2	41	-41
3	25	27
H=	16, K=	2
0	47	55
1	25*	-14
2	24	17
3	23*	-3
H=	16, K=	3
0	27	-19
1	90	-85
2	29	26
3	28	-17
H=	16, K=	4
0	101	-102
1	41	47
2	89	-97
3	68	67
H=	16, K=	5
0	99	98
1	25*	-7
2	31	36
3	34	-32
H=	16, K=	6
0	77	-81
1	24*	3
2	40	-34
H=	16, K=	7
0	28	23
1	23*	19
2	48	-51
H=	16, K=	8
0	49	46
1	22	14
H=	17, K=	0
2	57	60
H=	17, K=	1
1	28	32
2	25	-30
H=	17, K=	2
1	23*	-19
2	31	-29
H=	17, K=	3
1	24	-26
H=	17, K=	4
1	90	89

The molecule has fairly exact non-crystallographic mirror symmetry, and equality of related bond lengths is to be expected. However, inspection of Table 3 reveals differences of as much as 0·030 Å, whereas the conventionally estimated standard deviation of bond lengths is 0·003 to 0·004 Å. While the possibility that these differences are real cannot be ruled out, it seems likely that the e.s.d. is not in this case a realistic estimate of the precision achieved. It is known that in general the block-diagonal approximation gives e.s.d.'s which are lower than those given by the full-matrix calculation. Moreover, systematic errors may well be present in the intensity data, or the model, or both. The low yield of observed intensities and the severe, anisotropic, and possibly non-rigid thermal motion may be cited as possible sources of error. It is suggested that 0·014 Å and 0·7° are more realistic estimates of standard error for bond lengths and angles. An attempt was made to characterize the thermal motion in terms of rigid-body vibration, using the methods of Cruickshank (1956). The centre of libration was assumed, without proof (but with some justification from the relative orientations of the thermal ellipsoids, as shown in Fig. 1), to coincide with the centre of gravity of the carbon skeleton. The results provide a measure of support for the hypothesis of rigid-body motion. The root-mean-square deviation of calculated from observed values of U_{ij} is 0·006 Å^2. The mean value of the e.s.d.'s of U_{ij} (Table 1) is 0·0012 Å^2, but as we have seen, the parameter errors are seriously underestimated. The amplitudes of libration about the principal axes are 6·1, 4·7 and 3·4°. The axis of greatest libration corresponds to the direction of projection of Fig. 3(*b*). The appropriate bond-length corrections are shown in Table 3; the agreement between equivalent distances is not improved by the correction, but the mean values are probably a little more reliable.

Table 3. *Comparison of bond lengths*

Bond	Uncorrected length	E. s. d. (×10³)	Libration correction (×10³)	Corrected length	Mean
C(1)—C(2)	1·332 Å	4 Å	10 Å	1·342 Å	1·346 Å
C(9)—C(10)	1·340	4	10	1·350	
C(2)—C(3)	1·456	4	13	1·469	1·484
C(7)—C(9)	1·486	4	12	1·498	
C(3)—C(4)	1·419	3	9	1·428	1·413
C(6)—C(7)	1·389	3	9	1·398	
C(4)—C(5)	1·368	4	12	1·380	1·376
C(5)—C(6)	1·360	4	11	1·371	
C(3)—C(8)	1·385	3	11	1·396	1·400
C(7)—C(8)	1·392	3	12	1·404	
C(14)-C(1)	1·485	4	12	1·497	1·486
C(10)-C(11)	1·461	4	13	1·474	
C(11)-C(12)	1·401	3	9	1·410	1·405
C(13)-C(14)	1·391	3	9	1·400	
C(14)-C(15)	1·400	3	9	1·409	1·405
C(16)-C(11)	1·392	3	9	1·401	
C(12)-C(13)	1·376	4	11	1·387	
C(15)-C(16)	1·363	3	11	1·374	

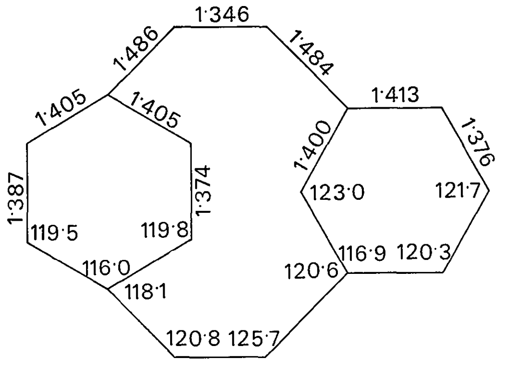


Fig. 2. Mean bond lengths (Å) and angles (°) assuming mirror symmetry.

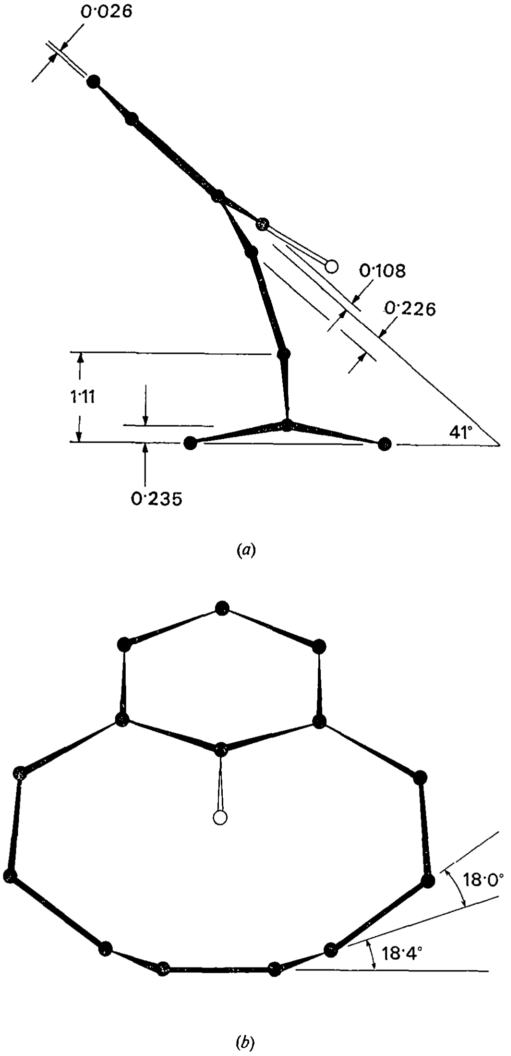


Fig. 3. Two views of the slightly idealized carbon skeleton. Distances shown are in Å. The open circle indicates the position of H(8). The directions of view are: (*a*) the normal to the approximate mirror plane; (*b*) the intersection of the mirror plane with the mean plane of the *para*-bridged ring.

Table 3 (*cont.*)

Mean aromatic bond distance	1·396 Å
Mean double-bond distance	1·346
Mean single-bond distance	1·485
Mean C–H bond distance	0·99 (0·91 to 1·07)

Table 4. *Comparison of bond angles*

Angle	Value	e.s.d.	Mean
C(14)-C(1)—C(2)	120·5°	0·3°	120·8°
C(9)—C(10)-C(11)	121·1	0·2	
C(1)—C(2)—C(3)	126·1	0·3	125·8
C(7)—C(9)—C(10)	125·4	0·2	
C(2)—C(3)—C(4)	122·6	0·2	122·3
C(6)—C(7)—C(9)	122·0	0·2	
C(2)—C(3)—C(8)	121·0	0·2	120·6
C(8)—C(7)—C(9)	120·2	0·2	
C(4)—C(3)—C(8)	116·3	0·2	116·9
C(6)—C(7)—C(8)	117·5	0·2	
C(3)—C(4)—C(5)	120·2	0·2	120·3
C(5)—C(6)—C(7)	120·4	0·2	
C(4)—C(5)—C(6)	121·7	0·2	
C(3)—C(8)—C(7)	123·0	0·2	
C(10)-C(11)-C(12)	121·9	0·2	121·6
C(13)-C(14)-C(1)	121·3	0·2	
C(10)-C(11)-C(16)	118·2	0·2	118·1
C(15)-C(14)-C(1)	118·0	0·2	
C(12)-C(11)-C(16)	116·9	0·2	117·0
C(13)-C(14)-C(15)	117·1	0·2	
C(11)-C(12)-C(13)	119·7	0·2	119·5
C(12)-C(13)-C(14)	119·3	0·2	
C(14)-C(15)-C(16)	119·8	0·2	119·8
C(15)-C(16)-C(11)	119·9	0·2	
Mean C–C–H angle			119 (116 to 123)

Discussion

The mean bond lengths and angles given in Table 3 and Fig. 2 are consistent with expectation and require no comment. Views of the molecule are given in Fig. 3. It is clear that whatever may be the case in solution, the molecule in the crystal does not have the predicted *mm*2 symmetry. Far from being mutually perpendicular, the aromatic rings are inclined to each other at 41°.

Significant strain may be inferred from the distortion from planarity of the aromatic rings and their substituents. The distortion of the *meta*-bridged ring is comparatively mild: the displacements noted in Fig. 3(*a*) (0·026, 0·108 and 0·226 Å) may be compared with the corresponding displacements of 0·042, 0·143 and 0·41 Å for [2.2]metacyclophane (Brown, 1953). The distortion of the *para*-bridged ring is much more severe: the displacements of the bridgehead atoms and their substituents from the mean plane of the unsubstituted atoms are 0·235 and 1·11 Å. The distortion may also be expressed in terms of the angles α and β, as in Fig. 3(*b*). The values given there (18·4 and 18·0°) may be compared with the corresponding values of 14 and 15° found for the highly strained compound [2.2]paracyclophane-1,9-diene (Coulter & Trueblood, 1963).

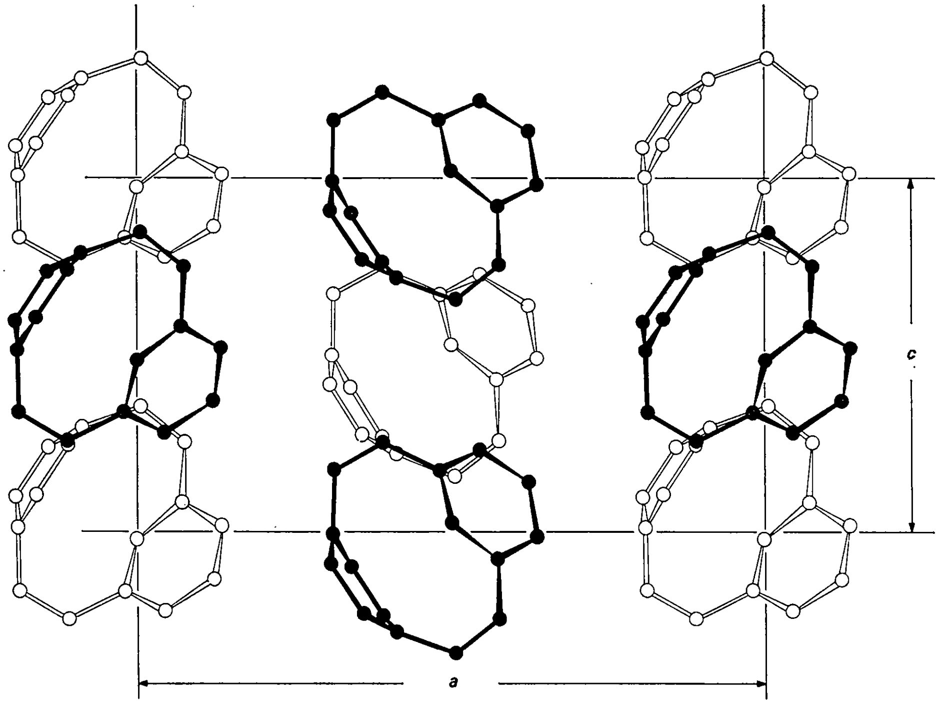


Fig. 4. A projection along **b** of that part of the structure lying between $y=0$ and $y=\frac{1}{2}$. The remainder is related to the part shown by a centre of symmetry at $\frac{1}{2},\frac{1}{2},\frac{1}{2}$.

In spite of this distortion C(8) lies only 2·71 Å, and H(8) only 2·16 Å, from the plane of the unsubstituted atoms of the *para*-bridged ring. If the predicted *mm*2 symmetry were to be adopted without further distortion, simply by constraining the *meta*-bridged ring and the bridging atoms to lie in a plane perpendicular to that of the *para*-bridged ring, these distances would be reduced to 2·57 and 1·59 Å. It seems certain that such a close approach would have to be avoided by further severe distortion of the molecule. Whether the energy required to cause such distortion would be sufficient to prevent significant 'flipping' of the molecule (in solution) is an open question.

[A distance of 0·98 Å has been assumed for C(8)–H(8). This is the distance actually observed; moreover it is in good agreement with C–H distances observed in many X-ray structure analyses. It does not represent internuclear distance, but rather the distance between centres of gravity of electron-density distributions. It is of course the electron-density distribution which is relevant in consideration of non-bonded contact].

The packing of the molecules is illustrated in Fig. 4. The shortest intermolecular contacts are: C–C, 3·59; C–H, 2·79; H–H, 2·46 Å.

The problem was suggested, and the specimen material supplied by Professor V. Boekelheide. Dr C. K. Johnson's (1965) *ORTEP* program was used for Fig. 1. All other computations (including the phasing of the reflexions) were carried out using the programs of Ahmed, Hall, Pippy & Huber (1966). The assistance of those named is gratefully acknowledged.

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