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The Structure of Methyl 1:2-benzanthraquinones. II. The Crystal and Molecular Structure of 5-Methyl-1:2-benzanthraquinone

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The crystal structure of 5-methyl-1:2-benzanthraquinone has been determined by two-dimensional Fourier methods, and accurate atomic coordinates were obtained by three-dimensional least-squares refinement. The crystals are orthorhombic with 4 molecules per unit cell:

$$a = 14{\cdot}13,\ b = 23{\cdot}27,\ c = 3{\cdot}94\ \text{Å},\ \text{space group}\ P2_1nb\ .$$

The reliability factor (R) for 635 independent reflexions is 0·082 and the standard deviations of the coordinates are approximately 0·01 Å. The C = O bonds are 1·218 Å, the C–C single bonds have a mean value of 1·491 Å, and there is one long aromatic bond of 1·472 Å which appears to be caused by steric interference between an oxygen atom and a hydrogen atom attached to one of the benzene rings. This oxygen atom is 0·173 Å distant from the mean plane and several carbon atoms have deviations of 0·05–0·15 Å from the plane. Asymmetrical temperature parameters were included in the refinement process and corrections to the coordinates have been calculated to allow for rotational-oscillation vibrations.

Introduction

The structure of 5-methyl-1:2-benzanthraquinone (Fig. 1) is of particular interest because it is a derivative of an active carcinogenic agent 5-methyl-1:2-benzanthracene; the compound whose structure is described in Part III is a quinone derived from an inactive methyl-substituted 1:2-benzanthracene. The unit cells and space groups of a series of substituted 1:2-benzanthraquinones were given in Part I (Ferrier & Iball, 1960).

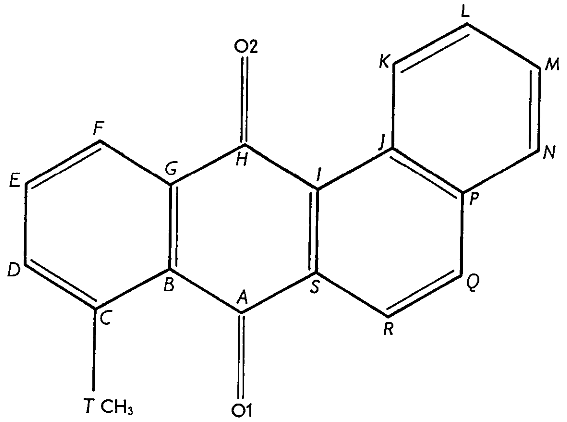

Fig. 1. 5-Methyl-1:2-benzanthraquinone ($C_{19}O_2H_{12}$).

Crystal data

The crystals obtained from ethyl-acetate solutions were thin yellow needles. They are orthorhombic with unit-cell dimensions

$$a = 14{\cdot}13 \pm 0{\cdot}01,\ b = 23{\cdot}27 \pm 0{\cdot}02,\ c = 3{\cdot}94 \pm 0{\cdot}01\ \text{Å}\ .$$

(The c axis is parallel to the axis of the needles). Space-group $P2_1nb$; 4 molecules per unit cell; d(obs.) (24·5 °C.) = 1·389 g.cm.$^{-3}$; d(calc.) = 1·395 g.cm.$^{-3}$. The systematic absences ($h0l$ when $h+l$ is odd and $hk0$ when k is odd) satisfy two space groups $P2_1nb$ and $Pmnb$ but the latter requires 8 asymmetric units and since the molecule cannot have any symmetry the former space group was adopted.

Intensity measurement

Weissenberg photographs were taken about the three axes, the zero, first, second and third layers of c, and the zero layers of a and b. Multiple film packs were used to record the strong and weak reflections and the Weissenberg camera was fitted with a device which gave an integration in the direction of travel. This was found to be a great advantage since it gave a spot with uniform intensity in one direction and it was much easier to estimate the intensities. The estimates were made by two observers independently with the normal calibration scale. In addition, if the amount of linear integration is suitably chosen in relation to the length of the crystal, it removes entirely the difficulty which arises in ordinary Weissenberg photographs of upper layer lines due to the contraction of spots on one half of the film and elongation on the other side. Cu $K\alpha$ radiation was used throughout and, in addition to the photographic method, some zero-level reflections on each axis were measured with a G.-M. counter spectrometer. The crystals were small and so absorption corrections could be ignored except for crystals mounted on the *a* axis and in this case corrections were calculated by means of the method of Albrecht (1939).

Determination of the structure

It was expected that a plot of the c^* zero layer of the weighted reciprocal lattice would show the orientation of the two molecules which are related by the glide plane. However only six groups of spots appeared on the plot near to the 'benzene ring circle' (Fig. 2). The two molecules must therefore have an orientation with one diameter of the benzene rings parallel to one axis of the cell. From the centres of gravity of the six groups of spots it was calculated that the molecule was tilted at an angle of approximately 25° about a line parallel to the *a*-axis. There are three ways in which the molecule can be arranged to satisfy the orientation indicated by Fig. 2. The position of the molecule along *a*, the screw-axis direction, is indeterminate and so the values of F_o for the $h00$ planes are dependent only on the orientation of the molecule. Each of the possible arrangements was tried in turn and only one gave reasonable agreement between calculated and observed structure factors. For these preliminary structure-factor calculations it was assumed that the molecule was planar and that the bond lengths and bond angles were similar to these found in other compounds. The scattering curves of Berghuis *et al.* (1955) were used with a temperature factor B of 3·7 Å^2 for all atoms. With the approximate orientation determined, the values of the $0k0$ structure factors were calculated for various positions along the *b* axis. The best agreement with the observed values was when the 'centre' of the molecule was at approximately $b/8$ from the screw axis. Further trials of small rotations of the molecule in the (001) plane showed that better agreement between F_o and F_c for $h00$ planes was obtained by (i) a rotation 1·5° clockwise or (ii) 2·5° anti-clockwise. An attempt to refine the atomic coordinates on the assumption that (i) was correct failed, but when (ii) was used the structure was determined in a straightforward manner. Sixty-five of the largest structure factors were calculated and the observed values put on the same scale. With the omission of three factors where agreement was bad, the R-factor $[R=\Sigma||F_o|-|F_c||/\Sigma|F_o|]$ was 0·15. An electron-density projection was computed with the calculated phases of these 62 structure factors and the map shown in Fig. 3 was obtained. The atoms were all resolved and the peak positions were determined by means of the semi-analytical method of Burns & Iball (1955). With the x and y coordinates obtained in this way all the structure factors for the $hk0$ reflections were calculated and when a temperature factor (B) of 4·5 was used, instead of the previous value of 3·7, the R-factor for all the observed reflections was 0·30. An (F_o-F_c) Fourier synthesis was now computed and the indicated shifts of atom positions were doubled. Re-calculation of the structure factors showed that for three reflections the agreement with the observed values was very poor. However, these three reflections are very sensitive to a slight movement of the whole molecule along the *b* axis direction and when all the y coordinates were reduced by $y/b=0{\cdot}003$ there was a marked improvement in the agreement. Successive F_o and (F_o-F_c) Fourier syntheses were now computed with increasing numbers of the observed structure factors and calculated phases. A value of 0·23 was obtained for the R-factor without any hydrogen atoms. The later (F_o-F_c) maps showed quite clearly approximate positions for the hydrogen atoms. Fig. 4 is such a map and on this the crosses represent calculated hydrogen positions assuming a C–H bond of 1·05 Å. The R-factor was reduced to 0·21 without the hydrogen atoms and it dropped only by 0·01 when they were included.

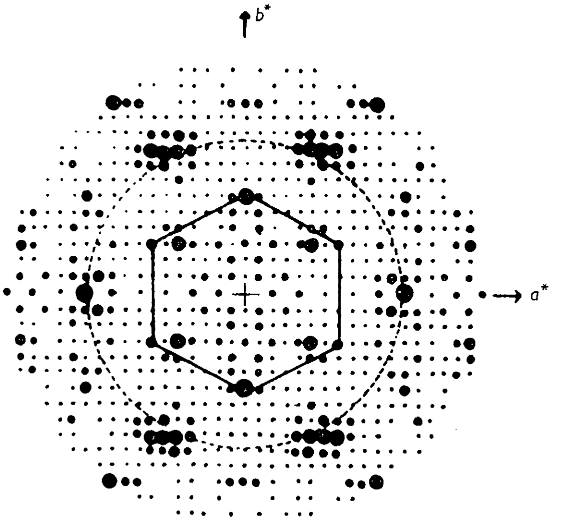

Fig. 2. $hk0$ weighted reciprocal lattice.

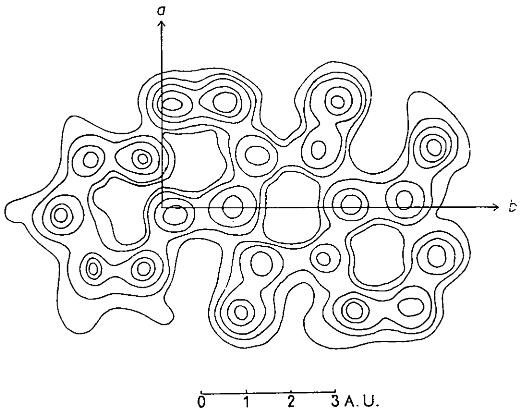

Fig. 3. First $hk0$ Fourier (contours at arbitrary intervals).

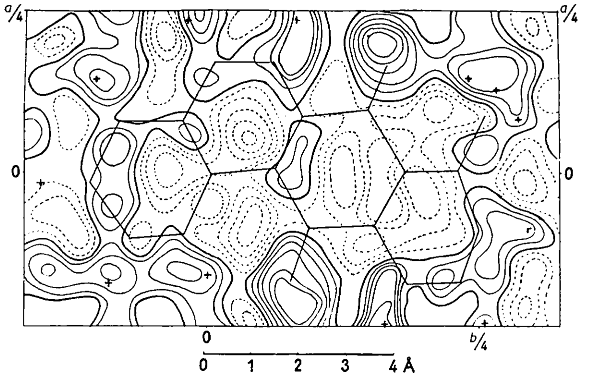

Fig. 4. $hk0$ $[F_o - F_c$ (carbon and oxygen)] Fourier. Contours at intervals of 0·2 e.Å^{-2}; the negative contours are in dotted lines. (The calculated hydrogen atom positions are shown by crosses).

The $0kl$ reflections were now considered. A tilt of the molecule of 26° about a line parallel to a was adopted and the position of the molecule along the c axis was found by calculating the structure factors of three reflections, 002, 004 and 062 for various positions from $z=0$ to $z=c/2$. It was found that the most probable position for the mass centre was $z/c=0{\cdot}09$. Calculation of all the observed $0kl$ structure factors gave an R-factor of 0·23. With the phases thus calculated an F_o Fourier map was computed and this confirmed the position and orientation of the molecule. It was concluded that the coordinates obtained at this stage were substantially correct and that a refinement by the least-squares techniques was justified.

Refinement by least-squares

The first stage of the refinement was carried out on the 'Pegasus' computer at Northampton Polytechnic, London with the program written by Dr Judith Milledge of University College, London. With the $hk0$ reflections, four cycles of refinement reduced the R-factor from 0·20 to 0·13. This program did not refine the individual temperature factors (B) so these were estimated from $(F_o - F_c)$ Fourier maps. A Fourier synthesis with the phases from the last cycle gave the map shown in Fig. 5. A refinement of the $0kl$ reflections was carried out in the same way but, since in this projection there is considerable overlap of the atoms, the y coordinates were not allowed to change.

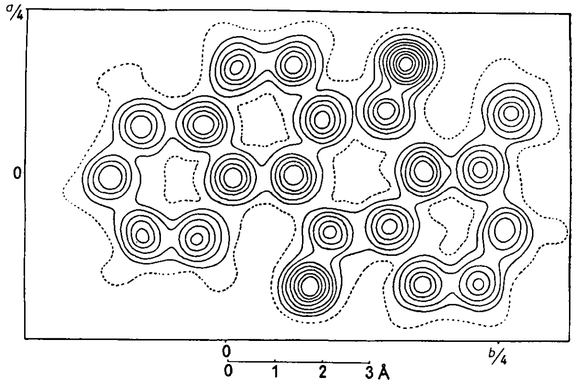

Fig. 5. Final $hk0$ Fourier. Contours are at 1 e.Å^{-2} and the 1e contour is dotted.

It was realized that the atomic vibrations were not isotropic and the subsequent refinement was done on the 'Pegasus' computer at Leeds University with the program written by Dr D. W. J. Cruickshank. With this program it is possible to refine with either isotropic or anisotropic thermal parameters. It was used with three-dimensional data (635 independent reflections). The number of reflections used is far smaller than the theoretical number for Cu $K\alpha$ radiation but the

Table 1. *Final carbon atom coordinates and standard deviations* (Å)

Atom	x		$\sigma(x)$	y		$\sigma(y)$	z		$\sigma(z)$
O1	2·418	(2·427)	0·007	3·860	(3·867)	0·006	0·698	(0·700)	0·009
O2	−2·413	(−2·425)	0·007	1·795	(1·794)	0·006	−0·217	(−0·218)	0·013
A	1·302	(1·308)	0·008	3·390	(3·395)	0·008	0·520	(0·522)	0·011
B	0·078	(0·078)	0·010	4·138	(4·141)	0·007	0·991	(0·992)	0·010
C	0·120	(0·120)	0·011	5·414	(5·418)	0·009	1·614	(1·616)	0·010
D	−1·082	(−1·083)	0·011	5·983	(5·986)	0·009	1·952	(1·954)	0·010
E	−2·316	(−2·319)	0·012	5·401	(5·406)	0·010	1·717	(1·719)	0·010
F	−2·345	(−2·350)	0·010	4·187	(4·192)	0·009	1·071	(1·073)	0·012
G	−1·156	(−1·158)	0·009	3·527	(3·529)	0·009	0·709	(0·710)	0·011
H	−1·310	(−1·318)	0·010	2·209	(2·209)	0·009	0·069	(0·070)	0·012
I	−0·069	(−0·069)	0·010	1·446	(1·445)	0·007	−0·304	(−0·304)	0·010
J	−0·123	(−0·124)	0·011	0·122	(0·119)	0·007	−0·944	(−0·945)	0·009
K	−1·370	(−1·373)	0·011	−0·621	(−0·628)	0·011	−1·069	(−1·072)	0·013
L	−1·298	(−1·299)	0·012	−1·827	(−1·834)	0·009	−1·702	(−1·705)	0·013
M	−0·093	(−0·093)	0·015	−2·464	(−2·470)	0·009	−2·143	(−2·145)	0·011
N	1·069	(1·070)	0·012	−1·760	(−1·764)	0·009	−1·983	(−1·985)	0·012
P	1·089	(1·090)	0·010	−0·457	(−0·460)	0·008	−1·306	(−1·308)	0·010
Q	2·303	(2·309)	0·009	0·187	(0·182)	0·009	−1·135	(−1·138)	0·011
R	2·345	(2·355)	0·010	1·439	(1·436)	0·009	−0·526	(−0·528)	0·012
S	1·167	(1·174)	0·009	2·044	(2·043)	0·008	−0·073	(−0·074)	0·010
T	1·407	(1·409)	0·013	6·076	(6·084)	0·010	1·877	(1·881)	0·013

(The figures in brackets are the coordinates after making corrections for rotational-oscillations).

thinness of the crystals made it difficult to record the weak reflections, especially those with an l index greater than 3.

The first cycle, with the estimated individual isotropic temperature factors gave $R = 0{\cdot}188$ and after three cycles of refinement and after re-scaling the observed structure factors to the calculated ones, the value of R fell to 0·11. The temperature parameters for the cycles after the first were used in the form

$$\exp -(b_{11}h^2 + b_{22}k^2 + b_{33}l^2 + b_{13}hl + b_{23}kl + b_{12}hk)$$

but the output gives the values of U_{ij} which are the components of the symmetric tensor describing the

Table 2. *Hydrogen atom coordinates* (Å)

Atom	x	y	z
H1 (T)	1·509	7·038	2·274
H2 (T)	2·125	6·118	1·102
H3 (T)	2·065	5·607	2·654
H4 (D)	−1·115	6·869	2·681
H5 (E)	−3·255	5·824	1·875
H6 (F)	−3·141	3·729	0·652
H7 (K)	−2·232	−0·386	−0·741
H8 (L)	−2·171	−2·452	−2·171
H9 (M)	−0·188	−3·089	−3·007
H10(N)	2·027	−2·215	−2·189
H11(Q)	3·211	−0·210	−1·185
H12(R)	3·251	1·817	−0·297

(The letters in brackets indicate the carbon atoms to which the hydrogen atoms are attached.)

Table 3. *Final asymmetric temperature parameters* (U_{ij}) ($Å^2 \times 10^{-2}$), *with respect to the unit cell axes*

Atom	U_{11}	U_{22}	U_{33}	$2U_{12}$	$2U_{23}$	$2U_{13}$
O1	5·01	6·12	12·25	−1·63	−2·69	−1·40
O2	5·05	5·87	17·50	0·28	−6·46	−3·00
A	3·89	5·35	6·95	0·50	2·77	2·72
B	4·85	4·32	6·68	0·12	4·70	0·13
C	6·36	5·66	5·66	1·11	2·65	−0·79
D	8·37	5·40	5·26	−0·23	−2·26	1·99
E	8·30	7·23	3·97	2·37	2·28	3·64
F	4·84	7·46	7·86	−0·15	0·34	5·28
G	5·00	5·99	5·75	0·06	5·64	−0·52
H	5·15	5·34	9·29	−1·21	5·49	0·26
I	4·87	3·90	5·60	0·78	1·51	−2·09
J	5·78	4·60	4·20	0·63	0·34	1·11
K	6·29	6·93	7·18	0·05	0·14	−4·04
L	7·47	6·09	7·62	−1·64	−3·49	−2·86
M	9·64	6·09	6·42	0·74	−0·48	−7·49
N	8·35	5·67	7·10	1·91	−0·04	−2·13
P	5·50	5·13	5·14	1·38	0·70	−1·66
Q	4·91	5·80	7·12	2·42	0·25	−0·72
R	5·63	5·33	7·20	0·57	4·15	1·37
S	3·91	5·64	5·61	0·01	5·25	4·09
T	8·51	6·03	8·06	−0·59	−3·46	0·14

Table 4. *Observed and calculated structure factors*

h k 0

h k	Fo	Fc	Ac	Bc
0 2	35.8	35.2	35.2	0
0 4	29.7	30.7	30.7	0
0 6	52.5	53.1	-53.1	0
0,10	9.3	11.8	-11.8	0
0,12	102.7	100.0	100.0	0
0,14	12.5	11.7	11.7	0
0,16	5.7	4.5	4.5	0
0,18	3.7	2.9	-2.9	0
0,20	6.9	7.9	-7.9	0
0,22	2.1	0.6	-0.6	0
0,24	6.4	6.9	-6.9	0
1 2	75.0	77.0	-1.5	77.0
1 4	52.8	51.6	-12.2	-50.1
1 6	7.5	5.9	5.9	0.4
1 8	45.6	43.3	-38.8	19.3
1,10	47.9	45.5	-0.4	-45.5
1,12	29.0	26.9	-1.6	-26.9
1,14	8.5	7.8	3.8	6.7
1,16	6.2	5.1	0.4	-5.1
1,18	5.5	4.9	4.9	0.2
1,20	8.6	9.7	-9.5	2.2
1,22	5.1	6.6	-0.7	6.5
1,24	11.0	12.9	-2.3	12.7
1,26	1.8	2.0	-1.8	-0.9
1,28	2.3	2.4	1.9	1.5
2 0	115.8	120.6	120.6	0.1
2 2	23.8	25.3	16.1	19.4
2 4	13.2	12.0	5.9	10.5
2 6	44.0	44.7	-25.4	36.7
2 8	23.9	26.9	25.4	8.8
2,10	12.2	12.3	7.2	-9.9
2,12	11.2	11.9	11.3	3.7
2,14	12.8	10.7	-2.9	10.3
2,16	5.9	6.4	1.8	6.2
2,18	10.1	10.4	-2.4	10.1
2,20	4.3	4.8	3.8	2.9
2,22	3.2	3.2	2.5	-1.9
2,24	5.9	6.0	-6.0	-0.6
2,26	2.3	3.6	-1.0	-3.5
2,28	1.7	3.1	2.1	-2.2
3 2	69.0	65.0	-4.2	-65.0
3 4	7.8	8.0	-4.5	-6.7
3 6	10.7	11.7	0.6	11.7
3,10	25.9	25.0	-0.3	25.0
3,12	2.7	2.4	2.1	-1.2
3,14	13.5	12.9	3.7	-12.4
3,16	2.2	3.1	-2.7	-1.5
3,18	2.6	2.3	0.7	2.2
3,20	7.8	9.0	7.6	-4.8
3,22	5.0	5.6	4.7	3.1
3,24	1.8	2.7	0.5	2.6
4 0	67.4	68.3	-68.3	-1.9
4 2	17.9	13.6	-3.9	-13.0
4 4	4.4	4.3	4.3	-0.7
4 6	41.3	37.2	16.7	-33.2
4 8	12.3	12.0	1.0	-11.9
4,10	18.0	16.9	13.1	10.7
4,12	19.0	18.9	-18.8	-1.2
4,14	7.8	8.2	-5.8	5.9
4,16	13.7	14.1	14.1	-0.1
4,18	21.6	24.3	19.2	-14.9
4,20	11.5	13.6	1.5	-13.5
4,22	6.9	6.5	3.6	5.3
4,24	3.1	3.7	-2.6	0.2
4,26	2.7	3.4	-0.9	3.3
4,28	0.2	2.2	2.2	0.3
5 2	33.0	27.9	-0.5	27.9
5 4	14.0	13.8	8.7	10.7
5 6	94.8	87.4	0.4	87.4
5 8	44.9	41.0	40.3	7.7
5,10	9.7	11.3	0.9	-11.3
5,12	12.0	12.8	3.5	-12.3
5,14	15.7	14.8	-0.8	14.8
5,16	6.3	6.4	5.6	-3.3
5,18	35.6	40.2	-2.8	40.1
5,20	16.5	17.3	16.5	5.0
5,22	5.6	4.4	3.1	-3.1
5,24	3.9	5.0	2.6	-4.3
5,26	2.1	2.1	0.6	2.1
5,28	2.0	2.6	-1.0	-2.4
6 0	30.7	30.5	30.5	-0.5
6 2	12.4	13.2	-6.9	11.2
6 4	16.1	15.4	15.0	-3.5
6 6	26.0	24.1	24.0	2.3
6 8	31.2	29.0	-27.4	-9.6
6,10	9.8	9.4	8.3	4.4
6,12	9.6	9.2	9.2	-0.5
6,14	4.4	4.8	-1.2	-4.7
6,16	24.7	27.1	26.2	-7.0
6,18	32.8	33.9	33.8	-3.2
6,20	8.8	9.6	-7.3	-6.2
6,22	4.0	4.7	4.6	-1.0
6,28	2.1	2.9	2.7	-1.2
7, 2	10.4	11.3	3.0	10.9
7, 4	19.5	18.3	17.4	5.7
7, 6	61.3	54.6	6.6	54.2
7, 8	11.8	11.1	-10.2	4.4
7,10	6.1	6.6	-4.5	-4.8
7,12	6.4	6.7	-3.4	-5.8
7,14	7.8	7.7	-2.3	7.3
7,16	12.9	13.8	13.4	-3.2
7,18	28.4	30.9	1.7	30.9
7,20	9.2	9.9	-9.1	3.9
7,22	6.0	6.6	-6.6	0
7,26	2.8	3.2	-3.0	1.0
8, 0	15.5	15.7	-15.7	0.8
8 2	5.6	5.8	-3.9	4.3
8 4	10.5	11.5	11.3	2.5
8 6	13.0	12.3	2.2	12.1
8 8	8.8	9.0	9.0	-1.0
8,10	7.4	7.8	6.7	-4.0
8,12	9.9	11.7	-11.5	1.7
8,14	8.1	8.7	-6.2	-6.1
8,16	9.7	10.0	9.5	-3.1
8,18	10.6	12.5	11.1	5.8
8,20	4.5	4.5	3.5	2.8
8,22	4.6	5.5	4.8	-2.8
8,24	4.3	4.1	-3.7	1.6
8,26	2.4	2.4	-0.7	-2.3
9 2	15.4	16.2	-0.6	-16.1
9 4	3.5	2.9	1.3	-2.6
9 6	5.8	6.0	-0.8	-5.9
9 8	2.3	2.0	1.9	-0.6
9,10	7.8	8.3	-1.7	8.1
9,14	5.8	5.9	-1.4	-5.7
9,16	2.2	2.4	2.3	0.6
9,18	3.4	3.6	-2.1	-2.9
9,20	3.5	3.6	-2.7	-2.4
9,22	2.8	3.7	-1.6	3.3
9,24	2.5	3.3	0.1	3.3
10, 0	31.5	33.3	33.0	-4.8
10,2	19.5	22.3	4.0	-21.9
10, 4	4.7	4.9	-4.9	0.5
10, 6	15.0	15.3	-6.1	-14.0
10, 8	4.8	4.7	4.3	-1.8
10,12	4.3	5.9	5.9	-0.1
10,14	4.5	5.4	0.9	-5.4
10,16	4.8	3.9	-3.9	-0.3
10,18	4.8	6.2	-2.6	-5.7
10,20	2.5	2.1	1.5	-1.5
10,22	3.3	3.1	0.9	3.0
10,24	3.5	4.1	-4.1	0.3
11, 2	24.9	27.1	-5.5	26.6
11, 4	5.5	6.5	2.6	-6.0
11, 6	2.2	2.3	-2.0	-1.2
11, 8	3.3	3.1	0.5	3.1
11,10	12.9	14.5	-0.8	-14.5
11,12	5.6	7.9	-1.3	-7.7
11,14	6.0	7.1	-3.4	6.2
11,16	1.8	2.6	-2.6	0.2
11,20	4.2	4.4	4.3	0.7
11,22	4.3	4.2	3.1	2.8
12, 0	64.6	63.7	63.6	-4.8
12, 2	18.8	21.5	7.5	-20.2
12, 4	3.1	2.5	-2.1	-1.3
12,6	2.9	3.8	-1.6	3.5
12, 8	7.0	6.5	-5.8	3.0
12,10	9.1	10.6	-1.3	-10.5
12,12	17.0	21.8	21.8	-0.1
12,14	4.3	6.3	5.5	-3.2
12,20	2.2	2.9	-2.7	1.1
13,2	10.9	13.3	-4.9	12.4
13, 4	4.3	4.2	2.1	-3.6
13, 6	2.8	3.4	0.3	3.4
13, 8	9.0	9.6	-9.5	1.3
13,10	5.2	7.0	-1.6	-6.8
13,12	4.1	4.7	0.4	-4.7
13,16	2.2	2.1	-1.2	-1.8
13,18	2.1	2.3	0.3	2.3
13,20	2.0	1.6	-1.5	-0.6
14, 0	3.4	4.4	4.4	0.1
14, 2	1.9	1.9	1.7	-0.9
14,4	2.9	3.5	3.3	1.2
14, 6	3.5	4.7	-1.1	4.6
14,8	2.2	3.3	2.6	2.0
14,10	2.2	3.1	1.8	-2.5
14,14	2.2	1.9	0.3	1.9
14,16	1.8	2.5	2.1	1.3
14,18	2.0	1.3	0.5	1.2
15, 2	8.1	7.9	-1.5	-7.8
15, 8	4.5	5.4	5.3	-0.5
15,10	3.1	3.3	1.1	3.1
15,14	3.0	3.1	-0.0	-3.1
16, 0	7.3	6.9	-6.8	1.3
16, 4	2.3	2.6	2.6	-0.3
16,6	4.8	4.7	0.0	-4.7
16, 8	4.1	3.5	-2.0	-2.8
16,10	4.6	3.8	-0.1	3.8
17, 2	1.8	1.2	0.5	1.1
17, 4	3.8	2.9	2.8	0.4
17, 6	7.9	7.0	1.2	6.9
17, 8	8.1	6.9	6.7	1.5
17,10	2.6	3.0	1.6	-2.6
18, 0	3.7	3.8	3.8	0.5
18, 4	2.5	3.0	3.0	0

Table 4 (*cont.*)

h k 1

h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc
0 1	26.0	30.7	30.7	0	2,13	3.3	2.7	0.8	2.6	5 9	7.8	7.9	6.4	4.6	8 2	8.6	9.8	7.7	-5.9
0 2	58.0	59.8	-59.8	0	2,14	7.0	6.0	-5.7	-1.9	5,10	7.8	8.4	-7.1	4.6	8 3	12.2	13.5	12.9	-4.1
0 3	25.4	24.8	-24.8	0	2,15	8.2	6.3	2.1	-5.9	5,11	7.4	7.8	1.6	-7.6	8 5	9.4	8.9	6.2	6.3
0 4	86.9	80.4	80.4	0	2,16	4.7	3.7	-3.7	-0.8	5,13	6.2	5.5	-4.0	-3.8	8 7	6.1	5.3	2.7	4.5
0 5	25.0	24.6	-24.6	0	2,17	5.9	5.9	0.1	5.9	5,14	22.8	23.4	-2.1	-23.3	8 8	9.8	9.7	9.7	-0.5
0 6	8.5	9.4	-9.4	0	2,18	4.3	4.9	-1.7	4.6	5,15	20.4	22.6	-7.1	-21.5	8 9	5.9	4.5	-4.2	1.7
0 7	11.3	8.6	-8.6	0	2,19	4.3	3.8	3.7	-0.8	5,16	14.1	14.4	-13.1	-6.0	8,10	7.2	5.7	2.7	5.0
0 8	52.7	49.5	-49.5	0	2,20	5.4	5.0	4.9	-0.5	5,17	9.0	9.4	-9.4	-0.5	8,13	7.8	7.4	-3.0	-6.7
0 9	37.8	34.7	-34.7	0	2,21	7.1	7.6	7.3	2.3	5,18	5.7	6.4	-5.0	-4.1	8,14	7.1	6.9	3.3	-6.1
0,12	8.8	11.1	-11.1	0	3 0	10.2	10.8	-10.8	0.1	5,19	9.5	8.9	3.5	8.1	8,15	8.5	9.0	6.7	-6.0
0,13	16.3	16.7	16.7	0	3 1	3.7	4.1	0	-4.1	5,20	15.3	15.6	-2.6	-15.4	8,19	5.5	4.7	0.9	4.6
0,14	24.8	22.9	-22.9	0	3 2	17.9	19.1	-3.0	18.8	5,21	9.7	9.3	1.6	9.2	8,20	5.7	5.4	5.2	1.6
0,15	5.8	5.3	5.3	0	3 3	13.2	13.6	-4.6	-12.8	5,22	5.4	5.4	-5.4	-0.1	9 0	4.2	4.0	-4.0	-0.4
0,16	10.4	9.3	9.3	0	3 4	6.6	8.2	-5.3	6.3	5,23	5.6	6.0	4.3	-4.2	9 1	5.4	5.5	5.2	-1.8
0,17	3.8	3.6	-3.6	0	3 5	3.1	3.3	-2.3	2.4	6 1	13.8	14.2	13.0	-5.6	9 5	5.4	5.2	3.1	-4.2
0,19	2.8	3.4	3.4	0	3 6	23.6	23.8	7.0	-22.8	6 2	13.1	12.2	11.4	-4.4	9 6	9.6	10.3	-2.8	-9.9
0,20	6.2	7.0	7.0	0	3 7	9.7	9.7	-5.5	-8.0	6 3	31.7	30.6	30.6	1.4	9 9	5.7	4.6	3.5	3.1
0,21	6.0	6.3	6.3	0	3 9	13.1	12.6	-3.4	12.2	6 4	49.2	47.3	47.2	-3.3	9,11	8.0	6.5	-1.9	6.2
0,22	5.6	5.8	5.8	0	3,10	5.8	6.0	0.2	6.0	6 5	8.1	6.6	-5.0	-4.2	9,12	3.8	3.2	-2.7	-1.6
0,24	3.2	2.4	2.4	0	3,11	15.4	16.0	3.6	15.6	6 6	9.3	8.5	-1.4	-8.4	9,21	5.0	4.2	1.4	3.9
0,26	5.7	5.7	5.7	0	3,12	5.5	6.7	2.5	-6.2	6 7	11.6	13.8	-11.9	6.9	10, 2	7.8	8.7	-8.2	-2.7
0,27	5.8	5.9	-5.9	0	3,15	8.0	8.6	-4.3	-7.4	6 8	12.9	11.5	10.7	-4.1	10, 3	6.0	6.5	-4.5	4.7
1 0	16.6	17.5	17.3	2.6	3,17	9.1	9.1	-7.8	-4.6	6 9	43.0	40.6	-40.5	2.5	10, 6	5.8	5.4	-1.2	-5.3
1 1	19.6	22.3	-17.2	14.1	3,18	5.7	5.8	3.1	-4.9	6,10	20.3	20.3	20.0	-3.6	10, 8	5.6	5.1	-4.6	2.3
1 2	92.5	105.9	9.1	-105.5	3,21	4.4	5.3	0.8	5.3	6,11	12.3	12.2	10.6	5.9	10,21	5.6	4.7	4.5	-1.4
1 3	138.7	155.6	17.9	154.6	4 1	8.7	9.5	-9.5	0.3	6,12	5.3	3.6	-1.7	-3.2	11, 0	16.7	19.4	-18.3	6.2
1 4	63.6	68.0	22.3	-64.2	4 2	18.5	21.3	20.3	6.6	6,13	7.0	7.9	0.9	-7.8	11, 1	16.1	15.2	11.6	9.9
1 5	11.2	11.3	-1.0	-11.2	4 3	27.7	27.8	26.8	7.5	6,14	5.2	5.4	3.6	-4.1	11, 2	20.2	21.9	-7.7	-20.5
1 6	19.5	11.4	5.7	9.9	4 4	8.5	7.5	7.1	-2.5	6,15	22.7	24.4	24.3	-1.7	11,3	27.3	28.9	-1.9	28.8
1 7	16.9	13.1	-8.5	10.0	4 5	22.2	21.6	11.5	-18.3	6,16	23.8	23.1	23.1	0.6	11, 4	15.1	17.0	-9.9	-13.8
1 8	14.1	12.4	3.9	-11.8	4 6	17.0	17.7	-4.7	-17.1	6,18	4.7	5.6	-4.4	-3.4	11, 5	10.4	10.0	10.0	-0.0
1 9	50.8	52.5	-13.2	-50.8	4 8	19.5	18.8	18.8	-0.8	6,19	5.8	5.9	-0.5	5.9	11, 6	10.6	9.8	-6.9	6.9
1,10	29.3	25.4	7.7	-24.2	4 9	17.5	18.6	-18.4	2.1	6,20	7.2	8.5	8.5	-0.4	11, 9	10.0	11.6	0.2	-11.6
1,11	8.9	8.5	5.3	-6.6	4,10	14.1	14.2	6.5	-12.7	6,21	20.4	20.3	-20.3	-0.9	11,10	6.7	7.3	-2.5	-6.9
1,12	2.9	2.8	-1.6	2.3	4,11	10.3	10.2	0.3	10.2	6,22	10.5	10.4	10.4	0.2	11,11	7.7	7.5	-3.8	-6.5
1,13	3.8	2.2	-0.3	-2.2	4,12	4.8	6.1	4.3	-4.3	7, 0	8.4	8.8	8.2	3.1	11,14	4.7	4.9	-1.9	-4.5
1,14	14.9	16.8	3.3	-16.5	4,13	4.3	3.0	-3.0	0.0	7 1	10.9	11.7	-7.1	-9.4	11,15	8.9	8.9	-3.4	8.3
1,15	24.3	23.9	4.1	23.6	4,14	8.0	8.3	5.1	6.5	7 2	19.0	19.8	10.1	-17.0	11,16	5.7	5.2	-3.5	-3.8
1,16	13.9	15.1	11.8	-9.3	4,15	16.8	16.8	15.0	7.6	7 3	14.1	14.5	12.3	-7.6	12, 1	8.9	8.7	8.7	1.1
1,18	6.4	6.5	4.8	4.4	4,16	8.0	7.8	7.5	-1.9	7 4	10.5	10.6	10.4	2.1	12, 2	10.0	10.7	-10.2	-3.2
1,19	5.0	3.3	-3.0	1.4	4,17	9.7	9.4	3.0	-8.9	7 8	10.8	10.0	-0.4	-10.0	12, 4	16.6	18.4	17.5	5.6
1,22	4.8	5.3	1.6	5.0	4,18	11.3	12.6	-5.4	-11.4	7 9	4.3	3.9	-2.1	3.3	12,5	10.3	9.4	-9.4	0.8
1,24	5.6	5.7	-4.5	-3.5	4,21	9.0	9.3	-9.3	-0.3	7,10	6.0	5.5	0.6	5.4	12, 7	5.6	5.3	-3.8	3.7
2 1	13.9	15.8	-15.1	-4.8	4,22	8.9	8.0	3.2	-7.3	7,11	2.9	2.7	1.9	-1.9	12, 8	10.1	9.9	-9.8	-0.5
2 2	17.3	16.4	-9.7	13.2	4,23	5.5	5.0	1.5	4.8	7,12	7.6	6.9	-6.7	-1.4	12, 9	8.4	7.9	-7.9	-0.5
2 3	22.2	25.4	-0.0	-25.4	4,24	6.3	5.7	-1.7	-5.5	7,13	7.5	7.0	-3.7	-5.9	12,13	6.1	5.4	5.0	2.1
2 4	13.3	13.4	0.6	13.4	5 0	15.6	16.4	-8.5	14.0	7,14	17.6	20.0	1.7	-19.6	12,14	6.8	5.8	-5.8	-0.2
2 5	18.2	15.4	11.0	10.9	5 1	4.0	5.4	4.4	-3.1	7,15	16.5	17.5	4.3	-16.9	13, 0	6.8	6.8	-6.8	0.2
2 6	21.8	25.1	-9.2	23.3	5 2	20.8	20.5	-2.3	-20.3	7,16	5.5	5.5	3.2	-4.4	13, 2	14.0	13.5	-1.0	-13.5
2 7	8.7	10.4	9.9	-3.3	5 3	19.5	20.9	-12.9	-16.5	7,17	5.7	5.6	5.2	-1.9	13, 3	16.4	16.1	4.0	15.6
2 8	5.6	6.7	-4.8	-4.8	5 4	19.3	18.8	-18.0	5.5	7,18	6.2	6.1	-4.1	-4.6	13, 4	9.1	8.7	1.7	-8.5
2 9	4.3	3.9	-1.0	-3.8	5 5	14.2	14.1	-11.7	7.9	7,19	5.8	5.4	-1.2	5.2	13, 5	7.5	7.5	7.2	1.9
2,10	7.6	7.0	-5.2	4.7	5 6	4.2	5.5	1.6	5.2	7,20	13.7	12.9	-1.7	-12.8	13, 7	6.6	5.1	-5.0	0.8
2,11	14.9	16.0	-10.4	-12.2	5 7	11.0	9.2	8.1	4.3	7,21	9.3	8.7	0.1	8.7	13, 9	7.3	7.0	-3.9	-5.8
2,12	3.2	1.3	-1.1	0.6	5 8	14.9	15.1	-1.2	-15.1	8 1	5.3	5.5	-4.2	-3.5	13,10	5.2	4.6	1.7	-4.3

h k 2

h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc
0 0	16.2	14.8	14.8	0	2 3	3.6	6.4	-5.5	3.3	4,11	4.5	4.5	-4.2	1.7	6,24	8.2	7.4	-7.2	1.5
0 1	7.3	7.1	7.1	0	2 4	12.0	13.2	-10.1	-8.5	4,12	14.6	14.6	-12.7	7.1	6,25	4.8	4.9	-4.9	-0.6
0 2	16.1	17.4	-17.4	0	2 5	16.4	15.7	13.8	7.4	4,13	13.3	12.6	-5.6	11.3	7 1	20.3	20.5	-4.2	-20.1
0 3					2 6	33.0	32.2	-31.4	7.3	4,14	11.1	10.8	-2.3	10.6	7 2	7.0	6.9	3.4	-6.1
0 4	14.9	14.4	-14.4	0	2 7	7.5	8.5	8.3	1.9	4,15	5.3	5.5	0.1	5.5	7 8	6.1	5.0	0.8	-5.0
0 5	65.0	58.6	58.6	0	2 8	9.7	9.7	-1.9	-9.5	4,16	6.1	5.8	-4.4	-3.8	7,10	5.4	6.4	-6.4	0.3
0 6	105.1	101.6	-101.6	0	2 9	4.7	4.7	1.4	4.5	4,22	8.3	6.3	-6.3	-0.3	7,11	12.0	9.8	-6.7	7.1
0 7	4.4	4.1	-4.1	0	2,10	4.5	2.7	-2.7	0.0	4,24	5.7	4.9	-4.3	2.4	7,12	23.4	24.9	-8.6	-23.3
0 9	14.1	10.9	-10.9	0	2,11	5.6	5.7	-1.0	5.6	5 1	25.4	26.1	4.9	-25.6	7,13	7.0	6.1	-2.8	-5.4
0,12	8.8	8.2	8.2	0	2,12	5.6	6.1	3.4	-5.1	5 2	11.6	11.7	-6.4	-9.8	7,23	7.1	7.1	-2.6	6.6
0,17	12.7	11.6	11.6	0	2,13	5.8	5.5	3.3	-4.5	5 3	11.9	12.2	-11.9	2.4	7,24	9.3	9.3	0.1	-9.3
0,18	12.4	12.4	-12.4	0	2,14	4.1	4.5	-3.7	-2.5	5 4	3.7	5.1	-4.6	2.1	8 0	13.0	15.3	-3.8	-14.8
0,19	10.6	10.2	10.2	0	2,19	5.6	5.8	5.6	1.6	5 5	5.2	6.3	4.0	4.9	8 1	6.1	6.1	4.9	-3.7
0,21	7.2	6.6	-6.6	0	3 1	5.6	6.8	1.4	-6.6	5 7	7.6	7.0	-0.1	7.0	8 3	6.1	6.8	-5.6	3.9
1 1	4.6	5.8	-4.2	4.0	3 2	4.4	3.7	-1.4	-3.4	5 8	9.3	9.0	0.5	-9.0	8,10	7.0	6.7	-5.3	4.1
1 2	14.5	14.3	4.7	-13.5	3 3	6.0	6.4	-3.4	5.5	5,10	8.0	8.2	-7.9	-2.4	8,13	8.1	7.8	-3.7	-6.9
1 3	7.4	9.4	7.1	-6.1	3 4	8.3	8.0	3.5	-7.2	5,11	11.5	11.4	4.7	10.4	9 5	7.2	7.3	-4.0	-6.1
1 4	25.8	24.6	9.0	22.9	3 5	12.8	11.7	-10.3	-5.6	5,12	29.0	31.1	2.7	-31.0	9 7	6.2	6.3	4.1	-4.8
1 5	17.9	17.9	-16.5	6.8	3 6	8.0	7.3	6.5	3.3	5,13	13.6	12.1	7.1	-9.8	10, 3	6.8	7.2	-4.0	-6.0
1 6	8.5	8.2	7.6	3.2	3 7	13.7	11.7	-8.8	-7.6	5,14	11.3	11.5	-10.5	-4.6	10, 4	11.8	11.8	-4.5	10.9
1 7	34.6	32.9	-9.7	31.5	3 8	10.7	10.8	-0.6	10.8	5,15	6.4	6.6	-6.6	0.7	10, 5	9.9	9.5	9.3	-1.9
1 8	8.5	7.2	-1.0	-7.2	3 9	4.4	3.5	-1.9	-2.9	5,23	8.0	7.9	2.6	7.4	10, 6	14.5	12.6	-12.5	1.0
1 9	7.7	7.5	-6.9	-3.0	3,11	5.5	4.6	4.1	-1.9	5,24	11.3	11.0	1.2	-10.9	10, 8	10.1	9.5	-1.4	9.4
1,10	12.6	12.1	5.2	10.9	3,12	5.6	5.7	1.8	-5.4	5,25	5.4	5.2	3.3	4.1	11, 4	11.4	11.6	-2.5	11.3
1,11	4.3	2.3	-2.0	1.2	4, 0	21.6	20.0	-8.8	18.0	6 0	27.8	25.6	-25.1	-4.9	11, 5	6.4	6.2	5.7	2.5
1,13	5.6	5.1	-4.7	2.1	4 1	13.5	14.4	6.4	12.9	6 1	13.6	14.4	13.6	4.6	11, 7	14.6	11.5	0.9	11.5
1,14	6.3	5.9	5.7	-1.6	4 2	12.9	11.5	5.9	9.9	6,10	12.9	11.8	-11.1	4.0	11, 8	8.1	7.0	1.9	-6.7
1,15	6.6	7.8	6.7	-3.9	4 3	9.7	10.1	-4.2	-9.2	6,11	12.0	13.0	-12.8	-2.1	12, 3	7.6	5.7	1.0	-5.6
1,16	7.8	6.8	0.0	6.8	4 4	4.9	4.2	-3.1	2.9	6,12	21.9	21.3	-21.2	2.1	12, 4	11.4	10.3	-3.4	9.7
1,17	13.6	12.6	-0.8	-12.6	4 5	6.5	6.8	-6.6	-1.4	6,13	16.4	16.1	-16.1	0.8	12, 5	16.6	14.7	14.7	1.1
1,18	7.4	7.3	2.1	-7.0	4 6	10.3	10.8	10.8	-1.0	6,14	4.7	5.1	2.5	4.5	12, 6	24.7	24.0	-23.7	3.6
2 0	17.3	15.6	14.0	-7.0	4 7	4.1	3.4	3.0	1.5	6,16	7.1	7.7	-7.6	0.9	12, 8	7.9	7.0	-1.1	6.9
2 1	11.1	13.0	2.6	-12.7	4 9	9.5	8.6	7.1	4.8	6,22	10.6	9.2	-9.0	2.2	13,7	10.8	9.5	-3.9	8.6
2 2	4.8	5.3	-5.2	-0.6	4,10	6.4	6.3	-5.7	-2.7	6,23	6.8	6.4	6.4	-0.1					

Table 4 (*cont.*)

$\underline{h}\,\underline{k}\,3$

h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc
0 2	10.6	9.7	9.7	0	1 1	6.4	6.3	1.1	-6.2	2,12	5.5	6.7	1.6	-6.5	5 4	5.3	6.9	-1.6	6.7
0 3	11.4	13.1	13.1	0	1 3	11.7	14.3	3.7	13.8	3 1	7.1	6.9	-3.6	6.0	5 6	7.5	7.4	6.5	-3.6
0 4	4.2	4.5	4.5	0	1 4	13.9	14.6	4.5	13.9	3 2	4.5	4.4	3.9	2.1	5 8	9.9	9.6	-1.5	9.5
0 5	6.5	6.6	6.6	0	1 6	6.1	6.2	-6.0	1.6	3 4	4.3	4.2	3.8	-1.8	5 9	13.2	14.7	3.4	14.3
0 7	3.8	5.2	-5.2	0	1 7	10.5	9.1	6.3	-6.6	3,10	5.6	6.3	3.5	5.2	5,10	8.6	8.9	6.2	6.4
0 8	6.4	6.0	6.0	0	1 8	22.4	25.2	2.0	25.1	3,11	6.7	7.4	6.6	-3.3	5,11	6.6	7.9	7.6	2.0
0 9	13.0	14.0	14.0	0	1 9	20.6	22.8	-1.6	-22.7	4 1	6.1	6.4	-3.5	5.3	6 1	7.6	5.3	-5.0	1.5
0,10	16.9	15.8	-15.8	0	1,10	9.5	6.2	-3.0	5.4	4 2	6.9	7.4	7.1	2.0	6 2	10.1	10.7	10.6	1.3
0,11	4.9	6.9	6.9	0	1,11	5.6	5.7	-0.4	5.7	4 3	6.4	5.6	-4.5	-3.3	6 3	5.5	5.6	-5.5	-1.4
0,12	4.9	4.6	4.6	0	1,12	5.5	4.6	-3.5	-3.0	4 9	9.0	9.0	-7.9	-4.2	6 9	7.0	6.7	-6.7	0.5
0,13	3.0	3.9	-3.9	0	2 2	3.9	4.4	4.4	0.1	4,10	9.9	8.5	-5.7	-6.4	6,10	13.9	14.1	-14.1	1.3
0,14	4.1	4.8	-4.8	0	2 3	4.8	5.5	2.4	5.0	4,11	7.0	7.0	-6.9	1.4	6,14	8.4	9.3	-9.2	0.8
0,15	5.6	4.9	-4.9	0	2 4	4.0	4.7	1.9	4.4	4,12	6.8	7.1	1.1	7.0	6,15	9.4	9.9	9.8	-1.1
0,16	3.4	3.1	-3.1	0	2 5	5.6	5.5	3.7	4.1	4,14	4.7	4.9	-4.9	0	7 3	7.8	8.0	5.5	-5.8
0,19	7.5	7.6	-7.6	0	2 7	7.0	7.6	-5.0	5.7	4,15	5.6	4.7	4.5	-1.4	7 4	6.1	6.4	-2.3	6.0
0,20	2.4	3.1	3.1	0	2 8	10.7	11.7	0.8	-11.6	5 1	8.0	8.0	-5.8	5.6	7 8	9.0	9.6	0.5	9.6
0,21	2.7	3.1	3.1	0	2 9	8.1	9.2	1.1	9.2	5 2	5.4	5.6	5.6	0.3	7 9	10.3	12.3	-3.6	11.7
0,22	3.0	4.0	-4.0	0	2,10	5.2	4.1	-3.7	-1.8	5 3	8.5	8.2	-1.4	-8.0					

$\underline{h}\,\underline{k}\,4$

h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc	h k	Fo	Fc	Ac	Bc
0 0	8.3	9.8	9.8	0	0,12	8.1	9.8	9.8	0	1,10	5.4	5.6	-4.1	-3.8	2 9	3.9	2.6	-1.1	-2.4
0 1	6.8	8.0	8.0	0	0,14	5.3	5.5	-5.5	0	1,11	5.6	5.5	5.1	2.1	2,10	5.5	4.8	3.4	3.5
0,10	6.4	6.1	6.1	0	0,15	3.1	4.2	4.2	0	1,12	8.9	10.2	-3.5	9.6	2,11	4.6	6.9	-6.7	1.5
0,11	9.8	11.6	-11.6	0	1 1	7.0	7.0	2.4	-6.6										

anisotropic vibrations of the atoms. The mean square amplitude of vibration in the direction of the unit vector l with components l_i is

$$u^{-2} = \sum_{i=1}^{3} \sum_{j=1}^{3} U_{ij} l_i l_j \, .$$

The relation between the U's and the b's is given by $b_{11} = 2\pi^2 a^{*2} U_{11}$, $b_{12} = 2(2\pi^2 a^* b^* U_{12})$ *etc.* Hydrogen-atom coordinates were calculated on the assumption that the C–H bond is 1·05 Å and that those hydrogen atoms bonded to carbons of aromatic rings are in a symmetrical position in the plane of the ring and that the hydrogens of the methyl groups are arranged tetrahedrally. The $(F_o - F_c)$ maps were used to fix the approximate coordinates of the methyl-group hydrogen atoms. Four further cycles of refinement were carried out and the final value of the R-factor was 0·082. For the final cycle only 0·4 of the full shift was accepted as there was a tendency in the later cycles for the parameter shifts to oscillate in sign.

The final carbon-atom and oxygen-atom coordinates with their standard deviations are given in Table 1. The hydrogen-atom coordinates which were used in the refinement are given in Table 2. Table 3 gives the final values for the asymmetric temperature parameters. The agreement between observed and calculated structure factors is shown in Table 4.

The mean plane of the carbon and oxygen atoms was computed on the University of Leeds 'Pegasus' computer with the program written by Dr A. M. B. Douglas. Dr D. W. J. Cruickshank computed the atomic and molecular thermal vibrations with his molecular analysis program.

The mean plane of all the carbon and oxygen atoms is represented by the equation

$$-0{\cdot}0496x + 0{\cdot}4312y - 0{\cdot}9009z + 0{\cdot}9165 = 0 \, . \quad (1)$$

In addition, two other planes were calculated. The mean plane of the carbon atoms B, C, D, E, F, G, which form a benzene ring, with the methyl group, T, has the following equation,

$$-0{\cdot}0359x + 0{\cdot}4476y - 0{\cdot}8935z + 0{\cdot}9795 = 0 \, . \quad (2)$$

The equation of the mean plane of the naphthalene nucleus (carbon atoms, $I, J, K, L, M, N, P, Q, R, S$) is

$$-0{\cdot}1031x + 0{\cdot}4355y - 0{\cdot}8943z + 0{\cdot}8662 = 0 \, . \quad (3)$$

Table 5. *Deviations* (Δ) *from mean planes* (Å)

Atom	Plane I	Plane II	Plane III
O1	−0·001	+0·038	−0·058
O2	+0·173	+0·105	+0·358
A	+0·012	+0·027	+0·019
B	−0·029	−0·015*	+0·042
C	−0·042	−0·002*	+0·036
D	−0·042	−0·007*	+0·105
E	−0·020	−0·013*	+0·189
F	+0·040	+0·022*	+0·241
G	+0·023	+0·007*	+0·155
H	+0·039	−0·006	+0·169
I	−0·016	−0·058	+0·042*
J	−0·008	−0·077	+0·044*
K	−0·153	−0·253	−0·040*
L	−0·106	−0·230	−0·006*
M	−0·044	−0·165	−0·013*
N	+0·058	−0·034	+0·031*
P	+0·009	−0·056	−0·009*
Q	+0·073	+0·036	−0·007*
R	+0·061	+0·050	−0·011*
S	−0·027	−0·041	−0·031*
T	−0·058	+0·013*	−0·044

(Plane I is the mean plane of all the atoms, Planes II and III are the mean planes of those atoms marked with asterisks.)

Table 6. *Observed and calculated* U_{ij} (*with respect to molecular inertial axes* ($Å^2\times 10^{-2}$))

	U_{11}		U_{22}		U_{33}		U_{23}		U_{13}		U_{12}	
Atom	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.	Obs.	Calc.
O1	6·37	7·09	4·90	5·02	12·11	12·68	−0·51	−0·62	−1·55	−1·73	1·02	0·81
O2	5·62	6·94	5·12	4·54	17·67	10·71	−1·83	−0·53	−2·64	−1·43	0·61	0·15
A	6·78	6·23	3·97	4·78	5·44	6·62	−1·30	−0·24	−1·57	−1·04	0·24	0·33
B	6·60	5·87	4·85	5·43	4·40	4·21	0·22	−0·27	−2·38	−0·73	−0·09	0·10
C	6·65	5·89	6·45	7·02	4·57	4·96	−0·45	−0·73	−0·88	−0·77	−0·31	0·26
D	4·46	5·97	8·31	8·15	6·26	4·34	0·99	−0·31	0·81	−0·64	−0·09	−0·65
E	7·25	6·65	8·44	7·29	3·81	6·93	1·30	0·47	0·57	−0·93	−1·82	−1·49
F	7·50	6·74	4·78	5·62	7·87	7·87	2·28	0·38	−0·15	−1·09	−1·27	−1·00
G	8·16	6·06	5·01	4·99	3·57	4·61	−0·06	0·11	−1·67	−0·77	−0·06	−0·32
H	8·30	6·17	5·04	4·50	6·43	5·61	0·54	−0·01	−3·20	−0·91	0·42	−0·04
I	4·83	5·86	4·95	4·59	4·59	3·81	−1·02	−0·01	−1·20	−0·71	0·17	0·02
J	4·59	5·87	5·81	5·40	4·18	4·25	0·44	−0·25	0·06	−0·74	−0·45	0·12
K	7·15	6·35	6·35	5·93	6·90	8·37	−1·85	−0·53	−0·23	−1·27	0·81	0·83
L	5·21	6·36	7·36	7·72	8·61	9·58	−1·02	−1·29	0·48	−1·36	1·50	1·26
M	6·16	5·90	9·77	9·24	6·22	5·90	−3·37	−1·25	−0·16	−0·84	1·56	0·44
N	5·88	5·97	8·52	8·27	6·72	4·35	−1·25	−0·33	−0·65	−0·64	−0·19	−0·66
P	5·37	6·01	5·63	6·16	4·77	4·28	−0·99	0·01	−0·30	−0·70	−0·23	−0·50
Q	6·01	6·74	5·13	5·63	6·69	7·80	−0·89	0·61	−0·70	−1·11	−0·98	−1·00
R	7·23	6·86	5·66	4·71	5·27	9·14	0·65	0·44	−2·01	−1·31	−0·57	−0·46
S	7·55	6·11	3·87	4·50	3·74	5·23	1·96	0·02	−1·53	−0·87	−1·08	−0·04
T	5·11	6·41	8·45	7·96	9·05	10·21	0·14	−1·78	0·31	−1·36	0·46	1·38

For each plane the axes are the unit-cell axes where x is parallel to a, y is parallel to b and z is parallel to c.

The deviations of all the carbon atoms and the oxygen atoms from each of the three planes were calculated and these are given in Table 5.

The molecular-analysis program of Cruickshank (1956*a*) converts the thermal parameters used in the least-squares refinement into atomic amplitudes of vibration U_{ij} referred to molecular axes. The molecule is then considered as a rigid body and two symmetric tensors are calculated, ($\mathbf{T}$) the translational vibrations of the mass centre and ($\boldsymbol{\omega}$) the angular oscillations about axes through the centre. The program then calculates from these tensors the individual U_{ij} and the agreement between these *calculated* U_{ij} and the observed U_{ij} is a measure of the validity of the assumption that the molecule does in fact vibrate as a rigid body.

The values of $\mathbf{T}_{ij}$ and $\boldsymbol{\omega}_{ij}$ are as follows, ($\mathbf{T}_{ij}$ in $Å^2\times 10^{-2}$ and $\boldsymbol{\omega}_{ij}$ in deg.^{2})

$$\mathbf{T}=\begin{pmatrix} 5·86 & 0 & -0·70 \\ & 4·49 & 0·02 \\ & & 3·74 \end{pmatrix},\quad \sigma(\mathbf{T})=\begin{pmatrix} 0·14 & 0·14 & 0·16 \\ & 0·18 & 0·18 \\ & & 0·27 \end{pmatrix}$$

$$\boldsymbol{\omega}=\begin{pmatrix} 37·92 & 4·95 & 4·27 \\ & 1·59 & 1·34 \\ & & 6·20 \end{pmatrix},\quad \sigma(\boldsymbol{\omega})=\begin{pmatrix} 2·10 & 0·63 & 1·07 \\ & 0·64 & 0·55 \\ & & 0·53 \end{pmatrix}.$$

The observed and calculated values of U_{ij} are given in Table 6 and it will be seen that the agreement is fairly good but not nearly so good as in the case of anthracene (Cruickshank, 1956*b*) or of chrysene (Burns & Iball, 1960). This is not surprising as one would expect in a molecule of this kind that, for example, the oxygen atoms and the methyl group would vibrate to some extent independently of the main part of the molecule.

It should be pointed out, in connection with the thermal parameters, that there is no evidence in this structure to support the suggestion put forward by Murty (1957*a*, *b*) that in anthraquinone the carbon and oxygen atoms of the C=O groups have different scattering curves from the normal carbon and oxygen atoms.

Discussion of the structure

The bond lengths are given in Table 7 and, with the bond angles, in Fig. 6. There is good agreement between the two C=O bonds (mean value 1·218 Å).

Table 7. *Bond-lengths* (Å)

Bond	Length	S.D.
A–O_1	1·224 (1·228)	0·011
H–O_2	1·212 (1·217)	0·012
A–*B*	1·510 (1·513)	0·013
B–*C*	1·420 (1·422)	0·012
C–*D*	1·372 (1·373)	0·015
D–*E*	1·384 (1·386)	0·016
E–*F*	1·376 (1·376)	0·014
F–*G*	1·407 (1·411)	0·014
G–*H*	1·473 (1·476)	0·013
H–*I*	1·504 (1·511)	0·014
I–*J*	1·472 (1·475)	0·011
J–*K*	1·456 (1·461)	0·015
K–*L*	1·364 (1·365)	0·015
L–*M*	1·432 (1·432)	0·018
M–*N*	1·368 (1·370)	0·018
N–*P*	1·469 (1·470)	0·013
P–*Q*	1·385 (1·388)	0·013
Q–*R*	1·393 (1·395)	0·013
R–*S*	1·399 (1·403)	0·013
S–*A*	1·477 (1·484)	0·012
C–*T*	1·510 (1·475)	0·016
B–*G*	1·405 (1·408)	0·013
I–*S*	1·392 (1·399)	0·013
J–*P*	1·391 (1·392)	0·014

(The values in brackets are the bond-lengths after corrections for rotational-oscillations of the atoms.)

Table 8. *Intermolecular contacts* (*less than* 3·6 Å)

Atoms	Distance	Atoms	Distance
Q–O2 (I)	3·343	*G*–*H* (III)	3·557
O1–*E* (II)	3·444	*I*–*J* (III)	3·556
C–*B* (III)	3·554	*K*–*L* (III)	3·520
E–*F* (III)	3·511	*P*–*N* (III)	3·514
F–O2 (III)	3·574	*R*–*Q* (III)	3·559

(I) refers to the molecule at $\frac{1}{2}+x$, $-y$, $-z$;
(II) $\frac{1}{2}+x$, $\frac{1}{2}-y$, $-\frac{1}{2}+z$;
(III) x, y, $1+z$.

The mean value for the four single bonds in ring II is 1·491 Å but while pairs of bonds which are on opposite sides of the ring [*GH* and *AS*; *HI* and *AB*] have almost identical lengths, the adjacent bonds [*GH* and *HI*; *AS* and *AB*] have different values. The difference (0·016 Å) from the mean is, however, not significant. There is a significant lengthening of the aromatic bond *IJ* (1·472 Å) due to steric interference between O2 and the hydrogen attached to atom K. The three aromatic rings have the following mean bond lengths, I (1·394), III (1·405), IV (1·413).

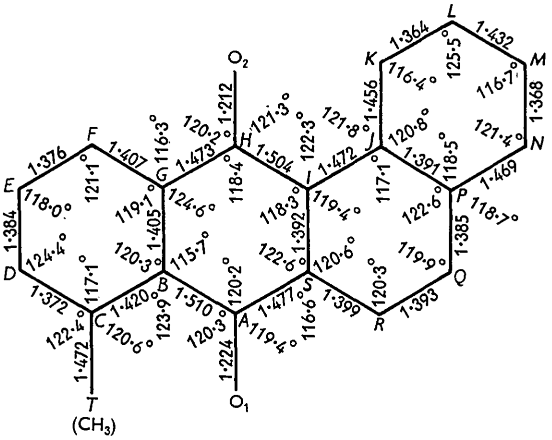

Fig. 6. Bond lengths (Å) and bond angles.

The short length of the *c* axis means that molecules at each end of this axis are already fairly close and the inclination of the molecular plane brings certain atoms in neighbouring molecules even closer. There are some other short intermolecular distances between the molecules related by the screw axis. Those intermolecular contacts (not including hydrogen atoms) which are less than 3·6 Å are listed in Table 8.

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Dynamical Diffraction Theory of Waves in Distorted Crystals. I. General Formulation and Treatment for Perfect Crystals

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A dynamical diffraction theory for distorted crystals is formulated for the Laue case based on a 'lamellar crystal' method originally given by C. G. Darwin (*Phil. Mag.* 1914, **27**, 325 and 675). In each lamellar crystal a Born approximation is assumed. Relations among two-dimensional Fourier transforms of wave functions on the successive boundaries between lamellae are obtained in terms of generalized matrix multiplication. In the two-beam case of a perfect crystal, results coincide with those of the ordinary Laue–Bethe theory.

The theory is applicable to asymmetrical cases with a large Bragg angle; namely to the general cases of X-rays and neutrons as well as electrons.

1. Introduction

Standard dynamical theories for diffraction phenomena in crystals are based on the Laue–Bethe formulation (Laue, 1931; Bethe, 1928). We can, however, treat the problem in an entirely different way which we may call 'lamellar crystal' approach. It seems