Acta Cryst. (1963). **16**, 513

The Structure of Methyl 1:2-benzanthraquinones. III. The Crystal and Molecular Structure of 2′-Methyl-1:2-benzanthraquinone

By R. P. Ferrier and J. Iball

Chemistry Department, Queen's College, Dundee, Scotland

(*Received* 19 *July* 1962)

The structure of 2′-methyl-1:2-benzanthraquinone has been determined by the use of a weighted reciprocal-lattice section and a Patterson projection. The unit cell is monoclinic with $a = 20{\cdot}67$, $b = 4{\cdot}06$, $c = 7{\cdot}77$ Å, $\beta = 90{\cdot}8°$; 2 mol. per unit cell; space group $= P2_1$. The carbon and oxygen atom coordinates were refined by means of 3-dimensional least-squares with anisotropic temperature parameters, and corrections for rotational oscillations were calculated. The standard deviations of bond lengths are approximately 0·01 Å.

Introduction

In Part II (Ferrier & Iball, 1963) the structure of 5-methyl-1:2-benzanthraquinone was described. The parent hydrocarbon of that compound has cancer-producing properties whereas the parent hydrocarbon of 2′-methyl-1:2-benzanthraquinone (Fig. 1) is inactive. These quinones had been prepared originally for an investigation of their oxidation-reduction potentials (Iball, 1940) and it was hoped that the O–*R* potentials of the quinones might give some correlation with the biological activity of the parent hydrocarbons. In addition it was considered of interest to see whether substitution of a methyl group in different positions made any appreciable change in the structure of the benzanthraquinone nucleus.

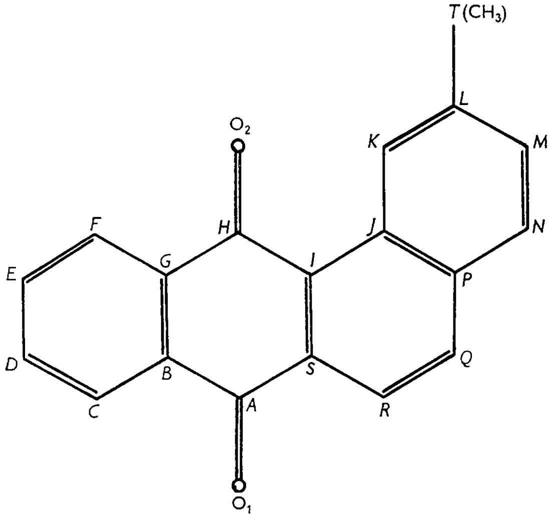

Fig. 1. 2′-Methyl-1:2-benzanthraquinone ($C_{19}O_2H_{12}$).

Crystal data

This compound crystallizes, as yellow needles, in two forms (Ferrier & Iball, 1960). Both forms are monoclinic but one form (I) has only 2 molecules per unit cell whereas the other form (II) has 8. The two forms can be obtained from a solution in methyl ethyl ketone. If the solution is maintained at near 0 °C, (I) is more likely to appear but at 20 °C form (II) is obtained. It is form (I) which is described in the present paper and the unit-cell dimensions are:

$$a = 20{\cdot}67,\ b = 4{\cdot}06,\ c = 7{\cdot}77\ \text{Å},\ \beta = 90{\cdot}8°.$$

(The *b* axis is parallel to the axis of the needles). The only systematic absences were $0k0$ when k is odd; the space group could therefore be $P2_1$ or $P2_1/m$. However $P2_1/m$ would require one molecule at 000 and a reflected molecule at $0\frac{1}{2}0$ and this is impossible with a *b* axis of only 4·06 Å. In addition, there are only 2 molecules per unit cell; d(obs.) (23·5 °C) = 1·380 g.cm^{-3}; d(calc.) = 1·386 g.cm^{-3}. The space group must therefore be $P2_1$.

Intensity measurement

Weissenberg photographs were taken about the *b* and *c* axes and intensities were measured by (i) visual estimation, (ii) a recording microdensitometer (see Part II) and (iii) for a limited number of strong reflections, G.–M. counter spectrometer.

Determination of the structure

Since there is a very short *b* axis it was clear that the molecules must be arranged so that there are no overlapping molecules in this direction and the plane of the molecule must be inclined at only a relatively small angle to the (010) plane. A good indication of the orientation of the benzene rings to the *a* and *c* axes was given by the weighted reciprocal lattice (Fig. 2) and this also gave a value of 30° for the angle of tilt of the molecular plane to the (010) plane. With the benzene ring orientation known, there are still five possible ways in which the molecule can be built up from such benzene rings and the position of the molecule with respect to the screw axis has to be determined. The answers to these questions were given by a two-dimensional Patterson synthesis. Fig. 3(*a*) is a Patterson $h0l$ projection, Fig. 3(*b*) is the molecular arrangement deduced from it and Fig. 3(*c*) is the vector diagram corresponding to 3(*b*). The large peak (A) in the Patterson synthesis showed quite clearly the distance apart of the two molecules related by the screw axis.

The Patterson synthesis gave x and y coordinates of $0{\cdot}317a$, $0{\cdot}105c$ for the mass centre of the molecule and with this and a tilt of 30°, atomic coordinates were calculated. Structure factors for 224 $h0l$ reflections with $\sin\theta < 0{\cdot}65$ were then computed and after scaling the F_o to the calculated values the reliability index ($R = \Sigma||F_o| - |F_c||/\Sigma|F_o|$) was 0·30. A Fourier synthesis with 130 of the largest observed structure factors gave a map with all the atoms resolved (Fig. 4). The coordinates of the peaks were determined by the method of Burns & Iball (1955) and a recalculation of the value of R gave 0·23. A second synthesis (Fig. 5) gave coordinates which reduced R to 0·18.

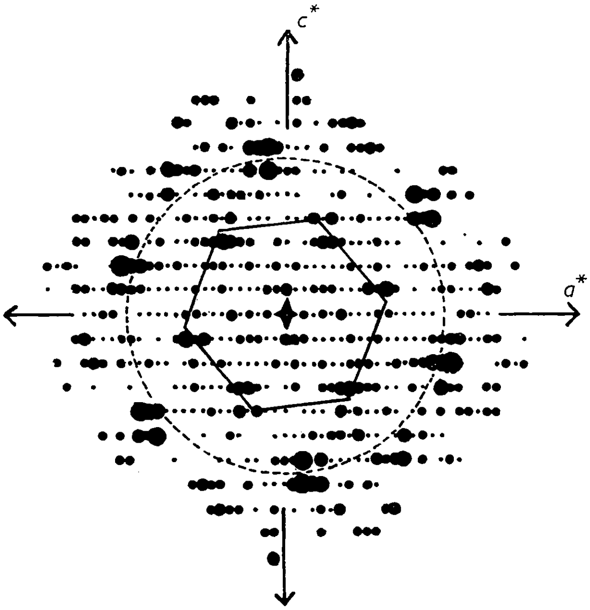

Fig. 2. ($h0l$) Weighted reciprocal lattice.

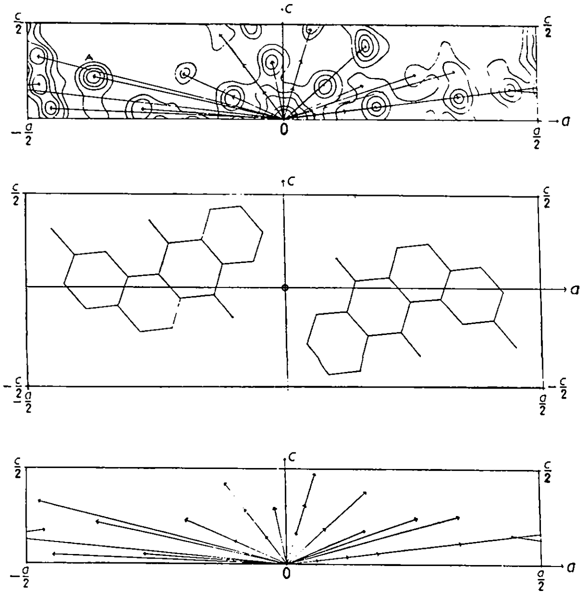

Fig. 3. (*a*) ($h0l$) Patterson projection, (*b*) molecular arrangement deduced from (*a*), (*c*) calculated vector diagram of (*b*) —each arrow head represents a peak.

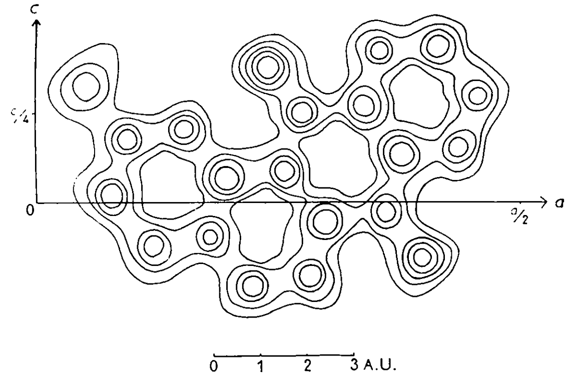

Fig. 4. First F_o Fourier synthesis ($h0l$). Contours at arbitrary intervals.

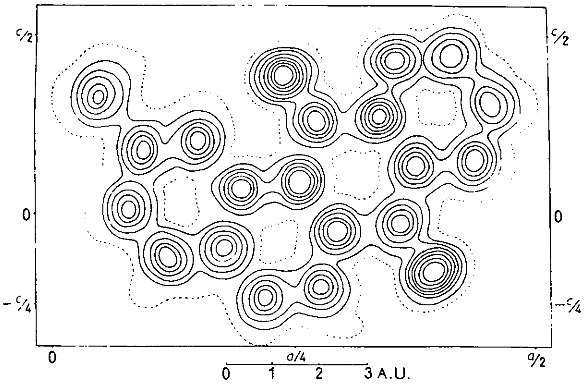

Fig. 5. Second F_o Fourier synthesis ($h0l$). Contours at intervals of 1·0 e.Å^{-2}: the 1-electron contour is dotted.

The y coordinates were determined by assuming a tilt of 30° to the plane (010) and calculation of the $hk0$ structure factors gave a value of 0·15 for R.

Refinement by least-squares

A two-dimensional refinement of the $h0l$ zone was carried out on the 'Pegasus' computer at the Northampton College of Advanced Technology, London with a program written by Dr J. H. Milledge, to whom we are very grateful for help in the early stages. Hydrogen-atom coordinates were calculated on the

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

Atom	x	$\sigma(x)$	y	$\sigma(y)$	z	$\sigma(z)$
O1	8·133 (8·138)	0·005	−0·234 (−0·234)	0·008	−1·238 (−1·245)	0·005
O2	4·948 (4·945)	0·004	0·043 (0·042)	0·008	2·994 (3·001)	0·004
A	7·423 (7·428)	0·006	−0·188 (−0·189)	0·010	−0·267 (−0·273)	0·007
B	7·814 (7·818)	0·006	−0·747 (−0·748)	0·009	1·022 (1·020)	0·007
C	9·057 (9·063)	0·006	−1·409 (−1·411)	0·009	1·171 (1·169)	0·008
D	9·412 (9·417)	0·007	−1·954 (−1·957)	0·011	2·384 (2·386)	0·008
E	8·577 (8·579)	0·007	−1·854 (−1·857)	0·011	3·459 (3·464)	0·008
F	7·363 (7·364)	0·006	−1·214 (−1·216)	0·009	3·336 (3·343)	0·007
G	6·973 (6·974)	0·006	−0·672 (−0·673)	0·009	2·123 (2·126)	0·007
H	5·648 (5·647)	0·006	0·045 (0·044)	0·009	2·007 (2·011)	0·006
I	5·232 (5·232)	0·006	0·646 (0·646)	0·009	0·715 (0·714)	0·006
J	4·018 (4·016)	0·006	1·369 (1·370)	0·009	0·533 (0·533)	0·007
K	3·101 (3·096)	0·006	1·650 (1·652)	0·010	1·586 (1·591)	0·007
L	1·950 (1·945)	0·007	2·358 (2·360)	0·009	1·360 (1·364)	0·007
M	1·643 (1·638)	0·007	2·811 (2·814)	0·010	0·083 (0·086)	0·009
N	2·475 (2·472)	0·007	2·583 (2·585)	0·010	−0·967 (−0·971)	0·008
P	3·688 (3·687)	0·007	1·832 (1·833)	0·010	−0·761 (0·766)	0·007
Q	4·557 (4·558)	0·007	1·665 (1·666)	0·010	−1·839 (−1·847)	0·007
R	5·737 (5·740)	0·007	1·005 (1·006)	0·009	−1·642 (−1·650)	0·007
S	6·087 (6·089)	0·006	0·517 (0·517)	0·009	−0·383 (−0·388)	0·006
T	1·023 (1·016)	0·008	2·687 (2·689)	0·013	2·489 (2·496)	0·009

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

Table 2. *Final asymmetric temperature parameters*

$$[2^{-(h^2b_{11}+k^2b_{22}+l^2b_{33}+klb_{23}+hlb_{13}+hkb_{12})}]$$

Atom	b_{11}	b_{22}	b_{33}	b_{12}	b_{13}	b_{23}
O1	0·0043	0·1976	0·0264	−0·0018	0·0087	0·0039
O2	0·0042	0·1887	0·0214	0·0126	0·0056	0·0123
A	0·0031	0·1220	0·0244	−0·0164	0·0034	−0·0089
B	0·0028	0·0928	0·0268	−0·0128	0·0012	−0·0079
C	0·0033	0·0906	0·0346	−0·0008	0·0044	0·0011
D	0·0038	0·1176	0·0380	−0·0157	−0·0004	0·0016
E	0·0063	0·1202	0·0334	0·0090	−0·0046	0·0044
F	0·0033	0·0785	0·0245	−0·0110	0·0004	−0·0051
G	0·0031	0·0859	0·0232	−0·0145	0·0010	−0·0082
H	0·0029	0·1045	0·0227	−0·0084	0·0010	−0·0026
I	0·0030	0·0890	0·0215	−0·0254	0·0000	−0·0086
J	0·0033	0·0973	0·0239	−0·0196	−0·0002	−0·0072
K	0·0030	0·1143	0·0277	−0·0064	0·0038	−0·0013
L	0·0044	0·0866	0·0317	0·0090	0·0023	−0·0008
M	0·0032	0·1197	0·0476	−0·0043	−0·0024	−0·0032
N	0·0047	0·1105	0·0307	−0·0048	−0·0017	−0·0054
P	0·0040	0·0808	0·0247	−0·0008	−0·0019	−0·0055
Q	0·0048	0·1175	0·0256	−0·0078	0·0013	−0·0069
R	0·0047	0·0916	0·0207	−0·0116	0·0040	−0·0066
S	0·0032	0·0833	0·0212	−0·0138	0·0017	−0·0070
T	0·0043	0·1868	0·0391	−0·0174	0·0054	0·0131

(The hydrogen atoms were given the temperature parameters of the carbon atoms to which they were attached).

basis of a C–H bond length of 1·05 Å and estimates of the individual carbon-atom isotropic temperature parameters were made from the peak heights of the Fourier map. With these additional parameters the value of R was reduced from 0·18 (see above) to 0·15 and after one cycle of least-squares refinement it fell to 0·11. A (F_o-F_c) Fourier map was then computed and this made it possible to estimate more accurately the individual atomic temperature parameters. It showed in addition that the oxygen atoms were vibrating anisotropically.

Subsequent refinement was carried out with the set of three-dimensional structure factors (863 independent reflections). The first cycles were computed

Table 3. *Hydrogen atom coordinates*

Atom*	x	y	z
H1 (*T*)	0·062 Å	2·452 Å	2·152 Å
H2 (*T*)	1·157	2·923	3·054
H3 (*T*)	1·157	1·981	3·054
H4 (*C*)	9·686	−1·494	0·412
H5 (*D*)	10·298	−2·399	2·479
H6 (*E*)	8·863	−2·233	4·320
H7 (*F*)	6·740	−1·117	4·110
H8 (*K*)	3·320	1·315	2·486
H9 (*M*)	0·796	3·301	−0·054
H10(*N*)	2·241	2·931	−1·880
H11(*Q*)	4·328	2·022	−2·766
H12(*R*)	6·354	0·869	−2·424

* The letter in brackets indicates the carbon atom to which the hydrogen atom is attached.

on the 'Deuce' machine at Glasgow University with the assistance of Dr David Watson and the later ones on the 'Mercury' computers at Manchester University and at Oxford University. The three-dimensional refinements were all done with the programs written by Dr J. S. Rollett.

The program refines the atomic coordinates and anisotropic temperature parameters given by the expression,

$$2^{-(h^2b_{11}+k^2b_{22}+l^2b_{33}+hkb_{12}+hlb_{13}+klb_{23})} .$$

The final value of R was 0·071.

The carbon and oxygen atom coordinates are given in Table 1 and the anisotropic temperature parameters in Table 2. The calculated hydrogen-atom coordinates are listed in Table 3. The observed and calculated structure factors are given in Table 4.

Analysis of thermal motion

As for 5-methyl-1:2-benzanthracene the thermal parameters were used with the molecular-analysis program of Dr D. W. J. Cruickshank on the 'Pegasus' machine at Leeds to compute the two symmetric tensors (T) and (ω) describing the translational vibrations and angular oscillations of the molecular mass centre about axes through the centre. The values obtained were as follows, ($\mathbf{T}_{ij}$ in Å$^2\times10^{-2}$; ω_{ij} in deg.^{2} with respect to molecular axes)

$$\mathbf{T}=\begin{pmatrix}5{\cdot}82 & 0{\cdot}55 & -0{\cdot}63\\ & 3{\cdot}59 & -0{\cdot}20\\ & & 4{\cdot}34\end{pmatrix},\quad \sigma(T)=\begin{pmatrix}0{\cdot}02 & 0{\cdot}02 & 0{\cdot}02\\ & 0{\cdot}03 & 0{\cdot}03\\ & & 0{\cdot}04\end{pmatrix}$$

$$\omega=\begin{pmatrix}20{\cdot}35 & 0{\cdot}46 & -3{\cdot}07\\ & 5{\cdot}76 & -0{\cdot}40\\ & & 3{\cdot}25\end{pmatrix},\quad \sigma(\omega)=\begin{pmatrix}0{\cdot}26 & 0{\cdot}07 & 0{\cdot}12\\ & 0{\cdot}08 & 0{\cdot}07\\ & & 0{\cdot}07\end{pmatrix}$$

From these tensors the program then calculates the individual atomic amplitudes of vibration U_{ij}. These calculated amplitudes (with respect to molecular axes) are given in Table 5 together with the observed values. The degree of agreement is rather better than in the case of 5-methyl-1:2-benzanthraquinone which

Table 4. *The observed and calculated structure amplitudes*

h0l

h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|
1 0	18.2	18.9	22 1	7.1	7.6	15 2	5.5	5.5	12 3	1.9	1.9	14 4	17.4	16.8	- 9 5	1.7	2.1	7 7	6.2	6.3
2 0	38.6	43.9	23 1	1.9	2.0	16 2	2.8	1.9	15 3	1.6	1.1	15 4	12.0	11.5	-10 5	1.7	1.5	8 7	3.0	3.1
3 0	11.0	11.3	- 1 1	39.7	42.4	17 2	2.5	2.4	17 3	1.9	1.9	16 4	18.8	18.9	-12 5	2.7	2.9	9 7	6.6	6.7
4 0	20.3	19.5	- 2 1	21.7	22.6	18 2	4.1	1.8	21 3	1.9	1.5	- 2 4	2.9	3.7	-13 5	12.9	13.2	10 7	3.1	3.3
5 0	10.9	9.9	- 3 1	13.8	14.4	20 2	2.5	2.9	24 3	2.2	2.4	- 3 4	2.0	4.7	-14 5	2.8	3.3	- 1 7	11.8	10.6
6 0	34.8	34.4	- 4 1	15.5	16.2	21 2	4.7	5.4	- 1 3	21.1	20.2	- 4 4	3.1	4.3	-15 5	3.9	3.7	- 2 7	18.4	17.7
7 0	8.3	8.5	- 5 1	12.3	13.2	22 2	2.6	2.7	- 2 3	7.1	6.7	- 6 4	15.7	13.9	-16 5	1.5	0.8	- 3 7	13.3	12.6
8 0	5.5	4.8	- 6 1	5.5	5.5	25 2	1.2	1.8	- 3 3	4.9	4.2	- 7 4	2.6	2.2	-17 5	1.5	1.5	- 4 7	14.5	13.6
9 0	7.1	6.9	- 7 1	13.8	13.2	- 1 2	30.5	31.1	- 4 3	14.6	14.0	- 8 4	8.5	7.1	0 6	4.4	5.4	- 6 7	1.9	2.4
10 0	11.0	10.8	- 8 1	10.8	10.1	- 2 2	14.9	15.5	- 5 3	11.4	10.9	- 9 4	2.5	2.0	1 6	4.6	4.3	- 7 7	3.4	3.0
11 0	15.5	13.8	- 9 1	4.3	4.6	- 3 2	4.5	4.0	- 6 3	32.7	31.6	-10 4	2.6	4.0	3 6	1.5	2.0	- 9 7	2.3	2.4
12 0	2.2	2.0	-10 1	13.9	14.7	- 4 2	20.1	20.1	- 7 3	36.6	35.4	-11 4	8.7	8.2	4 6	2.2	2.1	-10 7	2.9	1.8
13 0	3.3	2.1	-11 1	6.5	5.9	- 5 2	7.2	7.5	- 8 3	21.7	21.5	-12 4	3.4	1.9	6 6	2.9	3.1	-17 7	1.7	1.7
14 0	2.1	2.1	-12 1	2.1	1.0	- 6 2	6.6	6.1	- 9 3	7.3	7.5	-14 4	2.6	4.8	7 6	2.3	2.0	2 8	3.3	2.9
15 0	1.5	1.4	-13 1	2.5	2.8	- 7 2	12.1	11.9	-10 3	8.1	7.7	-15 4	3.1	3.8	11 6	1.6	1.0	3 8	1.5	1.1
16 0	1.9	1.4	-14 1	1.6	1.6	- 8 2	12.3	11.6	-11 3	2.5	2.7	-17 4	4.8	5.3	12 6	1.8	1.3	4 8	1.6	1.2
19 0	3.8	3.8	-15 1	1.5	1.9	- 9 2	8.6	8.2	-12 3	3.3	3.6	-19 4	4.2	4.5	13 6	1.5	1.1	5 8	1.2	0.9
20 0	2.6	3.0	-16 1	8.5	8.2	-10 2	2.5	2.6	-14 3	4.4	4.7	-20 4	2.6	2.7	17 6	4.3	4.0	6 8	2.2	1.3
21 0	2.1	2.2	-17 1	4.7	5.3	-11 2	1.9	1.6	-15 3	2.2	2.3	-21 4	2.0	1.9	18 6	2.5	0.8	7 8	2.8	2.8
0 1	49.8	53.6	-18 1	7.1	7.6	-12 2	11.8	11.8	-17 3	14.5	14.3	-22 4	2.7	2.5	- 1 6	4.5	4.6	8 8	5.4	5.6
1 1	13.2	12.5	-19 1	7.1	8.0	-13 2	3.6	3.5	-18 3	5.4	5.9	-23 4	2.1	1.9	- 2 6	31.9	32.1	9 8	2.0	2.0
2 1	12.0	12.1	-20 1	2.0	2.5	-14 2	8.5	8.9	-19 3	4.3	3.3	0 5	2.2	2.0	- 4 6	16.0	15.4	- 1 8	2.8	2.3
3 1	32.3	35.2	-21 1	2.1	2.3	-15 2	5.4	6.0	-20 3	2.1	1.7	1 5	1.7	1.7	- 5 6	2.4	2.6	- 3 8	2.3	2.2
4 1	25.0	25.9	-22 1	2.3	2.0	-16 2	17.8	17.8	-22 3	2.5	2.5	5 5	4.2	4.1	- 6 6	2.6	2.9	- 4 8	2.6	2.3
5 1	26.3	26.8	-23 1	2.3	2.5	-17 2	15.2	15.6	-23 3	2.1	1.9	6 5	9.4	7.8	- 7 6	1.6	1.6	- 5 8	2.4	2.4
6 1	4.6	4.8	0 2	17.0	17.4	-18 2	26.2	26.7	0 4	6.9	7.2	9 5	1.8	1.6	- 8 6	2.8	2.5	- 6 8	4.6	4.1
7 1	11.3	11.7	1 2	25.1	25.3	-19 2	1.9	1.2	1 4	3.3	2.9	14 5	18.5	17.5	- 9 6	2.4	2.9	-11 8	3.0	3.0
8 1	8.4	8.7	2 2	6.8	8.1	-24 2	2.1	2.4	2 4	4.2	4.1	15 5	4.3	4.7	-10 6	6.1	6.9	-12 8	4.3	4.1
9 1	30.5	30.7	3 2	10.9	10.9	0 3	7.9	8.2	3 4	19.3	18.0	16 5	9.5	8.8	-11 6	3.3	3.4	1 9	3.8	2.7
10 1	16.3	17.0	4 2	4.9	5.3	2 3	5.1	5.1	4 4	7.4	6.6	18 5	3.3	3.3	-12 6	8.1	8.6	2 9	2.5	1.7
11 1	43.1	42.9	5 2	9.4	10.0	3 3	21.9	20.6	5 4	29.3	27.4	20 5	2.7	1.0	-13 6	12.1	12.2	8 9	1.4	1.2
12 1	13.7	12.9	6 2	8.1	9.3	4 3	40.1	38.4	6 4	4.0	4.2	- 1 5	1.6	1.8	-15 6	3.9	3.8	- 3 9	2.8	2.5
14 1	5.0	3.3	7 2	14.5	14.6	5 3	33.6	32.8	7 4	2.0	1.3	- 2 5	5.5	6.0	-18 6	2.3	2.6	- 5 9	1.2	1.3
16 1	2.2	2.4	8 2	1.2	0.9	6 3	11.3	11.5	8 4	8.4	7.4	- 3 5	6.5	6.2	-19 6	1.5	1.7	- 6 9	1.1	0.7
17 1	5.0	5.6	9 2	5.0	5.0	7 3	3.1	2.2	9 4	1.6	1.4	- 4 5	4.4	4.6	0 7	1.8	2.0	- 8 9	2.2	1.9
18 1	3.8	3.6	10 2	9.5	9.3	8 3	2.5	2.8	10 4	2.6	2.6	- 5 5	9.6	9.3	1 7	2.8	1.5	- 9 9	3.0	2.6
19 1	1.7	0.9	11 2	6.6	6.0	9 3	2.3	1.7	11 4	1.8	1.3	- 6 5	6.1	5.6	2 7	1.8	1.0	-10 9	2.6	2.2
20 1	1.9	1.9	13 2	2.2	2.0	10 3	14.5	14.5	12 4	3.2	2.8	- 7 5	4.7	4.6	4 7	3.0	3.3	1 10	3.3	4.2
21 1	1.5	1.2	14 2	3.3	3.5	11 3	3.6	3.7	13 4	3.1	3.6	- 8 5	11.8	11.6						

Table 4 (*cont.*)

*h*1*l*

h ℓ	|Fo|	|Fo|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|
1 0	2.1	1.9	-1 1	11.0	9.0	9 2	7.2	6.8	1 3	25.4	24.9	2 4	8.9	8.6	0 5	3.0	2.7	-3 6	4.3	4.2
2 0	51.3	53.9	-2 1	4.1	3.8	11 2	5.2	4.6	2 3	23.5	23.0	4 4	5.4	4.7	1 5	3.3	3.4	-4 6	13.9	14.5
3 0	79.2	84.7	-3 1	15.3	14.5	12 2	7.8	6.6	3 3	21.4	21.1	5 4	6.4	5.4	2 5	2.8	2.3	-5 6	15.5	16.8
4 0	40.4	40.2	-4 1	17.3	14.8	13 2	13.5	13.4	4 3	6.0	5.5	6 4	15.1	14.5	3 5	5.4	4.5	-6 6	15.4	15.4
5 0	12.7	11.9	-5 1	5.1	4.7	14 2	9.2	8.7	5 3	9.6	8.9	7 4	17.2	16.3	4 5	3.5	3.8	-7 6	6.8	6.5
6 0	20.0	19.2	-6 1	7.4	7.8	15 2	3.7	3.2	6 3	9.3	9.4	8 4	10.1	9.6	5 5	3.3	3.3	-9 6	6.1	6.7
7 0	26.7	26.9	-7 1	7.7	7.2	17 2	3.8	3.4	7 3	8.7	8.4	9 4	8.0	7.9	6 5	5.9	5.8	-10 6	5.6	6.3
8 0	23.9	22.4	-8 1	6.5	5.8	23 2	2.5	2.0	8 3	3.6	3.3	11 4	12.7	13.2	7 5	4.8	4.0	-11 6	7.9	8.4
9 0	19.0	17.6	-9 1	4.3	3.9	24 2	3.4	3.2	9 3	3.4	3.1	12 4	17.4	17.7	8 5	3.1	2.9	-12 6	4.7	4.5
10 0	8.8	8.5	-10 1	3.9	3.4	25 2	2.0	2.6	10 3	2.9	2.8	13 4	11.4	11.7	11 5	4.7	4.3	-15 6	3.2	3.2
11 0	6.3	5.6	-11 1	7.0	6.4	-1 2	12.6	12.2	11 3	8.0	7.4	14 4	6.7	6.9	12 5	3.6	3.3	-16 6	3.5	2.8
12 0	5.8	5.3	-12 1	5.3	4.7	-3 2	4.9	4.6	12 3	8.5	8.6	16 4	3.1	3.2	14 5	3.4	3.3	0 7	13.8	15.2
13 0	3.4	3.9	-13 1	2.8	3.1	-4 2	2.8	2.5	13 3	8.1	7.8	17 4	3.8	3.6	16 5	6.4	6.1	1 7	11.8	11.6
19 0	4.1	3.8	-14 1	4.1	3.1	-5 2	5.6	4.7	14 3	7.3	7.1	18 4	3.9	4.2	17 5	7.8	8.1	2 7	6.8	6.5
0 1	27.2	30.0	-15 1	3.3	3.1	-6 2	15.4	13.5	15 3	3.2	3.0	19 4	4.2	5.0	18 5	8.9	8.6	5 7	5.8	5.9
1 1	26.9	28.7	-16 1	7.4	7.6	-7 2	14.5	13.8	-1 3	5.0	4.6	-1 4	8.0	8.2	19 5	4.3	4.2	6 7	5.9	6.2
2 1	75.8	86.2	-17 1	3.5	3.3	-8 2	8.4	7.6	-2 3	6.2	6.1	-2 4	2.0	2.3	-1 5	3.6	2.7	16 7	3.3	2.1
3 1	51.7	55.6	-18 1	8.7	7.4	-9 2	15.0	13.9	-3 3	14.2	12.9	-3 4	8.8	8.6	-2 5	6.1	6.1	17 7	2.6	2.1
4 1	32.9	32.5	-19 1	7.9	6.7	-10 2	14.2	13.5	-4 3	24.4	22.5	-4 4	20.0	20.7	-3 5	4.4	4.4	-1 7	7.8	7.9
5 1	3.7	3.7	-20 1	11.4	11.3	-11 2	9.9	9.4	-5 3	8.0	7.0	-5 4	13.9	11.9	-4 5	7.9	7.6	-2 7	5.7	5.5
6 1	7.9	7.4	-21 1	5.4	4.8	-13 2	9.4	9.6	-6 3	8.3	8.4	-6 4	5.3	5.0	-5 5	5.8	6.1	-4 7	2.6	2.1
7 1	23.5	22.8	-22 1	3.5	2.8	-14 2	11.9	11.6	-7 3	6.9	6.3	-7 4	6.3	5.6	-6 5	7.0	8.1	-5 7	4.1	3.8
8 1	26.2	26.4	-23 1	2.6	2.3	-15 2	13.7	13.8	-8 3	12.3	11.9	-8 4	2.9	1.9	-7 5	2.6	2.3	-6 7	4.3	4.0
9 1	9.5	8.2	-24 1	2.9	0.9	-16 2	7.4	6.7	-9 3	9.6	8.6	-9 4	2.4	2.6	-8 5	6.4	6.1	-7 7	3.8	2.9
10 1	6.2	6.2	-25 1	2.7	1.9	-17 2	5.6	5.4	-10 3	15.2	14.4	-10 4	6.6	7.1	-9 5	4.9	4.6	-8 7	4.8	4.3
11 1	7.7	7.2	0 2	12.3	12.6	-18 2	3.6	3.9	-11 3	11.1	10.8	-11 4	2.5	2.5	-10 5	4.3	3.8	-10 7	4.4	3.6
12 1	7.5	7.3	1 2	9.9	10.6	-19 2	9.1	8.2	-13 3	10.3	9.3	-12 4	4.2	4.0	0 6	3.3	3.5	-11 7	3.7	2.8
13 1	12.4	11.4	2 2	21.1	21.3	-20 2	8.4	8.5	-14 3	14.3	14.4	-13 4	3.5	3.8	1 6	9.7	9.2	-12 7	2.9	2.8
14 1	12.7	11.7	3 2	6.4	5.8	-21 2	8.2	8.1	-15 3	14.3	14.0	-14 4	3.1	3.4	9 6	3.3	3.2	10 8	2.8	2.6
15 1	6.2	5.9	4 2	6.3	6.6	-22 2	3.2	2.7	-16 3	12.0	12.0	-16 4	3.0	2.8	16 6	2.8	2.5	11 8	4.0	3.6
16 1	3.5	1.0	5 2	3.4	3.7	-24 2	3.2	3.6	-17 3	4.3	4.2	-17 4	3.4	3.0	17 6	3.6	3.0	-1 8	4.5	3.1
18 1	3.3	3.4	6 2	8.3	7.9	-25 2	3.2	3.1	-20 3	3.8	2.0	-18 4	2.9	2.0	-1 6	3.3	3.6	1 9	4.5	2.8
19 1	4.5	5.2	7 2	8.0	7.4	0 3	8.7	7.7	0 4	8.0	7.3	-19 4	2.7	2.4	-2 6	8.0	7.8	-1 9	6.1	4.2
20 1	3.4	3.8	8 2	15.4	14.7															

*h*2*l*

h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|	h ℓ	|Fo|	|Fc|
0 0	12.2	11.7	7 1	14.7	13.0	-23 1	5.3	5.5	-12 2	7.9	7.2	-12 3	13.0	12.5	-18 4	3.2	2.3	1 6	6.3	6.8
1 0	7.2	8.0	8 1	4.4	3.3	-24 1	4.0	5.2	-13 2	13.5	13.2	-13 3	12.4	12.1	-19 4	4.3	3.9	3 6	5.3	6.2
2 0	11.0	11.6	9 1	9.7	8.5	0 2	11.0	10.4	-14 2	6.4	5.9	-14 3	2.9	3.0	8 5	5.1	5.3	4 6	4.0	4.3
3 0	13.8	13.9	10 1	2.9	2.6	1 2	7.7	7.3	-15 2	3.4	3.6	-15 3	3.5	3.5	9 5	6.9	6.9	-4 6	3.3	3.3
4 0	9.3	9.3	11 1	3.8	3.7	2 2	8.7	8.8	-22 2	4.9	5.0	-23 3	2.1	2.7	10 5	3.3	3.8	-6 6	4.1	3.9
5 0	27.0	25.1	12 1	4.3	4.1	3 2	7.9	6.9	-24 2	1.5	2.5	0 4	6.1	5.4	11 5	7.5	7.7	-7 6	5.8	6.2
6 0	23.6	21.3	13 1	2.9	2.9	4 2	17.2	17.1	0 3	1.8	1.6	1 4	4.8	4.1	12 5	2.9	2.8	-8 6	10.5	10.9
7 0	13.4	12.8	15 1	4.5	4.4	5 2	12.9	12.6	1 3	2.3	1.6	2 4	6.7	6.8	19 5	4.0	2.9	-9 6	6.9	6.0
8 0	3.7	3.3	16 1	3.2	3.1	6 2	10.6	9.4	2 3	8.3	8.1	3 4	2.7	3.2	20 5	4.5	4.3	1 7	4.1	3.3
9 0	5.0	5.0	17 1	5.1	4.8	7 2	3.9	2.7	4 3	7.2	7.7	8 4	6.7	6.6	-1 5	2.2	2.3	2 7	3.5	3.1
10 0	2.4	2.4	-1 1	7.2	7.7	8 2	6.1	5.5	5 3	6.0	6.6	9 4	14.8	14.4	-2 5	5.4	5.2	3 7	10.4	10.7
11 0	4.5	4.2	-2 1	4.7	4.9	9 2	2.8	3.3	7 3	3.4	3.5	10 4	7.1	7.3	-3 5	2.7	2.5	4 7	6.3	5.6
12 0	2.2	2.5	-3 1	4.3	3.7	11 2	4.7	5.2	8 3	7.0	6.5	11 4	6.8	6.2	-4 5	4.6	4.1	5 7	3.8	3.4
13 0	5.0	4.7	-4 1	8.8	8.0	13 2	2.9	2.3	9 3	15.5	15.0	12 4	3.3	3.7	-5 5	6.3	5.8	6 7	2.9	2.4
14 0	4.3	3.8	-5 1	14.2	12.9	14 2	3.9	3.1	10 3	14.9	15.4	19 4	2.3	1.9	-6 5	6.9	6.4	13 7	3.2	1.9
15 0	4.2	4.2	-6 1	5.5	5.0	15 2	7.4	7.4	11 3	9.7	9.8	20 4	2.9	2.6	-7 5	10.1	10.1	14 7	2.2	1.5
16 0	4.2	3.9	-7 1	9.7	9.3	16 2	6.3	6.0	12 3	5.1	4.7	-1 4	12.2	11.8	-8 5	13.2	13.6	15 7	2.0	1.7
17 0	4.3	3.8	-8 1	6.3	5.6	17 2	5.6	5.7	13 3	3.1	3.4	-2 4	9.7	9.6	-9 5	6.0	5.8	-5 7	3.6	2.0
21 0	3.2	2.6	-9 1	3.8	3.5	-1 2	16.3	15.0	15 3	2.4	2.5	-3 4	5.7	5.1	-10 5	5.5	4.9	-6 7	5.6	3.9
22 0	4.4	3.9	-10 1	4.4	3.6	-2 2	13.7	12.7	16 3	3.7	3.4	-4 4	3.8	3.7	-11 5	4.3	4.4	-7 7	3.3	5.7
23 0	2.4	2.5	-11 1	4.9	4.1	-3 2	6.4	5.6	-1 3	17.8	16.5	-5 4	3.7	3.0	-13 5	2.8	2.8	-8 7	4.2	3.7
24 0	1.8	2.1	-12 1	5.4	4.7	-4 2	13.5	12.3	-2 3	7.6	6.8	-10 4	5.0	4.7	-14 5	3.5	2.9	1 8	2.9	3.0
0 1	6.4	6.1	-13 1	5.0	4.4	-5 2	6.2	5.5	-3 3	6.4	5.8	-11 4	5.8	5.2	-15 5	2.9	2.3	2 8	7.8	6.7
1 1	4.3	3.9	-14 1	3.3	3.2	-6 2	3.4	2.7	-4 3	6.4	5.7	-12 4	7.3	7.2	-16 5	4.4	4.2	3 8	6.6	5.9
2 1	6.1	5.7	-15 1	3.9	3.1	-7 2	6.9	6.0	-5 3	3.3	2.6	-13 4	5.4	5.3	-17 5	3.0	2.5	4 8	5.6	4.5
3 1	13.8	15.0	-19 1	3.2	3.1	-8 2	2.2	2.1	-6 3	4.5	3.7	-14 4	5.3	5.4	-18 5	2.9	2.4	-1 9	2.1	0.8
4 1	19.4	20.0	-20 1	2.5	2.5	-9 2	2.6	2.7	-9 3	4.8	4.6	-15 4	2.7	2.1	-19 5	3.8	3.5	-2 9	3.0	1.9
5 1	40.4	40.2	-21 1	5.5	4.8	-10 2	1.9	1.4	-10 3	6.3	5.2	-16 4	3.2	2.4	-20 5	2.4	2.3	-3 9	2.5	2.5
6 1	38.8	36.3	-22 1	6.0	6.4	-11 2	12.7	11.8	-11 3	15.1	15.3	-17 4	3.7	3.1						

Table 4 (*cont.*)

*h*3*l*

h l	|Fo|	|Fc|	h l	|Fo|	|Fc|	h l	|Fo|	|Fc|	h l	|Fo|	|Fc|	h l	|Fo|	|Fc|	h l	|Fo|	|Fc|	h l	|Fo|	|Fc|
1 0	4.1	4.0	8 1	11.7	11.7	-15 1	5.3	4.8	- 4 2	8.0	6.2	10 3	3.3	3.6	6 4	3.5	3.6	13 5	3.3	3.2
2 0	7.8	7.7	9 1	14.0	13.9	0 2	3.2	2.6	- 5 2	7.0	5.6	11 3	1.9	1.3	7 4	3.9	3.6	14 5	2.7	2.7
3 0	7.3	7.4	10 1	4.5	4.7	1 2	1.7	1.7	- 6 2	2.6	1.9	12 3	1.8	1.2	9 4	3.7	3.8	- 1 5	3.5	3.7
4 0	2.2	1.7	12 1	3.1	2.8	2 2	1.5	1.8	- 7 2	2.9	2.9	17 3	3.5	3.1	10 4	3.4	3.7	- 7 5	3.1	2.4
5 0	4.2	3.7	13 1	2.0	1.9	3 2	1.6	1.6	- 8 2	3.2	3.3	19 3	3.2	3.1	18 4	2.0	1.6	- 8 5	3.6	3.7
6 0	4.3	4.1	14 1	1.9	1.9	4 2	4.1	4.4	-10 2	3.6	4.0	- 1 3	2.3	2.5	- 1 4	3.2	2.8	- 9 5	6.3	6.2
7 0	5.1	5.4	16 1	2.8	2.8	5 2	6.1	6.1	-11 2	5.5	5.2	- 2 3	4.0	3.9	- 2 4	2.0	1.2	-10 5	6.7	6.5
8 0	3.1	2.6	- 1 1	6.8	6.3	6 2	8.7	8.7	-12 2	4.0	4.2	- 4 3	5.2	4.9	- 3 4	2.7	1.9	-11 5	7.1	6.8
9 0	2.2	0.9	- 2 1	6.8	7.1	7 2	9.4	9.5	-15 2	4.4	4.0	- 5 3	3.3	2.4	- 6 4	3.4	2.9	-12 5	5.3	4.4
10 0	2.1	1.4	- 3 1	11.4	11.2	8 2	18.9	19.9	-16 2	3.0	2.9	- 6 3	2.5	1.8	- 7 4	3.9	3.7	-13 5	1.9	1.4
11 0	2.7	2.7	- 4 1	9.6	9.6	9 2	5.6	5.5	0 3	2.5	2.2	- 7 3	3.3	2.8	- 8 4	6.1	5.2	0 6	3.6	3.8
12 0	3.4	3.7	- 5 1	3.9	3.2	10 2	5.6	5.0	1 3	2.0	1.4	- 8 3	6.8	6.6	- 9 4	6.8	7.2	1 6	2.7	3.1
13 0	4.1	3.9	- 6 1	3.2	2.5	11 2	2.0	1.2	2 3	2.2	1.8	- 9 3	10.0	9.7	-10 4	12.2	11.8	11 6	3.8	2.9
14 0	2.3	1.8	- 7 1	1.7	0.6	12 2	1.9	1.9	3 3	4.1	4.1	-10 3	3.9	3.4	-11 4	2.9	2.8	13 6	2.7	2.5
0 1	1.1	1.3	- 8 1	1.8	1.7	18 2	3.1	2.9	4 3	2.9	2.6	-11 3	2.6	2.2	-12 4	5.3	4.6	6 7	2.9	2.9
1 1	2.5	2.2	- 9 1	2.4	2.6	19 2	2.8	3.0	5 3	4.3	4.0	0 4	3.3	2.9	0 5	2.6	2.3	7 7	2.8	2.4
3 1	5.3	5.4	-10 1	3.2	3.3	20 2	2.6	2.4	6 3	11.8	11.2	1 4	5.2	4.5	1 5	4.7	4.8	8 7	3.1	1.4
4 1	5.3	4.8	-12 1	3.1	2.7	- 1 2	4.1	4.2	7 3	9.8	10.3	2 4	6.1	6.0	2 5	4.3	4.6	- 4 7	3.4	3.0
5 1	2.0	0.9	-13 1	2.8	2.3	- 2 2	4.7	4.5	8 3	6.3	6.2	3 4	3.8	3.2	11 5	2.5	2.6	- 5 7	3.4	2.9
6 1	2.7	2.6	-14 1	5.5	5.8	- 3 2	9.9	8.2	9 3	3.5	3.0	5 4	2.9	2.1	12 5	4.1	4.0	- 6 7	2.4	2.4
7 1	8.1	8.5																		

Table 5. *Observed and calculated* U_{ij} (*with respect to molecular inertial axes*)

	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	7·84	7·10	4·99	5·65	10·99	9·70	0·02	0·23	1·24	0·38	−2·16	−1·76
O2	6·26	6·73	4·25	4·69	11·64	6·63	−0·22	0·12	0·71	0·46	−0·91	−0·64
A	6·84	6·26	5·20	5·28	5·28	5·98	−0·09	0·01	1·37	0·44	−1·40	−1·16
B	5·79	5·86	5·90	5·93	4·03	4·55	0·13	0·00	0·90	0·49	−0·86	−0·81
C	5·27	5·87	7·05	7·65	5·68	5·68	0·98	0·16	−0·37	0·51	−1·35	−0·94
D	6·02	5·95	8·72	8·85	6·49	5·47	−0·33	0·11	0·26	0·55	−0·73	0·00
E	4·97	6·57	8·00	7·85	7·09	6·00	−0·04	−0·03	0·58	0·53	0·95	0·86
F	5·63	6·64	5·57	6·10	3·86	5·85	−0·09	−0·06	0·15	0·50	−0·37	0·41
G	5·98	6·01	5·22	5·42	3·70	4·19	−0·13	−0·07	0·64	0·50	−0·65	−0·31
H	5·35	6·06	5·08	4·73	5·22	4·28	−0·21	−0·07	0·78	0·49	−0·59	−0·56
I	6·03	5·85	5·15	4·70	3·45	3·66	−0·79	−0·11	0·87	0·49	−0·72	−0·65
J	6·18	5·84	5·64	5·24	4·17	3·92	−0·59	−0·07	0·83	0·50	−0·54	−0·63
K	5·50	6·07	5·57	5·84	6·34	5·65	0·87	−0·01	0·65	0·43	−0·96	−1·18
L	6·44	6·06	6·70	7·43	5·71	6·81	0·93	0·09	−0·83	0·41	−0·14	−1·44
M	5·80	5·92	10·73	8·30	6·05	6·43	0·73	0·03	1·11	0·37	−0·47	−1·16
N	7·56	6·15	7·23	7·45	5·74	5·09	−0·18	−0·02	0·20	0·54	0·41	0·27
P	6·33	6·16	5·86	5·79	4·25	4·60	−0·02	−0·13	−0·07	0·51	0·65	−0·07
Q	8·16	7·03	5·80	5·27	5·94	6·96	−0·25	−0·24	0·25	0·45	−0·15	0·17
R	7·83	7·05	4·43	4·71	4·88	7·48	−0·20	−0·19	−0·52	0·40	−0·63	−0·46
S	6·01	6·19	4·72	4·69	3·81	4·92	−0·19	−0·11	0·36	0·46	−0·64	−0·74
T	6·50	6·89	8·40	8·67	11·23	11·69	−0·70	0·02	0·45	0·17	−2·24	−2·70

suggests that in the present compound the molecule is vibrating more as a rigid body.

Discussion of the structure

The mean plane of the molecule had the following equation (x' is parallel to a, y' is parallel to b, z' is perpendicular to (001)).

$$0{\cdot}4637x' + 0{\cdot}8551y' + 0{\cdot}2319z' = 3{\cdot}1635 .$$

The deviations of the oxygen and carbon atoms from this plane are given in Table 6. It is interesting to note how the values increase and decrease as one moves round the molecule from *A* to *S* showing that the molecule is twisted. The benzene ring (atoms *B*, *C*, *D*, *E*, *F* and *G*) is strictly planar within experimental error (maximum deviation less than 0·01 Å). The aromatic naphthalene nucleus (atoms *I* to *S*) is not so planar, but the maximum deviation from its mean plane is only 0·046 Å while the carbon atom *T* is 0·062 Å out of the mean plane of the naphthalene nucleus. The main cause of the molecular distortion is clearly the steric interference between the hydrogen atom attached to atom *K* and the oxygen atom O2. In order to increase the distance between these atoms the part of the molecule *K*, *L*, *M*

Table 6. *Deviations from mean plane*

Atom	Δ	Atom	Δ	Atom	Δ
O1	−0·094 Å	*F*	0·038 Å	*M*	−0·016 Å
O2	0·154	*G*	0·035	*N*	0·040
A	−0·032	*H*	0·059	*P*	0·076
B	−0·033	*I*	0·036	*Q*	0·068
C	−0·074	*J*	0·020	*R*	0·043
D	−0·052	*K*	−0·043	*S*	0·008
E	−0·002	*L*	−0·067	*T*	−0·182

Table 7. *Bond lengths* (Å)

Bond	Length	S.D.
A–O1	1·211 (1·213)	0·008
H–O2	1·218 (1·222)	0·007
A–B	1·454 (1·457)	0·010
B–C	1·415 (1·416)	0·010
C–D	1·373 (1·376)	0·012
D–E	1·374 (1·379)	0·011
E–F	1·376 (1·377)	0·011
F–G	1·380 (1·384)	0·010
G–H	1·509 (1·511)	0·010
H–I	1·480 (1·484)	0·009
I–J	1·423 (1·424)	0·010
J–K	1·434 (1·439)	0·010
K–L	1·367 (1·368)	0·011
L–M	1·385 (1·387)	0·012
M–N	1·368 (1·372)	0·011
N–P	1·439 (1·441)	0·011
P–Q	1·403 (1·408)	0·010
Q–R	1·364 (1·366)	0·012
R–S	1·390 (1·393)	0·010
S–A	1·514 (1·516)	0·010
L–T	1·508 (1·510)	0·011
B–G	1·397 (1·402)	0·009
I–S	1·407 (1·412)	0·009
J–P	1·409 (1·413)	0·010

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

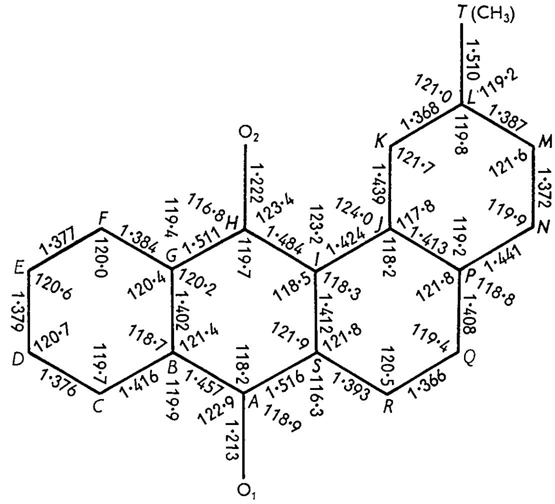

Fig. 6. Bond lengths (Å) and bond angles.

is pushed below the mean plane while O2 and atoms *F*, *G*, *H*, *I* are above the plane thus giving the molecule a twist.

The bond lengths, with their standard deviations are given in Table 7 and, together with bond angles, are shown in Fig. 6.

In Part II (Ferrier & Iball, 1963) it was pointed out that in 5-methyl-1:2-benzanthraquinone the bonds *AB* and *HI* in the quinone ring were almost equal in length (mean 1·507 Å) and the equivalent bonds *AS* and *GH* were also equal but with a mean value 1·475 Å. In the present compound the situation is reversed. We now have *AS* and *GH* almost equal with a mean length 1·511 Å while *AB* and *HI* are the shorter pair with a mean length 1·467 Å. It seems that the difference between the two pairs of bonds is a real difference and that the factor determining which is the longer pair is the position of the substituent CH_3 group. The mean C=O bond in this compound is 1·215 Å and in the 5-methyl compound it was 1·218 Å. One big difference between the two compounds is the length of the bond *IJ*, which was 1·472 Å in the 5-methyl compound but in the 2′-derivative this bond is 1·423 Å. This difference is probably related to the fact that in the 2′-compound the distance between O2 and atom *K* is 2·812 Å whereas in the 5-methyl compound this distance is only 2·766 Å.

We wish to thank the British Empire Cancer Campaign, the Royal Society and the Department of Scientific and Industrial Research for grants in support of this work. Mr David Guthrie gave valuable assistance in collecting the data and in many of the calculations, and we are indebted to Dr J. S. Rollett and Dr D. W. J. Cruickshank for the use of their programs and for advice and help in their use.

References

Burns, D. M. & Iball, J. (1955). *Proc. Roy. Soc.* A, **227**, 200.

Ferrier, R. P. & Iball, J. (1960). *Acta Cryst.* **13**, 162.

Ferrier, R. P. & Iball, J. (1963). *Acta Cryst.* **16**, 269.

Iball, J. (1940). *Amer. J. Cancer*, **38**, 372.