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The Crystal Structure of *N,N'*-Diphenyl-1,5-diaminoanthraquinone

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The crystal structure of *N,N'*-diphenyl-1,5-diaminoanthraquinone has been determined from three-dimensional X-ray diffraction data. A total of 771 structure amplitudes used in the refinement gave $R=7{\cdot}6\,\%$, and standard deviations of $\sim 0{\cdot}005$ Å in the atomic coordinates. The anthraquinone nucleus is planar while the substituent benzene rings are inclined to the central ring system by 62·4°. There is the possibility of intramolecular hydrogen bonding between the nitrogen and oxygen atoms, which are 2·602 Å apart. The other bond lengths and inter-bond angles are all within the usual range of values.

Introduction

In our programme of work on the determination of the crystal structures of substituted anthraquinones, results for indanthrone (Bailey, 1955), 1,5-dichloroanthraquinone (Bailey, 1958) and 1,5-dinitro-4,8-dihydroxyanthraquinone (Bailey & Brown, 1967) have already been published. This compound and the isomeric *N,N'*-diphenyl-1,8-diaminoanthraquinone have now been completed, and other determinations are in progress. The object was to find what influence various substituents have on the dimensions and configuration of the anthraquinone nucleus, and it was hoped that this information would later be of use in relating the shapes of larger molecules to their chemical reactivity and colour.

Experimental

Samples were obtained from the I.C.I. specimen collection, and the acicular crystals were quite suitable for X-ray photography without further treatment. Rotation and zero-level Weissenberg photographs were taken, in turn, with a crystal set up about each of its three principal axes. The angle β and values of $a \sin \beta$ and $c \sin \beta$ were determined from sets of reflexions on the Weissenberg photographs, and b was measured from a rotation photograph. Higher accuracy was not feasible owing to a lack of measurable high-order reflexions. The mean unit-cell dimensions adopted were

$$a=21{\cdot}02,\ b=4{\cdot}855,\ c=9{\cdot}34\ \text{Å},\ \beta=93°\,11'\ .$$

The space group was unambiguously $P2_1/n$, with two centrosymmetrical molecules in the unit cell. The density required by the X-ray data is 1·362, while that determined by flotation in sodium iodide solution was 1·360 g.cm^{-3}.

X-ray intensity data were obtained from Weissenberg photographs about **b** (zero and four layers) and **c** (zero layer only) by comparison with a calibrated film strip. The usual correction factors were applied and scaling was done on the computer.

Determination of the structure

A reasonable trial structure was obtained from space-filling considerations, together with the fact that the $(30\bar{1})$ plane gave the most intense reflexion on the **b** zero-level photograph, and the molecules would probably lie parallel to this plane in the unit cell. A rough diagram enabled a set of x and z coordinates to be obtained, from which a set of $F(h0l)$ were calculated which showed good agreement with the observed values. The x and z coordinates were refined first manually by successive Fourier syntheses, and then on the Pegasus computer. The y coordinates were determined by calculation assuming standard bond lengths and angles and a planar molecule. Refinement of the $\{hk0\}$ zone showed that the substituent phenyl group was twisted out of the plane of the anthraquinone nucleus, but little progress was possible two-dimensionally on account of overlapping atoms.

Three-dimensional refinement was continued by successive least-squares and structure factor calculations using the programs compiled by Cruickshank & Pilling (1961). Anisotropic thermal parameters were calculated and hydrogen atoms were included at their calculated positions assuming C–H and N–H to be 1·08 Å, but these were not refined. The refinement process was continued until the parameter shifts indicated were about one-fifth of the standard deviations. At this stage the R value over all the 771 structure amplitudes was 7·6%, and the process was considered to be complete. The final positional and thermal parameters are shown in Table 1, and the observed and calculated structure amplitudes in Table 2.

Description of the structure

Molecules of *N,N'*-diphenyl-1,5-diaminoanthraquinone are discrete, with no forces other than van der Waals between them. The nearest distances between non-hydrogen atoms in neighbouring molecules are 3·51 Å between C(2) and C(7) of the next molecule

Table 1. *Atomic parameters*

The values of B_{ij} are defined by the expression
$\exp\left[-\frac{1}{4}(h^2a^{*2}B_{11}+2hka^*b^*B_{12}+\ldots)\right]$
used in the structure amplitude calculations.

	x/a	y/b	z/c	B_{11}	B_{22}	B_{33}	B_{12}	B_{23}	B_{13}
C(1)	0·0860	0·3548	0·1675	4·12	3·16	5·50	0·41	0·04	−0·07
C(2)	0·1242	0·5385	0·0919	4·50	2·68	7·00	−0·28	0·11	0·23
C(3)	0·1217	0·5452	−0·0566	4·49	4·61	5·54	0·24	1·12	0·39
C(4)	0·0816	0·3712	−0·1343	4·18	3·82	4·93	−0·54	0·49	0·62
C(5)	0·0434	0·1906	−0·0636	3·61	3·62	5·50	0·44	0·24	0·09
C(6)	0·0432	0·1820	0·0871	3·52	3·30	5·05	0·26	0·36	0·54
C(7)	−0·0004	−0·0024	0·1542	3·89	4·31	4·20	0·47	0·49	0·28
C(8)	0·1330	0·4991	0·4046	5·37	4·50	4·60	−0·62	0·41	−0·26
C(9)	0·1983	0·4526	0·3987	5·16	4·33	7·30	0·28	−0·77	−0·09
C(10)	0·2408	0·5951	0·4894	5·50	5·29	7·67	0·12	−0·18	−0·65
C(11)	0·2179	0·7738	0·5875	6·89	6·04	5·51	−0·80	0·39	−1·39
C(12)	0·1528	0·8206	0·5926	8·02	4·95	5·38	−1·26	0·42	1·13
C(13)	0·1100	0·6801	0·5029	5·83	5·57	3·84	−0·21	0·44	0·56
N	0·0883	0·3449	0·3140	5·21	6·25	4·92	−1·46	−0·19	−0·13
O	−0·0041	−0·0115	0·2878	5·33	6·12	4·67	−1·16	0·49	0·34
H(1)	0·0546	0·2140	0·3647	Mean isotropic $B=5·30$					
H(2)	0·1560	0·6768	0·1514						
H(3)	0·1514	0·6878	−0·1114						
H(4)	0·0799	0·3755	−0·2500						
H(9)	0·2156	0·3054	0·3233	Mean isotropic $B=6·52$					
H(10)	0·2915	0·5658	0·4829						
H(11)	0·2508	0·8795	0·6617						
H(12)	0·1356	0·9686	0·6678						
H(13)	0·0594	0·7111	0·5094						

Mean e.s.d. (x,y,z) for carbon 0·006, nitrogen 0·005, oxygen 0·004 Å.
Mean e.s.d. (B_{ij}) diagonal terms for C, 0·30; N, 0·25; O, 0·25 Å^2.

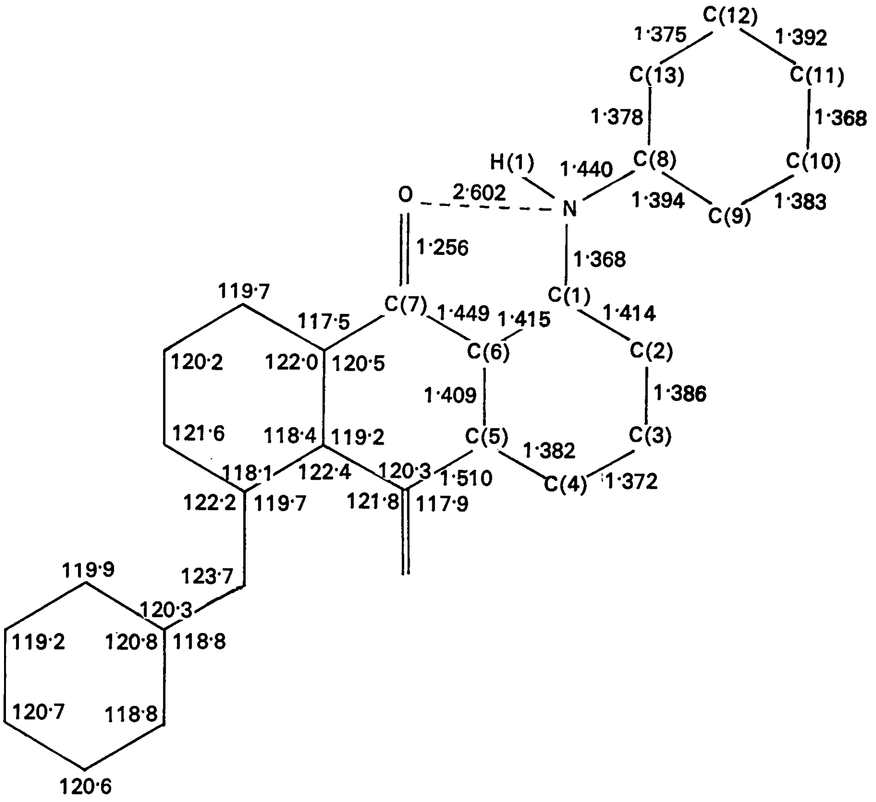

Fig. 1. Bond lengths and inter-bond angles.

Table 2. *Observed and calculated structure amplitudes*

H	K	L	F(OBS)	F(CALC)
0	0	2	45.7	48.2
0	0	4	37.8	-37.1
0	0	6	9.1	-6.9
0	0	10	6.3	6.4
0	1	1	18.3	18.7
0	1	2	47.0	46.1
0	1	3	20.4	19.1
0	1	4	8.3	7.9
0	1	7	10.3	9.6
0	2	0	19.8	21.4
0	2	1	6.6	6.4
0	2	2	14.0	-14.3
0	2	3	15.1	-15.8
0	2	4	4.7	-3.2
0	2	7	10.1	11.0
0	3	1	5.7	-5.4
0	3	2	2.2	2.2
0	3	3	18.6	-16.0
0	3	4	5.0	-3.8
0	3	5	12.2	-12.0
0	3	6	7.1	-8.5
0	3	7	9.7	9.3
0	4	0	5.0	5.1
0	4	3	6.6	5.6
0	6	0	6.0	7.6
1	0	-11	3.5	-2.5
1	0	-9	12.1	-13.2
1	0	-7	9.1	7.9
1	0	-5	40.0	-38.7
1	0	-3	12.9	-13.9
1	0	-1	47.0	48.4
1	0	1	13.3	14.1
1	0	5	19.8	-19.5
1	0	7	4.7	5.0
1	0	9	8.4	-9.5
1	1	-9	6.8	-7.2
1	1	-8	5.0	-4.6
1	1	-4	24.3	24.2
1	1	-3	6.5	-6.9
1	1	-2	9.4	10.3
1	1	-1	21.0	-22.5
1	1	0	22.2	-20.6
1	1	1	66.6	-72.2
1	1	2	66.6	-67.0
1	1	3	18.9	-18.9
1	1	5	11.6	9.5
1	2	-9	6.3	-5.8
1	2	-8	4.7	-4.4
1	2	-6	6.2	-5.2
1	2	-5	3.9	3.8
1	2	-4	23.2	-25.1
1	2	-3	27.6	31.7
1	2	-2	15.7	-16.5
1	2	-1	19.2	18.0
1	2	0	6.3	6.6
1	2	1	10.4	-10.1
1	2	2	35.0	-32.3
1	2	3	19.8	-21.2
1	2	4	11.5	-11.9
1	2	5	3.9	-3.6
1	2	7	16.0	18.1
1	3	-6	20.9	21.7
1	3	-5	6.6	-7.3
1	3	-4	7.6	7.9
1	3	-3	3.2	-2.7
1	3	-2	29.2	-27.3
1	3	-1	5.0	5.7
1	3	0	7.8	7.4
1	3	1	11.3	11.4
1	3	2	24.3	-22.3
1	3	4	6.2	6.6
1	3	5	11.1	-10.9
1	3	6	18.9	19.3
1	3	7	9.7	-10.2
1	4	-3	6.6	6.8
1	4	-2	2.5	2.2
1	4	0	6.9	-6.1
1	4	1	2.8	-2.1
1	4	2	4.5	-4.1
1	5	0	6.9	7.8
2	0	-8	8.4	-8.2
2	0	-6	4.3	3.6
2	0	-2	59.5	-65.8
2	0	0	58.7	55.4
2	0	2	37.4	-36.6
2	0	6	11.7	11.8
2	1	-7	10.3	9.0
2	1	-6	11.7	-9.4
2	1	-5	6.3	-5.7
2	1	-4	22.4	20.2
2	1	-3	18.9	-19.6
2	1	-2	48.3	52.4
2	1	-1	45.0	-48.6
2	1	0	11.7	-10.7
2	1	1	24.3	23.5
2	1	2	22.2	19.9
2	1	3	12.3	13.7
2	1	4	7.1	6.3
2	1	5	16.6	-15.8
2	1	6	10.8	11.2
2	1	9	11.7	-11.7
2	2	-7	6.5	7.3
2	2	-6	4.2	4.3
2	2	-5	9.3	8.2
2	2	-4	12.9	-14.1
2	2	-3	5.6	6.1
2	2	-2	39.4	-40.9
2	2	-1	22.2	-20.6
2	2	0	6.3	7.4
2	2	1	7.0	-6.7
2	2	2	9.0	-8.5
2	2	4	13.0	14.0
2	2	5	12.1	13.2
2	2	7	14.6	17.7
2	2	8	4.7	-3.9
2	3	-7	19.3	18.2
2	3	-5	5.5	3.8
2	3	-3	11.5	-9.0
2	3	-2	3.5	3.3
2	3	-1	10.1	-10.1
2	3	0	3.5	-2.4
2	3	1	16.6	-15.1
2	3	2	9.8	7.8
2	3	3	4.5	4.1
2	3	4	6.2	5.5
2	3	5	3.9	2.9
2	3	6	5.9	-4.9
2	4	-2	3.5	-2.8
2	4	1	5.9	-6.3
2	4	3	6.8	-6.0
2	4	4	7.5	6.2
2	4	7	3.5	-3.2
2	5	0	7.9	8.6
2	6	0	6.0	5.8
3	0	-11	5.3	3.9
3	0	-9	8.4	-8.8
3	0	-7	6.5	5.3
3	0	-5	40.9	-40.4
3	0	-3	6.5	-6.9
3	0	-1	93.4	94.5
3	0	1	37.2	36.5
3	0	5	14.4	-14.5
3	0	7	6.6	5.3
3	0	9	8.3	8.5
3	0	11	3.5	4.7
3	1	-8	5.0	-3.5
3	1	-7	4.7	-4.0
3	1	-6	4.2	-4.2
3	1	-4	12.5	-11.1
3	1	-3	17.2	-18.9
3	1	-2	5.6	6.1
3	1	-1	37.1	-38.8
3	1	0	97.4	101.2
3	1	1	3.2	-2.1
3	1	2	7.3	-7.3
3	1	3	15.4	15.4
3	1	4	7.3	-6.8
3	1	5	11.9	10.3
3	1	6	7.3	-6.4
3	2	-7	9.1	10.5
3	2	-6	13.5	14.1
3	2	-3	8.1	-8.6
3	2	-2	16.9	-19.4
3	2	-1	28.0	-26.0
3	2	0	11.3	12.5
3	2	1	8.0	-6.3
3	2	2	27.7	-26.6
3	2	3	7.1	-6.1
3	2	4	5.9	-5.7
3	2	5	3.9	-4.2
3	2	6	12.2	12.8
3	2	7	13.1	13.3
3	3	-5	5.7	5.2
3	3	-4	6.2	-6.0
3	3	-3	13.7	13.6
3	3	-2	11.5	-10.3
3	3	-1	5.3	5.2
3	3	0	23.5	21.8
3	3	1	8.3	8.7
3	3	2	4.7	-4.0
3	3	3	9.9	9.5
3	3	4	11.3	-11.4
3	3	5	3.9	4.3
3	3	6	7.3	-7.5
3	4	-3	4.5	3.7
3	4	1	2.2	2.1
3	4	2	12.1	13.3
3	4	3	9.9	-8.5
3	4	4	6.8	8.2
3	4	6	3.5	-5.3
3	5	0	7.9	8.3
3	6	0	7.9	-8.4
4	0	-4	16.1	16.0
4	0	-2	37.8	-41.1
4	0	0	23.2	-24.1
4	0	2	10.9	-10.4
4	0	4	16.6	17.1
4	0	6	4.3	5.0
4	1	-9	6.8	-6.2
4	1	-6	6.2	4.8
4	1	-5	14.6	-14.8
4	1	-4	19.5	18.9
4	1	-3	28.1	28.9
4	1	-2	7.6	7.4
4	1	-1	47.0	49.9
4	1	0	76.9	-76.1
4	1	1	33.2	34.2
4	1	2	6.2	-5.5
4	1	3	26.0	25.5
4	1	4	26.0	25.5
4	1	5	14.9	-14.1
4	1	6	6.2	6.5
4	1	7	14.9	16.2
4	1	8	6.8	6.6
4	1	9	6.6	-7.0
4	2	-7	13.0	14.7
4	2	-6	14.9	18.0
4	2	-5	3.9	-4.4
4	2	-4	17.2	20.1
4	2	-3	13.3	-13.4
4	2	-2	9.8	-7.9
4	2	-1	8.5	9.5
4	2	0	5.0	4.7
4	2	1	7.1	5.5
4	2	2	41.4	-41.1
4	2	3	5.3	-4.5
4	2	4	6.2	6.5
4	2	5	3.9	-4.1
4	2	6	15.2	17.9
4	2	8	6.6	6.3
4	3	-7	10.5	11.4
4	3	-5	3.9	2.9
4	3	-4	3.5	-2.5
4	3	-2	12.2	10.5
4	3	-1	3.5	3.0
4	3	0	6.3	5.1
4	3	1	3.5	-3.4
4	3	2	2.7	2.5
4	3	3	9.1	8.1
4	3	4	3.9	-3.6
4	3	5	4.2	4.6
4	3	6	6.2	-6.1
4	4	-4	3.5	-2.6
4	4	-2	7.8	-7.5
4	4	0	5.7	5.5
4	4	1	8.6	7.9
4	4	2	11.1	-11.5
4	4	5	6.3	-6.2
4	4	6	6.3	7.2
4	5	0	7.9	8.7
5	0	-11	3.5	3.0
5	0	-7	18.0	17.0
5	0	-5	14.7	14.5
5	0	-3	12.9	13.6
5	0	-1	27.6	-26.9
5	0	1	28.5	-27.0
5	0	3	23.3	22.5
5	0	5	15.1	15.2
5	0	7	4.7	5.1
5	0	11	3.2	2.3
5	1	-7	8.1	-7.5
5	1	-6	4.5	4.5
5	1	-5	10.8	9.1
5	1	-4	15.1	-13.4
5	1	-3	14.7	15.3
5	1	-2	18.3	-20.6
5	1	-1	39.5	-41.3
5	1	0	85.8	88.3
5	1	1	33.6	-30.7
5	1	2	13.3	-12.3
5	1	3	23.6	-21.4
5	1	4	6.2	4.8
5	1	5	3.9	4.5
5	1	6	4.5	4.8
5	1	7	13.5	13.4
5	1	8	5.0	-4.3
5	2	-7	14.6	13.7
5	2	-6	9.6	10.6
5	2	-5	3.9	-4.2
5	2	-3	13.8	-13.3
5	2	-2	24.3	-21.6
5	2	-1	26.7	-24.8
5	2	0	5.3	-4.1
5	2	2	14.0	-13.3
5	2	4	5.3	-5.3
5	2	5	11.5	-11.8
5	2	6	4.5	4.8
5	2	9	5.9	-6.0
5	3	-7	6.2	-5.4
5	3	-5	4.2	-4.0
5	3	-2	8.1	-6.2
5	3	-1	8.1	-8.3
5	3	0	6.2	-4.7
5	3	1	6.5	-6.0
5	3	3	5.9	-5.1
5	3	6	7.3	-7.2
5	4	-7	5.9	6.5
5	4	-3	8.0	-7.8
5	4	-2	8.3	9.0
5	4	-1	4.7	-4.2
5	4	0	9.9	9.8
5	4	1	6.2	4.7
5	4	2	3.2	3.2
5	4	4	6.2	-6.0
5	4	6	8.0	-9.8
5	4	7	5.7	5.3
5	5	0	6.9	7.2
6	0	-8	5.0	-4.7
6	0	-6	13.6	-13.5
6	0	-2	17.2	-17.8
6	0	0	23.8	-25.0
6	0	2	19.8	-19.7
6	0	4	11.2	-10.6
6	0	6	18.6	-18.5
6	1	-9	8.1	-7.7
6	1	-7	4.7	3.9
6	1	-6	8.7	6.4
6	1	-5	24.6	-24.3
6	1	-4	6.2	-6.2
6	1	-3	8.7	-9.0
6	1	-2	17.5	-18.8
6	1	-1	6.3	6.3
6	1	0	38.4	-39.2
6	1	1	3.5	-3.4
6	1	2	28.5	-25.7
6	1	3	22.4	-19.2
6	1	4	8.8	8.1
6	1	5	11.4	-12.6
6	1	7	13.5	14.0
6	2	-9	6.2	-5.1
6	2	-7	6.6	6.3
6	2	-6	9.9	11.9
6	2	-3	8.0	-7.1
6	2	-2	22.4	-21.2
6	2	0	36.4	32.3
6	2	1	17.2	-15.8
6	2	2	11.5	-9.7
6	2	4	3.9	-3.1
6	2	5	5.9	5.1
6	2	7	4.8	-4.3
6	2	8	4.7	-3.8
6	3	-3	8.3	7.1
6	3	-2	4.5	-2.9
6	3	-1	8.0	6.2
6	3	0	10.6	-9.1
6	3	1	11.2	9.2
6	3	2	3.2	-2.9
6	3	4	8.6	8.1
6	3	5	4.2	-4.2
6	3	6	6.2	5.4
6	4	-6	5.0	5.6
6	4	-3	4.7	3.2
6	4	-2	4.5	-3.3
6	4	-1	7.0	6.1
6	4	0	15.4	17.6
6	4	1	16.1	18.2
6	4	2	8.8	-8.4
6	4	3	6.8	6.8
6	4	4	5.0	-4.3
6	4	5	3.5	-3.2
6	4	6	7.0	5.5
6	4	7	3.2	-2.1
7	0	-3	14.2	15.2
7	0	-1	5.9	-6.5
7	0	1	21.0	21.4
7	0	5	4.3	-3.8
7	0	7	8.4	8.2
7	0	9	6.7	-6.4
7	1	-7	9.5	9.9
7	1	-6	8.8	8.7
7	1	-5	15.6	14.9
7	1	-4	3.5	-3.3
7	1	-3	10.3	10.3
7	1	-2	29.3	-33.1
7	1	-1	17.2	-18.2
7	1	0	37.8	37.6
7	1	1	9.7	10.1
7	1	2	37.1	-35.0
7	1	3	4.7	4.2
7	1	5	7.1	7.5
7	1	6	15.8	17.9
7	1	7	13.7	13.2
7	2	-8	6.5	5.7
7	2	-7	8.1	9.3
7	2	-6	4.5	4.2
7	2	-5	5.9	-5.7
7	2	-4	12.0	9.5
7	2	-2	5.5	5.3
7	2	-1	10.0	9.3
7	2	0	5.0	-4.3
7	2	1	8.3	8.3
7	2	2	24.6	26.3
7	2	4	13.4	12.8
7	2	5	7.3	-6.7
7	2	8	4.5	3.8
7	3	-2	4.7	-3.7
7	3	0	6.6	-5.4
7	3	1	7.5	6.6
7	3	2	4.7	-4.9
7	3	3	3.5	-2.9
7	3	4	8.8	8.5
7	4	-7	5.7	6.4
7	4	-6	5.0	-5.2
7	4	-4	5.0	-5.2
7	4	-3	5.9	-6.4
7	4	-2	4.5	4.2
7	4	-1	6.2	5.7
7	4	0	15.1	14.9
7	4	1	5.5	5.1
7	4	2	4.7	3.9
7	4	3	3.5	-3.2
7	4	6	5.0	-4.0
8	0	-4	6.3	6.8
8	0	-2	23.8	25.4
8	0	0	40.3	43.7
8	0	2	5.5	-5.5
8	0	4	19.3	21.9
8	0	6	4.7	3.3
8	0	8	3.5	3.2
8	1	-7	18.7	19.5
8	1	-6	11.1	10.7
8	1	-4	9.1	-8.8
8	1	-2	26.2	-25.1
8	1	-1	5.3	-5.1
8	1	0	4.2	3.8
8	1	1	11.7	-9.7
8	1	2	35.7	-32.9
8	1	3	8.0	6.5
8	1	4	9.5	-9.9
8	1	5	6.2	5.5
8	1	6	6.6	7.0
8	1	7	9.6	10.9
8	2	-5	4.2	4.5
8	2	-4	3.9	-3.5
8	2	-1	19.6	-17.8
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8	2	4	9.0	-9.1
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8	2	7	4.8	-4.4
8	2	8	4.5	-4.0
8	3	-3	3.9	2.9
8	3	-1	10.4	-10.3
8	3	0	12.5	10.9
8	3	1	4.7	3.5
8	3	2	3.5	-3.8
8	3	3	5.5	-5.0
8	3	4	5.7	-5.7
8	3	5	6.2	-7.4
8	3	6	4.5	-3.5
8	4	-7	3.2	-4.2
8	4	-4	6.3	-8.1
8	4	-3	5.0	4.9
8	4	-2	7.5	-7.8
8	4	-1	9.1	9.4
8	4	0	12.5	12.2
8	4	1	7.3	7.4
9	0	-7	5.0	-4.2
9	0	-5	10.3	-11.0
9	0	-1	34.0	-38.0
9	0	1	3.2	-2.9
9	0	3	14.1	-15.3
9	0	5	8.7	-9.3
9	0	7	15.6	16.4
9	0	9	7.6	-8.1
9	1	-7	11.8	12.2
9	1	-6	11.3	12.4
9	1	-4	5.5	5.2
9	1	-3	5.0	-5.3
9	1	-2	33.2	-34.5
9	1	-1	7.8	-7.7
9	1	0	9.9	-10.4
9	1	1	14.4	-14.2
9	1	2	29.8	-28.3
9	1	3	8.3	-7.6
9	2	-5	7.5	-6.8
9	2	-4	5.6	5.8
9	2	-3	9.1	7.6
9	2	-1	23.5	22.1
9	2	0	40.3	-38.7
9	2	1	15.1	16.8
9	2	4	8.3	7.8
9	2	5	6.3	-6.4
9	2	6	4.7	-4.3
9	3	-5	4.2	-4.6
9	3	-2	3.5	-2.3
9	3	0	17.2	16.6
9	3	1	3.5	3.8
9	3	2	3.9	-3.1
9	3	4	4.2	4.0
9	4	-8	3.5	-3.5
9	4	-1	4.7	4.4
9	4	2	3.5	-4.0
10	0	-6	10.4	11.9
10	0	-4	3.9	-4.0
10	0	-2	22.1	-21.5
10	0	0	23.2	23.5
10	0	2	43.8	-40.0
10	0	4	5.9	5.7
10	0	6	16.7	17.1
10	0	8	4.7	4.3
10	1	-7	15.4	14.7
10	1	-6	8.1	8.2
10	1	-5	4.5	-3.1
10	1	-3	12.9	-12.1
10	1	-2	3.5	-3.2
10	1	-1	10.6	-10.1
10	1	0	26.2	26.4
10	1	1	3.5	3.0
10	1	3	3.9	3.0
10	1	4	4.2	-3.8
10	1	5	4.5	-4.4
10	2	-6	6.6	-6.1
10	2	-5	9.9	9.2
10	2	-4	13.1	-12.8
10	2	-3	6.6	4.9
10	2	-2	8.3	-7.9
10	2	-1	22.4	-22.0
10	2	0	19.2	21.3
10	2	1	3.5	2.9
10	2	3	9.0	9.6
10	2	5	6.5	6.6
10	2	6	4.7	-4.5
10	3	-5	6.2	-5.3
10	3	-4	5.9	-5.4
10	3	-3	3.9	-3.0
10	3	0	12.6	13.2
10	3	1	12.9	12.1
10	3	2	3.9	4.0
10	3	5	7.3	-8.4
10	4	0	3.5	3.2
11	0	-7	7.0	6.2
11	0	-5	10.9	-11.1
11	0	-3	3.9	4.8
11	0	1	8.0	-6.5
11	0	3	15.6	-16.0
11	0	7	19.3	20.5
11	1	-7	8.4	6.9
11	1	-5	6.5	-6.3
11	1	-4	7.3	6.3
11	1	-3	15.2	-14.1
11	1	-1	5.3	5.0
11	1	0	12.4	11.6
11	1	2	3.9	-3.0
11	1	3	4.3	3.7
11	1	7	6.8	-7.6
11	2	-5	6.5	-5.6
11	2	-4	4.5	4.0
11	2	-3	5.9	5.7
11	2	-2	3.9	4.2
11	2	-1	6.5	6.3
11	2	0	11.8	-12.2
11	2	1	3.9	3.3
11	2	2	6.8	-6.9
11	2	3	5.9	-5.1
11	3	-4	7.3	-6.7
11	3	-1	10.9	11.4
11	3	0	33.2	34.8
11	3	1	8.7	8.4
11	3	2	5.7	-5.5
11	3	4	9.6	-10.3
12	0	-8	4.7	4.5
12	0	-6	24.3	24.6
12	0	-2	43.3	-46.0
12	0	2	31.0	-31.2
12	0	4	9.0	9.8
12	0	6	19.2	21.3
12	1	-6	6.8	6.5
12	1	-5	4.7	4.3
12	1	-4	9.8	8.8
12	1	-3	11.7	12.0
12	1	-1	6.6	-5.3
12	1	0	7.6	-7.1
12	2	-4	6.3	5.3
12	2	-3	6.2	7.0
12	2	-1	5.7	-4.7
12	2	0	3.9	4.0
12	2	1	5.7	6.3
12	2	2	9.3	-9.4
12	2	3	4.5	-4.2
12	2	4	4.5	5.1
12	3	-5	7.3	-7.1
12	3	-1	14.0	11.9
12	3	1	12.8	12.3
12	4	-1	5.3	-4.5
12	4	0	8.1	-8.2
12	4	1	6.3	-7.3
13	0	-7	24.8	26.5
13	0	-5	4.7	4.2
13	0	-3	13.5	-13.1
13	0	-1	13.9	-13.7
13	0	1	12.9	-13.4
13	0	3	4.5	-4.2
13	0	7	6.8	7.7
13	1	-4	6.5	-6.2
13	1	-2	4.2	-5.2
13	1	-1	4.2	3.5
13	1	0	14.9	14.6
13	1	5	5.0	-4.1
13	1	6	5.0	-6.2
13	1	7	6.6	-7.1
13	2	-5	10.4	-9.9
13	2	-4	6.5	-8.0
13	2	-3	7.5	-9.2
13	2	-2	7.3	-7.4
13	2	-1	5.9	-5.5
13	2	0	8.3	7.9
13	2	1	12.0	12.6
13	2	2	4.6	-4.0
13	2	5	8.1	-7.9
13	3	-1	10.1	-10.6
13	3	0	20.7	22.0
13	3	1	5.9	-5.8
13	3	3	4.5	4.2
13	3	4	7.3	-6.0
13	4	-1	5.3	-5.5
14	0	-4	4.7	-4.6
14	0	-2	13.6	-14.3
14	0	0	13.3	11.8
14	0	2	22.1	-24.7
14	0	4	16.6	-27.8
14	1	-7	4.7	-4.1
14	1	-6	5.0	-4.9

Table 2 (*cont.*)

H	K	L	F(OBS)	F(CALC)
14	1	-5	6.8	-6.4
14	1	-4	4.7	-3.4
14	1	-3	6.3	6.3
14	1	-1	6.2	-5.8
14	1	2	17.2	15.9
14	1	3	13.0	-11.9
14	1	5	5.0	-3.5
14	1	6	10.9	-10.5
14	1	7	7.8	8.7
14	2	-5	4.7	-4.6
14	2	-1	10.6	10.9
14	2	0	12.2	13.7
14	2	1	10.8	11.3
14	2	5	4.7	-4.2
14	3	-5	7.1	-7.4
14	3	-1	19.0	19.7
14	3	0	7.3	-7.3
14	3	1	7.3	7.1
14	3	3	4.5	-4.5
14	3	4	4.2	3.4
14	4	-1	3.5	2.0
14	4	0	3.5	4.1
14	4	1	5.0	4.3
15	0	-7	16.0	18.0
15	0	-5	7.0	6.2
15	0	-3	4.7	-4.6
15	0	3	13.5	12.5
15	0	5	7.0	6.4
15	1	-6	8.4	8.3
15	1	-5	6.8	6.5
15	1	-4	4.7	5.2
15	1	-3	9.3	8.0
15	1	-2	6.5	-6.3
15	1	1	6.3	5.5
15	1	2	13.1	-13.4
15	1	3	6.6	6.1
15	1	4	10.8	10.0
15	1	6	10.6	10.5
15	2	-3	4.7	5.7
15	2	-2	7.8	6.4
15	2	0	22.5	25.0
15	3	-2	6.2	-5.7
15	3	1	4.5	-4.0
16	0	-6	7.0	6.0
16	0	-2	4.7	-4.2
16	0	0	8.0	7.4
16	0	2	8.3	8.6
16	0	6	6.6	-6.5
16	1	-7	7.8	7.6
16	1	-6	6.8	-6.8
16	1	-3	4.7	-5.1
16	1	-2	6.7	6.7
16	1	-1	8.4	-7.1
16	1	0	4.7	-4.0
16	1	4	5.0	-3.7
16	1	6	9.1	-9.0
16	1	7	5.9	4.9
16	2	-5	6.5	-7.1
16	2	-4	8.1	-7.8
16	2	-3	6.6	-7.4
16	2	0	20.5	23.4
16	3	-2	4.5	-4.8
16	3	0	8.6	-8.6
16	4	-1	4.7	-4.4
16	4	0	5.9	7.7
16	4	1	3.5	-3.1
17	0	-7	4.5	-4.1
17	0	-5	5.0	-4.9
17	0	-3	5.0	5.3
17	0	3	12.1	-12.0
17	0	5	9.7	-9.8
17	1	-7	4.5	-4.8
17	1	-6	6.6	5.6
17	1	-5	5.0	-4.1
17	1	-4	5.0	-4.0
17	1	-3	5.0	4.1
17	1	-2	9.7	-8.4
17	1	-1	4.7	3.9
17	1	0	6.8	6.2
17	1	1	15.2	15.3
17	1	2	18.9	-17.8
17	1	3	5.0	4.1
17	1	4	6.8	-5.4
17	1	5	6.6	-6.3
17	1	6	4.5	3.3
17	1	7	3.9	-2.7
17	2	-5	4.5	-5.4
17	2	-1	6.6	6.3
17	2	0	6.6	6.9
17	2	1	6.6	5.1
17	3	-2	4.3	4.1
17	3	-1	4.2	-4.9
17	4	-2	4.5	-4.5
17	4	-1	4.5	5.8
17	4	0	3.2	-4.6
17	4	1	4.5	3.1
18	0	2	5.0	4.2
18	0	4	8.4	8.8
18	1	-7	8.1	8.1
18	1	-2	5.0	4.1
18	1	0	9.7	9.1
18	2	-5	4.5	-3.2
18	2	-2	6.6	-5.4
18	2	-1	8.1	7.3
18	2	2	8.0	-7.9
18	3	-2	5.7	5.8
18	3	-1	7.1	-8.1
18	4	0	5.3	6.6
18	4	1	4.2	-4.1
18	4	2	3.9	-3.2
19	0	-1	7.0	-8.5
19	0	1	7.0	6.7
19	1	-7	3.9	-4.3
19	1	-5	4.7	-4.2
19	1	-4	6.8	-6.1
19	1	-2	5.0	-4.4
19	1	-1	5.0	5.3
19	1	0	16.9	17.1
19	1	1	5.0	3.9
19	2	0	4.7	-4.3
19	4	0	2.8	-4.0
20	0	0	5.0	5.4
20	0	2	6.8	-5.9
20	1	-1	8.3	9.4
20	1	0	11.8	12.0
20	1	1	6.8	6.3
20	2	-2	6.3	-4.9
20	2	2	6.2	-5.3
21	1	-5	5.7	-5.1
21	1	-1	4.7	3.4
21	1	0	6.6	6.6
21	2	-1	7.3	-6.9
22	0	-4	4.2	-4.9
22	0	0	8.0	10.0
22	1	-5	3.9	-3.4
22	1	0	4.5	3.7
22	1	1	4.5	3.3
23	0	-1	8.7	9.2
23	0	1	8.4	9.3
24	0	-4	4.7	-4.5
24	0	0	12.5	13.4
25	0	-1	7.0	7.4
25	0	1	3.5	3.5
26	0	0	5.0	5.9

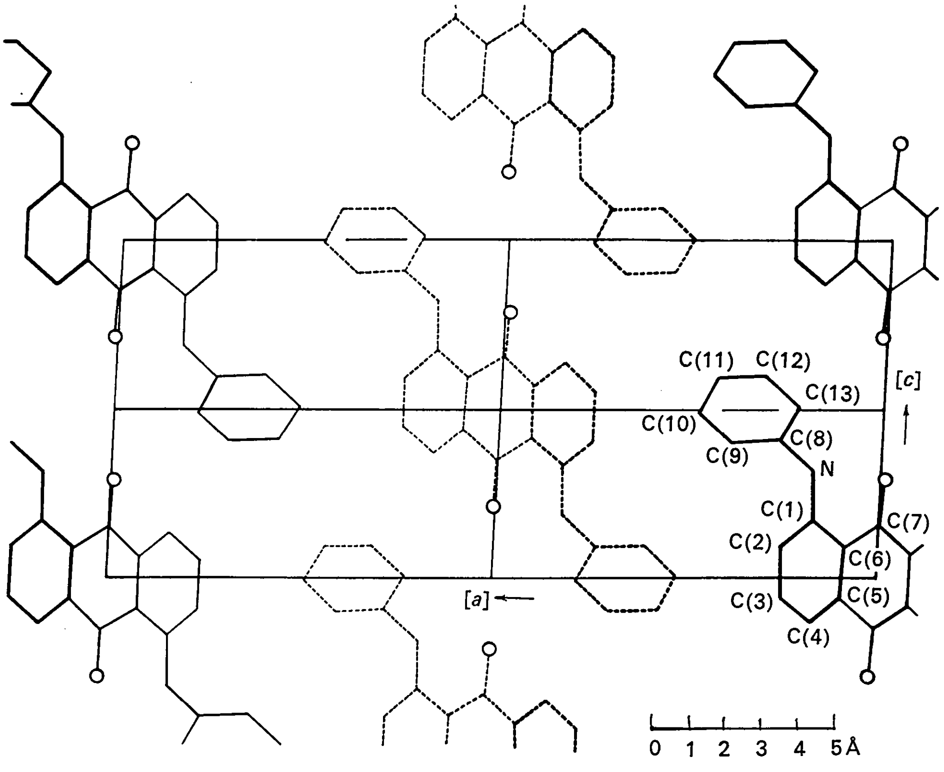

Fig. 2. Projection of structure along **b**.

along **b**, and 3·39 Å between C(13) and O(1), the doubly bound oxygen atom having a somewhat smaller radius than a carbon atom with an attached hydrogen atom. The anthraquinone nucleus is planar within experimental error and has the equation, referred to standard orthogonal axes:

$$0{\cdot}7121\,X-0{\cdot}7020\,Y+0{\cdot}0100\,Z=0\,.$$

The mean displacement of the carbon atoms out of this plane is 0·018 Å. The substituent phenyl ring has the equation:

$$0{\cdot}0849\,X+0{\cdot}7370\,Y-0{\cdot}6706\,Z=0{\cdot}5315$$

and the mean distance of the carbon atoms out of this plane is 0·008 Å. The normals to these two planes are inclined at 62·4°. The bonds from the nitrogen atom make angles of ~1° with the two planes, but this may not be significant.

The distance between the pairs of nitrogen and oxygen atoms lying on the same side of the anthraquinone nucleus is 2·602 Å, and this is convenient for hydrogen bond formation, although such a bond would probably

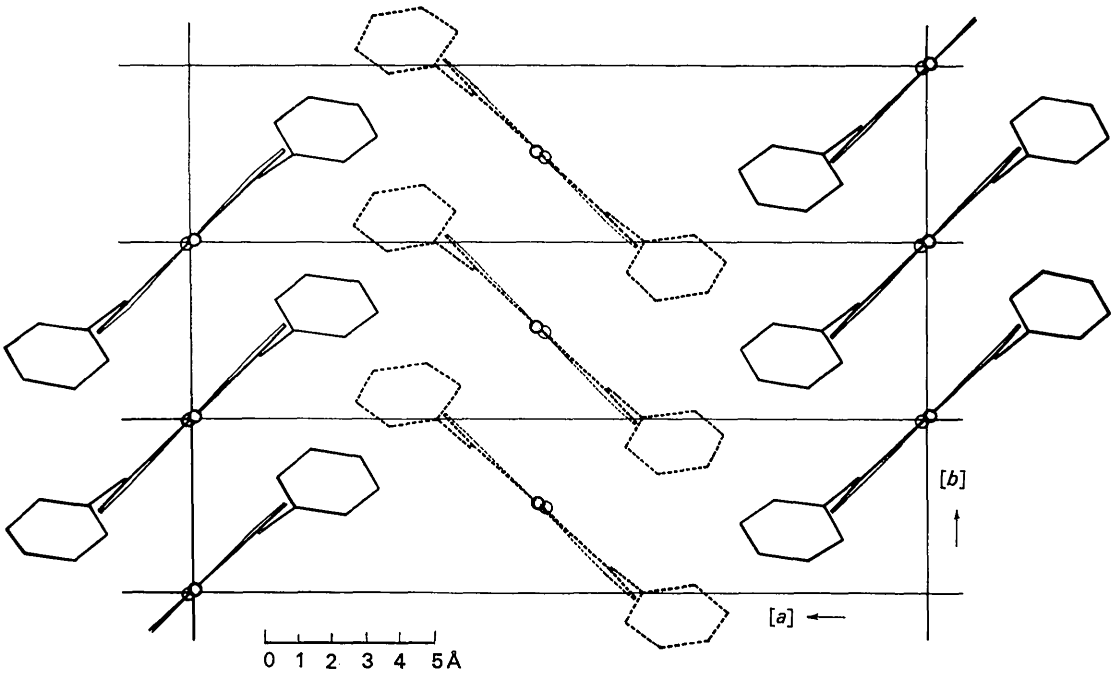

Fig. 3. Projection of structure along **c**.

not be linear. Whether they are formed or not has not been proved as the coordinates of the hydrogen atom H(1) have not been determined directly. If the N–H bond were to bisect the external angle between the N–C(1) and the N–C(8) bonds, the angle at the hydrogen atom would be 130°, and the overall (bent) length of the N–H–O bond would be 2·85 Å.

The intramolecular bond lengths and the inter-bond angles are shown in Fig. 1, as well as the numbering of the atoms used in this work. The mean C–C length in the phenyl ring is 1·382 Å, while that in the outer ring of the anthraquinone nucleus is 1·396 Å, compared with 1·393 Å in anthraquinone at room temperature (Lonsdale, Milledge & El Sayed, 1966). The C=O distance is 1·256 Å, as compared with 1·243 Å in anthraquinone and 1·25 Å in 1,5-dichloroanthraquinone (Bailey, 1958). The lack of equivalence in the lengths of two pairs of bonds, C(6)–C(7) and C(5)–C(7) at 1·449 and 1·510 Å, and N–C(1) and N–C(8) at 1·368 and 1·440 Å, which might be expected to be comparable, raises the question as to whether the ring containing C(1), N, H(1), O, C(7) and C(6) might be behaving as a resonating system with the surplus electrons from the oxygen and nitrogen atoms increasing the order of the C(6)–C(7) and C(1)–N bonds. Further work on other similar compounds needs to be done before this can be answered.

The projections of the structure along **b** and **c** are shown in Figs. 2 and 3. An attempt to analyse the thermal vibrations further led to inconclusive results as the molecule cannot be treated as a rigid body, and a more exhaustive analysis requires computer programs which are not yet available.

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