

1.14 (2) Å and the C—H bond lengths are both 0.95 (2) Å. The shortest intermolecular H—H distance is 2.49 (2) Å.

Table 3. Bond distances (Å) and angles (°)

(a) Distances in the *nido*-C₂H₁₁B₈ moiety

B(1')—B(2')	1.752 (3)	B(3')—B(8')	1.774 (2)
B(1')—B(3')	1.789 (2)	B(4')—B(8')	1.788 (2)
B(1')—B(4')	1.782 (2)	B(4')—B(9')	1.727 (2)
B(1')—C(5')	1.657 (2)	B(4')—B(10')	1.814 (2)
B(1')—B(10')	1.805 (2)	C(5')—C(6')	1.457 (2)
B(2')—B(3')	1.758 (2)	C(5')—B(10')	1.751 (2)
B(2')—C(5')	1.709 (3)	C(6')—B(7')	1.508 (2)
B(2')—C(6')	1.691 (2)	B(7')—B(8')	1.937 (2)
B(2')—B(7')	1.813 (2)	B(8')—B(9')	1.782 (2)
B(3')—B(4')	1.815 (2)	B(8')—B(3)	1.681 (2)
B(3')—B(7')	1.777 (2)	B(9')—B(10')	1.783 (2)

(b) Selected angles in the *nido*-C₂H₁₁B₈ moiety involving C atoms

B(2')—B(1')—C(5')	60.1 (1)	B(1')—C(5')—B(10')	63.9 (1)
C(5')—B(1')—B(10')	60.6 (1)	B(2')—C(5')—C(6')	64.0 (1)
B(1')—B(2')—C(5')	57.2 (1)	B(2')—C(6')—C(5')	65.3 (1)
C(5')—B(2')—C(6')	50.8 (1)	B(2')—C(6')—B(7')	68.8 (1)
C(6')—B(2')—B(7')	50.9 (1)	B(2')—B(7')—C(6')	60.4 (1)
B(1')—C(5')—B(2')	62.7 (1)	B(1')—B(10')—C(5')	55.5 (1)

(c) Distances in the *closo*-C₂H₁₁B₁₀ moiety

C(1)—C(2)	1.622 (2)	B(5)—B(6)	1.764 (3)
C(1)—B(3)	1.728 (2)	B(5)—B(9)	1.772 (3)
C(1)—B(4)	1.690 (2)	B(5)—B(10)	1.769 (3)
C(1)—B(5)	1.701 (2)	B(6)—B(10)	1.758 (3)
C(1)—B(6)	1.712 (2)	B(6)—B(11)	1.773 (3)
C(2)—B(3)	1.728 (2)	B(7)—B(8)	1.765 (3)
C(2)—B(6)	1.721 (2)	B(7)—B(11)	1.778 (2)
C(2)—B(7)	1.685 (2)	B(7)—B(12)	1.767 (2)
C(2)—B(11)	1.701 (2)	B(8)—B(9)	1.787 (3)
B(3)—B(4)	1.785 (2)	B(8)—B(12)	1.786 (2)
B(3)—B(7)	1.788 (2)	B(9)—B(10)	1.780 (2)
B(3)—B(8)	1.776 (2)	B(9)—B(12)	1.772 (3)
B(4)—B(5)	1.783 (3)	B(10)—B(11)	1.766 (3)
B(4)—B(8)	1.766 (3)	B(10)—B(12)	1.787 (3)
B(4)—B(9)	1.768 (2)	B(11)—B(12)	1.762 (3)

Acta Cryst. (1982). B38, 3149–3151

1,4-Bis(isopropylamino)anthraquinone

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(Received 12 February 1982; accepted 28 June 1982)

Abstract. C₂₀H₂₂N₂O₂, $M_r = 322.18$, trigonal, $R\bar{3}$, $a = 15.030$ (5) Å, $\alpha = 106.4$ (1)°, $V = 2872.65$ Å³, $D_m = 1.165$, $D_c = 1.118$ Mg m⁻³, $Z = 6$, λ (Mo) = 0.71069 Å, μ (Mo $K\alpha$) = 0.079 mm⁻¹, $F(000) = 1032$,

0567-7408/82/123149-03\$01.00

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$R = 0.056$ for 2307 observed reflexions. The structure comprises discrete molecules with two internal hydrogen bonds N—H...O per molecule but no intermolecular interactions other than van der Waals forces.

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Introduction. The title compound is a blue pigment of the solvent anthraquinone class (CI 61551). Our sample was supplied by Imperial Chemical Industries Limited (trade name 'Waxoline Blue') and was purified by repeated extraction with water to remove inorganic salts and then recrystallized from xylene. Two habits dominated, tabular and acicular, both exhibiting 60° angles between faces. Elemental analysis gave C 75.25, H 7.02, N 8.26% (required for $C_{20}H_{22}N_2O_2$: C 74.51, H 6.88, N 8.69%).

Unit-cell dimensions were obtained in the first instance from Weissenberg photographs which indicated a hexagonal lattice and only reflexions with $(-h + k + l) = 3n$. The parameters were subsequently refined from diffractometer measurements by least squares giving $a(\text{hex}) = 24.08(7)$ and $c(\text{hex}) = 17.15(3)$ Å. From these values the trigonal cell shown above was deduced. Of the five permissible rhombohedral space groups the probable choice lay between $R\bar{3}$ and $R\bar{3}m$ by virtue of the pyroelectric test and a statistical analysis of the intensity data; the first of these, $R\bar{3}$, was preferred and subsequently confirmed by the results.

Intensity measurements were obtained from a needle-shaped crystal, $4.0 \times 0.25 \times 0.25$ mm, mounted about $[111]$ on a Stoe Stadi-2 diffractometer using Weissenberg geometry and ω scans. 3776 independent reflexions were measured from the zero and 17 levels with $2\theta < 60^\circ$. Of these, 2307 were significantly above background and were used in the structure determination and refinement. The data were corrected for Lp effects but not for absorption which was negligible for Mo $K\alpha$.

The structure was solved by *MULTAN* (Main, Lessinger, Woolfson, Germain & Declercq, 1978). The *E* map showed 22 of the 24 non-H atoms; two C atoms were missing from an isopropyl group because the SEARCH scan used in *MULTAN* did not extend far enough in the *y* direction. These missing atoms were supplied by calculation. There were three permissible orientations which maintained a tetrahedral distribution around C(18) and the usual torsion angles about the C(18)–N(2) bond; the arrangement giving the best structure-factor agreement was accepted and this choice was confirmed both by the subsequent refinement and by the low value ($0.3 \text{ e } \text{Å}^{-3}$) of the residual electron density.

Difference Fourier syntheses failed to locate precise positions for the H atoms so these were calculated (C–H and N–H both = 1.08 Å, and appropriate torsion angles at the methyl groups) and included in the structure-factor calculations with $B_{\text{iso}} = 6.0 \text{ Å}^2$ for the trigonally bonded and 8.0 Å^2 for the tetrahedral. The weighting scheme in the least-squares refinement, which was carried out on a DEC-10 computer using the NRC suite of programs (Ahmed, 1970), was $w = (3.0 + F + 0.01F^2)^{-1}$ and, after a number of cycles first with B_{iso}

Table 1. Final atomic parameters and e.s.d.'s

$$B_{\text{eq}} = \frac{1}{3}(B_{11} + B_{22} + B_{33}) \text{ where } B_{11} = 4\beta_{11}/(a^*)^2 \text{ etc.}$$

	<i>x</i>	<i>y</i>	<i>z</i>	B_{eq} (Å ²)
C(1)	0.3364 (2)	−0.0452 (2)	0.6075 (2)	4.02 (12)
C(2)	0.3172 (2)	−0.1224 (2)	0.6464 (2)	4.86 (15)
C(3)	0.3910 (2)	−0.1487 (2)	0.6924 (2)	4.57 (14)
C(4)	0.4943 (2)	−0.0975 (2)	0.7090 (2)	4.35 (14)
C(5)	0.7418 (2)	0.1736 (2)	0.6731 (2)	4.55 (14)
C(6)	0.7650 (2)	0.2511 (2)	0.6362 (2)	5.61 (14)
C(7)	0.6860 (2)	0.2694 (2)	0.5808 (2)	4.63 (14)
C(8)	0.5867 (2)	0.2171 (2)	0.5638 (2)	4.00 (12)
C(9)	0.4586 (2)	0.0855 (2)	0.5839 (2)	3.25 (11)
C(10)	0.6221 (2)	0.0409 (2)	0.6953 (2)	4.00 (12)
C(11)	0.6433 (2)	0.1216 (2)	0.6568 (2)	3.66 (11)
C(12)	0.5658 (2)	0.1421 (2)	0.6022 (2)	3.22 (11)
C(13)	0.4368 (2)	0.0064 (2)	0.6228 (2)	3.08 (11)
C(14)	0.5175 (2)	−0.0165 (2)	0.6760 (2)	3.77 (12)
C(15)	0.1512 (2)	−0.0790 (2)	0.5364 (3)	5.62 (18)
C(16)	0.1318 (3)	−0.0349 (3)	0.6321 (3)	8.28 (27)
C(17)	0.0863 (2)	−0.0667 (3)	0.4471 (3)	6.99 (23)
C(18)	0.5522 (3)	−0.2149 (3)	0.7800 (3)	6.19 (22)
C(19)	0.5123 (3)	−0.3080 (3)	0.6823 (4)	7.32 (27)
C(20)	0.6494 (3)	−0.2023 (4)	0.8562 (3)	8.89 (35)
N(1)	0.2586 (2)	−0.0273 (2)	0.5559 (2)	4.52 (12)
N(2)	0.5680 (2)	−0.1226 (2)	0.7587 (2)	4.80 (13)
O(1)	0.3920 (1)	0.1078 (1)	0.5344 (1)	4.53 (10)
O(2)	0.6949 (1)	0.0241 (2)	0.7429 (2)	5.30 (10)

and later with B_{ij} , the parameter shifts were all less than 0.5σ . The final positional and isotropic thermal parameters are given in Table 1.*

Discussion. The numbering of the atoms used in this analysis is shown in Fig. 1, and the bond lengths and angles are given in Table 2. The geometry of the molecule is similar to that found in other anthraquinone derivatives (Bailey & Brown, 1967). The shortening of C(2)–C(3) to 1.354 Å was unexpected and may be due to conjugation resulting from the position of the bond between C(1)–N(1) and C(4)–N(2) both of which are shortened by the interaction of the N lone-pair electrons with the π electrons of the anthraquinone ring. The lengthening of C(13)–C(14) to 1.441 Å is not explained, but it is noteworthy that it is the diamino-substituted ring [C(1), C(2), C(3), C(4), C(14), C(13)] which contains the abnormal bond lengths, while the unsubstituted ring [C(5), C(6), C(7), C(8), C(12), C(11)] is necessarily much more regular.

The equation of the plane of the anthraquinone ring is $0.3642X - 0.3626Y - 0.8578Z + 4.4071 = 0$. The average deviation of atoms C(1)–(3) and C(5)–(14) from this mean plane is $0.021(2)$ Å; C(4) is $0.074(3)$ and N(2) $0.107(2)$ Å out of the plane. Evidently the

* Lists of structure factors, H-atom parameters and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38040 (20 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

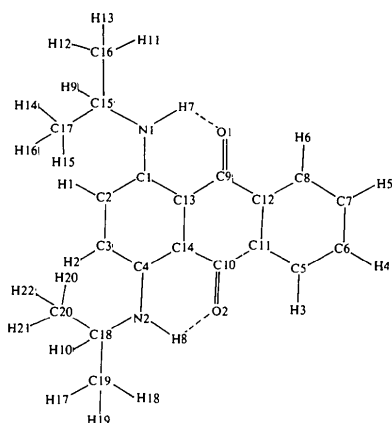
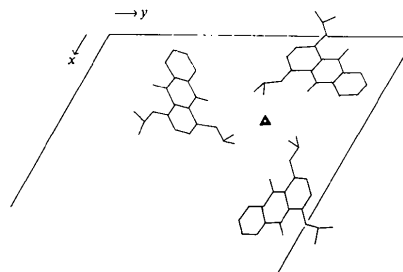


Fig. 1. Diagram of the molecule showing numbering of atoms.

Table 2. Bond lengths (Å) and inter-bond angles (°)

C(1)–C(2)	1.435 (4)	C(10)–C(14)	1.461 (4)
C(2)–C(3)	1.354 (4)	C(13)–C(9)	1.463 (3)
C(3)–C(4)	1.436 (4)	C(9)–C(12)	1.495 (4)
C(4)–C(14)	1.432 (4)	C(9)–O(1)	1.256 (3)
C(14)–C(13)	1.441 (3)	C(10)–O(2)	1.259 (4)
C(13)–C(1)	1.405 (4)	C(1)–N(1)	1.350 (4)
C(5)–C(6)	1.426 (4)	C(4)–N(2)	1.363 (4)
C(6)–C(7)	1.389 (4)	N(1)–C(15)	1.480 (4)
C(7)–C(8)	1.386 (4)	N(2)–C(18)	1.479 (5)
C(8)–C(12)	1.405 (4)	C(15)–C(16)	1.550 (6)
C(12)–C(11)	1.390 (4)	C(15)–C(17)	1.514 (6)
C(11)–C(5)	1.377 (4)	C(18)–C(19)	1.531 (6)
C(11)–C(10)	1.480 (4)	C(18)–C(20)	1.503 (6)
C(1)–C(2)–C(3)	122.3 (3)	C(9)–C(12)–C(11)	120.8 (2)
C(2)–C(3)–C(4)	121.6 (3)	C(12)–C(11)–C(5)	120.6 (2)
C(3)–C(4)–C(14)	117.9 (2)	C(12)–C(11)–C(10)	121.1 (2)
C(3)–C(4)–N(2)	120.7 (3)	C(5)–C(11)–C(10)	118.2 (2)
C(14)–C(4)–N(2)	121.4 (2)	C(6)–C(5)–C(11)	119.8 (2)
C(4)–C(14)–C(13)	119.2 (2)	C(12)–C(9)–C(13)	118.6 (2)
C(4)–C(14)–C(10)	119.8 (2)	C(12)–C(9)–O(1)	117.8 (2)
C(13)–C(14)–C(10)	120.9 (2)	C(13)–C(9)–O(1)	123.6 (2)
C(14)–C(13)–C(1)	121.4 (2)	C(11)–C(10)–C(14)	118.6 (2)
C(14)–C(13)–C(9)	119.9 (2)	C(11)–C(10)–O(2)	118.7 (2)
C(9)–C(13)–C(1)	118.6 (2)	C(14)–C(10)–O(2)	122.8 (2)
C(13)–C(1)–C(2)	117.4 (2)	C(1)–N(1)–C(15)	125.9 (2)
C(13)–C(1)–N(1)	122.9 (2)	N(1)–C(15)–C(16)	109.7 (3)
C(2)–C(1)–N(1)	119.6 (2)	N(1)–C(15)–C(17)	108.8 (3)
C(5)–C(6)–C(7)	118.5 (3)	C(16)–C(15)–C(17)	111.6 (3)
C(6)–C(7)–C(8)	122.0 (3)	C(4)–N(2)–C(18)	126.0 (3)
C(7)–C(8)–C(12)	118.5 (2)	N(2)–C(18)–C(19)	110.5 (3)
C(8)–C(12)–C(11)	120.6 (2)	N(2)–C(18)–C(20)	108.8 (3)
C(8)–C(12)–C(9)	118.6 (2)	C(19)–C(18)–C(20)	112.5 (4)

external pressures on the C(18)–(20) isopropyl group are sufficient to distort the C(4) region of the anthraquinone ring from true planarity. The planarity

Fig. 2. Arrangement of molecules with the same z coordinates in a hexagonal unit cell around $\bar{3}$ at $\frac{1}{3}, \frac{2}{3}, \frac{2}{3}$.

of the three six-membered rings was tested separately; the equation of (a) C(1)–(4), C(14), C(13) is $0.3335X - 0.3470Y - 0.8766Z + 4.7566 = 0$ and the average deviation 0.015 \AA . The equation of (b) C(9)–(14) is $0.3702X - 0.3599Y - 0.8564Z + 4.3782 = 0$ and the average deviation 0.008 \AA . The equation of (c) C(5)–(8), C(12), C(11) is $0.3734X - 0.3778Y - 0.8472Z + 4.2501 = 0$ and the average deviation 0.005 \AA . The dihedral angles between planes (a) and (b), (b) and (c), and (a) and (c) are respectively $2.51 (5)$, $1.16 (5)$, and $3.34 (5)^\circ$. It is evident that ring (a) shows the most departure from planarity; if ring (b) is taken as the norm, then C(1) is $-0.054 (2) \text{ \AA}$ out of the plane and C(4) is $+0.073 (3) \text{ \AA}$ out. This is consistent with the view that the two isopropyl groups are tending to distort ring (a) into a modified chair shape by pulling C(1) and C(4) in opposite directions.

Fig. 2 shows the distribution of three molecules whose corresponding atoms all have the same z (hexagonal) coordinates around one of the $\bar{3}$ axes; there are, of course, another three related by the $\bar{3}$ inversion centre.

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