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1-Amino-4-phenylamino-9,10-anthracenedione

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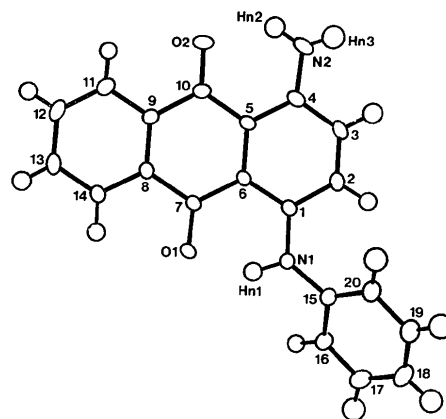
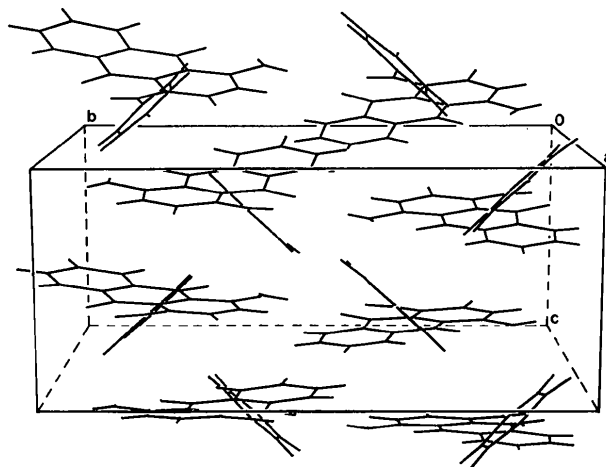
Abstract. C₂₀H₁₄N₂O₂, $M_r = 314.3$, monoclinic, $P2_1/c$, $a = 12.251$ (2), $b = 16.746$ (3), $c = 7.347$ (1) Å, $\beta = 91.69$ (1)°, $U = 1507$ (2) Å³, $Z = 4$, $D_m = 1.40$, $D_x = 1.385$ Mg m⁻³, Mo $K\alpha$ radiation, $\lambda = 0.7107$ Å, $\mu = 0.052$ mm⁻¹, $F(000) = 654$, $T = 295$ (2) K, $R = 0.050$ for 1573 reflections with $I \geq 2.5\sigma(I)$. The anthraquinone nucleus is nearly planar as exemplified by the dihedral angle of 5.3° formed between the outer six-membered rings; the nitrogen-bound phenyl group is not coplanar with the remaining molecule but forms a dihedral angle of 44.6°. Within the crystal lattice there are no significant intermolecular contacts less than those expected from normal van der Waals radii separations; the closest contact of 2.51 (5) Å occurs between O(1) and H(3')(1-x, ½+y, ½-z). Within the individual molecules however there are two close intramolecular hydrogen-bonding contacts of 1.76 (4) Å for N(1)-H(n1)···O(1) and 1.89 (4) Å for N(2)-H(n2)···O(2).

Experimental. C₂₀H₁₄N₂O₂ was obtained as well formed deep-coloured crystals from the recrystallization of a solution (benzene/80–120°-petroleum spirit) of technical-grade (Fluka A.G.) 1,4-diamino-

anthraquinone. The compound is registered as C. I. Disperse Blue-19. Density by flotation. Enraf-Nonius CAD-4F diffractometer controlled by a PDP8/A computer, graphite-monochromated Mo $K\alpha$ radiation; $\omega:2\theta$ scan technique. Cell parameters on crystal 0.44 × 0.31 × 0.11 mm from least-squares procedure (de Boer & Duisenberg, 1984) on 25 reflections ($8 \leq \theta \leq 12^\circ$). No absorption correction. Total of 3779 reflections ($1 \leq \theta \leq 27.5^\circ$) measured in the range $-15 \leq h \leq 1$, $-21 \leq k \leq 0$, $-9 \leq l \leq 9$. No significant variation in the intensities of three standards ($2\bar{4}2$, $\bar{1}15$, $\bar{1}72$) monitored every 7200 s. 3456 unique reflections ($R_{\text{int}} = 0.025$), 1573 satisfied $I \geq 2.5\sigma(I)$. Structure solved by direct methods with *SHELXS86* (Sheldrick, 1986), full-matrix least-squares refinement of 273 parameters based on F (Sheldrick, 1976). Anisotropic thermal parameters for non-H atoms, H atoms located from difference map and refined. At convergence $R = 0.050$, $wR = 0.054$, $w = 3.62/[\sigma^2(F) + 0.0015F^2]$, $S = 2.3$, $(\Delta/\sigma)_{\text{max}} \leq 0.001$, $(\Delta\rho)_{\text{max}} = 0.17$, $(\Delta\rho)_{\text{min}} = -0.28$ e Å⁻³; no extinction correction. Scattering factors for all atoms given in *SHELX76* (Sheldrick, 1976), all calculations on laboratory μ -VAX computer system. Atomic parameters given in Table 1, selected bond

Table 1. Fractional atomic coordinates and B_{eq} (\AA^2) values
$$B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3.$$

	x	y	z	B_{eq}
O(1)	0.5298 (2)	0.5463 (1)	0.2243 (3)	4.63
O(2)	0.7846 (2)	0.7956 (2)	0.0462 (4)	5.74
N(1)	0.3727 (2)	0.6405 (2)	0.3134 (4)	4.41
N(2)	0.6353 (4)	0.8978 (2)	0.1359 (5)	5.15
C(1)	0.4409 (3)	0.7021 (2)	0.2707 (4)	3.40
C(2)	0.4085 (3)	0.7826 (2)	0.2963 (5)	4.20
C(3)	0.4738 (3)	0.8438 (2)	0.2524 (5)	4.30
C(4)	0.5770 (3)	0.8322 (2)	0.1815 (5)	3.93
C(5)	0.6148 (3)	0.7537 (2)	0.1581 (4)	3.32
C(6)	0.5457 (3)	0.6875 (2)	0.2042 (4)	3.12
C(7)	0.5842 (3)	0.6058 (2)	0.1803 (4)	3.30
C(8)	0.6915 (3)	0.5929 (2)	0.0959 (4)	3.37
C(9)	0.7575 (3)	0.6572 (2)	0.0515 (4)	3.55
C(10)	0.7221 (3)	0.7402 (2)	0.0851 (5)	3.83
C(11)	0.8560 (3)	0.6432 (3)	-0.0306 (5)	4.91
C(12)	0.8881 (3)	0.5667 (3)	-0.0689 (6)	5.45
C(13)	0.8231 (3)	0.5033 (3)	-0.0241 (6)	5.27
C(14)	0.7249 (3)	0.5154 (2)	-0.0572 (5)	4.27
C(15)	0.2615 (3)	0.6436 (2)	0.3559 (5)	4.07
C(16)	0.1870 (3)	0.6938 (3)	0.2693 (6)	5.01
C(17)	0.0782 (4)	0.6899 (3)	0.3122 (7)	6.28
C(18)	0.0426 (4)	0.6347 (3)	0.4366 (8)	7.19
C(19)	0.1167 (4)	0.5843 (3)	0.5221 (7)	6.58
C(20)	0.2252 (3)	0.5882 (3)	0.4821 (5)	4.72
H(n1)	0.4116 (28)	0.5921 (23)	0.3081 (44)	4.84
H(n2)	0.7052 (34)	0.8886 (23)	0.1055 (51)	5.83
H(n3)	0.6063 (38)	0.9399 (32)	0.1626 (63)	8.01
H(2)	0.3387 (27)	0.7928 (20)	0.3479 (45)	4.49
H(3)	0.4517 (30)	0.8969 (25)	0.2738 (49)	5.92
H(11)	0.8986 (25)	0.6858 (20)	-0.0604 (42)	3.96
H(12)	0.9523 (29)	0.5575 (20)	-0.1189 (45)	4.42
H(13)	0.8437 (28)	0.4508 (23)	-0.0462 (46)	5.09
H(14)	0.6745 (26)	0.4717 (22)	0.0802 (43)	4.56
H(16)	0.2101 (33)	0.7318 (25)	0.1789 (59)	7.22
H(17)	0.0285 (36)	0.7254 (27)	0.2439 (62)	7.70
H(18)	-0.0333 (40)	0.6353 (24)	0.4591 (54)	7.41
H(19)	0.0948 (35)	0.5413 (27)	0.6035 (62)	7.64
H(20)	0.2785 (28)	0.5539 (20)	0.5428 (45)	4.39

Fig. 1. Molecular structure and crystallographic numbering scheme for $C_{20}H_{14}N_2O_2$. H atoms not indicated otherwise are numbered according to the C atom to which they are bonded (Johnson, 1971).Fig. 2. Unit-cell contents of $C_{20}H_{14}N_2O_2$.Table 2. Interatomic distances (\AA) and bond angles ($^\circ$)

O(1)—C(7)	1.246 (4)	O(2)—C(10)	1.241 (4)
N(1)—C(1)	1.370 (4)	N(2)—C(4)	1.359 (4)
C(1)—C(2)	1.418 (4)	C(1)—C(6)	1.409 (4)
C(2)—C(3)	1.346 (5)	C(3)—C(4)	1.395 (5)
C(4)—C(5)	1.406 (4)	C(5)—C(6)	1.442 (4)
C(5)—C(10)	1.452 (4)	C(6)—C(7)	1.459 (4)
C(7)—C(8)	1.485 (4)	C(8)—C(9)	1.391 (4)
C(8)—C(14)	1.393 (5)	C(9)—C(10)	1.479 (5)
C(9)—C(11)	1.385 (5)	C(11)—C(12)	1.372 (6)
C(12)—C(13)	1.374 (6)	C(13)—C(14)	1.374 (5)
N(1)—C(15)	1.408 (4)	C(15)—C(16)	1.382 (5)
C(15)—C(20)	1.393 (5)	C(16)—C(17)	1.381 (6)
C(17)—C(18)	1.380 (7)	C(18)—C(19)	1.377 (7)
C(19)—C(20)	1.372 (6)	N(1)—H(n1)	0.94 (4)
N(2)—H(n2)	0.90 (4)	N(2)—H(n3)	0.82 (5)
O(1)...H(n1)	1.76 (4)	O(2)...H(n2)	1.89 (4)
C(1)—N(1)—C(15)	128.5 (3)	C(1)—N(1)—H(n1)	109 (2)
C(15)—N(1)—H(n1)	122 (2)	C(4)—N(2)—H(n2)	116 (3)
C(4)—N(2)—H(n3)	114 (4)	H(n2)—N(2)—H(n3)	129 (4)
N(1)—C(1)—C(2)	120.7 (3)	N(1)—C(1)—C(6)	121.1 (3)
C(2)—C(1)—C(6)	118.2 (3)	C(1)—C(2)—C(3)	121.4 (3)
C(2)—C(3)—C(4)	122.3 (3)	C(3)—C(4)—N(2)	117.9 (3)
C(3)—C(4)—C(5)	118.8 (3)	N(2)—C(4)—C(5)	123.3 (4)
C(4)—C(5)—C(6)	119.5 (3)	C(4)—C(5)—C(10)	119.7 (3)
C(6)—C(5)—C(10)	120.8 (3)	C(5)—C(6)—C(1)	119.7 (3)
C(5)—C(6)—C(7)	119.9 (3)	C(1)—C(6)—C(7)	120.4 (3)
C(6)—C(7)—O(1)	122.8 (3)	C(6)—C(7)—C(8)	118.7 (3)
O(1)—C(7)—C(8)	118.6 (3)	C(7)—C(8)—C(9)	120.9 (3)
C(7)—C(8)—C(14)	119.3 (3)	C(9)—C(8)—C(14)	119.8 (3)
C(8)—C(9)—C(10)	120.9 (3)	C(8)—C(9)—C(11)	119.4 (3)
C(10)—C(9)—C(11)	119.7 (3)	C(9)—C(10)—O(2)	118.6 (3)
C(9)—C(10)—C(5)	118.7 (3)	O(2)—C(10)—C(5)	122.7 (3)
C(9)—C(11)—C(12)	120.4 (4)	C(11)—C(12)—C(13)	120.1 (4)
C(12)—C(13)—C(14)	120.7 (4)	C(13)—C(14)—C(8)	119.6 (4)
N(1)—C(15)—C(16)	123.4 (3)	N(1)—C(15)—C(20)	116.9 (3)
C(16)—C(15)—C(20)	119.5 (4)	C(15)—C(16)—C(17)	119.7 (4)
C(16)—C(17)—C(18)	120.6 (5)	C(17)—C(18)—C(19)	119.7 (5)
C(18)—C(19)—C(20)	120.2 (5)	C(19)—C(20)—C(15)	120.3 (5)

distances and angles in Table 2,* the numbering scheme used is shown in Fig. 1 and the unit-cell contents in Fig. 2.

Related literature. The overall planarity of the anthraquinone molecule and the nature of the hydrogen-bonding contacts found in the title compound are as expected from previous studies on related amine-substituted anthraquinone systems: for example in 1,5-bis(phenylamino)anthraquinone and its 1,8-isomer

* Lists of structure factors, anisotropic thermal parameters, interatomic parameters involving H atoms and mean-plane data have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44158 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

(Bailey & Brown, 1967*a,b*); 1,4-bis(isopropylamino)-anthraquinone (Brown & Mitchell, 1982); 1-[2-(diethylamino)ethylamino]anthraquinone and the 1,5-bis derivative (Almond *et al.*, 1983); 1-[4-(dimethylamino)phenylamino]-4-hydroxyanthraquinone (Foitzik, Paulus & Haase, 1986*a*); and in 1,5-bis-[4-(pentylxy)phenylamino]anthraquinone (Foitzik, Paulus & Haase, 1986*b*).

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Structure of 6-Hydroperoxy-5-hydroxythymine Hemihydrate (I) and *trans*-5,6-Dihydroxy-1,3-dimethylthymine (II)*

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Abstract. (I) $C_5H_8N_2O_5 \cdot \frac{1}{2}H_2O$, $M_r = 185.1$, triclinic, $P\bar{1}$, $a = 6.125$ (2), $b = 10.849$ (3), $c = 11.674$ (3) Å, $\alpha = 98.21$ (2), $\beta = 91.38$ (2), $\gamma = 94.66$ (2)°, $V = 764.7$ (2) Å³, $Z = 4$, $D_x = 1.61$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu = 1.31$ mm⁻¹, $F(000) = 388$, $T = 295$ K, final $R = 0.047$, $wR = 0.061$ for 1952 observed data with $F_o > 3\sigma(F_o)$. (II) $C_7H_{12}N_2O_4$, $M_r = 188.2$, orthorhombic, $Pbca$, $a = 8.265$ (2), $b = 21.128$ (3), $c = 10.587$ (6) Å, $V = 1848.8$ (2) Å³, $Z = 8$, $D_x = 1.35$ Mg m⁻³, $\lambda(Cu K\alpha) = 1.54178$ Å, $\mu = 0.96$ mm⁻¹, $F(000) = 800$, $T = 296$ K, final $R = 0.066$, $wR = 0.067$ for 913 observed data with $F_o > 3\sigma(F_o)$. The thymine ring has a half-chair conformation in both (I) and (II). The hydroxyl group on C(5) is *cis* to the peroxy group in (I) and *trans* to the hydroxyl on C(6) in (II). (I) shows extensive hydrogen bonding (ten bonds ranging from 2.67 to 2.98 Å) involving all possible donors and several close intermolecular approaches which may explain, in part, the rather high density. In (II) only two hydrogen bonds are possible [O(5)⋯O(4) at 2.82 Å and O(6)⋯O(2) at 2.70 Å] and its density is that more normally found for molecules of this type.

* 5,6-Dihydro-6-hydroperoxy-5-hydroxy-5-methyl-2,4(1*H*,3*H*)-pyrimidinedione hemihydrate (I) and 5,6-dihydro-5,6-dihydroxy-1,3-dimethyl-2,4(1*H*,3*H*)-pyrimidinedione (II).

Experimental. Crystals of both (I) and (II) supplied by the late S. Y. Wang (formerly of Johns Hopkins University). Crystals used had dimensions 0.20 × 0.25 × 0.15 (I) and 0.30 × 0.25 × 0.08 (II). Automated Nicolet P3F diffractometer with incident-beam monochromator, 20 centered reflections with $8 < 2\theta < 60^\circ$ for (I) and 25 centered reflections with $8 < 2\theta < 70^\circ$ for (II) used for determining lattice parameters. Data corrected for Lorentz and polarization effects, absorption ignored. $[(\sin\theta)/\lambda]_{\max} = 0.547$ for both, range of hkl : $-6 \leq h \leq 0$, $-8 \leq k \leq 11$, $-12 \leq l \leq 12$ for (I); $-8 \leq h \leq 8$, $0 \leq k \leq 22$, $0 \leq l \leq 11$ for (II). For (I), standards 300, 004, 247 monitored every 60 reflections showed a linear decrease of 10% and were then used to scale the data, θ - 2θ scan technique ($2.0 + \Delta\alpha_1\alpha_2$), constant scan rate of 30° min⁻¹ (crystals were unstable). 2347 reflections measured, 1999 unique, 1952 considered observed [$|F_o| > 3\sigma(|F_o|)$] and used for refinement. For (II): standards, 400, 0,10,0, 0,0,14 monitored every 60 reflections showed a 2.5% random variation over data collection, θ - 2θ mode, same scan width as (I), scan rate a function of count rate (4° min⁻¹ minimum, 30° min⁻¹ maximum), 3014 reflections measured, 1154 unique, 913 considered observed [$|F_o| > 3\sigma(|F_o|)$] and used for refinement.