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2-Aminoanthraquinone

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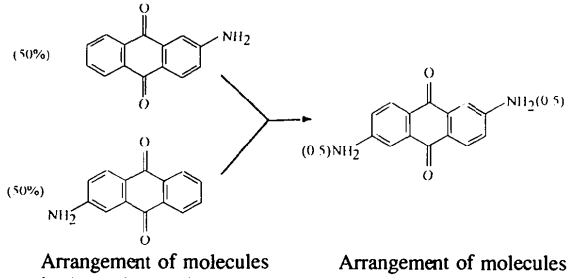
Abstract

The molecule of 2-aminoanthraquinone, $C_{14}H_9NO_2$, is nearly planar, with the non-H atoms exhibiting a mean distance of 0.022 Å from their best plane. The statistical disorder of the 2-aminoanthraquinone molecules is located around the centre of symmetry in space group $P2_1/c$. Weak intermolecular hydrogen bonds (N—H···N and N—H···O) link the molecules into a three-dimensional network.

Comment

This paper reports on the statistically disordered structure of 2-aminoanthraquinone. The planar molecules possess a centre of symmetry and have an occupancy factor of 0.5 for the randomly disordered NH_2 groups [atom H(2) is also disordered]. This accounts for the C—N bond distance of 1.222 (9) Å which is about 0.1 Å shorter than the C—N distances in other diaminoanthraquinone derivatives (Bailey & Brown, 1967*a*,*b*; Brown & Mitchell, 1982; Chippendalle, Mathias, Aujla, Harris, Packer & Say, 1983; Kashino, Senoo & Haisa, 1988). One type of molecular disorder is presented in the scheme below. The C—C and C=O bond distances are comparable to those observed in non-substituted anthraquinone (Lonsdale, Milledge & Sayed, 1966; Lonsdale, Walley & Sayed, 1966; Lonsdale, 1966; Murty, 1960; Prakash, 1967).



Arrangement of molecules in the real crystals — Arrangement of molecules resulting from X-ray analysis

(I)

The crystal structure consists of the two parallel sheets of planar 2-aminoanthraquinone molecules (Fig. 2). The distance between two successive parallel planes is 3.488 (6) Å, which is slightly longer than the van der Waals distance (3.4 Å) for aromatic C atoms (Pauling, 1960). The angle between the planes of two neighbouring sheets is 56.2 (5)°. The shortest intermolecular contacts between N and H, and O and H atoms are 2.36 (7)

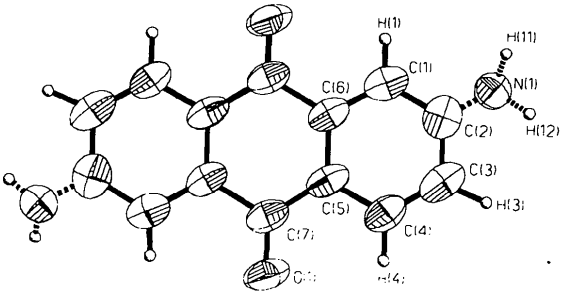


Fig. 1. View of the title compound showing the numbering scheme with displacement ellipsoids drawn at the 50% probability level. H atoms are drawn as circles of arbitrary radii. Atom H(2) has been omitted for clarity.

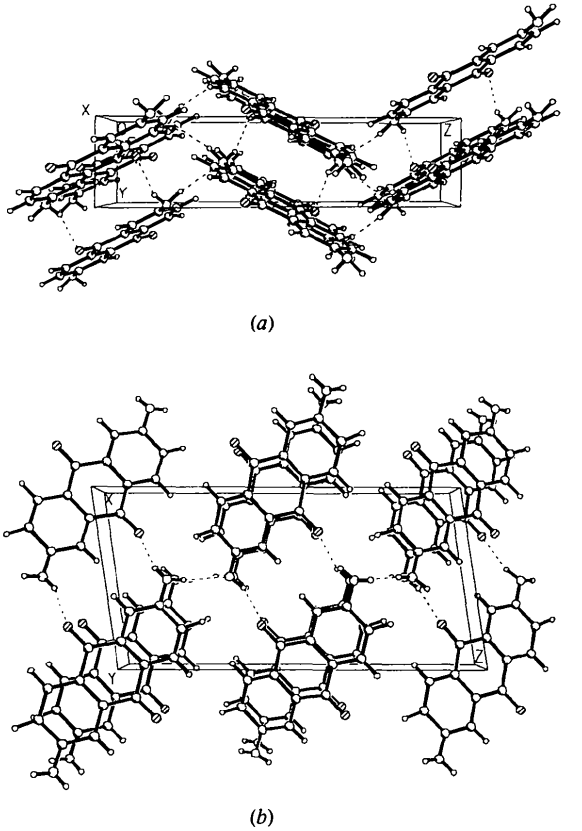


Fig. 2. Packing of the molecules in the unit cell shown by (*a*) a *bc* projection and (*b*) an *ac* projection.

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and 2.54 (7) Å, respectively (see dashed lines on Fig. 2). These values point to the existence of weak intermolecular interactions.

Experimental

The 2-aminoanthraquinone starting material was purchased from the Aldrich Chemical Company Inc. The red single crystals used for the X-ray measurements were obtained by heating the starting material at 473 K in an evacuated sealed glass ampoule for one day.

Crystal data

$C_{14}H_9NO_2$
$M_r = 223.2$
Monoclinic
$P2_1/c$
$a = 8.082\ (2)$ Å
$b = 3.954\ (1)$ Å
$c = 16.103\ (3)$ Å
$\beta = 98.54\ (3)°$
$V = 508.9$ Å^3
$Z = 2$
$D_x = 1.457$ Mg m^{-3}
$D_m = 1.45$ Mg m^{-3}
Cu $K\alpha$ radiation
$\lambda = 1.54184$ Å
Cell parameters from 20 reflections
$\theta = 20$–$35°$
$\mu = 0.761$ mm^{-1}
$T = 295$ K
Parallelepiped
0.50 × 0.35 × 0.30 mm
Red

Data collection

Kuma KM-4 computer-controlled four-circle κ-axis diffractometer
$\omega/2\theta$ scans
Absorption correction: none
1664 measured reflections
936 independent reflections
704 observed reflections [$F > 4\sigma(F)$]
$R_{int} = 0.0345$
$\theta_{max} = 75°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 5$
$l = -20 \rightarrow 20$
2 standard reflections monitored every 50 reflections
intensity decay: <1%

Refinement

Refinement on F
$R = 0.0616$
$wR = 0.0563$
$S = 0.55$
704 reflections
103 parameters
Weighting scheme based on measured e.s.d.'s
$(\Delta/\sigma)_{max} = 0.017$
$\Delta\rho_{max} = 0.15$ e Å^{-3}
$\Delta\rho_{min} = -0.26$ e Å^{-3}
Atomic scattering factors from *SHELXTL/PC* (Sheldrick, 1990)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
C(1)	0.3234 (6)	0.2508 (11)	0.5613 (2)	0.066 (2)
C(2)	0.3537 (6)	0.4057 (11)	0.6392 (3)	0.070 (2)
C(3)	0.2266 (7)	0.4433 (12)	0.6880 (3)	0.074 (2)
C(4)	0.0704 (6)	0.3259 (11)	0.6582 (2)	0.065 (2)
C(5)	0.0362 (5)	0.1671 (10)	0.5807 (3)	0.056 (1)
C(6)	0.1658 (5)	0.1303 (10)	0.5316 (2)	0.055 (1)
C(7)	−0.1330 (5)	0.0377 (11)	0.5517 (2)	0.060 (1)
O(1)	−0.2444 (4)	0.0648 (9)	0.5950 (2)	0.086 (1)
N(1)	0.4946 (10)	0.498 (2)	0.6688 (6)	0.067 (3)

Table 2. *Selected geometric parameters* (Å, °)

C(1)—C(2)	1.384 (6)	C(5)—C(6)	1.411 (6)
C(1)—C(6)	1.378 (6)	C(5)—C(7)	1.470 (5)
C(2)—C(3)	1.391 (7)	C(6)—C(7^i)	1.485 (5)
C(2)—N(1)	1.222 (9)	C(7)—O(1)	1.223 (5)
C(3)—C(4)	1.363 (7)	C(7)—C(6^i)	1.485 (5)
C(4)—C(5)	1.387 (5)		
C(2)—C(1)—C(6)	119.9 (4)	C(1)—C(2)—C(3)	120.9 (4)
C(1)—C(2)—N(1)	120.9 (6)	C(3)—C(2)—N(1)	118.1 (6)
C(2)—C(3)—C(4)	119.2 (4)	C(3)—C(4)—C(5)	121.4 (4)
C(4)—C(5)—C(6)	119.1 (4)	C(4)—C(5)—C(7)	119.7 (4)
C(6)—C(5)—C(7)	121.2 (3)	C(1)—C(6)—C(5)	119.6 (3)
C(1)—C(6)—C(7^i)	120.1 (4)	C(5)—C(6)—C(7^i)	120.3 (3)
C(5)—C(7)—O(1)	121.2 (3)	C(5)—C(7)—C(6^i)	118.4 (3)
O(1)—C(7)—C(6^i)	120.4 (4)		

Symmetry code: (i) $-x, -y, 1-z$.

Preliminary rotation and Weissenberg photographs indicated space group $P2_1/c$. The structure was solved by direct methods. Difference maps showed maxima in positions consistent with the expected locations of the H atoms. The positional and displacement parameters of the H atoms [except those of atoms H(2), H(11) and H(12), whose temperature factors were fixed at $U = 0.12$ Å^2] were refined.

Data collection, cell refinement and data reduction: Kuma KM-4 diffractometer software (Kuma, 1992). Structure solution and refinement, molecular graphics: *SHELXTL/PC* (Sheldrick, 1990).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: KA1101). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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