Supplementary data for this paper are available from the IUCr electronic archives (Reference: CF1257). Services for accessing these data are described at the back of the journal.

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7,8,10,11,12,13-Hexahydro-4-methylnaphtho[2,3-*c*]acridine

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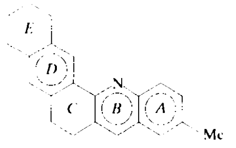
Abstract

In the title molecule, $C_{22}H_{21}N$, one of the two cyclohexane rings shows conformational disorder, with both major and minor conformers in half-chair conformations; the other cyclohexane ring adopts a conformation intermediate between sofa and half-chair.

Comment

The synthesis and biological evaluation of polycyclic azaarenes (PAA) with bay regions (Pullman & Pullman, 1955) have acquired great importance, due to their presence in the biosphere as serious environmental pollutants (Dong *et al.*, 1977; Schmeltz & Hoffman, 1977). These compounds reach the environment mainly from energy industries, automobile exhausts, cigarette smoke and the incomplete combustion of fossil fuels. Such PAA's and their oxidative metabolites have been reported to possess mutagenic and/or carcinogenic properties (Jerina, 1986; Wood *et al.*, 1989; Ray & Kar, 1993). Isolation of such compounds in sufficient amounts and in pure form from environmental sediments is difficult. In order to prepare sufficient material, Ray and his group have been working towards the synthesis and characterization of such compounds (Kar, Karmakar & Ray, 1989; Kar, Sami & Ray, 1992; Ray *et al.*, 1996). As a part of this programme, the crystal structure of the title compound, (I), was determined.



(I)

Bond lengths and angles in the title compound agree with those observed in a chloro-derivative of this structure (Ray *et al.*, 1995). One of the cyclohexane rings (*C*) present in the structure shows conformational disorder; both the major and minor conformers adopt half-chair conformations, but in opposite orientations. The conformation of the other cyclohexane ring (*E*) lies between sofa and half-chair, with asymmetry parameters $\Delta C_s(\mathrm{C10}) = 0.050\,(2)$ and $\Delta C_2(\mathrm{C10—C15}) = 0.054\,(1)$ (Nardelli, 1983). The mean plane through the major conformer of ring *C* forms dihedral angles of 6.60 (7) and 4.82 (7)°, respectively, with the planes through the rings *A*, *B* and *D*; the dihedral angle between rings *D* and

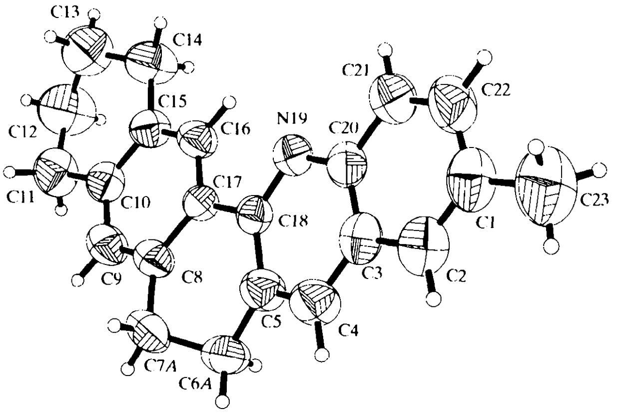


Fig. 1. The structure of the title compound, showing 50% probability displacement ellipsoids and the atom-numbering scheme. For clarity, only the major (80%) conformer of ring *C* is shown.

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E is 4.01 (8)°. In the crystal, the inversion-related molecules form a pair, with C4···C16($-x + 1$, $-y$, $-z + 1$) [3.397 (4) Å] being the shortest contact, and the glide-related pairs are packed at angle of 60.79°.

Experimental

The title compound was synthesized in 95–96% yield, by thermolysis (513–543 K for 3–4 min) of 3,4,6,7,8,9-hexahydro-1-(*p*-tolyliminomethyl)anthracene hydrochloride, which in turn was obtained by reaction of two equivalents of *p*-toluidine with 1-chloro-3,4,6,7,8,9-hexahydroanthracene-2-aldehyde. The compound (m.p. 415–416 K) was first purified by column chromatography [neutral alumina/petroleum ether (333–353 K)], and then single crystals were obtained by slow evaporation from solution in a mixture of petroleum ether (333–353 K) and chloroform.

Crystal data

$C_{22}H_{21}N$
$M_r = 299.40$
Monoclinic
$P2_1/c$
$a = 12.2516\,(8)$ Å
$b = 13.2439\,(9)$ Å
$c = 10.1913\,(7)$ Å
$\beta = 90.291\,(2)°$
$V = 1653.61\,(19)$ Å^3
$Z = 4$
$D_x = 1.203$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 2875 reflections
$\theta = 3.02$–$28.5°$
$\mu = 0.069$ mm^{-1}
$T = 293\,(2)$ K
Block
0.50 × 0.26 × 0.20 mm
Light yellow

Data collection

Siemens SMART CCD area-detector diffractometer
ω scans
Absorption correction: none
11 105 measured reflections
4159 independent reflections
1839 reflections with $I > 2\sigma(I)$

$R_{\rm int} = 0.045$
$\theta_{\rm max} = 28.5°$
$h = -16 \rightarrow 16$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 13$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.074$
$wR(F^2) = 0.241$
$S = 1.021$
4159 reflections
227 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0964P)^2 + 0.318P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.30$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.21$ e Å^{-3}
Extinction correction: *SHELXTL* (Sheldrick, 1996)
Extinction coefficient: 0.006 (3)
Scattering factors from *International Tables for Crystallography* (Vol. C)

The data collection covered more than a hemisphere of reciprocal space by a combination of three sets of exposures; each set had a different φ angle (0, 88 and 180°) for the crystal and each exposure of 30 s covered 0.3° in ω. The crystal-to-detector distance was 4 cm and the detector swing angle was −35°. Coverage of the unique set is over 99% complete. Crystal decay was monitored by repeating 30 initial frames at the end of data collection and analysing the duplicate reflections, and was found to be negligible. The structure was solved by direct methods and refined by full-matrix least-squares techniques. Atoms C6 and C7 of the cyclohexane ring (*C*) were found to be disordered. The occupancies of the disordered positions, C6*A*, C6*B*, C7*A* and C7*B*, were initially refined and later fixed at 80/20. The corresponding C—C distances in the major and minor conformers were restrained to be equal, with an effective standard deviation of 0.01 Å. The C12—C13 distance is shorter than expected, at 1.466 (5) Å. This may be due to incipient disorder, but no valid model could be developed. The anisotropic displacement parameters of the disordered positions were also restrained to be equal, with an effective standard deviation of 0.01 Å^2. Due to the low *r*/*p* ratio of observed reflections, all H atoms were geometrically fixed and allowed to ride on the atoms to which they are attached, apart from those on C23, which were refined as part of a rigid group. We attribute the somewhat elevated final *R* index to two factors, namely, the poor diffraction quality of the crystal [only some 44% of the reflections had $I > 2\sigma(I)$], and the presence of disorder. Outside the disordered region, standard uncertainties on C—C distances do not exceed 0.05 Å.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Sheldrick, 1996). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL* and *PARST* (Nardelli, 1995).

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2-Aminopyrimidine and *p*-phenylenediacetic acid (1:1) co-crystal

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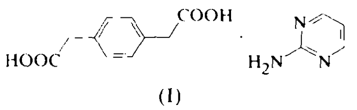
Abstract

In the title co-crystal, each 2-aminopyrimidine molecule, $C_4H_5N_3$, participates in two eight-membered hydrogen-bonded rings with carboxylic acid groups from two different phenylenediacetic acid molecules, $C_{10}H_{10}O_4$. This results in infinite hydrogen-bonded chains. In the supramolecular structure, the chains are held together by weak C—H···O contacts.

Comment

Hydrogen bonds are used extensively as a tool to design the structure of molecular crystals, because of their strength, as well as their directional nature, compared to other intermolecular non-covalent interactions (Lehn, 1995). Most of the supramolecular crystals originate from strong N—H···*X* and O—H···*X* (*X* = O, N) hydrogen bonds. Weak C—H···O bonds are known to play a significant role in determining the molecular packing of organic solids (Taylor & Kennard, 1982). 2-Aminopyrimidine forms heterodimeric structures with different mono- and dicarboxylic acids (Etter & Adsmond, 1990; Etter *et al.*, 1990), rather than their individual self assembly (Scheinbeim *et al.*, 1976). We have also recently shown (Goswami *et al.*, 1999) that terephthalic acid forms chain-like heteroassemblies with 2-aminopyrimidine, similar to those formed by 2-aminopyrimidine and succinic acid (Etter *et al.*, 1990). In this paper, we report the supramolecular structure of the 2-aminopyrimidine–*p*-phenylenediacetic acid co-crystal, (I), *via* C—H···O hydrogen bonding.



(I)

The asymmetric unit consists of one half-molecule each of 2-aminopyrimidine and *p*-phenylenediacetic acid; the 2-aminopyrimidine lies on a crystallographic twofold axis passing through atoms N2, C6 and C8, and the *p*-phenylenediacetic acid is on an inversion centre.

Bond lengths and angles in the 2-aminopyrimidine agree with other reported values (Byriel *et al.*, 1992; Lynch *et al.*, 1994). The carboxylic acid group (O1, O2, C4 and C5) makes dihedral angles of 77.6 (1) and 2.5 (1)° with the planes of the phenyl ring and the 2-aminopyrimidine moiety, respectively. The planar 2-aminopyrimidine is linked to the *p*-phenylenediacetic acid by N2—H1N2···O1 and O2—H1O2···N1 intermolecular hydrogen bonds; along with C5 and C6, they form an eight-membered ring, which is coplanar with the pyrimidine moiety. Since both molecules lie across crystallographic symmetry elements, the hydrogen-bond system is extended to form an infinite chain-like structure in the solid state. Neighbouring chains along the *a* axis are linked by weak C4—H4*B*···O1^{i} contacts to form a supramolecular assembly (Fig. 2), in which the pyrimidine and phenyl rings are stacked with perpendicular separations of 3.605 (4) and 3.614 (4) Å, respectively [symmetry code: (i) $x - 1, y, z$].

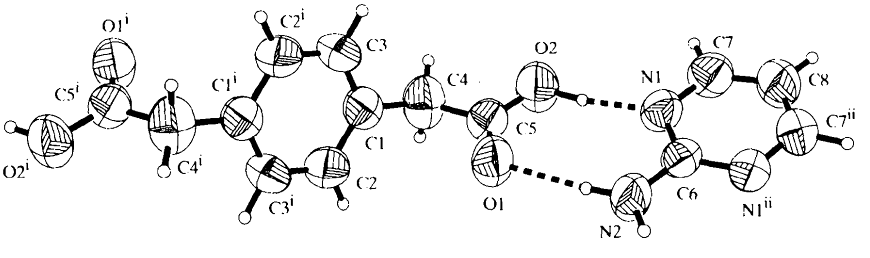


Fig. 1. The structure of the title co-crystal, showing 50% probability displacement ellipsoids and the atom-numbering scheme [symmetry codes: (i) $-x, 1 - y, 1 - z$; (ii) $1 - x, y, \frac{3}{2} - z$].

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