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3-Amino-5,5-dimethyl-2-phenylcyclohex-2-en-1-one

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Key indicators

Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.005 Å
R factor = 0.052
wR factor = 0.147
Data-to-parameter ratio = 11.1

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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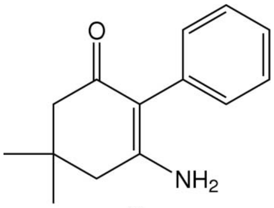
In the title compound, $C_{14}H_{17}NO$, there are two crystallographically independent molecules in the asymmetric unit. The cyclohexene rings adopt an envelope conformation. Adjacent molecules are linked *via* N—H···O hydrogen bonds to form dimers and these form one-dimensional chains along the [20$\overline{1}$] direction. These chains are interconnected by weak intermolecular C—H···O interactions. N—H···π interactions are also observed in the crystal structure.

Comment

Arynes used as reagents in synthetic organic chemistry have received much attention. There has recently been a renewed interest in exploring new benzyne reactions after Kobayashi described a mild method for the *in situ* preparation of benzyne at moderate temperatures that exploits the fluoride-induced elimination of *ortho*-silyl aryl triflates (Himeshima *et al.*, 1983). Here we report the crystal structure of the title compound, (I), which was obtained by the reaction of benzyne with the cyclic enaminone 3-amino-5,5-dimethylcyclohex-2-enone.



(I)

The asymmetric unit of (I) contains two molecules, *A* and *B*, in which the orientation of the benzene ring with respect to the cyclohexene ring is different (Fig. 1). The corresponding bond lengths and angles of these two molecules agree with each other and show normal values (Allen *et al.*, 1987). In both molecules *A* and *B*, the cyclohexene rings adopt an envelope conformation, with puckering parameters (Cremer & Pople 1975) Q = 0.450 (4) Å, θ = 52.3 (4)° and φ = 117.9 (6)° in molecule *A* [Q = 0.452 (4) Å, θ = 129.1 (5)° and φ = 297.1 (6)° in molecule *B*] and atom C3 having the maximum deviation of 0.318 (4) Å in molecule *A* [0.319 (4) Å in molecule *B*] from the mean plane of C1/C2/C4–C6. The C7–C12 benzene rings are oriented at angles of 73.63 (14)° in molecule *A* [79.56 (12)° in molecule *B*] with respect to the cyclohexene ring. The dihedral angle between the C7–C12 benzene ring and the mean plane of C1/C2/C4–C7/N1/O1 is 73.63 (14)° in molecule *A* [79.56 (12)° in molecule *B*]. The dihedral angle between the two benzene rings of molecules *A* and *B* is 39.86 (15)°.

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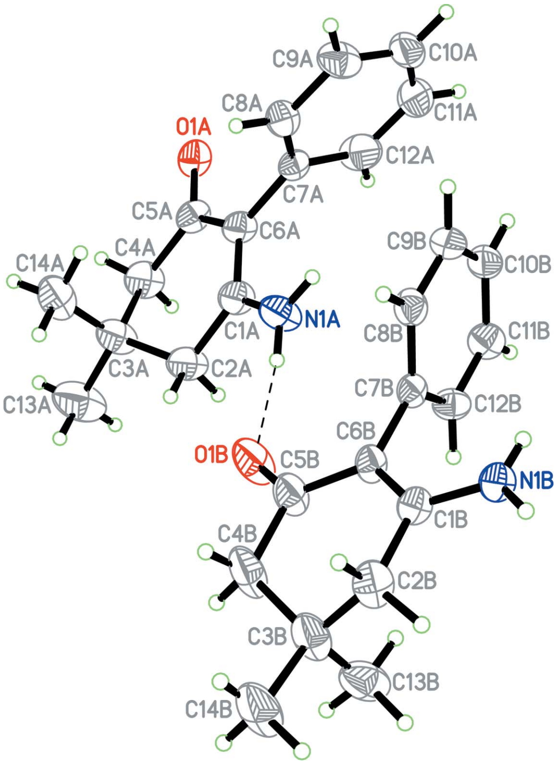

Figure 1
The asymmetric unit of (I), showing 60% probability displacement ellipsoids and the atomic numbering. The dashed line indicates a hydrogen bond.

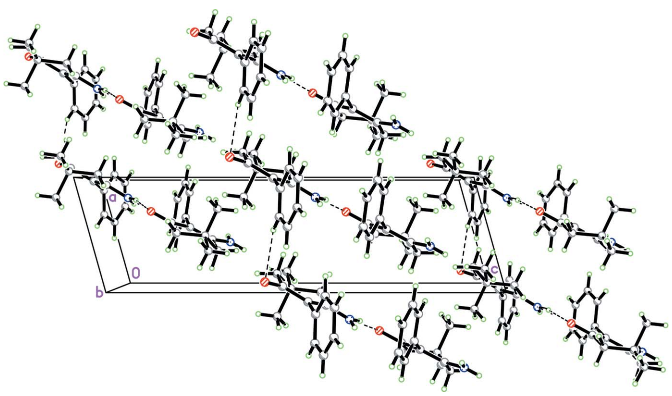

Figure 2
The crystal packing of (I), viewed down the *b* axis. Hydrogen bonds are shown as dashed lines.

In the crystal the two unique molecules in the asymmetric unit are linked into dimers by N—H···O hydrogen bonds (Table 1) and these dimers are arranged into one-dimensional chains along the [$20\overline{1}$] direction. The chains are interconnected by weak C—H···O interactions (Fig. 2). N—H ···π interactions were also observed (Table 2); *Cg*1 is the centroid of the C7*B*–C12*B* benzene ring.

Experimental

Compound (I) was prepared by the reaction of 3-amino-5,5-dimethylcyclohex-2-enone (43.2 µl, 0.4 mmol, 1.0 equiv.) with 2-(trimethylsilyl)phenyl trifluoromethanesulfonate (121.4 µl, 0.5 mmol, 1.25 equiv.) in the presence of caesium fluoride (152 mg, 1.0 mmol, 2.5 equiv.). The title compound was isolated using silica gel column chromatography using petroleum ether (b.p. 333–363 K) and ethyl acetate as eluents. Pale-yellow single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvent from a petroleum ether/ethyl acetate solution (3:1 *v*/*v*), m.p. 443–444 K.

Crystal data

$C_{14}H_{17}NO$
M_r = 215.29
Monoclinic, *Pc*
a = 6.1044 (1) Å
b = 10.1495 (2) Å
c = 20.4177 (4) Å
β = 105.691 (1)°
V = 1217.87 (4) Å^3
Z = 4
D_x = 1.174 Mg m^{-3}
Mo *K*α radiation
μ = 0.07 mm^{-1}
T = 100.0 (1) K
Block, pale yellow
0.60 × 0.20 × 0.12 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.957, T_{max} = 0.991
20888 measured reflections
3248 independent reflections
2641 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 29.0°

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.147
S = 1.06
3248 reflections
293 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0821P)^2 + 0.1433P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.33 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1*A*—H2NA···O1*B*	0.86	1.99	2.839 (4)	169
N1*B*—H2NB···O1*A*^{i}	0.86	2.03	2.877 (3)	171
C8*A*—H8*AA*···O1*A*^{ii}	0.93	2.51	3.296 (4)	143
N1*A*—H1NA···*Cg*1	0.86	2.61	3.111 (3)	118

Symmetry codes: (i) $x-1, -y+1, z+\frac{1}{2}$; (ii) $x-1, y, z$.

All H atoms were positioned geometrically and refined using a riding model with C—H = 0.93 Å, U_{iso}(H) =1.2U_{eq}(C) for aromatic, C—H = 0.97 Å, U_{iso}(H) = 1.2U_{eq}(C) for CH_2, C—H = 0.96 Å, U_{iso}(H) = 1.5U_{eq}(C) for CH_3 and N—H = 0.86 Å, U_{iso}(H) = 1.2U_{eq}(N) for NH atoms. A rotating group model was used for the methyl groups. A total of 2769 Friedel pairs were merged before final refinement as there is no significant anomalous dispersion for the determination of the absolute structure.

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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