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3-Anilino-5,5-dimethylcyclohex-2-enone

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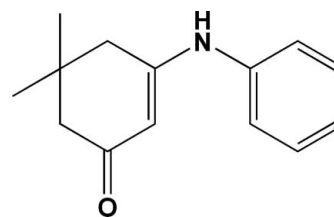
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Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.001$ Å; R factor = 0.047; wR factor = 0.138; data-to-parameter ratio = 34.2.

In the title molecule, $\text{C}_{14}\text{H}_{17}\text{NO}$, the 5,5-dimethylcyclohex-2-enone moiety is attached to an aniline group, the dihedral angle subtended [$54.43(3)^\circ$] indicating a significant twist. The hexaneone ring has a half-chair conformation with the C atom bearing two methyl groups lying $0.6384(8)$ Å above the plane of the five remaining atoms (r.m.s. deviation = 0.0107 Å). The crystal packing can be described as alternating layers parallel to $(\bar{1}01)$, which are consolidated by $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds and $\text{C}-\text{H}\cdots\pi$ interactions.

Related literature

For the synthesis of the title compound, see: Amini *et al.* (2013); Machacek *et al.* (2002). For its reactivity, see: Wang *et al.* (2007); Mohammadzadeh *et al.* (2009); Gao *et al.* (2008). For our previous work [inspired by Assy (1996)] on the preparation and the reactivity of imidazole derivatives, see: Zama *et al.* (2013a,b); Chelghoum *et al.* (2011); Bahnous *et al.* (2012) For enamine derivatives as precursors in the synthesis of compounds of pharmaceutical interest, see: Palko *et al.* (2008) Park & Jahng (1998); Tadesse *et al.* (1999); Thummel & Jahng (1985); When enamines are treated with alkyl halides, an alkylation occurs to give an iminium salt, see: Adams (2000); Kempf *et al.* (2003).



Experimental

Crystal data

$\text{C}_{14}\text{H}_{17}\text{NO}$
 $M_r = 215.29$
Monoclinic, $P2_1/c$
 $a = 10.1766(19)$ Å
 $b = 13.159(2)$ Å
 $c = 9.2877(17)$ Å
 $\beta = 104.062(7)^\circ$

$V = 1206.5(4)$ Å³
 $Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.07$ mm⁻¹
 $T = 150$ K
 $0.15 \times 0.12 \times 0.09$ mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan
(SADABS; Sheldrick, 2002)
 $T_{\min} = 0.667$, $T_{\max} = 0.747$

12403 measured reflections
5021 independent reflections
3873 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.138$
 $S = 1.04$
5021 reflections

147 parameters
H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.45$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.20$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

Cg1 is the centroid of C10–C15 ring.

| $D-\text{H}\cdots A$ | $D-\text{H}$ | $\text{H}\cdots A$ | $D\cdots A$ | $D-\text{H}\cdots A$ |
|--|--------------|--------------------|-------------|----------------------|
| $\text{N1}-\text{H1}\cdots\text{O1}^{\text{i}}$ | 0.86 | 2.02 | 2.8587 (11) | 165 |
| $\text{C8}-\text{H8A}\cdots\text{Cg1}^{\text{ii}}$ | 0.96 | 2.61 | 3.5547 (12) | 157 |

Symmetry codes: (i) $-x + 1, y + \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 1, -y + 2, -z + 1$.

Data collection: APEX2 (Bruker, 2001); cell refinement: SAINT (Bruker, 2001); data reduction: SAINT; program(s) used to solve structure: SIR2002 (Burla *et al.*, 2005); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 2012) and DIAMOND (Brandenburg & Berndt, 2001); software used to prepare material for publication: WinGX (Farrugia, 2012) and CRYSCAL (T. Roisnel, local program).

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Supporting information for this paper is available from the IUCr electronic archives (Reference: BQ2392).

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supporting information

Acta Cryst. (2014). E70, o233–o234 [doi:10.1107/S160053681400186X]

3-Anilino-5,5-dimethylcyclohex-2-enone

Housseem Boulebd, Abdelmalek Bouraiou, Sofiane Bouacida, Hocine Merazig and Ali Belfaitah

S1. Comment

The reaction of primary amines with ketones leads to imines; however, secondary amines give enamines. The preparation of enamines takes place when an aldehyde or ketone containing an α hydrogen is treated with a secondary amine. When enamines are treated with alkyl halides, an alkylation occurs to give an iminium salt *via* an electron transfer from the electron pair on nitrogen, through the C=C to the electrophilic carbon of the alkyl halide (Adams, 2000). In fact, an enamine behaves as a "nitrogen enolate" and generally react as carbon nucleophiles (Kempf *et al.*, 2003). The use of enamine derivatives as intermediates in organic synthesis has been extensively investigated and they proved to be versatile precursors in the synthesis of a large variety of compounds of pharmaceutical interest (Tadesse *et al.*, 1999; Park *et al.*, 1998; Thummel & Jahng 1985; Palko *et al.*, 2008). Inspired by the works of Assy 1996 and also as an extension of our ongoing research on the preparation and the reactivity of imidazole derivatives (Zama *et al.*, 2013a; Zama *et al.*, 2013b; Chelghoum *et al.*, 2011; Bahnous *et al.*, 2012), we describe herein the single-crystal X-ray structure of the enamine 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone (I). This latter has been recovered from our attempt to coupling the dihydropyridine entity with imidazole unit using a "One Pot reaction" strategy implicating aniline, 5,5-dimethyl-1,3-cyclohexandione and *N*-methylimidazolmethylenemalononitrile. The molecular geometry and the atom-numbering scheme of (I) are shown in Fig. 1. The asymmetric unit of (I) consists of the 5,5-dimethyl-cyclohex-2-enone moiety and its attached phenylamino group. The crystal packing can be described as alternating layers parallel to the (-101)(Fig. 2). It is stabilized by N—H \cdots O hydrogen bond (Fig.3) and C—H \cdots π interactions (Table. 1). These interaction bonds link the molecules within the layers and also link the layers together, reinforcing the cohesion of the structure.

S2. Experimental

5,5-dimethyl-3-(phenylamino)cyclohex-2-enone (I) has been obtained from the reaction of aniline and 5,5-dimethyl-1,3-cyclohexandione. In a typical reaction, 1 mmol of 5,5-dimethylcyclohex-1,3-dione, and 1 mmol aniline, in ethanol were placed in a 10 ml round-bottomed flask fitted with a condenser and a magnetic stirrer bar. The reaction mixture was stirred at reflux for 24 h, then 1 mmol of *N*-methylimidazolmethylenemalononitrile was added to this solution and the reaction mixture was heated for an additional 24 h. The solvent was distilled off and flash chromatographic purification furnished the 5,5-dimethyl-3-(phenylamino)cyclohex-2-enone I and the recovered *N*-methylimidazolmethylenemalononitrile. Suitable crystals for X-ray experiments of I were obtained by slow evaporation from an ethanol/CH₂Cl₂ solution at room temperature.

S3. Refinement

Approximate positions for all the H atoms were first obtained from the difference electron density map. However, the H atoms were situated into idealized positions and the H-atoms have been refined within the riding atom approximation. The applied constraints were as follow: C_{aryl}—H_{aryl} = 0.93 Å; C_{methylene}—H_{methylene} = 0.97 Å; C_{methyl}—H_{methyl} = 0.96 Å and N

—H = 0.86 Å; The idealized methyl group was allowed to rotate about the C—C bond during the refinement by application of the command AFIX 137 in *SHELXL97* (Sheldrick, 2008). $U_{\text{iso}}(\text{H}_{\text{methyl}}) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ or $U_{\text{iso}}(\text{H}_{\text{aryl, methylene, amine}}) = 1.2 U_{\text{eq}}(\text{C}_{\text{aryl, methylene or N}})$.

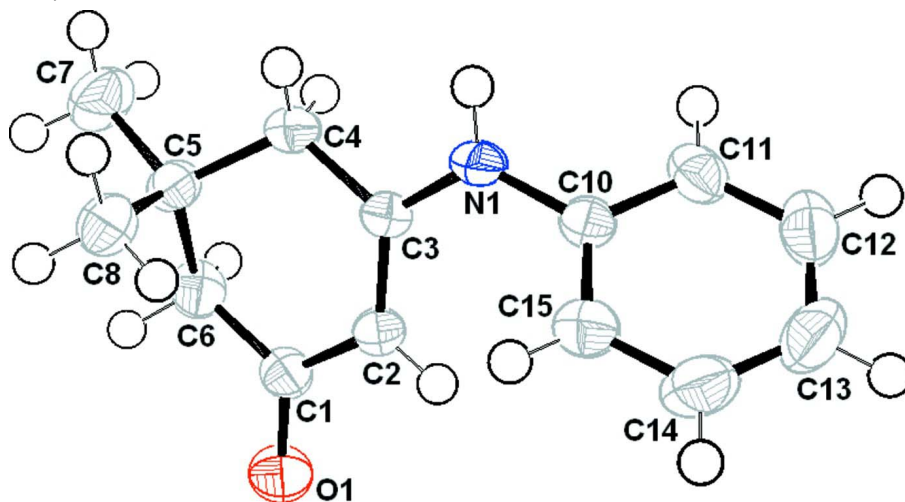


Figure 1

(Farrugia, 2012) The molecular geometry of (I) with the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as small spheres of arbitrary radius.

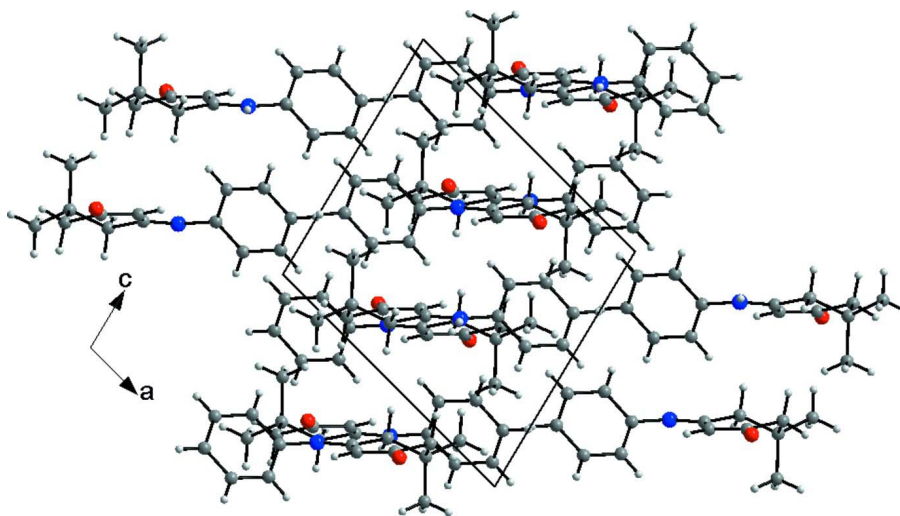


Figure 2

(Brandenburg & Berndt, 2001) Alternating layers parallel to (-101) planes of (I) viewed down the *b* axis.

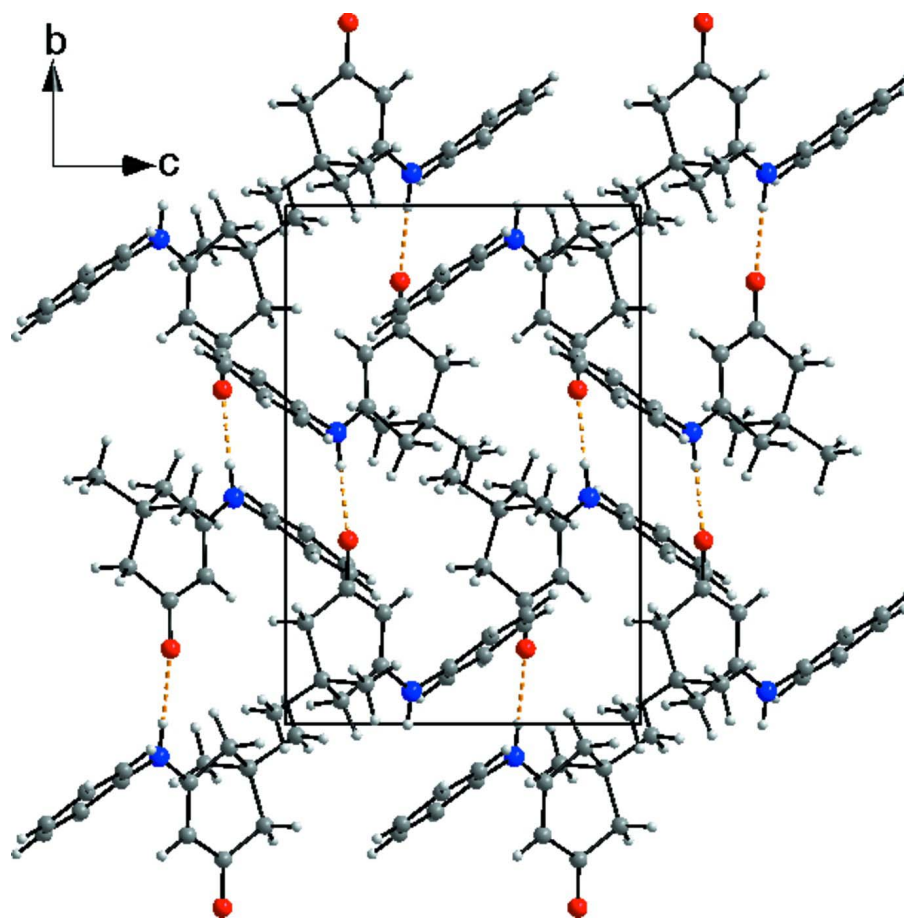


Figure 3

(Brandenburg & Berndt, 2001) A diagram of the layered crystal packing of (I) viewed down the a axis showing hydrogen bond as dashed line.

3-Anilino-5,5-dimethylcyclohex-2-enone

Crystal data

$C_{14}H_{17}NO$

$M_r = 215.29$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2_1/c$

$a = 10.1766$ (19) Å

$b = 13.159$ (2) Å

$c = 9.2877$ (17) Å

$\beta = 104.062$ (7)°

$V = 1206.5$ (4) Å³

$Z = 4$

$F(000) = 464$

$D_x = 1.185$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 4703 reflections

$\theta = 2.6$ – 34.3 °

$\mu = 0.07$ mm⁻¹

$T = 150$ K

Prism, colorless

$0.15 \times 0.12 \times 0.09$ mm

Data collection

Bruker APEXII

diffractometer

Graphite monochromator

CCD rotation images, thin slices scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 2002)

$T_{\min} = 0.667$, $T_{\max} = 0.747$

12403 measured reflections

5021 independent reflections

3873 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.030$
 $\theta_{\text{max}} = 34.3^\circ$, $\theta_{\text{min}} = 2.6^\circ$

$h = -16 \rightarrow 14$
 $k = -13 \rightarrow 20$
 $l = -12 \rightarrow 14$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.047$
 $wR(F^2) = 0.138$
 $S = 1.04$
 5021 reflections
 147 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: inferred from
 neighbouring sites
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0725P)^2 + 0.1791P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.45 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.20 \text{ e } \text{\AA}^{-3}$

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) *etc.* and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ |
|-----|--------------|-------------|--------------|----------------------------------|
| C1 | 0.64385 (8) | 0.73838 (6) | 0.82567 (9) | 0.01988 (15) |
| C2 | 0.53143 (8) | 0.78529 (6) | 0.72449 (9) | 0.01973 (15) |
| H2 | 0.4742 | 0.7455 | 0.6531 | 0.024* |
| C3 | 0.50602 (8) | 0.88768 (6) | 0.73022 (9) | 0.01767 (14) |
| C4 | 0.59803 (8) | 0.95491 (6) | 0.84239 (10) | 0.02194 (16) |
| H4A | 0.5593 | 0.963 | 0.9274 | 0.026* |
| H4B | 0.6012 | 1.0216 | 0.7987 | 0.026* |
| C5 | 0.74309 (8) | 0.91494 (6) | 0.89647 (9) | 0.01943 (15) |
| C6 | 0.73478 (9) | 0.80376 (6) | 0.94261 (9) | 0.02232 (16) |
| H6A | 0.8253 | 0.775 | 0.9661 | 0.027* |
| H6B | 0.7019 | 0.8017 | 1.0322 | 0.027* |
| C7 | 0.81684 (10) | 0.97776 (8) | 1.03068 (11) | 0.0320 (2) |
| H7A | 0.7703 | 0.9715 | 1.1086 | 0.048* |
| H7B | 0.8185 | 1.0478 | 1.0024 | 0.048* |
| H7C | 0.908 | 0.9533 | 1.0652 | 0.048* |
| C8 | 0.81959 (9) | 0.92285 (7) | 0.77337 (10) | 0.02490 (17) |
| H8A | 0.8268 | 0.9929 | 0.7477 | 0.037* |
| H8B | 0.7712 | 0.886 | 0.6876 | 0.037* |
| H8C | 0.9086 | 0.8945 | 0.8079 | 0.037* |
| C10 | 0.29024 (8) | 0.89121 (6) | 0.53767 (9) | 0.01887 (15) |
| C11 | 0.15721 (8) | 0.90959 (7) | 0.54586 (10) | 0.02316 (16) |
| H11 | 0.1403 | 0.95 | 0.6215 | 0.028* |

| | | | | |
|-----|--------------|-------------|--------------|--------------|
| C12 | 0.04984 (9) | 0.86746 (7) | 0.44081 (12) | 0.0306 (2) |
| H12 | −0.0388 | 0.88 | 0.4461 | 0.037* |
| C13 | 0.07470 (11) | 0.80686 (7) | 0.32830 (13) | 0.0351 (2) |
| H13 | 0.003 | 0.7781 | 0.2587 | 0.042* |
| C14 | 0.20729 (11) | 0.78931 (8) | 0.31997 (12) | 0.0343 (2) |
| H14 | 0.2239 | 0.7488 | 0.2443 | 0.041* |
| C15 | 0.31524 (9) | 0.83168 (7) | 0.42365 (10) | 0.02586 (18) |
| H15 | 0.4037 | 0.8203 | 0.4168 | 0.031* |
| N1 | 0.39813 (7) | 0.93664 (5) | 0.64459 (8) | 0.02126 (14) |
| H1 | 0.3945 | 1.0014 | 0.6559 | 0.026* |
| O1 | 0.66676 (7) | 0.64544 (5) | 0.82303 (9) | 0.03086 (17) |

Atomic displacement parameters (Å²)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|------------|------------|------------|-------------|-------------|-------------|
| C1 | 0.0190 (3) | 0.0142 (3) | 0.0280 (4) | −0.0002 (2) | 0.0086 (3) | 0.0044 (3) |
| C2 | 0.0182 (3) | 0.0134 (3) | 0.0268 (4) | −0.0001 (2) | 0.0040 (3) | −0.0003 (3) |
| C3 | 0.0170 (3) | 0.0144 (3) | 0.0217 (3) | 0.0010 (2) | 0.0048 (3) | −0.0012 (2) |
| C4 | 0.0214 (3) | 0.0170 (3) | 0.0261 (4) | 0.0017 (3) | 0.0032 (3) | −0.0060 (3) |
| C5 | 0.0198 (3) | 0.0172 (3) | 0.0202 (3) | −0.0011 (3) | 0.0027 (3) | −0.0009 (3) |
| C6 | 0.0225 (3) | 0.0210 (4) | 0.0226 (3) | 0.0010 (3) | 0.0036 (3) | 0.0053 (3) |
| C7 | 0.0320 (4) | 0.0310 (5) | 0.0280 (4) | −0.0039 (4) | −0.0024 (4) | −0.0070 (4) |
| C8 | 0.0245 (4) | 0.0214 (4) | 0.0300 (4) | −0.0041 (3) | 0.0092 (3) | 0.0014 (3) |
| C10 | 0.0191 (3) | 0.0144 (3) | 0.0221 (3) | 0.0013 (2) | 0.0031 (3) | 0.0013 (2) |
| C11 | 0.0212 (3) | 0.0206 (3) | 0.0278 (4) | 0.0043 (3) | 0.0061 (3) | 0.0046 (3) |
| C12 | 0.0201 (4) | 0.0254 (4) | 0.0437 (5) | −0.0011 (3) | 0.0025 (3) | 0.0103 (4) |
| C13 | 0.0317 (5) | 0.0228 (4) | 0.0420 (5) | −0.0068 (3) | −0.0080 (4) | 0.0015 (4) |
| C14 | 0.0397 (5) | 0.0256 (4) | 0.0328 (5) | 0.0008 (4) | −0.0004 (4) | −0.0097 (4) |
| C15 | 0.0254 (4) | 0.0237 (4) | 0.0278 (4) | 0.0026 (3) | 0.0052 (3) | −0.0053 (3) |
| N1 | 0.0210 (3) | 0.0135 (3) | 0.0266 (3) | 0.0034 (2) | 0.0007 (3) | −0.0026 (2) |
| O1 | 0.0290 (3) | 0.0135 (3) | 0.0487 (4) | 0.0018 (2) | 0.0067 (3) | 0.0065 (3) |

Geometric parameters (Å, °)

| | | | |
|--------|-------------|---------|-------------|
| C1—O1 | 1.2463 (10) | C7—H7C | 0.96 |
| C1—C2 | 1.4320 (11) | C8—H8A | 0.96 |
| C1—C6 | 1.5126 (12) | C8—H8B | 0.96 |
| C2—C3 | 1.3754 (11) | C8—H8C | 0.96 |
| C2—H2 | 0.93 | C10—C15 | 1.3895 (12) |
| C3—N1 | 1.3523 (10) | C10—C11 | 1.3954 (12) |
| C3—C4 | 1.5069 (11) | C10—N1 | 1.4209 (10) |
| C4—C5 | 1.5323 (12) | C11—C12 | 1.3911 (13) |
| C4—H4A | 0.97 | C11—H11 | 0.93 |
| C4—H4B | 0.97 | C12—C13 | 1.3857 (16) |
| C5—C7 | 1.5316 (12) | C12—H12 | 0.93 |
| C5—C6 | 1.5326 (12) | C13—C14 | 1.3895 (16) |
| C5—C8 | 1.5351 (12) | C13—H13 | 0.93 |
| C6—H6A | 0.97 | C14—C15 | 1.3896 (13) |

| | | | |
|-------------|-------------|-----------------|-------------|
| C6—H6B | 0.97 | C14—H14 | 0.93 |
| C7—H7A | 0.96 | C15—H15 | 0.93 |
| C7—H7B | 0.96 | N1—H1 | 0.86 |
| O1—C1—C2 | 122.25 (8) | C5—C7—H7C | 109.5 |
| O1—C1—C6 | 119.18 (7) | H7A—C7—H7C | 109.5 |
| C2—C1—C6 | 118.54 (7) | H7B—C7—H7C | 109.5 |
| C3—C2—C1 | 121.65 (7) | C5—C8—H8A | 109.5 |
| C3—C2—H2 | 119.2 | C5—C8—H8B | 109.5 |
| C1—C2—H2 | 119.2 | H8A—C8—H8B | 109.5 |
| N1—C3—C2 | 125.26 (7) | C5—C8—H8C | 109.5 |
| N1—C3—C4 | 113.97 (7) | H8A—C8—H8C | 109.5 |
| C2—C3—C4 | 120.72 (7) | H8B—C8—H8C | 109.5 |
| C3—C4—C5 | 114.36 (7) | C15—C10—C11 | 119.93 (8) |
| C3—C4—H4A | 108.7 | C15—C10—N1 | 121.09 (7) |
| C5—C4—H4A | 108.7 | C11—C10—N1 | 118.95 (8) |
| C3—C4—H4B | 108.7 | C12—C11—C10 | 119.99 (9) |
| C5—C4—H4B | 108.7 | C12—C11—H11 | 120 |
| H4A—C4—H4B | 107.6 | C10—C11—H11 | 120 |
| C7—C5—C4 | 108.91 (7) | C13—C12—C11 | 120.13 (9) |
| C7—C5—C6 | 109.70 (7) | C13—C12—H12 | 119.9 |
| C4—C5—C6 | 107.77 (7) | C11—C12—H12 | 119.9 |
| C7—C5—C8 | 109.43 (7) | C12—C13—C14 | 119.69 (9) |
| C4—C5—C8 | 110.80 (7) | C12—C13—H13 | 120.2 |
| C6—C5—C8 | 110.20 (7) | C14—C13—H13 | 120.2 |
| C1—C6—C5 | 114.07 (7) | C13—C14—C15 | 120.64 (10) |
| C1—C6—H6A | 108.7 | C13—C14—H14 | 119.7 |
| C5—C6—H6A | 108.7 | C15—C14—H14 | 119.7 |
| C1—C6—H6B | 108.7 | C10—C15—C14 | 119.61 (9) |
| C5—C6—H6B | 108.7 | C10—C15—H15 | 120.2 |
| H6A—C6—H6B | 107.6 | C14—C15—H15 | 120.2 |
| C5—C7—H7A | 109.5 | C3—N1—C10 | 126.13 (7) |
| C5—C7—H7B | 109.5 | C3—N1—H1 | 116.9 |
| H7A—C7—H7B | 109.5 | C10—N1—H1 | 116.9 |
| O1—C1—C2—C3 | 178.90 (8) | C8—C5—C6—C1 | -69.18 (9) |
| C6—C1—C2—C3 | 0.93 (12) | C15—C10—C11—C12 | 0.66 (12) |
| C1—C2—C3—N1 | -176.05 (8) | N1—C10—C11—C12 | 178.82 (7) |
| C1—C2—C3—C4 | 1.17 (12) | C10—C11—C12—C13 | 0.27 (13) |
| N1—C3—C4—C5 | -157.43 (7) | C11—C12—C13—C14 | -0.70 (15) |
| C2—C3—C4—C5 | 25.06 (11) | C12—C13—C14—C15 | 0.19 (16) |
| C3—C4—C5—C7 | -168.72 (7) | C11—C10—C15—C14 | -1.17 (13) |
| C3—C4—C5—C6 | -49.78 (9) | N1—C10—C15—C14 | -179.28 (8) |
| C3—C4—C5—C8 | 70.86 (9) | C13—C14—C15—C10 | 0.74 (15) |
| O1—C1—C6—C5 | 152.80 (8) | C2—C3—N1—C10 | 2.19 (14) |
| C2—C1—C6—C5 | -29.16 (11) | C4—C3—N1—C10 | -175.20 (7) |
| C7—C5—C6—C1 | 170.28 (7) | C15—C10—N1—C3 | -54.91 (12) |
| C4—C5—C6—C1 | 51.84 (9) | C11—C10—N1—C3 | 126.96 (9) |

Hydrogen-bond geometry (Å, °)

Cg1 is the centroid of C10–C15 ring.

| <i>D</i> —H··· <i>A</i> | <i>D</i> —H | H··· <i>A</i> | <i>D</i> ··· <i>A</i> | <i>D</i> —H··· <i>A</i> |
|-------------------------------------|-------------|---------------|-----------------------|-------------------------|
| N1—H1···O1 ⁱ | 0.86 | 2.02 | 2.8587 (11) | 165 |
| C8—H8 <i>A</i> ···Cg1 ⁱⁱ | 0.96 | 2.61 | 3.5547 (12) | 157 |

Symmetry codes: (i) $-x+1, y+1/2, -z+3/2$; (ii) $-x+1, -y+2, -z+1$.