# Crystal structure of 3-amino-5,5-dimethyl-2-[(E)-2-nitroethenyl]cyclohex-2-en-1-one

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The asymmetric unit of the title compound, $C_{10}H_{14}N_2O_3$, contains two independent molecules with similar conformations. In the both molecules, the cyclohexene rings adopt the same envelope conformation with the flap C atoms lying 0.658 (3) and 0.668 (3) Å from the mean planes formed by the remaining atoms. In the crystal, adjacent molecules are connected via N—H···O hydrogen bonds and weak C—H···O interactions, forming supramolecular layers parallel to ($\bar{1}$01).

## 1. Chemical context

sec-Nitrodienamines appear to be potentially useful synthons in organic synthesis due to the enaminic, dienic and 'push–pull' character of these molecules (Koike et al., 2000). Several methods are available for the synthesis of nitrodienamines, which include the reaction of acetaldehydes with 1-dimethyl-amino-2-nitroethylen followed by treatment with amines (Severin et al., 1971), the reaction of aminoacrolein with di-methylamine and subsequent treatment of the vinylamidinium salt with nitromethane (Takeuchi et al., 1988) and nitro-alkenylation reactions of 2-methylindolines with nitroenam-ines (Attanasi et al., 2006).
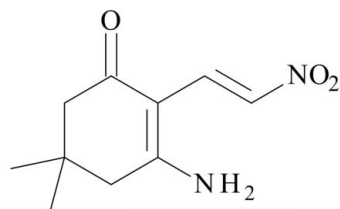
Previously, we found that alpha-nitro acetaldehyde under-goes an unusual condensation with aldehydes and ammonium acetate to afford 3,5-dinitro-1,2-dihydropyridines (Vigante et al., 1993). Afterwards, the synthesis of N-substituted 1,2-di-hydropyridines by heterocyclic annulation reaction of sec-nitrodienamines with acetaldehyde was reported (Koike et al., 1999). As part of our studies of synthetic pathways to fused 1,2-dihydropyridines, the title compound was synthesized and we report herein on its molecular and crystal structure.

## 2. Structural commentary

The asymmetric unit of the title compound (Fig. 1) contains two independent molecules (A and B) having coincident geometry. The bond lengths in the molecules are close to standard values. The cyclohexene rings adopt an envelope conformation, with flap atoms C3A and C3B lying 0.658 (3) and 0.668 (3) Å from the mean planes formed by the remaining atoms in molecules A and B, respectively.
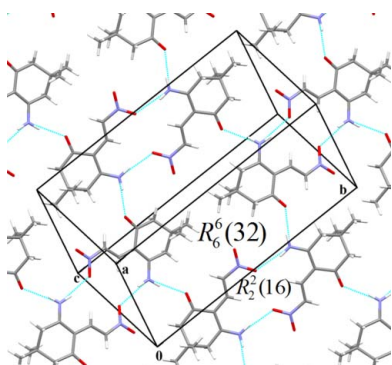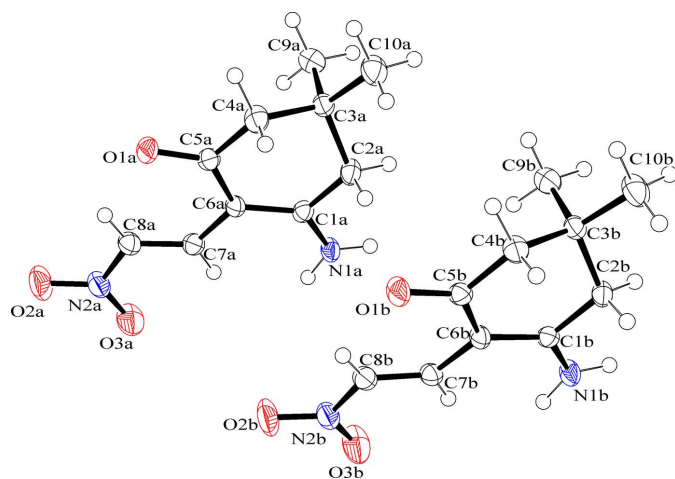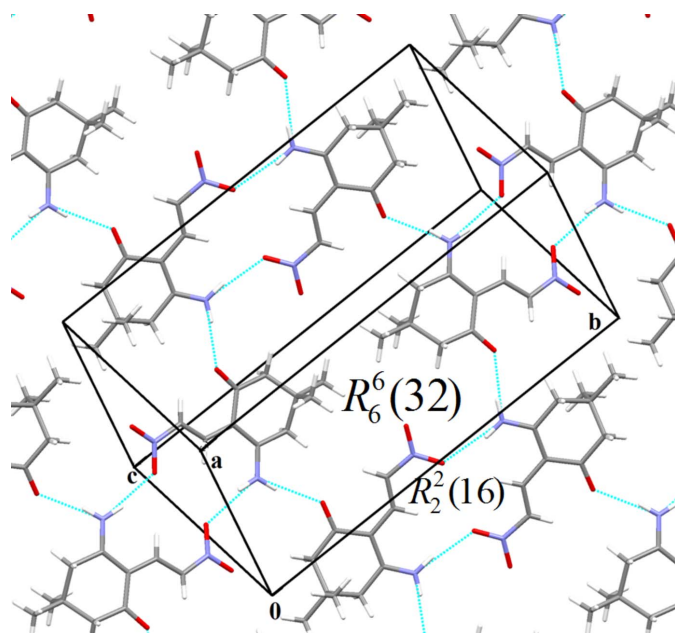
**Figure 1**
The asymmetric unit of the title compound, showing 50% probability displacement ellipsoids and the atomic numbering

## 3. Supramolecular features

In the crystal, the molecules form sheets parallel to ($\bar{1}$01) by means of N—H···O hydrogen bonds. The network consists of two hydrogen-bond motifs, $R_2^2(16)$ and $R_6^6(32)$ (Fig. 2). Weak C—H···O interactions are also observed in the supramolecular networks (Table 1).

## 4. Database survey

A search of the Cambridge Structural Database (Version 5.35; Groom & Allen, 2014) for 5,5-dimethylcyclohex-2-enones



**Figure 2**
The crystal packing of the title compound showing sheets parallel to ($\bar{1}$01).

gave 609 hits. Only one of these is a 3-amino-5,5-dimethyl-cyclohex-2-enone, namely, 3-amino-5,5-dimethyl-2-phenyl-cyclohex-2-enone (Fun *et al.*, 2007). The conformation of the cyclohexene ring is identical to that found in the title compound.

## 5. Synthesis and crystallization

A mixture of 3-amino-5,5-dimethylcyclohex-2-enone (140 mg, 1 mmol) and potassium salt of alpha-nitro acetaldehyde (190 mg, 1.5 mmol) in methanol (2 mL) and acetic acid (2 mL) was stirred for 5 days at room temperature. The solvents were removed under reduced pressure and the residue was purified by flash chromatography on silica gel, eluent: chloroform, hexane, acetone, methanol (9:7:1:1). The appropriate fraction

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N1$A$—H1$NA$···O1$B^i$ | 0.84 (2) | 2.06 (3) | 2.873 (2) | 162 (2) |
| N1$A$—H2$NA$···O3$B^{ii}$ | 0.88 (2) | 2.10 (3) | 2.961 (2) | 167 (2) |
| N1$B$—H1$NB$···O3$A^{ii}$ | 0.92 (2) | 2.03 (3) | 2.942 (2) | 168 (2) |
| N1$B$—H2$NB$···O1$A^{iii}$ | 0.85 (3) | 2.04 (2) | 2.858 (2) | 162 (2) |
| C2$A$—H2$A$1···O1$B^i$ | 0.97 | 2.42 | 3.262 (2) | 145 |
| C2$B$—H2$B$1···O1$A^{iii}$ | 0.97 | 2.46 | 3.298 (2) | 144 |
| C7$A$—H7$A$···O3$B^{ii}$ | 0.93 | 2.54 | 3.469 (2) | 173 |
| C7$B$—H7$B$···O3$A^{ii}$ | 0.93 | 2.55 | 3.469 (2) | 171 |

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

**Table 2**
Experimental details.

| | |
|---|---|
| **Crystal data** | |
| Chemical formula | $C_{10}H_{14}N_2O_3$ |
| $M_r$ | 210.23 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 173 |
| $a, b, c$ (Å) | 11.3545 (3), 18.1097 (5), 10.4689 (3) |
| $\beta$ (°) | 100.119 (2) |
| $V$ (Å$^3$) | 2119.20 (10) |
| $Z$ | 8 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.10 |
| Crystal size (mm) | $0.35 \times 0.25 \times 0.01$ |
| | |
| **Data collection** | |
| Diffractometer | Nonius KappaCCD |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 10741, 6174, 3235 |
| $R_{int}$ | 0.068 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.705 |
| | |
| **Refinement** | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.069, 0.158, 1.01 |
| No. of reflections | 6174 |
| No. of parameters | 287 |
| H-atom treatment | H atoms treated by a mixture of independent and constrained refinement |
| $\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å$^{-3}$) | 0.28, −0.29 |

Computer programs: *KappaCCD Server Software* (Nonius, 1997), *HKL DENZO SCALEPACK* (Otwinowski & Minor, 1997), *SIR2011* (Burla *et al.*, 2012), *ORTEP-3 for Windows* (Farrugia, 2012), *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

was collected and crystallized from methanol, yielding 116 mg (55%) of bright-yellow crystals (m.p. 503 K).

MS (+ESI) $m/z$ (relative intensity): 211.2 ($[M+H]^+$, 100). $^1$H NMR (400 MHz, DMSO-$d_6$): $\delta$ 0.96 (s, 6H), 2.20 (s, 2H), 2.53 (s, 2H), 8.12 (d, $J$ = 12.4 Hz, 1H), 8.39 (d, $J$ = 12.4 Hz, 1H), 8.48 (s, 1H), 8.74 (s, 1H).

$^{13}$C NMR (100.56 MHz, DMSO-$d_6$): $\delta$ 27.94, 31.41, 44.12, 51.46, 100.02, 131.82, 132.15, 172.30, 193.93. Analysis calculated for $C_{10}H_{14}N_2O_3$: C, 57.13; H, 6.71; N, 13.32; found: C, 56.98; H, 6.78; N, 13.16.

## 6. Refinement

Hydrogens on the amino group were located in a difference Fourier map and freely refined. The C-bound hydrogen atoms were positioned geometrically with C—H distances ranging from 0.93 to 0.97 Å and refined as riding on their parent atoms with $U_{iso}(H) = 1.5U_{eq}(C)$ for methyl groups and $U_{iso}(H) = 1.2U_{eq}(C)$ for other H atoms. The reflection whose intensity was affected by the beamstop was removed from the final refinement. Crystal data, data collection and structure refinement details are summarized in Table 2.

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# supporting information

# Crystal structure of 3-amino-5,5-dimethyl-2-[(*E*)-2-nitroethenyl]cyclohex-2-en-1-one

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### Computing details

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *HKL SCALEPACK* (Otwinowski & Minor, 1997); data reduction: *HKL DENZO* and *SCALEPACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR2011* (Burla *et al.*, 2012); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* (Sheldrick, 2008), *PLATON* (Spek, 2009) and *publCIF* (Westrip, 2010).

### 3-Amino-5,5-dimethyl-2-[(*E*)-2-nitroethenyl]cyclohex-2-en-1-one

*Crystal data*

| | |
|---|---|
| $C_{10}H_{14}N_2O_3$ | $F(000) = 896$ |
| $M_r = 210.23$ | $D_x = 1.318$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Melting point: 503 K |
| Hall symbol: -P 2ybc | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 11.3545$ (3) Å | Cell parameters from 11852 reflections |
| $b = 18.1097$ (5) Å | $\theta = 1.0$–30.0° |
| $c = 10.4689$ (3) Å | $\mu = 0.10$ mm$^{-1}$ |
| $\beta = 100.119$ (2)° | $T = 173$ K |
| $V = 2119.20$ (10) Å$^3$ | Plate, yellow |
| $Z = 8$ | $0.35 \times 0.25 \times 0.01$ mm |

*Data collection*

| | |
|---|---|
| Nonius KappaCCD diffractometer | 3235 reflections with $I > 2\sigma(I)$ |
| Radiation source: fine-focus sealed tube | $R_{int} = 0.068$ |
| Graphite monochromator | $\theta_{max} = 30.1°$, $\theta_{min} = 2.7°$ |
| CCD scans | $h = -15 \rightarrow 15$ |
| 10741 measured reflections | $k = -25 \rightarrow 23$ |
| 6174 independent reflections | $l = -14 \rightarrow 14$ |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Primary atom site location: structure-invariant direct methods |
| Least-squares matrix: full | |
| $R[F^2 > 2\sigma(F^2)] = 0.069$ | Secondary atom site location: difference Fourier map |
| $wR(F^2) = 0.158$ | |
| $S = 1.01$ | Hydrogen site location: inferred from neighbouring sites |
| 6174 reflections | |
| 287 parameters | H atoms treated by a mixture of independent and constrained refinement |
| 0 restraints | |

$w = 1/[\sigma^2(F_o^2) + (0.0554P)^2 + 0.6139P]$
  where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.28$ e Å$^{-3}$
$\Delta\rho_{min} = -0.28$ e Å$^{-3}$

*Special details*

**Geometry**. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)*

|  | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1A | 0.44423 (12) | 0.26776 (8) | 0.25500 (13) | 0.0295 (4) |
| O2A | 0.55773 (14) | 0.50926 (9) | 0.20293 (17) | 0.0471 (5) |
| O3A | 0.38755 (15) | 0.52829 (9) | 0.07970 (17) | 0.0470 (5) |
| N1A | 0.09527 (15) | 0.33969 (10) | −0.03942 (16) | 0.0254 (4) |
| H1NA | 0.034 (2) | 0.3223 (13) | −0.087 (2) | 0.030* |
| H2NA | 0.1126 (19) | 0.3869 (14) | −0.043 (2) | 0.030* |
| N2A | 0.45967 (16) | 0.48703 (10) | 0.14688 (17) | 0.0307 (4) |
| C1A | 0.16406 (16) | 0.29331 (11) | 0.03826 (18) | 0.0209 (4) |
| C2A | 0.11184 (17) | 0.21747 (11) | 0.0458 (2) | 0.0247 (5) |
| H2A1 | 0.0619 | 0.2062 | −0.0369 | 0.030* |
| H2A2 | 0.0608 | 0.2179 | 0.1110 | 0.030* |
| C3A | 0.20444 (16) | 0.15635 (11) | 0.07868 (19) | 0.0229 (4) |
| C4A | 0.28813 (18) | 0.18035 (11) | 0.20157 (19) | 0.0271 (5) |
| H4A1 | 0.2440 | 0.1807 | 0.2729 | 0.032* |
| H4A2 | 0.3518 | 0.1442 | 0.2217 | 0.032* |
| C5A | 0.34312 (16) | 0.25560 (11) | 0.19190 (17) | 0.0215 (4) |
| C6A | 0.27637 (16) | 0.31205 (11) | 0.11171 (17) | 0.0207 (4) |
| C7A | 0.32346 (17) | 0.38535 (11) | 0.10523 (18) | 0.0232 (4) |
| H7A | 0.2738 | 0.4188 | 0.0543 | 0.028* |
| C8A | 0.42972 (18) | 0.41153 (11) | 0.16320 (19) | 0.0259 (5) |
| H8A | 0.4840 | 0.3805 | 0.2141 | 0.031* |
| C9A | 0.27396 (19) | 0.14516 (13) | −0.0321 (2) | 0.0329 (5) |
| H9A1 | 0.3107 | 0.1909 | −0.0500 | 0.049* |
| H9A2 | 0.2202 | 0.1292 | −0.1083 | 0.049* |
| H9A3 | 0.3347 | 0.1084 | −0.0078 | 0.049* |
| C10A | 0.14236 (19) | 0.08427 (12) | 0.1036 (2) | 0.0356 (6) |
| H10A | 0.2015 | 0.0475 | 0.1344 | 0.053* |
| H10B | 0.0938 | 0.0676 | 0.0244 | 0.053* |
| H10C | 0.0927 | 0.0924 | 0.1677 | 0.053* |
| O1B | 0.92300 (13) | 0.24275 (8) | 0.28241 (13) | 0.0322 (4) |
| O2B | 1.04280 (14) | 0.48418 (9) | 0.22269 (17) | 0.0487 (5) |
| O3B | 0.88174 (16) | 0.49987 (9) | 0.08349 (18) | 0.0521 (5) |

| | | | | |
|---|---|---|---|---|
| N1B | 0.59376 (15) | 0.31138 (10) | −0.04246 (17) | 0.0263 (4) |
| H1NB | 0.6104 (19) | 0.3611 (14) | −0.046 (2) | 0.032* |
| H2NB | 0.537 (2) | 0.2930 (12) | −0.097 (2) | 0.032* |
| N2B | 0.94809 (15) | 0.46084 (10) | 0.16108 (17) | 0.0315 (4) |
| C1B | 0.66003 (16) | 0.26509 (11) | 0.03703 (18) | 0.0208 (4) |
| C2B | 0.61249 (16) | 0.18770 (11) | 0.03415 (19) | 0.0233 (4) |
| H2B1 | 0.5699 | 0.1773 | −0.0526 | 0.028* |
| H2B2 | 0.5553 | 0.1847 | 0.0927 | 0.028* |
| C3B | 0.70757 (16) | 0.12831 (11) | 0.07177 (19) | 0.0227 (4) |
| C4B | 0.77921 (18) | 0.15161 (11) | 0.20286 (19) | 0.0258 (5) |
| H4B1 | 0.7281 | 0.1477 | 0.2677 | 0.031* |
| H4B2 | 0.8451 | 0.1174 | 0.2267 | 0.031* |
| C5B | 0.82867 (17) | 0.22863 (11) | 0.20607 (18) | 0.0224 (4) |
| C6B | 0.76642 (16) | 0.28498 (11) | 0.12114 (18) | 0.0213 (4) |
| C7B | 0.81399 (17) | 0.35818 (11) | 0.11657 (18) | 0.0236 (4) |
| H7B | 0.7678 | 0.3906 | 0.0596 | 0.028* |
| C8B | 0.91564 (18) | 0.38621 (11) | 0.18294 (19) | 0.0258 (5) |
| H8B | 0.9651 | 0.3572 | 0.2432 | 0.031* |
| C9B | 0.78776 (19) | 0.12275 (13) | −0.0302 (2) | 0.0341 (5) |
| H9B1 | 0.8237 | 0.1699 | −0.0400 | 0.051* |
| H9B2 | 0.7408 | 0.1080 | −0.1116 | 0.051* |
| H9B3 | 0.8493 | 0.0868 | −0.0033 | 0.051* |
| C10B | 0.6467 (2) | 0.05431 (12) | 0.0841 (2) | 0.0357 (6) |
| H10D | 0.7056 | 0.0184 | 0.1199 | 0.054* |
| H10E | 0.6074 | 0.0383 | 0.0000 | 0.054* |
| H10F | 0.5888 | 0.0596 | 0.1402 | 0.054* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1A | 0.0257 (8) | 0.0261 (9) | 0.0313 (8) | 0.0026 (6) | −0.0102 (6) | 0.0006 (6) |
| O2A | 0.0386 (9) | 0.0392 (11) | 0.0589 (11) | −0.0172 (8) | −0.0045 (8) | −0.0057 (8) |
| O3A | 0.0501 (10) | 0.0252 (9) | 0.0583 (11) | −0.0029 (7) | −0.0110 (9) | 0.0062 (8) |
| N1A | 0.0226 (9) | 0.0198 (10) | 0.0294 (9) | −0.0021 (7) | −0.0073 (7) | 0.0016 (7) |
| N2A | 0.0321 (10) | 0.0267 (11) | 0.0314 (10) | −0.0052 (8) | 0.0002 (8) | −0.0035 (8) |
| C1A | 0.0194 (9) | 0.0242 (12) | 0.0187 (9) | 0.0018 (8) | 0.0021 (8) | −0.0004 (8) |
| C2A | 0.0196 (10) | 0.0243 (12) | 0.0280 (10) | −0.0023 (8) | −0.0020 (8) | 0.0045 (8) |
| C3A | 0.0217 (10) | 0.0193 (11) | 0.0258 (10) | −0.0003 (8) | −0.0009 (8) | 0.0010 (8) |
| C4A | 0.0284 (11) | 0.0238 (12) | 0.0258 (10) | −0.0005 (9) | −0.0039 (9) | 0.0059 (8) |
| C5A | 0.0226 (10) | 0.0244 (11) | 0.0165 (9) | 0.0035 (8) | 0.0007 (8) | −0.0021 (8) |
| C6A | 0.0206 (9) | 0.0195 (11) | 0.0201 (9) | 0.0007 (8) | −0.0012 (8) | −0.0009 (7) |
| C7A | 0.0248 (10) | 0.0226 (12) | 0.0207 (10) | 0.0033 (8) | −0.0006 (8) | −0.0005 (8) |
| C8A | 0.0275 (10) | 0.0198 (12) | 0.0286 (11) | −0.0003 (8) | 0.0000 (9) | 0.0000 (8) |
| C9A | 0.0339 (11) | 0.0270 (13) | 0.0376 (12) | −0.0004 (9) | 0.0061 (10) | −0.0051 (10) |
| C10A | 0.0330 (12) | 0.0248 (13) | 0.0458 (14) | −0.0049 (9) | −0.0016 (11) | 0.0055 (10) |
| O1B | 0.0286 (8) | 0.0280 (9) | 0.0332 (8) | 0.0016 (6) | −0.0131 (7) | −0.0021 (6) |
| O2B | 0.0387 (9) | 0.0368 (11) | 0.0635 (12) | −0.0169 (8) | −0.0108 (9) | −0.0058 (8) |
| O3B | 0.0520 (11) | 0.0271 (10) | 0.0665 (12) | −0.0061 (8) | −0.0193 (9) | 0.0124 (8) |

| N1B | 0.0229 (9) | 0.0220 (10) | 0.0292 (9) | −0.0008 (7) | −0.0090 (7) | 0.0009 (7) |
|---|---|---|---|---|---|---|
| N2B | 0.0302 (10) | 0.0253 (11) | 0.0361 (10) | −0.0038 (8) | −0.0018 (8) | −0.0039 (8) |
| C1B | 0.0203 (10) | 0.0211 (11) | 0.0204 (9) | 0.0023 (8) | 0.0015 (8) | −0.0028 (8) |
| C2B | 0.0189 (9) | 0.0215 (11) | 0.0276 (10) | −0.0024 (8) | −0.0013 (8) | 0.0024 (8) |
| C3B | 0.0216 (10) | 0.0183 (11) | 0.0269 (10) | −0.0013 (8) | 0.0006 (8) | −0.0006 (8) |
| C4B | 0.0274 (10) | 0.0204 (11) | 0.0271 (11) | 0.0003 (8) | −0.0022 (9) | 0.0051 (8) |
| C5B | 0.0227 (10) | 0.0226 (12) | 0.0209 (10) | 0.0027 (8) | 0.0011 (8) | −0.0031 (8) |
| C6B | 0.0203 (10) | 0.0209 (11) | 0.0207 (10) | −0.0003 (8) | −0.0017 (8) | −0.0010 (8) |
| C7B | 0.0242 (10) | 0.0206 (12) | 0.0242 (10) | 0.0024 (8) | −0.0007 (8) | −0.0019 (8) |
| C8B | 0.0274 (10) | 0.0230 (12) | 0.0257 (10) | 0.0013 (8) | 0.0007 (9) | −0.0022 (8) |
| C9B | 0.0329 (12) | 0.0297 (14) | 0.0405 (13) | −0.0033 (9) | 0.0091 (10) | −0.0092 (10) |
| C10B | 0.0378 (12) | 0.0243 (13) | 0.0419 (13) | −0.0072 (10) | −0.0017 (11) | 0.0036 (10) |

## Geometric parameters (Å, º)

| | | | |
|---|---|---|---|
| O1A—C5A | 1.240 (2) | O1B—C5B | 1.245 (2) |
| O2A—N2A | 1.231 (2) | O2B—N2B | 1.228 (2) |
| O3A—N2A | 1.233 (2) | O3B—N2B | 1.230 (2) |
| N1A—C1A | 1.324 (2) | N1B—C1B | 1.320 (2) |
| N1A—H1NA | 0.84 (2) | N1B—H1NB | 0.92 (2) |
| N1A—H2NA | 0.88 (2) | N1B—H2NB | 0.86 (2) |
| N2A—C8A | 1.426 (3) | N2B—C8B | 1.430 (3) |
| C1A—C6A | 1.410 (2) | C1B—C6B | 1.411 (2) |
| C1A—C2A | 1.504 (3) | C1B—C2B | 1.500 (3) |
| C2A—C3A | 1.524 (3) | C2B—C3B | 1.526 (3) |
| C2A—H2A1 | 0.9700 | C2B—H2B1 | 0.9700 |
| C2A—H2A2 | 0.9700 | C2B—H2B2 | 0.9700 |
| C3A—C4A | 1.522 (3) | C3B—C9B | 1.523 (3) |
| C3A—C9A | 1.527 (3) | C3B—C10B | 1.524 (3) |
| C3A—C10A | 1.528 (3) | C3B—C4B | 1.527 (3) |
| C4A—C5A | 1.510 (3) | C4B—C5B | 1.502 (3) |
| C4A—H4A1 | 0.9700 | C4B—H4B1 | 0.9700 |
| C4A—H4A2 | 0.9700 | C4B—H4B2 | 0.9700 |
| C5A—C6A | 1.449 (3) | C5B—C6B | 1.453 (3) |
| C6A—C7A | 1.437 (3) | C6B—C7B | 1.435 (3) |
| C7A—C8A | 1.338 (3) | C7B—C8B | 1.338 (3) |
| C7A—H7A | 0.9300 | C7B—H7B | 0.9300 |
| C8A—H8A | 0.9300 | C8B—H8B | 0.9300 |
| C9A—H9A1 | 0.9600 | C9B—H9B1 | 0.9600 |
| C9A—H9A2 | 0.9600 | C9B—H9B2 | 0.9600 |
| C9A—H9A3 | 0.9600 | C9B—H9B3 | 0.9600 |
| C10A—H10A | 0.9600 | C10B—H10D | 0.9600 |
| C10A—H10B | 0.9600 | C10B—H10E | 0.9600 |
| C10A—H10C | 0.9600 | C10B—H10F | 0.9600 |
| | | | |
| C1A—N1A—H1NA | 117.6 (15) | C1B—N1B—H1NB | 123.2 (12) |
| C1A—N1A—H2NA | 122.0 (13) | C1B—N1B—H2NB | 117.2 (15) |
| H1NA—N1A—H2NA | 120 (2) | H1NB—N1B—H2NB | 119.4 (19) |

| | | | |
|---|---|---|---|
| O2A—N2A—O3A | 121.86 (19) | O2B—N2B—O3B | 121.85 (19) |
| O2A—N2A—C8A | 118.09 (17) | O2B—N2B—C8B | 117.90 (17) |
| O3A—N2A—C8A | 120.05 (16) | O3B—N2B—C8B | 120.26 (16) |
| N1A—C1A—C6A | 124.16 (19) | N1B—C1B—C6B | 124.21 (19) |
| N1A—C1A—C2A | 114.59 (16) | N1B—C1B—C2B | 114.67 (16) |
| C6A—C1A—C2A | 121.23 (16) | C6B—C1B—C2B | 121.11 (16) |
| C1A—C2A—C3A | 114.37 (16) | C1B—C2B—C3B | 114.68 (15) |
| C1A—C2A—H2A1 | 108.7 | C1B—C2B—H2B1 | 108.6 |
| C3A—C2A—H2A1 | 108.7 | C3B—C2B—H2B1 | 108.6 |
| C1A—C2A—H2A2 | 108.7 | C1B—C2B—H2B2 | 108.6 |
| C3A—C2A—H2A2 | 108.7 | C3B—C2B—H2B2 | 108.6 |
| H2A1—C2A—H2A2 | 107.6 | H2B1—C2B—H2B2 | 107.6 |
| C4A—C3A—C2A | 106.68 (16) | C9B—C3B—C10B | 109.70 (18) |
| C4A—C3A—C9A | 110.52 (16) | C9B—C3B—C2B | 110.40 (17) |
| C2A—C3A—C9A | 110.67 (17) | C10B—C3B—C2B | 109.33 (16) |
| C4A—C3A—C10A | 109.75 (16) | C9B—C3B—C4B | 111.01 (16) |
| C2A—C3A—C10A | 109.79 (16) | C10B—C3B—C4B | 110.17 (17) |
| C9A—C3A—C10A | 109.39 (18) | C2B—C3B—C4B | 106.16 (16) |
| C5A—C4A—C3A | 113.90 (16) | C5B—C4B—C3B | 114.51 (16) |
| C5A—C4A—H4A1 | 108.8 | C5B—C4B—H4B1 | 108.6 |
| C3A—C4A—H4A1 | 108.8 | C3B—C4B—H4B1 | 108.6 |
| C5A—C4A—H4A2 | 108.8 | C5B—C4B—H4B2 | 108.6 |
| C3A—C4A—H4A2 | 108.8 | C3B—C4B—H4B2 | 108.6 |
| H4A1—C4A—H4A2 | 107.7 | H4B1—C4B—H4B2 | 107.6 |
| O1A—C5A—C6A | 121.64 (18) | O1B—C5B—C6B | 121.31 (18) |
| O1A—C5A—C4A | 118.67 (16) | O1B—C5B—C4B | 118.76 (17) |
| C6A—C5A—C4A | 119.68 (16) | C6B—C5B—C4B | 119.93 (16) |
| C1A—C6A—C7A | 120.33 (17) | C1B—C6B—C7B | 120.06 (17) |
| C1A—C6A—C5A | 118.48 (17) | C1B—C6B—C5B | 118.28 (17) |
| C7A—C6A—C5A | 121.18 (16) | C7B—C6B—C5B | 121.58 (16) |
| C8A—C7A—C6A | 128.25 (18) | C8B—C7B—C6B | 128.93 (18) |
| C8A—C7A—H7A | 115.9 | C8B—C7B—H7B | 115.5 |
| C6A—C7A—H7A | 115.9 | C6B—C7B—H7B | 115.5 |
| C7A—C8A—N2A | 120.01 (18) | C7B—C8B—N2B | 119.90 (18) |
| C7A—C8A—H8A | 120.0 | C7B—C8B—H8B | 120.0 |
| N2A—C8A—H8A | 120.0 | N2B—C8B—H8B | 120.0 |
| C3A—C9A—H9A1 | 109.5 | C3B—C9B—H9B1 | 109.5 |
| C3A—C9A—H9A2 | 109.5 | C3B—C9B—H9B2 | 109.5 |
| H9A1—C9A—H9A2 | 109.5 | H9B1—C9B—H9B2 | 109.5 |
| C3A—C9A—H9A3 | 109.5 | C3B—C9B—H9B3 | 109.5 |
| H9A1—C9A—H9A3 | 109.5 | H9B1—C9B—H9B3 | 109.5 |
| H9A2—C9A—H9A3 | 109.5 | H9B2—C9B—H9B3 | 109.5 |
| C3A—C10A—H10A | 109.5 | C3B—C10B—H10D | 109.5 |
| C3A—C10A—H10B | 109.5 | C3B—C10B—H10E | 109.5 |
| H10A—C10A—H10B | 109.5 | H10D—C10B—H10E | 109.5 |
| C3A—C10A—H10C | 109.5 | C3B—C10B—H10F | 109.5 |
| H10A—C10A—H10C | 109.5 | H10D—C10B—H10F | 109.5 |
| H10B—C10A—H10C | 109.5 | H10E—C10B—H10F | 109.5 |

| | | | |
|---|---|---|---|
| N1A—C1A—C2A—C3A | 152.82 (18) | N1B—C1B—C2B—C3B | 152.97 (18) |
| C6A—C1A—C2A—C3A | −28.5 (3) | C6B—C1B—C2B—C3B | −28.1 (3) |
| C1A—C2A—C3A—C4A | 52.7 (2) | C1B—C2B—C3B—C9B | −67.4 (2) |
| C1A—C2A—C3A—C9A | −67.5 (2) | C1B—C2B—C3B—C10B | 171.80 (17) |
| C1A—C2A—C3A—C10A | 171.61 (17) | C1B—C2B—C3B—C4B | 53.0 (2) |
| C2A—C3A—C4A—C5A | −54.4 (2) | C9B—C3B—C4B—C5B | 66.5 (2) |
| C9A—C3A—C4A—C5A | 66.0 (2) | C10B—C3B—C4B—C5B | −171.79 (18) |
| C10A—C3A—C4A—C5A | −173.26 (18) | C2B—C3B—C4B—C5B | −53.5 (2) |
| C3A—C4A—C5A—O1A | −149.19 (18) | C3B—C4B—C5B—O1B | −150.83 (19) |
| C3A—C4A—C5A—C6A | 31.9 (3) | C3B—C4B—C5B—C6B | 29.2 (3) |
| N1A—C1A—C6A—C7A | −0.1 (3) | N1B—C1B—C6B—C7B | −5.0 (3) |
| C2A—C1A—C6A—C7A | −178.63 (19) | C2B—C1B—C6B—C7B | 176.23 (19) |
| N1A—C1A—C6A—C5A | −179.33 (19) | N1B—C1B—C6B—C5B | 178.3 (2) |
| C2A—C1A—C6A—C5A | 2.2 (3) | C2B—C1B—C6B—C5B | −0.5 (3) |
| O1A—C5A—C6A—C1A | 177.29 (19) | O1B—C5B—C6B—C1B | 179.96 (19) |
| C4A—C5A—C6A—C1A | −3.9 (3) | C4B—C5B—C6B—C1B | −0.1 (3) |
| O1A—C5A—C6A—C7A | −1.9 (3) | O1B—C5B—C6B—C7B | 3.3 (3) |
| C4A—C5A—C6A—C7A | 176.91 (19) | C4B—C5B—C6B—C7B | −176.77 (19) |
| C1A—C6A—C7A—C8A | −176.7 (2) | C1B—C6B—C7B—C8B | −176.3 (2) |
| C5A—C6A—C7A—C8A | 2.5 (3) | C5B—C6B—C7B—C8B | 0.3 (3) |
| C6A—C7A—C8A—N2A | −178.8 (2) | C6B—C7B—C8B—N2B | 178.4 (2) |
| O2A—N2A—C8A—C7A | 179.2 (2) | O2B—N2B—C8B—C7B | −179.1 (2) |
| O3A—N2A—C8A—C7A | 0.2 (3) | O3B—N2B—C8B—C7B | 1.5 (3) |

*Hydrogen-bond geometry (Å, º)*

| D—H···A | D—H | H···A | D···A | D—H···A |
|---|---|---|---|---|
| N1A—H1NA···O1Bⁱ | 0.84 (2) | 2.06 (3) | 2.873 (2) | 162 (2) |
| N1A—H2NA···O3Bⁱⁱ | 0.88 (2) | 2.10 (3) | 2.961 (2) | 167 (2) |
| N1B—H1NB···O3Aⁱⁱ | 0.92 (2) | 2.03 (3) | 2.942 (2) | 168 (2) |
| N1B—H2NB···O1Aⁱⁱⁱ | 0.85 (3) | 2.04 (2) | 2.858 (2) | 162 (2) |
| C2A—H2A1···O1Bⁱ | 0.97 | 2.42 | 3.262 (2) | 145 |
| C2B—H2B1···O1Aⁱⁱⁱ | 0.97 | 2.46 | 3.298 (2) | 144 |
| C7A—H7A···O3Bⁱⁱ | 0.93 | 2.54 | 3.469 (2) | 173 |
| C7B—H7B···O3Aⁱⁱ | 0.93 | 2.55 | 3.469 (2) | 171 |

Symmetry codes: (i) *x*−1, −*y*+1/2, *z*−1/2; (ii) −*x*+1, −*y*+1, −*z*; (iii) *x*, −*y*+1/2, *z*−1/2.