Acta Crystallographica Section E
**Structure Reports Online**
ISSN 1600-5368

# 1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5*H*-1,5-benzodiazepin-2(1*H*)-one

**Hanane Benzeid,[a] Nathalie Saffon,[b] Bernard Garrigues,[c] El Mokhtar Essassi[a] and Seik Weng Ng[d]***

[a]Laboratoire de Chimie Organique Hétérocyclique, Pôle de Compétences Pharmacochimie, Université Mohammed V-Agdal, BP 1014 Avenue Ibn Batout, Rabat, Morocco, [b]Service Commun Rayons X, Université Paul Sabatier, Bâtiment 2R1, 118 route de Narbonne, 31062 Toulouse, France, [c]Hétérochimie Fondamentale et Appliquée, Université Paul Sabatier, UMR 5069, 118 Route de Narbonne, 31062 Toulouse, France, and [d]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
Correspondence e-mail: seikweng@um.edu.my

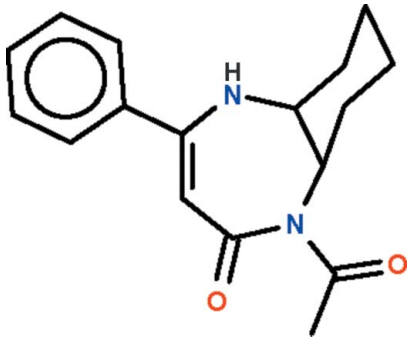
Received 29 September 2009; accepted 1 October 2009

Key indicators: single-crystal X-ray study; $T$ = 193 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.046; $wR$ factor = 0.139; data-to-parameter ratio = 23.5.

The seven-membered ring of the title compound, $C_{17}H_{20}N_2O_2$, adopts an approximate boat conformation while the cyclohexyl ring adopts a chair conformation. In the crystal, adjacent molecules are linked by N—H···O hydrogen bonds into a zigzag chain running along the $c$ axis of the monoclinic unit cell.

## Related literature

For the crystal structures of anhydrous and hydrated 7-phenyl-1,2, 3,4-tetrahydro-1,4-diazepin-5-ones, see: Clark *et al.* (1999); Chammache *et al.* (2001).




## Experimental

### Crystal data

$C_{17}H_{20}N_2O_2$
$M_r$ = 284.35
Monoclinic, $P2_1/c$
$a$ = 9.6794 (2) Å
$b$ = 14.0095 (3) Å
$c$ = 11.2832 (2) Å
$\beta$ = 98.053 (1)°
$V$ = 1514.95 (5) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.08 mm$^{-1}$
$T$ = 193 K
0.6 × 0.6 × 0.6 mm

### Data collection

Bruker APEXII diffractometer
Absorption correction: none
27399 measured reflections
4591 independent reflections
3878 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.024

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.139
$S$ = 1.05
4591 reflections
195 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.38 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.30 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O1$^{i}$ | 0.88 (1) | 2.001 (9) | 2.854 (1) | 164 (1) |

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

We thank Université Mohammed V-Agdal and the University of Malaya for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2546).

## References

Barbour, L. J. (2001). *J. Supramol. Chem.* **1**, 189–191.
Bruker (2005). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Chammache, M., Essassi, E. M. & Pierrot, M. (2001). *Z. Kristallogr. New Cryst. Struct.* **216**, 101–102.
Clark, B. A. J., Evans, M. C., Lloyd, D., McNab, H. & Parsons, S. (1999). *Acta Cryst.* C**55**, 1725–1727.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2009). *publCIF*. In preparation.

# supplementary materials

*Acta Cryst.* (2009). E**65**, o2657 [ **doi:10.1107/S1600536809039932** ]

# 1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5*H*-1,5-benzodiazepin-2(1*H*)-one

**H. Benzeid, N. Saffon, B. Garrigues, E. M. Essassi and S. W. Ng**

## Experimental

4-Phenyl-5*a*,6,7,8,9,9*a*-hexahydro-1*H*-benzo[*b*][1,4]diazepin-2(3*H*)-one (1 g) was refluxed in acetic acid (20 ml) for 12 h. The precipate was collected and recrystallized from ethanol to afford colorless crystals in 90% yield.

## Refinement

Carbon-bound H-atoms were placed in calculated positions (C—H 0.95 to 0.99 Å) and were included in the refinement in the riding model approximation, with $U_{iso}$(H) set to 1.2–1.5$U_{eq}$(C). The amino H-atom was located in a difference Fourier map and was refined with an N–H 0.88±0.01 Å restraint.

## Figures

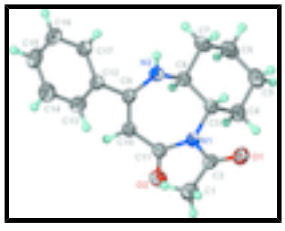


Fig. 1. Thermal ellipsoid plot (Barbour, 2001) of $C_{17}H_{20}N_2O_2$ at the 70% probability level; hydrogen atoms are drawn as spheres of arbitrary radius.

## 1-Acetyl-4-phenyl-5a,6,7,8,9,9a-hexahydro-5*H*- 1,5-benzodiazepin-2(1*H*)-one

*Crystal data*

| | |
|---|---|
| $C_{17}H_{20}N_2O_2$ | $F_{000}$ = 608 |
| $M_r$ = 284.35 | $D_x$ = 1.247 Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, λ = 0.71073 Å |
| Hall symbol: -P 2ybc | Cell parameters from 9928 reflections |
| $a$ = 9.6794 (2) Å | θ = 5.2–30.6º |
| $b$ = 14.0095 (3) Å | μ = 0.08 mm$^{-1}$ |
| $c$ = 11.2832 (2) Å | $T$ = 193 K |
| β = 98.053 (1)º | Block, colorless |
| $V$ = 1514.95 (5) Å$^3$ | 0.6 × 0.6 × 0.6 mm |
| $Z$ = 4 | |

*Data collection*

| | |
|---|---|
| Bruker APEXII diffractometer | 3878 reflections with $I > 2\sigma(I)$ |

| | |
|---|---|
| Radiation source: fine-focus sealed tube | $R_{int} = 0.024$ |
| Monochromator: graphite | $\theta_{max} = 30.5°$ |
| $T = 193$ K | $\theta_{min} = 5.2°$ |
| φ and ω scans | $h = -13 \rightarrow 13$ |
| Absorption correction: None | $k = 0 \rightarrow 20$ |
| 27399 measured reflections | $l = 0 \rightarrow 16$ |
| 4591 independent reflections | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.046$ | H atoms treated by a mixture of independent and constrained refinement |
| $wR(F^2) = 0.139$ | $w = 1/[\sigma^2(F_o^2) + (0.0824P)^2 + 0.2971P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.05$ | $(\Delta/\sigma)_{max} = 0.001$ |
| 4591 reflections | $\Delta\rho_{max} = 0.38$ e Å$^{-3}$ |
| 195 parameters | $\Delta\rho_{min} = -0.30$ e Å$^{-3}$ |
| 1 restraint | Extinction correction: none |
| Primary atom site location: structure-invariant direct methods | |

*Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 0.38581 (9) | 0.66240 (6) | 0.46797 (7) | 0.03445 (19) |
| O2 | 0.32929 (9) | 0.42092 (6) | 0.27399 (8) | 0.0390 (2) |
| N1 | 0.40005 (8) | 0.57744 (6) | 0.29988 (7) | 0.02455 (17) |
| N2 | 0.44012 (9) | 0.65352 (6) | 0.07259 (8) | 0.02644 (18) |
| H2 | 0.4411 (15) | 0.7098 (7) | 0.0384 (12) | 0.038 (4)* |
| C1 | 0.18397 (12) | 0.57212 (10) | 0.39317 (10) | 0.0398 (3) |
| H1A | 0.1299 | 0.6227 | 0.4252 | 0.060* |
| H1B | 0.1843 | 0.5152 | 0.4436 | 0.060* |
| H1C | 0.1417 | 0.5566 | 0.3114 | 0.060* |
| C2 | 0.33153 (10) | 0.60571 (7) | 0.39210 (8) | 0.0266 (2) |
| C3 | 0.53214 (10) | 0.62624 (7) | 0.28648 (9) | 0.02480 (19) |
| H3 | 0.5220 | 0.6951 | 0.3071 | 0.030* |
| C4 | 0.65550 (11) | 0.58553 (8) | 0.37093 (10) | 0.0321 (2) |
| H4A | 0.6663 | 0.5168 | 0.3538 | 0.039* |
| H4B | 0.6375 | 0.5918 | 0.4548 | 0.039* |
| C5 | 0.78980 (12) | 0.63873 (10) | 0.35493 (12) | 0.0412 (3) |
| H5A | 0.8697 | 0.6087 | 0.4059 | 0.049* |
| H5B | 0.7828 | 0.7058 | 0.3812 | 0.049* |
| C6 | 0.81573 (12) | 0.63687 (9) | 0.22446 (12) | 0.0382 (3) |
| H6A | 0.8330 | 0.5703 | 0.2009 | 0.046* |
| H6B | 0.8999 | 0.6750 | 0.2160 | 0.046* |

| | | | | |
|---|---|---|---|---|
| C7 | 0.69074 (11) | 0.67727 (8) | 0.14134 (10) | 0.0322 (2) |
| H7A | 0.7086 | 0.6732 | 0.0572 | 0.039* |
| H7B | 0.6774 | 0.7453 | 0.1607 | 0.039* |
| C8 | 0.55891 (10) | 0.62073 (7) | 0.15649 (9) | 0.02496 (19) |
| H8 | 0.5762 | 0.5523 | 0.1379 | 0.030* |
| C9 | 0.33011 (10) | 0.59655 (7) | 0.03819 (8) | 0.02568 (19) |
| C10 | 0.30297 (11) | 0.51532 (7) | 0.10041 (9) | 0.0280 (2) |
| H10 | 0.2498 | 0.4668 | 0.0563 | 0.034* |
| C11 | 0.34703 (10) | 0.49695 (7) | 0.22489 (9) | 0.02630 (19) |
| C12 | 0.24115 (11) | 0.62065 (7) | −0.07600 (9) | 0.0269 (2) |
| C13 | 0.09583 (12) | 0.61485 (8) | −0.08454 (10) | 0.0333 (2) |
| H13 | 0.0541 | 0.5972 | −0.0164 | 0.040* |
| C14 | 0.01201 (13) | 0.63481 (9) | −0.19226 (11) | 0.0390 (3) |
| H14 | −0.0867 | 0.6311 | −0.1976 | 0.047* |
| C15 | 0.07343 (14) | 0.66027 (9) | −0.29194 (10) | 0.0400 (3) |
| H15 | 0.0164 | 0.6734 | −0.3657 | 0.048* |
| C16 | 0.21775 (14) | 0.66671 (8) | −0.28440 (10) | 0.0365 (2) |
| H16 | 0.2590 | 0.6844 | −0.3527 | 0.044* |
| C17 | 0.30146 (12) | 0.64719 (7) | −0.17679 (9) | 0.0308 (2) |
| H17 | 0.4000 | 0.6519 | −0.1716 | 0.037* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| O1 | 0.0456 (5) | 0.0305 (4) | 0.0279 (4) | −0.0012 (3) | 0.0075 (3) | −0.0051 (3) |
| O2 | 0.0502 (5) | 0.0266 (4) | 0.0384 (4) | −0.0081 (3) | −0.0005 (4) | 0.0075 (3) |
| N1 | 0.0264 (4) | 0.0238 (4) | 0.0234 (4) | −0.0018 (3) | 0.0033 (3) | −0.0015 (3) |
| N2 | 0.0308 (4) | 0.0209 (4) | 0.0275 (4) | −0.0017 (3) | 0.0038 (3) | 0.0017 (3) |
| C1 | 0.0286 (5) | 0.0589 (7) | 0.0326 (5) | −0.0009 (5) | 0.0063 (4) | 0.0009 (5) |
| C2 | 0.0301 (5) | 0.0263 (4) | 0.0236 (4) | 0.0030 (3) | 0.0038 (3) | 0.0026 (3) |
| C3 | 0.0256 (4) | 0.0214 (4) | 0.0273 (4) | −0.0012 (3) | 0.0034 (3) | −0.0019 (3) |
| C4 | 0.0281 (5) | 0.0344 (5) | 0.0324 (5) | 0.0006 (4) | −0.0010 (4) | −0.0002 (4) |
| C5 | 0.0286 (5) | 0.0494 (7) | 0.0440 (6) | −0.0053 (5) | −0.0006 (4) | −0.0051 (5) |
| C6 | 0.0272 (5) | 0.0390 (6) | 0.0492 (7) | −0.0038 (4) | 0.0080 (4) | −0.0051 (5) |
| C7 | 0.0314 (5) | 0.0278 (5) | 0.0387 (5) | −0.0050 (4) | 0.0096 (4) | −0.0028 (4) |
| C8 | 0.0258 (4) | 0.0208 (4) | 0.0286 (4) | −0.0003 (3) | 0.0049 (3) | −0.0016 (3) |
| C9 | 0.0310 (5) | 0.0233 (4) | 0.0229 (4) | −0.0006 (3) | 0.0047 (3) | −0.0026 (3) |
| C10 | 0.0357 (5) | 0.0228 (4) | 0.0251 (4) | −0.0053 (3) | 0.0030 (4) | −0.0028 (3) |
| C11 | 0.0290 (4) | 0.0219 (4) | 0.0277 (4) | −0.0014 (3) | 0.0029 (3) | −0.0005 (3) |
| C12 | 0.0326 (5) | 0.0246 (4) | 0.0235 (4) | −0.0022 (3) | 0.0036 (3) | −0.0012 (3) |
| C13 | 0.0335 (5) | 0.0366 (5) | 0.0300 (5) | −0.0015 (4) | 0.0056 (4) | 0.0012 (4) |
| C14 | 0.0347 (5) | 0.0422 (6) | 0.0383 (6) | 0.0006 (4) | −0.0006 (4) | 0.0002 (5) |
| C15 | 0.0506 (7) | 0.0390 (6) | 0.0277 (5) | 0.0019 (5) | −0.0041 (5) | −0.0006 (4) |
| C16 | 0.0519 (7) | 0.0348 (5) | 0.0233 (4) | −0.0036 (5) | 0.0067 (4) | −0.0006 (4) |
| C17 | 0.0383 (5) | 0.0283 (5) | 0.0263 (4) | −0.0042 (4) | 0.0065 (4) | −0.0023 (4) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| O1—C2 | 1.2303 (13) | C6—H6A | 0.9900 |

| | | | |
|---|---|---|---|
| O2—C11 | 1.2238 (12) | C6—H6B | 0.9900 |
| N1—C2 | 1.3686 (12) | C7—C8 | 1.5317 (14) |
| N1—C11 | 1.4591 (12) | C7—H7A | 0.9900 |
| N1—C3 | 1.4761 (12) | C7—H7B | 0.9900 |
| N2—C9 | 1.3438 (13) | C8—H8 | 1.0000 |
| N2—C8 | 1.4574 (13) | C9—C10 | 1.3815 (13) |
| N2—H2 | 0.878 (9) | C9—C12 | 1.4849 (14) |
| C1—C2 | 1.5055 (15) | C10—C11 | 1.4328 (14) |
| C1—H1A | 0.9800 | C10—H10 | 0.9500 |
| C1—H1B | 0.9800 | C12—C13 | 1.3987 (15) |
| C1—H1C | 0.9800 | C12—C17 | 1.3995 (14) |
| C3—C4 | 1.5298 (14) | C13—C14 | 1.3919 (16) |
| C3—C8 | 1.5268 (13) | C13—H13 | 0.9500 |
| C3—H3 | 1.0000 | C14—C15 | 1.3904 (17) |
| C4—C5 | 1.5309 (16) | C14—H14 | 0.9500 |
| C4—H4A | 0.9900 | C15—C16 | 1.3904 (19) |
| C4—H4B | 0.9900 | C15—H15 | 0.9500 |
| C5—C6 | 1.5279 (18) | C16—C17 | 1.3892 (16) |
| C5—H5A | 0.9900 | C16—H16 | 0.9500 |
| C5—H5B | 0.9900 | C17—H17 | 0.9500 |
| C6—C7 | 1.5315 (17) | | |
| C2—N1—C11 | 119.73 (8) | C6—C7—C8 | 109.85 (9) |
| C2—N1—C3 | 117.57 (8) | C6—C7—H7A | 109.7 |
| C11—N1—C3 | 122.54 (8) | C8—C7—H7A | 109.7 |
| C9—N2—C8 | 121.44 (8) | C6—C7—H7B | 109.7 |
| C9—N2—H2 | 117.6 (10) | C8—C7—H7B | 109.7 |
| C8—N2—H2 | 120.8 (10) | H7A—C7—H7B | 108.2 |
| C2—C1—H1A | 109.5 | N2—C8—C3 | 112.54 (8) |
| C2—C1—H1B | 109.5 | N2—C8—C7 | 111.00 (8) |
| H1A—C1—H1B | 109.5 | C3—C8—C7 | 109.71 (8) |
| C2—C1—H1C | 109.5 | N2—C8—H8 | 107.8 |
| H1A—C1—H1C | 109.5 | C3—C8—H8 | 107.8 |
| H1B—C1—H1C | 109.5 | C7—C8—H8 | 107.8 |
| O1—C2—N1 | 120.71 (9) | N2—C9—C10 | 122.81 (9) |
| O1—C2—C1 | 120.62 (9) | N2—C9—C12 | 117.08 (8) |
| N1—C2—C1 | 118.49 (9) | C10—C9—C12 | 119.99 (9) |
| N1—C3—C4 | 112.03 (8) | C9—C10—C11 | 126.47 (9) |
| N1—C3—C8 | 109.94 (7) | C9—C10—H10 | 116.8 |
| C4—C3—C8 | 110.90 (8) | C11—C10—H10 | 116.8 |
| N1—C3—H3 | 107.9 | O2—C11—C10 | 123.97 (9) |
| C4—C3—H3 | 107.9 | O2—C11—N1 | 118.06 (9) |
| C8—C3—H3 | 107.9 | C10—C11—N1 | 117.51 (8) |
| C3—C4—C5 | 110.28 (9) | C13—C12—C17 | 119.30 (10) |
| C3—C4—H4A | 109.6 | C13—C12—C9 | 120.13 (9) |
| C5—C4—H4A | 109.6 | C17—C12—C9 | 120.55 (9) |
| C3—C4—H4B | 109.6 | C14—C13—C12 | 120.40 (10) |
| C5—C4—H4B | 109.6 | C14—C13—H13 | 119.8 |
| H4A—C4—H4B | 108.1 | C12—C13—H13 | 119.8 |
| C4—C5—C6 | 111.23 (10) | C15—C14—C13 | 119.66 (11) |

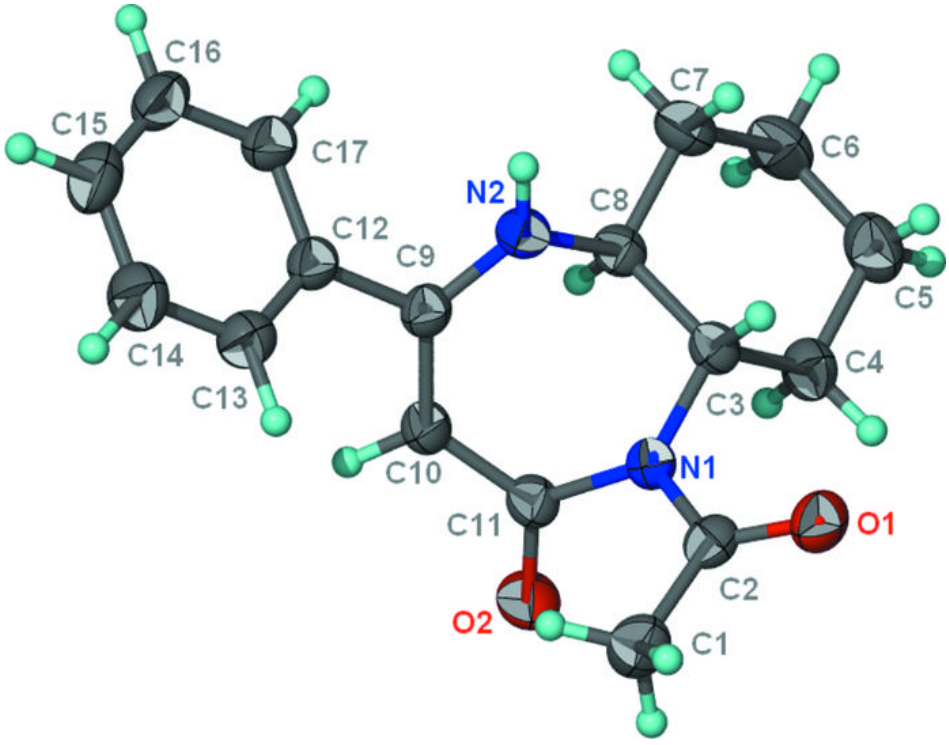
| | | | |
|---|---|---|---|
| C4—C5—H5A | 109.4 | C15—C14—H14 | 120.2 |
| C6—C5—H5A | 109.4 | C13—C14—H14 | 120.2 |
| C4—C5—H5B | 109.4 | C14—C15—C16 | 120.46 (11) |
| C6—C5—H5B | 109.4 | C14—C15—H15 | 119.8 |
| H5A—C5—H5B | 108.0 | C16—C15—H15 | 119.8 |
| C5—C6—C7 | 111.19 (9) | C17—C16—C15 | 119.92 (10) |
| C5—C6—H6A | 109.4 | C17—C16—H16 | 120.0 |
| C7—C6—H6A | 109.4 | C15—C16—H16 | 120.0 |
| C5—C6—H6B | 109.4 | C16—C17—C12 | 120.26 (11) |
| C7—C6—H6B | 109.4 | C16—C17—H17 | 119.9 |
| H6A—C6—H6B | 108.0 | C12—C17—H17 | 119.9 |
| C11—N1—C2—O1 | 168.01 (9) | C8—N2—C9—C12 | −158.47 (9) |
| C3—N1—C2—O1 | −7.51 (14) | N2—C9—C10—C11 | 24.81 (16) |
| C11—N1—C2—C1 | −16.81 (14) | C12—C9—C10—C11 | −159.20 (10) |
| C3—N1—C2—C1 | 167.66 (9) | C9—C10—C11—O2 | −174.30 (11) |
| C2—N1—C3—C4 | 81.59 (10) | C9—C10—C11—N1 | 13.72 (15) |
| C11—N1—C3—C4 | −93.80 (10) | C2—N1—C11—O2 | −55.02 (13) |
| C2—N1—C3—C8 | −154.61 (8) | C3—N1—C11—O2 | 120.27 (11) |
| C11—N1—C3—C8 | 30.00 (11) | C2—N1—C11—C10 | 117.44 (10) |
| N1—C3—C4—C5 | −179.46 (9) | C3—N1—C11—C10 | −67.27 (12) |
| C8—C3—C4—C5 | 57.29 (11) | N2—C9—C12—C13 | −138.24 (10) |
| C3—C4—C5—C6 | −55.11 (13) | C10—C9—C12—C13 | 45.54 (14) |
| C4—C5—C6—C7 | 55.70 (14) | N2—C9—C12—C17 | 43.26 (13) |
| C5—C6—C7—C8 | −57.34 (12) | C10—C9—C12—C17 | −132.96 (10) |
| C9—N2—C8—C3 | −80.75 (11) | C17—C12—C13—C14 | 0.29 (16) |
| C9—N2—C8—C7 | 155.86 (9) | C9—C12—C13—C14 | −178.23 (10) |
| N1—C3—C8—N2 | 52.05 (10) | C12—C13—C14—C15 | 0.27 (18) |
| C4—C3—C8—N2 | 176.50 (8) | C13—C14—C15—C16 | −0.56 (19) |
| N1—C3—C8—C7 | 176.16 (8) | C14—C15—C16—C17 | 0.29 (18) |
| C4—C3—C8—C7 | −59.39 (10) | C15—C16—C17—C12 | 0.27 (17) |
| C6—C7—C8—N2 | −176.16 (8) | C13—C12—C17—C16 | −0.56 (16) |
| C6—C7—C8—C3 | 58.84 (11) | C9—C12—C17—C16 | 177.95 (9) |
| C8—N2—C9—C10 | 17.64 (14) | | |

*Hydrogen-bond geometry (Å, °)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2···O1$^{i}$ | 0.88 (1) | 2.001 (9) | 2.854 (1) | 164 (1) |

Symmetry codes: (i) *x*, −*y*+3/2, *z*−1/2.

**Fig. 1**


C16
C15
C17
C7
C6
N2
C8
C12
C9
C5
C14
C4
C13
C3
C10
N1
C11
O1
C2
O2
C1