

(E)-Methyl 2-chloro-4-dicyclohexyl-amino-4-oxobut-2-enoate

Cai-Mei Liu, Fu-Ling Xue, Jian-Hua Fu and Zhao-Yang Wang*

School of Chemistry and Environment, South China Normal University, Guangzhou 510006, People's Republic of China

Correspondence e-mail: wangwangzhaoyang@tom.com

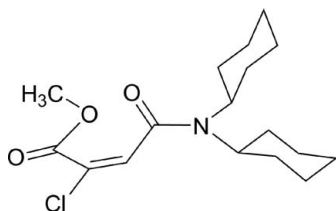
Received 11 May 2011; accepted 11 July 2011

Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.053; wR factor = 0.160; data-to-parameter ratio = 19.3.

In the title compound, $\text{C}_{17}\text{H}_{26}\text{ClNO}_3$, both cyclohexyl rings have chair conformations. In the crystal, molecules are linked by weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ hydrogen bonds.

Related literature

For the synthesis, see: Song *et al.* (2009). For the biological activity of 2(5*H*)-furanones, see: Lattmann *et al.* (2005); Rowland *et al.* (2007); Kim *et al.* (2002). For chemical, pharmaceutical and agrochemical applications of 3,4-amino-2(5*H*)-furanones, see: Kimura *et al.* (2000); Tanoury *et al.* (2008). For related structures, see: Lattmann *et al.* (1999, 2006).



Experimental

Crystal data

$\text{C}_{17}\text{H}_{26}\text{ClNO}_3$
 $M_r = 327.84$
 Monoclinic, $P2_1/c$
 $a = 8.8291$ (19) Å
 $b = 10.533$ (2) Å
 $c = 19.139$ (4) Å
 $\beta = 92.955$ (3)°

$V = 1777.5$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 0.23$ mm⁻¹
 $T = 298$ K
 $0.32 \times 0.22 \times 0.20$ mm

Data collection

Bruker APEXII area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{\min} = 0.931$, $T_{\max} = 0.956$

8635 measured reflections
 3886 independent reflections
 2457 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.051$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.160$
 $S = 1.02$
 3886 reflections

201 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.24$ e Å⁻³
 $\Delta\rho_{\min} = -0.28$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{C17}-\text{H17A}\cdots\text{O1}^{\text{i}}$	0.96	2.44	3.323 (4)	153
$\text{C9}-\text{H9}\cdots\text{O2}^{\text{ii}}$	0.93	2.50	3.389 (3)	160

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

Data collection: APEX2 (Bruker, 2008); cell refinement: SAINT (Bruker, 2008); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: SHELXL97.

This work was supported by the National Natural Science Foundation of China (grant No. 20772035) and the Natural Science Foundation of Guangdong Province, China (grant No. 5300082).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LX2191).

References

- Bruker (2008). APEX2 and SAINT. Bruker AXS Inc., Madison, Wisconsin, USA.
- Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
- Kim, Y., Nam, N.-H., You, Y.-J. & Ahn, B.-Z. (2002). *Bioorg. Med. Chem. Lett.* **12**, 719–722.
- Kimura, Y., Mizuno, T., Kawano, T., Okada, K. & Shimada, A. (2000). *Phytochemistry*, **53**, 829–831.
- Lattmann, E., Billington, D. C. & Langley, C. A. (1999). *Drug Des. Discov.* **16**, 243–250.
- Lattmann, E., Dunn, S., Niamsanit, S. & Sattayasai, N. (2005). *Bioorg. Med. Chem. Lett.* **15**, 919–921.
- Lattmann, E., Sattayasai, N., Schwalbe, C. S., Niamsanit, S., Billington, D. C., Lattmann, P., Langley, C. A., Singh, H. & Dunn, S. (2006). *Curr. Drug Discov. Technol.* **3**, 125–134.
- Rowland, S., Clark, P., Gordon, R., Mullen, A., Guay, J., Dufresne, L., Brideau, C., Cote, B., Ducharme, Y. & Mancini, J. (2007). *Eur. J. Pharmacol.* **560**, 216–224.
- Sheldrick, G. M. (1996). SADABS. University of Göttingen, Germany.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.
- Song, X.-M., Wang, Z.-Y., Li, J.-X. & Fu, J.-H. (2009). *Chin. J. Org. Chem.* **11**, 1804–1810.
- Tanoury, G. J., Chen, M. Z., Dong, Y., Forslund, R. E. & Magdziak, D. (2008). *Org. Lett.* **10**, 185–188.

supporting information

Acta Cryst. (2011). E67, o2051 [doi:10.1107/S1600536811027760]

(E)-Methyl 2-chloro-4-dicyclohexylamino-4-oxobut-2-enoate

Cai-Mei Liu, Fu-Ling Xue, Jian-Hua Fu and Zhao-Yang Wang

S1. Comment

Molecules possessing 2(5H)-furanone moiety are useful heterocyclic compounds due to their valuable biological activities such as antibacterial, anti-inflammatory and antitumor (Lattmann *et al.*, 2005; Rowland *et al.*, 2007; Kim *et al.*, 2002). The 5-alkoxy-3,4-dihalo-2(5H)-furanones being a kind of synthons are widely used in tandem Michael addition-elimination reactions (Song *et al.*, 2009). The 4-amino-2(5H)-furanones exhibit an antibiotic activity against MRSA (Lattmann *et al.*, 1999; Lattmann *et al.*, 2006). Therefore, we are interested in the tandem Michael addition-elimination reaction of the synthon 3,4-dichloro-5-methoxyfuran-2(5H)-one with secondary amines in the present of potassium fluoride. However, we obtained an unanticipated product, (E)-methyl 2-chloro-4-(dicyclohexylamino)-4-oxobut-2-enoate, instead of the expected compound 3-chloro-4-(dicyclohexylamino)-5-methoxyfuran-2(5H)-one (Lattmann *et al.*, 1999). Herein, we report the crystal structure of the title compound.

In the title compound (Fig. 1), the both cyclohexyl rings are in the chair form. The molecular packing (Fig. 2) is stabilized by weak intermolecular C—H \cdots O hydrogen bonds; the first one between the H atom of the vinyl group and the O atom of the ester group (Table 1; C9—H9 \cdots O2ⁱⁱ), and a methyl H atom and the O atom of the amide group (Table 1; C17—H17A \cdots O1ⁱ).

S2. Experimental

The precursor 3,4-dichloro-5-methoxyfuran-2(5H)-one was prepared according to the literature procedure (Song *et al.*, 2009). The solution of dicyclohexylamine (2.736 g, 3.0 mL) in tetrahydrofuran (3.0 mL) was added to a stirred solution of 3,4-dichloro-5-methoxyfuran-2(5H)-one (36.39 mg, 2.0 mmol) and potassium fluoride (34.85 mg, 6.0 mmol) in tetrahydrofuran (3.0 mL) under nitrogen atmosphere. After being stirred at room temperature for 24 h, the solvent was removed under reduced pressure. The residual solid was dissolved in dichloromethane and then the combined organic layer was concentrated under reduced pressure. The residue was purified by silica gel column chromatography with the gradient mixture of petroleum ether and ethyl acetate to give the title compound (yield 22.6%). Single crystals suitable for X-ray diffraction were prepared by slow evaporation of a solution of the title compound in (insert proper solvent) at room temperature.

S3. Refinement

H atoms were positioned in calculated positions with C—H = 0.93–0.98 Å and were refined using a riding model, with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl and $1.2U_{\text{eq}}(\text{C})$ for the others.

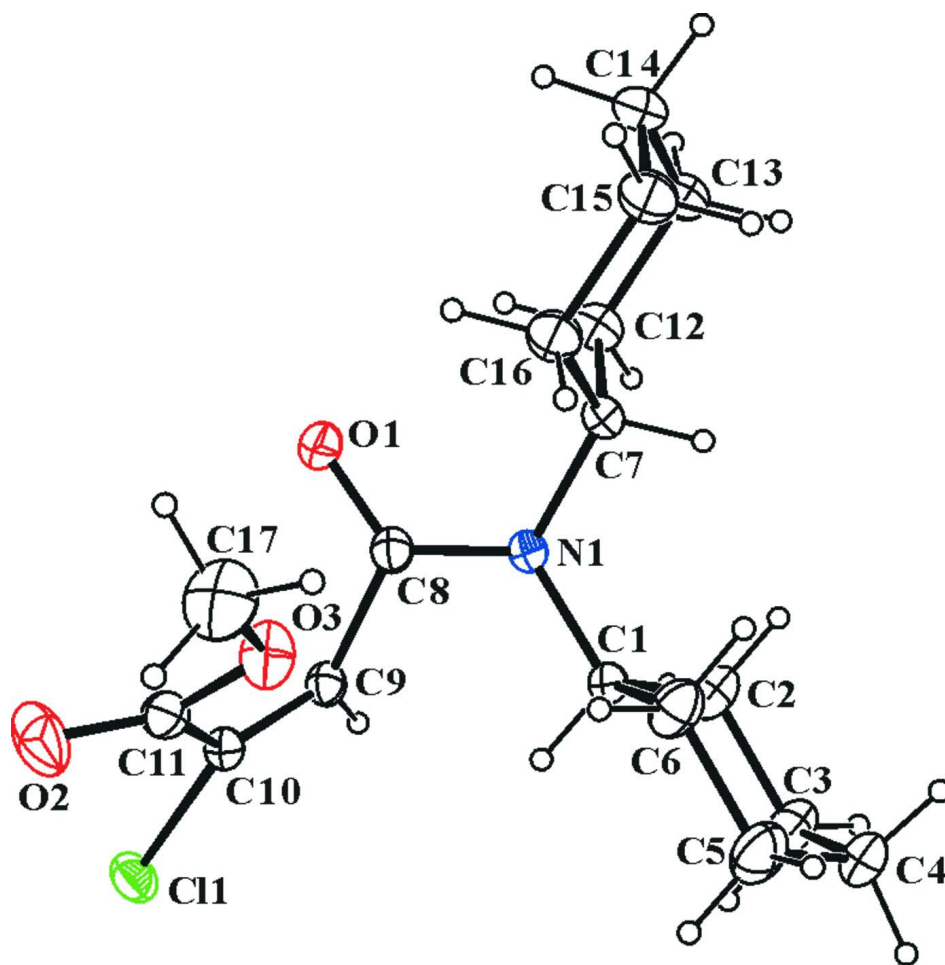
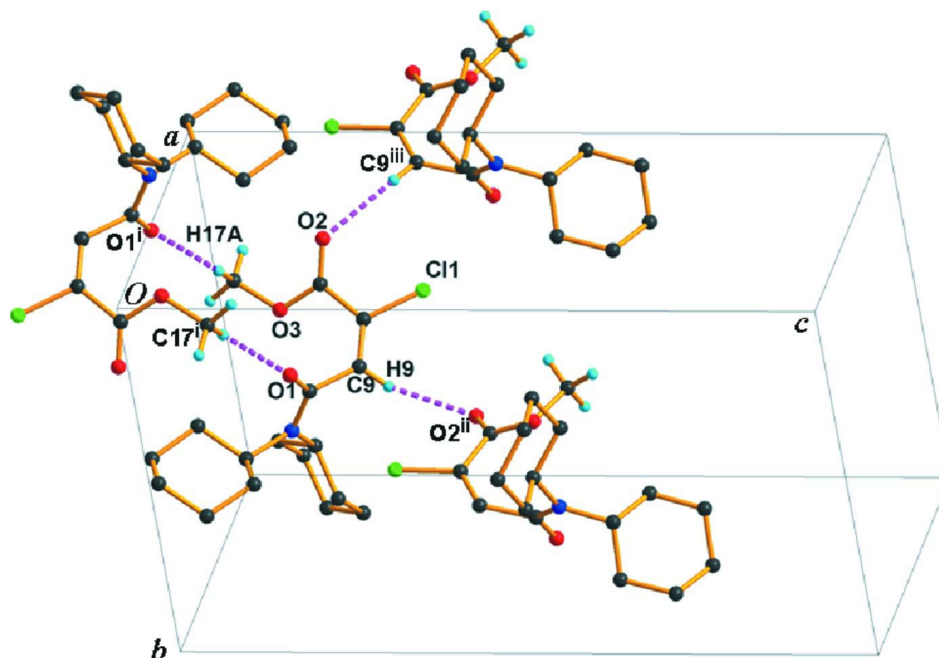


Figure 1

The molecular structure of the title compound showing the atom-labelling scheme. Ellipsoids are drawn at the 50% probability level. H atoms are presented as a small spheres of arbitrary radius.

**Figure 2**

A view of the C—H...O hydrogen bonds (dotted lines) in the crystal structure of the title compound. [Symmetry codes: (i) $-x + 2, -y + 1, -z$; (ii) $-x + 2, y + 1/2, -z + 1/2$; (iii) $-x + 2, y - 1/2, -z + 1/2$.]

(E)-Methyl 2-chloro-4-dicyclohexylamino-4-oxobut-2-enoate

Crystal data

$C_{17}H_{26}ClNO_3$

$M_r = 327.84$

Monoclinic, $P2_1/c$

Hall symbol: $-P\ 2ybc$

$a = 8.8291(19)\ \text{\AA}$

$b = 10.533(2)\ \text{\AA}$

$c = 19.139(4)\ \text{\AA}$

$\beta = 92.955(3)^\circ$

$V = 1777.5(6)\ \text{\AA}^3$

$Z = 4$

$F(000) = 704$

$D_x = 1.225\ \text{Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073\ \text{\AA}$

Cell parameters from 2253 reflections

$\theta = 2.2\text{--}23.5^\circ$

$\mu = 0.23\ \text{mm}^{-1}$

$T = 298\ \text{K}$

Block, colourless

$0.32 \times 0.22 \times 0.20\ \text{mm}$

Data collection

Bruker APEXII area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: $10.0\ \text{pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Sheldrick, 1996)

$T_{\min} = 0.931, T_{\max} = 0.956$

8635 measured reflections

3886 independent reflections

2457 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.051$

$\theta_{\max} = 27.0^\circ, \theta_{\min} = 2.1^\circ$

$h = -4 \rightarrow 11$

$k = -13 \rightarrow 12$

$l = -24 \rightarrow 24$

Refinement

Refinement on F^2
 Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.053$
 $wR(F^2) = 0.160$
 $S = 1.02$
 3886 reflections
 201 parameters
 0 restraints
 Primary atom site location: structure-invariant
 direct methods

Secondary atom site location: difference Fourier
 map
 Hydrogen site location: difference Fourier map
 H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.070P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -0.28 \text{ e } \text{\AA}^{-3}$
 Extinction correction: *SHELXL97* (Sheldrick,
 2008), $F_c^* = kF_c[1 + 0.001x F_c^2 \lambda^3 / \sin(2\theta)]^{-1/4}$
 Extinction coefficient: 0.015 (3)

Special details

Experimental. Data for (I): ^1H NMR (400 MHz, CDCl_3 , TMS): 1.073-1.564 (12H, *m*, 6 CH_2), 1.681-2.436 (8H, *m*, 4 CH_2), 2.973-3.124 (1H, *m*, CH), 3.350-3.417 (1H, *m*, CH), 3.813 (3H, *s*, CH_3), ESI-MS, *m/z* (%): Calcd for $\text{C}_{17}\text{H}_{27}\text{ClNO}_3^+([\text{M}+\text{H}]^+)$: 328.16(100.0), 330.16(32.6), found: 328.39(15.0), 330.43(5.0).

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 $F^2 > 2\sigma(F^2)$ 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>	$U_{\text{iso}}^*/U_{\text{eq}}$
Cl1	1.02330 (7)	0.47301 (6)	0.29004 (3)	0.0574 (2)
N1	0.7284 (2)	0.73071 (19)	0.10932 (8)	0.0427 (5)
C1	0.6049 (2)	0.7104 (2)	0.15716 (10)	0.0452 (6)
H1	0.6483	0.6628	0.1974	0.054*
C6	0.4768 (3)	0.6305 (3)	0.12475 (14)	0.0623 (7)
H6A	0.4338	0.6728	0.0833	0.075*
H6B	0.5167	0.5492	0.1105	0.075*
C3	0.4221 (3)	0.8114 (3)	0.23589 (14)	0.0711 (8)
H3A	0.4656	0.7696	0.2774	0.085*
H3B	0.3815	0.8924	0.2501	0.085*
C2	0.5460 (3)	0.8343 (3)	0.18519 (12)	0.0588 (7)
H2A	0.5062	0.8861	0.1465	0.071*
H2B	0.6288	0.8804	0.2088	0.071*
C4	0.2953 (3)	0.7311 (3)	0.20438 (14)	0.0662 (8)
H4A	0.2238	0.7126	0.2398	0.079*
H4B	0.2421	0.7781	0.1671	0.079*
C5	0.3537 (3)	0.6090 (3)	0.17577 (18)	0.0805 (9)
H5A	0.2704	0.5633	0.1523	0.097*
H5B	0.3937	0.5568	0.2142	0.097*
C9	0.9064 (2)	0.6357 (2)	0.19601 (10)	0.0407 (5)
H9	0.9020	0.6907	0.2338	0.049*

C10	0.9445 (2)	0.5170 (2)	0.20899 (10)	0.0414 (5)
C8	0.8696 (2)	0.6886 (2)	0.12389 (10)	0.0431 (5)
C7	0.6909 (3)	0.7947 (2)	0.04169 (10)	0.0469 (6)
H7	0.5843	0.8202	0.0426	0.056*
C11	0.9352 (3)	0.4105 (2)	0.15775 (11)	0.0519 (6)
C12	0.7807 (3)	0.9153 (3)	0.03266 (13)	0.0625 (7)
H12A	0.8878	0.8952	0.0320	0.075*
H12B	0.7662	0.9716	0.0718	0.075*
C16	0.7013 (3)	0.7045 (3)	-0.02024 (11)	0.0617 (7)
H16A	0.6369	0.6312	-0.0140	0.074*
H16B	0.8049	0.6752	-0.0233	0.074*
C13	0.7293 (4)	0.9818 (3)	-0.03560 (14)	0.0788 (10)
H13A	0.6254	1.0104	-0.0327	0.095*
H13B	0.7924	1.0557	-0.0423	0.095*
C15	0.6504 (4)	0.7749 (4)	-0.08734 (13)	0.0845 (11)
H15A	0.6612	0.7190	-0.1271	0.101*
H15B	0.5439	0.7967	-0.0855	0.101*
C14	0.7399 (4)	0.8930 (4)	-0.09763 (14)	0.0881 (12)
H14A	0.7018	0.9357	-0.1399	0.106*
H14B	0.8452	0.8710	-0.1033	0.106*
O1	0.97348 (18)	0.69654 (19)	0.08431 (8)	0.0573 (5)
O3	0.8279 (2)	0.43037 (19)	0.10887 (9)	0.0648 (5)
O2	1.0144 (3)	0.3193 (2)	0.16138 (10)	0.0985 (9)
C17	0.8142 (4)	0.3387 (4)	0.05276 (16)	0.0938 (11)
H17A	0.8939	0.3520	0.0213	0.141*
H17B	0.7177	0.3487	0.0279	0.141*
H17C	0.8218	0.2545	0.0718	0.141*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C11	0.0631 (4)	0.0582 (4)	0.0499 (3)	0.0121 (3)	-0.0066 (3)	0.0093 (2)
N1	0.0360 (10)	0.0521 (12)	0.0399 (9)	0.0072 (8)	0.0010 (7)	0.0075 (7)
C1	0.0338 (11)	0.0604 (16)	0.0417 (11)	0.0065 (10)	0.0025 (8)	0.0121 (10)
C6	0.0532 (15)	0.0544 (18)	0.0806 (17)	-0.0074 (12)	0.0148 (13)	-0.0088 (13)
C3	0.0494 (15)	0.105 (3)	0.0594 (14)	-0.0008 (15)	0.0108 (12)	-0.0137 (15)
C2	0.0438 (13)	0.0717 (19)	0.0617 (14)	-0.0059 (12)	0.0110 (11)	-0.0175 (12)
C4	0.0413 (14)	0.084 (2)	0.0744 (17)	0.0032 (13)	0.0129 (12)	0.0066 (14)
C5	0.0538 (17)	0.071 (2)	0.118 (2)	-0.0079 (15)	0.0236 (16)	0.0114 (17)
C9	0.0358 (11)	0.0439 (14)	0.0422 (10)	0.0024 (9)	-0.0014 (8)	-0.0004 (9)
C10	0.0362 (11)	0.0462 (14)	0.0417 (10)	0.0056 (9)	0.0023 (8)	0.0038 (9)
C8	0.0410 (12)	0.0448 (14)	0.0434 (11)	0.0036 (10)	-0.0004 (9)	-0.0023 (9)
C7	0.0440 (12)	0.0558 (15)	0.0408 (10)	0.0090 (11)	0.0004 (9)	0.0082 (9)
C11	0.0612 (15)	0.0469 (15)	0.0486 (12)	0.0088 (12)	0.0128 (11)	0.0018 (10)
C12	0.0655 (17)	0.0614 (19)	0.0607 (14)	0.0049 (14)	0.0044 (12)	0.0136 (12)
C16	0.0622 (16)	0.078 (2)	0.0440 (12)	0.0043 (14)	-0.0025 (11)	-0.0009 (11)
C13	0.081 (2)	0.079 (2)	0.0781 (19)	0.0246 (17)	0.0235 (16)	0.0392 (16)
C15	0.080 (2)	0.129 (3)	0.0432 (13)	0.022 (2)	-0.0034 (13)	0.0081 (15)

C14	0.092 (2)	0.119 (3)	0.0547 (15)	0.040 (2)	0.0198 (15)	0.0367 (17)
O1	0.0416 (9)	0.0774 (14)	0.0534 (9)	0.0111 (8)	0.0070 (7)	0.0084 (8)
O3	0.0602 (11)	0.0674 (13)	0.0654 (10)	0.0056 (9)	-0.0101 (9)	-0.0238 (9)
O2	0.150 (2)	0.0773 (17)	0.0678 (12)	0.0605 (16)	-0.0023 (13)	-0.0091 (10)
C17	0.107 (3)	0.096 (3)	0.0781 (19)	0.003 (2)	-0.0031 (18)	-0.0433 (18)

Geometric parameters (Å, °)

C11—C10	1.730 (2)	C8—O1	1.222 (2)
N1—C8	1.339 (3)	C7—C12	1.512 (4)
N1—C1	1.475 (3)	C7—C16	1.525 (3)
N1—C7	1.481 (3)	C7—H7	0.9800
C1—C2	1.514 (4)	C11—O2	1.189 (3)
C1—C6	1.517 (3)	C11—O3	1.313 (3)
C1—H1	0.9800	C12—C13	1.530 (3)
C6—C5	1.515 (3)	C12—H12A	0.9700
C6—H6A	0.9700	C12—H12B	0.9700
C6—H6B	0.9700	C16—C15	1.530 (4)
C3—C4	1.504 (4)	C16—H16A	0.9700
C3—C2	1.518 (3)	C16—H16B	0.9700
C3—H3A	0.9700	C13—C14	1.518 (5)
C3—H3B	0.9700	C13—H13A	0.9700
C2—H2A	0.9700	C13—H13B	0.9700
C2—H2B	0.9700	C15—C14	1.493 (5)
C4—C5	1.500 (4)	C15—H15A	0.9700
C4—H4A	0.9700	C15—H15B	0.9700
C4—H4B	0.9700	C14—H14A	0.9700
C5—H5A	0.9700	C14—H14B	0.9700
C5—H5B	0.9700	O3—C17	1.444 (3)
C9—C10	1.315 (3)	C17—H17A	0.9600
C9—C8	1.508 (3)	C17—H17B	0.9600
C9—H9	0.9300	C17—H17C	0.9600
C10—C11	1.489 (3)		
C8—N1—C1	122.19 (17)	N1—C8—C9	117.89 (18)
C8—N1—C7	119.78 (17)	N1—C7—C12	112.77 (19)
C1—N1—C7	117.97 (16)	N1—C7—C16	112.1 (2)
N1—C1—C2	111.9 (2)	C12—C7—C16	112.4 (2)
N1—C1—C6	112.71 (18)	N1—C7—H7	106.3
C2—C1—C6	111.2 (2)	C12—C7—H7	106.3
N1—C1—H1	106.9	C16—C7—H7	106.3
C2—C1—H1	106.9	O2—C11—O3	124.8 (2)
C6—C1—H1	106.9	O2—C11—C10	123.9 (2)
C5—C6—C1	111.3 (2)	O3—C11—C10	111.3 (2)
C5—C6—H6A	109.4	C7—C12—C13	110.4 (2)
C1—C6—H6A	109.4	C7—C12—H12A	109.6
C5—C6—H6B	109.4	C13—C12—H12A	109.6
C1—C6—H6B	109.4	C7—C12—H12B	109.6

H6A—C6—H6B	108.0	C13—C12—H12B	109.6
C4—C3—C2	112.3 (2)	H12A—C12—H12B	108.1
C4—C3—H3A	109.2	C7—C16—C15	108.9 (2)
C2—C3—H3A	109.2	C7—C16—H16A	109.9
C4—C3—H3B	109.2	C15—C16—H16A	109.9
C2—C3—H3B	109.2	C7—C16—H16B	109.9
H3A—C3—H3B	107.9	C15—C16—H16B	109.9
C1—C2—C3	111.2 (2)	H16A—C16—H16B	108.3
C1—C2—H2A	109.4	C14—C13—C12	111.0 (2)
C3—C2—H2A	109.4	C14—C13—H13A	109.4
C1—C2—H2B	109.4	C12—C13—H13A	109.4
C3—C2—H2B	109.4	C14—C13—H13B	109.4
H2A—C2—H2B	108.0	C12—C13—H13B	109.4
C5—C4—C3	111.5 (2)	H13A—C13—H13B	108.0
C5—C4—H4A	109.3	C14—C15—C16	112.3 (3)
C3—C4—H4A	109.3	C14—C15—H15A	109.1
C5—C4—H4B	109.3	C16—C15—H15A	109.1
C3—C4—H4B	109.3	C14—C15—H15B	109.1
H4A—C4—H4B	108.0	C16—C15—H15B	109.1
C4—C5—C6	112.2 (3)	H15A—C15—H15B	107.9
C4—C5—H5A	109.2	C15—C14—C13	110.9 (2)
C6—C5—H5A	109.2	C15—C14—H14A	109.5
C4—C5—H5B	109.2	C13—C14—H14A	109.5
C6—C5—H5B	109.2	C15—C14—H14B	109.5
H5A—C5—H5B	107.9	C13—C14—H14B	109.5
C10—C9—C8	124.41 (19)	H14A—C14—H14B	108.1
C10—C9—H9	117.8	C11—O3—C17	116.9 (2)
C8—C9—H9	117.8	O3—C17—H17A	109.5
C9—C10—C11	125.92 (19)	O3—C17—H17B	109.5
C9—C10—C11	120.73 (17)	H17A—C17—H17B	109.5
C11—C10—C11	113.26 (17)	O3—C17—H17C	109.5
O1—C8—N1	124.64 (19)	H17A—C17—H17C	109.5
O1—C8—C9	117.33 (19)	H17B—C17—H17C	109.5
C8—N1—C1—C2	116.2 (2)	C10—C9—C8—N1	115.7 (3)
C7—N1—C1—C2	-66.8 (3)	C8—N1—C7—C12	-61.9 (3)
C8—N1—C1—C6	-117.6 (2)	C1—N1—C7—C12	121.0 (2)
C7—N1—C1—C6	59.4 (3)	C8—N1—C7—C16	66.2 (3)
N1—C1—C6—C5	178.7 (2)	C1—N1—C7—C16	-110.9 (2)
C2—C1—C6—C5	-54.7 (3)	C9—C10—C11—O2	152.5 (3)
N1—C1—C2—C3	-178.38 (19)	C11—C10—C11—O2	-23.9 (3)
C6—C1—C2—C3	54.6 (3)	C9—C10—C11—O3	-28.0 (3)
C4—C3—C2—C1	-54.5 (3)	C11—C10—C11—O3	155.65 (17)
C2—C3—C4—C5	53.9 (4)	N1—C7—C12—C13	-176.0 (2)
C3—C4—C5—C6	-53.9 (3)	C16—C7—C12—C13	56.0 (3)
C1—C6—C5—C4	54.5 (3)	N1—C7—C16—C15	176.1 (2)
C8—C9—C10—C11	-10.0 (4)	C12—C7—C16—C15	-55.7 (3)
C8—C9—C10—C11	166.08 (16)	C7—C12—C13—C14	-55.2 (3)

C1—N1—C8—O1	175.6 (2)	C7—C16—C15—C14	56.3 (3)
C7—N1—C8—O1	-1.4 (4)	C16—C15—C14—C13	-57.4 (3)
C1—N1—C8—C9	-8.9 (3)	C12—C13—C14—C15	56.2 (4)
C7—N1—C8—C9	174.2 (2)	O2—C11—O3—C17	-5.0 (4)
C10—C9—C8—O1	-68.4 (3)	C10—C11—O3—C17	175.5 (2)

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C17—H17A \cdots O1 ⁱ	0.96	2.44	3.323 (4)	153
C9—H9 \cdots O2 ⁱⁱ	0.93	2.50	3.389 (3)	160

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