IUCrData

ISSN 2414-3146

2-(4-Hydroxyphenyl)-4,6-dimethyl-2,3-dihydropyrimidin-1-ium acetate

Kedila Rasheeda,[a] Seranthimata Samshuddin,[b] Phadke N. Swathi,[b] Vijaya D. P. Alva,[a] Joel T. Mague[c] and Youssef Ramli[d]*

[a]Department of Chemistry, Shree Devi Institute of Technology, Kenjar, Mangalore, Karnataka 574 142, India, [b]Department of Chemistry, Sri Dharmasthala Manjunatheshwara Institute of, Technology, Ujire, Karnataka 574 240, India, [c]Department of Chemistry, Tulane University, New Orleans, LA 70118, USA, and [d]Laboratory of Medicinal Chemistry, Faculty of Medicine and Pharmacy, Research Center for Medicinal Sciences Mohammed V University, Rabat, Morocco. *Correspondence e-mail: y.ramli@um5s.net.ma

Received 9 July 2018
Accepted 20 July 2018

Edited by L. Van Meervelt, Katholieke Universiteit Leuven, Belgium

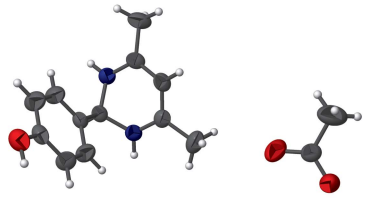
Keywords: crystal structure; hydrogen bond; dihydropyrimidinium.

CCDC reference: 1857057

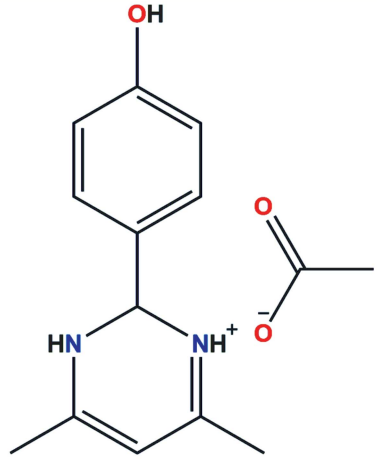
Structural data: full structural data are available from iucrdata.iucr.org

In the title compound, $C_{12}H_{15}N_2O^+\cdot C_2H_3O_2^-$, the phenoxy group is nearly perpendicular [80.73 (11)°] to the dihydropyrimidinium ring. In the crystal, O—H···O, N—H···O and C—H···O hydrogen bonds form corrugated layers parallel to the *ac* plane.

3D view



Chemical scheme



Structure description

Pyrimidine and its derivatives are bioactive molecules and play an important role in several biological processes (Selvam *et al.*, 2012). These derivatives have also been used in coordination chemistry and in corrosion inhibitors (Ansari *et al.*, 2015). Several methods have been proposed for the synthesis of pyrimidine derivatives (Gore & Rajput, 2013). The crystal structures of several pyrimidine derivatives have been reported (Fun *et al.*, 2012). In view of the importance of pyrimidine derivatives, a new pyrimidine derivative is synthesized and the crystal structure has been determined (Fig. 1).

The dihydropyrimidinium ring adopts an envelope conformation with puckering parameters Q = 0.419 (2) Å, θ = 108.3 (3)° and φ = 237.7 (3)°. The phenoxy ring is nearly perpendicular to the dihydropyrimidinium ring, as indicated by the dihedral angle of 80.73 (11)° between the mean planes of the two rings. In the crystal, O1—H1*B*···O3, N1—H1···O2, N2—H2···O3 and C1—H1*A*···O1 hydrogen bonds (Table 1) link the molecules into corrugated layers parallel to the *ac* plane with one of the methyl groups on the dihydropyrimidinium ring protruding from each surface of the layer (Figs. 2 and 3).

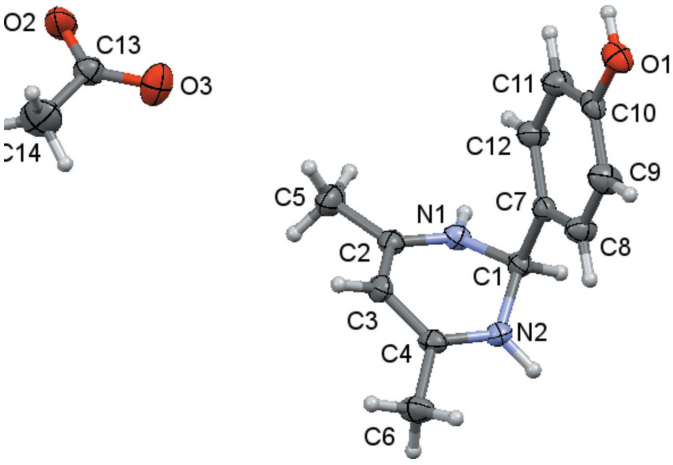


Figure 1
The title molecule with labeling scheme and 30% probability ellipsoids.

Synthesis and crystallization

A mixture of 4-hydroxy benzaldehyde (0.01 mol), acetyl acetone (0.01 mol) and ammonium acetate (5 g) was refluxed for 8 h in 30 ml of acetic acid. The reaction mixture was cooled to room temperature and the solid product obtained was filtered and recrystallized from ethanol. Single crystals were grown from ethanol by the slow evaporation method (yield 67%, m.p. 529 K).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

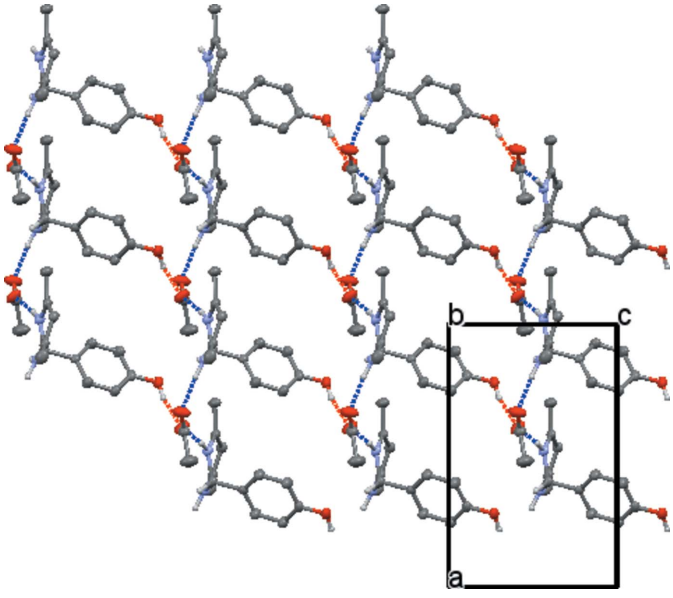


Figure 2
A portion of one corrugated layer viewed along the *b*-axis direction. O—H···O and N—H···O hydrogen bonds are shown, respectively, by red and blue dashed lines. The C—H···O hydrogen bonds are omitted for clarity.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O3^{i}	0.87	1.76	2.609 (3)	165
N1—H1···O2^{ii}	0.91	1.83	2.731 (3)	172
N2—H2···O3^{iii}	0.91	1.87	2.771 (2)	170
C1—H1*A*···O1^{iv}	0.98	2.31	3.214 (3)	154

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

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{15}N_2O^+\cdot C_2H_3O_2^-$
M_r	262.30
Crystal system, space group	Orthorhombic, *Pna*2$_1$
Temperature (K)	296
a, *b*, *c* (Å)	12.2836 (4), 14.5343 (5), 7.8596 (3)
V (Å^3)	1403.20 (9)
Z	4
Radiation type	Cu *K*α
μ (mm^{-1})	0.72
Crystal size (mm)	0.35 × 0.24 × 0.06
Data collection	
Diffractometer	Bruker D8 VENTURE PHOTON 100 CMOS
Absorption correction	Multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min}, T_{max}	0.79, 0.95
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	9762, 2702, 2611
R_{int}	0.042
(sin θ/λ)$_{max}$ (Å^{-1})	0.618
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.035, 0.093, 1.08
No. of reflections	2702
No. of parameters	176
No. of restraints	1
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.16, −0.15
Absolute structure	Flack *x* determined using 1136 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	0.03 (8)

Computer programs: *APEX3* and *SAINT* (Bruker, 2016), *SHELXT* (Sheldrick, 2015*a*), *SHELXL2018* (Sheldrick, 2015*b*), *Mercury* (Macrae *et al.*, 2008) and *SHELXTL* (Sheldrick, 2008).

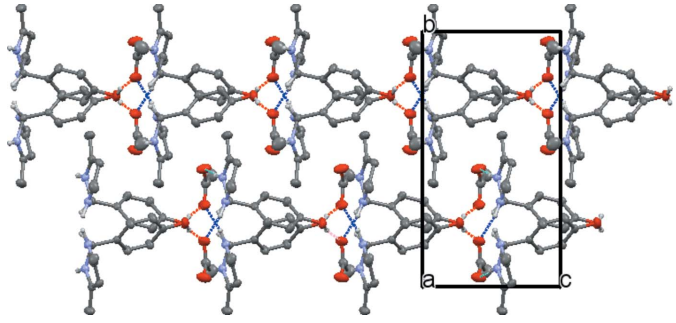


Figure 3
Elevation view of two layers seen along the *a*-axis direction. Hydrogen bonds are depicted as in Fig. 2.

Funding information

The support of NSF–MRI grant No. 1228232 for the purchase of the diffractometer and Tulane University for support of the Tulane Crystallography Laboratory are gratefully acknowledged. KR is grateful to the Directorate of Minorities, Government of Karnataka, for providing a research fellowship.

References

Ansari, K. R., Sudheer, Singh, A. & Quraishi, M. A. (2015). *J. Dispersion Sci. Technol.* **36**, 908–917.
Bruker (2016). *APEX3*, *SADABS* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fun, H.-K., Chia, T. S., Samshuddin, S., Narayana, B. & Sarojini, B. K. (2012). *Acta Cryst.* E**68**, o807–o808.
Gore, R. P. & Rajput, A. P. (2013). *Drug Invention Today*, **5**, 148–152.
Krause, L., Herbst-Irmer, R., Sheldrick, G. M. & Stalke, D. (2015). *J. Appl. Cryst.* **48**, 3–10.
Macrae, C. F., Bruno, I. J., Chisholm, J. A., Edgington, P. R., McCabe, P., Pidcock, E., Rodriguez-Monge, L., Taylor, R., van de Streek, J. & Wood, P. A. (2008). *J. Appl. Cryst.* **41**, 466–470.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Selvam, T. P., James, C. R., Dniandev, P. V. & Valzita, S. K. (2012). *Res. Pharm.* **2**, 1–9.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.

full crystallographic data

IUCrData (2018). **3**, x181046 [https://doi.org/10.1107/S2414314618010465]

2-(4-Hydroxyphenyl)-4,6-dimethyl-2,3-dihydropyrimidin-1-ium acetate

Kedila Rasheeda, Seranthimata Samshuddin, Phadke N. Swathi, Vijaya D. P. Alva, Joel T. Mague and Youssef Ramli

2-(4-Hydroxyphenyl)-4,6-dimethyl-2,3-dihydropyrimidin-1-ium acetate

Crystal data

$C_{12}H_{15}N_2O^+·C_2H_3O_2^-$
M_r = 262.30
Orthorhombic, $Pna2_1$
a = 12.2836 (4) Å
b = 14.5343 (5) Å
c = 7.8596 (3) Å
V = 1403.20 (9) Å^3
Z = 4
F(000) = 560
D_x = 1.242 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 9919 reflections
θ = 3.0–72.5°
μ = 0.72 mm^{-1}
T = 296 K
Plate, amber
0.35 × 0.24 × 0.06 mm

Data collection

Bruker D8 VENTURE PHOTON 100 CMOS diffractometer
Radiation source: INCOATEC IμS micro-focus source
Mirror monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Krause *et al.*, 2015)
T_{min} = 0.79, T_{max} = 0.95
9762 measured reflections
2702 independent reflections
2611 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 72.4°, θ_{min} = 4.7°
h = −14→15
k = −17→14
l = −9→9

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.035
$wR(F^2)$ = 0.093
S = 1.08
2702 reflections
176 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: mixed
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0477P)^2 + 0.1696P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.16 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Extinction correction: *SHELXL2018* (Sheldrick, 2015*b*), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.044 (3)
Absolute structure: Flack x determined using 1136 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter: 0.03 (8)

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. H-atoms attached to carbon were placed in calculated positions (C—H = 0.95 - 1.00 Å) while those attached to nitrogen and oxygen were placed in locations derived from a difference map and their coordinates adjusted to give N—H = 0.91 and O—H = 0.87 %A. All were included as riding contributions with isotropic displacement parameters 1.2 - 1.5 times those of the attached atoms.

H-atoms attached to carbon were placed in calculated positions while those attached to nitrogen and oxygen were placed in locations derived from a difference map and their coordinates adjusted to give N—H = 0.91 and O—H = 0.87 Å. All were included as riding contributions.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.27724 (14)	0.75133 (12)	0.7694 (2)	0.0546 (4)
H1B	0.217782	0.722953	0.798867	0.082*
N1	0.36392 (14)	0.57083 (11)	0.0439 (2)	0.0420 (4)
H1	0.298478	0.563493	−0.008622	0.050*
N2	0.51651 (14)	0.66729 (12)	0.0645 (2)	0.0420 (4)
H2	0.545084	0.721104	0.025708	0.050*
C1	0.39948 (16)	0.66268 (13)	0.0918 (3)	0.0390 (4)
H1A	0.364086	0.707373	0.016533	0.047*
C2	0.42312 (18)	0.49979 (14)	0.0923 (3)	0.0433 (5)
C3	0.52904 (18)	0.51327 (15)	0.1474 (3)	0.0462 (5)
H3	0.565920	0.467961	0.207922	0.055*
C4	0.57816 (17)	0.59665 (16)	0.1094 (3)	0.0434 (5)
C5	0.3741 (2)	0.40638 (17)	0.0759 (4)	0.0654 (7)
H5A	0.300785	0.411806	0.034710	0.098*
H5B	0.373633	0.376778	0.185129	0.098*
H5C	0.416204	0.370418	−0.002539	0.098*
C6	0.69929 (19)	0.6085 (2)	0.1048 (4)	0.0627 (7)
H6A	0.723330	0.611386	−0.011310	0.094*
H6B	0.733401	0.557281	0.160521	0.094*
H6C	0.718748	0.664425	0.162220	0.094*
C7	0.36783 (16)	0.68454 (13)	0.2754 (3)	0.0378 (4)
C8	0.42471 (19)	0.74749 (17)	0.3707 (3)	0.0520 (5)
H8	0.485363	0.776132	0.323659	0.062*
C9	0.3937 (2)	0.76929 (18)	0.5356 (3)	0.0594 (7)
H9	0.433786	0.811862	0.597828	0.071*
C10	0.30388 (18)	0.72830 (14)	0.6075 (3)	0.0428 (5)
C11	0.2443 (2)	0.66604 (16)	0.5124 (3)	0.0523 (6)
H11	0.182600	0.638735	0.558644	0.063*
C12	0.2766 (2)	0.64423 (17)	0.3479 (3)	0.0519 (6)

H12	0.236315	0.601912	0.285269	0.062*
O2	0.33630 (14)	0.03653 (13)	0.4068 (4)	0.0796 (7)
O3	0.39509 (17)	0.17870 (13)	0.4094 (3)	0.0729 (6)
C13	0.4095 (2)	0.09335 (17)	0.4226 (3)	0.0529 (5)
C14	0.5234 (3)	0.0608 (3)	0.4529 (7)	0.0972 (13)
H14A	0.573848	0.106519	0.413293	0.146*
H14B	0.535285	0.004326	0.392359	0.146*
H14C	0.534262	0.050737	0.572402	0.146*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0600 (10)	0.0655 (10)	0.0384 (8)	0.0097 (8)	0.0013 (7)	−0.0062 (7)
N1	0.0390 (9)	0.0462 (9)	0.0406 (9)	0.0000 (7)	0.0013 (7)	−0.0045 (7)
N2	0.0417 (9)	0.0411 (8)	0.0432 (9)	−0.0018 (7)	0.0082 (8)	0.0061 (7)
C1	0.0404 (10)	0.0396 (9)	0.0370 (9)	0.0037 (8)	0.0009 (8)	0.0027 (8)
C2	0.0484 (11)	0.0427 (10)	0.0387 (10)	−0.0018 (9)	0.0100 (9)	−0.0010 (8)
C3	0.0478 (12)	0.0442 (11)	0.0467 (11)	0.0080 (9)	0.0029 (9)	0.0104 (9)
C4	0.0389 (10)	0.0543 (11)	0.0371 (9)	0.0017 (9)	0.0046 (9)	0.0048 (8)
C5	0.0803 (18)	0.0455 (12)	0.0705 (17)	−0.0120 (12)	0.0148 (15)	−0.0064 (12)
C6	0.0378 (11)	0.0830 (17)	0.0674 (15)	0.0003 (11)	0.0060 (11)	0.0124 (14)
C7	0.0367 (9)	0.0366 (8)	0.0401 (10)	0.0066 (7)	0.0015 (8)	0.0008 (8)
C8	0.0480 (12)	0.0565 (12)	0.0514 (12)	−0.0106 (10)	0.0064 (10)	−0.0084 (11)
C9	0.0567 (14)	0.0671 (15)	0.0545 (14)	−0.0102 (12)	0.0038 (11)	−0.0207 (12)
C10	0.0481 (11)	0.0443 (10)	0.0362 (10)	0.0132 (9)	−0.0017 (9)	−0.0005 (8)
C11	0.0503 (12)	0.0588 (12)	0.0478 (13)	−0.0075 (10)	0.0104 (10)	−0.0066 (10)
C12	0.0515 (13)	0.0561 (13)	0.0480 (12)	−0.0117 (11)	0.0078 (10)	−0.0114 (10)
O2	0.0478 (10)	0.0576 (11)	0.133 (2)	−0.0061 (8)	0.0135 (12)	−0.0193 (12)
O3	0.0754 (12)	0.0562 (10)	0.0873 (15)	−0.0097 (9)	−0.0357 (11)	−0.0046 (9)
C13	0.0454 (12)	0.0571 (13)	0.0562 (13)	−0.0040 (10)	−0.0018 (11)	−0.0058 (11)
C14	0.0515 (16)	0.111 (3)	0.129 (4)	0.0017 (16)	−0.0141 (19)	0.022 (3)

Geometric parameters (Å, º)

O1—C10	1.356 (3)	C6—H6B	0.9600
O1—H1B	0.8700	C6—H6C	0.9600
N1—C2	1.319 (3)	C7—C8	1.373 (3)
N1—C1	1.454 (3)	C7—C12	1.387 (3)
N1—H1	0.9099	C8—C9	1.388 (3)
N2—C4	1.324 (3)	C8—H8	0.9300
N2—C1	1.455 (3)	C9—C10	1.375 (3)
N2—H2	0.9100	C9—H9	0.9300
C1—C7	1.528 (3)	C10—C11	1.383 (3)
C1—H1A	0.9800	C11—C12	1.389 (3)
C2—C3	1.385 (3)	C11—H11	0.9300
C2—C5	1.491 (3)	C12—H12	0.9300
C3—C4	1.386 (3)	O2—C13	1.227 (3)
C3—H3	0.9300	O3—C13	1.257 (3)

C4—C6	1.498 (3)	C13—C14	1.497 (4)
C5—H5A	0.9600	C14—H14A	0.9600
C5—H5B	0.9600	C14—H14B	0.9600
C5—H5C	0.9600	C14—H14C	0.9600
C6—H6A	0.9600		

C10—O1—H1B	109.6	C4—C6—H6C	109.5
C2—N1—C1	118.59 (18)	H6A—C6—H6C	109.5
C2—N1—H1	121.8	H6B—C6—H6C	109.5
C1—N1—H1	119.4	C8—C7—C12	118.0 (2)
C4—N2—C1	119.38 (17)	C8—C7—C1	121.60 (19)
C4—N2—H2	122.4	C12—C7—C1	120.37 (19)
C1—N2—H2	118.0	C7—C8—C9	121.4 (2)
N1—C1—N2	107.52 (16)	C7—C8—H8	119.3
N1—C1—C7	111.02 (16)	C9—C8—H8	119.3
N2—C1—C7	112.37 (17)	C10—C9—C8	120.4 (2)
N1—C1—H1A	108.6	C10—C9—H9	119.8
N2—C1—H1A	108.6	C8—C9—H9	119.8
C7—C1—H1A	108.6	O1—C10—C9	118.2 (2)
N1—C2—C3	119.82 (19)	O1—C10—C11	122.8 (2)
N1—C2—C5	117.7 (2)	C9—C10—C11	119.0 (2)
C3—C2—C5	122.4 (2)	C10—C11—C12	120.1 (2)
C2—C3—C4	117.71 (19)	C10—C11—H11	119.9
C2—C3—H3	121.1	C12—C11—H11	119.9
C4—C3—H3	121.1	C7—C12—C11	121.1 (2)
N2—C4—C3	119.11 (19)	C7—C12—H12	119.5
N2—C4—C6	118.2 (2)	C11—C12—H12	119.5
C3—C4—C6	122.5 (2)	O2—C13—O3	123.6 (2)
C2—C5—H5A	109.5	O2—C13—C14	119.3 (3)
C2—C5—H5B	109.5	O3—C13—C14	117.1 (2)
H5A—C5—H5B	109.5	C13—C14—H14A	109.5
C2—C5—H5C	109.5	C13—C14—H14B	109.5
H5A—C5—H5C	109.5	H14A—C14—H14B	109.5
H5B—C5—H5C	109.5	C13—C14—H14C	109.5
C4—C6—H6A	109.5	H14A—C14—H14C	109.5
C4—C6—H6B	109.5	H14B—C14—H14C	109.5
H6A—C6—H6B	109.5		

C2—N1—C1—N2	43.2 (2)	N2—C1—C7—C8	34.0 (3)
C2—N1—C1—C7	−80.1 (2)	N1—C1—C7—C12	−28.8 (3)
C4—N2—C1—N1	−41.8 (3)	N2—C1—C7—C12	−149.3 (2)
C4—N2—C1—C7	80.6 (2)	C12—C7—C8—C9	1.2 (4)
C1—N1—C2—C3	−16.4 (3)	C1—C7—C8—C9	177.9 (2)
C1—N1—C2—C5	166.9 (2)	C7—C8—C9—C10	−0.4 (4)
N1—C2—C3—C4	−16.2 (3)	C8—C9—C10—O1	179.5 (2)
C5—C2—C3—C4	160.4 (2)	C8—C9—C10—C11	−0.8 (4)
C1—N2—C4—C3	13.4 (3)	O1—C10—C11—C12	−179.1 (2)
C1—N2—C4—C6	−170.9 (2)	C9—C10—C11—C12	1.3 (4)

C2—C3—C4—N2	17.7 (3)	C8—C7—C12—C11	−0.7 (4)
C2—C3—C4—C6	−157.9 (2)	C1—C7—C12—C11	−177.5 (2)
N1—C1—C7—C8	154.5 (2)	C10—C11—C12—C7	−0.5 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*B*···O3^{i}	0.87	1.76	2.609 (3)	165
N1—H1···O2^{ii}	0.91	1.83	2.731 (3)	172
N2—H2···O3^{iii}	0.91	1.87	2.771 (2)	170
C1—H1*A*···O1^{iv}	0.98	2.31	3.214 (3)	154

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