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rac-Ethyl 4-hydroxy-6-(2-hydroxyphenyl)-2-oxo-4-(trifluoromethyl)perhydropyrimidine-5-carboxylate

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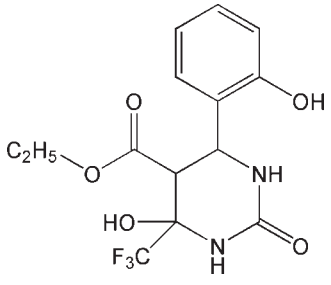
Key indicators: single-crystal X-ray study; T = 113 K; mean σ(C–C) = 0.002 Å; R factor = 0.033; wR factor = 0.075; data-to-parameter ratio = 15.2.

In the title compound, $C_{14}H_{15}F_3N_2O_5$, prepared by reaction of 2-hydroxybenzaldehyde, ethyl 4,4,4-trifluoro-3-oxobutanoate and urea, the tetrapyrimidine ring adopts a half-chair conformation. The crystal structure is stabilized by five intermolecular hydrogen bonds, three O—H···O and two N—H···O, giving cyclic dimers (through three hydrogen bonds) which are further extended into a two-dimensional network.

Related literature

For the bioactivity of dihydropyrimidines, see: Brier *et al.* (2004); Cochran *et al.* (2005); Moran *et al.* (2007); Zorkun *et al.* (2006). For the bioactivity of organofluorine compounds, see: Hermann *et al.* (2003); Ulrich (2004).



Experimental

Crystal data

$C_{14}H_{15}F_3N_2O_5$
M_r = 348.28
Monoclinic, $P2_1/n$
a = 12.0940 (15) Å
b = 8.665 (1) Å
c = 14.3110 (18) Å
β = 93.987 (6)°
V = 1496.1 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 113 K
0.26 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
T_{min} = 0.965, T_{max} = 0.973
15222 measured reflections
3558 independent reflections
2461 reflections with $I > 2\sigma(I)$
R_{int} = 0.043

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.96
3558 reflections
234 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H4···O1^{i}	0.836 (14)	2.244 (14)	3.0760 (13)	174.1 (14)
N2—H5···O2^{ii}	0.867 (14)	2.040 (15)	2.9064 (14)	177.4 (13)
O1—H1···O5^{i}	0.858 (17)	2.588 (16)	3.0983 (13)	119.2 (13)
O1—H1···O3^{i}	0.858 (17)	2.013 (17)	2.8232 (13)	157.1 (15)
O5—H6···O2^{iii}	0.912 (16)	1.759 (17)	2.6685 (12)	175.8 (14)

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

Data collection: *CrystalClear-SM Expert* (Rigaku, 2009); cell refinement: *CrystalClear-SM Expert*; data reduction: *CrystalClear-SM Expert*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalStructure* (Rigaku, 2009); software used to prepare material for publication: *CrystalStructure*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZS2042).

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supporting information

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rac-Ethyl 4-hydroxy-6-(2-hydroxyphenyl)-2-oxo-4-(trifluoromethyl)perhydropyrimidine-5-carboxylate

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S1. Comment

Dihydropyrimidine (DHPM) derivatives can be used as potential calcium channel blockers (Zorkun *et al.*, 2006), inhibitors of mitotic kinesin Eg5 for treating cancer (Cochran *et al.*, 2005; Brier *et al.*, 2004) and as TRPA1 modulators for treating pain (Moran *et al.*, 2007). In addition, compounds that contain fluorine have special bioactivity, *e.g.* flumioxazin is a widely used herbicide (Hermann *et al.*, 2003; Ulrich, 2004). This led us to focus our attention on the synthesis and bioactivity of these important fused perfluoroalkylated heterocyclic compounds. During the synthesis of DHPM derivatives, the title compound, an intermediate $C_{14}H_{15}F_3N_2O_5$ (I) was isolated and the structure confirmed by X-ray diffraction, in order to elucidate the reaction mechanism.

In the structure of the title molecule, the dihydropyrimidine ring adopts a half-chair conformation. The crystal structure is stabilized by five intermolecular hydrogen bonds, three O—H···O and two N—H···O (Table 1), giving cyclic dimers which are further extended into a two-dimensional network (Fig. 2).

dimension?

S2. Experimental

The title compound was synthesized refluxing for 3 h, a stirred solution of 2-hydroxybenzaldehyde (0.61 g, 5 mmol), ethyl 4,4,4-trifluoro-3-oxobutanoate (1.11 g, 6 mmol) and urea (0.45 g, 7.5 mmol) in 5 ml of ethanol, the reaction catalyzed by sulfamic acid (0.15 g). The solvent was evaporated *in vacuo* and the residue was washed with water. The title compound was recrystallized from 50% aqueous ethanol and single crystals of (I) were obtained by slow evaporation.

S3. Refinement

Hydrogen atoms involved in hydrogen-bonding inetractions were located by difference methods and their positional and isotropic displacement parameters were refined. Other H atoms were placed in calculated positions, with C—H(aromatic) = 0.95 Å and C—H(aliphatic) = 0.98 or 0.99 Å, and treated as riding, with U_{iso}(H) = 1.2Ueq(C).

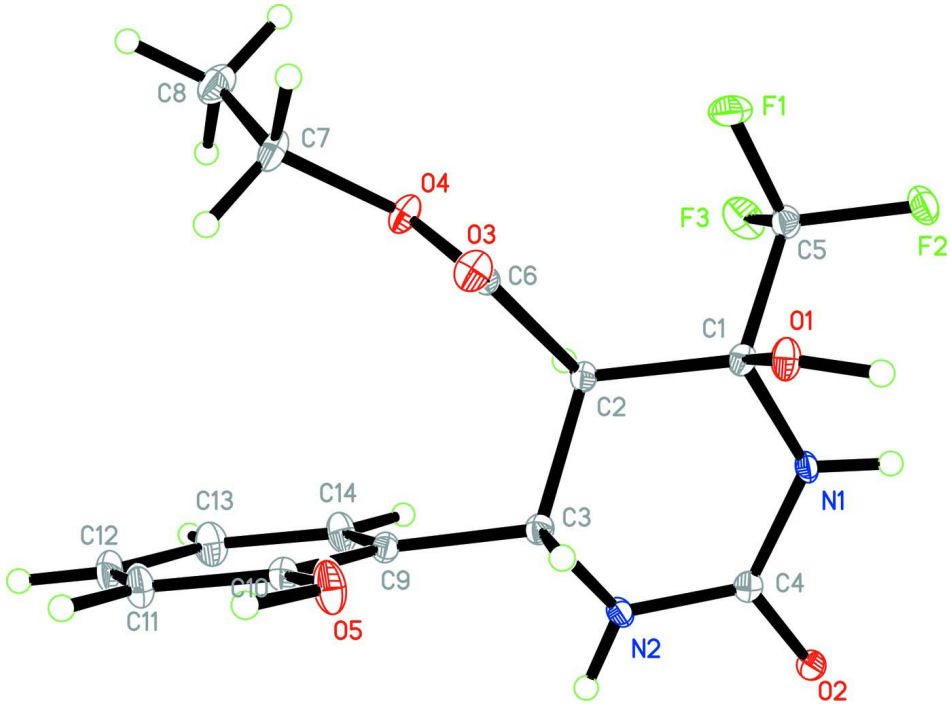

Figure 1

Molecular configuration and atom numbering scheme for (I), with displacement ellipsoids drawn at the 30% probability level.

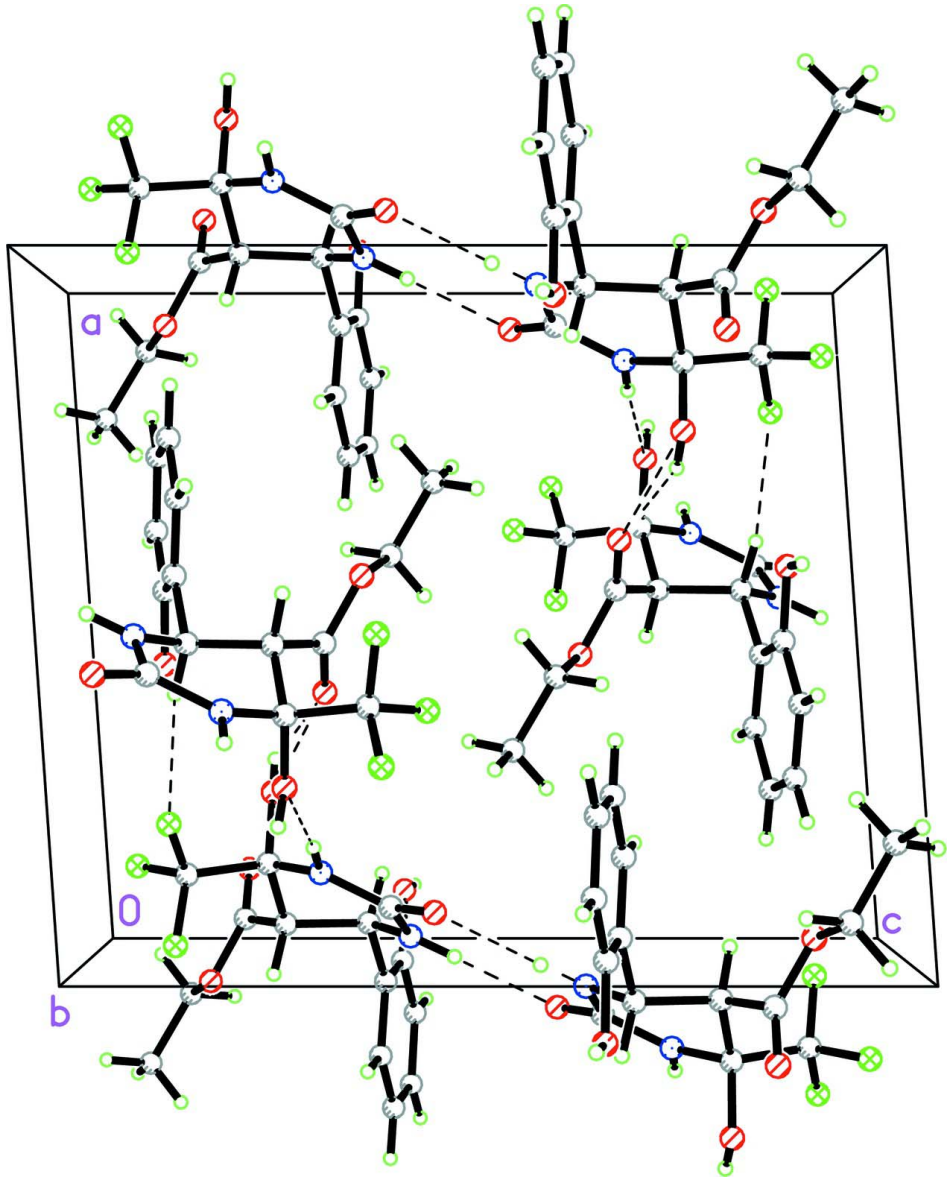

Figure 2

The packing diagram of the title compound. Intermolecular hydrogen bonds are shown as dashed line.

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Crystal data

$C_{14}H_{15}F_3N_2O_5$
$M_r = 348.28$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 12.0940\ (15)$ Å
$b = 8.665\ (1)$ Å
$c = 14.3110\ (18)$ Å
$\beta = 93.987\ (6)°$
$V = 1496.1\ (3)$ Å^3
$Z = 4$

$F(000) = 720$
$D_x = 1.546$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71075$ Å
Cell parameters from 4600 reflections
$\theta = 1.7–28.0°$
$\mu = 0.14$ mm^{-1}
$T = 113$ K
Prism, colorless
0.26 × 0.22 × 0.20 mm

Data collection

Rigaku Saturn724 CCD diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.222 pixels mm^{-1}
ω scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku, 2009)
T_{min} = 0.965, T_{max} = 0.973
15222 measured reflections
3558 independent reflections
2461 reflections with $I > 2\sigma(I)'$
R_{int} = 0.043
θ_{max} = 27.9°, θ_{min} = 2.1°
h = −15→15
k = −10→11
l = −18→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.075
S = 0.96
3558 reflections
234 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0366P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.29 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-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 (Å²)

	x	y	z	U_{iso}*/U_{eq}
F1	0.63046 (7)	0.13597 (8)	0.56068 (5)	0.0284 (2)
F2	0.70104 (6)	−0.07799 (8)	0.61474 (5)	0.02243 (19)
F3	0.52483 (6)	−0.04190 (9)	0.61064 (5)	0.0278 (2)
O1	0.73825 (7)	0.17971 (10)	0.73408 (6)	0.0166 (2)
O2	0.57743 (7)	−0.12700 (9)	0.92883 (6)	0.0155 (2)
O3	0.61021 (7)	0.46053 (9)	0.68641 (6)	0.0188 (2)
O4	0.44283 (7)	0.37553 (9)	0.63011 (6)	0.0189 (2)
O5	0.57096 (8)	0.57378 (10)	0.88466 (7)	0.0226 (2)
N1	0.62882 (9)	−0.02277 (12)	0.79262 (7)	0.0144 (2)
N2	0.52802 (9)	0.11947 (11)	0.89483 (8)	0.0151 (2)
C1	0.63604 (10)	0.10070 (14)	0.72522 (9)	0.0136 (3)
C2	0.54011 (10)	0.21315 (13)	0.73746 (8)	0.0128 (3)
H2	0.4693	0.1572	0.7201	0.015*
C3	0.53875 (10)	0.26147 (13)	0.84121 (8)	0.0139 (3)
H3	0.6103	0.3134	0.8614	0.017*

C4	0.57828 (10)	−0.01283 (14)	0.87496 (9)	0.0137 (3)
C5	0.62326 (10)	0.02811 (14)	0.62678 (9)	0.0169 (3)
C6	0.53918 (10)	0.36240 (14)	0.68090 (8)	0.0144 (3)
C7	0.41216 (11)	0.52976 (14)	0.59613 (10)	0.0219 (3)
H7A	0.4704	0.5723	0.5582	0.026*
H7B	0.4024	0.6003	0.6494	0.026*
C8	0.30531 (11)	0.51220 (17)	0.53746 (10)	0.0273 (3)
H8A	0.3166	0.4433	0.4847	0.033*
H8B	0.2806	0.6135	0.5137	0.033*
H8C	0.2489	0.4683	0.5757	0.033*
C9	0.44302 (10)	0.37055 (13)	0.85540 (8)	0.0145 (3)
C10	0.46361 (10)	0.52609 (14)	0.87614 (8)	0.0157 (3)
C11	0.37451 (11)	0.62576 (15)	0.88661 (9)	0.0203 (3)
H11	0.3882	0.7312	0.9014	0.024*
C12	0.26690 (11)	0.57248 (15)	0.87573 (9)	0.0233 (3)
H12	0.2071	0.6412	0.8836	0.028*
C13	0.24560 (12)	0.41890 (15)	0.85339 (10)	0.0248 (3)
H13	0.1715	0.3824	0.8452	0.030*
C14	0.33378 (11)	0.31964 (15)	0.84326 (9)	0.0204 (3)
H14	0.3194	0.2146	0.8277	0.024*
H1	0.7889 (15)	0.1108 (18)	0.7422 (12)	0.046 (5)*
H4	0.6684 (12)	−0.1011 (15)	0.7884 (9)	0.021 (4)*
H5	0.4973 (12)	0.1248 (15)	0.9477 (11)	0.025 (4)*
H6	0.5734 (13)	0.6771 (19)	0.8967 (11)	0.043 (5)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0430 (5)	0.0269 (4)	0.0157 (4)	0.0048 (4)	0.0052 (4)	0.0046 (3)
F2	0.0214 (4)	0.0232 (4)	0.0232 (4)	0.0060 (3)	0.0047 (3)	−0.0049 (3)
F3	0.0180 (4)	0.0365 (5)	0.0285 (5)	−0.0054 (4)	−0.0001 (3)	−0.0131 (4)
O1	0.0097 (4)	0.0138 (5)	0.0264 (5)	−0.0005 (4)	0.0010 (4)	0.0003 (4)
O2	0.0196 (5)	0.0110 (4)	0.0161 (5)	0.0002 (4)	0.0029 (4)	0.0015 (4)
O3	0.0161 (5)	0.0168 (5)	0.0234 (5)	−0.0030 (4)	−0.0004 (4)	0.0028 (4)
O4	0.0151 (5)	0.0168 (5)	0.0241 (5)	0.0006 (4)	−0.0041 (4)	0.0051 (4)
O5	0.0165 (5)	0.0134 (5)	0.0381 (6)	−0.0009 (4)	0.0021 (4)	−0.0064 (4)
N1	0.0154 (5)	0.0115 (5)	0.0168 (6)	0.0045 (4)	0.0049 (4)	0.0010 (4)
N2	0.0197 (6)	0.0118 (5)	0.0146 (6)	0.0027 (4)	0.0065 (4)	0.0009 (4)
C1	0.0105 (6)	0.0133 (6)	0.0172 (7)	−0.0007 (5)	0.0014 (5)	0.0020 (5)
C2	0.0114 (6)	0.0120 (6)	0.0150 (7)	0.0009 (5)	0.0013 (5)	0.0010 (5)
C3	0.0135 (6)	0.0131 (6)	0.0152 (7)	0.0001 (5)	0.0017 (5)	0.0014 (5)
C4	0.0113 (6)	0.0132 (6)	0.0163 (7)	−0.0022 (5)	−0.0009 (5)	−0.0015 (5)
C5	0.0148 (6)	0.0171 (6)	0.0189 (7)	0.0016 (5)	0.0027 (5)	0.0010 (5)
C6	0.0128 (6)	0.0166 (7)	0.0138 (7)	0.0021 (5)	0.0020 (5)	−0.0009 (5)
C7	0.0209 (7)	0.0161 (7)	0.0282 (8)	0.0041 (5)	−0.0009 (6)	0.0072 (6)
C8	0.0217 (7)	0.0306 (8)	0.0291 (8)	0.0048 (6)	−0.0013 (6)	0.0094 (6)
C9	0.0148 (6)	0.0139 (6)	0.0153 (7)	0.0023 (5)	0.0036 (5)	0.0024 (5)
C10	0.0149 (6)	0.0150 (6)	0.0172 (7)	0.0009 (5)	0.0015 (5)	0.0004 (5)

C11	0.0209 (7)	0.0143 (7)	0.0257 (8)	0.0033 (5)	0.0018 (6)	−0.0028 (6)
C12	0.0178 (7)	0.0223 (7)	0.0302 (8)	0.0081 (6)	0.0045 (6)	−0.0003 (6)
C13	0.0150 (7)	0.0246 (7)	0.0353 (8)	0.0003 (6)	0.0049 (6)	−0.0016 (6)
C14	0.0183 (7)	0.0147 (7)	0.0289 (8)	−0.0005 (5)	0.0059 (6)	−0.0017 (6)

Geometric parameters (Å, º)

F1—C5	1.3370 (14)	C2—C3	1.5438 (17)
F2—C5	1.3350 (14)	C2—H2	1.0000
F3—C5	1.3418 (14)	C3—C9	1.5191 (16)
O1—C1	1.4110 (14)	C3—H3	1.0000
O1—H1	0.858 (17)	C7—C8	1.4990 (19)
O2—C4	1.2547 (14)	C7—H7A	0.9900
O3—C6	1.2073 (14)	C7—H7B	0.9900
O4—C6	1.3347 (15)	C8—H8A	0.9800
O4—C7	1.4611 (14)	C8—H8B	0.9800
O5—C10	1.3599 (15)	C8—H8C	0.9800
O5—H6	0.912 (16)	C9—C14	1.3922 (17)
N1—C4	1.3674 (15)	C9—C10	1.3986 (17)
N1—C1	1.4472 (16)	C10—C11	1.3971 (17)
N1—H4	0.836 (14)	C11—C12	1.3795 (18)
N2—C4	1.3373 (16)	C11—H11	0.9500
N2—C3	1.4606 (15)	C12—C13	1.3886 (19)
N2—H5	0.867 (14)	C12—H12	0.9500
C1—C2	1.5344 (16)	C13—C14	1.3856 (18)
C1—C5	1.5407 (18)	C13—H13	0.9500
C2—C6	1.5254 (16)	C14—H14	0.9500
C1—O1—H1	106.7 (11)	F3—C5—C1	111.93 (10)
C6—O4—C7	116.81 (10)	O3—C6—O4	124.46 (11)
C10—O5—H6	109.5 (10)	O3—C6—C2	125.69 (12)
C4—N1—C1	125.39 (10)	O4—C6—C2	109.59 (10)
C4—N1—H4	113.9 (9)	O4—C7—C8	106.41 (10)
C1—N1—H4	119.4 (9)	O4—C7—H7A	110.4
C4—N2—C3	123.51 (10)	C8—C7—H7A	110.4
C4—N2—H5	117.4 (9)	O4—C7—H7B	110.4
C3—N2—H5	118.2 (9)	C8—C7—H7B	110.4
O1—C1—N1	113.07 (10)	H7A—C7—H7B	108.6
O1—C1—C2	110.26 (10)	C7—C8—H8A	109.5
N1—C1—C2	108.19 (9)	C7—C8—H8B	109.5
O1—C1—C5	108.17 (9)	H8A—C8—H8B	109.5
N1—C1—C5	107.50 (10)	C7—C8—H8C	109.5
C2—C1—C5	109.58 (10)	H8A—C8—H8C	109.5
C6—C2—C1	117.10 (9)	H8B—C8—H8C	109.5
C6—C2—C3	106.28 (10)	C14—C9—C10	119.03 (11)
C1—C2—C3	109.92 (10)	C14—C9—C3	120.77 (11)
C6—C2—H2	107.7	C10—C9—C3	120.12 (11)
C1—C2—H2	107.7	O5—C10—C11	122.76 (11)

C3—C2—H2	107.7	O5—C10—C9	117.82 (10)
N2—C3—C9	110.95 (9)	C11—C10—C9	119.42 (11)
N2—C3—C2	106.50 (10)	C12—C11—C10	120.64 (12)
C9—C3—C2	110.89 (10)	C12—C11—H11	119.7
N2—C3—H3	109.5	C10—C11—H11	119.7
C9—C3—H3	109.5	C11—C12—C13	120.35 (12)
C2—C3—H3	109.5	C11—C12—H12	119.8
O2—C4—N2	121.47 (11)	C13—C12—H12	119.8
O2—C4—N1	120.20 (11)	C14—C13—C12	119.15 (13)
N2—C4—N1	118.31 (11)	C14—C13—H13	120.4
F2—C5—F1	107.99 (10)	C12—C13—H13	120.4
F2—C5—F3	106.92 (10)	C13—C14—C9	121.39 (12)
F1—C5—F3	107.15 (10)	C13—C14—H14	119.3
F2—C5—C1	111.86 (10)	C9—C14—H14	119.3
F1—C5—C1	110.74 (10)		

C4—N1—C1—O1	98.15 (14)	O1—C1—C5—F3	−176.35 (10)
C4—N1—C1—C2	−24.26 (16)	N1—C1—C5—F3	61.24 (13)
C4—N1—C1—C5	−142.52 (11)	C2—C1—C5—F3	−56.12 (13)
O1—C1—C2—C6	49.05 (14)	C7—O4—C6—O3	−13.82 (17)
N1—C1—C2—C6	173.17 (10)	C7—O4—C6—C2	160.62 (10)
C5—C1—C2—C6	−69.90 (13)	C1—C2—C6—O3	−60.93 (16)
O1—C1—C2—C3	−72.30 (12)	C3—C2—C6—O3	62.31 (15)
N1—C1—C2—C3	51.82 (13)	C1—C2—C6—O4	124.72 (11)
C5—C1—C2—C3	168.75 (9)	C3—C2—C6—O4	−112.04 (11)
C4—N2—C3—C9	158.86 (11)	C6—O4—C7—C8	176.95 (10)
C4—N2—C3—C2	38.09 (16)	N2—C3—C9—C14	−51.23 (16)
C6—C2—C3—N2	174.26 (10)	C2—C3—C9—C14	66.90 (14)
C1—C2—C3—N2	−58.11 (12)	N2—C3—C9—C10	132.13 (12)
C6—C2—C3—C9	53.45 (12)	C2—C3—C9—C10	−109.73 (13)
C1—C2—C3—C9	−178.92 (9)	C14—C9—C10—O5	−177.92 (11)
C3—N2—C4—O2	171.65 (11)	C3—C9—C10—O5	−1.22 (17)
C3—N2—C4—N1	−10.02 (18)	C14—C9—C10—C11	1.67 (18)
C1—N1—C4—O2	−179.52 (11)	C3—C9—C10—C11	178.36 (11)
C1—N1—C4—N2	2.12 (18)	O5—C10—C11—C12	178.89 (12)
O1—C1—C5—F2	63.67 (12)	C9—C10—C11—C12	−0.68 (19)
N1—C1—C5—F2	−58.74 (13)	C10—C11—C12—C13	−0.5 (2)
C2—C1—C5—F2	−176.10 (9)	C11—C12—C13—C14	0.7 (2)
O1—C1—C5—F1	−56.85 (13)	C12—C13—C14—C9	0.3 (2)
N1—C1—C5—F1	−179.26 (10)	C10—C9—C14—C13	−1.49 (19)
C2—C1—C5—F1	63.38 (12)	C3—C9—C14—C13	−178.16 (12)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H4···O1^{i}	0.836 (14)	2.244 (14)	3.0760 (13)	174.1 (14)
N2—H5···O2^{ii}	0.867 (14)	2.040 (15)	2.9064 (14)	177.4 (13)
O1—H1···O5^{i}	0.858 (17)	2.588 (16)	3.0983 (13)	119.2 (13)

O1—H1···O3^{i}	0.858 (17)	2.013 (17)	2.8232 (13)	157.1 (15)
O5—H6···O2^{iii}	0.912 (16)	1.759 (17)	2.6685 (12)	175.8 (14)

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