

1-(5-Hydroxy-1-phenyl-3-trifluoromethyl-1H-pyrazol-1-yl)ethanone

Hugo Gallardo, Edivandro Giroto and Adailton J. Bortoluzzi*

Depto. de Química - Universidade Federal de Santa Catarina, 88040-900 - Florianópolis, SC, Brazil

Correspondence e-mail: adajb@qmc.ufsc.br

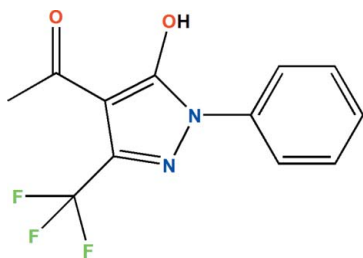
Received 2 November 2009; accepted 1 December 2009

Key indicators: single-crystal X-ray study; $T = 293$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; disorder in main residue; R factor = 0.055; wR factor = 0.165; data-to-parameter ratio = 12.2.

The crystal structure of the title compound, $\text{C}_{12}\text{H}_9\text{F}_3\text{N}_2\text{O}_2$, contains two independent molecules in the asymmetric unit. The molecules are chemically identical but exhibit a significant difference in the dihedral angles between the mean planes of the phenyl and pyrazole rings, with values of 11.62 (13) and 18.17 (11)°. Moreover, the trifluoromethyl group in one of the molecules shows rotational disorder of the F atoms, with site occupancy factors of 0.929 (6) and 0.071 (6). The hydroxyl group in each of the molecules shows a strong intramolecular hydrogen bond with the carbonyl O atom, forming a six-membered ring and forcing the formyl group and pyrazole ring to be coplanar, showing C—C—C—O torsion angles of 70.3(5)° and 0°. Weak intermolecular C—H···O and C—H···F interactions contribute to the stabilization of the crystal packing.

Related literature

For the pharmaceutical activity of pyrazole derivatives, see: Belmar *et al.* (2001). For related structures, see: Gallardo *et al.* (2009); Belmar *et al.* (2006a,b); Pérez *et al.* (2005). For the melting point, see: Bieringer & Holzer (2006).



Experimental

Crystal data

$\text{C}_{12}\text{H}_9\text{F}_3\text{N}_2\text{O}_2$	$\gamma = 78.791$ (18)°
$M_r = 270.21$	$V = 1178.2$ (4) Å ³
Triclinic, $P\bar{1}$	$Z = 4$
$a = 7.4779$ (19) Å	Mo $K\alpha$ radiation
$b = 11.9390$ (18) Å	$\mu = 0.14$ mm ⁻¹
$c = 13.8587$ (14) Å	$T = 293$ K
$\alpha = 78.591$ (11)°	$0.40 \times 0.33 \times 0.13$ mm
$\beta = 80.090$ (17)°	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.025$
4766 measured reflections	3 standard reflections every 200 reflections
4567 independent reflections	intensity decay: 1%
2268 reflections with $I > 2\sigma(I)$	

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.055$	81 restraints
$wR(F^2) = 0.165$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 0.20$ e Å ⁻³
4567 reflections	$\Delta\rho_{\text{min}} = -0.21$ e Å ⁻³
373 parameters	

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O5}-\text{H5}\cdots\text{O13}$	1.18	1.60	2.548 (3)	132
$\text{O5}'-\text{H5}'\cdots\text{O13}'$	1.07	1.65	2.560 (4)	139
$\text{C14}-\text{H14B}\cdots\text{F1}^{\text{ii}}$	0.96	2.55	3.394 (5)	146
$\text{C7}-\text{H7}\cdots\text{O13}^{\text{ii}}$	0.93	2.64	3.446 (4)	145
$\text{C7}'-\text{H7}'\cdots\text{O13}^{\text{iii}}$	0.93	2.57	3.406 (4)	149

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *HELENA* (Spek, 1996); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXL97*.

The authors are grateful to the Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq), the Coordenação de Aperfeiçoamento de Pessoal de Nível Superior (CAPES) and the Instituto Nacional de Ciência e Tecnologia (INCT) - Catalize for financial assistance.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PV2233).

References

- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G. L., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Belmar, J., Alderete, J., Zuñiga, C., Jimenez, C., Jimenez, V., Núñez, H., Grandy, R. & Yori, A. (2001). *Bol. Soc. Chil. Quím.* **46**, 459–470.
- Belmar, J., Jiménez, C., Ortiz, L., Garland, M. T. & Baggio, R. (2006a). *Acta Cryst.* **C62**, o76–o78.
- Belmar, J., Jiménez, C., Ruiz-Pérez, C., Delgado, F. S. & Baggio, R. (2006b). *Acta Cryst.* **C62**, o599–o601.
- Bieringer, S. & Holzer, W. (2006). *Heterocycles*, **68**, 1825–1836.

Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.

Gallardo, H., Giroto, E., Bortoluzzi, A. J. & Terra, G. G. (2009). *Acta Cryst.* **E65**, o2040–o2041.

Pérez, F. R., Belmar, J., Jiménez, C., Moreno, Y., Hermosilla, P. & Baggio, R. (2005). *Acta Cryst.* **C61**, m318–m320.

Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

Spek, A. L. (1996). *HELENA*. University of Utrecht, The Netherlands.

Spek, A. L. (2009). *Acta Cryst.* **D65**, 148–155.

supplementary materials

Acta Cryst. (2010). E66, o75-o76 [doi:10.1107/S1600536809051745]

1-(5-Hydroxy-1-phenyl-3-trifluoromethyl-1*H*-pyrazol-1-yl)ethanone

H. Gallardo, E. Girotto and A. J. Bortoluzzi

Comment

The pyrazolones are derivatives of pyrazole having an additional carbonyl/hydroxy group. Some derivatives of pyrazolone show analgesic and anti-inflammatory properties (Belmar *et al.*, 2001). In this article, the crystal structure of the title compound (I), which is a derivative of pyrazole is presented.

The asymmetric unit of the title compound contains two independent molecules (Fig. 1). The molecules are chemically identical, but show different spacial conformation. This can be evidenced by the different dihedral angle between the mean plane of the phenyl and pyrazole rings of 11.62 (13)° and 18.17 (11)°. In the pyrazole rings of the two molecules the single bond distances are shorter and the double bond distances are longer than expected values indicating that the pyrazole rings are delocalized π -systems. A cyclic six-membered intramolecular hydrogen bond between hydroxyl and acyl groups force the acyl groups and the pyrazole rings to be coplanar, in both molecules. Several weak intermolecular C—H \cdots O and C—H \cdots F hydrogen bonds are involved in the stabilization of the crystal packing. The crystal structures of a few compounds closely related to (I) have been reported (Gallardo *et al.*, 2009; Belmar *et al.*, 2006*a,b*); Pérez *et al.*, 2005).

Experimental

In a three necked flask of 50 ml equipped with condenser, were transferred 2-phenyl-5-(trifluoromethyl)pyrazol-3(2*H*)-one (1.5 g, 6.57 mmol), 1,4-dioxane (dried) (25 ml) and Ca(OH)₂ (0.67 g, 13.1 mmol). The reaction mixture was heated to 373 K with stirring for 15 min, then cooled to room temperature and added slowly ac. acetyl chloride (0.56 g, 7.2 mmol) and allowed to reflux for 24 h. After the reaction was completed, the product was poured into 40 ml of HCl solution (3 mol l⁻¹) and ice and stirred for 30 min, resulting in precipitate which was filtered. The compound was purified through a silica gel chromatographic column (eluent: hexane/AcOEt 3%) which yielded a yellow solid (0.525 g) of the title compound. In the purification process, 0.752 g of 2-phenyl-5-(trifluoromethyl)pyrazol-3(2*H*)-one was also recovered.

Refinement

H atoms were placed at their idealized positions with distances of 0.93 and 0.96 Å and U_{eq} fixed at 1.2 and 1.5 times U_{iso} of the preceding atom for C—H_{Ar} and CH₃, respectively. The H atoms of the hydroxyl groups were found from Fourier difference map and fixed at those positions with U_{eq} fixed at 1.2 times U_{iso} of the parent atom. Trifluoromethyl group of one molecule exhibited rotational disorder with two alternative positions for F atoms. The refined site occupancies for disordered atoms are 0.929 (6) and 0.071 (6).

Figures

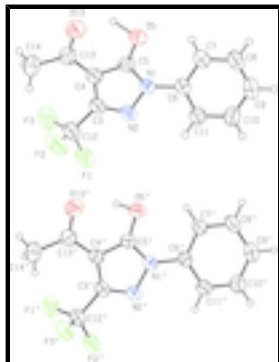


Fig. 1. The structure of the two molecules of the title compound with labeling scheme; displacement ellipsoids are shown at the 40% probability level and atoms F1", F2" and F3" representing the smaller fraction of the disordered trifluoromethyl group have been excluded.

1-(5-Hydroxy-1-phenyl-3-trifluoromethyl-1H-pyrazol-1-yl)ethanone

Crystal data

$C_{12}H_9F_3N_2O_2$

$M_r = 270.21$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4779 (19) \text{ \AA}$

$b = 11.9390 (18) \text{ \AA}$

$c = 13.8587 (14) \text{ \AA}$

$\alpha = 78.591 (11)^\circ$

$\beta = 80.090 (17)^\circ$

$\gamma = 78.791 (18)^\circ$

$V = 1178.2 (4) \text{ \AA}^3$

$Z = 4$

$F(000) = 552$

$D_x = 1.523 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71069 \text{ \AA}$

Cell parameters from 25 reflections

$\theta = 5.4\text{--}18.6^\circ$

$\mu = 0.14 \text{ mm}^{-1}$

$T = 293 \text{ K}$

Prismatic, yellow

$0.40 \times 0.33 \times 0.13 \text{ mm}$

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube
graphite

$\omega/2\theta$ scans

4766 measured reflections

4567 independent reflections

2268 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.025$

$\theta_{\text{max}} = 26.0^\circ$, $\theta_{\text{min}} = 1.5^\circ$

$h = -9 \rightarrow 9$

$k = -14 \rightarrow 14$

$l = -17 \rightarrow 0$

3 standard reflections every 200 reflections

intensity decay: 1%

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

$wR(F^2) = 0.165$

$S = 1.04$

4567 reflections

373 parameters

81 restraints

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.4943P]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

$$(\Delta/\sigma)_{\max} = 0.001$$

$$\Delta\rho_{\max} = 0.20 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -0.20 \text{ e } \text{\AA}^{-3}$$

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
N1	0.4429 (3)	0.5800 (2)	0.73982 (18)	0.0517 (7)	
N2	0.4653 (4)	0.6690 (2)	0.66084 (18)	0.0585 (7)	
C3	0.4755 (5)	0.7585 (3)	0.7002 (2)	0.0557 (8)	
C4	0.4619 (4)	0.7333 (3)	0.8049 (2)	0.0524 (8)	
C5	0.4399 (4)	0.6175 (3)	0.8262 (2)	0.0536 (8)	
O5	0.4173 (3)	0.55236 (19)	0.91394 (16)	0.0695 (7)	
H5	0.4804	0.6027	0.9610	0.083*	
C6	0.4214 (4)	0.4699 (3)	0.7213 (2)	0.0508 (8)	
C7	0.4301 (5)	0.3738 (3)	0.7950 (3)	0.0668 (10)	
H7	0.4494	0.3792	0.8583	0.080*	
C8	0.4099 (5)	0.2695 (3)	0.7735 (3)	0.0749 (11)	
H8	0.4140	0.2046	0.8233	0.090*	
C9	0.3840 (5)	0.2594 (3)	0.6807 (3)	0.0738 (11)	
H9	0.3725	0.1881	0.6670	0.089*	
C10	0.3752 (5)	0.3559 (3)	0.6079 (3)	0.0718 (10)	
H10	0.3567	0.3499	0.5446	0.086*	
C11	0.3934 (5)	0.4620 (3)	0.6275 (2)	0.0633 (9)	
H11	0.3869	0.5271	0.5779	0.076*	
C12	0.4960 (7)	0.8687 (3)	0.6304 (3)	0.0751 (11)	
F1	0.5010 (8)	0.8597 (3)	0.5363 (2)	0.1229 (17)	0.929 (6)
F1''	0.362 (5)	0.899 (4)	0.585 (3)	0.119 (13)	0.071 (6)
F2	0.3541 (5)	0.9518 (2)	0.6488 (3)	0.1112 (14)	0.929 (6)
F2''	0.508 (7)	0.955 (3)	0.667 (3)	0.106 (11)	0.071 (6)
F3	0.6442 (6)	0.9097 (3)	0.6361 (3)	0.1119 (14)	0.929 (6)
F3''	0.637 (6)	0.850 (3)	0.573 (3)	0.096 (12)	0.071 (6)
C13	0.4653 (5)	0.7924 (3)	0.8855 (3)	0.0643 (9)	
O13	0.4473 (4)	0.7366 (2)	0.97198 (18)	0.0839 (8)	
C14	0.4909 (6)	0.9153 (3)	0.8704 (3)	0.0849 (12)	
H14A	0.4708	0.9407	0.9335	0.127*	
H14B	0.4046	0.9625	0.8290	0.127*	
H14C	0.6140	0.9221	0.8388	0.127*	
N1'	0.9141 (4)	0.4419 (2)	0.7688 (2)	0.0561 (7)	
N2'	0.9250 (4)	0.3460 (2)	0.8425 (2)	0.0612 (7)	
C3'	0.9172 (4)	0.2592 (3)	0.7996 (3)	0.0587 (9)	
C4'	0.9010 (4)	0.2929 (3)	0.6972 (2)	0.0578 (9)	
C5'	0.8996 (4)	0.4117 (3)	0.6816 (2)	0.0569 (8)	
O5'	0.8858 (3)	0.4856 (2)	0.59906 (17)	0.0747 (7)	

supplementary materials

H5'	0.8542	0.4376	0.5487	0.090*
C6'	0.9283 (4)	0.5517 (3)	0.7911 (2)	0.0536 (8)
C7'	0.9738 (5)	0.6410 (3)	0.7169 (3)	0.0655 (9)
H7'	0.9917	0.6317	0.6506	0.079*
C8'	0.9925 (5)	0.7435 (3)	0.7409 (3)	0.0737 (11)
H8'	1.0230	0.8038	0.6908	0.088*
C9'	0.9668 (5)	0.7580 (3)	0.8383 (3)	0.0778 (11)
H9'	0.9813	0.8276	0.8542	0.093*
C10'	0.9198 (5)	0.6702 (3)	0.9119 (3)	0.0768 (11)
H10'	0.9013	0.6805	0.9779	0.092*
C11'	0.8993 (5)	0.5662 (3)	0.8896 (3)	0.0652 (9)
H11'	0.8666	0.5067	0.9399	0.078*
C12'	0.9344 (6)	0.1406 (3)	0.8617 (3)	0.0736 (10)
F1'	1.0741 (4)	0.06809 (19)	0.8220 (2)	0.1054 (8)
F2'	0.9629 (4)	0.14090 (19)	0.95283 (18)	0.1084 (9)
F3'	0.7860 (3)	0.09262 (18)	0.86974 (18)	0.0964 (8)
C13'	0.8886 (5)	0.2391 (3)	0.6145 (3)	0.0726 (10)
O13'	0.8789 (4)	0.3019 (3)	0.5317 (2)	0.0953 (9)
C14'	0.8891 (7)	0.1121 (3)	0.6241 (3)	0.0989 (14)
H14D	0.8621	0.0961	0.5635	0.148*
H14E	0.7975	0.0886	0.6780	0.148*
H14F	1.0081	0.0701	0.6371	0.148*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0614 (17)	0.0456 (15)	0.0463 (16)	-0.0126 (12)	-0.0084 (13)	0.0007 (12)
N2	0.077 (2)	0.0473 (16)	0.0473 (16)	-0.0152 (13)	-0.0068 (13)	0.0045 (13)
C3	0.068 (2)	0.0430 (18)	0.054 (2)	-0.0103 (16)	-0.0102 (17)	-0.0010 (16)
C4	0.055 (2)	0.0486 (18)	0.053 (2)	-0.0092 (15)	-0.0099 (15)	-0.0037 (15)
C5	0.055 (2)	0.056 (2)	0.0487 (19)	-0.0117 (16)	-0.0067 (15)	-0.0035 (16)
O5	0.0961 (19)	0.0660 (15)	0.0460 (13)	-0.0251 (13)	-0.0112 (12)	0.0044 (11)
C6	0.050 (2)	0.0445 (18)	0.056 (2)	-0.0085 (14)	-0.0029 (15)	-0.0060 (15)
C7	0.077 (3)	0.055 (2)	0.066 (2)	-0.0159 (18)	-0.0112 (19)	0.0008 (18)
C8	0.084 (3)	0.050 (2)	0.086 (3)	-0.0157 (19)	-0.011 (2)	0.002 (2)
C9	0.073 (3)	0.052 (2)	0.098 (3)	-0.0164 (18)	-0.002 (2)	-0.019 (2)
C10	0.086 (3)	0.064 (2)	0.070 (2)	-0.022 (2)	-0.006 (2)	-0.018 (2)
C11	0.079 (3)	0.054 (2)	0.057 (2)	-0.0170 (18)	-0.0042 (18)	-0.0073 (17)
C12	0.107 (4)	0.053 (2)	0.064 (3)	-0.017 (2)	-0.013 (3)	-0.002 (2)
F1	0.244 (5)	0.0707 (18)	0.0569 (19)	-0.053 (2)	-0.025 (2)	0.0120 (14)
F1''	0.18 (2)	0.10 (2)	0.07 (2)	-0.04 (2)	-0.05 (2)	0.033 (19)
F2	0.137 (3)	0.0565 (17)	0.121 (3)	0.0069 (18)	-0.022 (2)	0.0097 (16)
F2''	0.15 (3)	0.059 (18)	0.12 (2)	-0.049 (19)	-0.05 (2)	0.007 (16)
F3	0.118 (3)	0.087 (2)	0.135 (3)	-0.059 (2)	-0.030 (2)	0.023 (2)
F3''	0.14 (2)	0.060 (18)	0.08 (2)	-0.040 (17)	-0.042 (19)	0.052 (16)
C13	0.064 (2)	0.062 (2)	0.069 (2)	-0.0067 (17)	-0.0114 (18)	-0.0165 (19)
O13	0.118 (2)	0.0789 (18)	0.0542 (16)	-0.0138 (15)	-0.0083 (15)	-0.0153 (14)
C14	0.107 (3)	0.064 (2)	0.093 (3)	-0.014 (2)	-0.018 (2)	-0.032 (2)

N1'	0.0623 (18)	0.0467 (16)	0.0565 (17)	-0.0101 (13)	-0.0075 (14)	-0.0023 (13)
N2'	0.0710 (19)	0.0489 (17)	0.0611 (18)	-0.0104 (14)	-0.0097 (14)	-0.0021 (14)
C3'	0.057 (2)	0.049 (2)	0.070 (2)	-0.0112 (16)	-0.0084 (17)	-0.0059 (17)
C4'	0.057 (2)	0.057 (2)	0.061 (2)	-0.0148 (16)	-0.0053 (17)	-0.0095 (17)
C5'	0.053 (2)	0.061 (2)	0.054 (2)	-0.0096 (16)	-0.0055 (16)	-0.0046 (17)
O5'	0.0982 (19)	0.0699 (16)	0.0563 (15)	-0.0246 (14)	-0.0168 (13)	0.0045 (13)
C6'	0.0479 (19)	0.0483 (19)	0.062 (2)	-0.0051 (15)	-0.0083 (16)	-0.0067 (16)
C7'	0.070 (2)	0.054 (2)	0.067 (2)	-0.0113 (17)	-0.0032 (18)	-0.0036 (18)
C8'	0.077 (3)	0.050 (2)	0.088 (3)	-0.0139 (18)	-0.008 (2)	0.002 (2)
C9'	0.080 (3)	0.057 (2)	0.102 (3)	-0.0088 (19)	-0.022 (2)	-0.020 (2)
C10'	0.089 (3)	0.065 (3)	0.080 (3)	-0.008 (2)	-0.021 (2)	-0.019 (2)
C11'	0.072 (2)	0.058 (2)	0.064 (2)	-0.0108 (18)	-0.0118 (18)	-0.0035 (18)
C12'	0.086 (3)	0.056 (2)	0.080 (3)	-0.015 (2)	-0.017 (2)	-0.008 (2)
F1'	0.1063 (19)	0.0639 (14)	0.130 (2)	0.0090 (13)	-0.0078 (16)	-0.0090 (14)
F2'	0.175 (3)	0.0663 (14)	0.0878 (18)	-0.0275 (15)	-0.0512 (17)	0.0124 (13)
F3'	0.1083 (19)	0.0687 (14)	0.1132 (18)	-0.0376 (13)	-0.0114 (14)	0.0012 (13)
C13'	0.073 (3)	0.075 (3)	0.075 (3)	-0.024 (2)	-0.006 (2)	-0.016 (2)
O13'	0.131 (3)	0.098 (2)	0.0654 (18)	-0.0395 (18)	-0.0116 (17)	-0.0173 (16)
C14'	0.125 (4)	0.083 (3)	0.104 (3)	-0.031 (3)	-0.018 (3)	-0.036 (3)

Geometric parameters (Å, °)

N1—C5	1.353 (4)	C14—H14C	0.9600
N1—N2	1.378 (3)	N1'—C5'	1.354 (4)
N1—C6	1.432 (4)	N1'—N2'	1.374 (3)
N2—C3	1.312 (4)	N1'—C6'	1.432 (4)
C3—C4	1.413 (4)	N2'—C3'	1.308 (4)
C3—C12	1.488 (5)	C3'—C4'	1.416 (5)
C4—C5	1.392 (4)	C3'—C12'	1.499 (5)
C4—C13	1.440 (5)	C4'—C5'	1.390 (4)
C5—O5	1.311 (4)	C4'—C13'	1.445 (5)
O5—H5	1.1757	C5'—O5'	1.306 (4)
C6—C11	1.375 (4)	O5'—H5'	1.0662
C6—C7	1.376 (4)	C6'—C7'	1.377 (4)
C7—C8	1.377 (5)	C6'—C11'	1.385 (4)
C7—H7	0.9300	C7'—C8'	1.368 (5)
C8—C9	1.364 (5)	C7'—H7'	0.9300
C8—H8	0.9300	C8'—C9'	1.371 (5)
C9—C10	1.373 (5)	C8'—H8'	0.9300
C9—H9	0.9300	C9'—C10'	1.364 (5)
C10—C11	1.383 (4)	C9'—H9'	0.9300
C10—H10	0.9300	C10'—C11'	1.380 (5)
C11—H11	0.9300	C10'—H10'	0.9300
C12—F3''	1.22 (3)	C11'—H11'	0.9300
C12—F1''	1.24 (3)	C12'—F2'	1.317 (4)
C12—F2''	1.26 (3)	C12'—F3'	1.324 (4)
C12—F3	1.315 (5)	C12'—F1'	1.335 (4)
C12—F1	1.323 (5)	C13'—O13'	1.246 (4)
C12—F2	1.331 (5)	C13'—C14'	1.494 (5)

supplementary materials

C13—O13	1.249 (4)	C14'—H14D	0.9600
C13—C14	1.486 (5)	C14'—H14E	0.9600
C14—H14A	0.9600	C14'—H14F	0.9600
C14—H14B	0.9600		
C5—N1—N2	109.9 (2)	C13—C14—H14C	109.5
C5—N1—C6	130.8 (3)	H14A—C14—H14C	109.5
N2—N1—C6	119.3 (2)	H14B—C14—H14C	109.5
C3—N2—N1	105.6 (2)	C5'—N1'—N2'	110.4 (3)
N2—C3—C4	112.9 (3)	C5'—N1'—C6'	130.3 (3)
N2—C3—C12	116.8 (3)	N2'—N1'—C6'	119.2 (3)
C4—C3—C12	130.3 (3)	C3'—N2'—N1'	105.4 (3)
C5—C4—C3	102.8 (3)	N2'—C3'—C4'	113.0 (3)
C5—C4—C13	119.1 (3)	N2'—C3'—C12'	117.5 (3)
C3—C4—C13	138.1 (3)	C4'—C3'—C12'	129.4 (3)
O5—C5—N1	123.7 (3)	C5'—C4'—C3'	102.8 (3)
O5—C5—C4	127.5 (3)	C5'—C4'—C13'	119.1 (3)
N1—C5—C4	108.8 (3)	C3'—C4'—C13'	138.1 (3)
C5—O5—H5	100.5	O5'—C5'—N1'	123.6 (3)
C11—C6—C7	120.5 (3)	O5'—C5'—C4'	127.9 (3)
C11—C6—N1	118.4 (3)	N1'—C5'—C4'	108.4 (3)
C7—C6—N1	121.1 (3)	C5'—O5'—H5'	104.8
C6—C7—C8	119.0 (3)	C7'—C6'—C11'	120.1 (3)
C6—C7—H7	120.5	C7'—C6'—N1'	121.2 (3)
C8—C7—H7	120.5	C11'—C6'—N1'	118.7 (3)
C9—C8—C7	121.4 (4)	C8'—C7'—C6'	119.8 (3)
C9—C8—H8	119.3	C8'—C7'—H7'	120.1
C7—C8—H8	119.3	C6'—C7'—H7'	120.1
C8—C9—C10	119.0 (3)	C7'—C8'—C9'	120.6 (3)
C8—C9—H9	120.5	C7'—C8'—H8'	119.7
C10—C9—H9	120.5	C9'—C8'—H8'	119.7
C9—C10—C11	120.8 (4)	C10'—C9'—C8'	119.8 (4)
C9—C10—H10	119.6	C10'—C9'—H9'	120.1
C11—C10—H10	119.6	C8'—C9'—H9'	120.1
C6—C11—C10	119.2 (3)	C9'—C10'—C11'	120.7 (4)
C6—C11—H11	120.4	C9'—C10'—H10'	119.6
C10—C11—H11	120.4	C11'—C10'—H10'	119.6
F3"—C12—F1"	110.4 (15)	C10'—C11'—C6'	119.1 (3)
F3"—C12—F2"	106.8 (15)	C10'—C11'—H11'	120.5
F1"—C12—F2"	105.9 (17)	C6'—C11'—H11'	120.5
F3—C12—F1	107.1 (4)	F2'—C12'—F3'	106.7 (3)
F3—C12—F2	106.0 (4)	F2'—C12'—F1'	107.2 (3)
F1—C12—F2	105.5 (4)	F3'—C12'—F1'	105.9 (3)
F3"—C12—C3	107.1 (14)	F2'—C12'—C3'	112.6 (3)
F1"—C12—C3	109.2 (19)	F3'—C12'—C3'	112.7 (3)
F2"—C12—C3	117.4 (19)	F1'—C12'—C3'	111.3 (3)
F3—C12—C3	113.5 (4)	O13'—C13'—C4'	118.0 (3)
F1—C12—C3	113.1 (3)	O13'—C13'—C14'	119.2 (4)
F2—C12—C3	111.1 (3)	C4'—C13'—C14'	122.9 (4)
O13—C13—C4	117.9 (3)	C13'—C14'—H14D	109.5

O13—C13—C14	118.9 (3)	C13'—C14'—H14E	109.5
C4—C13—C14	123.1 (3)	H14D—C14'—H14E	109.5
C13—C14—H14A	109.5	C13'—C14'—H14F	109.5
C13—C14—H14B	109.5	H14D—C14'—H14F	109.5
H14A—C14—H14B	109.5	H14E—C14'—H14F	109.5
C5—N1—N2—C3	0.0 (3)	C3—C4—C13—O13	179.8 (4)
C6—N1—N2—C3	178.1 (3)	C5—C4—C13—C14	179.1 (3)
N1—N2—C3—C4	0.4 (4)	C3—C4—C13—C14	-0.8 (6)
N1—N2—C3—C12	-178.7 (3)	C5'—N1'—N2'—C3'	0.1 (3)
N2—C3—C4—C5	-0.6 (4)	C6'—N1'—N2'—C3'	-176.9 (3)
C12—C3—C4—C5	178.3 (4)	N1'—N2'—C3'—C4'	0.0 (4)
N2—C3—C4—C13	179.3 (4)	N1'—N2'—C3'—C12'	177.4 (3)
C12—C3—C4—C13	-1.9 (7)	N2'—C3'—C4'—C5'	0.0 (4)
N2—N1—C5—O5	178.9 (3)	C12'—C3'—C4'—C5'	-177.1 (3)
C6—N1—C5—O5	1.1 (5)	N2'—C3'—C4'—C13'	179.4 (4)
N2—N1—C5—C4	-0.4 (4)	C12'—C3'—C4'—C13'	2.3 (7)
C6—N1—C5—C4	-178.2 (3)	N2'—N1'—C5'—O5'	179.5 (3)
C3—C4—C5—O5	-178.6 (3)	C6'—N1'—C5'—O5'	-3.9 (5)
C13—C4—C5—O5	1.4 (5)	N2'—N1'—C5'—C4'	-0.1 (4)
C3—C4—C5—N1	0.6 (3)	C6'—N1'—C5'—C4'	176.5 (3)
C13—C4—C5—N1	-179.3 (3)	C3'—C4'—C5'—O5'	-179.5 (3)
C5—N1—C6—C11	167.1 (3)	C13'—C4'—C5'—O5'	0.9 (5)
N2—N1—C6—C11	-10.6 (4)	C3'—C4'—C5'—N1'	0.1 (4)
C5—N1—C6—C7	-13.6 (5)	C13'—C4'—C5'—N1'	-179.5 (3)
N2—N1—C6—C7	168.8 (3)	C5'—N1'—C6'—C7'	-16.1 (5)
C11—C6—C7—C8	-0.2 (5)	N2'—N1'—C6'—C7'	160.2 (3)
N1—C6—C7—C8	-179.6 (3)	C5'—N1'—C6'—C11'	165.3 (3)
C6—C7—C8—C9	0.9 (6)	N2'—N1'—C6'—C11'	-18.4 (4)
C7—C8—C9—C10	-1.0 (6)	C11'—C6'—C7'—C8'	0.8 (5)
C8—C9—C10—C11	0.5 (6)	N1'—C6'—C7'—C8'	-177.7 (3)
C7—C6—C11—C10	-0.3 (5)	C6'—C7'—C8'—C9'	0.1 (6)
N1—C6—C11—C10	179.0 (3)	C7'—C8'—C9'—C10'	-0.8 (6)
C9—C10—C11—C6	0.2 (5)	C8'—C9'—C10'—C11'	0.6 (6)
N2—C3—C12—F3''	-59 (3)	C9'—C10'—C11'—C6'	0.4 (6)
C4—C3—C12—F3''	122 (3)	C7'—C6'—C11'—C10'	-1.1 (5)
N2—C3—C12—F1''	60 (3)	N1'—C6'—C11'—C10'	177.5 (3)
C4—C3—C12—F1''	-119 (3)	N2'—C3'—C12'—F2'	-2.5 (5)
N2—C3—C12—F2''	-179 (3)	C4'—C3'—C12'—F2'	174.5 (4)
C4—C3—C12—F2''	2(3)	N2'—C3'—C12'—F3'	118.3 (4)
N2—C3—C12—F3	-122.2 (5)	C4'—C3'—C12'—F3'	-64.8 (5)
C4—C3—C12—F3	59.0 (6)	N2'—C3'—C12'—F1'	-122.9 (4)
N2—C3—C12—F1	0.0 (6)	C4'—C3'—C12'—F1'	54.0 (5)
C4—C3—C12—F1	-178.9 (4)	C5'—C4'—C13'—O13'	0.7 (5)
N2—C3—C12—F2	118.4 (4)	C3'—C4'—C13'—O13'	-178.6 (4)
C4—C3—C12—F2	-60.4 (6)	C5'—C4'—C13'—C14'	180.0 (4)
C5—C4—C13—O13	-0.3 (5)	C3'—C4'—C13'—C14'	0.7 (7)

supplementary materials

Hydrogen-bond geometry (Å, °)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O5—H5 \cdots O13	1.18	1.60	2.548 (3)	132
O5'—H5' \cdots O13'	1.07	1.65	2.560 (4)	139
C14—H14B \cdots F1 ⁱ	0.96	2.55	3.394 (5)	146
C7—H7 \cdots O13 ⁱⁱ	0.93	2.64	3.446 (4)	145
C7'—H7' \cdots O13 ⁱⁱⁱ	0.93	2.57	3.406 (4)	149

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

Fig. 1

