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

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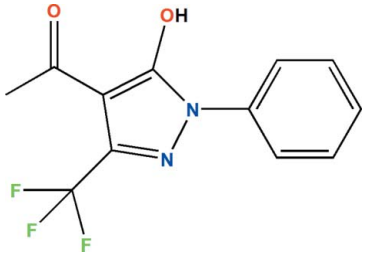
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.055; wR factor = 0.165; data-to-parameter ratio = 12.2.

The crystal structure of the title compound, $C_{12}H_9F_3N_2O_2$, 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 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 coplanarshowing C—C—C—O torsion angles of ?0.3(5)o 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.* (2006*a*,*b*); Pérez *et al.* (2005). For the melting point, see: Bieringer & Holzer (2006).



Experimental

Crystal data

$C_{12}H_9F_3N_2O_2$
M_r = 270.21
Triclinic, $P\overline{1}$
a = 7.4779 (19) Å
b = 11.9390 (18) Å
c = 13.8587 (14) Å
α = 78.591 (11)°
β = 80.090 (17)°
γ = 78.791 (18)°
V = 1178.2 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.14 mm^{-1}
T = 293 K
0.40 × 0.33 × 0.13 mm

Data collection

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

Refinement

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O5—H5···O13	1.18	1.60	2.548 (3)	132
O5′—H5′···O13′	1.07	1.65	2.560 (4)	139
C14—H14B···F1′^{i}	0.96	2.55	3.394 (5)	146
C7—H7···O13^{ii}	0.93	2.64	3.446 (4)	145
C7′—H7′···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) $-x+2, -y+1, -z+1$.

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*.

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

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

Hugo Gallardo, Edivandro Girotto and Adailton J. Bortoluzzi

S1. Comment

The pyrazolones are derivatives of pyrazole having an additional carbonyl/hydroxy group. Some derivatives of pyrazolone show analgesic and anti-inflamatory 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 that 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···O and C—H···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).

S2. 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.

S3. 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 CH_3, 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).

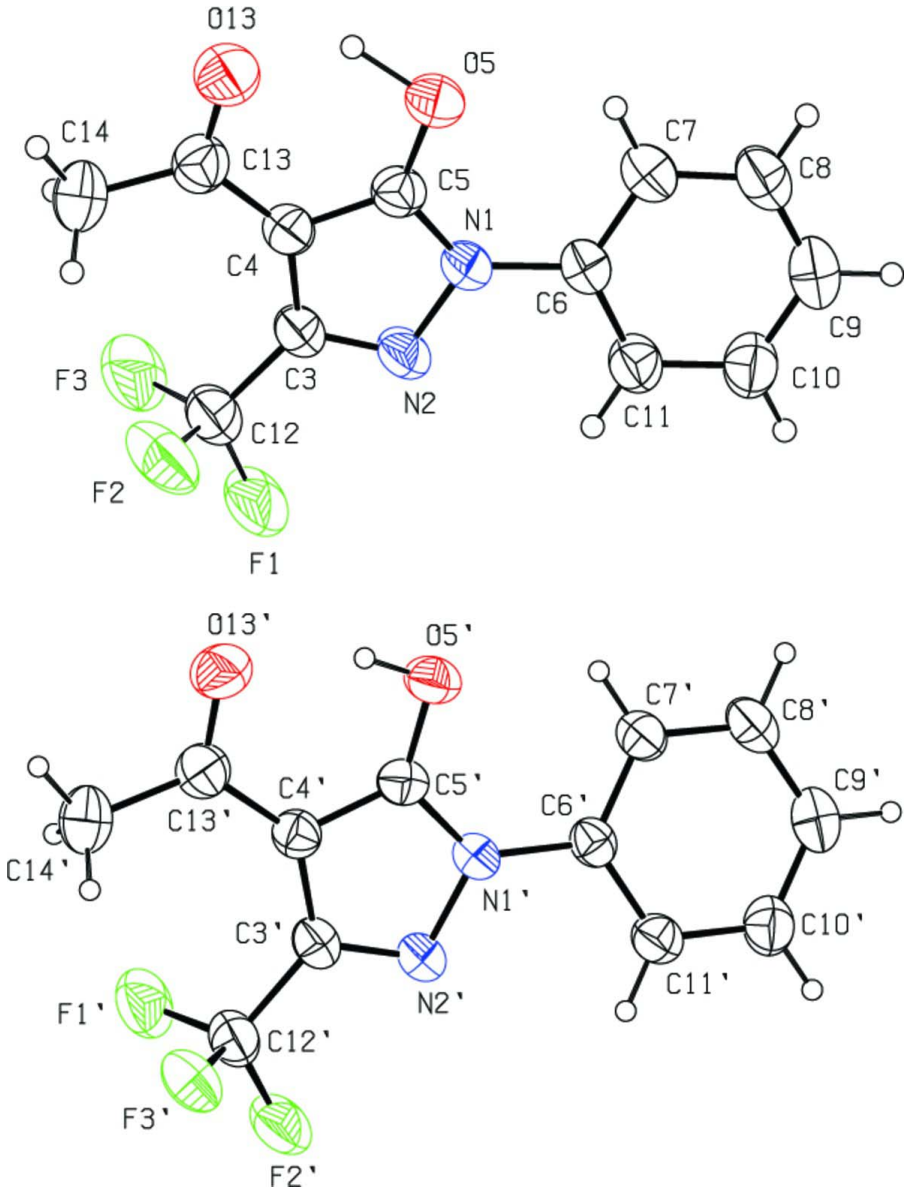

Figure 1

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-1*H*-pyrazol-1-yl)ethanone

Crystal data

$C_{12}H_9F_3N_2O_2$
$M_r = 270.21$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 7.4779\ (19)$ Å
$b = 11.9390\ (18)$ Å
$c = 13.8587\ (14)$ Å
$\alpha = 78.591\ (11)°$
$\beta = 80.090\ (17)°$
$\gamma = 78.791\ (18)°$
$V = 1178.2\ (4)$ Å^3
$Z = 4$
$F(000) = 552$
$D_x = 1.523$ Mg m^{-3}

Mo $K\alpha$ radiation, λ = 0.71069 Å
Cell parameters from 25 reflections
θ = 5.4–18.6°
μ = 0.14 mm^{-1}
T = 293 K
Prismatic, yellow
0.40 × 0.33 × 0.13 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
4766 measured reflections
4567 independent reflections
2268 reflections with $I > 2\sigma(I)$
R_{int} = 0.025
θ_{max} = 26.0°, θ_{min} = 1.5°
h = −9→9
k = −14→14
l = −17→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
$wR(F^2)$ = 0.165
S = 1.04
4567 reflections
373 parameters
81 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 0.4943P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.21 e Å^{-3}

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

	x	y	z	U_{iso}*/U_{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)	
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 (Å²)

	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, º)

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)
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)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O5—H5···O13	1.18	1.60	2.548 (3)	132
O5′—H5′···O13′	1.07	1.65	2.560 (4)	139
C14—H14*B*···F1′^{i}	0.96	2.55	3.394 (5)	146
C7—H7···O13^{ii}	0.93	2.64	3.446 (4)	145
C7′—H7′···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.