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(*E*)-*N*′-(2,4,5-Trifluorobenzylidene)-isonicotinohydrazide monohydrate

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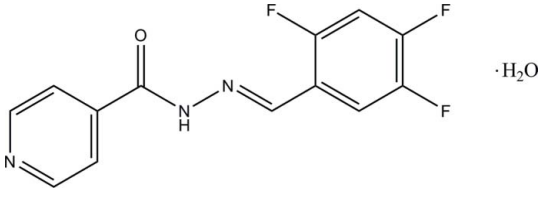
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.002 Å; R factor = 0.046; wR factor = 0.146; data-to-parameter ratio = 17.5.

In the Schiff base molecule of the title compound, $C_{13}H_8F_3N_3O\cdot H_2O$, the benzene ring and the pyridine ring are nearly coplanar, making a dihedral angle of 6.64 (7)°. The molecule exists in an *E* configuration with respect to the C=N double bond. In the crystal structure, molecules are linked *via* the water molecules into two-dimensional planes parallel to the *ab* plane through intermolecular N—H···O, O—H···O O—H···N and C—H···O hydrogen bonds.

Related literature

For applications of isoniazid (isonicotinylhydrazine) derivatives, see: Janin (2007); Maccari *et al.* (2005); Slayden & Barry (2000); Kahwa *et al.* (1986). For the preparation of the title compound, see: Lourenco *et al.* (2008). For related structures, see: Naveenkumar *et al.* (2009, 2010); Shi (2005).



Experimental

Crystal data

$C_{13}H_8F_3N_3O\cdot H_2O$
M_r = 297.24
Triclinic, $P\overline{1}$
a = 4.9241 (1) Å
b = 6.3915 (1) Å
c = 21.3387 (2) Å
α = 88.616 (1)°
β = 86.556 (1)°
γ = 76.056 (1)°
V = 650.58 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.13 mm^{-1}
T = 296 K
0.32 × 0.32 × 0.13 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.958, T_{max} = 0.984
14710 measured reflections
4024 independent reflections
2730 reflections with $I > 2\sigma(I)$
R_{int} = 0.022

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.146
S = 1.05
4024 reflections
230 parameters
All H-atom parameters refined
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H1*N2*···O1*W*	0.883 (19)	1.921 (19)	2.7910 (13)	168.1 (16)
O1*W*—H1*W*1···O1^{i}	0.79 (2)	2.03 (2)	2.8263 (17)	176.3 (18)
O1*W*—H2*W*1···O1^{ii}	0.84 (2)	2.19 (2)	2.9670 (17)	155 (2)
O1*W*—H2*W*1···N1^{ii}	0.84 (2)	2.47 (2)	3.1024 (14)	133.7 (19)
C7—H7*A*···O1*W*	0.967 (13)	2.492 (14)	3.2576 (16)	135.9 (12)
C13—H13*A*···O1*W*	0.963 (19)	2.427 (18)	3.3308 (19)	156.2 (14)

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2725).

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supporting information

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(*E*)-*N*′-(2,4,5-Trifluorobenzylidene)isonicotinohydrazide monohydrate

H. S. Naveenkumar, Amirin Sadikun, Pazilah Ibrahim, Chin Sing Yeap and Hoong-Kun Fun

S1. Comment

In the search for new biologically active compounds, isoniazid (isonicotinylhydrazine) derivatives have been found to possess potential tuberculostatic activity (Janin, 2007; Maccari *et al.*, 2005; Slayden & Barry, 2000). As part of our current work on the synthesis of (*E*)-*N*′-substituted isonicotinohydrazide derivatives, in this paper we present the crystal structure of the title compound, (I).

The asymmetric unit consists of one Schiff base molecule and one water molecule (Fig. 1). The geometric parameters are comparable to those related structures (Naveenkumar *et al.*, 2009, 2010; Shi, 2005). The molecule is nearly coplanar with a dihedral angle between the benzene ring and the pyridine ring of 6.64 (7)°. The molecule exists in an *E* configuration with respect to the C7=N1 double bond. In the crystal structure, the water molecules link the molecules into two-dimensional planes parallel to the *ab* plane through intermolecular N–H···O, O–H···O O–H···N and C–H···O hydrogen bonds (Fig. 2, Table 1).

S2. Experimental

The isoniazid derivative was prepared following a procedure reported by Lourenco *et al.*, (2008). 2,4,5-triflurobenzaldehyde (1.0 eq) was added to isoniazid (1.0 eq) in ethanol/water. After stirring for 1-3 h at room temperature, the resulting mixture was concentrated under reduced pressure. The residue was purified by washing with cold ethanol and diethyl ether to give the pure derivative. Colourless single crystals suitable for X-ray analysis were obtained by re-crystallization from methanol.

S3. Refinement

All hydrogen atoms were located from the difference Fourier map and refined freely.

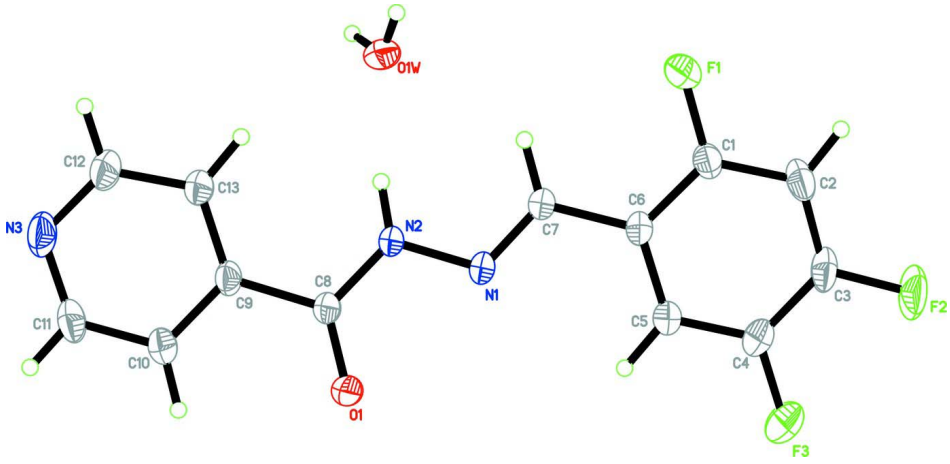

Figure 1

The molecular structure of (I) with atom labels and 50% probability ellipsoids for non-H atoms.

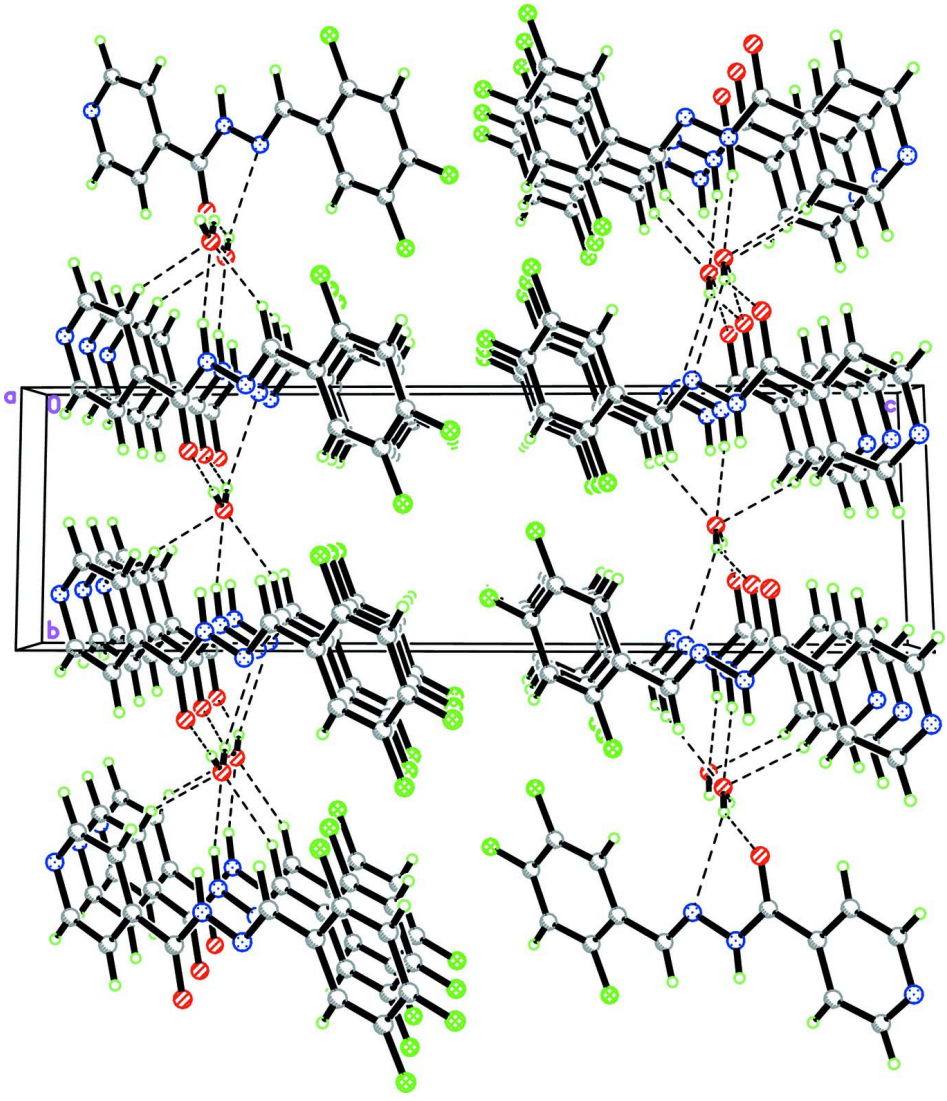

Figure 2

The crystal packing of (I), viewed down the *a* axis, showing the molecules are linked into 2-dimensional planes parallel to the *ab* plane. Intermolecular hydrogen bonds are shown as dashed lines.

(*E*)-*N*′-(2,4,5-Trifluorobenzylidene)isonicotinohydrazide monohydrate

Crystal data

$C_{13}H_8F_3N_3O\cdot H_2O$
$M_r = 297.24$
Triclinic, $P\overline{1}$
Hall symbol: -P 1
$a = 4.9241\ (1)$ Å
$b = 6.3915\ (1)$ Å
$c = 21.3387\ (2)$ Å
$\alpha = 88.616\ (1)°$
$\beta = 86.556\ (1)°$
$\gamma = 76.056\ (1)°$
$V = 650.58\ (2)$ Å^3
$Z = 2$
$F(000) = 304$
$D_x = 1.517$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 6031 reflections
$\theta = 2.9–30.8°$
$\mu = 0.13$ mm^{-1}
$T = 296$ K
Plate, colourless
0.32 × 0.32 × 0.13 mm

Data collection

Bruker SMART APEXII CCD area-detector diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2009)
T_{min} = 0.958, T_{max} = 0.984
14710 measured reflections
4024 independent reflections
2730 reflections with $I > 2\sigma(I)$
R_{int} = 0.022
θ_{max} = 30.8°, θ_{min} = 2.9°
h = −6→7
k = −9→9
l = −30→29

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.046
$wR(F^2)$ = 0.146
S = 1.05
4024 reflections
230 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
All H-atom parameters refined
$w = 1/[\sigma^2(F_o^2) + (0.0764P)^2 + 0.0515P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.27 e Å^{-3}
$\Delta\rho_{min}$ = −0.22 e Å^{-3}

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.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	1.20930 (18)	0.63383 (14)	0.34682 (4)	0.0667 (3)
F2	1.2581 (2)	1.19398 (18)	0.48033 (4)	0.0824 (3)
F3	0.8111 (2)	1.44958 (15)	0.42966 (5)	0.0834 (3)
O1	0.1646 (2)	1.24750 (13)	0.19113 (5)	0.0552 (3)
N1	0.5532 (2)	1.00798 (15)	0.25981 (4)	0.0386 (2)
N2	0.4249 (2)	0.91747 (15)	0.21550 (4)	0.0377 (2)
N3	−0.1882 (3)	0.7926 (2)	0.04246 (6)	0.0592 (3)
C1	1.1071 (3)	0.8366 (2)	0.36730 (6)	0.0431 (3)
C2	1.2389 (3)	0.9080 (3)	0.41488 (6)	0.0532 (3)
C3	1.1375 (3)	1.1151 (3)	0.43436 (6)	0.0533 (3)
C4	0.9082 (3)	1.2479 (2)	0.40781 (6)	0.0520 (3)
C5	0.7792 (3)	1.1753 (2)	0.36057 (6)	0.0465 (3)
C6	0.8779 (2)	0.96531 (19)	0.33900 (5)	0.0381 (3)
C7	0.7429 (2)	0.87952 (19)	0.28944 (5)	0.0391 (3)
C8	0.2279 (2)	1.05161 (17)	0.18276 (5)	0.0381 (3)
C9	0.0888 (2)	0.95209 (18)	0.13457 (5)	0.0381 (3)

C10	−0.1271 (3)	1.0858 (2)	0.10359 (6)	0.0469 (3)
C11	−0.2587 (3)	0.9994 (3)	0.05883 (6)	0.0549 (3)
C12	0.0208 (4)	0.6670 (3)	0.07203 (7)	0.0645 (4)
C13	0.1642 (3)	0.7368 (2)	0.11825 (7)	0.0555 (4)
O1W	0.6042 (3)	0.46898 (15)	0.22162 (5)	0.0567 (3)
H2A	1.388 (4)	0.807 (3)	0.4315 (9)	0.079 (5)*
H5A	0.619 (4)	1.275 (3)	0.3436 (8)	0.069 (5)*
H7A	0.798 (3)	0.727 (2)	0.2805 (7)	0.054 (4)*
H10A	−0.178 (3)	1.237 (3)	0.1110 (7)	0.061 (4)*
H11A	−0.398 (4)	1.092 (3)	0.0388 (8)	0.069 (5)*
H12A	0.087 (4)	0.519 (3)	0.0585 (9)	0.084 (6)*
H13A	0.316 (4)	0.636 (3)	0.1371 (8)	0.073 (5)*
H1N2	0.479 (3)	0.776 (3)	0.2116 (8)	0.067 (5)*
H1W1	0.759 (4)	0.402 (3)	0.2127 (8)	0.071 (6)*
H2W1	0.499 (4)	0.384 (4)	0.2232 (10)	0.099 (7)*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0610 (5)	0.0571 (5)	0.0724 (6)	0.0110 (4)	−0.0253 (4)	−0.0153 (4)
F2	0.0779 (6)	0.1120 (8)	0.0649 (6)	−0.0278 (6)	−0.0330 (5)	−0.0306 (5)
F3	0.0999 (8)	0.0612 (5)	0.0897 (7)	−0.0114 (5)	−0.0295 (6)	−0.0347 (5)
O1	0.0565 (6)	0.0337 (4)	0.0772 (6)	−0.0068 (4)	−0.0346 (5)	−0.0034 (4)
N1	0.0405 (5)	0.0378 (5)	0.0402 (5)	−0.0114 (4)	−0.0145 (4)	−0.0050 (4)
N2	0.0408 (5)	0.0331 (5)	0.0412 (5)	−0.0087 (4)	−0.0177 (4)	−0.0039 (4)
N3	0.0653 (8)	0.0712 (8)	0.0501 (6)	−0.0285 (6)	−0.0241 (5)	−0.0041 (6)
C1	0.0381 (6)	0.0491 (7)	0.0410 (6)	−0.0062 (5)	−0.0103 (5)	−0.0040 (5)
C2	0.0407 (7)	0.0730 (9)	0.0446 (7)	−0.0073 (6)	−0.0172 (5)	−0.0031 (6)
C3	0.0490 (7)	0.0758 (9)	0.0409 (6)	−0.0220 (7)	−0.0140 (5)	−0.0131 (6)
C4	0.0593 (8)	0.0503 (7)	0.0499 (7)	−0.0161 (6)	−0.0125 (6)	−0.0129 (5)
C5	0.0492 (7)	0.0431 (6)	0.0480 (7)	−0.0086 (5)	−0.0183 (5)	−0.0049 (5)
C6	0.0377 (6)	0.0422 (6)	0.0367 (5)	−0.0119 (4)	−0.0110 (4)	−0.0020 (4)
C7	0.0411 (6)	0.0361 (5)	0.0415 (6)	−0.0091 (4)	−0.0139 (5)	−0.0039 (4)
C8	0.0372 (6)	0.0355 (5)	0.0442 (6)	−0.0107 (4)	−0.0138 (5)	−0.0013 (4)
C9	0.0378 (6)	0.0433 (6)	0.0375 (6)	−0.0153 (5)	−0.0126 (4)	−0.0005 (4)
C10	0.0449 (7)	0.0509 (7)	0.0451 (7)	−0.0084 (5)	−0.0161 (5)	−0.0017 (5)
C11	0.0484 (7)	0.0724 (9)	0.0459 (7)	−0.0138 (7)	−0.0216 (6)	0.0011 (6)
C12	0.0858 (11)	0.0528 (8)	0.0630 (9)	−0.0250 (8)	−0.0335 (8)	−0.0060 (7)
C13	0.0671 (9)	0.0427 (7)	0.0603 (8)	−0.0132 (6)	−0.0322 (7)	−0.0036 (6)
O1W	0.0608 (7)	0.0322 (4)	0.0771 (7)	−0.0067 (4)	−0.0194 (5)	−0.0063 (4)

Geometric parameters (Å, º)

F1—C1	1.3454 (15)	C5—C6	1.3922 (17)
F2—C3	1.3445 (13)	C5—H5A	0.969 (18)
F3—C4	1.3463 (16)	C6—C7	1.4660 (14)
O1—C8	1.2299 (13)	C7—H7A	0.965 (15)
N1—C7	1.2737 (15)	C8—C9	1.5025 (14)

N1—N2	1.3791 (11)	C9—C13	1.3835 (17)
N2—C8	1.3477 (14)	C9—C10	1.3843 (17)
N2—H1N2	0.881 (18)	C10—C11	1.3821 (17)
N3—C12	1.326 (2)	C10—H10A	0.953 (16)
N3—C11	1.3324 (19)	C11—H11A	0.911 (19)
C1—C2	1.3800 (17)	C12—C13	1.3882 (17)
C1—C6	1.3877 (16)	C12—H12A	0.966 (19)
C2—C3	1.363 (2)	C13—H13A	0.963 (18)
C2—H2A	0.93 (2)	O1W—H1W1	0.79 (2)
C3—C4	1.381 (2)	O1W—H2W1	0.83 (2)
C4—C5	1.3699 (16)		

C7—N1—N2	116.55 (9)	N1—C7—C6	118.93 (10)
C8—N2—N1	117.36 (9)	N1—C7—H7A	121.8 (9)
C8—N2—H1N2	126.2 (11)	C6—C7—H7A	119.2 (9)
N1—N2—H1N2	116.5 (11)	O1—C8—N2	122.04 (9)
C12—N3—C11	116.24 (11)	O1—C8—C9	120.84 (10)
F1—C1—C2	118.25 (11)	N2—C8—C9	117.12 (9)
F1—C1—C6	118.70 (10)	C13—C9—C10	117.67 (10)
C2—C1—C6	123.04 (12)	C13—C9—C8	124.60 (10)
C3—C2—C1	117.80 (12)	C10—C9—C8	117.72 (10)
C3—C2—H2A	126.1 (11)	C11—C10—C9	119.15 (12)
C1—C2—H2A	116.1 (11)	C11—C10—H10A	119.8 (9)
F2—C3—C2	120.48 (12)	C9—C10—H10A	121.0 (9)
F2—C3—C4	118.53 (13)	N3—C11—C10	123.93 (13)
C2—C3—C4	120.99 (11)	N3—C11—H11A	119.1 (11)
F3—C4—C5	120.41 (12)	C10—C11—H11A	116.9 (11)
F3—C4—C3	118.89 (11)	N3—C12—C13	124.42 (14)
C5—C4—C3	120.70 (12)	N3—C12—H12A	117.9 (11)
C4—C5—C6	120.06 (12)	C13—C12—H12A	117.5 (11)
C4—C5—H5A	117.3 (10)	C9—C13—C12	118.58 (13)
C6—C5—H5A	122.7 (10)	C9—C13—H13A	121.8 (10)
C1—C6—C5	117.41 (10)	C12—C13—H13A	119.6 (10)
C1—C6—C7	120.67 (11)	H1W1—O1W—H2W1	108.0 (19)
C5—C6—C7	121.89 (10)		

C7—N1—N2—C8	−178.64 (11)	N2—N1—C7—C6	−177.37 (10)
F1—C1—C2—C3	178.60 (12)	C1—C6—C7—N1	−172.44 (12)
C6—C1—C2—C3	−0.2 (2)	C5—C6—C7—N1	9.12 (19)
C1—C2—C3—F2	−179.70 (12)	N1—N2—C8—O1	0.66 (18)
C1—C2—C3—C4	0.8 (2)	N1—N2—C8—C9	−179.99 (9)
F2—C3—C4—F3	−1.2 (2)	O1—C8—C9—C13	174.03 (13)
C2—C3—C4—F3	178.33 (14)	N2—C8—C9—C13	−5.32 (19)
F2—C3—C4—C5	179.61 (13)	O1—C8—C9—C10	−4.90 (18)
C2—C3—C4—C5	−0.8 (2)	N2—C8—C9—C10	175.74 (11)
F3—C4—C5—C6	−178.86 (13)	C13—C9—C10—C11	0.8 (2)
C3—C4—C5—C6	0.3 (2)	C8—C9—C10—C11	179.78 (12)
F1—C1—C6—C5	−179.11 (12)	C12—N3—C11—C10	0.3 (2)

C2—C1—C6—C5	−0.3 (2)	C9—C10—C11—N3	−1.0 (2)
F1—C1—C6—C7	2.38 (18)	C11—N3—C12—C13	0.7 (3)
C2—C1—C6—C7	−178.84 (12)	C10—C9—C13—C12	0.1 (2)
C4—C5—C6—C1	0.3 (2)	C8—C9—C13—C12	−178.85 (13)
C4—C5—C6—C7	178.75 (12)	N3—C12—C13—C9	−0.9 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H1*N*2···O1*W*	0.883 (19)	1.921 (19)	2.7910 (13)	168.1 (16)
O1*W*—H1*W*1···O1^{i}	0.79 (2)	2.03 (2)	2.8263 (17)	176.3 (18)
O1*W*—H2*W*1···O1^{ii}	0.84 (2)	2.19 (2)	2.9670 (17)	155 (2)
O1*W*—H2*W*1···N1^{ii}	0.84 (2)	2.47 (2)	3.1024 (14)	133.7 (19)
C7—H7*A*···O1*W*	0.967 (13)	2.492 (14)	3.2576 (16)	135.9 (12)
C13—H13*A*···O1*W*	0.963 (19)	2.427 (18)	3.3308 (19)	156.2 (14)

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