Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

1-(4-Fluorophenyl)-2-(1*H*-1,2,4-triazol-1-yl)ethanone hemihydrate

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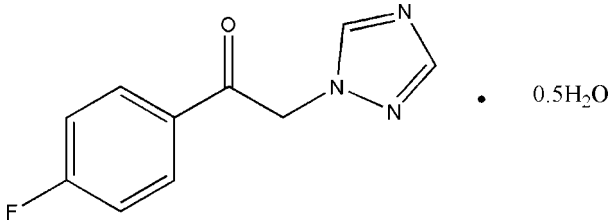
Received 26 October 2011; accepted 29 October 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.054; wR factor = 0.160; data-to-parameter ratio = 12.7.

In the title compound, $C_{10}H_8FN_3O\cdot0.5H_2O$, the dihedral angle between the mean planes of the rings is 99.80 (4)°. The water molecule lies on a twofold axis. Weak intermolecular O—H···N and C—H···O hydrogen bonds link one water molecule with four phenylethanone molecules, while intermolecular C—H···O hydrogen bonds involving the ketone group link phenylethanone molecules into layers parallel to (100).

Related literature

For related compounds containing a 2-(1*H*-1,2,4-triazol-1-yl)-1-phenylethanone fragment, see: Akira *et al.* (1985); Yoshimi *et al.* (2000); Yuan *et al.* (2007); Tao *et al.* (2007). For standard bond lengths, see: Allen *et al.* (1987).



Experimental

Crystal data

$C_{10}H_8FN_3O\cdot0.5H_2O$
M_r = 214.2
Orthorhombic, *Pbcn*
a = 24.419 (5) Å
b = 10.147 (2) Å
c = 8.2410 (16) Å
V = 2042.0 (7) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.11 mm^{-1}
T = 293 K
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.968, T_{max} = 0.978
3626 measured reflections
1844 independent reflections
1087 reflections with $I > 2\sigma(I)$
R_{int} = 0.057
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.054
$wR(F^2)$ = 0.160
S = 1.00
1844 reflections
145 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.17 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
OW—HWA···N1	0.80 (4)	2.18 (4)	2.957 (3)	166 (4)
C2—H2B···O^{i}	0.93	2.54	3.309 (3)	140
C3—H3B···O^{ii}	0.97	2.51	3.459 (3)	166
C7—H7A···OW^{iii}	0.93	2.49	3.407 (4)	169

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

Data collection: *CAD-4 EXPRESS* (Enraf–Nonius, 1994); cell refinement: *CAD-4 EXPRESS*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *PLATON* (Spek, 2009); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank the Center of Testing and Analysis, Nanjing University, for support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2131).

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supporting information

Acta Cryst. (2011). E**67**, o3170 [https://doi.org/10.1107/S1600536811045429]

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S1. Comment

The title compound is the key intermediate in the synthesis of a new kind of antifungal drug (Yoshimi *et al.*, 2000; Akira *et al.*, 1985). The crystal structure determination has been carried out in order to elucidate the molecular conformation.

The molecular structure of the title compound is reported in Fig. 1. The bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. The dihedral angle between the mean planes of the N1/N2/C9–C11 and C1–C6 rings is 99.80 (4)°. The solvent molecules of water lie on two-fold axes. The second position of the hydrogen atom is reproduced by the symmetry operation (-x, y, 0.5-z). Weak intermolecular O—H···N (Fig. 2) and C—H···O hydrogen bonds link one isolated water molecule with four phenylethanone molecules while intermolecular C—H···O hydrogen bonds involving the ketone group link phenylethanone molecules together into layers parallel to (100).

S2. Experimental

Sodium hydride (4.8 g, 120 mmol) was suspended in dimethylformamide (DMF, 30 ml). Triazole (8.28 g, 120 mmol) dissolved in DMF (30 ml) was slowly added dropwise at 273 K, and reacted at room temperature for 30 min. 2-Chloro-1-(4-fluorophenyl)ethanone (15.48 g, 90 mmol) dissolved in DMF (30 ml) was then slowly added dropwise, and reacted at room temperature for 4 h. The mixture was placed in ice-water (300 ml), and 1 mol hydrochloric acid (50 ml) was then added. After filtration, the filtrate was neutralized with sodium bicarbonate to pH = 6, and a yellow deposit was obtained. Recrystallization with ethanol yielded a white deposit (m.p. 397–400 K). Crystals suitable for a X-ray analysis study were obtained by dissolving the crude product (1.0 g) in 95% ethanol (30 ml) and then allowing the solution to evaporate slowly at room temperature for about 7 days.

S3. Refinement

The water H atom (the second position being obtained by a symmetry operation) was located in a Fourier difference map and freely refined with U_{iso}(H) = 1.2U_{eq}(O). The other H atoms were positioned geometrically with C—H = 0.93 Å (aromatic) and 0.97 Å (methylene) and constrained to ride on their parent atoms with U_{iso}(H) = 1.2U_{eq}(C).

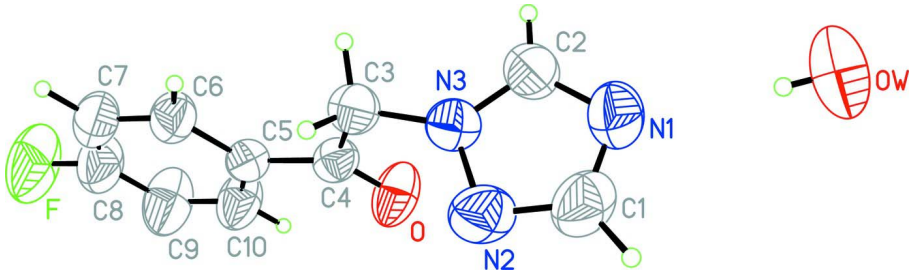

Figure 1

The molecular structure of the title compound with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level.

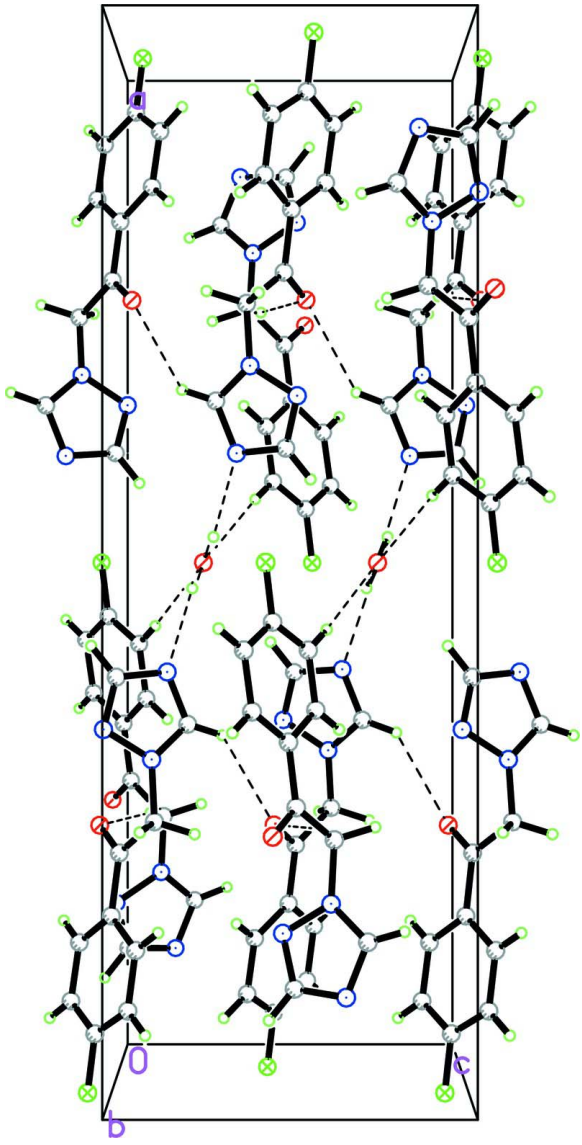

Figure 2

A packing diagram of the title compound showing the intermolecular O—H···N hydrogen bonds (dashed lines).

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Crystal data

$C_{10}H_8FN_3O·0.5H_2O$
$M_r = 214.2$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 24.419$ (5) Å
$b = 10.147$ (2) Å
$c = 8.2410$ (16) Å
$V = 2042.0$ (7) Å^3
$Z = 8$
$F(000) = 888$
$D_x = 1.394$ Mg m^{-3}
Melting point: 397 K
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–13°
$\mu = 0.11$ mm^{-1}
$T = 293$ K
Prism, yellow
0.3 × 0.2 × 0.2 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.968$, $T_{max} = 0.978$
3626 measured reflections
1844 independent reflections
1087 reflections with $I > 2\sigma(I)$
$R_{int} = 0.057$
$\theta_{max} = 25.4°$, $\theta_{min} = 1.7°$
$h = -29\rightarrow29$
$k = -12\rightarrow0$
$l = 0\rightarrow9$
3 standard reflections every 200 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.160$
$S = 1.00$
1844 reflections
145 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
$w = 1/[\sigma^2(F_o^2) + (0.084P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.17$ e Å^{-3}
$\Delta\rho_{min} = -0.15$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.062 (7)

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
O	0.25394 (7)	0.00400 (18)	−0.0459 (2)	0.0749 (6)
F	0.50013 (8)	0.1731 (3)	−0.0667 (3)	0.1359 (10)

N1	0.10453 (9)	0.0848 (3)	0.1516 (3)	0.0769 (8)
C1	0.10833 (13)	0.1521 (3)	0.0121 (4)	0.0774 (9)
H1B	0.0787	0.1626	−0.0575	0.093*
N2	0.15669 (10)	0.2021 (3)	−0.0199 (3)	0.0722 (7)
C2	0.15429 (11)	0.0941 (3)	0.2103 (3)	0.0640 (7)
H2B	0.1658	0.0569	0.3076	0.077*
N3	0.18595 (8)	0.16374 (19)	0.1122 (2)	0.0546 (6)
C3	0.24381 (10)	0.1914 (2)	0.1215 (3)	0.0539 (7)
H3A	0.2558	0.1837	0.2333	0.065*
H3B	0.2504	0.2812	0.0864	0.065*
C4	0.27685 (10)	0.0978 (2)	0.0168 (3)	0.0492 (6)
C5	0.33581 (10)	0.1223 (2)	−0.0041 (3)	0.0496 (7)
C6	0.36302 (11)	0.2249 (3)	0.0712 (3)	0.0608 (7)
H6A	0.3436	0.2831	0.1367	0.073*
C7	0.41853 (13)	0.2421 (3)	0.0504 (4)	0.0759 (9)
H7A	0.4370	0.3103	0.1024	0.091*
C8	0.44541 (13)	0.1573 (4)	−0.0475 (4)	0.0867 (10)
C9	0.42071 (14)	0.0552 (3)	−0.1240 (4)	0.1019 (12)
H9A	0.4407	−0.0014	−0.1901	0.122*
C10	0.36552 (12)	0.0370 (3)	−0.1017 (3)	0.0778 (9)
H10A	0.3479	−0.0330	−0.1525	0.093*
OW	0.0000	−0.0387 (3)	0.2500	0.1023 (13)
HWA	0.0248 (15)	0.006 (4)	0.220 (5)	0.123*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O	0.0593 (11)	0.0603 (11)	0.1051 (14)	−0.0056 (10)	−0.0064 (11)	−0.0288 (11)
F	0.0577 (12)	0.172 (2)	0.178 (2)	−0.0314 (14)	0.0201 (13)	−0.0477 (18)
N1	0.0560 (16)	0.0907 (18)	0.0842 (17)	0.0002 (13)	0.0055 (13)	−0.0031 (15)
C1	0.0541 (19)	0.099 (2)	0.079 (2)	0.0195 (17)	−0.0088 (15)	−0.0097 (19)
N2	0.0639 (16)	0.0880 (17)	0.0647 (15)	0.0182 (13)	−0.0064 (12)	0.0102 (12)
C2	0.0618 (18)	0.0686 (17)	0.0616 (16)	0.0001 (14)	0.0035 (14)	0.0044 (14)
N3	0.0553 (13)	0.0557 (12)	0.0528 (12)	0.0055 (10)	−0.0002 (10)	0.0006 (10)
C3	0.0585 (16)	0.0502 (13)	0.0530 (13)	−0.0014 (11)	0.0003 (12)	−0.0006 (12)
C4	0.0551 (15)	0.0431 (12)	0.0495 (14)	−0.0019 (11)	−0.0063 (11)	0.0005 (11)
C5	0.0520 (15)	0.0504 (14)	0.0465 (13)	−0.0037 (11)	−0.0024 (11)	0.0004 (11)
C6	0.0620 (17)	0.0580 (15)	0.0623 (15)	−0.0105 (13)	0.0025 (13)	−0.0093 (13)
C7	0.069 (2)	0.080 (2)	0.0786 (19)	−0.0229 (16)	0.0005 (16)	−0.0123 (16)
C8	0.057 (2)	0.107 (3)	0.096 (2)	−0.0199 (18)	0.0096 (17)	−0.015 (2)
C9	0.066 (2)	0.113 (3)	0.126 (3)	−0.0109 (19)	0.027 (2)	−0.053 (2)
C10	0.0627 (19)	0.082 (2)	0.088 (2)	−0.0122 (15)	0.0084 (16)	−0.0342 (17)
OW	0.071 (2)	0.085 (2)	0.150 (3)	0.000	0.034 (2)	0.000

Geometric parameters (Å, º)

O—C4	1.219 (3)	C4—C5	1.471 (3)
F—C8	1.355 (3)	C5—C6	1.382 (3)

N1—C2	1.311 (3)	C5—C10	1.387 (3)
N1—C1	1.341 (4)	C6—C7	1.377 (4)
C1—N2	1.312 (4)	C6—H6A	0.9300
C1—H1B	0.9300	C7—C8	1.350 (4)
N2—N3	1.359 (3)	C7—H7A	0.9300
C2—N3	1.323 (3)	C8—C9	1.354 (4)
C2—H2B	0.9300	C9—C10	1.372 (4)
N3—C3	1.443 (3)	C9—H9A	0.9300
C3—C4	1.516 (3)	C10—H10A	0.9300
C3—H3A	0.9700	OW—HWA	0.79 (4)
C3—H3B	0.9700		

C2—N1—C1	102.4 (2)	C5—C4—C3	118.8 (2)
N2—C1—N1	115.6 (3)	C6—C5—C10	118.6 (2)
N2—C1—H1B	122.2	C6—C5—C4	123.1 (2)
N1—C1—H1B	122.2	C10—C5—C4	118.3 (2)
C1—N2—N3	101.6 (2)	C7—C6—C5	120.9 (3)
N1—C2—N3	110.8 (2)	C7—C6—H6A	119.6
N1—C2—H2B	124.6	C5—C6—H6A	119.6
N3—C2—H2B	124.6	C8—C7—C6	118.1 (3)
C2—N3—N2	109.6 (2)	C8—C7—H7A	120.9
C2—N3—C3	130.1 (2)	C6—C7—H7A	120.9
N2—N3—C3	120.1 (2)	C7—C8—C9	123.3 (3)
N3—C3—C4	111.66 (19)	C7—C8—F	118.3 (3)
N3—C3—H3A	109.3	C9—C8—F	118.4 (3)
C4—C3—H3A	109.3	C8—C9—C10	118.6 (3)
N3—C3—H3B	109.3	C8—C9—H9A	120.7
C4—C3—H3B	109.3	C10—C9—H9A	120.7
H3A—C3—H3B	107.9	C9—C10—C5	120.5 (3)
O—C4—C5	122.1 (2)	C9—C10—H10A	119.8
O—C4—C3	119.1 (2)	C5—C10—H10A	119.8

C2—N1—C1—N2	0.1 (3)	O—C4—C5—C10	1.6 (4)
N1—C1—N2—N3	−0.5 (3)	C3—C4—C5—C10	−179.2 (2)
C1—N1—C2—N3	0.4 (3)	C10—C5—C6—C7	−0.2 (4)
N1—C2—N3—N2	−0.8 (3)	C4—C5—C6—C7	178.6 (2)
N1—C2—N3—C3	−175.2 (2)	C5—C6—C7—C8	1.0 (4)
C1—N2—N3—C2	0.7 (3)	C6—C7—C8—C9	−1.1 (5)
C1—N2—N3—C3	175.8 (2)	C6—C7—C8—F	−179.2 (3)
C2—N3—C3—C4	97.0 (3)	C7—C8—C9—C10	0.3 (6)
N2—N3—C3—C4	−76.9 (3)	F—C8—C9—C10	178.5 (3)
N3—C3—C4—O	−8.4 (3)	C8—C9—C10—C5	0.5 (5)
N3—C3—C4—C5	172.43 (19)	C6—C5—C10—C9	−0.6 (4)
O—C4—C5—C6	−177.3 (2)	C4—C5—C10—C9	−179.5 (3)
C3—C4—C5—C6	1.9 (3)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O*W*—H*WA*···N1	0.80 (4)	2.18 (4)	2.957 (3)	166 (4)
C2—H2*B*···O^{i}	0.93	2.54	3.309 (3)	140
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