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1-Phenyl-5-[4-(trifluoromethyl)phenyl]-pyrazolidin-3-one monohydrate

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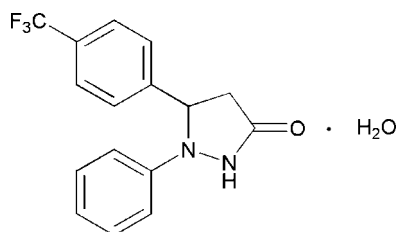
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 Key indicators: single-crystal X-ray study; $T = 294$ K; mean $\sigma(\text{C}-\text{C}) = 0.006$ Å; disorder in solvent or counterion; R factor = 0.077; wR factor = 0.186; data-to-parameter ratio = 15.1.

 In the molecule of the title compound, $\text{C}_{16}\text{H}_{13}\text{F}_3\text{N}_2\text{O}\cdot\text{H}_2\text{O}$, the two benzene rings are oriented at a dihedral angle of $82.55(3)^\circ$ and the pyrazole ring adopts an envelope conformation. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{F}$ hydrogen bonds link the molecules into chains.

Related literature

 For general background, see: Menozzi *et al.* (1990); James & William (2003); Shi *et al.* (2006). For related literature, see: Jia *et al.* (2008). For bond-length data, see: Allen *et al.* (1987).


Experimental

Crystal data

 $\text{C}_{16}\text{H}_{13}\text{F}_3\text{N}_2\text{O}\cdot\text{H}_2\text{O}$
 $M_r = 324.30$
 Triclinic, $P\bar{1}$
 $a = 7.4960(15)$ Å
 $b = 9.794(2)$ Å
 $c = 13.319(3)$ Å
 $\alpha = 97.70(3)^\circ$
 $\beta = 101.58(3)^\circ$
 $\gamma = 107.97(3)^\circ$
 $V = 890.8(4)$ Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.10$ mm⁻¹
 $T = 294(2)$ K
 $0.20 \times 0.10 \times 0.05$ mm

Data collection

 Enraf–Nonius CAD-4
 diffractometer
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\min} = 0.980$, $T_{\max} = 0.995$
 3463 measured reflections

 3196 independent reflections
 1611 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.068$
 3 standard reflections
 frequency: 120 min
 intensity decay: none

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.077$
 $wR(F^2) = 0.186$
 $S = 1.00$
 3196 reflections
 211 parameters

 48 restraints
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.21$ e Å⁻³
 $\Delta\rho_{\min} = -0.23$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|-----------------------------------|-------|-------------|-------------|---------------|
| OW2—HW2B \cdots OW1 | 0.85 | 1.99 | 2.50 (3) | 118 |
| C14—H14A \cdots F3 ⁱ | 0.93 | 2.54 | 3.262 (6) | 134 |
| N1—H1A \cdots O ⁱⁱ | 0.86 | 1.98 | 2.811 (6) | 161 |
| C9—H9B \cdots O ⁱⁱⁱ | 0.97 | 2.60 | 3.555 (7) | 168 |

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

 Data collection: *CAD-4 Software* (Enraf–Nonius, 1985); cell refinement: *CAD-4 Software*; 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: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HK2518).

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supplementary materials

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1-Phenyl-5-[4-(trifluoromethyl)phenyl]pyrazolidin-3-one monohydrate

Y.-Y. Liu, Z.-Y. Wu, H. Shi, Q.-Y. Chu and H.-J. Zhu

Comment

Nowadays, heterocyclic compounds as medicines and pesticides have been developed most quickly. Among them, pyrazole and its derivatives exhibit better bioactivity, such as antipyretic (Menozzi *et al.*, 1990) and anticancer (James & William, 2003) activities. They are also useful in the treatment of inflammation and related disorders (Shi *et al.*, 2006). We report herein the crystal structure of the title compound.

In the molecule of the title compound (Fig. 1), the bond lengths (Allen *et al.*, 1987) and angles are within normal ranges. Rings A (C2-C7) and C (C11-C16) are, of course, planar and the dihedral angle between them is 82.55 (3)°. Ring B (N1/N2/C8-C10) is not planar, and adopts envelope conformation with C8 atom displaced by 0.358 (3) Å from the plane of the other ring atoms. The intramolecular C-H...N and C-H...F hydrogen bonds (Table 1) result in the formation of two planar five-membered rings D (N2/C4/C5/C8/H4A) and E (F3/C1/C2/C7/H7A). They are oriented with respect to ring A at dihedral angles of 3.20 (3)° and 2.57 (3)°, respectively. So, rings A, D and E are nearly coplanar.

In the crystal structure, intermolecular C-H...F hydrogen bonds (Table 1) link the molecules into chains (Fig. 2), in which they may be effective in the stabilization of the structure.

Experimental

The title compound was prepared by the literature method (Jia *et al.*, 2008). For the preparation of the title compound, n-butanol (40 ml) and ethanolamine (4 ml) were added to a solution of sodium (40 mmol) in anhydrous methanol (9 ml). Then, methanol was removed by distillation and methyl 3-(4-(trifluoromethyl)phenyl)acrylate (30 mmol) was added. The mixture was refluxed for 1.5 h at 385 K, after which phenylhydrazine (4 ml) was added. The mixture was refluxed for another 10 h, and then left to cool to room temperature. It was then acidified with acetic acid (50%), allowed to stand and filtered. The filter cake was chromatographed over silica gel (500 g) eluting with a mixture of ethyl acetate and petroleum ether to give the title compound (m.p. 413-415 K). Crystals suitable for x-ray analysis were obtained by dissolving the title compound (1.5 g) in ethyl acetate (25 ml) and evaporating the solvent slowly at room temperature for about 10 d.

Refinement

The water molecule was disordered. During the refinement process the disordered O and H atoms were refined with occupancies of 0.50. H atoms were positioned geometrically, with O-H = 0.85 Å (for H₂O), N-H = 0.86 (for NH) and C-H = 0.93, 0.98 and 0.97 Å for aromatic, methine and methylene H, respectively, and constrained to ride on their parent atoms with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C,N,O})$.

Figures

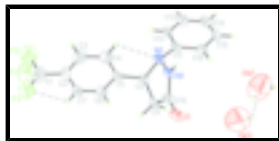


Fig. 1. The molecular structure of the title molecule, with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. Hydrogen bonds are shown as dotted lines.

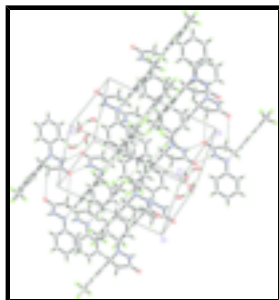


Fig. 2. A packing diagram of the title compound. Hydrogen bonds are shown as dashed lines.

1-Phenyl-5-[4-(trifluoromethyl)phenyl]pyrazolidin-3-one monohydrate

Crystal data

$C_{16}H_{13}F_3N_2O \cdot H_2O$

$M_r = 324.30$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 7.4960$ (15) Å

$b = 9.794$ (2) Å

$c = 13.319$ (3) Å

$\alpha = 97.70$ (3)°

$\beta = 101.58$ (3)°

$\gamma = 107.97$ (3)°

$V = 890.8$ (4) Å³

$Z = 2$

$F_{000} = 336$

$D_x = 1.209$ Mg m⁻³

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 0.10$ mm⁻¹

$T = 294$ (2) K

Needle, colorless

$0.20 \times 0.10 \times 0.05$ mm

Data collection

Enraf-Nonius CAD-4
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 294$ (2) K

$\omega/2\theta$ scans

Absorption correction: ψ scan
(North *et al.*, 1968)

$T_{\min} = 0.980$, $T_{\max} = 0.995$

3463 measured reflections

3196 independent reflections

1611 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.068$

$\theta_{\max} = 25.2^\circ$

$\theta_{\min} = 1.6^\circ$

$h = -8 \rightarrow 8$

$k = -11 \rightarrow 11$

$l = 0 \rightarrow 15$

3 standard reflections

every 120 min

intensity decay: none

Refinement

| | |
|--|--|
| Refinement on F^2 | Secondary atom site location: difference Fourier map |
| Least-squares matrix: full | Hydrogen site location: inferred from neighbouring sites |
| $R[F^2 > 2\sigma(F^2)] = 0.077$ | H-atom parameters constrained |
| $wR(F^2) = 0.186$ | $w = 1/[\sigma^2(F_o^2) + (0.06P)^2 + 0.58P]$ |
| $S = 1.00$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| 3196 reflections | $(\Delta/\sigma)_{\max} < 0.001$ |
| 211 parameters | $\Delta\rho_{\max} = 0.21 \text{ e } \text{\AA}^{-3}$ |
| 48 restraints | $\Delta\rho_{\min} = -0.23 \text{ e } \text{\AA}^{-3}$ |
| Primary atom site location: structure-invariant direct methods | Extinction correction: none |

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 (\AA^2)

| | <i>x</i> | <i>y</i> | <i>z</i> | $U_{\text{iso}}^*/U_{\text{eq}}$ | Occ. (<1) |
|------|------------|-------------|-------------|----------------------------------|-----------|
| OW1 | 0.464 (3) | 1.4138 (15) | 1.0235 (11) | 0.288 (8) | 0.50 |
| HW1A | 0.5305 | 1.3813 | 1.0675 | 0.345* | 0.50 |
| HW1B | 0.4367 | 1.4891 | 1.0439 | 0.345* | 0.50 |
| OW2 | 0.805 (3) | 1.5022 (19) | 1.0058 (13) | 0.328 (9) | 0.50 |
| HW2A | 0.8673 | 1.5642 | 0.9752 | 0.394* | 0.50 |
| HW2B | 0.7436 | 1.5280 | 1.0477 | 0.394* | 0.50 |
| O | 0.7711 (4) | 1.0639 (3) | 1.0297 (2) | 0.0741 (9) | |
| N1 | 0.5378 (4) | 0.9682 (3) | 0.8750 (2) | 0.0520 (8) | |
| H1A | 0.4422 | 0.9388 | 0.9027 | 0.062* | |
| N2 | 0.5122 (4) | 0.9482 (3) | 0.7661 (2) | 0.0495 (7) | |
| F1 | 0.8128 (5) | 0.3357 (4) | 0.6898 (3) | 0.1429 (12) | |
| F2 | 0.7729 (6) | 0.3635 (4) | 0.5389 (3) | 0.1549 (14) | |
| F3 | 1.0292 (5) | 0.4452 (4) | 0.6316 (3) | 0.1343 (11) | |
| C1 | 0.8642 (9) | 0.4424 (7) | 0.6409 (5) | 0.097 | |
| C2 | 0.8153 (7) | 0.5692 (4) | 0.6625 (3) | 0.0695 (11) | |
| C3 | 0.6372 (7) | 0.5629 (5) | 0.6753 (4) | 0.0924 (15) | |
| H3A | 0.5419 | 0.4721 | 0.6676 | 0.111* | |

supplementary materials

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|------|------------|------------|------------|-------------|
| C4 | 0.5972 (6) | 0.6888 (5) | 0.6994 (4) | 0.0869 (14) |
| H4A | 0.4717 | 0.6827 | 0.7014 | 0.104* |
| C5 | 0.7395 (5) | 0.8228 (4) | 0.7207 (3) | 0.0480 (9) |
| C6 | 0.9191 (5) | 0.8233 (4) | 0.7059 (3) | 0.0654 (11) |
| H6A | 1.0176 | 0.9128 | 0.7150 | 0.079* |
| C7 | 0.9539 (6) | 0.6985 (5) | 0.6791 (3) | 0.0660 (11) |
| H7A | 1.0764 | 0.7030 | 0.6721 | 0.079* |
| C8 | 0.7150 (4) | 0.9668 (4) | 0.7541 (2) | 0.0443 (8) |
| H8A | 0.7384 | 1.0243 | 0.7003 | 0.053* |
| C9 | 0.8485 (5) | 1.0570 (4) | 0.8573 (2) | 0.0512 (9) |
| H9A | 0.9001 | 1.1596 | 0.8538 | 0.061* |
| H9B | 0.9556 | 1.0222 | 0.8777 | 0.061* |
| C10 | 0.7225 (5) | 1.0368 (4) | 0.9337 (3) | 0.0568 (10) |
| C11 | 0.4190 (4) | 1.0326 (4) | 0.7171 (3) | 0.0485 (9) |
| C12 | 0.3624 (5) | 1.0040 (4) | 0.6080 (3) | 0.0587 (10) |
| H12A | 0.3921 | 0.9310 | 0.5698 | 0.070* |
| C13 | 0.2660 (6) | 1.0793 (5) | 0.5571 (3) | 0.0694 (12) |
| H13A | 0.2318 | 1.0592 | 0.4842 | 0.083* |
| C14 | 0.2170 (6) | 1.1866 (5) | 0.6116 (4) | 0.0759 (12) |
| H14A | 0.1510 | 1.2393 | 0.5760 | 0.091* |
| C15 | 0.2666 (6) | 1.2142 (4) | 0.7181 (4) | 0.0716 (12) |
| H15A | 0.2292 | 1.2840 | 0.7545 | 0.086* |
| C16 | 0.3695 (5) | 1.1430 (4) | 0.7737 (3) | 0.0557 (10) |
| H16A | 0.4060 | 1.1662 | 0.8466 | 0.067* |

Atomic displacement parameters (\AA^2)

| | U^{11} | U^{22} | U^{33} | U^{12} | U^{13} | U^{23} |
|-----|-------------|-------------|-------------|-------------|-------------|--------------|
| OW1 | 0.37 (2) | 0.239 (16) | 0.249 (16) | 0.119 (17) | 0.069 (15) | 0.038 (12) |
| OW2 | 0.31 (2) | 0.312 (19) | 0.33 (2) | 0.089 (17) | 0.053 (17) | 0.039 (15) |
| O | 0.0489 (17) | 0.132 (3) | 0.0524 (17) | 0.0415 (16) | 0.0149 (13) | 0.0292 (16) |
| N1 | 0.0365 (17) | 0.082 (2) | 0.0465 (17) | 0.0262 (16) | 0.0146 (14) | 0.0257 (15) |
| N2 | 0.0361 (16) | 0.0695 (19) | 0.0517 (18) | 0.0246 (15) | 0.0166 (14) | 0.0191 (15) |
| F1 | 0.161 (3) | 0.128 (3) | 0.146 (3) | 0.051 (2) | 0.048 (3) | 0.037 (2) |
| F2 | 0.168 (4) | 0.152 (3) | 0.143 (3) | 0.061 (3) | 0.033 (3) | 0.020 (2) |
| F3 | 0.134 (3) | 0.131 (3) | 0.149 (3) | 0.054 (2) | 0.046 (2) | 0.030 (2) |
| C1 | 0.106 | 0.099 | 0.095 | 0.045 | 0.038 | 0.010 |
| C2 | 0.080 (3) | 0.060 (2) | 0.079 (3) | 0.037 (2) | 0.025 (2) | 0.013 (2) |
| C3 | 0.084 (3) | 0.070 (3) | 0.119 (4) | 0.013 (2) | 0.043 (3) | 0.009 (3) |
| C4 | 0.053 (3) | 0.076 (3) | 0.124 (4) | 0.011 (2) | 0.037 (3) | 0.004 (3) |
| C5 | 0.0356 (19) | 0.057 (2) | 0.056 (2) | 0.0196 (17) | 0.0133 (16) | 0.0184 (17) |
| C6 | 0.039 (2) | 0.071 (2) | 0.083 (3) | 0.0130 (19) | 0.023 (2) | 0.007 (2) |
| C7 | 0.053 (2) | 0.078 (3) | 0.082 (3) | 0.036 (2) | 0.026 (2) | 0.022 (2) |
| C8 | 0.0291 (18) | 0.068 (2) | 0.0437 (19) | 0.0187 (17) | 0.0230 (15) | 0.0132 (17) |
| C9 | 0.0347 (19) | 0.070 (2) | 0.046 (2) | 0.0167 (18) | 0.0150 (16) | -0.0019 (17) |
| C10 | 0.036 (2) | 0.087 (3) | 0.053 (2) | 0.021 (2) | 0.0159 (18) | 0.026 (2) |
| C11 | 0.0298 (19) | 0.064 (2) | 0.058 (2) | 0.0191 (17) | 0.0170 (17) | 0.0159 (18) |
| C12 | 0.054 (2) | 0.088 (3) | 0.043 (2) | 0.040 (2) | 0.0123 (18) | 0.0052 (19) |

| | | | | | | |
|-----|-----------|-----------|-----------|-----------|-------------|-------------|
| C13 | 0.069 (3) | 0.110 (3) | 0.040 (2) | 0.039 (3) | 0.018 (2) | 0.025 (2) |
| C14 | 0.065 (3) | 0.104 (3) | 0.074 (3) | 0.043 (3) | 0.019 (2) | 0.039 (3) |
| C15 | 0.065 (3) | 0.069 (3) | 0.095 (3) | 0.040 (2) | 0.029 (2) | 0.012 (2) |
| C16 | 0.044 (2) | 0.072 (2) | 0.053 (2) | 0.025 (2) | 0.0106 (18) | 0.0093 (19) |

Geometric parameters (Å, °)

| | | | |
|---------------|-----------|--------------|-----------|
| OW1—HW1A | 0.8502 | C5—C8 | 1.499 (4) |
| OW1—HW1B | 0.8499 | C6—C7 | 1.344 (5) |
| OW2—HW2A | 0.8500 | C6—H6A | 0.9300 |
| OW2—HW2B | 0.8499 | C7—H7A | 0.9300 |
| O—C10 | 1.226 (4) | C8—C9 | 1.499 (4) |
| N1—C10 | 1.352 (4) | C8—H8A | 0.9800 |
| N1—N2 | 1.404 (4) | C9—C10 | 1.513 (4) |
| N1—H1A | 0.8600 | C9—H9A | 0.9700 |
| N2—C11 | 1.384 (4) | C9—H9B | 0.9700 |
| N2—C8 | 1.518 (4) | C11—C12 | 1.392 (5) |
| F1—C1 | 1.303 (6) | C11—C16 | 1.420 (5) |
| F2—C1 | 1.389 (6) | C12—C13 | 1.339 (5) |
| F3—C1 | 1.260 (6) | C12—H12A | 0.9300 |
| C1—C2 | 1.409 (6) | C13—C14 | 1.381 (5) |
| C2—C7 | 1.324 (5) | C13—H13A | 0.9300 |
| C2—C3 | 1.363 (6) | C14—C15 | 1.358 (5) |
| C3—C4 | 1.369 (6) | C14—H14A | 0.9300 |
| C3—H3A | 0.9300 | C15—C16 | 1.368 (5) |
| C4—C5 | 1.363 (5) | C15—H15A | 0.9300 |
| C4—H4A | 0.9300 | C16—H16A | 0.9300 |
| C5—C6 | 1.398 (4) | | |
| HW1A—OW1—HW1B | 120.0 | C5—C8—N2 | 112.4 (3) |
| HW2A—OW2—HW2B | 120.0 | C9—C8—N2 | 105.1 (2) |
| C10—N1—N2 | 115.7 (3) | C5—C8—H8A | 108.2 |
| C10—N1—H1A | 122.2 | C9—C8—H8A | 108.2 |
| N2—N1—H1A | 122.2 | N2—C8—H8A | 108.2 |
| C11—N2—N1 | 115.6 (3) | C8—C9—C10 | 104.5 (3) |
| C11—N2—C8 | 116.8 (3) | C8—C9—H9A | 110.9 |
| N1—N2—C8 | 102.8 (2) | C10—C9—H9A | 110.9 |
| F3—C1—F1 | 103.2 (5) | C8—C9—H9B | 110.9 |
| F3—C1—F2 | 92.8 (4) | C10—C9—H9B | 110.9 |
| F1—C1—F2 | 98.4 (5) | H9A—C9—H9B | 108.9 |
| F3—C1—C2 | 123.8 (6) | O—C10—N1 | 124.4 (3) |
| F1—C1—C2 | 120.7 (5) | O—C10—C9 | 129.1 (3) |
| F2—C1—C2 | 111.8 (5) | N1—C10—C9 | 106.2 (3) |
| C7—C2—C3 | 119.5 (4) | N2—C11—C12 | 119.1 (3) |
| C7—C2—C1 | 117.8 (5) | N2—C11—C16 | 122.5 (3) |
| C3—C2—C1 | 122.4 (5) | C12—C11—C16 | 118.3 (3) |
| C2—C3—C4 | 120.7 (4) | C13—C12—C11 | 121.3 (3) |
| C2—C3—H3A | 119.7 | C13—C12—H12A | 119.3 |
| C4—C3—H3A | 119.7 | C11—C12—H12A | 119.3 |
| C5—C4—C3 | 120.7 (4) | C12—C13—C14 | 120.7 (4) |

supplementary materials

| | | | |
|---------------|------------|-----------------|------------|
| C5—C4—H4A | 119.7 | C12—C13—H13A | 119.6 |
| C3—C4—H4A | 119.7 | C14—C13—H13A | 119.6 |
| C4—C5—C6 | 116.1 (4) | C15—C14—C13 | 119.0 (4) |
| C4—C5—C8 | 125.2 (3) | C15—C14—H14A | 120.5 |
| C6—C5—C8 | 118.7 (3) | C13—C14—H14A | 120.5 |
| C7—C6—C5 | 122.1 (4) | C14—C15—C16 | 122.4 (4) |
| C7—C6—H6A | 118.9 | C14—C15—H15A | 118.8 |
| C5—C6—H6A | 118.9 | C16—C15—H15A | 118.8 |
| C2—C7—C6 | 120.6 (4) | C15—C16—C11 | 118.1 (3) |
| C2—C7—H7A | 119.7 | C15—C16—H16A | 120.9 |
| C6—C7—H7A | 119.7 | C11—C16—H16A | 120.9 |
| C5—C8—C9 | 114.7 (3) | | |
| C10—N1—N2—C11 | -109.8 (3) | C11—N2—C8—C5 | -130.5 (3) |
| C10—N1—N2—C8 | 18.6 (4) | N1—N2—C8—C5 | 101.8 (3) |
| F3—C1—C2—C7 | -3.6 (8) | C11—N2—C8—C9 | 104.2 (3) |
| F1—C1—C2—C7 | 132.2 (6) | N1—N2—C8—C9 | -23.5 (3) |
| F2—C1—C2—C7 | -112.9 (5) | C5—C8—C9—C10 | -103.1 (3) |
| F3—C1—C2—C3 | -178.0 (5) | N2—C8—C9—C10 | 20.8 (4) |
| F1—C1—C2—C3 | -42.2 (8) | N2—N1—C10—O | 179.7 (3) |
| F2—C1—C2—C3 | 72.7 (7) | N2—N1—C10—C9 | -5.5 (4) |
| C7—C2—C3—C4 | 3.6 (7) | C8—C9—C10—O | 164.2 (4) |
| C1—C2—C3—C4 | 177.9 (5) | C8—C9—C10—N1 | -10.4 (4) |
| C2—C3—C4—C5 | -6.1 (7) | N1—N2—C11—C12 | -170.6 (3) |
| C3—C4—C5—C6 | 6.2 (6) | C8—N2—C11—C12 | 68.2 (4) |
| C3—C4—C5—C8 | -176.1 (4) | N1—N2—C11—C16 | 6.1 (5) |
| C4—C5—C6—C7 | -4.2 (6) | C8—N2—C11—C16 | -115.1 (3) |
| C8—C5—C6—C7 | 177.9 (3) | N2—C11—C12—C13 | 177.7 (4) |
| C3—C2—C7—C6 | -1.6 (7) | C16—C11—C12—C13 | 0.9 (6) |
| C1—C2—C7—C6 | -176.2 (4) | C11—C12—C13—C14 | -1.1 (6) |
| C5—C6—C7—C2 | 2.0 (6) | C12—C13—C14—C15 | -0.5 (6) |
| C4—C5—C8—C9 | 121.9 (4) | C13—C14—C15—C16 | 2.3 (7) |
| C6—C5—C8—C9 | -60.5 (4) | C14—C15—C16—C11 | -2.4 (6) |
| C4—C5—C8—N2 | 2.0 (5) | N2—C11—C16—C15 | -175.9 (3) |
| C6—C5—C8—N2 | 179.6 (3) | C12—C11—C16—C15 | 0.8 (5) |

Hydrogen-bond geometry (\AA , $^\circ$)

| <i>D</i> —H \cdots <i>A</i> | <i>D</i> —H | H \cdots <i>A</i> | <i>D</i> \cdots <i>A</i> | <i>D</i> —H \cdots <i>A</i> |
|-----------------------------------|-------------|---------------------|----------------------------|-------------------------------|
| OW2—HW2B \cdots OW1 | 0.85 | 1.99 | 2.50 (3) | 118 |
| C4—H4A \cdots N2 | 0.93 | 2.53 | 2.875 (6) | 102 |
| C7—H7A \cdots F3 | 0.93 | 2.41 | 2.731 (6) | 100 |
| C14—H14A \cdots F3 ⁱ | 0.93 | 2.54 | 3.262 (6) | 134 |
| N1—H1A \cdots O ⁱⁱ | 0.86 | 1.98 | 2.811 (6) | 161 |
| C9—H9B \cdots O ⁱⁱⁱ | 0.97 | 2.60 | 3.555 (7) | 168 |

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

Fig. 1

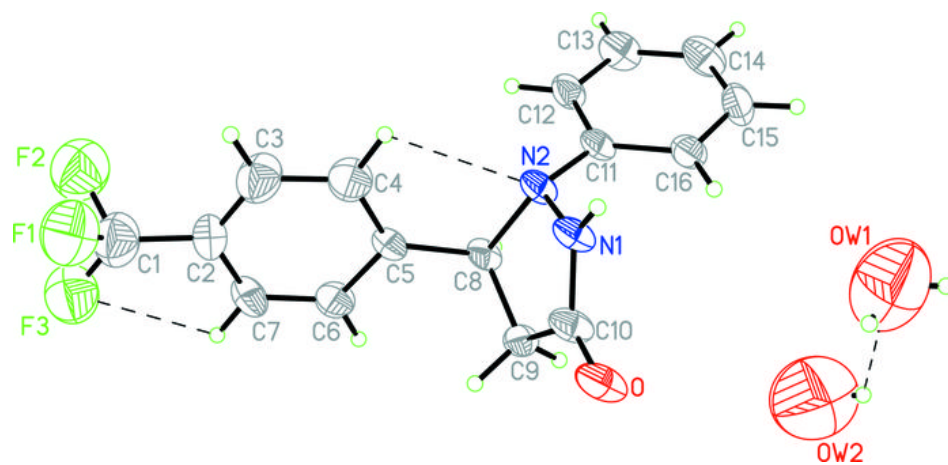


Fig. 2

