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# 1-(3-Fluorophenyl)-3-(4-nitrophenyl)urea

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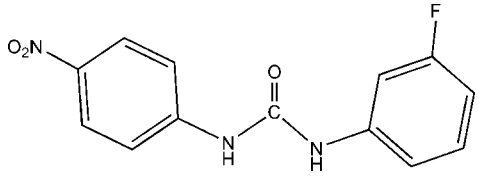
Key indicators: single-crystal X-ray study; $T$ = 113 K; mean $\sigma$(C–C) = 0.002 Å; $R$ factor = 0.052; $wR$ factor = 0.131; data-to-parameter ratio = 15.3.

In the title compound, $C_{13}H_{10}FN_3O_3$, the dihedral angle between the fluorophenyl and nitrophenyl ring planes is 6.51 (9)°. The crystal structure features N—H···O hydrogen bonds.

## Related literature

The title compound is an activated fragment of sorafenib derivatives. Sorafenib is a VEGFR-2 inhibitor (Ferrara *et al.*, 2003; Peruzzi *et al.*, 2006) that has good therapeutic effect for renal carcinoma and liver cancer (Wan *et al.*, 2004; Wilhelm *et al.*, 2004).




## Experimental

### Crystal data

$C_{13}H_{10}FN_3O_3$
$M_r$ = 275.24
Monoclinic, $P2_1/n$
$a$ = 8.351 (4) Å
$b$ = 12.461 (6) Å
$c$ = 11.912 (6) Å
$\beta$ = 100.315 (9)°
$V$ = 1219.5 (11) Å$^3$
$Z$ = 4
Mo $K\alpha$ radiation
$\mu$ = 0.12 mm$^{-1}$
$T$ = 113 K
0.24 × 0.22 × 0.20 mm

### Data collection

Rigaku Saturn CCD area-detector diffractometer
Absorption correction: multi-scan *CrystalClear*
$T_{min}$ = 0.972, $T_{max}$ = 0.977
12466 measured reflections
2900 independent reflections
2459 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045

### Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.131
$S$ = 1.12
2900 reflections
189 parameters
2 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2$A$···O1$^{i}$ | 0.91 (1) | 1.99 (1) | 2.890 (2) | 170 (2) |
| N3—H3$A$···O2$^{i}$ | 0.90 (1) | 2.28 (1) | 3.157 (2) | 168 (2) |

Symmetry code: (i) $x+\frac{1}{2}, -y+\frac{1}{2}, z-\frac{1}{2}$.

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZJ2072).

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# supplementary materials

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## 1-(3-Fluorophenyl)-3-(4-nitrophenyl)urea

**Mu-Sen Lin, Yu Shi, Shi-Yu Zhang and Yi-Liang Li**

### Comment

Sorafenib is a VEGFR-2 inhibitor (Ferrara *et al.*, 2003; Peruzzi *et al.*, 2006) that has good therapeutic effect for renal carcinoma and liver cancer (Wan *et al.*, 2004; Wilhelm *et al.*, 2004). 1-(3-fluorophenyl)-3-(4-nitrophenyl) urea is an important activated fragment of sorafenib derivatives. We present here the structure characterization of the title compound.

In the molecule of the title compound (Fig.1) bond lengths and angles have normal values. The interplanar angle between the fluorobenzyl and nitrobenzyl ring planes is 6.51 (9)°. The crystal structure is stabilized by the intermolecular N—H···O hydrogen bonds. The crystal structure (Fig.2) is stabilized by intermolecular N—H···O hydrogen bonds (table 1).

### Experimental

A solution of 4-nitroaniline (1.38 g, 10 mmol) in DCM (100 ml) was added dropwise to a stirred solution of bis(trichloromethyl) carbonate (5.92 g, 20 mmol) in DCM (20 ml) at the atmosphere of ice-bath.The reaction mixture was stirred for 2 hrs at 0–5°C. Then the reaction mixture was added drpwise to a refluxed and stirred solution of 3-fluoroaniline (1.11 g, 10 mmol) in DCM (40 ml).The reaction was completed within 2 hrs at the reflux temperature.The solvent was removed under reduced pressure.Acetone (100 ml) and $H_2O$ (300 ml) was added to the mixture. The solid was collected and washed with $H_2O$,then gave a yellow solid. The yield was 2.08 g (75.6%). Put about 0.3 g of the product in the ampoule bottle and add 10 ml absolute ethyl alcohol, yellow single crystals suitable for X-ray diffraction analysis were obtained by slow evaporation of the solvent at room temperature after 3 weeks.

### Refinement

All H atoms were detected in a difference map, nevertheless, the H-atoms attached to the nitrogen atoms were refined freely, and the H-atoms attached to the carbon atoms were placed in calculated positions and refined using a riding motion approximation, with C—H=0.95 Å, with $U_{iso}$(H)=1.2$U_{eq}$(C).

### Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL/PC* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL/PC* (Sheldrick, 2008).

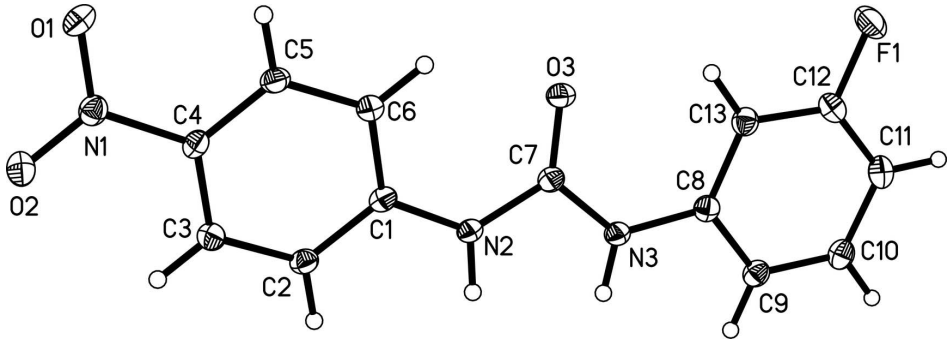


**Figure 1**

The molecular structure of the title compound, showing the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

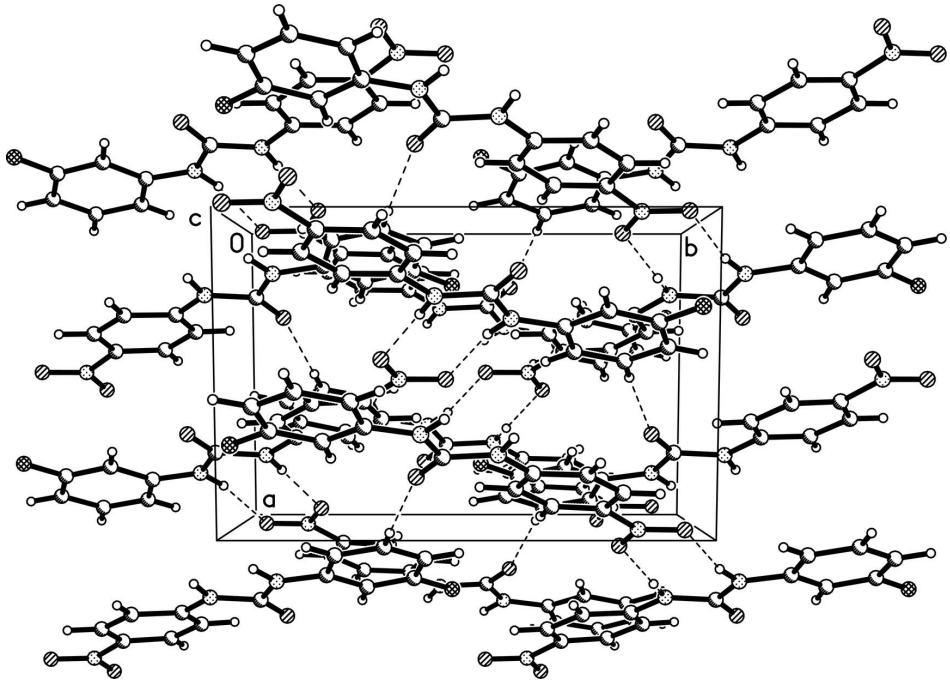


**Figure 2**

Packing diagram of the title compound viewed along the *c* axis. Hydrogen bonds are shown as dashed lines.

### 1-(3-Fluorophenyl)-3-(4-nitrophenyl)urea

*Crystal data*

$C_{13}H_{10}FN_3O_3$
$M_r = 275.24$
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
$a = 8.351$ (4) Å
$b = 12.461$ (6) Å
$c = 11.912$ (6) Å
$\beta = 100.315$ (9)°
$V = 1219.5$ (11) Å$^3$
$Z = 4$
$F(000) = 568$
$D_x = 1.499$ Mg m$^{-3}$
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4180 reflections
$\theta = 1.6$–27.8°
$\mu = 0.12$ mm$^{-1}$
$T = 113$ K
Prism, yellow
0.24 × 0.22 × 0.20 mm

*Data collection*

Rigaku Saturn CCD area-detector diffractometer
Radiation source: rotating anode
Multilayer monochromator
Detector resolution: 14.63 pixels mm$^{-1}$
$\omega$ and $\varphi$ scans
Absorption correction: multi-scan *CrystalClear*
$T_{min}$ = 0.972, $T_{max}$ = 0.977
12466 measured reflections
2900 independent reflections
2459 reflections with $I > 2\sigma(I)$
$R_{int}$ = 0.045
$\theta_{max}$ = 27.8°, $\theta_{min}$ = 2.4°
$h$ = −10→10
$k$ = −16→16
$l$ = −15→15

*Refinement*

Refinement on $F^2$
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.052
$wR(F^2)$ = 0.131
$S$ = 1.12
2900 reflections
189 parameters
2 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
$w = 1/[\sigma^2(F_o^2) + (0.063P)^2 + 0.1263P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.003
$\Delta\rho_{max}$ = 0.30 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.26 e Å$^{-3}$

*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$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| F1 | 0.29184 (14) | 0.96861 (8) | −0.02312 (9) | 0.0449 (3) |
| O1 | −0.04969 (14) | 0.17411 (10) | 0.34582 (10) | 0.0322 (3) |
| O2 | 0.01132 (14) | 0.04318 (10) | 0.24132 (10) | 0.0340 (3) |
| O3 | 0.19105 (13) | 0.60131 (9) | 0.05581 (9) | 0.0277 (3) |
| N1 | 0.01105 (16) | 0.13960 (12) | 0.26533 (12) | 0.0265 (3) |
| N2 | 0.26220 (16) | 0.43681 (11) | −0.00884 (11) | 0.0255 (3) |
| N3 | 0.32644 (17) | 0.58967 (11) | −0.09569 (11) | 0.0258 (3) |
| C1 | 0.20093 (18) | 0.36633 (13) | 0.06336 (13) | 0.0236 (3) |
| C2 | 0.19815 (19) | 0.25683 (13) | 0.03495 (13) | 0.0252 (4) |
| H2 | 0.2380 | 0.2346 | −0.0312 | 0.030* |
| C3 | 0.13910 (19) | 0.18127 (13) | 0.10071 (14) | 0.0264 (4) |
| H3 | 0.1371 | 0.1074 | 0.0808 | 0.032* |
| C4 | 0.08209 (18) | 0.21605 (13) | 0.19783 (13) | 0.0232 (3) |
| C5 | 0.08776 (19) | 0.32275 (13) | 0.22924 (14) | 0.0258 (4) |
| H5 | 0.0505 | 0.3439 | 0.2967 | 0.031* |
| C6 | 0.14717 (19) | 0.39910 (13) | 0.16335 (13) | 0.0258 (4) |

| | | | | |
|---|---|---|---|---|
| H6 | 0.1517 | 0.4725 | 0.1852 | 0.031* |
| C7 | 0.25437 (18) | 0.54780 (13) | −0.01016 (13) | 0.0234 (3) |
| C8 | 0.35025 (18) | 0.69898 (13) | −0.11978 (13) | 0.0246 (4) |
| C9 | 0.42473 (19) | 0.72135 (14) | −0.21284 (14) | 0.0268 (4) |
| H9 | 0.4554 | 0.6642 | −0.2573 | 0.032* |
| C10 | 0.4543 (2) | 0.82664 (14) | −0.24089 (14) | 0.0297 (4) |
| H10 | 0.5056 | 0.8406 | −0.3044 | 0.036* |
| C11 | 0.4106 (2) | 0.91169 (15) | −0.17805 (15) | 0.0327 (4) |
| H11 | 0.4305 | 0.9840 | −0.1969 | 0.039* |
| C12 | 0.3367 (2) | 0.88660 (13) | −0.08702 (15) | 0.0307 (4) |
| C13 | 0.30351 (19) | 0.78355 (14) | −0.05540 (14) | 0.0274 (4) |
| H13 | 0.2509 | 0.7705 | 0.0077 | 0.033* |
| H2A | 0.314 (2) | 0.4069 (14) | −0.0618 (13) | 0.037 (5)* |
| H3A | 0.372 (2) | 0.5429 (15) | −0.1377 (15) | 0.056 (7)* |

*Atomic displacement parameters (Å²)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| F1 | 0.0653 (8) | 0.0268 (6) | 0.0451 (7) | −0.0026 (5) | 0.0172 (6) | −0.0077 (5) |
| O1 | 0.0318 (6) | 0.0391 (7) | 0.0288 (7) | −0.0028 (5) | 0.0136 (5) | 0.0027 (5) |
| O2 | 0.0370 (7) | 0.0269 (6) | 0.0393 (7) | −0.0018 (5) | 0.0096 (5) | 0.0044 (5) |
| O3 | 0.0300 (6) | 0.0275 (6) | 0.0278 (6) | 0.0027 (5) | 0.0116 (5) | −0.0012 (5) |
| N1 | 0.0231 (7) | 0.0283 (7) | 0.0281 (7) | −0.0001 (6) | 0.0044 (5) | 0.0032 (6) |
| N2 | 0.0292 (7) | 0.0241 (7) | 0.0265 (7) | 0.0010 (6) | 0.0141 (6) | 0.0009 (6) |
| N3 | 0.0303 (7) | 0.0235 (7) | 0.0265 (7) | 0.0016 (6) | 0.0130 (6) | 0.0012 (6) |
| C1 | 0.0206 (7) | 0.0276 (8) | 0.0236 (8) | 0.0013 (6) | 0.0060 (6) | 0.0015 (6) |
| C2 | 0.0259 (8) | 0.0284 (8) | 0.0228 (8) | 0.0016 (6) | 0.0084 (6) | −0.0001 (6) |
| C3 | 0.0257 (8) | 0.0250 (8) | 0.0287 (9) | 0.0008 (6) | 0.0056 (7) | −0.0005 (6) |
| C4 | 0.0213 (7) | 0.0262 (8) | 0.0228 (8) | −0.0009 (6) | 0.0057 (6) | 0.0044 (6) |
| C5 | 0.0259 (8) | 0.0285 (9) | 0.0250 (8) | 0.0023 (6) | 0.0097 (6) | −0.0002 (6) |
| C6 | 0.0281 (8) | 0.0245 (8) | 0.0267 (8) | 0.0002 (6) | 0.0099 (7) | −0.0007 (6) |
| C7 | 0.0205 (7) | 0.0257 (8) | 0.0239 (8) | 0.0009 (6) | 0.0042 (6) | 0.0006 (6) |
| C8 | 0.0208 (7) | 0.0254 (8) | 0.0266 (8) | 0.0015 (6) | 0.0018 (6) | 0.0022 (6) |
| C9 | 0.0250 (8) | 0.0292 (9) | 0.0263 (8) | 0.0008 (6) | 0.0051 (6) | 0.0037 (7) |
| C10 | 0.0278 (8) | 0.0320 (9) | 0.0287 (9) | −0.0021 (7) | 0.0032 (7) | 0.0051 (7) |
| C11 | 0.0352 (9) | 0.0275 (9) | 0.0342 (9) | −0.0054 (7) | 0.0027 (7) | 0.0030 (7) |
| C12 | 0.0344 (9) | 0.0250 (8) | 0.0315 (9) | −0.0005 (7) | 0.0029 (7) | −0.0035 (7) |
| C13 | 0.0286 (8) | 0.0279 (8) | 0.0257 (8) | −0.0018 (7) | 0.0048 (7) | 0.0011 (7) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C12 | 1.366 (2) | C3—H3 | 0.9500 |
| O1—N1 | 1.2392 (18) | C4—C5 | 1.380 (2) |
| O2—N1 | 1.235 (2) | C5—C6 | 1.381 (2) |
| O3—C7 | 1.2209 (19) | C5—H5 | 0.9500 |
| N1—C4 | 1.441 (2) | C6—H6 | 0.9500 |
| N2—C7 | 1.384 (2) | C8—C9 | 1.393 (2) |
| N2—C1 | 1.389 (2) | C8—C13 | 1.399 (2) |
| N2—H2A | 0.907 (9) | C9—C10 | 1.387 (2) |
| N3—C7 | 1.376 (2) | C9—H9 | 0.9500 |

| | | | |
|---|---|---|---|
| N3—C8 | 1.413 (2) | C10—C11 | 1.384 (2) |
| N3—H3A | 0.897 (9) | C10—H10 | 0.9500 |
| C1—C2 | 1.405 (2) | C11—C12 | 1.376 (3) |
| C1—C6 | 1.407 (2) | C11—H11 | 0.9500 |
| C2—C3 | 1.372 (2) | C12—C13 | 1.380 (2) |
| C2—H2 | 0.9500 | C13—H13 | 0.9500 |
| C3—C4 | 1.397 (2) | | |

| | | | |
|---|---|---|---|
| O2—N1—O1 | 122.33 (14) | C5—C6—C1 | 118.87 (16) |
| O2—N1—C4 | 119.72 (14) | C5—C6—H6 | 120.6 |
| O1—N1—C4 | 117.95 (14) | C1—C6—H6 | 120.6 |
| C7—N2—C1 | 128.07 (14) | O3—C7—N3 | 124.54 (16) |
| C7—N2—H2A | 115.5 (12) | O3—C7—N2 | 124.28 (15) |
| C1—N2—H2A | 116.4 (12) | N3—C7—N2 | 111.18 (14) |
| C7—N3—C8 | 127.73 (14) | C9—C8—C13 | 119.58 (15) |
| C7—N3—H3A | 116.9 (14) | C9—C8—N3 | 116.97 (15) |
| C8—N3—H3A | 115.1 (14) | C13—C8—N3 | 123.46 (15) |
| N2—C1—C2 | 117.18 (14) | C10—C9—C8 | 120.35 (16) |
| N2—C1—C6 | 123.33 (15) | C10—C9—H9 | 119.8 |
| C2—C1—C6 | 119.47 (15) | C8—C9—H9 | 119.8 |
| C3—C2—C1 | 121.41 (15) | C11—C10—C9 | 121.22 (16) |
| C3—C2—H2 | 119.3 | C11—C10—H10 | 119.4 |
| C1—C2—H2 | 119.3 | C9—C10—H10 | 119.4 |
| C2—C3—C4 | 118.09 (15) | C12—C11—C10 | 116.83 (16) |
| C2—C3—H3 | 121.0 | C12—C11—H11 | 121.6 |
| C4—C3—H3 | 121.0 | C10—C11—H11 | 121.6 |
| C5—C4—C3 | 121.57 (15) | F1—C12—C11 | 118.40 (15) |
| C5—C4—N1 | 118.89 (14) | F1—C12—C13 | 117.07 (15) |
| C3—C4—N1 | 119.51 (15) | C11—C12—C13 | 124.52 (16) |
| C4—C5—C6 | 120.54 (15) | C12—C13—C8 | 117.49 (16) |
| C4—C5—H5 | 119.7 | C12—C13—H13 | 121.3 |
| C6—C5—H5 | 119.7 | C8—C13—H13 | 121.3 |

| | | | |
|---|---|---|---|
| C7—N2—C1—C2 | 168.79 (15) | C8—N3—C7—O3 | 4.1 (3) |
| C7—N2—C1—C6 | −13.1 (2) | C8—N3—C7—N2 | −176.20 (14) |
| N2—C1—C2—C3 | −179.65 (14) | C1—N2—C7—O3 | 0.8 (3) |
| C6—C1—C2—C3 | 2.2 (2) | C1—N2—C7—N3 | −178.89 (14) |
| C1—C2—C3—C4 | −0.3 (2) | C7—N3—C8—C9 | −179.05 (15) |
| C2—C3—C4—C5 | −1.5 (2) | C7—N3—C8—C13 | 1.1 (3) |
| C2—C3—C4—N1 | 176.62 (13) | C13—C8—C9—C10 | 0.8 (2) |
| O2—N1—C4—C5 | −176.89 (14) | N3—C8—C9—C10 | −179.09 (14) |
| O1—N1—C4—C5 | 3.6 (2) | C8—C9—C10—C11 | −0.2 (2) |
| O2—N1—C4—C3 | 4.9 (2) | C9—C10—C11—C12 | 0.0 (2) |
| O1—N1—C4—C3 | −174.56 (13) | C10—C11—C12—F1 | −179.98 (14) |
| C3—C4—C5—C6 | 1.5 (2) | C10—C11—C12—C13 | −0.3 (3) |
| N1—C4—C5—C6 | −176.63 (14) | F1—C12—C13—C8 | −179.49 (14) |
| C4—C5—C6—C1 | 0.4 (2) | C11—C12—C13—C8 | 0.8 (3) |
| N2—C1—C6—C5 | 179.79 (14) | C9—C8—C13—C12 | −1.0 (2) |
| C2—C1—C6—C5 | −2.1 (2) | N3—C8—C13—C12 | 178.82 (15) |

*Hydrogen-bond geometry (Å, º)*

| *D*—H···*A* | *D*—H | H···*A* | *D*···*A* | *D*—H···*A* |
|---|---|---|---|---|
| N2—H2*A*···O1$^{i}$ | 0.91 (1) | 1.99 (1) | 2.890 (2) | 170 (2) |
| N3—H3*A*···O2$^{i}$ | 0.90 (1) | 2.28 (1) | 3.157 (2) | 168 (2) |

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