# 5-(2-Ethoxy-4-fluorophenyl)-1,2,4-triazolo[1,5-a]-pyrimidine

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In the title compound, $C_{13}H_{11}FN_4O$, the dihedral angle between the triazolopyrimidine ring system and fluorophenyl ring is 39.16 (12)°. In the crystal, C—H···N hydrogen bonds link the molecules resulting in $R_2^2(8)$ ring motifs and $C(8)$ chain motifs.

**3D view**



**Chemical scheme**
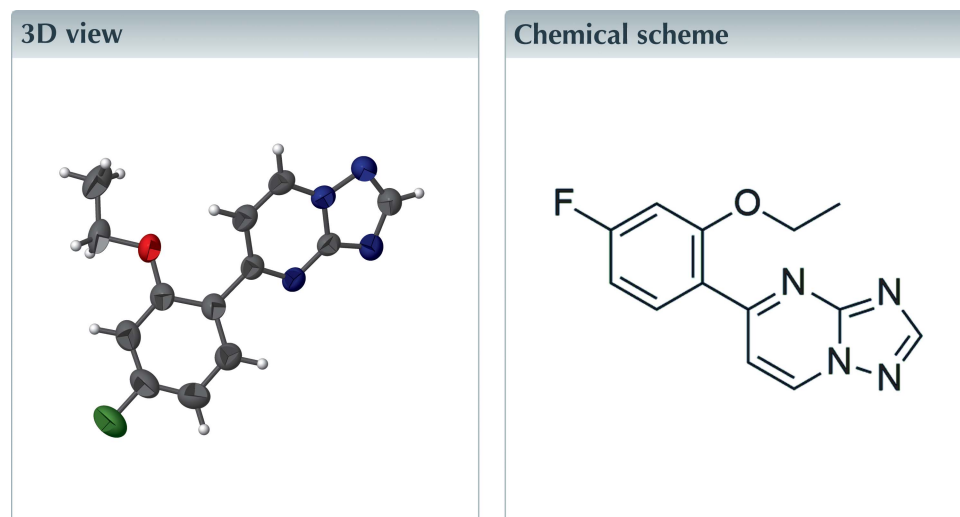


## Structure description

1,2,4-Triazolo[1,5-a]pyrimidine derivatives are used in the field of pharmaceutics, agriculture and other areas (Mopper & Zhou, 1990). As part of our work on the synthesis and crystal structure determination of 1,2,4-triazolo[1,5-a]pyrimidine derivatives (Gilandoust et al., 2016), the title compound is reported here.

Fig. 1 represents the *ORTEP* drawing of the title compound of which the geometric parameters (bond lengths and bond angles) are in the normal range. The dihedral angle between the triazolopyrimidine and fluorophenyl rings is 39.16 (12)°. An intramolecular C17—H17···O1 contact (Table 1) is observed.

The crystal structure features C—H···N hydrogen bonds (Fig. 2, Table 1). The C5—H5···N4 and C16—H16···N2 hydrogen bonds lead to the formation of infinite chains along the b axis [$C(8)$ chain motifs] and inversion dimers [$R_2^2(8)$ ring motifs], respectively.

## Synthesis and crystallization

5-Bromo-[1,2,4]-triazolo[1,5-a]pyrimidines (1 mmol), 2-ethoxy-4-fluorobenzenboronic acid (1.2 mmol) and $K_2CO_3$ (3 mmol) were added to a mixture of ethanol, water and 1,4-dioxan in the ratio (1:1:5). The reaction mixture was stirred in a sealed tube for 15 min in
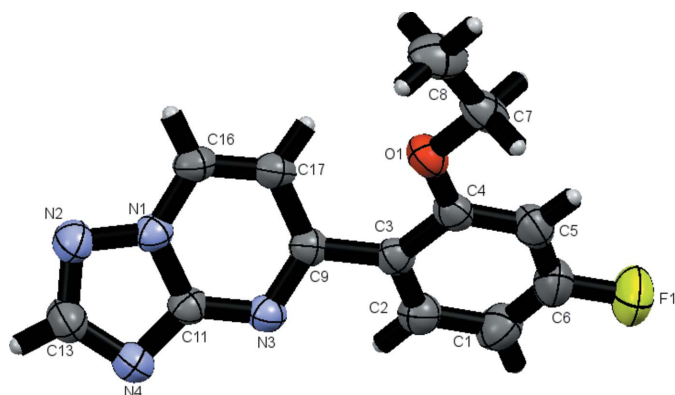
**Figure 1**
A view of the title molecule, showing the atom labelling. Displacement ellipsoids are drawn at the 50% probability level and the intramolecular hydrogen bond is shown as a dashed line.

the presence of nitrogen gas to create an inert atmosphere after which the catalyst [PdCl$_2$(PPh$_3$)$_2$] was added (0.1 mmol). The reaction mass was heated to 120–130°C for 35 min in a sealed tube and the progress of the reaction was monitored by TLC. The resultant mixture was filtered through a Celite bed and the filtrate was concentrated under reduced pressure to remove the ethanol by using a rotary evaporator. The reaction mass was extracted with ethyl acetate followed by a brine wash and dried over anhydrous sodium sulfate. The organic layer was evaporated under reduced pressure to get the crude product, which was purified by column chromatography using 60:120 mesh silica gel and EtOAc:hexane as eluent (40:60 ml) to get the desired triazolopyrimidine as a white solid. Good quality single crystals suitable for X-ray diffraction studies were obtained by the slow evaporation method using ethanol as a solvent.
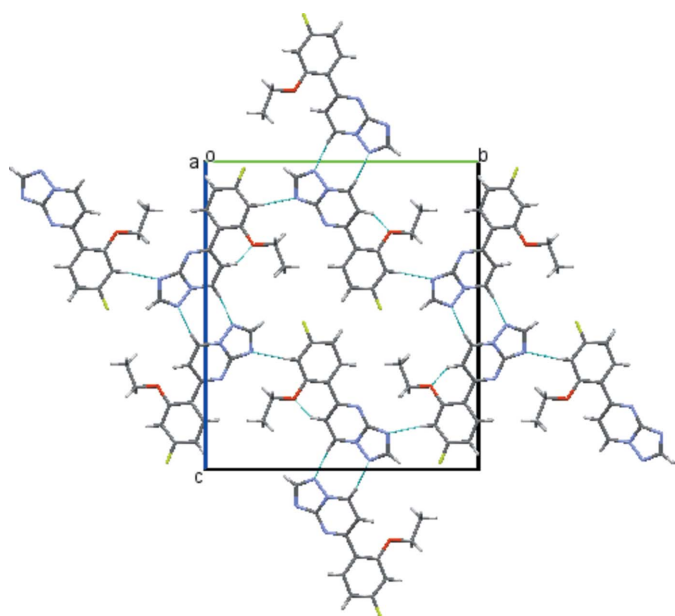


**Figure 2**
A view along the *a* axis of the crystal packing of the title compound. Hydrogen bonds are drawn as dashed lines.

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

| $D$—H$\cdots A$ | $D$—H | H$\cdots A$ | $D\cdots A$ | $D$—H$\cdots A$ |
|---|---|---|---|---|
| C5—H5$\cdots$N4$^i$ | 0.93 | 2.60 | 3.492 (4) | 162 |
| C16—H16$\cdots$N2$^{ii}$ | 0.93 | 2.49 | 3.407 (4) | 168 |
| C17—H17$\cdots$O1 | 0.93 | 2.39 | 2.811 (3) | 107 |

Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{3}{2}$; (ii) $-x + 3, -y + 1, -z + 2$.

**Table 2**
Experimental details.

| | |
|---|---|
| Crystal data | |
| Chemical formula | C$_{13}$H$_{11}$FN$_4$O |
| $M_r$ | 258.26 |
| Crystal system, space group | Monoclinic, $P2_1/c$ |
| Temperature (K) | 293 |
| $a, b, c$ (Å) | 3.9166 (3), 16.6608 (11), 18.8096 (14) |
| $\beta$ (°) | 93.232 (7) |
| $V$ (Å$^3$) | 1225.44 (16) |
| $Z$ | 4 |
| Radiation type | Mo $K\alpha$ |
| $\mu$ (mm$^{-1}$) | 0.10 |
| Crystal size (mm) | 0.32 × 0.23 × 0.21 |
| | |
| Data collection | |
| Diffractometer | Rigaku Saturn724+ |
| Absorption correction | Multi-scan (*NUMABS*; Rigaku, 1999) |
| $T_{min}, T_{max}$ | 0.966, 0.979 |
| No. of measured, independent and observed [$I > 2\sigma(I)$] reflections | 10165, 2171, 1458 |
| $R_{int}$ | 0.059 |
| $(\sin \theta/\lambda)_{max}$ (Å$^{-1}$) | 0.595 |
| | |
| Refinement | |
| $R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, $S$ | 0.061, 0.182, 1.08 |
| No. of reflections | 2171 |
| No. of parameters | 174 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta\rho_{max}, \Delta\rho_{min}$ (e Å$^{-3}$) | 0.24, −0.26 |

Computer programs: *CrystalClear SM Expert* (Rigaku, 2011), *SHELXS97* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *Mercury* (Macrae *et al.*, 2008) and *OLEX2* (Dolomanov *et al.*, 2009).

## Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2.

## References

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Sheldrick, G. M. (2015). *Acta Cryst.* C**71**, 3–8.

# full crystallographic data

## 5-(2-Ethoxy-4-fluorophenyl)-1,2,4-triazolo[1,5-*a*]pyrimidine

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### 5-(2-Ethoxy-4-fluorophenyl)-1,2,4-triazolo[1,5-*a*]pyrimidine

*Crystal data*

| | |
|---|---|
| $C_{13}H_{11}FN_4O$ | $F(000) = 536$ |
| $M_r = 258.26$ | $D_x = 1.400$ Mg m$^{-3}$ |
| Monoclinic, $P2_1/c$ | Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å |
| $a = 3.9166$ (3) Å | Cell parameters from 2171 reflections |
| $b = 16.6608$ (11) Å | $\theta = 2.2$–25.0° |
| $c = 18.8096$ (14) Å | $\mu = 0.10$ mm$^{-1}$ |
| $\beta = 93.232$ (7)° | $T = 293$ K |
| $V = 1225.44$ (16) Å$^3$ | Block, yellow |
| $Z = 4$ | $0.32 \times 0.23 \times 0.21$ mm |

*Data collection*

| | |
|---|---|
| Rigaku Saturn724+ diffractometer | 1458 reflections with $I > 2\sigma(I)$ |
| profile data from $\omega$–scans | $R_{int} = 0.059$ |
| Absorption correction: multi-scan (NUMABS; Rigaku, 1999) | $\theta_{max} = 25.0°$, $\theta_{min} = 2.2°$ |
| $T_{min} = 0.966$, $T_{max} = 0.979$ | $h = -4 \rightarrow 3$ |
| 10165 measured reflections | $k = -19 \rightarrow 19$ |
| 2171 independent reflections | $l = -22 \rightarrow 22$ |
| | 2171 standard reflections |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | H-atom parameters constrained |
| Least-squares matrix: full | $w = 1/[\sigma^2(F_o^2) + (0.0817P)^2 + 0.3483P]$ where $P = (F_o^2 + 2F_c^2)/3$ |
| $R[F^2 > 2\sigma(F^2)] = 0.061$ | |
| $wR(F^2) = 0.182$ | $(\Delta/\sigma)_{max} < 0.001$ |
| $S = 1.08$ | $\Delta\rho_{max} = 0.24$ e Å$^{-3}$ |
| 2171 reflections | $\Delta\rho_{min} = -0.26$ e Å$^{-3}$ |
| 174 parameters | Extinction correction: SHELXL2014 (Sheldrick, 2015), |
| 0 restraints | $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$ |
| Primary atom site location: iterative | Extinction coefficient: 0.023 (4) |
| Hydrogen site location: inferred from neighbouring sites | |

*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.

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

|      | x           | y            | z            | $U_{iso}$*/$U_{eq}$ |
|------|-------------|--------------|--------------|---------------------|
| F1   | 0.5117 (7)  | 0.35926 (13) | 0.52220 (10) | 0.1067 (9)          |
| O1   | 0.7212 (5)  | 0.32471 (10) | 0.77097 (10) | 0.0567 (6)          |
| N1   | 1.1380 (5)  | 0.55271 (12) | 0.91136 (11) | 0.0459 (6)          |
| N2   | 1.1990 (6)  | 0.60040 (14) | 0.97014 (12) | 0.0574 (7)          |
| N3   | 0.8590 (5)  | 0.56069 (12) | 0.79604 (11) | 0.0464 (6)          |
| N4   | 0.8923 (6)  | 0.66793 (13) | 0.88210 (12) | 0.0575 (7)          |
| C1   | 0.7414 (9)  | 0.4662 (2)   | 0.59031 (16) | 0.0701 (9)          |
| H1   | 0.7509      | 0.4977       | 0.5497       | 0.084*              |
| C2   | 0.8464 (7)  | 0.49545 (17) | 0.65655 (15) | 0.0573 (8)          |
| H2   | 0.9233      | 0.5482       | 0.6605       | 0.069*              |
| C3   | 0.8405 (7)  | 0.44865 (15) | 0.71738 (14) | 0.0468 (7)          |
| C4   | 0.7224 (7)  | 0.36906 (16) | 0.71033 (14) | 0.0503 (7)          |
| C5   | 0.6084 (8)  | 0.33987 (18) | 0.64441 (16) | 0.0607 (8)          |
| H5   | 0.5237      | 0.2879       | 0.6395       | 0.073*              |
| C6   | 0.6231 (9)  | 0.3892 (2)   | 0.58674 (17) | 0.0707 (9)          |
| C7   | 0.5874 (8)  | 0.24438 (16) | 0.76772 (18) | 0.0627 (9)          |
| H7A  | 0.7172      | 0.2116       | 0.7363       | 0.075*              |
| H7B  | 0.3500      | 0.2448       | 0.7500       | 0.075*              |
| C8   | 0.6178 (9)  | 0.2119 (2)   | 0.8419 (2)   | 0.0806 (11)         |
| H8A  | 0.4932      | 0.2458       | 0.8727       | 0.121*              |
| H8B  | 0.8542      | 0.2106       | 0.8584       | 0.121*              |
| H8C  | 0.5253      | 0.1586       | 0.8424       | 0.121*              |
| C9   | 0.9482 (6)  | 0.48491 (14) | 0.78707 (13) | 0.0436 (6)          |
| C11  | 0.9540 (6)  | 0.59429 (15) | 0.85917 (13) | 0.0447 (7)          |
| C13  | 1.0413 (8)  | 0.66697 (18) | 0.94821 (16) | 0.0619 (8)          |
| H13  | 1.0338      | 0.7117       | 0.9777       | 0.074*              |
| C16  | 1.2387 (7)  | 0.47580 (16) | 0.90249 (15) | 0.0499 (7)          |
| H16  | 1.3677      | 0.4485       | 0.9378       | 0.060*              |
| C17  | 1.1421 (7)  | 0.44085 (16) | 0.83985 (14) | 0.0494 (7)          |
| H17  | 1.2026      | 0.3879       | 0.8312       | 0.059*              |

*Atomic displacement parameters (Å²)*

|     | $U^{11}$    | $U^{22}$    | $U^{33}$    | $U^{12}$     | $U^{13}$     | $U^{23}$     |
|-----|-------------|-------------|-------------|--------------|--------------|--------------|
| F1  | 0.152 (2)   | 0.0993 (17) | 0.0656 (13) | 0.0004 (14)  | −0.0248 (13) | −0.0214 (11) |
| O1  | 0.0669 (13) | 0.0402 (10) | 0.0630 (13) | −0.0063 (9)  | 0.0050 (10)  | −0.0024 (9)  |
| N1  | 0.0472 (13) | 0.0443 (13) | 0.0459 (13) | −0.0022 (10) | −0.0002 (10) | 0.0059 (10)  |
| N2  | 0.0691 (17) | 0.0534 (15) | 0.0491 (14) | −0.0032 (12) | −0.0021 (12) | 0.0003 (11)  |
| N3  | 0.0482 (13) | 0.0396 (12) | 0.0511 (13) | 0.0002 (9)   | −0.0006 (11) | 0.0040 (9)   |
| N4  | 0.0680 (17) | 0.0449 (14) | 0.0591 (15) | 0.0030 (11)  | −0.0016 (13) | −0.0019 (11) |
| C1  | 0.078 (2)   | 0.078 (2)   | 0.0537 (19) | 0.0043 (18)  | −0.0027 (17) | 0.0062 (16)  |
| C2  | 0.0580 (18) | 0.0527 (17) | 0.0609 (18) | 0.0015 (13)  | −0.0006 (14) | 0.0026 (14)  |
| C3  | 0.0402 (14) | 0.0441 (15) | 0.0557 (16) | 0.0026 (11)  | 0.0000 (12)  | −0.0014 (12) |
| C4  | 0.0502 (16) | 0.0438 (15) | 0.0572 (17) | 0.0068 (12)  | 0.0053 (13)  | −0.0050 (13) |
| C5  | 0.0619 (19) | 0.0533 (18) | 0.067 (2)   | 0.0035 (14)  | 0.0006 (16)  | −0.0133 (15) |

| C6 | 0.079 (2) | 0.075 (2) | 0.0562 (19) | 0.0072 (18) | −0.0095 (17) | −0.0152 (17) |
| C7 | 0.0519 (17) | 0.0401 (15) | 0.097 (2) | −0.0062 (13) | 0.0091 (16) | −0.0051 (15) |
| C8 | 0.070 (2) | 0.0553 (19) | 0.116 (3) | −0.0083 (16) | 0.007 (2) | 0.0229 (19) |
| C9 | 0.0376 (14) | 0.0410 (14) | 0.0521 (15) | −0.0030 (11) | 0.0025 (11) | 0.0028 (11) |
| C11 | 0.0452 (15) | 0.0416 (14) | 0.0471 (15) | −0.0040 (11) | 0.0015 (12) | 0.0068 (12) |
| C13 | 0.078 (2) | 0.0495 (18) | 0.0576 (18) | −0.0016 (15) | 0.0008 (16) | −0.0050 (14) |
| C16 | 0.0480 (16) | 0.0453 (15) | 0.0559 (17) | 0.0032 (12) | −0.0011 (13) | 0.0113 (13) |
| C17 | 0.0456 (15) | 0.0415 (15) | 0.0610 (18) | 0.0033 (11) | 0.0020 (13) | 0.0055 (12) |

*Geometric parameters (Å, º)*

| | | | |
|---|---|---|---|
| F1—C6 | 1.361 (3) | C3—C4 | 1.408 (4) |
| O1—C4 | 1.359 (3) | C3—C9 | 1.483 (3) |
| O1—C7 | 1.437 (3) | C4—C5 | 1.382 (4) |
| N1—N2 | 1.371 (3) | C5—H5 | 0.9300 |
| N1—C11 | 1.372 (3) | C5—C6 | 1.365 (4) |
| N1—C16 | 1.354 (3) | C7—H7A | 0.9700 |
| N2—C13 | 1.324 (4) | C7—H7B | 0.9700 |
| N3—C9 | 1.323 (3) | C7—C8 | 1.495 (4) |
| N3—C11 | 1.346 (3) | C8—H8A | 0.9600 |
| N4—C11 | 1.327 (3) | C8—H8B | 0.9600 |
| N4—C13 | 1.344 (3) | C8—H8C | 0.9600 |
| C1—H1 | 0.9300 | C9—C17 | 1.420 (3) |
| C1—C2 | 1.379 (4) | C13—H13 | 0.9300 |
| C1—C6 | 1.364 (5) | C16—H16 | 0.9300 |
| C2—H2 | 0.9300 | C16—C17 | 1.349 (4) |
| C2—C3 | 1.386 (4) | C17—H17 | 0.9300 |
| | | | |
| C4—O1—C7 | 119.3 (2) | O1—C7—H7B | 110.4 |
| N2—N1—C11 | 110.2 (2) | O1—C7—C8 | 106.7 (2) |
| C16—N1—N2 | 127.5 (2) | H7A—C7—H7B | 108.6 |
| C16—N1—C11 | 122.3 (2) | C8—C7—H7A | 110.4 |
| C13—N2—N1 | 100.2 (2) | C8—C7—H7B | 110.4 |
| C9—N3—C11 | 116.6 (2) | C7—C8—H8A | 109.5 |
| C11—N4—C13 | 102.3 (2) | C7—C8—H8B | 109.5 |
| C2—C1—H1 | 121.3 | C7—C8—H8C | 109.5 |
| C6—C1—H1 | 121.3 | H8A—C8—H8B | 109.5 |
| C6—C1—C2 | 117.4 (3) | H8A—C8—H8C | 109.5 |
| C1—C2—H2 | 119.0 | H8B—C8—H8C | 109.5 |
| C1—C2—C3 | 121.9 (3) | N3—C9—C3 | 115.9 (2) |
| C3—C2—H2 | 119.0 | N3—C9—C17 | 122.6 (2) |
| C2—C3—C4 | 118.2 (2) | C17—C9—C3 | 121.4 (2) |
| C2—C3—C9 | 118.9 (2) | N3—C11—N1 | 121.9 (2) |
| C4—C3—C9 | 122.9 (2) | N4—C11—N1 | 109.4 (2) |
| O1—C4—C3 | 116.7 (2) | N4—C11—N3 | 128.7 (2) |
| O1—C4—C5 | 123.0 (3) | N2—C13—N4 | 118.0 (3) |
| C5—C4—C3 | 120.2 (3) | N2—C13—H13 | 121.0 |
| C4—C5—H5 | 120.8 | N4—C13—H13 | 121.0 |

| C6—C5—C4 | 118.4 (3) | N1—C16—H16 | 121.7 |
| C6—C5—H5 | 120.8 | C17—C16—N1 | 116.5 (2) |
| F1—C6—C1 | 118.6 (3) | C17—C16—H16 | 121.7 |
| F1—C6—C5 | 117.5 (3) | C9—C17—H17 | 120.0 |
| C1—C6—C5 | 123.8 (3) | C16—C17—C9 | 120.0 (2) |
| O1—C7—H7A | 110.4 | C16—C17—H17 | 120.0 |
| | | | |
| O1—C4—C5—C6 | −179.5 (3) | C4—C5—C6—F1 | 179.6 (3) |
| N1—N2—C13—N4 | −1.2 (4) | C4—C5—C6—C1 | −0.6 (5) |
| N1—C16—C17—C9 | 0.7 (4) | C6—C1—C2—C3 | 1.4 (5) |
| N2—N1—C11—N3 | −179.6 (2) | C7—O1—C4—C3 | 176.9 (2) |
| N2—N1—C11—N4 | 0.0 (3) | C7—O1—C4—C5 | −1.8 (4) |
| N2—N1—C16—C17 | 179.0 (2) | C9—N3—C11—N1 | 0.9 (4) |
| N3—C9—C17—C16 | 0.9 (4) | C9—N3—C11—N4 | −178.6 (3) |
| C1—C2—C3—C4 | −0.1 (4) | C9—C3—C4—O1 | −2.1 (4) |
| C1—C2—C3—C9 | −178.3 (3) | C9—C3—C4—C5 | 176.6 (3) |
| C2—C1—C6—F1 | 178.7 (3) | C11—N1—N2—C13 | 0.7 (3) |
| C2—C1—C6—C5 | −1.0 (5) | C11—N1—C16—C17 | −1.5 (4) |
| C2—C3—C4—O1 | 179.8 (2) | C11—N3—C9—C3 | 179.7 (2) |
| C2—C3—C4—C5 | −1.5 (4) | C11—N3—C9—C17 | −1.7 (4) |
| C2—C3—C9—N3 | 37.3 (4) | C11—N4—C13—N2 | 1.2 (4) |
| C2—C3—C9—C17 | −141.3 (3) | C13—N4—C11—N1 | −0.6 (3) |
| C3—C4—C5—C6 | 1.9 (4) | C13—N4—C11—N3 | 179.0 (3) |
| C3—C9—C17—C16 | 179.4 (2) | C16—N1—N2—C13 | −179.7 (3) |
| C4—O1—C7—C8 | −179.5 (2) | C16—N1—C11—N3 | 0.7 (4) |
| C4—C3—C9—N3 | −140.8 (3) | C16—N1—C11—N4 | −179.7 (2) |
| C4—C3—C9—C17 | 40.6 (4) | | |

*Hydrogen-bond geometry (Å, °)*

| D—H···A | D—H | H···A | D···A | D—H···A |
| --- | --- | --- | --- | --- |
| C5—H5···N4[i] | 0.93 | 2.60 | 3.492 (4) | 162 |
| C16—H16···N2[ii] | 0.93 | 2.49 | 3.407 (4) | 168 |
| C17—H17···O1 | 0.93 | 2.39 | 2.811 (3) | 107 |

Symmetry codes: (i) −x+1, y−1/2, −z+3/2; (ii) −x+3, −y+1, −z+2.