

1-[4-(Difluoromethoxy)phenyl]-N-(2,3-dimethylphenyl)-1H-1,2,4-triazole-3-carboxamide

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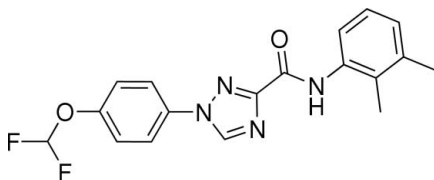
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Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; R factor = 0.042; wR factor = 0.123; data-to-parameter ratio = 13.1.

In the molecule of the title compound, $\text{C}_{18}\text{H}_{16}\text{F}_2\text{N}_4\text{O}_2$, the 1,2,4-triazole ring forms dihedral angles of 3.6 (2) and 14.9 (6)° with the 4-difluoromethoxy-substituted benzene ring and the 2,3-dimethyl-substituted benzene ring, respectively. The OCHF_2 group is twisted away from the plane of the benzene ring, as shown by the $\text{C}-\text{O}-\text{C}-\text{C}$ torsion angle of 145.8 (2)°. The conformation is stabilized by an intermolecular $\text{N}-\text{H}\cdots\text{N}$ hydrogen bond. In the crystal, short $\text{C}-\text{H}\cdots\text{O}$ interactions lead to chains of molecules.

Related literature

For general background regarding the biological and pharmacological activities of 1,2,4-triazoles and their derivatives, see: Wahbi *et al.* (1995); Chai *et al.* (2003); Hashimoto *et al.* (1990); Kalluraya *et al.* (1996); Almasirad *et al.* (2004); Amir & Shikha (2004); Kanazawa *et al.* (1988); Vlasova *et al.* (1971); Labanauskas *et al.* (2004); Tozkoparan *et al.* (2007). For a related synthesis, see: Drutkowski *et al.* (2002); Frohberg *et al.* (2002).



Experimental

Crystal data

$\text{C}_{18}\text{H}_{16}\text{F}_2\text{N}_4\text{O}_2$
 $M_r = 358.35$
 Triclinic, $P\bar{1}$
 $a = 7.5543$ (10) Å
 $b = 7.8132$ (10) Å
 $c = 14.8190$ (19) Å
 $\alpha = 95.974$ (2)°
 $\beta = 98.593$ (1)°
 $\gamma = 101.523$ (1)°
 $V = 839.28$ (19) Å³
 $Z = 2$
 Mo $K\alpha$ radiation
 $\mu = 0.11$ mm⁻¹
 $T = 296$ K
 $0.49 \times 0.31 \times 0.10$ mm

Data collection

Bruker APEXII CCD diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2004)
 $T_{\min} = 0.948$, $T_{\max} = 0.990$
 6446 measured reflections
 3115 independent reflections
 2366 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.017$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.042$
 $wR(F^2) = 0.123$
 $S = 1.06$
 3115 reflections
 238 parameters
 H-atom parameters constrained
 $\Delta\rho_{\text{max}} = 0.23$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.21$ e Å⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N4}-\text{H4D}\cdots\text{N3}$	0.86	2.27	2.717 (2)	113
$\text{C6}-\text{H6}\cdots\text{O2}^i$	0.93	2.43	3.344 (2)	169
$\text{C8}-\text{H8}\cdots\text{O2}^i$	0.93	2.26	3.159 (2)	162

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

Data collection: APEX2 (Bruker, 2004); cell refinement: SAINT (Bruker, 2004); data reduction: SAINT; 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 and PLATON (Spek, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2162).

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supporting information

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1-[4-(Difluoromethoxy)phenyl]-*N*-(2,3-dimethylphenyl)-1*H*-1,2,4-triazole-3-carboxamide

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S1. Comment

1,2,4-Triazoles and their derivatives have long been known to exhibit diverse biological and pharmacological activities, such as antitubercular, anticancer (Vlasova *et al.*, 1971; Kalluraya *et al.*, 1996), anticonvulsant (Almasirad *et al.*, 2004; Kanazawa *et al.*, 1988; Chai *et al.*, 2003; Hashimoto *et al.*, 1990), anti-inflammatory (Labanauskas *et al.*, 2004), herbicidal, and analgesic properties (Tozkoparan *et al.*, 2007; Amir & Shikha, 2004). Also, antifungal activity of aromatic ethers possessing a 1*H*-1,2,4-triazole ring has been reported (Wahbi *et al.*, 1995). Herein, we report the synthesis and crystal structure of the title compound, (I).

In the molecule of the title compound (Fig. 1) the bond lengths and angles are generally within normal ranges. The planar 1,2,4-triazole ring is oriented at dihedral angles of 3.6 (2)° and 14.9 (6)° with respect to the 4-difluoromethoxy-substituted benzene ring and 2,3-dimethyl-substituted benzene ring, respectively. The CHF₂ group is twisted away from the plane of the benzene ring, as shown by the C1—O1—C2—C3 torsion angle [145.8 (2)°].

S2. Experimental

The general procedure to synthesize the title compound: 2-amine-*N*-(2,3-dimethyl-phenyl)-2-[(4-difluoromethoxy-phenyl)hydrazono]acetamide (10 mmol), 1.5 mL of a 37%-solution of formaldehyde (20 mmol) and 0.1 g *p*-toluene sulfonic acid were refluxed in approximately 50 mL ethanol. The reaction was complete after 10 h. The mixture was cooled to room temperature and the solvent was evaporated. The solid product was collected and recrystallized from 2-propanol (Drutkowski *et al.*, 2002; Frohberg *et al.*, 2002).

S3. Refinement

H atoms were placed in calculated positions with C—H = 0.95–0.99 Å, and refined in riding mode with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

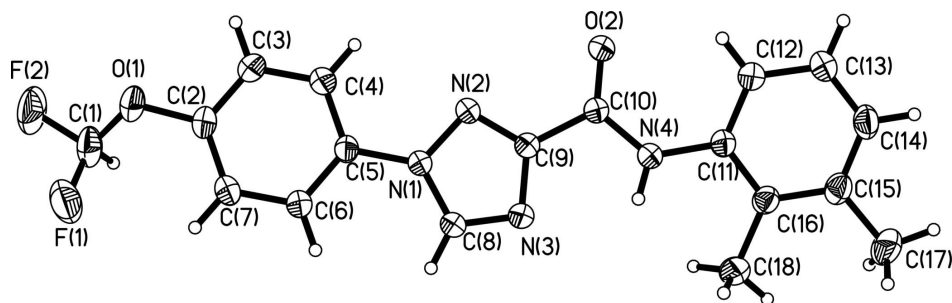


Figure 1

View of the title compound showing the atom numbering scheme and the ellipsoids at the 50% probability level.

1-[4-(Difluoromethoxy)phenyl]-N-(2,3-dimethylphenyl)-1H-1,2,4- triazole-3-carboxamide*Crystal data*C₁₈H₁₆F₂N₄O₂M_r = 358.35Triclinic, *P* $\bar{1}$ *a* = 7.5543 (10) Å*b* = 7.8132 (10) Å*c* = 14.8190 (19) Å α = 95.974 (2)° β = 98.593 (1)° γ = 101.523 (1)°*V* = 839.28 (19) Å³*Z* = 2*F*(000) = 372*D*_x = 1.418 Mg m⁻³Mo *K*α radiation, λ = 0.71073 Åμ = 0.11 mm⁻¹*T* = 296 K

Block, white

0.49 × 0.31 × 0.10 mm

*Data collection*Bruker APEXII CCD
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

φ and ω scans

Absorption correction: multi-scan

(SADABS; Bruker, 2004)

*T*_{min} = 0.948, *T*_{max} = 0.990

6446 measured reflections

3115 independent reflections

2366 reflections with *I* > 2σ(*I*)*R*_{int} = 0.017θ_{max} = 25.5°, θ_{min} = 2.7°*h* = -9→9*k* = -9→9*l* = -17→17*Refinement*Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2σ(*F*²)] = 0.042*wR*(*F*²) = 0.123*S* = 1.06

3115 reflections

238 parameters

0 restraints

Primary atom site location: structure-invariant
direct methodsSecondary atom site location: difference Fourier
mapHydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

w = 1/[σ²(*F*_o²) + (0.0593*P*)² + 0.1647*P*]where *P* = (*F*_o² + 2*F*_c²)/3(Δ/σ)_{max} < 0.001Δρ_{max} = 0.23 e Å⁻³Δρ_{min} = -0.21 e Å⁻³

Extinction correction: SHELXL,

*F*_c* = *kF*_c[1 + 0.001 × *F*_c²λ³/sin(2θ)]^{-1/4}

Extinction coefficient: 0.016 (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 > σ(*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>	$U_{\text{iso}}^*/U_{\text{eq}}$
F1	-0.9182 (2)	0.3392 (2)	0.14577 (12)	0.1146 (6)
F2	-0.8560 (2)	0.1332 (2)	0.05859 (11)	0.1113 (6)
O1	-0.6301 (2)	0.3298 (2)	0.13688 (9)	0.0742 (4)
O2	0.41124 (16)	0.80678 (19)	0.54210 (9)	0.0634 (4)
N1	-0.15235 (18)	0.67315 (18)	0.45595 (9)	0.0458 (4)
N2	0.03287 (19)	0.69741 (19)	0.46115 (10)	0.0472 (4)
N3	-0.0355 (2)	0.8275 (2)	0.59091 (11)	0.0663 (5)
N4	0.32482 (19)	0.9655 (2)	0.66010 (10)	0.0513 (4)
H4D	0.2285	0.9901	0.6779	0.062*
C1	-0.7981 (3)	0.2345 (3)	0.13967 (17)	0.0766 (7)
H1	-0.7944	0.1632	0.1903	0.092*
C2	-0.5184 (3)	0.4106 (2)	0.21999 (13)	0.0541 (5)
C3	-0.3332 (3)	0.4212 (3)	0.22448 (13)	0.0611 (5)
H3	-0.2899	0.3708	0.1749	0.073*
C4	-0.2118 (3)	0.5063 (3)	0.30237 (13)	0.0556 (5)
H4	-0.0866	0.5130	0.3057	0.067*
C5	-0.2770 (2)	0.5815 (2)	0.37546 (11)	0.0444 (4)
C6	-0.4633 (2)	0.5707 (2)	0.37090 (13)	0.0539 (5)
H6	-0.5069	0.6212	0.4203	0.065*
C7	-0.5839 (3)	0.4846 (3)	0.29260 (13)	0.0577 (5)
H7	-0.7093	0.4768	0.2891	0.069*
C8	-0.1882 (3)	0.7512 (3)	0.53408 (14)	0.0649 (6)
H8	-0.3053	0.7513	0.5464	0.078*
C9	0.0950 (2)	0.7909 (2)	0.54299 (11)	0.0460 (4)
C10	0.2942 (2)	0.8537 (2)	0.58047 (11)	0.0448 (4)
C11	0.4936 (2)	1.0475 (2)	0.71818 (12)	0.0464 (4)
C12	0.6569 (2)	1.0709 (2)	0.68418 (13)	0.0538 (5)
H12	0.6575	1.0310	0.6229	0.065*
C13	0.8177 (3)	1.1539 (3)	0.74204 (14)	0.0599 (5)
H13	0.9280	1.1682	0.7200	0.072*
C14	0.8166 (3)	1.2157 (3)	0.83215 (14)	0.0629 (5)
H14	0.9266	1.2713	0.8704	0.076*
C15	0.6546 (3)	1.1965 (3)	0.86695 (13)	0.0572 (5)
C16	0.4892 (2)	1.1100 (2)	0.80967 (12)	0.0506 (4)
C17	0.6597 (3)	1.2699 (3)	0.96572 (15)	0.0823 (7)
H17A	0.7840	1.3231	0.9941	0.124*
H17B	0.6118	1.1762	0.9984	0.124*
H17C	0.5864	1.3570	0.9675	0.124*
C18	0.3094 (3)	1.0884 (3)	0.84471 (14)	0.0703 (6)
H18A	0.2420	1.1706	0.8208	0.105*
H18B	0.3330	1.1105	0.9108	0.105*
H18C	0.2389	0.9703	0.8248	0.105*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0681 (9)	0.1287 (13)	0.1263 (13)	0.0247 (9)	-0.0216 (9)	-0.0270 (10)
F2	0.1052 (12)	0.1003 (11)	0.0926 (11)	-0.0021 (9)	-0.0311 (9)	-0.0318 (8)
O1	0.0631 (9)	0.0896 (11)	0.0523 (8)	0.0000 (8)	-0.0083 (7)	-0.0111 (7)
O2	0.0403 (7)	0.0849 (10)	0.0585 (8)	0.0131 (7)	0.0075 (6)	-0.0162 (7)
N1	0.0359 (8)	0.0537 (8)	0.0444 (8)	0.0091 (6)	0.0045 (6)	-0.0034 (6)
N2	0.0356 (8)	0.0569 (9)	0.0460 (8)	0.0084 (6)	0.0055 (6)	-0.0017 (7)
N3	0.0412 (9)	0.0964 (13)	0.0542 (10)	0.0167 (8)	0.0046 (7)	-0.0201 (9)
N4	0.0378 (8)	0.0640 (10)	0.0482 (9)	0.0110 (7)	0.0053 (6)	-0.0065 (7)
C1	0.0699 (15)	0.0672 (14)	0.0725 (15)	0.0012 (12)	-0.0212 (11)	-0.0079 (11)
C2	0.0521 (11)	0.0548 (11)	0.0466 (10)	0.0044 (8)	-0.0037 (8)	-0.0005 (8)
C3	0.0570 (12)	0.0698 (13)	0.0502 (11)	0.0090 (10)	0.0090 (9)	-0.0105 (9)
C4	0.0431 (10)	0.0625 (11)	0.0562 (11)	0.0076 (8)	0.0075 (8)	-0.0050 (9)
C5	0.0410 (9)	0.0456 (9)	0.0425 (9)	0.0072 (7)	0.0015 (7)	0.0009 (7)
C6	0.0436 (10)	0.0648 (12)	0.0495 (11)	0.0138 (9)	0.0027 (8)	-0.0050 (9)
C7	0.0410 (10)	0.0693 (12)	0.0582 (12)	0.0123 (9)	-0.0005 (8)	-0.0001 (9)
C8	0.0379 (10)	0.0943 (15)	0.0557 (11)	0.0151 (10)	0.0055 (8)	-0.0180 (10)
C9	0.0397 (9)	0.0541 (10)	0.0428 (9)	0.0117 (8)	0.0056 (7)	-0.0004 (8)
C10	0.0396 (9)	0.0511 (10)	0.0421 (9)	0.0100 (7)	0.0054 (7)	0.0012 (7)
C11	0.0413 (9)	0.0497 (10)	0.0451 (10)	0.0091 (7)	0.0030 (7)	0.0013 (8)
C12	0.0469 (10)	0.0614 (11)	0.0496 (10)	0.0084 (8)	0.0078 (8)	-0.0010 (8)
C13	0.0433 (10)	0.0661 (12)	0.0638 (12)	0.0047 (9)	0.0059 (9)	-0.0008 (10)
C14	0.0485 (11)	0.0651 (12)	0.0638 (13)	0.0056 (9)	-0.0084 (9)	-0.0033 (10)
C15	0.0601 (12)	0.0597 (11)	0.0472 (10)	0.0145 (9)	-0.0024 (9)	0.0008 (9)
C16	0.0514 (10)	0.0557 (11)	0.0436 (10)	0.0150 (8)	0.0037 (8)	0.0018 (8)
C17	0.0873 (17)	0.0976 (18)	0.0509 (12)	0.0169 (14)	-0.0047 (11)	-0.0101 (11)
C18	0.0616 (13)	0.0961 (16)	0.0510 (12)	0.0169 (11)	0.0128 (10)	-0.0033 (11)

Geometric parameters (Å, °)

F1—C1	1.345 (3)	C6—C7	1.381 (2)
F2—C1	1.330 (3)	C6—H6	0.9300
O1—C1	1.346 (3)	C7—H7	0.9300
O1—C2	1.394 (2)	C8—H8	0.9300
O2—C10	1.215 (2)	C9—C10	1.487 (2)
N1—C8	1.341 (2)	C11—C12	1.387 (2)
N1—N2	1.3632 (19)	C11—C16	1.400 (2)
N1—C5	1.428 (2)	C12—C13	1.374 (3)
N2—C9	1.316 (2)	C12—H12	0.9300
N3—C8	1.314 (2)	C13—C14	1.374 (3)
N3—C9	1.355 (2)	C13—H13	0.9300
N4—C10	1.353 (2)	C14—C15	1.385 (3)
N4—C11	1.417 (2)	C14—H14	0.9300
N4—H4D	0.8600	C15—C16	1.402 (3)
C1—H1	0.9800	C15—C17	1.507 (3)
C2—C7	1.372 (3)	C16—C18	1.510 (3)

C2—C3	1.376 (3)	C17—H17A	0.9600
C3—C4	1.377 (3)	C17—H17B	0.9600
C3—H3	0.9300	C17—H17C	0.9600
C4—C5	1.380 (2)	C18—H18A	0.9600
C4—H4	0.9300	C18—H18B	0.9600
C5—C6	1.384 (2)	C18—H18C	0.9600
C1—O1—C2	118.10 (17)	N2—C9—N3	115.33 (15)
C8—N1—N2	109.18 (14)	N2—C9—C10	122.68 (15)
C8—N1—C5	129.19 (15)	N3—C9—C10	121.99 (15)
N2—N1—C5	121.60 (13)	O2—C10—N4	125.87 (16)
C9—N2—N1	102.16 (13)	O2—C10—C9	122.19 (15)
C8—N3—C9	102.47 (15)	N4—C10—C9	111.94 (14)
C10—N4—C11	129.00 (15)	C12—C11—C16	121.23 (16)
C10—N4—H4D	115.5	C12—C11—N4	120.80 (16)
C11—N4—H4D	115.5	C16—C11—N4	117.93 (15)
F2—C1—F1	105.50 (18)	C13—C12—C11	119.26 (17)
F2—C1—O1	106.7 (2)	C13—C12—H12	120.4
F1—C1—O1	110.9 (2)	C11—C12—H12	120.4
F2—C1—H1	111.2	C14—C13—C12	120.48 (18)
F1—C1—H1	111.2	C14—C13—H13	119.8
O1—C1—H1	111.2	C12—C13—H13	119.8
C7—C2—C3	120.57 (17)	C13—C14—C15	121.14 (18)
C7—C2—O1	123.26 (18)	C13—C14—H14	119.4
C3—C2—O1	116.09 (17)	C15—C14—H14	119.4
C2—C3—C4	120.04 (18)	C14—C15—C16	119.45 (18)
C2—C3—H3	120.0	C14—C15—C17	119.27 (19)
C4—C3—H3	120.0	C16—C15—C17	121.28 (19)
C3—C4—C5	119.59 (18)	C11—C16—C15	118.43 (17)
C3—C4—H4	120.2	C11—C16—C18	120.51 (16)
C5—C4—H4	120.2	C15—C16—C18	121.04 (17)
C4—C5—C6	120.32 (16)	C15—C17—H17A	109.5
C4—C5—N1	120.17 (15)	C15—C17—H17B	109.5
C6—C5—N1	119.50 (15)	H17A—C17—H17B	109.5
C7—C6—C5	119.65 (17)	C15—C17—H17C	109.5
C7—C6—H6	120.2	H17A—C17—H17C	109.5
C5—C6—H6	120.2	H17B—C17—H17C	109.5
C2—C7—C6	119.82 (18)	C16—C18—H18A	109.5
C2—C7—H7	120.1	C16—C18—H18B	109.5
C6—C7—H7	120.1	H18A—C18—H18B	109.5
N3—C8—N1	110.85 (16)	C16—C18—H18C	109.5
N3—C8—H8	124.6	H18A—C18—H18C	109.5
N1—C8—H8	124.6	H18B—C18—H18C	109.5
C8—N1—N2—C9	-0.5 (2)	C8—N3—C9—N2	-0.1 (2)
C5—N1—N2—C9	177.77 (15)	C8—N3—C9—C10	179.36 (18)
C2—O1—C1—F2	-162.99 (17)	C11—N4—C10—O2	-2.5 (3)
C2—O1—C1—F1	82.6 (2)	C11—N4—C10—C9	177.38 (16)

C1—O1—C2—C7	-37.5 (3)	N2—C9—C10—O2	-8.1 (3)
C1—O1—C2—C3	145.8 (2)	N3—C9—C10—O2	172.46 (18)
C7—C2—C3—C4	0.1 (3)	N2—C9—C10—N4	172.08 (16)
O1—C2—C3—C4	176.96 (18)	N3—C9—C10—N4	-7.4 (2)
C2—C3—C4—C5	-0.4 (3)	C10—N4—C11—C12	22.9 (3)
C3—C4—C5—C6	0.4 (3)	C10—N4—C11—C16	-159.57 (17)
C3—C4—C5—N1	-178.75 (17)	C16—C11—C12—C13	1.4 (3)
C8—N1—C5—C4	-178.5 (2)	N4—C11—C12—C13	178.84 (17)
N2—N1—C5—C4	3.6 (2)	C11—C12—C13—C14	-1.1 (3)
C8—N1—C5—C6	2.4 (3)	C12—C13—C14—C15	-0.1 (3)
N2—N1—C5—C6	-175.54 (16)	C13—C14—C15—C16	1.1 (3)
C4—C5—C6—C7	-0.2 (3)	C13—C14—C15—C17	-178.7 (2)
N1—C5—C6—C7	178.98 (16)	C12—C11—C16—C15	-0.4 (3)
C3—C2—C7—C6	0.1 (3)	N4—C11—C16—C15	-177.95 (16)
O1—C2—C7—C6	-176.50 (17)	C12—C11—C16—C18	178.06 (18)
C5—C6—C7—C2	-0.1 (3)	N4—C11—C16—C18	0.5 (3)
C9—N3—C8—N1	-0.2 (2)	C14—C15—C16—C11	-0.8 (3)
N2—N1—C8—N3	0.5 (2)	C17—C15—C16—C11	178.95 (19)
C5—N1—C8—N3	-177.64 (17)	C14—C15—C16—C18	-179.26 (19)
N1—N2—C9—N3	0.4 (2)	C17—C15—C16—C18	0.5 (3)
N1—N2—C9—C10	-179.09 (15)		

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
N4—H4D \cdots N3	0.86	2.27	2.717 (2)	113
C6—H6 \cdots O2 ⁱ	0.93	2.43	3.344 (2)	169
C8—H8 \cdots O2 ⁱ	0.93	2.26	3.159 (2)	162

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