data reports

Crystal structure of 2-{[(naphthalen-1-yl)-oxy]methyl}-5-(2,4,5-trifluorophenyl)-1,3,4-oxadiazole

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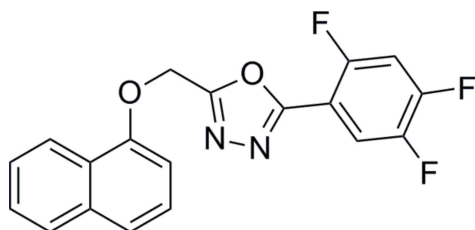
In the title compound $C_{19}H_{11}F_3N_2O_2$, the oxadiazole ring and the naphthalene ring system are approximately planar (r.m.s. deviations of 0.001 and 0.020 Å, respectively) and the oxadiazole ring makes dihedral angles of 13.11 (1) and 7.59 (1)° with the naphthalene ring system and the trifluoro-phenyl ring, respectively. In the crystal, C—H···N hydrogen bonds link molecules into chains along the *a*-axis direction, while C—H···F contacts form additional chains along the *ac* diagonal. These contacts generate sheets of molecules approximately parallel to the (011) plane.

Keywords: crystal structure; naphthalen-1-yloxy; trifluorophenyl; 1,3,4-oxadiazole; hydrogen bonding.

1. Related literature

For the biological activity and other applications of triazole derivatives, see: Desai *et al.* (2014); Bhat *et al.* (2011); Katrin *et al.* (2005); Shailaja *et al.* (2010).

2. Experimental

2.1. Crystal data

$C_{19}H_{11}F_3N_2O_2$	$\gamma = 65.370\ (3)°$
$M_r = 356.30$	$V = 792.36\ (9)\ Å^3$
Triclinic, $P\bar{1}$	$Z = 2$
$a = 7.4817\ (5)\ Å$	Mo $K\alpha$ radiation
$b = 7.5928\ (5)\ Å$	$\mu = 0.12\ mm^{-1}$
$c = 15.7908\ (10)\ Å$	$T = 293\ K$
$\alpha = 78.673\ (3)°$	$0.25 \times 0.15 \times 0.10\ mm$
$\beta = 78.404\ (3)°$	

2.2. Data collection

Bruker SMART APEXII area-detector diffractometer	11539 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)	3330 independent reflections
$T_{min} = 0.970$, $T_{max} = 0.988$	2402 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.031$

2.3. Refinement

$R[F^2 > 2\sigma(F^2)] = 0.047$	236 parameters
$wR(F^2) = 0.140$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{max} = 0.22\ e\ Å^{-3}$
3330 reflections	$\Delta\rho_{min} = -0.31\ e\ Å^{-3}$

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N2^i	0.93	2.61	3.449 (3)	151
C5—H5···F2^{ii}	0.93	2.51	3.290 (3)	141

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

Data collection: *APEX2* (Bruker, 2008); cell refinement: *SAINT* (Bruker, 2008); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEP-3 for Windows* (Farrugia, 2012); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2009).

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supporting information

Crystal structure of 2-{[(naphthalen-1-yl)oxy]methyl}-5-(2,4,5-trifluoro-phenyl)-1,3,4-oxadiazole

Muniyappan Govindhan, Kathavarayan Subramanian, Vijayan Viswanathan and Devadasan Velmurugan

S1. Comment

1,3,4-Oxadiazoles are a class of 5-membered heterocyclic compounds that have a wide range of biological activity as well as polymer and material science applications (Shailaja *et al.*, 2010). Their derivatives are commonly used pharmacophores due to their metabolic profile and ability to engage in hydrogen bonding. Their pharmaceutical applications include use as anti-inflammatory, analgesic, anti-HIV, antimycobacterial agents, cathepsin K, tyrosinase and monoamine oxidase (MAO) inhibitors (Desai *et al.*, 2014). They are also used as anticonvulsant, anticancer, antifungal and tuberculostatic drugs (Bhat *et al.*,2011). They also have analgesic, antiplatelet and antithrombotic activities (Katrin *et al.*, 2005). Moreover, their amino derivatives are very commonly used as antimicrobial and germicidal agents.

In the title compound $C_{19}H_{11}N_2O_2F_3$, Fig. 1, the oxadiazole, napthalene and trifluorophenyl rings are planar and the oxadiazole ring (C12/N1/N2/C13/O2) makes dihedral angles of 13.11 (1)° and 7.59 (1)° with the naphthalene (C1-C10) ring system and the trifluorophenyl (C14—C19) ring respectively. The fluorine F1, F2 and F3 atoms deviate from the benzene ring by -0.0017 Å, 0.0175 Å and -0.0163 Å respectively.
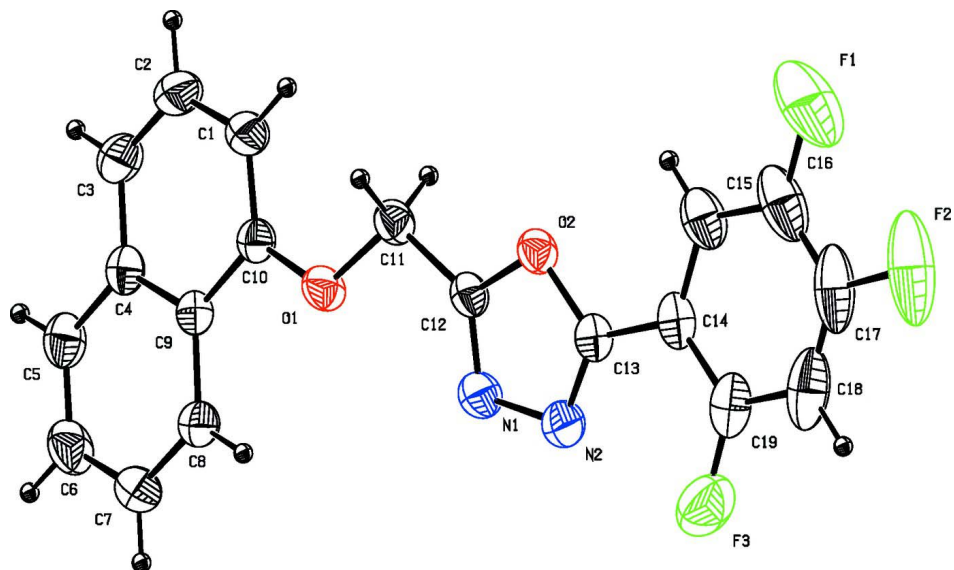
In the crystal C1–H1···N2 hydrogen bonds link molecules into chains along *a* while C5–H5···F2 contacts form additional chains along the *ac* diagonal. These contacts generate sheets of molecules approximately parallel to the (011) plane. Within these sheets groups of four molecules form $R^4_4(37)$ ring motifs.
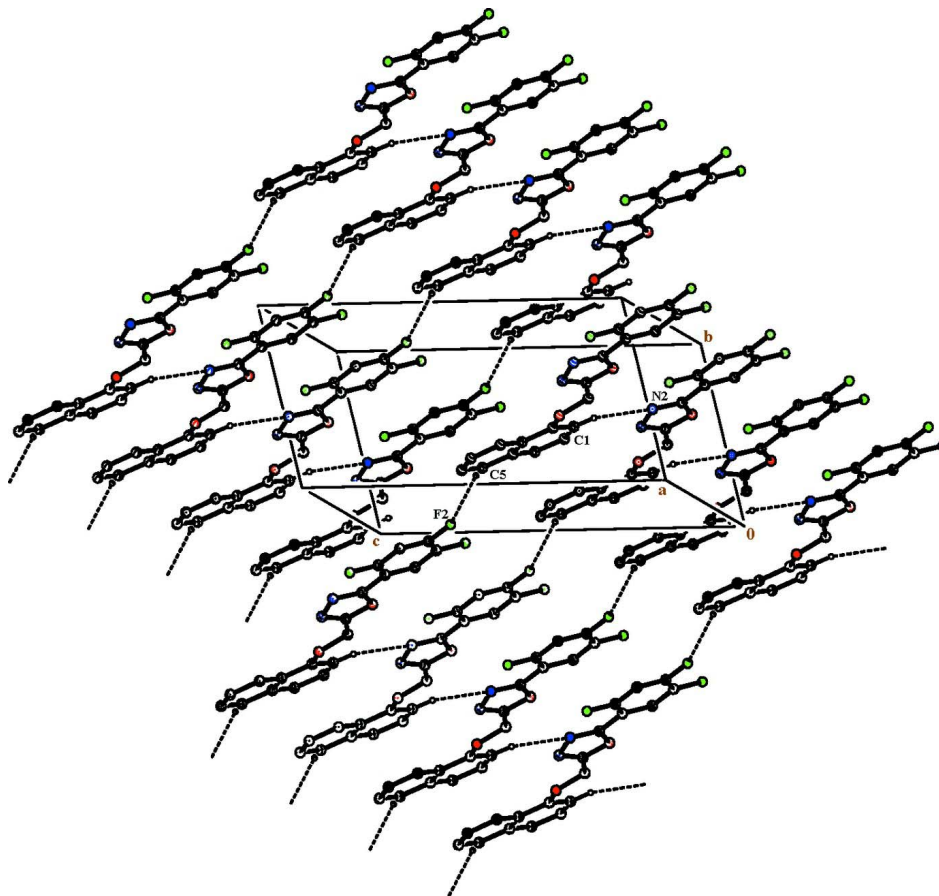
S2. Experimental

Iodobenzene diacetate (2.0 mol eq) was added to a solution of naphthalen-1-yloxy-acetic acid (2, 4, 5-trifluoro-benzylidene)-hydrazide (1.0 mole eq) in dioxane (10mL) at 25-30 ° C and stirred at the same temperature for 15-30 minutes. Completion of the reaction was confirmed by TLC (mobile phase ethyl acetate/hexane, 3:7). The dioxane was distilled off under vacuum. The resulting residue was dissolved in ethyl acetate and washed with saturated sodium bicarbonate solution, followed by water and brine solution. The separated organic layer was dried over anhydrous sodium sulfate and distilled under vacuum. The crude product was purified by column chromatography over silica gel (60-120 mesh) using hexane and ethyl acetate (9:1) as eluent to afford the pure product of as an off-white solid. After purification the compound was crystallized from methanol by the slow evaporation method.

S3. Refinement

The hydrogen atoms were placed in calculated positions with C—H = 0.93Å, refined in the riding model with fixed isotropic displacement parameters: $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl group and $U_{iso}(H) = 1.2U_{eq}(C)$ for other H atoms.

Figure 1

The molecular structure of the title compound, showing the atomic numbering with displacement ellipsoids drawn at the 30% probability level.

Figure 2
Crystal packing of the title compound viewed along the *a* axis H-atoms not involved in H-bonds have been excluded for clarity.

2-{[(Naphthalen-1-yl)oxy]methyl}-5-(2,4,5-trifluorophenyl)-1,3,4-oxadiazole

Crystal data

$C_{19}H_{11}F_3N_2O_2$	$Z = 2$
$M_r = 356.30$	$F(000) = 364$
Triclinic, $P\bar{1}$	$D_x = 1.493$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 7.4817$ (5) Å	Cell parameters from 3330 reflections
$b = 7.5928$ (5) Å	$\theta = 1.3–26.8°$
$c = 15.7908$ (10) Å	$\mu = 0.12$ mm^{-1}
$\alpha = 78.673$ (3)°	$T = 293$ K
$\beta = 78.404$ (3)°	Block, colourless
$\gamma = 65.370$ (3)°	$0.25 \times 0.15 \times 0.10$ mm
$V = 792.36$ (9) Å^3	

Data collection

Bruker SMART APEXII area-detector diffractometer	Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
Radiation source: fine-focus sealed tube	$T_{min} = 0.970$, $T_{max} = 0.988$
Graphite monochromator	11539 measured reflections
ω and φ scans	3330 independent reflections

2402 reflections with $I > 2\sigma(I)$

$R_{int} = 0.031$

$\theta_{max} = 26.8°$, $\theta_{min} = 1.3°$

$h = -9\rightarrow9$

$k = -9\rightarrow9$

$l = -19\rightarrow19$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.047$	$w = 1/[\sigma^2(F_o^2) + (0.062P)^2 + 0.2065P]$
$wR(F^2) = 0.140$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3330 reflections	$\Delta\rho_{max} = 0.22$ e Å^{-3}
236 parameters	$\Delta\rho_{min} = -0.31$ e Å^{-3}
0 restraints	Extinction correction: *SHELXL97* (Sheldrick,
Primary atom site location: structure-invariant direct methods	2008), Fc*=kFc[1+0.001xFc$^2\lambda^3$/sin(2θ)]$^{-1/4}$
Secondary atom site location: difference Fourier map	Extinction coefficient: 0.006 (2)

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.

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 > 2sigma(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}
C1	0.4888 (3)	0.3315 (3)	0.37814 (13)	0.0588 (5)
H1	0.5114	0.2802	0.4354	0.071*
C2	0.6415 (3)	0.3567 (3)	0.31604 (15)	0.0691 (6)
H2	0.7656	0.3212	0.3326	0.083*
C3	0.6121 (3)	0.4313 (3)	0.23273 (14)	0.0673 (5)
H3	0.7168	0.4439	0.1925	0.081*
C4	0.4239 (3)	0.4907 (3)	0.20584 (12)	0.0543 (5)
C5	0.3867 (3)	0.5717 (3)	0.12011 (13)	0.0670 (5)
H5	0.4891	0.5862	0.0789	0.080*
C6	0.2045 (4)	0.6293 (3)	0.09616 (14)	0.0744 (6)
H6	0.1830	0.6829	0.0390	0.089*
C7	0.0489 (3)	0.6080 (3)	0.15737 (14)	0.0699 (6)
H7	−0.0759	0.6483	0.1406	0.084*
C8	0.0783 (3)	0.5289 (3)	0.24135 (13)	0.0578 (5)
H8	−0.0265	0.5158	0.2814	0.069*
C9	0.2666 (3)	0.4668 (2)	0.26779 (11)	0.0474 (4)
C10	0.3070 (3)	0.3827 (2)	0.35414 (11)	0.0483 (4)
C11	0.1842 (3)	0.2579 (3)	0.49334 (11)	0.0540 (4)
H11A	0.2063	0.3365	0.5285	0.065*
H11B	0.2998	0.1361	0.4898	0.065*

C12	0.0033 (3)	0.2197 (2)	0.53195 (11)	0.0491 (4)
C13	−0.1893 (3)	0.1278 (2)	0.63030 (12)	0.0516 (4)
C14	−0.2556 (3)	0.0518 (3)	0.71692 (12)	0.0595 (5)
C15	−0.1309 (4)	−0.0186 (3)	0.78032 (14)	0.0767 (6)
H15	−0.0047	−0.0168	0.7679	0.092*
C16	−0.1951 (6)	−0.0916 (4)	0.86218 (16)	0.1020 (10)
C17	−0.3818 (6)	−0.0941 (4)	0.88135 (19)	0.1061 (12)
C18	−0.5058 (5)	−0.0245 (4)	0.8207 (2)	0.1034 (11)
H18	−0.6323	−0.0254	0.8342	0.124*
C19	−0.4444 (4)	0.0477 (3)	0.73917 (17)	0.0761 (7)
N1	−0.1442 (3)	0.2435 (2)	0.49547 (10)	0.0622 (4)
N2	−0.2727 (2)	0.1818 (2)	0.56086 (11)	0.0620 (4)
O1	0.14915 (18)	0.35881 (19)	0.40929 (8)	0.0582 (4)
O2	−0.01186 (18)	0.14762 (17)	0.61719 (7)	0.0520 (3)
F1	−0.0726 (4)	−0.1601 (3)	0.92306 (11)	0.1607 (9)
F2	−0.4342 (4)	−0.1687 (3)	0.96228 (11)	0.1684 (11)
F3	−0.5703 (2)	0.1171 (3)	0.68014 (13)	0.1095 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0581 (11)	0.0638 (11)	0.0548 (11)	−0.0257 (9)	−0.0118 (9)	0.0004 (9)
C2	0.0548 (11)	0.0827 (14)	0.0737 (14)	−0.0332 (10)	−0.0113 (10)	−0.0018 (11)
C3	0.0564 (11)	0.0796 (13)	0.0672 (13)	−0.0357 (10)	0.0033 (10)	−0.0038 (10)
C4	0.0632 (11)	0.0516 (9)	0.0494 (10)	−0.0280 (8)	0.0000 (8)	−0.0044 (8)
C5	0.0804 (14)	0.0697 (12)	0.0523 (11)	−0.0389 (11)	0.0030 (10)	−0.0017 (9)
C6	0.0996 (18)	0.0763 (14)	0.0483 (11)	−0.0395 (13)	−0.0165 (11)	0.0081 (10)
C7	0.0737 (13)	0.0743 (13)	0.0643 (13)	−0.0317 (11)	−0.0240 (11)	0.0069 (10)
C8	0.0586 (11)	0.0608 (11)	0.0557 (11)	−0.0285 (9)	−0.0075 (9)	0.0000 (8)
C9	0.0538 (10)	0.0431 (8)	0.0461 (9)	−0.0215 (7)	−0.0049 (7)	−0.0038 (7)
C10	0.0498 (10)	0.0478 (9)	0.0468 (10)	−0.0212 (7)	−0.0020 (8)	−0.0047 (7)
C11	0.0594 (11)	0.0543 (10)	0.0452 (10)	−0.0225 (8)	−0.0082 (8)	0.0021 (8)
C12	0.0587 (10)	0.0461 (9)	0.0404 (9)	−0.0205 (8)	−0.0072 (8)	−0.0006 (7)
C13	0.0597 (11)	0.0440 (9)	0.0495 (10)	−0.0213 (8)	−0.0028 (8)	−0.0049 (7)
C14	0.0797 (13)	0.0438 (9)	0.0496 (11)	−0.0250 (9)	0.0047 (9)	−0.0061 (8)
C15	0.1047 (18)	0.0658 (12)	0.0487 (12)	−0.0290 (12)	−0.0033 (11)	−0.0001 (9)
C16	0.170 (3)	0.0668 (14)	0.0494 (13)	−0.0327 (17)	−0.0085 (17)	0.0005 (11)
C17	0.174 (3)	0.0633 (14)	0.0662 (17)	−0.0576 (18)	0.047 (2)	−0.0151 (12)
C18	0.130 (3)	0.0742 (16)	0.100 (2)	−0.0583 (17)	0.0498 (19)	−0.0246 (15)
C19	0.0887 (16)	0.0575 (12)	0.0788 (15)	−0.0364 (11)	0.0182 (13)	−0.0137 (11)
N1	0.0677 (10)	0.0713 (10)	0.0480 (9)	−0.0315 (8)	−0.0122 (8)	0.0060 (7)
N2	0.0668 (10)	0.0677 (10)	0.0549 (10)	−0.0327 (8)	−0.0108 (8)	0.0020 (8)
O1	0.0534 (7)	0.0692 (8)	0.0444 (7)	−0.0241 (6)	−0.0046 (5)	0.0083 (6)
O2	0.0606 (8)	0.0538 (7)	0.0410 (7)	−0.0243 (6)	−0.0080 (5)	0.0010 (5)
F1	0.249 (3)	0.1436 (16)	0.0596 (10)	−0.0549 (17)	−0.0400 (13)	0.0235 (10)
F2	0.303 (3)	0.1016 (12)	0.0759 (11)	−0.1005 (16)	0.0777 (14)	−0.0141 (9)
F3	0.0853 (10)	0.1222 (13)	0.1295 (14)	−0.0566 (9)	0.0005 (10)	−0.0139 (11)

Geometric parameters (Å, º)

C1—C10	1.361 (3)	C11—C12	1.487 (3)
C1—C2	1.401 (3)	C11—H11A	0.9700
C1—H1	0.9300	C11—H11B	0.9700
C2—C3	1.349 (3)	C12—N1	1.277 (2)
C2—H2	0.9300	C12—O2	1.351 (2)
C3—C4	1.415 (3)	C13—N2	1.283 (2)
C3—H3	0.9300	C13—O2	1.368 (2)
C4—C5	1.404 (3)	C13—C14	1.451 (3)
C4—C9	1.421 (2)	C14—C15	1.381 (3)
C5—C6	1.355 (3)	C14—C19	1.398 (3)
C5—H5	0.9300	C15—C16	1.378 (3)
C6—C7	1.400 (3)	C15—H15	0.9300
C6—H6	0.9300	C16—F1	1.340 (4)
C7—C8	1.364 (3)	C16—C17	1.376 (5)
C7—H7	0.9300	C17—F2	1.344 (3)
C8—C9	1.410 (3)	C17—C18	1.344 (5)
C8—H8	0.9300	C18—C19	1.366 (4)
C9—C10	1.422 (2)	C18—H18	0.9300
C10—O1	1.375 (2)	C19—F3	1.334 (3)
C11—O1	1.411 (2)	N1—N2	1.413 (2)
C10—C1—C2	119.51 (18)	C12—C11—H11A	110.5
C10—C1—H1	120.2	O1—C11—H11B	110.5
C2—C1—H1	120.2	C12—C11—H11B	110.5
C3—C2—C1	121.32 (19)	H11A—C11—H11B	108.7
C3—C2—H2	119.3	N1—C12—O2	113.26 (16)
C1—C2—H2	119.3	N1—C12—C11	129.06 (16)
C2—C3—C4	120.79 (18)	O2—C12—C11	117.67 (15)
C2—C3—H3	119.6	N2—C13—O2	112.38 (16)
C4—C3—H3	119.6	N2—C13—C14	129.71 (19)
C5—C4—C3	122.47 (18)	O2—C13—C14	117.91 (17)
C5—C4—C9	118.68 (18)	C15—C14—C19	118.1 (2)
C3—C4—C9	118.85 (18)	C15—C14—C13	120.0 (2)
C6—C5—C4	121.36 (19)	C19—C14—C13	122.0 (2)
C6—C5—H5	119.3	C16—C15—C14	119.4 (3)
C4—C5—H5	119.3	C16—C15—H15	120.3
C5—C6—C7	120.0 (2)	C14—C15—H15	120.3
C5—C6—H6	120.0	F1—C16—C17	120.4 (3)
C7—C6—H6	120.0	F1—C16—C15	118.9 (3)
C8—C7—C6	120.7 (2)	C17—C16—C15	120.7 (3)
C8—C7—H7	119.6	F2—C17—C18	121.7 (4)
C6—C7—H7	119.6	F2—C17—C16	117.6 (4)
C7—C8—C9	120.36 (18)	C18—C17—C16	120.7 (3)
C7—C8—H8	119.8	C17—C18—C19	119.4 (3)
C9—C8—H8	119.8	C17—C18—H18	120.3
C8—C9—C4	118.84 (17)	C19—C18—H18	120.3

C8—C9—C10	123.12 (16)	F3—C19—C18	118.4 (3)
C4—C9—C10	118.04 (16)	F3—C19—C14	119.9 (2)
C1—C10—O1	124.10 (16)	C18—C19—C14	121.7 (3)
C1—C10—C9	121.46 (16)	C12—N1—N2	105.99 (15)
O1—C10—C9	114.45 (15)	C13—N2—N1	106.01 (15)
O1—C11—C12	106.06 (14)	C10—O1—C11	117.48 (14)
O1—C11—H11A	110.5	C12—O2—C13	102.37 (13)
C10—C1—C2—C3	0.2 (3)	C13—C14—C15—C16	−179.59 (18)
C1—C2—C3—C4	1.4 (3)	C14—C15—C16—F1	179.8 (2)
C2—C3—C4—C5	179.02 (19)	C14—C15—C16—C17	−0.3 (4)
C2—C3—C4—C9	−1.2 (3)	F1—C16—C17—F2	−0.6 (4)
C3—C4—C5—C6	−179.3 (2)	C15—C16—C17—F2	179.5 (2)
C9—C4—C5—C6	0.9 (3)	F1—C16—C17—C18	179.6 (2)
C4—C5—C6—C7	−0.2 (3)	C15—C16—C17—C18	−0.3 (4)
C5—C6—C7—C8	−0.3 (3)	F2—C17—C18—C19	−179.3 (2)
C6—C7—C8—C9	−0.1 (3)	C16—C17—C18—C19	0.6 (4)
C7—C8—C9—C4	0.9 (3)	C17—C18—C19—F3	−179.6 (2)
C7—C8—C9—C10	−179.79 (17)	C17—C18—C19—C14	−0.2 (4)
C5—C4—C9—C8	−1.3 (3)	C15—C14—C19—F3	179.00 (19)
C3—C4—C9—C8	178.98 (17)	C13—C14—C19—F3	−0.7 (3)
C5—C4—C9—C10	179.36 (16)	C15—C14—C19—C18	−0.4 (3)
C3—C4—C9—C10	−0.4 (2)	C13—C14—C19—C18	179.84 (19)
C2—C1—C10—O1	178.07 (17)	O2—C12—N1—N2	−0.3 (2)
C2—C1—C10—C9	−1.9 (3)	C11—C12—N1—N2	178.92 (17)
C8—C9—C10—C1	−177.39 (17)	O2—C13—N2—N1	−0.1 (2)
C4—C9—C10—C1	2.0 (2)	C14—C13—N2—N1	179.94 (17)
C8—C9—C10—O1	2.7 (2)	C12—N1—N2—C13	0.2 (2)
C4—C9—C10—O1	−177.99 (14)	C1—C10—O1—C11	−6.4 (3)
O1—C11—C12—N1	9.7 (3)	C9—C10—O1—C11	173.59 (14)
O1—C11—C12—O2	−171.20 (14)	C12—C11—O1—C10	−169.31 (14)
N2—C13—C14—C15	172.59 (19)	N1—C12—O2—C13	0.18 (19)
O2—C13—C14—C15	−7.3 (3)	C11—C12—O2—C13	−179.10 (14)
N2—C13—C14—C19	−7.7 (3)	N2—C13—O2—C12	−0.01 (19)
O2—C13—C14—C19	172.39 (16)	C14—C13—O2—C12	179.92 (14)
C19—C14—C15—C16	0.7 (3)		

Hydrogen-bond geometry (Å, º)

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···N2[i]	0.93	2.61	3.449 (3)	151
C5—H5···F2[ii]	0.93	2.51	3.290 (3)	141

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