2-Amino-7,7-dimethyl-5-oxo-4-[3-(trifluoromethyl)phenyl]-5,6,7,8-tetrahydro-4H-chromene-3-carbonitrile

Rajni Kant,[a]* Vivek K. Gupta,[a] Kamini Kapoor,[a] D. R. Patil,[b] D. R. Chandam[b] and Madhukar B. Deshmukh[b]

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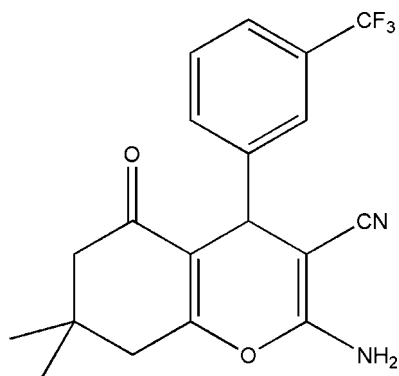Correspondence e-mail: rkvk.paper11@gmail.com

In the title molecule, $C_{19}H_{17}F_3N_2O_2$, the fused cyclohexene and pyran rings adopt sofa and flattened boat conformations, respectively. The four essentially planar atoms of the pyran ring [maximum deviation = 0.008 (2) Å] form a dihedral angle of 88.13 (9)° with the benzene ring. The F atoms of the trifluoromethyl group were refined as disordered over three sets of sites in a 0.507 (7):0.330 (7):0.163 (3) ratio. In the crystal, molecules are connected into inversion dimers via pairs of N—H⋯N hydrogen bonds and these dimers are further linked by N—H⋯O hydrogen bonds into a two-dimensional network parallel to (100).

Related literature

For the biological activity of 4H-pyran derivatives, see: Bhattacharyya et al.(2012); Khaksar et al. (2012); Fotouhi et al. (2007). For related structures, see: Wang (2011); Anthal et al. (2012); Kant et al. (2013). For ring conformations, see: Duax & Norton (1975).

Experimental

Crystal data

$C_{19}H_{17}F_3N_2O_2$	V = 3530.33 (15) Å³
M_r = 362.35	Z = 8
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
a = 23.7543 (6) Å	μ = 0.11 mm⁻¹
b = 9.3871 (2) Å	T = 293 K
c = 15.8857 (4) Å	$0.3 \times 0.2 \times 0.2$ mm
β = 94.704 (2)°	

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	40937 measured reflections
Absorption correction: multi-scan (CrysAlis PRO; Oxford Diffraction, 2010)	3467 independent reflections
	2538 reflections with $I > 2\sigma(I)$
T_{min} = 0.766, T_{max} = 1.000	R_{int} = 0.065

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061	10 restraints
$wR(F^2)$ = 0.126	H-atom parameters constrained
S = 1.03	$\Delta\rho_{max}$ = 0.26 e Å⁻³
3467 reflections	$\Delta\rho_{min}$ = −0.32 e Å⁻³
258 parameters	

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

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N21—H21A⋯N20ⁱ	0.86	2.17	3.025 (3)	171
N21—H21B⋯O2ⁱⁱ	0.86	2.10	2.934 (2)	163

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

Data collection: CrysAlis PRO (Oxford Diffraction, 2010); cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; 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) and PLATON (Spek, 2009); software used to prepare material for publication: PLATON.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5585).

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supporting information

2-Amino-7,7-dimethyl-5-oxo-4-[3-(trifluoromethyl)phenyl]-5,6,7,8-tetrahydro-4*H*-chromene-3-carbonitrile

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S1. Comment

Polyfunctionalized 4H-pyran derivatives are used as anti-coagulants, anticancer agents, spasmolytics, anti-anaphylactics, anti-microbial and immunomodulating activities (Khaksar *et al.*, 2012; Bhattacharyya *et al.*, 2012). Furthermore, these compounds can be employed as pigments, photoactive materials and used as biodegradable agrochemicals (Fotouhi *et al.*, 2007). In this paper, we report the crystal structure of the title compound, (I).

In (I) (Fig.1), all bond lengths and angles are normal and correspond to those observed in related structures (Wang *et al.*,2011; Anthal *et al.*, 2012; Kant *et al.*,2013). The cyclohexene ring (C5/C6/C7/C8/C8A/C4A) and and pyran ring (O1/C2/C3/C4/C4A/C8A) exhibit sofa and boat conformations, respectively, with asymmetry parameters ($\Delta Cs(C7) = 9.78$ & $\Delta Cs(C4) = 2.36$, $\Delta Cs(C2-C3) = 9.4$)(Duax & Norton, 1975) with atom C7 forming the flap in the cyclohexene ring. The four essentially planar atoms (C2/C3/C4A/C8A) of pyran ring (maximum deviation = -0.008 (2)Å for C8A) form a dihedral angle of 88.13 (9)° with benzene the ring. The F atoms of the trifluoromethyl group were refined as disordered over three sets of sites in a 0.507 (7) : 0.330 (7) : 0.163 (3) ratio. In the crystal, molecules are connected into dimers via N21—H21A⋯N20[i] hydrogen bonds and these dimers are further connected by N21—H21B⋯O2[ii] (Table 1) hydrogen bonds into a two-dimensional network (Fig. 2) parallel to (100).

S2. Experimental

In a 50 ml round bottom flask charged with 1mmole of dimedone, 1 mmole of 3-(trifluoromethyl)benzaldehyde and 1 mmole of malononitrile were added. Then 5 ml of aqueous ethanol (1:1) and 20 mol% of NH₄Cl was added and the reaction mixture stirred for 30-45 min. at 323-328 K. The reaction was monitored by TLC. After completion of the reaction, the mixture was poured onto crushed ice and stirred. The solid precipitated was filtered and recrystallized from ethanol to afford pure product as crystal suitable for X-ray diffraction.

m.p.: 503-504 K, Yield: 82%.

1H NMR (300MHz,DMSO-d6): δ 0.94(s, 3H, CH3), 1.02(s, 3H, CH3), 2.05-2.20(m,2H, CH2), 2.44-2.49(m, 2H, CH2), 4.18(s, 1H, CH), 6.73(s, 2H,NH2), 6.81-6.87(m, 2H, Ar-H), 6.92-6.95(m, 1H, Ar-H), 7.18-7.25(m, 1H, Ar-H).

S3. Refinement

All H atoms were positioned geometrically and were treated as riding on their parent C/N atoms, with C—H distances of 0.93–0.98 Å, N—H distances of 0.86 Å and with $U_{iso}(H) = 1.2U_{eq}(C/N)$ or $1.5U_{eq}$(methyl C).
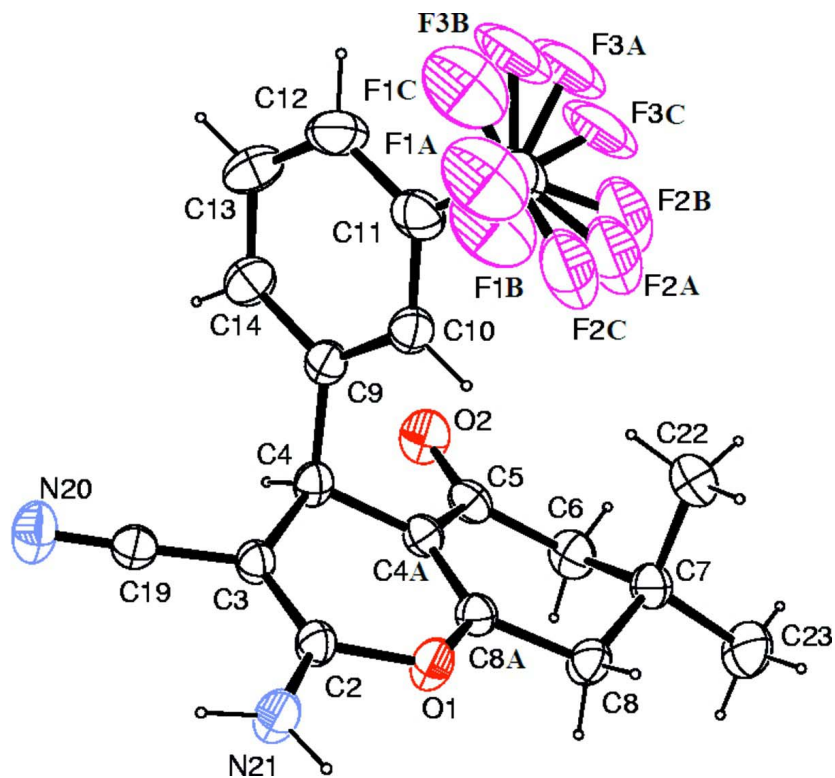
Figure 1

The molecular structure of the title compound with ellipsoids drawn at the 40% probability level. H atoms are shown as small spheres of arbitrary radii. The F atoms are disorded over three sets of sites.
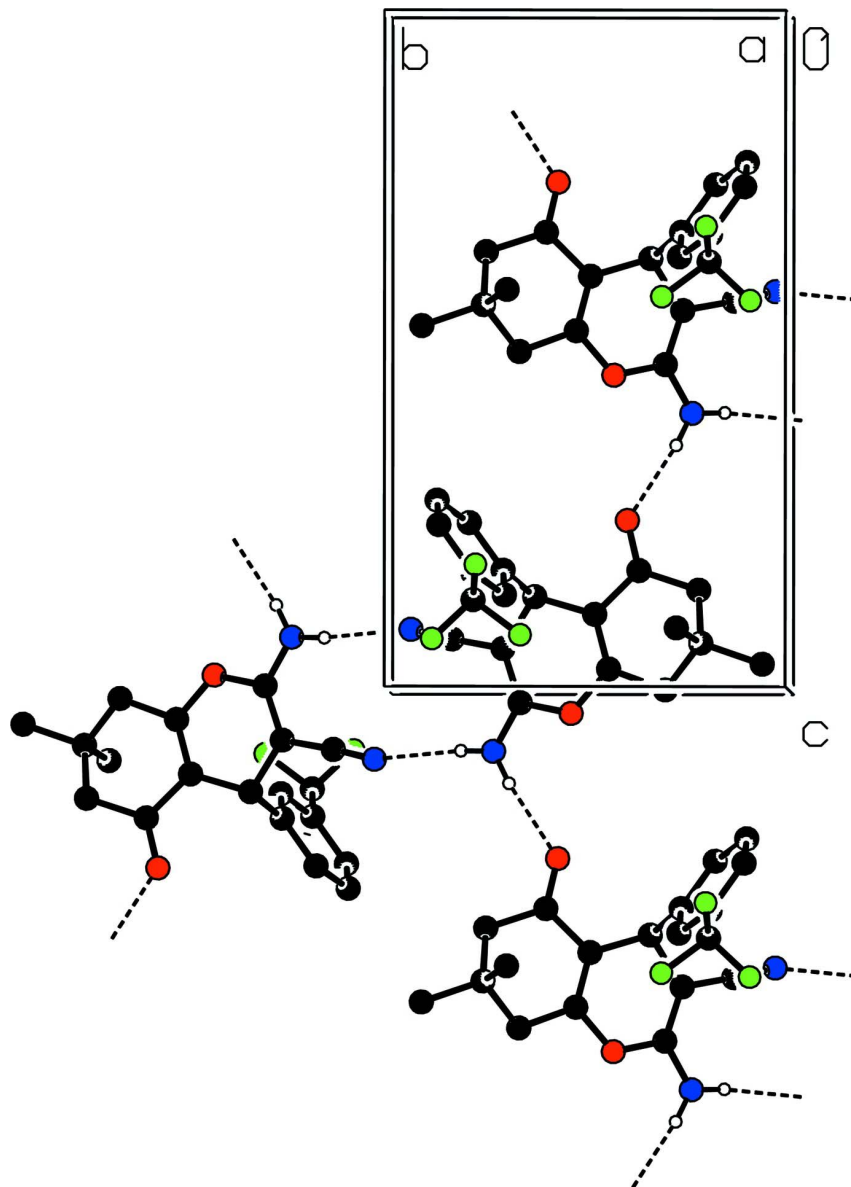
Figure 2

The packing arrangement of molecules viewed along the *a* axis. The dashed lines show intermolecular N—H···O and N—H···N hydrogen bonds. The disorder is not shown.

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$M_r = 362.35$	$Z = 8$
Monoclinic, $C2/c$	$F(000) = 1504$
Hall symbol: -C 2yc	$D_x = 1.363$ Mg m^{-3}
$a = 23.7543\,(6)$ Å	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$b = 9.3871\,(2)$ Å	Cell parameters from 18267 reflections
$c = 15.8857\,(4)$ Å	$\theta = 3.4$–$29.1°$
$\beta = 94.704\,(2)°$	$\mu = 0.11$ mm^{-1}

T = 293 K

Block, colorless

0.3 × 0.2 × 0.2 mm

Data collection

Oxford Diffraction Xcalibur Sapphire3 diffractometer	40937 measured reflections
Radiation source: fine-focus sealed tube	3467 independent reflections
Graphite monochromator	2538 reflections with $I > 2\sigma(I)$
Detector resolution: 16.1049 pixels mm^{-1}	R_{int} = 0.065
ω scans	θ_{max} = 26.0°, θ_{min} = 3.4°
Absorption correction: multi-scan (*CrysAlis PRO*; Oxford Diffraction, 2010)	$h = -29 \rightarrow 29$
	$k = -11 \rightarrow 11$
T_{min} = 0.766, T_{max} = 1.000	$l = -19 \rightarrow 19$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)]$ = 0.061	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.126	
S = 1.03	H-atom parameters constrained
3467 reflections	$w = 1/[\sigma^2(F_o^2) + (0.0389P)^2 + 5.1587P]$ where $P = (F_o^2 + 2F_c^2)/3$
258 parameters	
10 restraints	$(\Delta/\sigma)_{max}$ = 0.001
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max}$ = 0.26 e Å^{-3}
	$\Delta\rho_{min}$ = −0.32 e Å^{-3}

Special details

Experimental. CrysAlisPro, Oxford Diffraction Ltd., Version 1.171.34.40 (release 27-08-2010 CrysAlis171 .NET) (compiled Aug 27 2010,11:50:40) Empirical absorption correction using spherical harmonics, implemented in SCALE3 ABSPACK scaling algorithm.

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}	Occ. (<1)
O1	0.08902 (7)	0.55303 (15)	1.02901 (8)	0.0401 (4)	
C2	0.06233 (9)	0.6806 (2)	1.01297 (12)	0.0324 (5)	
O2	0.06995 (8)	0.41341 (18)	0.74350 (9)	0.0506 (5)	
C3	0.04766 (9)	0.7238 (2)	0.93258 (12)	0.0308 (5)	
C4	0.06583 (9)	0.6445 (2)	0.85626 (12)	0.0319 (5)	
H4	0.0335	0.6405	0.8138	0.038*	
C4A	0.08096 (9)	0.4945 (2)	0.88253 (12)	0.0313 (5)	
C5	0.08182 (9)	0.3842 (2)	0.81766 (13)	0.0358 (5)	
C6	0.09581 (11)	0.2348 (2)	0.84666 (15)	0.0431 (6)	
H6A	0.1117	0.1836	0.8011	0.052*	
H6B	0.0612	0.1869	0.8588	0.052*	

C7	0.13766 (10)	0.2287 (2)	0.92539 (14)	0.0394 (5)
C8	0.11327 (11)	0.3177 (2)	0.99442 (14)	0.0402 (6)
H8A	0.0821	0.2663	1.0161	0.048*
H8B	0.1421	0.3309	1.0407	0.048*
C8A	0.09300 (9)	0.4593 (2)	0.96338 (13)	0.0319 (5)
C9	0.11372 (10)	0.7223 (2)	0.81771 (12)	0.0337 (5)
C10	0.16874 (10)	0.7180 (2)	0.85477 (13)	0.0377 (5)
H10	0.1772	0.6617	0.9023	0.045*
C11	0.21092 (11)	0.7965 (3)	0.82186 (15)	0.0454 (6)
C12	0.19901 (13)	0.8810 (3)	0.75148 (17)	0.0575 (7)
H12	0.2275	0.9335	0.7292	0.069*
C13	0.14458 (14)	0.8865 (3)	0.71472 (16)	0.0636 (8)
H13	0.1360	0.9440	0.6677	0.076*
C14	0.10264 (11)	0.8071 (3)	0.74738 (14)	0.0498 (7)
H14	0.0661	0.8107	0.7214	0.060*
C15	0.26941 (13)	0.7941 (4)	0.8631 (2)	0.0643 (8)
C19	0.01734 (9)	0.8518 (2)	0.91848 (12)	0.0328 (5)
N20	−0.00772 (9)	0.9545 (2)	0.90387 (12)	0.0495 (6)
N21	0.05451 (9)	0.7470 (2)	1.08480 (11)	0.0446 (5)
H21A	0.0381	0.8288	1.0839	0.054*
H21B	0.0658	0.7083	1.1323	0.054*
C22	0.19433 (11)	0.2886 (3)	0.90370 (18)	0.0583 (7)
H22A	0.1887	0.3805	0.8779	0.088*
H22B	0.2109	0.2254	0.8652	0.088*
H22C	0.2190	0.2975	0.9544	0.088*
C23	0.14521 (13)	0.0752 (3)	0.95659 (17)	0.0592 (8)
H23A	0.1580	0.0171	0.9122	0.089*
H23B	0.1098	0.0395	0.9727	0.089*
H23C	0.1726	0.0727	1.0044	0.089*
F1A	0.2811 (5)	0.8968 (12)	0.9208 (6)	0.089 (2)	0.507 (7)
F2A	0.2806 (3)	0.6766 (7)	0.9151 (5)	0.0813 (17)	0.507 (7)
F3A	0.3113 (2)	0.7874 (11)	0.8116 (4)	0.091 (2)	0.507 (7)
F1B	0.2694 (8)	0.861 (2)	0.9364 (9)	0.089 (2)	0.330 (7)
F2B	0.2877 (5)	0.6587 (9)	0.8792 (8)	0.0813 (17)	0.330 (7)
F3B	0.3049 (4)	0.8668 (14)	0.8182 (7)	0.091 (2)	0.330 (7)
F1C	0.2904 (6)	0.9254 (11)	0.8551 (10)	0.089 (2)	0.163 (3)
F2C	0.2730 (6)	0.7466 (19)	0.9427 (6)	0.0813 (17)	0.163 (3)
F3C	0.2982 (6)	0.7011 (16)	0.8215 (10)	0.091 (2)	0.163 (3)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0604 (11)	0.0333 (8)	0.0262 (7)	0.0141 (7)	0.0006 (7)	−0.0023 (6)
C2	0.0373 (13)	0.0274 (10)	0.0326 (11)	0.0049 (9)	0.0042 (9)	−0.0011 (9)
O2	0.0679 (12)	0.0506 (10)	0.0325 (9)	0.0070 (9)	−0.0012 (8)	−0.0119 (8)
C3	0.0333 (12)	0.0291 (10)	0.0299 (11)	0.0049 (9)	0.0027 (8)	−0.0011 (8)
C4	0.0365 (13)	0.0338 (11)	0.0246 (10)	0.0056 (9)	−0.0024 (8)	−0.0015 (8)
C4A	0.0303 (12)	0.0325 (11)	0.0311 (11)	0.0007 (9)	0.0023 (8)	−0.0036 (9)

C5	0.0316 (12)	0.0403 (12)	0.0358 (12)	−0.0004 (10)	0.0042 (9)	−0.0079 (10)
C6	0.0495 (15)	0.0323 (12)	0.0480 (13)	−0.0001 (11)	0.0073 (11)	−0.0114 (10)
C7	0.0436 (14)	0.0307 (11)	0.0450 (13)	0.0061 (10)	0.0101 (10)	−0.0006 (10)
C8	0.0524 (15)	0.0311 (11)	0.0377 (12)	0.0061 (11)	0.0075 (10)	0.0026 (10)
C8A	0.0344 (13)	0.0288 (11)	0.0328 (11)	0.0009 (9)	0.0055 (9)	−0.0025 (9)
C9	0.0438 (14)	0.0323 (11)	0.0255 (10)	0.0066 (10)	0.0057 (9)	−0.0034 (9)
C10	0.0438 (14)	0.0376 (12)	0.0322 (11)	0.0059 (10)	0.0058 (10)	0.0014 (10)
C11	0.0484 (15)	0.0464 (14)	0.0431 (13)	0.0029 (12)	0.0151 (11)	−0.0079 (11)
C12	0.063 (2)	0.0597 (17)	0.0531 (16)	−0.0032 (14)	0.0267 (14)	0.0068 (13)
C13	0.078 (2)	0.0712 (19)	0.0429 (15)	0.0045 (16)	0.0142 (14)	0.0259 (14)
C14	0.0539 (17)	0.0607 (16)	0.0345 (13)	0.0068 (13)	0.0022 (11)	0.0116 (12)
C15	0.0492 (18)	0.077 (2)	0.0679 (19)	−0.0027 (16)	0.0137 (14)	0.0023 (17)
C19	0.0381 (13)	0.0354 (12)	0.0248 (10)	0.0012 (10)	0.0023 (9)	−0.0033 (9)
N20	0.0636 (15)	0.0412 (12)	0.0423 (11)	0.0166 (11)	−0.0040 (10)	−0.0042 (9)
N21	0.0708 (15)	0.0362 (10)	0.0270 (9)	0.0175 (10)	0.0058 (9)	−0.0005 (8)
C22	0.0380 (15)	0.0703 (18)	0.0675 (17)	0.0098 (14)	0.0094 (12)	0.0119 (15)
C23	0.080 (2)	0.0344 (13)	0.0647 (17)	0.0141 (13)	0.0142 (15)	−0.0008 (12)
F1A	0.061 (5)	0.119 (6)	0.085 (3)	−0.017 (3)	−0.006 (3)	−0.031 (5)
F2A	0.057 (3)	0.098 (3)	0.089 (5)	0.029 (2)	0.005 (3)	−0.004 (3)
F3A	0.0386 (17)	0.154 (7)	0.0855 (18)	0.008 (3)	0.0329 (12)	0.012 (4)
F1B	0.061 (5)	0.119 (6)	0.085 (3)	−0.017 (3)	−0.006 (3)	−0.031 (5)
F2B	0.057 (3)	0.098 (3)	0.089 (5)	0.029 (2)	0.005 (3)	−0.004 (3)
F3B	0.0386 (17)	0.154 (7)	0.0855 (18)	0.008 (3)	0.0329 (12)	0.012 (4)
F1C	0.061 (5)	0.119 (6)	0.085 (3)	−0.017 (3)	−0.006 (3)	−0.031 (5)
F2C	0.057 (3)	0.098 (3)	0.089 (5)	0.029 (2)	0.005 (3)	−0.004 (3)
F3C	0.0386 (17)	0.154 (7)	0.0855 (18)	0.008 (3)	0.0329 (12)	0.012 (4)

Geometric parameters (Å, º)

O1—C2	1.369 (2)	C11—C12	1.381 (4)
O1—C8A	1.373 (2)	C11—C15	1.487 (4)
C2—N21	1.327 (3)	C12—C13	1.375 (4)
C2—C3	1.358 (3)	C12—H12	0.9300
O2—C5	1.220 (3)	C13—C14	1.379 (4)
C3—C19	1.409 (3)	C13—H13	0.9300
C3—C4	1.515 (3)	C14—H14	0.9300
C4—C4A	1.504 (3)	C15—F3C	1.320 (8)
C4—C9	1.522 (3)	C15—F1B	1.321 (8)
C4—H4	0.9800	C15—F3B	1.336 (7)
C4A—C8A	1.334 (3)	C15—F2C	1.337 (8)
C4A—C5	1.462 (3)	C15—F3A	1.339 (5)
C5—C6	1.505 (3)	C15—F1C	1.340 (8)
C6—C7	1.534 (3)	C15—F1A	1.344 (5)
C6—H6A	0.9700	C15—F2B	1.360 (7)
C6—H6B	0.9700	C15—F2A	1.389 (5)
C7—C22	1.524 (3)	C19—N20	1.147 (3)
C7—C23	1.529 (3)	N21—H21A	0.8600
C7—C8	1.530 (3)	N21—H21B	0.8600

C8—C8A	1.484 (3)	C22—H22A	0.9600
C8—H8A	0.9700	C22—H22B	0.9600
C8—H8B	0.9700	C22—H22C	0.9600
C9—C14	1.379 (3)	C23—H23A	0.9600
C9—C10	1.389 (3)	C23—H23B	0.9600
C10—C11	1.380 (3)	C23—H23C	0.9600
C10—H10	0.9300		
C2—O1—C8A	118.60 (15)	C9—C14—C13	121.4 (2)
N21—C2—C3	128.71 (19)	C9—C14—H14	119.3
N21—C2—O1	110.26 (17)	C13—C14—H14	119.3
C3—C2—O1	121.03 (18)	F3C—C15—F1B	142.4 (12)
C2—C3—C19	119.51 (18)	F3C—C15—F3B	72.2 (8)
C2—C3—C4	122.53 (18)	F1B—C15—F3B	105.9 (9)
C19—C3—C4	117.84 (17)	F3C—C15—F2C	104.9 (10)
C4A—C4—C3	108.37 (16)	F3B—C15—F2C	132.9 (9)
C4A—C4—C9	113.05 (18)	F3C—C15—F3A	127.9 (8)
C3—C4—C9	110.95 (17)	F2C—C15—F3A	124.6 (7)
C4A—C4—H4	108.1	F3C—C15—F1C	110.4 (10)
C3—C4—H4	108.1	F1B—C15—F1C	71.1 (10)
C9—C4—H4	108.1	F2C—C15—F1C	113.6 (10)
C8A—C4A—C5	119.27 (19)	F3A—C15—F1C	71.6 (8)
C8A—C4A—C4	121.77 (18)	F3C—C15—F1A	136.9 (9)
C5—C4A—C4	118.96 (17)	F3B—C15—F1A	83.9 (6)
O2—C5—C4A	120.4 (2)	F2C—C15—F1A	66.4 (10)
O2—C5—C6	122.18 (19)	F3A—C15—F1A	109.1 (5)
C4A—C5—C6	117.37 (18)	F1B—C15—F2B	107.4 (12)
C5—C6—C7	113.39 (18)	F3B—C15—F2B	111.8 (7)
C5—C6—H6A	108.9	F2C—C15—F2B	61.1 (8)
C7—C6—H6A	108.9	F3A—C15—F2B	80.2 (5)
C5—C6—H6B	108.9	F1C—C15—F2B	139.6 (8)
C7—C6—H6B	108.9	F1A—C15—F2B	119.8 (9)
H6A—C6—H6B	107.7	F3C—C15—F2A	72.0 (8)
C22—C7—C23	109.8 (2)	F1B—C15—F2A	82.3 (11)
C22—C7—C8	110.7 (2)	F3B—C15—F2A	128.6 (6)
C23—C7—C8	108.87 (19)	F3A—C15—F2A	102.1 (4)
C22—C7—C6	109.1 (2)	F1C—C15—F2A	137.0 (7)
C23—C7—C6	110.5 (2)	F1A—C15—F2A	98.3 (7)
C8—C7—C6	107.79 (19)	F3C—C15—C11	107.1 (7)
C8A—C8—C7	112.50 (18)	F1B—C15—C11	108.2 (9)
C8A—C8—H8A	109.1	F3B—C15—C11	111.4 (6)
C7—C8—H8A	109.1	F2C—C15—C11	114.0 (7)
C8A—C8—H8B	109.1	F3A—C15—C11	116.4 (4)
C7—C8—H8B	109.1	F1C—C15—C11	106.6 (6)
H8A—C8—H8B	107.8	F1A—C15—C11	115.0 (6)
C4A—C8A—O1	123.33 (18)	F2B—C15—C11	111.7 (6)
C4A—C8A—C8	125.43 (19)	F2A—C15—C11	113.6 (4)
O1—C8A—C8	111.23 (17)	N20—C19—C3	177.4 (2)

C14—C9—C10	118.1 (2)	C2—N21—H21A	120.0
C14—C9—C4	120.2 (2)	C2—N21—H21B	120.0
C10—C9—C4	121.58 (18)	H21A—N21—H21B	120.0
C11—C10—C9	120.7 (2)	C7—C22—H22A	109.5
C11—C10—H10	119.6	C7—C22—H22B	109.5
C9—C10—H10	119.6	H22A—C22—H22B	109.5
C10—C11—C12	120.4 (2)	C7—C22—H22C	109.5
C10—C11—C15	120.4 (2)	H22A—C22—H22C	109.5
C12—C11—C15	119.2 (2)	H22B—C22—H22C	109.5
C13—C12—C11	119.2 (2)	C7—C23—H23A	109.5
C13—C12—H12	120.4	C7—C23—H23B	109.5
C11—C12—H12	120.4	H23A—C23—H23B	109.5
C12—C13—C14	120.2 (2)	C7—C23—H23C	109.5
C12—C13—H13	119.9	H23A—C23—H23C	109.5
C14—C13—H13	119.9	H23B—C23—H23C	109.5
C8A—O1—C2—N21	170.12 (19)	C7—C8—C8A—O1	−159.98 (19)
C8A—O1—C2—C3	−9.9 (3)	C4A—C4—C9—C14	138.3 (2)
N21—C2—C3—C19	−3.7 (4)	C3—C4—C9—C14	−99.8 (2)
O1—C2—C3—C19	176.4 (2)	C4A—C4—C9—C10	−46.0 (3)
N21—C2—C3—C4	172.3 (2)	C3—C4—C9—C10	76.0 (2)
O1—C2—C3—C4	−7.7 (3)	C14—C9—C10—C11	0.0 (3)
C2—C3—C4—C4A	21.0 (3)	C4—C9—C10—C11	−175.90 (19)
C19—C3—C4—C4A	−163.03 (19)	C9—C10—C11—C12	0.2 (3)
C2—C3—C4—C9	−103.7 (2)	C9—C10—C11—C15	178.5 (2)
C19—C3—C4—C9	72.3 (2)	C10—C11—C12—C13	0.3 (4)
C3—C4—C4A—C8A	−19.2 (3)	C15—C11—C12—C13	−178.1 (3)
C9—C4—C4A—C8A	104.2 (2)	C11—C12—C13—C14	−0.8 (4)
C3—C4—C4A—C5	160.06 (18)	C10—C9—C14—C13	−0.5 (4)
C9—C4—C4A—C5	−76.5 (2)	C4—C9—C14—C13	175.4 (2)
C8A—C4A—C5—O2	178.7 (2)	C12—C13—C14—C9	0.9 (4)
C4—C4A—C5—O2	−0.7 (3)	C10—C11—C15—F3C	97.7 (9)
C8A—C4A—C5—C6	0.7 (3)	C12—C11—C15—F3C	−83.9 (9)
C4—C4A—C5—C6	−178.6 (2)	C10—C11—C15—F1B	−69.1 (11)
O2—C5—C6—C7	149.3 (2)	C12—C11—C15—F1B	109.3 (10)
C4A—C5—C6—C7	−32.8 (3)	C10—C11—C15—F3B	174.8 (7)
C5—C6—C7—C22	−65.0 (3)	C12—C11—C15—F3B	−6.8 (8)
C5—C6—C7—C23	174.1 (2)	C10—C11—C15—F2C	−17.9 (10)
C5—C6—C7—C8	55.3 (3)	C12—C11—C15—F2C	160.5 (9)
C22—C7—C8—C8A	71.6 (3)	C10—C11—C15—F3A	138.6 (6)
C23—C7—C8—C8A	−167.6 (2)	C12—C11—C15—F3A	−43.0 (6)
C6—C7—C8—C8A	−47.7 (3)	C10—C11—C15—F1C	−144.1 (8)
C5—C4A—C8A—O1	−174.82 (19)	C12—C11—C15—F1C	34.3 (9)
C4—C4A—C8A—O1	4.5 (3)	C10—C11—C15—F1A	−91.9 (6)
C5—C4A—C8A—C8	6.5 (3)	C12—C11—C15—F1A	86.5 (6)
C4—C4A—C8A—C8	−174.2 (2)	C10—C11—C15—F2B	49.0 (7)
C2—O1—C8A—C4A	11.7 (3)	C12—C11—C15—F2B	−132.6 (6)
C2—O1—C8A—C8	−169.41 (19)	C10—C11—C15—F2A	20.3 (5)

C7—C8—C8A—C4A	18.8 (3)	C12—C11—C15—F2A	−161.3 (4)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N21—H21*A*···N20ⁱ	0.86	2.17	3.025 (3)	171
N21—H21*B*···O2ⁱⁱ	0.86	2.10	2.934 (2)	163

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