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Cytenamide trifluoroacetic acid solvate

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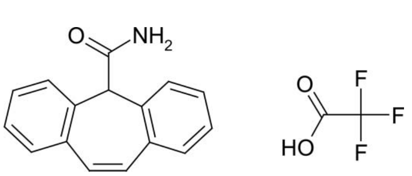
Key indicators: single-crystal X-ray study; T = 160 K; mean σ(C–C) = 0.006 Å; disorder in solvent or counterion; R factor = 0.080; wR factor = 0.178; data-to-parameter ratio = 12.6.

Cytenamide forms a 1:1 solvate with trifluoroacetic acid (systematic name: 5*H*-dibenzo[*a*,*d*]cycloheptatriene-5-carboxamide trifluoroacetic acid solvate), $C_{16}H_{13}NO\cdot C_2HF_3O_2$. The compound crystallizes with one molecule of cytenamide and one of trifluoroacetic acid in the asymmetric unit; these are linked by O—H···O and N—H···O hydrogen bonds to form an $R_2^2(8)$ motif. The trifluoromethyl group of the solvent molecule displays rotational disorder over two sites, with site-occupancy factors of 0.964 (4) and 0.036 (4).

Related literature

For details on the experimental methods used to obtain this form, see: Davis *et al.* (1964); Florence *et al.* (2003); Florence, Johnston, Fernandes *et al.* (2006). For literature on carbamazepine and other related structures, see: Cyr *et al.* (1987); Fleischman *et al.* (2003); Florence, Johnston, Price *et al.* (2006); Florence, Leech *et al.* (2006); Bandoli *et al.* (1992); Harrison *et al.* (2006); Leech *et al.* (2007); Florence, Bedford *et al.* (2008); Florence, Shankland *et al.* (2008); Fernandes *et al.* (2007). For hydrogen-bond motifs, see: Etter (1990); Bernstein *et al.* (1995).



Experimental

Crystal data

$C_{16}H_{13}NO\cdot C_2HF_3O_2$	V = 1614.9 (2) Å^3
M_r = 349.31	Z = 4
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation
a = 12.1673 (11) Å	μ = 0.12 mm^{-1}
b = 6.3235 (6) Å	T = 160 K
c = 21.4525 (15) Å	0.16 × 0.13 × 0.08 mm
β = 101.932 (8)°	

Data collection

Oxford Diffraction Gemini S diffractometer	10796 measured reflections
Absorption correction: multi-scan (*ABSPACK*/*CrysAlis RED*; Oxford Diffraction, 2006)	2995 independent reflections
T_{min} = 0.83, T_{max} = 0.99	1423 reflections with $I > 2\sigma(I)$
	R_{int} = 0.094

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.080	24 restraints
$wR(F^2)$ = 0.178	H-atom parameters not refined
S = 1.04	$\Delta\rho_{max}$ = 0.73 e Å^{-3}
2984 reflections	$\Delta\rho_{min}$ = −0.60 e Å^{-3}
236 parameters	

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

D—H···A	D—H	H···A	D···A	D—H···A
O3—H5···O2^{i}	0.89	1.58	2.462 (4)	173
N1—H11···O1^{ii}	0.86	2.23	2.976 (4)	144
N1—H12···O1^{iii}	0.87	2.16	2.982 (5)	159

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

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEP-3* (Farrugia, 1997) and *Mercury* (Macrae *et al.*, 2006); software used to prepare material for publication: *publCIF* (Westrip, 2008) and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CF2202).

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Cytenamide trifluoroacetic acid solvate

Andrea Johnston, Alastair J. Florence, Francesca J. A. Fabbiani, Kenneth Shankland, Colin T. Bedford and Julie Bardin

S1. Comment

Cytenamide (CYT) is an analogue of carbamazepine (CBZ), a dibenzazepine drug used to control seizures (Cyr *et al.*, 1987). Cytenamide-trifluoroacetic acid solvate (CYT-TFAA) was produced during an automated parallel crystallization study (Florence, Johnston, Fernandes *et al.*, 2006) of CYT as part of a wider investigation that couples automated parallel crystallization with crystal structure prediction methodology to investigate the basic science underlying the solid-state diversity of CBZ (Florence, Johnston, Price *et al.*, 2006; Florence, Leech *et al.*, 2006) and its closely related analogues: CYT (Florence, Bedford *et al.,* 2008), 10,11-dihydrocarbamazepine (Bandoli *et al.*, 1992; Harrison *et al.*, 2006; Leech *et al.*, 2007) and cyheptamide (Florence, Shankland *et al.*, 2008). The sample was identified as a new form using multi-sample foil transmission X-ray powder diffraction analysis (Florence *et al.*, 2003). Subsequent manual recrystallization from a saturated TFAA solution by slow evaporation at 278 K yielded a sample suitable for single-crystal X-ray diffraction (Fig. 1).

The compound crystallizes in space group $P2_1/n$ with one molecule of CBZ and one molecule of TFAA in the asymmetric unit. As in the structure of CBZ-TFAA solvate (Fernandes *et al.*, 2007) the solvent molecule displays rotational disorder and the fluorine atoms were refined over two sites yielding site occupancy factors 0.964 (4), 0.036 (4) and 0.53 (1), 0.47 (1) for CYT-TFAA and CBZ-TFAA respectively. The molecules also adopt a hydrogen-bonded arrangement similar to that observed in CBZ-TFAA solvate whereby the amide group of each CYT molecule is connected to the carboxylic acid group of a TFAA molecule by N–H···O and O–H···O contacts (Table 1) to form an $R_2^2(8)$ hydrogen-bonded motif (Etter, 1990; Bernstein *et al.*, 1995). CYT also forms a second N—H···O contact with an adjacent solvent molecule to form a chain extending along the [010] direction.

S2. Experimental

A sample of cytenamide was synthesized according to a modification of the published method (Davis *et al.*, 1964). A single-crystal sample of cytenamide-TFAA was grown from a saturated TFAA solution by isothermal solvent evaporation at 278 K.

S3. Refinement

Owing to the weak scattering, data were integrated applying a theta cut off of 25°. All non-hydrogen atoms were modelled with anisotropic displacement parameters with the exception of the minor component of the disordered site in the TFAA molecule, for which one common isotropic displacement parameter was calculated and fixed during refinement. Bond-length restraints were applied to C—F bond lengths involving atoms F1, F4, F5 and F6. 3-Fold symmetry was imposed on the disordered minor site of the the TFAA molecule by the use of restraints. H-atoms were found in a difference Fourier map and were initially refined with soft restraints on the bond lengths and angles to regularize their geometry and U_{iso}(H) (in the range 1.2–1.5 times U_{eq} of the parent atom), after which the positions were fixed. Eleven reflections were suppressed as outliers in an analysis of the data.

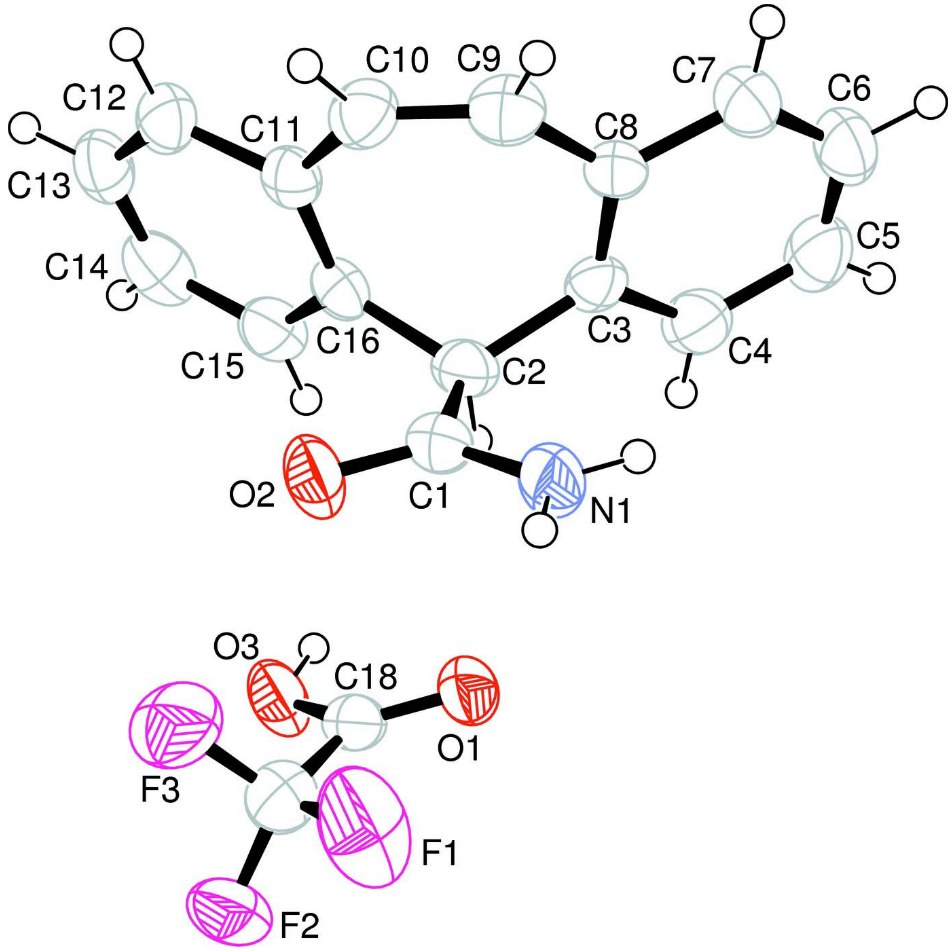

Figure 1

The molecular structure of CYT-TFAA, showing 50% probablility displacement ellipsoids. The lower occupancy fluorine atoms have beem omitted for clarity.

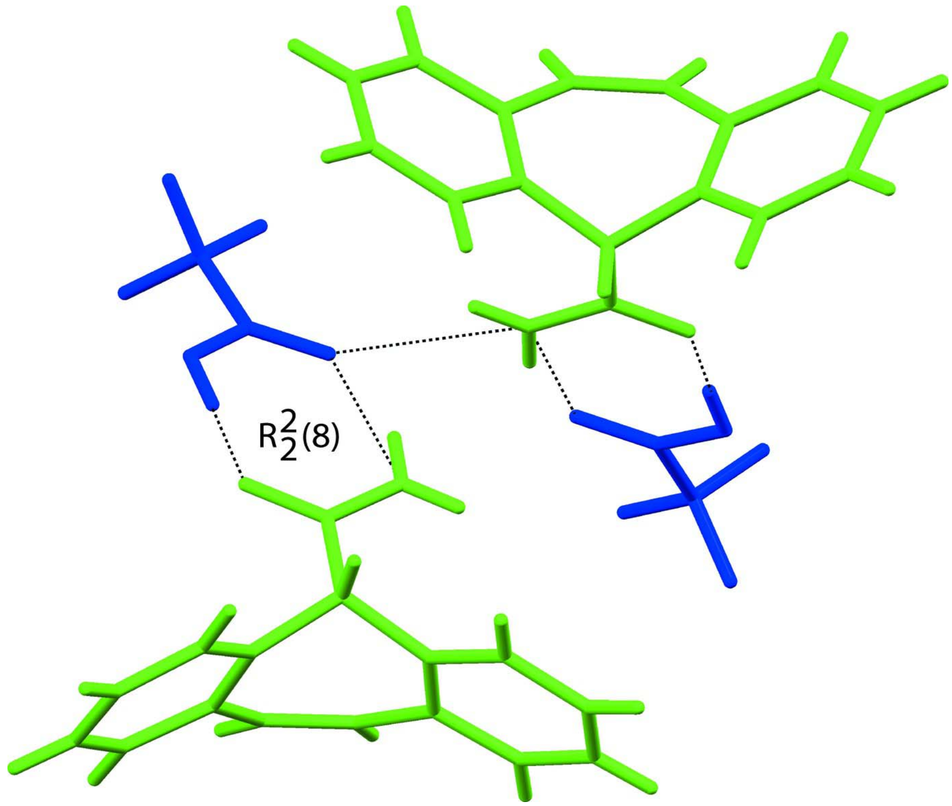

Figure 2

Hydrogen-bonded contacts in CYT-TFAA, showing the adjacent $R_2^2(8)$ CYT-TFAA units further linked by an N—H···O interaction. Minor ocupancy components have been omitted for clarity. CYT and TFAA molecules are coloured according to symmetry equivalence (green and blue respectively) and hydrogen bonds are shown as dashed lines.

5*H*-dibenzo[*a*,*d*]cycloheptatriene-5-carboxamide trifluoroacetic acid solvate

Crystal data

$C_{16}H_{13}NO·C_2HF_3O_2$
M_r = 349.31
Monoclinic, $P2_1/n$
Hall symbol: -P 2yn
a = 12.1673 (11) Å
b = 6.3235 (6) Å
c = 21.4525 (15) Å
β = 101.932 (8)°
V = 1614.9 (2) Å^3
Z = 4
$F(000)$ = 720
D_x = 1.437 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 1137 reflections
θ = 3–25°
μ = 0.12 mm^{-1}
T = 160 K
Block, colourless
0.16 × 0.13 × 0.08 mm

Data collection

Oxford Diffraction Gemini S diffractometer
Radiation source: Enhance (Mo) X-ray Source
Graphite monochromator
Detector resolution: 15.9745 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (ABSPACK/*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.83, T_{max} = 0.99
10796 measured reflections
2995 independent reflections
1423 reflections with $I > 2\sigma(I)$

R_{int} = 0.094
θ_{max} = 25.5°, θ_{min} = 3.1°
h = −14→14
k = 0→7
l = 0→25

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.080
$wR(F^2)$ = 0.178
S = 1.05
2984 reflections
236 parameters
24 restraints
Primary atom site location: structure-invariant direct methods
Hydrogen site location: inferred from neighbouring sites
H-atom parameters not refined
Method = Modified Sheldrick $w = 1/[\sigma^2(F^2) + (0.06P)^2 + 0.42P]$, where $P = (\max(F_o^2,0) + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.000413
$\Delta\rho_{max}$ = 0.73 e Å^{-3}
$\Delta\rho_{min}$ = −0.60 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
C1	0.6284 (3)	0.6720 (7)	0.3296 (2)	0.0461	
C2	0.5488 (4)	0.4854 (7)	0.31163 (18)	0.0431	
C3	0.4806 (3)	0.4971 (7)	0.24379 (18)	0.0380	
C4	0.4965 (4)	0.3481 (7)	0.1996 (2)	0.0507	
C5	0.4406 (4)	0.3618 (8)	0.1366 (2)	0.0578	
C6	0.3726 (4)	0.5332 (9)	0.1180 (2)	0.0576	
C7	0.3553 (4)	0.6826 (8)	0.16104 (19)	0.0519	
C8	0.4077 (3)	0.6646 (7)	0.22567 (17)	0.0393	
C9	0.3794 (4)	0.8214 (7)	0.2691 (2)	0.0475	
C10	0.3773 (3)	0.7968 (7)	0.33147 (19)	0.0456	
C11	0.4028 (3)	0.6095 (7)	0.37105 (18)	0.0369	
C12	0.3488 (4)	0.5854 (7)	0.42250 (19)	0.0470	
C13	0.3646 (4)	0.4094 (8)	0.45999 (18)	0.0515	
C14	0.4337 (4)	0.2524 (8)	0.4474 (2)	0.0570	
C15	0.4912 (4)	0.2755 (7)	0.3988 (2)	0.0497	
C16	0.4774 (3)	0.4536 (7)	0.36104 (17)	0.0374	
C17	0.8909 (4)	0.3112 (8)	0.4387 (2)	0.0572	
C18	0.8197 (3)	0.1386 (7)	0.4008 (2)	0.0461	
N1	0.6679 (3)	0.7697 (6)	0.28446 (15)	0.0533	
O1	0.7936 (2)	0.1490 (5)	0.34338 (13)	0.0564	
O2	0.6593 (2)	0.7215 (5)	0.38684 (12)	0.0591	
O3	0.7904 (3)	−0.0019 (5)	0.43735 (12)	0.0618	
F1	0.9217 (4)	0.4524 (6)	0.40116 (15)	0.1040	0.964 (4)
F2	0.9828 (2)	0.2331 (5)	0.47540 (13)	0.0714	0.964 (4)
F3	0.8361 (3)	0.4082 (5)	0.47792 (16)	0.0845	0.964 (4)
F4	0.846 (3)	0.502 (2)	0.429 (3)	0.0800*	0.036 (4)
F5	0.992 (2)	0.330 (8)	0.425 (3)	0.0800*	0.036 (4)
F6	0.910 (5)	0.282 (6)	0.5010 (4)	0.0800*	0.036 (4)
H11	0.6461	0.7332	0.2450	0.0619*	
H12	0.7150	0.8730	0.2945	0.0619*	
H21	0.5969	0.3601	0.3139	0.0495*	

H41	0.5449	0.2331	0.2125	0.0596*
H51	0.4499	0.2559	0.1077	0.0680*
H61	0.3386	0.5487	0.0754	0.0651*
H71	0.3068	0.7953	0.1473	0.0581*
H91	0.3579	0.9563	0.2521	0.0539*
H101	0.3556	0.9169	0.3516	0.0541*
H121	0.3022	0.6959	0.4312	0.0539*
H131	0.3278	0.3947	0.4943	0.0619*
H141	0.4423	0.1260	0.4713	0.0647*
H151	0.5414	0.1676	0.3914	0.0557*
H5	0.7404	−0.0935	0.4169	0.0888*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.033 (2)	0.065 (3)	0.040 (3)	−0.002 (2)	0.005 (2)	0.003 (2)
C2	0.036 (2)	0.047 (3)	0.044 (2)	0.008 (2)	0.002 (2)	−0.006 (2)
C3	0.028 (2)	0.046 (3)	0.041 (2)	−0.007 (2)	0.0095 (19)	−0.007 (2)
C4	0.044 (3)	0.056 (3)	0.052 (3)	0.000 (2)	0.011 (2)	−0.009 (2)
C5	0.056 (3)	0.072 (4)	0.048 (3)	−0.012 (3)	0.015 (2)	−0.021 (3)
C6	0.053 (3)	0.081 (4)	0.036 (3)	−0.011 (3)	0.002 (2)	−0.003 (3)
C7	0.044 (3)	0.070 (3)	0.041 (3)	−0.004 (2)	0.004 (2)	0.002 (3)
C8	0.030 (2)	0.049 (3)	0.038 (2)	−0.003 (2)	0.0052 (19)	0.000 (2)
C9	0.047 (3)	0.041 (3)	0.055 (3)	0.002 (2)	0.009 (2)	−0.001 (2)
C10	0.047 (3)	0.045 (3)	0.047 (3)	0.003 (2)	0.013 (2)	−0.006 (2)
C11	0.036 (2)	0.037 (3)	0.035 (2)	−0.006 (2)	0.0022 (19)	−0.006 (2)
C12	0.047 (3)	0.056 (3)	0.038 (2)	−0.005 (2)	0.008 (2)	−0.011 (2)
C13	0.050 (3)	0.069 (4)	0.035 (2)	−0.014 (3)	0.007 (2)	0.006 (3)
C14	0.058 (3)	0.057 (3)	0.050 (3)	−0.010 (3)	−0.004 (2)	0.011 (3)
C15	0.043 (3)	0.055 (3)	0.048 (3)	−0.003 (2)	0.001 (2)	0.002 (2)
C16	0.040 (3)	0.036 (3)	0.032 (2)	−0.002 (2)	−0.0011 (19)	−0.003 (2)
C17	0.054 (3)	0.064 (4)	0.055 (3)	−0.003 (3)	0.014 (3)	−0.002 (3)
C18	0.034 (3)	0.060 (3)	0.044 (3)	0.002 (2)	0.007 (2)	0.008 (3)
N1	0.043 (2)	0.078 (3)	0.038 (2)	−0.017 (2)	0.0045 (17)	−0.009 (2)
O1	0.0488 (19)	0.082 (2)	0.0375 (17)	−0.0082 (17)	0.0064 (14)	0.0091 (17)
O2	0.054 (2)	0.090 (3)	0.0300 (17)	−0.0283 (18)	0.0024 (14)	−0.0002 (16)
O3	0.060 (2)	0.085 (2)	0.0362 (17)	−0.0297 (19)	−0.0009 (15)	0.0067 (17)
F1	0.132 (3)	0.101 (3)	0.074 (2)	−0.059 (3)	0.008 (2)	0.020 (2)
F2	0.0448 (18)	0.091 (2)	0.0715 (19)	−0.0066 (16)	−0.0045 (14)	−0.0160 (17)
F3	0.075 (2)	0.086 (2)	0.095 (2)	0.0090 (19)	0.0230 (19)	−0.027 (2)

Geometric parameters (Å, º)

C1—C2	1.525 (6)	C11—C16	1.386 (5)
C1—N1	1.321 (5)	C12—C13	1.363 (6)
C1—O2	1.247 (4)	C12—H121	0.942
C2—C3	1.521 (5)	C13—C14	1.364 (6)
C2—C16	1.517 (6)	C13—H131	0.942

C2—H21	0.980	C14—C15	1.378 (6)
C3—C4	1.379 (6)	C14—H141	0.945
C3—C8	1.384 (5)	C15—C16	1.376 (5)
C4—C5	1.383 (6)	C15—H151	0.951
C4—H41	0.941	C17—C18	1.520 (5)
C5—C6	1.372 (7)	C17—F1	1.307 (4)
C5—H51	0.936	C17—F2	1.322 (5)
C6—C7	1.368 (6)	C17—F3	1.328 (5)
C6—H61	0.928	C17—C18	1.520 (5)
C7—C8	1.406 (5)	C17—F4	1.323 (7)
C7—H71	0.934	C17—F5	1.323 (7)
C8—C9	1.451 (5)	C17—F6	1.323 (7)
C9—C10	1.352 (5)	C18—O1	1.209 (4)
C9—H91	0.944	C18—O3	1.283 (5)
C10—C11	1.453 (6)	N1—H11	0.865
C10—H101	0.938	N1—H12	0.867
C11—C12	1.405 (5)	O3—H5	0.887

C2—C1—N1	119.0 (4)	C12—C11—C16	118.2 (4)
C2—C1—O2	119.3 (4)	C11—C12—C13	121.4 (4)
N1—C1—O2	121.6 (4)	C11—C12—H121	118.3
C1—C2—C3	113.4 (4)	C13—C12—H121	120.4
C1—C2—C16	110.5 (3)	C12—C13—C14	119.6 (4)
C3—C2—C16	113.4 (3)	C12—C13—H131	120.7
C1—C2—H21	105.8	C14—C13—H131	119.7
C3—C2—H21	106.9	C13—C14—C15	120.2 (4)
C16—C2—H21	106.2	C13—C14—H141	120.7
C2—C3—C4	119.9 (4)	C15—C14—H141	119.0
C2—C3—C8	119.8 (4)	C14—C15—C16	120.8 (4)
C4—C3—C8	120.2 (4)	C14—C15—H151	119.6
C3—C4—C5	121.2 (4)	C16—C15—H151	119.6
C3—C4—H41	119.6	C2—C16—C11	120.1 (4)
C5—C4—H41	119.2	C2—C16—C15	120.1 (4)
C4—C5—C6	118.6 (4)	C11—C16—C15	119.7 (4)
C4—C5—H51	120.0	C18—C17—F1	111.5 (4)
C6—C5—H51	121.3	C18—C17—F2	111.6 (4)
C5—C6—C7	121.1 (4)	F1—C17—F2	107.9 (4)
C5—C6—H61	119.4	C18—C17—F3	111.4 (4)
C7—C6—H61	119.5	F1—C17—F3	108.7 (4)
C6—C7—C8	120.6 (4)	F2—C17—F3	105.6 (4)
C6—C7—H71	119.3	C18—C17—F4	113.56 (6)
C8—C7—H71	120.1	C18—C17—F5	113.57 (6)
C7—C8—C3	118.2 (4)	F4—C17—F5	105.08 (7)
C7—C8—C9	117.4 (4)	C18—C17—F6	113.57 (6)
C3—C8—C9	124.4 (4)	F4—C17—F6	105.08 (7)
C8—C9—C10	127.8 (4)	F5—C17—F6	105.08 (7)
C8—C9—H91	116.8	C17—C18—O1	120.4 (4)
C10—C9—H91	115.3	C17—C18—O3	111.8 (4)

C9—C10—C11	128.8 (4)	O1—C18—O3	127.8 (4)
C9—C10—H101	115.3	C1—N1—H11	120.6
C11—C10—H101	115.9	C1—N1—H12	119.5
C10—C11—C12	118.0 (4)	H11—N1—H12	119.9
C10—C11—C16	123.9 (4)	C18—O3—H5	113.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O3—H5···O2^{i}	0.89	1.58	2.462 (4)	173
N1—H11···O1^{ii}	0.86	2.23	2.976 (4)	144
N1—H12···O1^{iii}	0.87	2.16	2.982 (5)	159

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