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1-(4-Chlorophenyl)-8-(4-fluorophenyl)-4-[(*E*)-(4-fluorophenyl)methylidene]-6-methyl-4,5,6,7,7a,8-hexahydro-1,2,4-oxadiazolo[5,4-*d*]pyrido[3,4-*c*][1,5]-benzothiazepine

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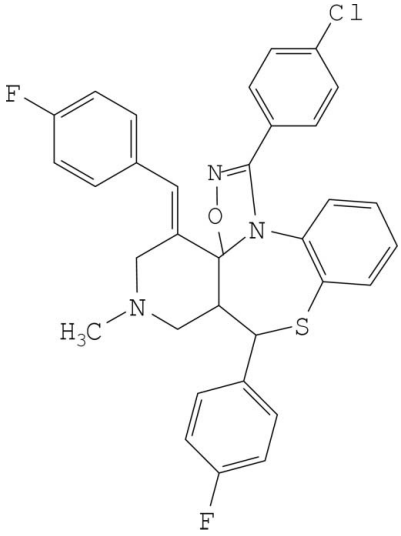
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.045; wR factor = 0.128; data-to-parameter ratio = 22.7.

In the title compound, $C_{33}H_{26}ClF_2N_3OS$, the oxadiazole, piperidine and benzothiazepine rings adopt envelope, chair and twist-boat conformations, respectively. The molecular aggregation in the crystal is characterized by linear chains of centrosymmetrically related pairs of molecules connected through C—H···F and C—H···N hydrogen bonds.

Related literature

For biological properties of related compounds, see: Budriesi *et al.* (2007); Şahin *et al.* (2002). For conformational analysis of ring systems, see: Cremer & Pople (1975); Boeyens (1978).



Experimental

Crystal data

$C_{33}H_{26}ClF_2N_3OS$
M_r = 586.08
Monoclinic, $P2_1/n$
a = 10.836 (5) Å
b = 11.366 (4) Å
c = 23.100 (3) Å
β = 92.63 (1)°
V = 2842.0 (17) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 298 (2) K
0.30 × 0.20 × 0.20 mm

Data collection

Bruker Kappa-APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.93, T_{max} = 0.96
36502 measured reflections
8394 independent reflections
5384 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.045
$wR(F^2)$ = 0.128
S = 1.01
8394 reflections
370 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.34 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
C61—H61*C*···F2^{i}	0.96	2.50	3.435 (3)	166
C83—H83···N2^{ii}	0.93	2.61	3.488 (3)	157

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

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *SAINT-Plus* (Bruker–Nonius, 2004); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2135).

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supplementary materials

Acta Cryst. (2007). E**63**, o4268 [**doi:10.1107/S1600536807048465**]

1-(4-Chlorophenyl)-8-(4-fluorophenyl)-4-[(*E*)-(4-fluorophenyl)methylidene]-6-methyl-4,5,6,7,7a,8-hexahydro-1,2,4-oxadiazolo[5,4-*d*]pyrido[3,4-*c*][1,5]benzothiazepine

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Comment

The title compound, $C_{33}H_{26}N_3OF_2SCl$, belongs to an important class of heterocycles which are expected to possess anti-hypertensive properties, and is made of three fused ring systems *viz*. a benzothiazepine, an oxadiazole and a methyl piperidine ring. Interestingly, benzothiazepines have been established as a class of calcium channel blockers (Budriesi *et al.*, 2007), oxadiazole derivatives have been found to possess antimicrobial activity (Şahin *et al.*, 2002) and piperidine ring occurs in many natural alkaloids and are constituents of a number of pharmaceutical drugs. Also, the role of non-conventional hydrogen bonds *viz*. C—H···*X* (*X*= N, O, Cl, F, *etc*.) can be accurately assessed through precise single-crystal structure determinations of these compounds which, in turn, may provide insights into design of drugs with a wide range of biological activities.

Least-squares plane calculations show that the 4-fluorophenyl attached to thiazepine, 4-flurophenyl attached to piperidine and 4-chlorophenyl rings make dihedral angles of 40.0 (1), 67.6 (1) and 71.5 (1)°, respectively, with respect to the benzene fused to the thiazepine ring. The torsion angles about the methylidene bond C4—C40—C41—C42 = −39.7 (3)° and C4—C40—C41—C46 = 144.01 (19)° indicate a significant twist of the 4-fluorophenyl ring which may be attributed to steric factors. The oxadiazole, piperidine and benzothiazepine rings adopt the envelope [Q = 0.299 (2) Å, φ = 150.5 (3)°] (Cremer & Pople, 1975), chair [Q = 0.597 (2) Å, θ = 1.6 (2)°, φ = 251 (5)°] (Boeyens, 1978) and twist-boat conformations, respectively. The molecular aggregation is characterized by linear chains of centrosymmetrically related pairs extending along the [010] axis, connected through a C—H···F and a C—H···N bonds (Table 1). The interactions between these linear chains are purely van der Waal's in nature. No significant π···π and C—H···π interactions were found in the crystal structure.

Experimental

2-Methyl-11-(4-fluorophenyl)-4-[(*E*)-(4-fluorophenyl)methylidene]- 1,2,3,4,11,11*a*-hexahydro-pyrido[3,4-*c*][1,5]benzothiazepine (1 mmol) and 4-chloro-*N*-hydroxybenzenecarboxymidoyl chloride (1 mmol) were dissolved in benzene (15 ml). Triethylamine (1 mmol) was added to the above mixture and refluxed for 30 min. After completion of the reaction the triethylamine hydrochloride was filtered off, solvent evaporated, and the product was purified by column chromatography using petroleum ether:ethyl acetate (90:10 *v*/*v*) mixture and finally recrystallized from ethyl acetate to obtain pure 1-(4-chlorophenyl)-8-(4-fluorophenyl)-4-[(*E*)-(4-fluorophenyl)methylidene]- 6-methyl-4,5,6,7,7a,8-hexahydro[1,2,4]oxadiazolo[5,4-*d*]pyrido[3,4-*c*][1,5] benzothiazepine as colourless crystals.

Refinement

H atoms were placed geometrically and refined using a riding model with C—H = 0.95–0.99 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

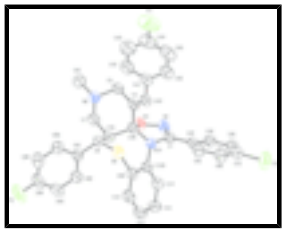

Fig. 1. The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids. H atoms have been omitted for clarity.

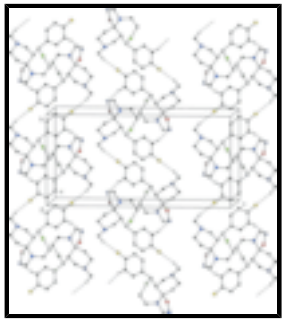

Fig. 2. A view of the molecular aggregation down the [100] axis. Ring systems and H atoms that are not involved in hydrogen bonding have been omitted for clarity.

1-(4-Chlorophenyl)-8-(4-fluorophenyl)-4-[(*E*)-(4- fluorophenyl)methylidene]-6-methyl-4,5,6,7,7a,8-hexahydro-1,2,4-oxadiazolo[5,4-*d*]pyrido[3,4-*c*][1,5]benzothiazepine

Crystal data

$C_{33}H_{26}ClF_2N_3OS$	F_{000} = 1216
M_r = 586.08	D_x = 1.370 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 5122 reflections
a = 10.836 (5) Å	θ = 2.0–30.0º
b = 11.366 (4) Å	μ = 0.25 mm^{-1}
c = 23.100 (3) Å	T = 298 (2) K
β = 92.63 (1)º	Plate, colourless
V = 2842.0 (17) Å^3	0.30 × 0.20 × 0.20 mm
Z = 4	

Data collection

Bruker Kappa-APEX2 CCD diffractometer	8394 independent reflections
Radiation source: fine-focus sealed tube	5384 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.029
T = 298(2) K	θ_{max} = 30.2º
ω and φ scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −14→15
T_{min} = 0.93, T_{max} = 0.96	k = −16→15
36502 measured reflections	l = −32→30

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.045$	H-atom parameters constrained
$wR(F^2) = 0.128$	$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.8797P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.01$	$(\Delta/\sigma)_{max} = 0.001$
8394 reflections	$\Delta\rho_{max} = 0.32$ e Å^{-3}
370 parameters	$\Delta\rho_{min} = -0.34$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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

	x	*y*	*z*	U_{iso}*/U_{eq}
C1	0.26565 (15)	−0.17757 (14)	0.62165 (7)	0.0415 (3)
N2	0.18066 (13)	−0.16509 (12)	0.65790 (6)	0.0463 (3)
O3	0.13128 (10)	−0.04971 (10)	0.64893 (5)	0.0464 (3)
C3A	0.22839 (14)	0.01637 (13)	0.62121 (6)	0.0391 (3)
C4	0.31070 (15)	0.06934 (13)	0.66958 (6)	0.0399 (3)
C5	0.23745 (16)	0.14586 (15)	0.70822 (7)	0.0458 (4)
H5A	0.1761	0.0986	0.7268	0.055*
H5B	0.2920	0.1797	0.7383	0.055*
N6	0.17576 (14)	0.24050 (13)	0.67469 (6)	0.0491 (3)
C7	0.09347 (16)	0.19098 (16)	0.62914 (7)	0.0481 (4)
H7A	0.0515	0.2543	0.6081	0.058*
H7B	0.0313	0.1427	0.6466	0.058*
C7A	0.16481 (14)	0.11610 (13)	0.58675 (6)	0.0390 (3)
H71A	0.1059	0.0818	0.5580	0.047*
C8	0.25323 (15)	0.19818 (13)	0.55535 (7)	0.0406 (3)
H8	0.2868	0.2524	0.5851	0.049*
S9	0.38729 (4)	0.12699 (4)	0.525049 (19)	0.04702 (12)
C9A	0.31578 (15)	0.00373 (15)	0.49202 (7)	0.0429 (4)
C10	0.30426 (17)	−0.00698 (17)	0.43211 (7)	0.0517 (4)
H10	0.3324	0.0532	0.4088	0.062*
C11	0.25179 (19)	−0.10530 (18)	0.40693 (8)	0.0582 (5)
H11	0.2471	−0.1128	0.3668	0.070*
C12	0.20602 (17)	−0.19283 (17)	0.44107 (8)	0.0544 (4)
H12	0.1700	−0.2592	0.4239	0.065*
C13	0.21341 (16)	−0.18245 (15)	0.50082 (7)	0.0469 (4)
H13	0.1800	−0.2407	0.5236	0.056*
C13A	0.27046 (14)	−0.08553 (14)	0.52684 (7)	0.0404 (3)
N14	0.28845 (12)	−0.07636 (11)	0.58843 (5)	0.0396 (3)
C1E	0.47422 (19)	−0.48916 (16)	0.60873 (8)	0.0560 (5)
C2E	0.36580 (19)	−0.49215 (15)	0.63745 (8)	0.0535 (4)

H2E	0.3392	−0.5617	0.6541	0.064*
C3E	0.29710 (17)	−0.39048 (14)	0.64122 (7)	0.0470 (4)
H3E	0.2230	−0.3917	0.6600	0.056*
C4E	0.33800 (16)	−0.28603 (14)	0.61706 (7)	0.0426 (4)
C5E	0.44779 (17)	−0.28544 (16)	0.58912 (8)	0.0546 (4)
H5E	0.4760	−0.2157	0.5733	0.066*
C6E	0.51637 (19)	−0.38703 (17)	0.58441 (9)	0.0611 (5)
H6E	0.5898	−0.3865	0.5651	0.073*
C40	0.43178 (15)	0.04950 (14)	0.67434 (7)	0.0438 (4)
H40	0.4622	−0.0044	0.6483	0.053*
C41	0.52401 (16)	0.10201 (17)	0.71562 (7)	0.0492 (4)
C42	0.5219 (2)	0.21982 (18)	0.73168 (9)	0.0641 (5)
H42	0.4578	0.2676	0.7174	0.077*
C43	0.6129 (2)	0.2676 (2)	0.76843 (11)	0.0807 (7)
H43	0.6101	0.3463	0.7792	0.097*
C44	0.7058 (2)	0.1977 (3)	0.78831 (11)	0.0828 (7)
C45	0.7134 (2)	0.0817 (3)	0.77427 (10)	0.0809 (7)
H45	0.7784	0.0356	0.7890	0.097*
C46	0.62175 (17)	0.0339 (2)	0.73741 (9)	0.0619 (5)
H46	0.6260	−0.0451	0.7272	0.074*
C61	0.1071 (2)	0.3164 (2)	0.71289 (9)	0.0737 (6)
H61A	0.1624	0.3477	0.7427	0.111*
H61B	0.0436	0.2714	0.7303	0.111*
H61C	0.0702	0.3798	0.6908	0.111*
C81	0.18678 (15)	0.27456 (14)	0.51049 (7)	0.0405 (3)
C82	0.11326 (16)	0.22881 (16)	0.46533 (7)	0.0507 (4)
H82	0.1009	0.1479	0.4630	0.061*
C83	0.05769 (18)	0.3015 (2)	0.42351 (8)	0.0603 (5)
H83	0.0090	0.2702	0.3931	0.072*
C84	0.0760 (2)	0.4192 (2)	0.42803 (9)	0.0670 (6)
C85	0.1463 (2)	0.46790 (19)	0.47118 (11)	0.0792 (7)
H85	0.1573	0.5490	0.4731	0.095*
C86	0.2018 (2)	0.39493 (16)	0.51271 (9)	0.0622 (5)
H86	0.2504	0.4279	0.5428	0.075*
F1	0.79600 (16)	0.24504 (19)	0.82413 (8)	0.1296 (7)
F2	0.02109 (15)	0.49074 (14)	0.38734 (6)	0.1075 (5)
CL1	0.55743 (6)	−0.61865 (5)	0.60066 (3)	0.0882 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0473 (9)	0.0431 (8)	0.0338 (8)	−0.0042 (7)	−0.0001 (7)	0.0055 (6)
N2	0.0513 (8)	0.0449 (7)	0.0429 (8)	−0.0018 (6)	0.0041 (6)	0.0078 (6)
O3	0.0455 (6)	0.0478 (6)	0.0465 (6)	−0.0002 (5)	0.0097 (5)	0.0072 (5)
C3A	0.0404 (8)	0.0430 (8)	0.0341 (8)	0.0006 (6)	0.0042 (6)	0.0043 (6)
C4	0.0457 (9)	0.0423 (8)	0.0315 (7)	0.0019 (6)	0.0007 (6)	0.0066 (6)
C5	0.0497 (9)	0.0533 (9)	0.0342 (8)	0.0067 (7)	0.0008 (7)	0.0020 (7)
N6	0.0583 (9)	0.0519 (8)	0.0370 (7)	0.0130 (7)	0.0009 (6)	−0.0011 (6)

C7	0.0459 (9)	0.0579 (10)	0.0407 (9)	0.0104 (7)	0.0027 (7)	0.0055 (7)
C7A	0.0385 (8)	0.0455 (8)	0.0326 (8)	0.0007 (6)	−0.0020 (6)	0.0033 (6)
C8	0.0441 (9)	0.0414 (8)	0.0362 (8)	0.0009 (6)	−0.0013 (7)	0.0018 (6)
S9	0.0408 (2)	0.0489 (2)	0.0518 (3)	−0.00048 (17)	0.00653 (18)	0.01041 (18)
C9A	0.0408 (8)	0.0493 (9)	0.0392 (8)	0.0071 (7)	0.0081 (7)	0.0073 (7)
C10	0.0529 (10)	0.0627 (11)	0.0402 (9)	0.0078 (8)	0.0111 (8)	0.0107 (8)
C11	0.0636 (12)	0.0768 (13)	0.0341 (9)	0.0095 (10)	0.0024 (8)	0.0012 (8)
C12	0.0583 (11)	0.0605 (11)	0.0438 (10)	0.0031 (8)	−0.0054 (8)	−0.0053 (8)
C13	0.0494 (10)	0.0504 (9)	0.0408 (9)	−0.0002 (7)	0.0011 (7)	0.0026 (7)
C13A	0.0411 (8)	0.0464 (8)	0.0338 (8)	0.0055 (6)	0.0036 (6)	0.0034 (6)
N14	0.0473 (7)	0.0382 (6)	0.0332 (7)	0.0015 (5)	0.0028 (5)	0.0048 (5)
C1E	0.0650 (12)	0.0476 (9)	0.0547 (11)	0.0095 (8)	−0.0054 (9)	0.0014 (8)
C2E	0.0753 (13)	0.0404 (8)	0.0445 (9)	−0.0027 (8)	−0.0010 (9)	0.0055 (7)
C3E	0.0601 (10)	0.0447 (9)	0.0363 (8)	−0.0046 (7)	0.0046 (7)	0.0032 (6)
C4E	0.0518 (9)	0.0417 (8)	0.0339 (8)	−0.0013 (7)	−0.0016 (7)	0.0042 (6)
C5E	0.0556 (11)	0.0474 (9)	0.0611 (11)	−0.0006 (8)	0.0065 (9)	0.0116 (8)
C6E	0.0558 (11)	0.0600 (11)	0.0682 (13)	0.0070 (9)	0.0092 (10)	0.0086 (9)
C40	0.0447 (9)	0.0484 (9)	0.0383 (8)	0.0022 (7)	0.0012 (7)	0.0066 (7)
C41	0.0446 (9)	0.0627 (11)	0.0400 (9)	−0.0014 (8)	−0.0004 (7)	0.0105 (7)
C42	0.0634 (12)	0.0601 (12)	0.0675 (13)	−0.0060 (9)	−0.0096 (10)	0.0087 (10)
C43	0.0832 (16)	0.0760 (15)	0.0812 (16)	−0.0228 (13)	−0.0137 (13)	−0.0014 (12)
C44	0.0693 (15)	0.110 (2)	0.0672 (14)	−0.0264 (14)	−0.0187 (12)	0.0072 (14)
C45	0.0529 (12)	0.116 (2)	0.0717 (15)	0.0024 (13)	−0.0179 (11)	0.0199 (14)
C46	0.0501 (11)	0.0755 (13)	0.0597 (12)	0.0049 (9)	−0.0027 (9)	0.0105 (10)
C61	0.0951 (17)	0.0759 (14)	0.0501 (11)	0.0348 (12)	0.0032 (11)	−0.0070 (10)
C81	0.0426 (8)	0.0442 (8)	0.0350 (8)	0.0034 (6)	0.0040 (7)	0.0042 (6)
C82	0.0543 (10)	0.0515 (10)	0.0455 (10)	−0.0002 (8)	−0.0044 (8)	0.0032 (7)
C83	0.0560 (11)	0.0831 (14)	0.0412 (10)	0.0075 (10)	−0.0055 (8)	0.0089 (9)
C84	0.0723 (14)	0.0732 (13)	0.0556 (12)	0.0189 (11)	0.0033 (10)	0.0271 (10)
C85	0.1073 (19)	0.0467 (11)	0.0828 (16)	0.0116 (11)	−0.0055 (14)	0.0163 (10)
C86	0.0826 (14)	0.0457 (10)	0.0573 (12)	−0.0001 (9)	−0.0093 (10)	0.0019 (8)
F1	0.1032 (12)	0.1675 (18)	0.1129 (13)	−0.0444 (12)	−0.0530 (10)	−0.0030 (12)
F2	0.1228 (12)	0.1098 (11)	0.0883 (10)	0.0369 (10)	−0.0117 (9)	0.0513 (9)
CL1	0.0964 (4)	0.0586 (3)	0.1103 (5)	0.0258 (3)	0.0137 (4)	0.0067 (3)

Geometric parameters (Å, °)

C1—N2	1.281 (2)	C1E—CL1	1.740 (2)
C1—N14	1.4108 (19)	C2E—C3E	1.380 (3)
C1—C4E	1.467 (2)	C2E—H2E	0.9300
N2—O3	1.4279 (18)	C3E—C4E	1.393 (2)
O3—C3A	1.4639 (19)	C3E—H3E	0.9300
C3A—N14	1.467 (2)	C4E—C5E	1.379 (3)
C3A—C4	1.521 (2)	C5E—C6E	1.380 (3)
C3A—C7A	1.531 (2)	C5E—H5E	0.9300
C4—C40	1.331 (2)	C6E—H6E	0.9300
C4—C5	1.499 (2)	C40—C41	1.475 (2)
C5—N6	1.468 (2)	C40—H40	0.9300
C5—H5A	0.9700	C41—C46	1.388 (3)

C5—H5B	0.9700	C41—C42	1.390 (3)
N6—C7	1.461 (2)	C42—C43	1.383 (3)
N6—C61	1.461 (2)	C42—H42	0.9300
C7—C7A	1.533 (2)	C43—C44	1.346 (4)
C7—H7A	0.9700	C43—H43	0.9300
C7—H7B	0.9700	C44—C45	1.361 (4)
C7A—C8	1.542 (2)	C44—F1	1.362 (3)
C7A—H71A	0.9800	C45—C46	1.389 (3)
C8—C81	1.509 (2)	C45—H45	0.9300
C8—S9	1.8298 (17)	C46—H46	0.9300
C8—H8	0.9800	C61—H61A	0.9600
S9—C9A	1.7582 (19)	C61—H61B	0.9600
C9A—C10	1.389 (2)	C61—H61C	0.9600
C9A—C13A	1.398 (2)	C81—C86	1.379 (2)
C10—C11	1.371 (3)	C81—C82	1.385 (2)
C10—H10	0.9300	C82—C83	1.388 (2)
C11—C12	1.376 (3)	C82—H82	0.9300
C11—H11	0.9300	C83—C84	1.356 (3)
C12—C13	1.384 (2)	C83—H83	0.9300
C12—H12	0.9300	C84—C85	1.345 (3)
C13—C13A	1.387 (2)	C84—F2	1.359 (2)
C13—H13	0.9300	C85—C86	1.385 (3)
C13A—N14	1.431 (2)	C85—H85	0.9300
C1E—C2E	1.376 (3)	C86—H86	0.9300
C1E—C6E	1.377 (3)		
N2—C1—N14	114.48 (14)	C2E—C1E—C6E	121.62 (17)
N2—C1—C4E	122.87 (14)	C2E—C1E—CL1	119.26 (15)
N14—C1—C4E	122.57 (14)	C6E—C1E—CL1	119.08 (16)
C1—N2—O3	106.42 (12)	C1E—C2E—C3E	119.01 (16)
N2—O3—C3A	105.22 (11)	C1E—C2E—H2E	120.5
O3—C3A—N14	101.65 (12)	C3E—C2E—H2E	120.5
O3—C3A—C4	106.91 (12)	C2E—C3E—C4E	120.40 (17)
N14—C3A—C4	113.85 (13)	C2E—C3E—H3E	119.8
O3—C3A—C7A	106.98 (12)	C4E—C3E—H3E	119.8
N14—C3A—C7A	117.59 (12)	C5E—C4E—C3E	119.26 (16)
C4—C3A—C7A	108.88 (13)	C5E—C4E—C1	120.32 (15)
C40—C4—C5	126.58 (15)	C3E—C4E—C1	120.42 (16)
C40—C4—C3A	122.42 (15)	C4E—C5E—C6E	120.79 (17)
C5—C4—C3A	110.99 (14)	C4E—C5E—H5E	119.6
N6—C5—C4	110.59 (13)	C6E—C5E—H5E	119.6
N6—C5—H5A	109.5	C1E—C6E—C5E	118.91 (19)
C4—C5—H5A	109.5	C1E—C6E—H6E	120.5
N6—C5—H5B	109.5	C5E—C6E—H6E	120.5
C4—C5—H5B	109.5	C4—C40—C41	128.37 (16)
H5A—C5—H5B	108.1	C4—C40—H40	115.8
C7—N6—C61	110.58 (15)	C41—C40—H40	115.8
C7—N6—C5	110.23 (14)	C46—C41—C42	117.55 (18)
C61—N6—C5	110.24 (14)	C46—C41—C40	119.45 (18)
N6—C7—C7A	111.41 (14)	C42—C41—C40	122.90 (16)

N6—C7—H7A	109.3	C43—C42—C41	121.5 (2)
C7A—C7—H7A	109.3	C43—C42—H42	119.3
N6—C7—H7B	109.3	C41—C42—H42	119.3
C7A—C7—H7B	109.3	C44—C43—C42	118.6 (2)
H7A—C7—H7B	108.0	C44—C43—H43	120.7
C3A—C7A—C7	107.88 (12)	C42—C43—H43	120.7
C3A—C7A—C8	114.71 (13)	C43—C44—C45	122.8 (2)
C7—C7A—C8	107.87 (13)	C43—C44—F1	118.6 (3)
C3A—C7A—H71A	108.7	C45—C44—F1	118.5 (2)
C7—C7A—H71A	108.7	C44—C45—C46	118.4 (2)
C8—C7A—H71A	108.7	C44—C45—H45	120.8
C81—C8—C7A	112.57 (13)	C46—C45—H45	120.8
C81—C8—S9	110.72 (11)	C41—C46—C45	121.1 (2)
C7A—C8—S9	115.70 (11)	C41—C46—H46	119.5
C81—C8—H8	105.7	C45—C46—H46	119.5
C7A—C8—H8	105.7	N6—C61—H61A	109.5
S9—C8—H8	105.7	N6—C61—H61B	109.5
C9A—S9—C8	100.17 (8)	H61A—C61—H61B	109.5
C10—C9A—C13A	119.46 (16)	N6—C61—H61C	109.5
C10—C9A—S9	121.32 (13)	H61A—C61—H61C	109.5
C13A—C9A—S9	119.22 (12)	H61B—C61—H61C	109.5
C11—C10—C9A	120.71 (17)	C86—C81—C82	117.63 (16)
C11—C10—H10	119.6	C86—C81—C8	119.57 (15)
C9A—C10—H10	119.6	C82—C81—C8	122.77 (15)
C10—C11—C12	119.95 (17)	C81—C82—C83	121.15 (18)
C10—C11—H11	120.0	C81—C82—H82	119.4
C12—C11—H11	120.0	C83—C82—H82	119.4
C11—C12—C13	120.28 (18)	C84—C83—C82	118.47 (19)
C11—C12—H12	119.9	C84—C83—H83	120.8
C13—C12—H12	119.9	C82—C83—H83	120.8
C12—C13—C13A	120.28 (16)	C85—C84—C83	122.59 (18)
C12—C13—H13	119.9	C85—C84—F2	118.8 (2)
C13A—C13—H13	119.9	C83—C84—F2	118.6 (2)
C13—C13A—C9A	119.24 (15)	C84—C85—C86	118.7 (2)
C13—C13A—N14	121.77 (14)	C84—C85—H85	120.6
C9A—C13A—N14	118.94 (14)	C86—C85—H85	120.6
C1—N14—C13A	117.52 (13)	C81—C86—C85	121.44 (19)
C1—N14—C3A	102.24 (12)	C81—C86—H86	119.3
C13A—N14—C3A	121.47 (12)	C85—C86—H86	119.3
N14—C1—N2—O3	3.69 (18)	C13—C13A—N14—C3A	−116.19 (17)
C4E—C1—N2—O3	−179.38 (14)	C9A—C13A—N14—C3A	66.2 (2)
C1—N2—O3—C3A	−22.32 (15)	O3—C3A—N14—C1	−27.85 (14)
N2—O3—C3A—N14	31.02 (13)	C4—C3A—N14—C1	86.72 (15)
N2—O3—C3A—C4	−88.59 (14)	C7A—C3A—N14—C1	−144.21 (14)
N2—O3—C3A—C7A	154.89 (12)	O3—C3A—N14—C13A	105.57 (14)
O3—C3A—C4—C40	123.58 (16)	C4—C3A—N14—C13A	−139.87 (14)
N14—C3A—C4—C40	12.2 (2)	C7A—C3A—N14—C13A	−10.8 (2)
C7A—C3A—C4—C40	−121.17 (16)	C6E—C1E—C2E—C3E	0.9 (3)
O3—C3A—C4—C5	−57.68 (16)	CL1—C1E—C2E—C3E	−176.81 (14)

N14—C3A—C4—C5	−169.09 (12)	C1E—C2E—C3E—C4E	−1.0 (3)
C7A—C3A—C4—C5	57.57 (16)	C2E—C3E—C4E—C5E	0.2 (3)
C40—C4—C5—N6	120.48 (18)	C2E—C3E—C4E—C1	−179.30 (15)
C3A—C4—C5—N6	−58.19 (18)	N2—C1—C4E—C5E	−162.90 (17)
C4—C5—N6—C7	59.06 (18)	N14—C1—C4E—C5E	13.8 (2)
C4—C5—N6—C61	−178.59 (17)	N2—C1—C4E—C3E	16.6 (2)
C61—N6—C7—C7A	177.10 (15)	N14—C1—C4E—C3E	−166.68 (15)
C5—N6—C7—C7A	−60.75 (17)	C3E—C4E—C5E—C6E	0.7 (3)
O3—C3A—C7A—C7	58.34 (16)	C1—C4E—C5E—C6E	−179.80 (17)
N14—C3A—C7A—C7	171.77 (13)	C2E—C1E—C6E—C5E	0.0 (3)
C4—C3A—C7A—C7	−56.86 (16)	CL1—C1E—C6E—C5E	177.70 (16)
O3—C3A—C7A—C8	178.57 (12)	C4E—C5E—C6E—C1E	−0.8 (3)
N14—C3A—C7A—C8	−67.99 (18)	C5—C4—C40—C41	−4.0 (3)
C4—C3A—C7A—C8	63.37 (16)	C3A—C4—C40—C41	174.52 (15)
N6—C7—C7A—C3A	59.72 (17)	C4—C40—C41—C46	144.01 (19)
N6—C7—C7A—C8	−64.73 (16)	C4—C40—C41—C42	−39.7 (3)
C3A—C7A—C8—C81	165.43 (13)	C46—C41—C42—C43	−0.6 (3)
C7—C7A—C8—C81	−74.34 (16)	C40—C41—C42—C43	−176.99 (19)
C3A—C7A—C8—S9	36.72 (17)	C41—C42—C43—C44	0.7 (4)
C7—C7A—C8—S9	156.96 (11)	C42—C43—C44—C45	−0.7 (4)
C81—C8—S9—C9A	−84.45 (12)	C42—C43—C44—F1	179.5 (2)
C7A—C8—S9—C9A	45.15 (12)	C43—C44—C45—C46	0.6 (4)
C8—S9—C9A—C10	110.28 (14)	F1—C44—C45—C46	−179.6 (2)
C8—S9—C9A—C13A	−70.17 (14)	C42—C41—C46—C45	0.5 (3)
C13A—C9A—C10—C11	−1.6 (3)	C40—C41—C46—C45	177.01 (19)
S9—C9A—C10—C11	177.92 (14)	C44—C45—C46—C41	−0.5 (3)
C9A—C10—C11—C12	2.3 (3)	C7A—C8—C81—C86	125.69 (18)
C10—C11—C12—C13	−0.4 (3)	S9—C8—C81—C86	−103.06 (17)
C11—C12—C13—C13A	−2.0 (3)	C7A—C8—C81—C82	−56.5 (2)
C12—C13—C13A—C9A	2.6 (2)	S9—C8—C81—C82	74.79 (18)
C12—C13—C13A—N14	−174.97 (16)	C86—C81—C82—C83	0.6 (3)
C10—C9A—C13A—C13	−0.8 (2)	C8—C81—C82—C83	−177.30 (16)
S9—C9A—C13A—C13	179.63 (12)	C81—C82—C83—C84	−0.6 (3)
C10—C9A—C13A—N14	176.86 (14)	C82—C83—C84—C85	0.4 (3)
S9—C9A—C13A—N14	−2.7 (2)	C82—C83—C84—F2	−179.60 (18)
N2—C1—N14—C13A	−119.50 (16)	C83—C84—C85—C86	−0.2 (4)
C4E—C1—N14—C13A	63.6 (2)	F2—C84—C85—C86	179.8 (2)
N2—C1—N14—C3A	16.18 (17)	C82—C81—C86—C85	−0.4 (3)
C4E—C1—N14—C3A	−160.76 (14)	C8—C81—C86—C85	177.58 (19)
C13—C13A—N14—C1	10.6 (2)	C84—C85—C86—C81	0.2 (4)
C9A—C13A—N14—C1	−166.97 (15)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C61—H61C···F2^{i}	0.96	2.50	3.435 (3)	166
C83—H83···N2^{ii}	0.93	2.61	3.488 (3)	157

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

Fig. 1

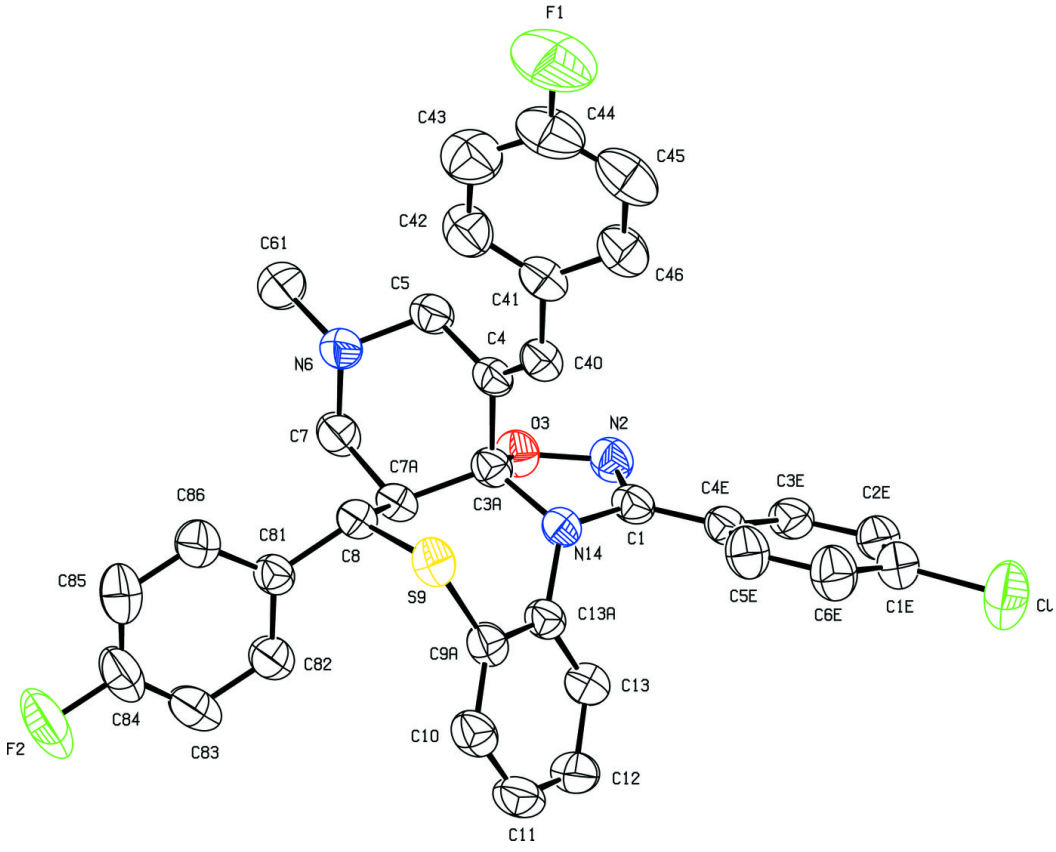
F1
C43
C44
C45
C42
C46
C61
C5
C41
C4
N6
C40
C7
O3
N2
C7A
C3A
C4E
C3E
C2E
C86
C81
C8
N14
C1
C85
S9
C5E
C6E
C1E
Cl
C13A
C9A
C82
C13
C84
C10
C12
F2
C83
C11

Fig. 2