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(9*S*,13*R*,14*S*)-7,8-Didehydro-4-(4-fluorobenzyloxy)-3,7-dimethoxy-17-methylmorphinan-6-one sesquihydrate

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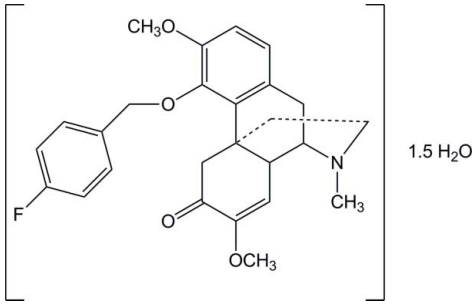
Key indicators: single-crystal X-ray study; T = 133 K; mean σ(C–C) = 0.002 Å; R factor = 0.028; wR factor = 0.081; data-to-parameter ratio = 10.9.

In the title sinomenine derivative, $C_{26}H_{28}FNO_4\cdot1.5H_2O$, the dihedral angle between the two aromatic rings is 55.32 (6)°. The N-containing ring has an approximate chair conformation, while other two rings have approximate envelope and half-chair conformations. One water molecule is located on a twofold symmetry axis. In the crystal, the water molecules form O—H···O and O—H···N hydrogen bonds, bridging symmetry-related main molecules.

Related literature

For background to the biological activity of sinomenine derivatives and other related compounds, see: Liu *et al.* (1994, 1996, 1997); Mark *et al.* (2003); Ye *et al.* (2004). For the synthesis of the title compound, see: Mitsunobu (1981). For related structures, see: Li *et al.* (2009); Batterham *et al.* (1965); Zheng & Jiang (2010); Zheng *et al.* (2011).



Experimental

Crystal data

$C_{26}H_{28}FNO_4\cdot1.5H_2O$
M_r = 464.52
Monoclinic, $C2$
a = 18.0155 (3) Å
b = 7.6776 (1) Å
c = 18.1506 (4) Å
β = 109.324 (1)°
V = 2369.08 (7) Å^3
Z = 4
Cu $K\alpha$ radiation
μ = 0.79 mm^{-1}
T = 133 K
0.25 × 0.15 × 0.10 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)
T_{min} = 0.826, T_{max} = 0.925
7232 measured reflections
3415 independent reflections
3402 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.028
$wR(F^2)$ = 0.081
S = 1.04
3415 reflections
312 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.20 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack (1983), 1359 Friedel pairs
Flack parameter: 0.05 (12)

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

D—H···A	D—H	H···A	D···A	D—H···A
O2*S*—H22*S*···O4	0.91	2.33	2.9805 (16)	128
O2*S*—H22*S*···O3	0.91	2.54	3.417 (2)	164
O1*S*—H11*S*···O2*S*	0.90 (3)	1.94 (3)	2.8342 (19)	172 (2)
O2*S*—H21*S*···N1^{i}	0.97	1.81	2.7736 (19)	170

Symmetry code: (i) $x+\frac{1}{2}, y+\frac{1}{2}, z$.

Data collection: *APEX2* (Bruker, 2000); cell refinement: *SAINT* (Bruker, 2000); 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* (Sheldrick, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BH2361).

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supplementary materials

Acta Cryst. (2011). E**67**, o1988 [**doi:10.1107/S1600536811026183**]

(9*S*,13*R*,14*S*)-7,8-Didehydro-4-(4-fluorobenzyloxy)-3,7-dimethoxy-17-methylmorphinan-6-one sesquihydrate

X.-L. Zheng, N.-F. Jiang, D. Luo, H.-S. Gao and A.-S. Ding

Comment

We synthesized a new sinomenine derivative sesquihydrate. Herein, its crystal structure is reported. Biological effects of sinomenine derivatives and related compounds have been described (Liu *et al.*, 1994, 1996, 1997; Mark *et al.*, 2003; Ye *et al.*, 2004).

The molecular structure of the title compound is shown in Fig. 1. The crystal structure is stabilized by O—H···O and O—H···N hydrogen bonds linking the sinomenine derivative and the water molecules, and weak C—H···O hydrogen bonds between molecules (Fig. 2). Significant aromatic stacking interactions were not found. There exist two planes in the molecule of the title compound: atoms C1···C6 form the benzene plane (I), and atoms C21···C26 form the benzene plane substituted by fluorine (II). The angle between the two planes (I) and (II) is 55.32 (6)°. Rings *C* [C7/C8/C11/C12/C13/C14] and *B* [C5···C10] in the molecule approximate envelope and half-chair conformations, respectively. In contrast, ring *D* [C9/N1/C16/C15/C7/C8] exhibits an almost regular chair conformation. Similar features have been described in related compounds (Zheng & Jiang, 2010; Zheng *et al.*, 2011; Li *et al.*, 2009; Batterham *et al.*, 1965).

Experimental

The title compound was obtained according to the method of Mitsunobu (1981). Colorless blocks were grown from an acetic ether solution.

Refinement

The water H atoms (H21S, H22S and H11S) were located in a difference map. H11S was refined with free coordinates and isotropic displacement parameter. H21S and H22S were fixed in their as found positions and allowed to ride on O2S, and their displacement parameters were refined. Other H atoms were positioned geometrically, with C—H = 0.95 (aromatic CH), 0.98 (methyl CH_3), 0.99 (methylene CH_2) or 1.00 Å (methine CH), and were constrained to ride on their parent atoms, with U_{iso}(H) = 1.2U_{eq}(carrier C) or U_{iso}(H) = 1.5U_{eq}(carrier C17 C18 C19). 1359 Friedel pairs were used for the Flack parameter refinement.

Figures

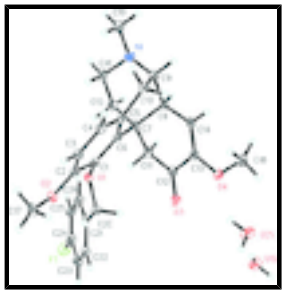

Fig. 1. The molecular structure of the title compound showing 50% probability displacement ellipsoids.

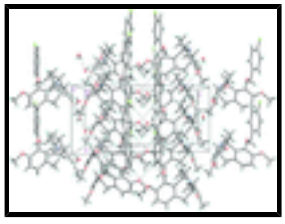

Fig. 2. A part of the crystal structure of the title compound.

(9*S*,13*R*,14*S*)-7,8-Didehydro-4-(4-fluorobenzyloxy)-3,7- dimethoxy-17-methylmorphinan-6-one sesquihydrate

Crystal data

$C_{26}H_{28}FNO_4 \cdot 1.5H_2O$	$F(000) = 988$
$M_r = 464.52$	$D_x = 1.302$ Mg m^{-3}
Monoclinic, *C*2	Cu *K*α radiation, λ = 1.54178 Å
Hall symbol: C 2y	Cell parameters from 6582 reflections
$a = 18.0155$ (3) Å	θ = 2.6–64.7°
$b = 7.6776$ (1) Å	μ = 0.79 mm^{-1}
$c = 18.1506$ (4) Å	$T = 133$ K
β = 109.324 (1)°	Block, colourless
$V = 2369.08$ (7) Å^3	0.25 × 0.15 × 0.10 mm
$Z = 4$	

Data collection

Bruker APEXII CCD diffractometer	3415 independent reflections
Radiation source: fine-focus sealed tube	3402 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.024$
φ and ω scans	$\theta_{max} = 65.0°$, $\theta_{min} = 2.6°$
Absorption correction: multi-scan (*SADABS*; Bruker, 2000)	$h = -20 \rightarrow 21$
$T_{min} = 0.826$, $T_{max} = 0.925$	$k = -9 \rightarrow 9$
7232 measured reflections	$l = -18 \rightarrow 20$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.028$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.081$	$w = 1/[\sigma^2(F_o^2) + (0.0555P)^2 + 0.6114P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.04$	$(\Delta/\sigma)_{max} < 0.001$
3415 reflections	$\Delta\rho_{max} = 0.20$ e Å^{-3}
312 parameters	$\Delta\rho_{min} = -0.18$ e Å^{-3}
1 restraint	Absolute structure: Flack (1983), 1359 Friedel pairs
0 constraints	Flack parameter: 0.05 (12)
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}
F1	0.49485 (7)	−0.26627 (16)	0.40332 (8)	0.0571 (3)
N1	−0.03356 (8)	0.4513 (2)	0.15607 (8)	0.0314 (3)
O1	0.26142 (6)	0.36402 (13)	0.34083 (6)	0.0237 (2)
O2	0.29280 (6)	0.57951 (14)	0.46321 (6)	0.0268 (2)
O3	0.32140 (7)	0.48228 (18)	0.15878 (7)	0.0392 (3)
O4	0.24651 (6)	0.74481 (15)	0.07306 (7)	0.0307 (3)
C1	0.21885 (8)	0.51681 (19)	0.33202 (9)	0.0217 (3)
C2	0.23288 (8)	0.6279 (2)	0.39717 (9)	0.0233 (3)
C3	0.18682 (9)	0.7742 (2)	0.39246 (9)	0.0265 (3)
H3	0.1968	0.8511	0.4357	0.032*
C4	0.12611 (8)	0.80691 (19)	0.32395 (9)	0.0253 (3)
H4	0.0943	0.9072	0.3209	0.030*
C5	0.10990 (8)	0.6993 (2)	0.25971 (9)	0.0239 (3)
C6	0.15775 (8)	0.55115 (19)	0.26184 (9)	0.0220 (3)
C7	0.13574 (8)	0.4223 (2)	0.19297 (9)	0.0254 (3)
C8	0.08177 (8)	0.5102 (2)	0.11800 (9)	0.0264 (3)
H8	0.0620	0.4171	0.0777	0.032*
C9	0.01057 (9)	0.5906 (2)	0.13270 (9)	0.0285 (3)
H9	−0.0244	0.6395	0.0820	0.034*
C10	0.03877 (8)	0.7424 (2)	0.18927 (9)	0.0279 (3)
H10A	0.0520	0.8416	0.1610	0.033*
H10B	−0.0048	0.7799	0.2075	0.033*
C11	0.20578 (9)	0.3494 (2)	0.17160 (9)	0.0275 (3)
H11A	0.2421	0.2888	0.2176	0.033*
H11B	0.1861	0.2628	0.1292	0.033*
C12	0.25034 (9)	0.4894 (2)	0.14564 (9)	0.0281 (3)
C13	0.20238 (9)	0.6325 (2)	0.09902 (9)	0.0266 (3)
C14	0.12457 (9)	0.6423 (2)	0.08562 (9)	0.0259 (3)
H14	0.0957	0.7353	0.0548	0.031*
C15	0.08761 (10)	0.2737 (2)	0.21185 (10)	0.0316 (4)
H15A	0.0736	0.1877	0.1688	0.038*

H15B	0.1198	0.2137	0.2601	0.038*
C16	0.01249 (10)	0.3444 (2)	0.22285 (10)	0.0330 (4)
H16A	−0.0202	0.2457	0.2293	0.040*
H16B	0.0267	0.4156	0.2710	0.040*
C17	0.31222 (10)	0.6993 (2)	0.52708 (9)	0.0332 (4)
H17A	0.3228	0.8142	0.5092	0.050*
H17B	0.3590	0.6580	0.5686	0.050*
H17C	0.2681	0.7079	0.5471	0.050*
C18	0.20598 (10)	0.8932 (2)	0.03173 (10)	0.0355 (4)
H18A	0.1646	0.8552	−0.0158	0.053*
H18B	0.2431	0.9685	0.0177	0.053*
H18C	0.1823	0.9579	0.0649	0.053*
C19	−0.10438 (10)	0.5180 (3)	0.16953 (12)	0.0392 (4)
H19A	−0.1362	0.4201	0.1767	0.059*
H19B	−0.1352	0.5874	0.1245	0.059*
H19C	−0.0890	0.5911	0.2164	0.059*
C20	0.34415 (9)	0.3842 (2)	0.35220 (9)	0.0273 (3)
H20A	0.3687	0.4621	0.3971	0.033*
H20B	0.3512	0.4367	0.3051	0.033*
C21	0.38241 (9)	0.2075 (2)	0.36728 (9)	0.0262 (3)
C22	0.46433 (10)	0.1988 (2)	0.39458 (10)	0.0323 (4)
H22	0.4942	0.3033	0.4049	0.039*
C23	0.50269 (10)	0.0401 (3)	0.40683 (11)	0.0394 (4)
H23	0.5585	0.0343	0.4250	0.047*
C24	0.45800 (11)	−0.1091 (3)	0.39200 (12)	0.0398 (4)
C25	0.37742 (11)	−0.1056 (2)	0.36530 (12)	0.0397 (4)
H25	0.3480	−0.2107	0.3556	0.048*
C26	0.33978 (10)	0.0545 (2)	0.35278 (11)	0.0335 (4)
H26	0.2839	0.0590	0.3340	0.040*
O2S	0.39668 (7)	0.7129 (2)	0.03873 (8)	0.0485 (4)
H21S	0.4268	0.7898	0.0802	0.043 (6)*
H22S	0.3668	0.6573	0.0628	0.131 (15)*
O1S	0.5000	0.4796 (3)	0.0000	0.0471 (5)
H11S	0.4712 (14)	0.555 (4)	0.0168 (15)	0.059 (7)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
F1	0.0562 (6)	0.0372 (6)	0.0885 (9)	0.0217 (5)	0.0383 (6)	0.0200 (6)
N1	0.0288 (6)	0.0374 (8)	0.0294 (8)	−0.0071 (6)	0.0115 (5)	−0.0058 (6)
O1	0.0254 (5)	0.0206 (5)	0.0261 (6)	0.0017 (4)	0.0101 (4)	0.0017 (4)
O2	0.0308 (5)	0.0273 (5)	0.0195 (6)	0.0014 (4)	0.0045 (4)	−0.0017 (4)
O3	0.0355 (6)	0.0483 (8)	0.0397 (7)	0.0102 (5)	0.0205 (5)	0.0108 (6)
O4	0.0313 (5)	0.0316 (6)	0.0331 (6)	−0.0011 (5)	0.0158 (4)	0.0059 (5)
C1	0.0241 (6)	0.0198 (7)	0.0241 (8)	−0.0004 (5)	0.0117 (5)	0.0022 (6)
C2	0.0244 (7)	0.0244 (7)	0.0219 (8)	−0.0044 (6)	0.0088 (6)	0.0001 (6)
C3	0.0298 (7)	0.0260 (8)	0.0253 (8)	−0.0044 (6)	0.0113 (6)	−0.0051 (6)
C4	0.0258 (7)	0.0220 (7)	0.0299 (9)	0.0000 (6)	0.0117 (6)	−0.0004 (6)

C5	0.0223 (6)	0.0252 (8)	0.0251 (8)	−0.0033 (6)	0.0091 (6)	0.0035 (6)
C6	0.0248 (7)	0.0221 (7)	0.0215 (8)	−0.0046 (6)	0.0110 (6)	0.0005 (6)
C7	0.0310 (7)	0.0261 (8)	0.0194 (8)	−0.0040 (6)	0.0086 (6)	−0.0019 (6)
C8	0.0290 (7)	0.0287 (8)	0.0208 (8)	−0.0043 (6)	0.0072 (6)	−0.0022 (6)
C9	0.0263 (7)	0.0342 (9)	0.0230 (9)	−0.0044 (7)	0.0056 (6)	−0.0004 (7)
C10	0.0259 (7)	0.0294 (8)	0.0271 (9)	0.0014 (6)	0.0070 (6)	0.0018 (6)
C11	0.0398 (8)	0.0236 (8)	0.0207 (8)	0.0012 (6)	0.0120 (6)	−0.0038 (6)
C12	0.0346 (8)	0.0314 (9)	0.0214 (8)	0.0022 (7)	0.0138 (6)	−0.0038 (6)
C13	0.0330 (7)	0.0301 (8)	0.0188 (8)	−0.0025 (6)	0.0113 (6)	−0.0019 (6)
C14	0.0311 (7)	0.0286 (8)	0.0165 (8)	−0.0042 (6)	0.0060 (6)	−0.0013 (6)
C15	0.0391 (8)	0.0270 (8)	0.0289 (9)	−0.0083 (7)	0.0115 (7)	−0.0035 (7)
C16	0.0371 (8)	0.0350 (9)	0.0296 (9)	−0.0130 (7)	0.0145 (6)	−0.0028 (7)
C17	0.0426 (9)	0.0300 (8)	0.0216 (9)	−0.0040 (7)	0.0034 (6)	−0.0033 (6)
C18	0.0374 (8)	0.0350 (9)	0.0336 (10)	−0.0049 (8)	0.0109 (7)	0.0074 (7)
C19	0.0319 (8)	0.0491 (11)	0.0393 (10)	−0.0082 (8)	0.0154 (7)	−0.0081 (8)
C20	0.0284 (7)	0.0261 (8)	0.0310 (9)	−0.0007 (6)	0.0146 (6)	0.0020 (7)
C21	0.0332 (7)	0.0289 (8)	0.0215 (8)	0.0050 (6)	0.0156 (6)	0.0034 (6)
C22	0.0334 (8)	0.0355 (9)	0.0283 (9)	−0.0011 (7)	0.0104 (6)	−0.0009 (7)
C23	0.0343 (9)	0.0478 (11)	0.0369 (10)	0.0089 (8)	0.0128 (7)	0.0037 (8)
C24	0.0459 (9)	0.0355 (9)	0.0466 (11)	0.0143 (8)	0.0267 (8)	0.0149 (8)
C25	0.0448 (9)	0.0258 (8)	0.0596 (12)	0.0024 (8)	0.0322 (9)	0.0078 (8)
C26	0.0318 (8)	0.0301 (9)	0.0446 (10)	0.0029 (7)	0.0208 (7)	0.0054 (7)
O2S	0.0406 (6)	0.0605 (9)	0.0520 (8)	−0.0194 (7)	0.0256 (6)	−0.0300 (7)
O1S	0.0402 (10)	0.0401 (11)	0.0665 (14)	0.000	0.0251 (10)	0.000

Geometric parameters (Å, °)

F1—C24	1.360 (2)	C12—C13	1.479 (2)
N1—C19	1.469 (2)	C13—C14	1.343 (2)
N1—C16	1.471 (2)	C14—H14	0.9500
N1—C9	1.476 (2)	C15—C16	1.531 (2)
O1—C1	1.3815 (18)	C15—H15A	0.9900
O1—C20	1.4438 (17)	C15—H15B	0.9900
O2—C2	1.3725 (18)	C16—H16A	0.9900
O2—C17	1.430 (2)	C16—H16B	0.9900
O3—C12	1.2233 (19)	C17—H17A	0.9800
O4—C13	1.3585 (19)	C17—H17B	0.9800
O4—C18	1.425 (2)	C17—H17C	0.9800
C1—C6	1.406 (2)	C18—H18A	0.9800
C1—C2	1.411 (2)	C18—H18B	0.9800
C2—C3	1.383 (2)	C18—H18C	0.9800
C3—C4	1.380 (2)	C19—H19A	0.9800
C3—H3	0.9500	C19—H19B	0.9800
C4—C5	1.379 (2)	C19—H19C	0.9800
C4—H4	0.9500	C20—C21	1.505 (2)
C5—C6	1.420 (2)	C20—H20A	0.9900
C5—C10	1.517 (2)	C20—H20B	0.9900
C6—C7	1.540 (2)	C21—C26	1.381 (2)
C7—C15	1.539 (2)	C21—C22	1.394 (2)

C7—C8	1.542 (2)	C22—C23	1.381 (3)
C7—C11	1.543 (2)	C22—H22	0.9500
C8—C14	1.506 (2)	C23—C24	1.375 (3)
C8—C9	1.525 (2)	C23—H23	0.9500
C8—H8	1.0000	C24—C25	1.370 (3)
C9—C10	1.525 (2)	C25—C26	1.386 (3)
C9—H9	1.0000	C25—H25	0.9500
C10—H10A	0.9900	C26—H26	0.9500
C10—H10B	0.9900	O2S—H21S	0.9694
C11—C12	1.507 (2)	O2S—H22S	0.9051
C11—H11A	0.9900	O1S—H11S	0.90 (3)
C11—H11B	0.9900		
C19—N1—C16	109.97 (14)	O4—C13—C12	111.83 (13)
C19—N1—C9	112.02 (14)	C13—C14—C8	122.02 (15)
C16—N1—C9	115.49 (12)	C13—C14—H14	119.0
C1—O1—C20	115.64 (11)	C8—C14—H14	119.0
C2—O2—C17	116.41 (12)	C16—C15—C7	110.70 (13)
C13—O4—C18	115.67 (12)	C16—C15—H15A	109.5
O1—C1—C6	120.15 (13)	C7—C15—H15A	109.5
O1—C1—C2	118.52 (12)	C16—C15—H15B	109.5
C6—C1—C2	121.03 (14)	C7—C15—H15B	109.5
O2—C2—C3	123.80 (14)	H15A—C15—H15B	108.1
O2—C2—C1	116.07 (13)	N1—C16—C15	111.84 (13)
C3—C2—C1	120.13 (13)	N1—C16—H16A	109.2
C4—C3—C2	118.93 (14)	C15—C16—H16A	109.2
C4—C3—H3	120.5	N1—C16—H16B	109.2
C2—C3—H3	120.5	C15—C16—H16B	109.2
C5—C4—C3	122.39 (14)	H16A—C16—H16B	107.9
C5—C4—H4	118.8	O2—C17—H17A	109.5
C3—C4—H4	118.8	O2—C17—H17B	109.5
C4—C5—C6	120.01 (13)	H17A—C17—H17B	109.5
C4—C5—C10	117.71 (14)	O2—C17—H17C	109.5
C6—C5—C10	122.25 (14)	H17A—C17—H17C	109.5
C1—C6—C5	117.47 (13)	H17B—C17—H17C	109.5
C1—C6—C7	121.96 (13)	O4—C18—H18A	109.5
C5—C6—C7	120.11 (13)	O4—C18—H18B	109.5
C15—C7—C6	107.95 (12)	H18A—C18—H18B	109.5
C15—C7—C8	106.49 (12)	O4—C18—H18C	109.5
C6—C7—C8	110.68 (13)	H18A—C18—H18C	109.5
C15—C7—C11	110.81 (13)	H18B—C18—H18C	109.5
C6—C7—C11	115.14 (12)	N1—C19—H19A	109.5
C8—C7—C11	105.44 (12)	N1—C19—H19B	109.5
C14—C8—C9	111.39 (13)	H19A—C19—H19B	109.5
C14—C8—C7	112.60 (12)	N1—C19—H19C	109.5
C9—C8—C7	110.14 (12)	H19A—C19—H19C	109.5
C14—C8—H8	107.5	H19B—C19—H19C	109.5
C9—C8—H8	107.5	O1—C20—C21	108.57 (12)
C7—C8—H8	107.5	O1—C20—H20A	110.0
N1—C9—C8	108.72 (13)	C21—C20—H20A	110.0

N1—C9—C10	116.85 (14)	O1—C20—H20B	110.0
C8—C9—C10	108.37 (12)	C21—C20—H20B	110.0
N1—C9—H9	107.5	H20A—C20—H20B	108.4
C8—C9—H9	107.5	C26—C21—C22	118.90 (15)
C10—C9—H9	107.5	C26—C21—C20	122.68 (13)
C5—C10—C9	113.63 (14)	C22—C21—C20	118.39 (15)
C5—C10—H10A	108.8	C23—C22—C21	120.94 (16)
C9—C10—H10A	108.8	C23—C22—H22	119.5
C5—C10—H10B	108.8	C21—C22—H22	119.5
C9—C10—H10B	108.8	C24—C23—C22	118.28 (15)
H10A—C10—H10B	107.7	C24—C23—H23	120.9
C12—C11—C7	112.63 (13)	C22—C23—H23	120.9
C12—C11—H11A	109.1	F1—C24—C25	118.55 (17)
C7—C11—H11A	109.1	F1—C24—C23	119.01 (15)
C12—C11—H11B	109.1	C25—C24—C23	122.44 (17)
C7—C11—H11B	109.1	C24—C25—C26	118.62 (17)
H11A—C11—H11B	107.8	C24—C25—H25	120.7
O3—C12—C13	121.36 (15)	C26—C25—H25	120.7
O3—C12—C11	122.58 (15)	C21—C26—C25	120.83 (15)
C13—C12—C11	115.97 (13)	C21—C26—H26	119.6
C14—C13—O4	126.68 (15)	C25—C26—H26	119.6
C14—C13—C12	121.49 (14)	H21S—O2S—H22S	100.4
C20—O1—C1—C6	117.00 (14)	C7—C8—C9—C10	67.54 (17)
C20—O1—C1—C2	−69.19 (16)	C4—C5—C10—C9	−161.88 (13)
C17—O2—C2—C3	−6.5 (2)	C6—C5—C10—C9	16.3 (2)
C17—O2—C2—C1	174.39 (13)	N1—C9—C10—C5	76.09 (17)
O1—C1—C2—O2	4.70 (18)	C8—C9—C10—C5	−47.08 (17)
C6—C1—C2—O2	178.46 (12)	C15—C7—C11—C12	174.84 (13)
O1—C1—C2—C3	−174.47 (12)	C6—C7—C11—C12	−62.30 (18)
C6—C1—C2—C3	−0.7 (2)	C8—C7—C11—C12	59.99 (16)
O2—C2—C3—C4	−177.53 (14)	C7—C11—C12—O3	146.72 (15)
C1—C2—C3—C4	1.6 (2)	C7—C11—C12—C13	−36.64 (19)
C2—C3—C4—C5	−0.4 (2)	C18—O4—C13—C14	4.3 (2)
C3—C4—C5—C6	−1.7 (2)	C18—O4—C13—C12	−176.15 (14)
C3—C4—C5—C10	176.50 (14)	O3—C12—C13—C14	−178.85 (14)
O1—C1—C6—C5	172.34 (12)	C11—C12—C13—C14	4.5 (2)
C2—C1—C6—C5	−1.31 (19)	O3—C12—C13—O4	1.6 (2)
O1—C1—C6—C7	0.16 (19)	C11—C12—C13—O4	−175.12 (12)
C2—C1—C6—C7	−173.49 (13)	O4—C13—C14—C8	−179.60 (15)
C4—C5—C6—C1	2.50 (19)	C12—C13—C14—C8	0.9 (2)
C10—C5—C6—C1	−175.63 (13)	C9—C8—C14—C13	150.28 (14)
C4—C5—C6—C7	174.83 (13)	C7—C8—C14—C13	26.0 (2)
C10—C5—C6—C7	−3.3 (2)	C6—C7—C15—C16	60.26 (16)
C1—C6—C7—C15	77.55 (16)	C8—C7—C15—C16	−58.62 (17)
C5—C6—C7—C15	−94.43 (16)	C11—C7—C15—C16	−172.80 (13)
C1—C6—C7—C8	−166.27 (12)	C19—N1—C16—C15	−179.28 (13)
C5—C6—C7—C8	21.76 (18)	C9—N1—C16—C15	−51.32 (19)
C1—C6—C7—C11	−46.83 (19)	C7—C15—C16—N1	52.98 (18)
C5—C6—C7—C11	141.20 (14)	C1—O1—C20—C21	175.38 (12)

C15—C7—C8—C14	−171.75 (13)	O1—C20—C21—C26	14.3 (2)
C6—C7—C8—C14	71.16 (16)	O1—C20—C21—C22	−167.91 (13)
C11—C7—C8—C14	−53.97 (17)	C26—C21—C22—C23	0.2 (2)
C15—C7—C8—C9	63.25 (16)	C20—C21—C22—C23	−177.70 (16)
C6—C7—C8—C9	−53.84 (16)	C21—C22—C23—C24	−0.5 (3)
C11—C7—C8—C9	−178.96 (12)	C22—C23—C24—F1	179.61 (18)
C19—N1—C9—C8	−178.64 (13)	C22—C23—C24—C25	0.4 (3)
C16—N1—C9—C8	54.42 (18)	F1—C24—C25—C26	−179.18 (17)
C19—N1—C9—C10	58.37 (18)	C23—C24—C25—C26	0.0 (3)
C16—N1—C9—C10	−68.57 (18)	C22—C21—C26—C25	0.2 (3)
C14—C8—C9—N1	173.90 (13)	C20—C21—C26—C25	178.05 (17)
C7—C8—C9—N1	−60.41 (17)	C24—C25—C26—C21	−0.4 (3)
C14—C8—C9—C10	−58.15 (17)		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2S—H22S···O4	0.91	2.33	2.9805 (16)	128
O2S—H22S···O3	0.91	2.54	3.417 (2)	164
O1S—H11S···O2S	0.90 (3)	1.94 (3)	2.8342 (19)	172 (2)
O2S—H21S···N1^{i}	0.97	1.81	2.7736 (19)	170

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

Fig. 1

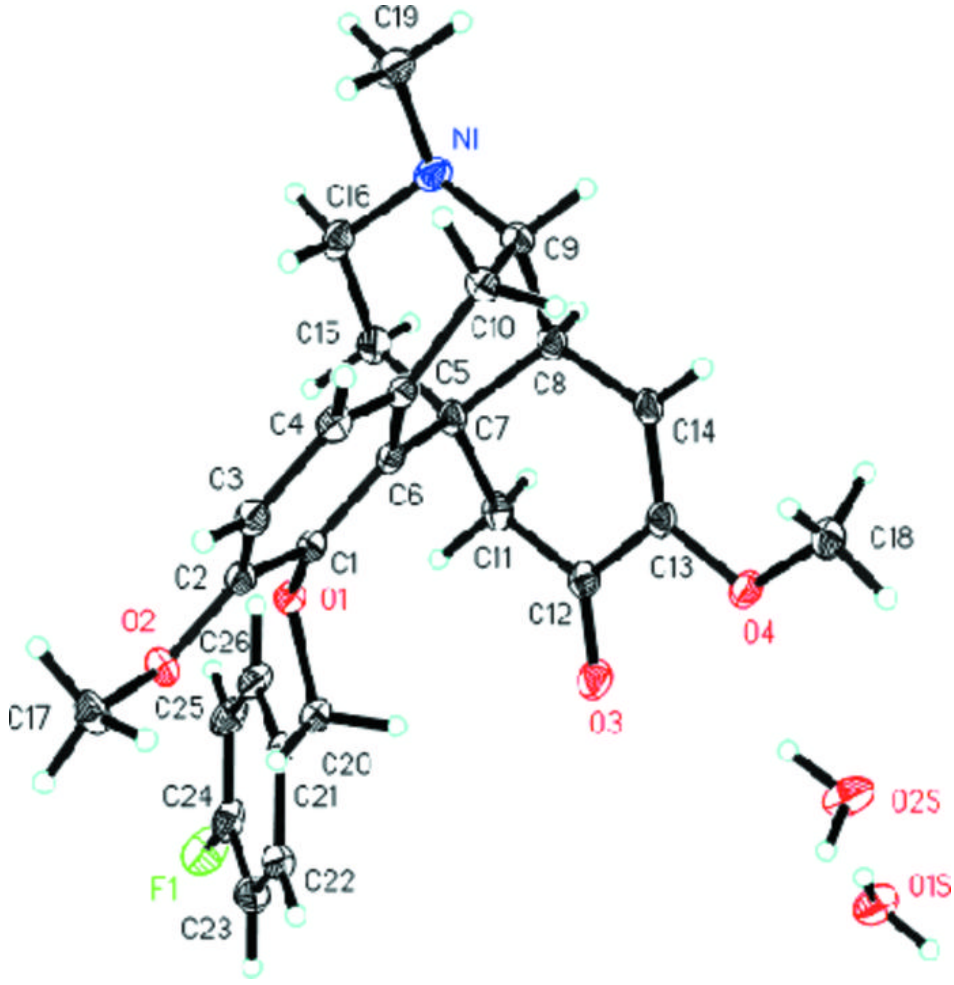
C19
NI
C16
C9
C15
C10
C5
C8
C4
C7
C14
C3
C6
C18
C2
C1
C11
C13
O2
O1
C12
O4
C26
C17
C25
O3
C20
C24
C21
O2S
F1
C22
O1S
C23

Fig. 2