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Ethyl 6-ethoxy-10-(3-methoxypropyl)-9-methyl-11-thioxo-8-oxa-10,12-diaza-tricyclo[7.3.1.0^{2,7}]trideca-2,4,6-triene-13-carboxylate

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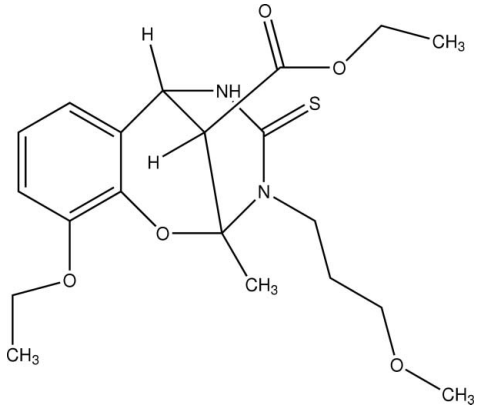
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.003 Å; R factor = 0.038; wR factor = 0.066; data-to-parameter ratio = 14.5.

Comparison of the title compound, $C_{20}H_{28}N_2O_5S$, with related structures shows that the presence of the alkyl substituents influences the conformation of the rings in the frame fragment. The molecules form hydrogen-bonded centrosymmetric dimers due to an N—H···S intermolecular interaction.

Related literature

For related literature, see: Baldwin *et al.* (1986); Biginelli, (1893); Fu *et al.* (2002); Kettmann & Svetlík (1996, 1997); Kettmann *et al.* (1996); Zefirov & Zorky (1995).



Experimental

Crystal data

$C_{20}H_{28}N_2O_5S$
M_r = 408.50
Triclinic, $P\overline{1}$
a = 9.7612 (16) Å
b = 10.3405 (17) Å
c = 12.309 (2) Å
α = 107.354 (13)°
β = 109.709 (13)°
γ = 100.096 (12)°
V = 1062.5 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 293 (2) K
0.40 × 0.20 × 0.20 mm

Data collection

Siemens $P3$/PC diffractometer
Absorption correction: none
3927 measured reflections
3737 independent reflections
2151 reflections with $I > 2\sigma(I)$
R_{int} = 0.053
2 standard reflections
every 98 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.066
S = 0.89
3737 reflections
257 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.12 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1N···S1^{i}	0.86	2.62	3.3973 (17)	152

Symmetry code: (i) $-x+1, -y+1, -z+1$.

Data collection: *P3* (Siemens, 1989); cell refinement: *P3*; data reduction: *XDISK* (Siemens, 1991); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

The authors thank Svitlana V. Shishkina for help in the preparation of the manuscript and for valuable discussions.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BG2144).

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supplementary materials

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Ethyl 6-ethoxy-10-(3-methoxypropyl)-9-methyl-11-thioxo-8-oxa-10,12-diazatricyclo[7.3.1.$0^{2,7}$]trideca-2,4,6-triene-13-carboxylate

I. S. Konovalova, O. V. Zaremba, S. S. Kovalenko, V. P. Chernykh, S. M. Kovalenko, V. N. Baumer and O. V. Shishkin

Comment

Some derivatives of 2,6-methano-2*H*-1,3,5-benzothiadiazocine and -benzoxadiazocine have been studied due to their biological activity as perspective calcium channel blockers (Baldwin *et al.*, 1986; Kettmann *et al.*, 1996; Kettmann & Svetlík, 1996, 1997). We have performed an X-ray diffraction study of a new member of this group, the title compound $C_{20}H_{28}N_2O_5S_1$ (I). Tetrahydropyrimidine-2-thione and tetrahydropyran rings adopt in (I) a conformation which is intermediate between half-chair and sofa. Maximum deviations from the tetrahydropyrimidine and the tetrahydropyrane mean planes correspond to atoms C1, C10 (0.28 A°, −0.54 A°) and C8,C10 (0.21 A°, −0.59 A°), respectively. Comparison of the ring conformations in (I) and some of its analogues (Kettmann *et al.*, 1996*b*; Fu *et al.*, 2002; Kettmann *et al.*, 1997) indicates that the presence of substituent at the nitrogen atoms is probably the reason for some deformation in the heterocycles conformation. The ester substituent in (I) is planar within 0.01 A° and coplanar to the C8—C10 bond (the C8—C10—C11—O2 torsion angle is 2.2 (3) °). The methoxybuthyl substituent adopts instead an orthogonal orientation with respect to the tetrahydropyrimidine ring (the C9—N2—C16—C17 torsion angle is −92.7 (2) °) probably due to the repulsion between some of its atoms and the tione and the methyl groups (as sugested by the short intramolecular contacts C20···H16A 2.43 Å (van der Waals radii sum is 2.87 Å, Zefirov & Zorky, 1995), H16A···H20C 2.12 Å [2.32 Å], S1···H16B is 2.53 Å [3.01 Å]). The ethoxy substituent is turned with respect to the plane of the aromatic ring (the C5—C6—O4—C14 torsion angle is 17.6 (3) °) probably due to the repulsion between atoms of these fragments (shortened intramolecular contacts C14···H5 2.59 Å [van der Waals radii sum is 2.87 Å], C5···H14A 2.71 Å [2.87 Å]). In the crystal phase the molecules of (I) form centrosymmetric dimers due to a weak intermolecular hydrogen bond (Table 1).

Experimental

The synthesis was carried out according to the Biginelli reaction (Biginelli, 1893). 2-Hydroxy-3-etoxysalicylic aldehyde (0,01 mol), *N*-(3-methoxypropyl)-thiourea and ethyl acetoacetate (0,015 mol) were mixed in 10 ml of ethanol. 3 drops of concentrated HCL were added. Raw crystals of the title compound were grown during 2 days at room temperature; they were further filtered, washed in ethanol and dried at room temperature. Crystals apt for *x*-ray diffraction were obtained by quick evaporation of a solution of (I) in dimethylformamide.

Refinement

All hydrogen atoms were located from electron density difference maps, idealized and included in the refinement in the riding approximation with U_{iso} constrained to be 1.5 times U_{eq} of the carrier atom for the methyl groups and 1.2 times U_{eq} of the carrier atom for the other atoms.

Figures

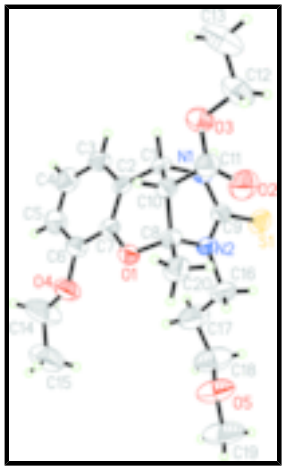


Fig. 1. Molecular view of the title compound with atomic numbering. Displacement ellipsoids drawn at the 50% probability level.

Ethyl 6-ethoxy-10-(3-methoxypropyl)-9-methyl-11-thioxo-8-oxa-10,12- diazatricyclo[7.3.1.$0^{2,7}$]trideca-2,4,6-triene-13-carboxylate

Crystal data

$C_{20}H_{28}N_2O_5S$	$Z = 2$
$M_r = 408.50$	$F_{000} = 436$
Triclinic, $P\overline{1}$	$D_x = 1.277$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 9.7612$ (16) Å	Cell parameters from 36 reflections
$b = 10.3405$ (17) Å	$\theta = 8$–22º
$c = 12.309$ (2) Å	$\mu = 0.19$ mm^{-1}
$\alpha = 107.354$ (13)º	$T = 293$ (2) K
$\beta = 109.709$ (13)º	Block, colourless
$\gamma = 100.096$ (12)º	0.40 × 0.20 × 0.20 mm
$V = 1062.5$ (4) Å^3	

Data collection

Siemens P3/PC diffractometer	$R_{int} = 0.053$
Radiation source: fine-focus sealed tube	$\theta_{max} = 25.0$º
Monochromator: graphite	$\theta_{min} = 2.2$º
$T = 293(2)$ K	$h = -11 \rightarrow 10$
$\theta/2\theta$ scans	$k = -12 \rightarrow 11$
Absorption correction: none	$l = 0 \rightarrow 14$
3927 measured reflections	2 standard reflections
3737 independent reflections	every 98 reflections
2151 reflections with $I > 2\sigma(I)$	intensity decay: ?

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	H-atom parameters constrained
$wR(F^2) = 0.066$	$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.89$	$(\Delta/\sigma)_{max} = 0.001$
3737 reflections	$\Delta\rho_{max} = 0.19$ e Å^{-3}
257 parameters	$\Delta\rho_{min} = -0.12$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s 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 $F^2 > 2$sigma(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}
S1	0.33397 (6)	0.30050 (6)	0.46821 (5)	0.05561 (17)
C1	0.51711 (19)	0.33456 (17)	0.22987 (15)	0.0366 (4)
H1	0.5496	0.4221	0.2172	0.044*
C2	0.64837 (19)	0.27649 (17)	0.26528 (15)	0.0360 (4)
C3	0.8001 (2)	0.36494 (19)	0.33397 (16)	0.0447 (5)
H3	0.8220	0.4630	0.3576	0.054*
C4	0.9156 (2)	0.3074 (2)	0.36622 (18)	0.0559 (6)
H4	1.0160	0.3669	0.4110	0.067*
C5	0.8859 (2)	0.1622 (2)	0.33345 (18)	0.0560 (6)
H5	0.9659	0.1246	0.3567	0.067*
C6	0.7364 (2)	0.0724 (2)	0.26571 (17)	0.0471 (5)
C7	0.61870 (19)	0.13107 (18)	0.23001 (16)	0.0365 (4)
O1	0.47460 (13)	0.03496 (12)	0.15539 (11)	0.0435 (3)
C8	0.34663 (19)	0.09174 (18)	0.13639 (16)	0.0404 (5)
N2	0.31829 (16)	0.12553 (14)	0.25062 (13)	0.0401 (4)
C9	0.37102 (19)	0.25931 (18)	0.33937 (16)	0.0384 (4)
N1	0.45580 (16)	0.36240 (14)	0.32376 (13)	0.0405 (4)
H1N	0.4750	0.4493	0.3715	0.049*
C10	0.38594 (19)	0.22459 (17)	0.11061 (16)	0.0382 (4)

H10	0.4230	0.2022	0.0446	0.046*
C11	0.2556 (2)	0.2852 (2)	0.07013 (18)	0.0513 (5)
O2	0.12758 (17)	0.24063 (17)	0.06021 (17)	0.0882 (5)
O3	0.30182 (15)	0.39880 (15)	0.04911 (12)	0.0590 (4)
C12	0.1892 (3)	0.4702 (3)	0.0106 (2)	0.0770 (7)
H12A	0.1508	0.5006	0.0740	0.115*
H12B	0.1038	0.4057	−0.0671	0.115*
C13	0.2648 (3)	0.5951 (3)	−0.0066 (3)	0.1075 (10)
H13A	0.3445	0.6614	0.0721	0.161*
H13B	0.1910	0.6403	−0.0375	0.161*
H13C	0.3081	0.5645	−0.0656	0.161*
O4	0.69411 (16)	−0.07243 (14)	0.22771 (13)	0.0640 (4)
C14	0.7999 (3)	−0.1341 (3)	0.2869 (3)	0.1045 (10)
H14A	0.8463	−0.0810	0.3767	0.157*
H14B	0.8803	−0.1313	0.2573	0.157*
C15	0.7188 (4)	−0.2819 (3)	0.2576 (2)	0.1044 (10)
H15A	0.6465	−0.2834	0.2946	0.157*
H15B	0.7909	−0.3275	0.2906	0.157*
H15C	0.6658	−0.3316	0.1686	0.157*
C16	0.2414 (2)	0.0061 (2)	0.27311 (19)	0.0579 (6)
H16A	0.1618	−0.0635	0.1944	0.087*
H16B	0.1933	0.0414	0.3282	0.087*
C17	0.3498 (3)	−0.0645 (3)	0.3302 (2)	0.0807 (7)
H17A	0.4396	0.0082	0.3992	0.121*
H17B	0.3825	−0.1153	0.2684	0.121*
C18	0.2826 (3)	−0.1691 (3)	0.3784 (3)	0.0983 (9)
H18A	0.3637	−0.1959	0.4291	0.148*
H18B	0.2353	−0.1236	0.4304	0.148*
O5	0.17768 (19)	−0.28630 (17)	0.28194 (14)	0.0856 (5)
C19	0.1400 (4)	−0.3987 (3)	0.3224 (3)	0.1141 (11)
H19A	0.2311	−0.4201	0.3624	0.171*
H19B	0.0684	−0.4819	0.2514	0.171*
H19C	0.0954	−0.3692	0.3805	0.171*
C20	0.2177 (2)	−0.02969 (19)	0.02500 (17)	0.0551 (6)
H20A	0.2327	−0.0366	−0.0495	0.083*
H20B	0.1220	−0.0122	0.0166	0.083*
H20C	0.2168	−0.1172	0.0373	0.083*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S1	0.0581 (3)	0.0510 (3)	0.0540 (3)	0.0063 (3)	0.0338 (3)	0.0092 (3)
C1	0.0397 (11)	0.0283 (9)	0.0453 (11)	0.0049 (8)	0.0210 (9)	0.0179 (9)
C2	0.0405 (11)	0.0346 (10)	0.0368 (10)	0.0070 (9)	0.0215 (9)	0.0151 (8)
C3	0.0422 (12)	0.0348 (10)	0.0487 (12)	0.0044 (9)	0.0149 (10)	0.0137 (9)
C4	0.0366 (12)	0.0634 (15)	0.0571 (14)	0.0079 (11)	0.0116 (10)	0.0225 (11)
C5	0.0446 (13)	0.0622 (14)	0.0630 (14)	0.0221 (11)	0.0191 (11)	0.0268 (11)
C6	0.0553 (13)	0.0416 (12)	0.0527 (13)	0.0192 (11)	0.0271 (11)	0.0211 (10)

C7	0.0349 (10)	0.0351 (10)	0.0399 (11)	0.0082 (9)	0.0162 (9)	0.0153 (9)
O1	0.0399 (8)	0.0322 (7)	0.0547 (8)	0.0098 (6)	0.0201 (6)	0.0120 (6)
C8	0.0342 (11)	0.0348 (10)	0.0453 (12)	0.0045 (9)	0.0151 (9)	0.0113 (9)
N2	0.0434 (9)	0.0289 (8)	0.0488 (9)	0.0050 (7)	0.0250 (8)	0.0131 (7)
C9	0.0329 (10)	0.0321 (10)	0.0455 (11)	0.0074 (8)	0.0140 (9)	0.0129 (9)
N1	0.0485 (10)	0.0244 (8)	0.0482 (10)	0.0090 (7)	0.0260 (8)	0.0082 (7)
C10	0.0358 (10)	0.0355 (10)	0.0365 (11)	0.0043 (8)	0.0128 (9)	0.0115 (8)
C11	0.0490 (14)	0.0498 (13)	0.0529 (13)	0.0130 (11)	0.0176 (11)	0.0222 (10)
O2	0.0411 (9)	0.0877 (12)	0.1408 (16)	0.0196 (8)	0.0280 (10)	0.0619 (11)
O3	0.0648 (9)	0.0600 (9)	0.0669 (10)	0.0277 (8)	0.0284 (8)	0.0378 (8)
C12	0.102 (2)	0.0726 (17)	0.0708 (16)	0.0537 (16)	0.0323 (15)	0.0360 (14)
C13	0.141 (3)	0.0629 (18)	0.111 (2)	0.0521 (18)	0.035 (2)	0.0318 (17)
O4	0.0663 (10)	0.0432 (9)	0.0851 (11)	0.0258 (7)	0.0271 (8)	0.0281 (8)
C14	0.118 (2)	0.0620 (18)	0.132 (2)	0.0471 (17)	0.032 (2)	0.0467 (17)
C15	0.164 (3)	0.0660 (18)	0.110 (2)	0.0559 (18)	0.068 (2)	0.0463 (16)
C16	0.0695 (15)	0.0403 (12)	0.0606 (14)	0.0024 (11)	0.0344 (12)	0.0147 (10)
C17	0.0720 (16)	0.0715 (16)	0.0985 (19)	0.0092 (14)	0.0266 (15)	0.0502 (15)
C18	0.109 (2)	0.0844 (19)	0.095 (2)	0.0057 (17)	0.0273 (18)	0.0573 (17)
O5	0.1038 (13)	0.0639 (11)	0.0701 (11)	−0.0084 (10)	0.0246 (10)	0.0334 (9)
C19	0.164 (3)	0.0719 (18)	0.115 (2)	0.0061 (19)	0.063 (2)	0.0579 (17)
C20	0.0458 (12)	0.0484 (13)	0.0493 (12)	0.0006 (10)	0.0119 (10)	0.0067 (10)

Geometric parameters (Å, °)

S1—C9	1.6927 (18)	C12—C13	1.481 (3)
C1—N1	1.451 (2)	C12—H12A	0.9700
C1—C2	1.497 (2)	C12—H12B	0.9700
C1—C10	1.521 (2)	C13—H13A	0.9600
C1—H1	0.9800	C13—H13B	0.9600
C2—C7	1.381 (2)	C13—H13C	0.9600
C2—C3	1.401 (2)	O4—C14	1.416 (3)
C3—C4	1.362 (3)	C14—C15	1.466 (3)
C3—H3	0.9300	C14—H14A	0.9700
C4—C5	1.382 (3)	C14—H14B	0.9700
C4—H4	0.9300	C15—H15A	0.9600
C5—C6	1.389 (3)	C15—H15B	0.9600
C5—H5	0.9300	C15—H15C	0.9600
C6—O4	1.367 (2)	C16—C17	1.490 (3)
C6—C7	1.394 (2)	C16—H16A	0.9700
C7—O1	1.3798 (19)	C16—H16B	0.9700
O1—C8	1.450 (2)	C17—C18	1.532 (3)
C8—N2	1.476 (2)	C17—H17A	0.9700
C8—C10	1.514 (2)	C17—H17B	0.9700
C8—C20	1.515 (2)	C18—O5	1.346 (3)
N2—C9	1.357 (2)	C18—H18A	0.9700
N2—C16	1.478 (2)	C18—H18B	0.9700
C9—N1	1.334 (2)	O5—C19	1.432 (2)
N1—H1N	0.8600	C19—H19A	0.9600
C10—C11	1.516 (3)	C19—H19B	0.9600

C10—H10	0.9800	C19—H19C	0.9600
C11—O2	1.204 (2)	C20—H20A	0.9600
C11—O3	1.313 (2)	C20—H20B	0.9600
O3—C12	1.456 (2)	C20—H20C	0.9600
N1—C1—C2	111.76 (14)	O3—C12—H12B	110.1
N1—C1—C10	105.55 (13)	C13—C12—H12B	110.1
C2—C1—C10	109.75 (14)	H12A—C12—H12B	108.4
N1—C1—H1	109.9	C12—C13—H13A	109.5
C2—C1—H1	109.9	C12—C13—H13B	109.5
C10—C1—H1	109.9	H13A—C13—H13B	109.5
C7—C2—C3	118.97 (17)	C12—C13—H13C	109.5
C7—C2—C1	118.86 (15)	H13A—C13—H13C	109.5
C3—C2—C1	122.17 (16)	H13B—C13—H13C	109.5
C4—C3—C2	120.08 (18)	C6—O4—C14	118.18 (17)
C4—C3—H3	120.0	O4—C14—C15	108.7 (2)
C2—C3—H3	120.0	O4—C14—H14A	109.9
C3—C4—C5	121.07 (19)	C15—C14—H14A	109.9
C3—C4—H4	119.5	O4—C14—H14B	109.9
C5—C4—H4	119.5	C15—C14—H14B	109.9
C4—C5—C6	119.90 (19)	H14A—C14—H14B	108.3
C4—C5—H5	120.1	C14—C15—H15A	109.5
C6—C5—H5	120.1	C14—C15—H15B	109.5
O4—C6—C5	124.83 (18)	H15A—C15—H15B	109.5
O4—C6—C7	116.18 (17)	C14—C15—H15C	109.5
C5—C6—C7	118.97 (18)	H15A—C15—H15C	109.5
O1—C7—C2	123.19 (16)	H15B—C15—H15C	109.5
O1—C7—C6	115.82 (15)	N2—C16—C17	112.23 (17)
C2—C7—C6	120.96 (16)	N2—C16—H16A	109.2
C7—O1—C8	117.03 (13)	C17—C16—H16A	109.2
O1—C8—N2	108.40 (14)	N2—C16—H16B	109.2
O1—C8—C10	108.89 (13)	C17—C16—H16B	109.2
N2—C8—C10	110.53 (14)	H16A—C16—H16B	107.9
O1—C8—C20	102.78 (14)	C16—C17—C18	114.0 (2)
N2—C8—C20	112.72 (15)	C16—C17—H17A	108.8
C10—C8—C20	113.09 (15)	C18—C17—H17A	108.8
C9—N2—C8	122.51 (14)	C16—C17—H17B	108.8
C9—N2—C16	119.32 (15)	C18—C17—H17B	108.8
C8—N2—C16	117.94 (14)	H17A—C17—H17B	107.7
N1—C9—N2	118.16 (16)	O5—C18—C17	110.4 (2)
N1—C9—S1	118.88 (13)	O5—C18—H18A	109.6
N2—C9—S1	122.96 (13)	C17—C18—H18A	109.6
C9—N1—C1	122.96 (14)	O5—C18—H18B	109.6
C9—N1—H1N	118.5	C17—C18—H18B	109.6
C1—N1—H1N	118.5	H18A—C18—H18B	108.1
C8—C10—C11	115.46 (15)	C18—O5—C19	111.30 (18)
C8—C10—C1	106.56 (14)	O5—C19—H19A	109.5
C11—C10—C1	110.17 (14)	O5—C19—H19B	109.5
C8—C10—H10	108.1	H19A—C19—H19B	109.5
C11—C10—H10	108.1	O5—C19—H19C	109.5

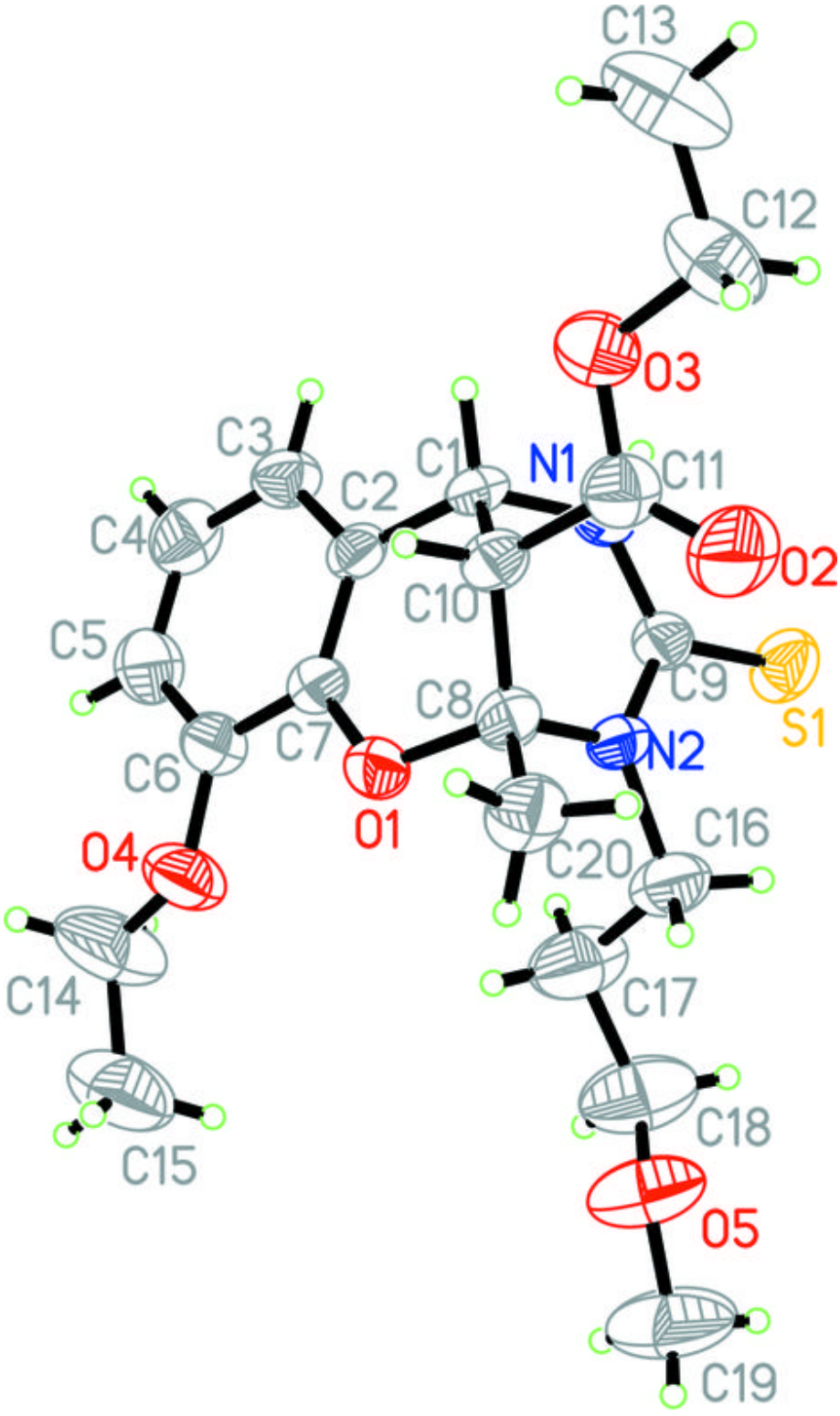
C1—C10—H10	108.1	H19A—C19—H19C	109.5
O2—C11—O3	123.1 (2)	H19B—C19—H19C	109.5
O2—C11—C10	126.59 (19)	C8—C20—H20A	109.5
O3—C11—C10	110.29 (17)	C8—C20—H20B	109.5
C11—O3—C12	116.75 (17)	H20A—C20—H20B	109.5
O3—C12—C13	108.0 (2)	C8—C20—H20C	109.5
O3—C12—H12A	110.1	H20A—C20—H20C	109.5
C13—C12—H12A	110.1	H20B—C20—H20C	109.5
N1—C1—C2—C7	88.77 (18)	C8—N2—C9—S1	177.75 (13)
C10—C1—C2—C7	−28.0 (2)	C16—N2—C9—S1	−7.9 (2)
N1—C1—C2—C3	−90.64 (19)	N2—C9—N1—C1	−11.6 (2)
C10—C1—C2—C3	152.62 (16)	S1—C9—N1—C1	168.40 (12)
C7—C2—C3—C4	−0.7 (3)	C2—C1—N1—C9	−74.0 (2)
C1—C2—C3—C4	178.67 (16)	C10—C1—N1—C9	45.3 (2)
C2—C3—C4—C5	−0.7 (3)	O1—C8—C10—C11	171.51 (14)
C3—C4—C5—C6	0.5 (3)	N2—C8—C10—C11	−69.51 (19)
C4—C5—C6—O4	179.55 (18)	C20—C8—C10—C11	57.9 (2)
C4—C5—C6—C7	1.2 (3)	O1—C8—C10—C1	−65.80 (17)
C3—C2—C7—O1	−175.56 (15)	N2—C8—C10—C1	53.17 (18)
C1—C2—C7—O1	5.0 (2)	C20—C8—C10—C1	−179.38 (15)
C3—C2—C7—C6	2.4 (2)	N1—C1—C10—C8	−63.55 (17)
C1—C2—C7—C6	−177.02 (16)	C2—C1—C10—C8	57.03 (17)
O4—C6—C7—O1	−3.0 (2)	N1—C1—C10—C11	62.40 (18)
C5—C6—C7—O1	175.50 (16)	C2—C1—C10—C11	−177.02 (15)
O4—C6—C7—C2	178.85 (15)	C8—C10—C11—O2	2.2 (3)
C5—C6—C7—C2	−2.6 (3)	C1—C10—C11—O2	−118.6 (2)
C2—C7—O1—C8	−13.4 (2)	C8—C10—C11—O3	−179.32 (15)
C6—C7—O1—C8	168.57 (15)	C1—C10—C11—O3	59.9 (2)
C7—O1—C8—N2	−76.22 (17)	O2—C11—O3—C12	−0.8 (3)
C7—O1—C8—C10	44.07 (19)	C10—C11—O3—C12	−179.37 (15)
C7—O1—C8—C20	164.25 (14)	C11—O3—C12—C13	179.40 (18)
O1—C8—N2—C9	98.62 (17)	C5—C6—O4—C14	17.6 (3)
C10—C8—N2—C9	−20.7 (2)	C7—C6—O4—C14	−164.0 (2)
C20—C8—N2—C9	−148.30 (16)	C6—O4—C14—C15	164.75 (18)
O1—C8—N2—C16	−75.83 (18)	C9—N2—C16—C17	−92.7 (2)
C10—C8—N2—C16	164.90 (15)	C8—N2—C16—C17	81.9 (2)
C20—C8—N2—C16	37.2 (2)	N2—C16—C17—C18	168.78 (19)
C8—N2—C9—N1	−2.3 (2)	C16—C17—C18—O5	70.9 (3)
C16—N2—C9—N1	172.10 (16)	C17—C18—O5—C19	166.2 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1N···S1^{i}	0.86	2.62	3.3973 (17)	152

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

Fig. 1


C13
C12
O3
C3
C1
N1
C11
C2
C4
O2
C10
C5
C9
S1
C8
C7
N2
C6
O1
C16
O4
C20
C14
C17
C18
C15
O5
C19