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Crystal structure of (1*S*,2*R*,4*R*,9*S*,11*S*,12*R*)-9α-hydroxy-4,8-dimethyl-12-[(thiomorpholin-4-yl)methyl]-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one

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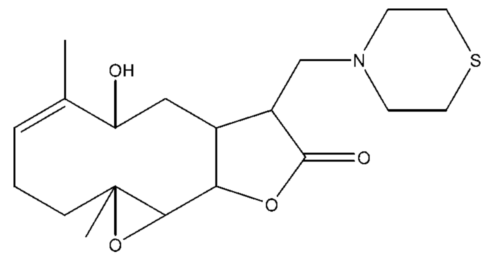
The title compound, $C_{19}H_{29}NO_4S$, was synthesised from 9α-hydroxyparthenolide (9α-hydroxy-4,8-dimethyl-12-methylene-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one), which was isolated from the chloroform extract of the aerial parts of the plant *Anvillea radiata*. The molecule is built up from two fused five- and ten-membered rings, with an additional epoxy ring system and a thiomorpholine group as a substituent. The ten-membered ring adopts an approximate chair–chair conformation, while the thiomorpholine ring displays a chair conformation and the five-membered ring has an envelope conformation, with the C atom closest to the hydroxy group forming the flap. An intramolecular O—H···N hydrogen bond closes an *S*(8) ring. The crystal structure features weak C—H···O hydrogen-bonding interactions, which link the molecules into [010] chains.

Keywords: crystal structure; *Anvillea radiata*; medicinal compound; hydrogen bonding.

CCDC reference: 1045641

1. Related literature

For background to the medicinal uses of the plant *Anvillea radiata*, see: El Hassany *et al.* (2004); Abdel Sattar *et al.* (1996). For the reactivity of this sesquiterpene, see: Hwang *et al.* (2006); Neelakantan *et al.* (2009); Loubidi *et al.* (2014).



2. Experimental

2.1. Crystal data

$C_{19}H_{29}NO_4S$
M_r = 367.49
Monoclinic, $P2_1$
a = 11.920 (2) Å
b = 6.7919 (13) Å
c = 12.144 (3) Å
β = 101.659 (6)°
V = 962.9 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.19 mm^{-1}
T = 296 K
0.33 × 0.17 × 0.04 mm

2.2. Data collection

Bruker APEXII CCD diffractometer
12288 measured reflections
3940 independent reflections
3749 reflections with $I > 2\sigma(I)$
R_{int} = 0.033

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.03
3940 reflections
229 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack & Bernardinelli (2000), 1799 Friedel pairs
Absolute structure parameter: 0.04 (7)

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4···N2	0.82	2.21	3.0278 (18)	176
C2—H2···O1^{i}	0.98	2.51	3.2948 (19)	137
C6—H6···O2^{ii}	0.93	2.59	3.2526 (19)	129

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT-Plus* (Bruker, 2009); data reduction: *SAINT-Plus*; 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: *WinGX* (Farrugia, 2012).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: HB7355).

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supporting information

Acta Cryst. (2015). E**71**, o140–o141 [doi:10.1107/S205698901500170X]

Crystal structure of (1*S*,2*R*,4*R*,9*S*,11*S*,12*R*)-9*α*-hydroxy-4,8-dimethyl-12-[(thiomorpholin-4-yl)methyl]-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one

Ahmed Benharref, Mohamed Akssira, Lahcen El Ammari, Mohamed Saadi and Moha Berraho

S1. Comment

The natural sesquiterpene lactone, 9*α* -hydroxypartenolide is the main constituent of the chloroform extract of the aerial parts of Anvillea radiata (El Hassany et al., 2004; Qureshi et al.) and of Anvillea garcini (Abdel Sattar et al.,1996). The reactivity of this sesquiterpene lactone and its derivatives have been the subject of several studies (Hwang et al., 2006; Neelakantan et al., 2009; Loubidi et al., 2014), in order to prepare products with high value which can be used in the pharmacological industry. In this context, we have treated the 9*α*-hydroxy-parthenolide with an equivalent amount of thiomorpholine and prepared the 9*α*-Hydroxy-12- [thiomorpholin-N- methyl]-4,8-dimethyl-3,14-dioxatricyclo [9.3.0.0^2,^{4}] tetradec-7-en-13-one. The structure of this new product was confirmed by its single crystal X-ray structure. The molecule contains a fused ring system and thiomorpholin group as a substituent to a lactone ring. The molecular structure of (I), Fig.1, shows the lactone ring to adopt an envelope conformation, as indicated by puckering parameters Q = 0.2241 (15) Å and φ = 291.3 (4)°. The atom C10 deviate from the mean plane through other four atoms in the ring by 0.3601 (13)Å. The ten-membered ring displays an approximate chair-chair conformation, while the thiomorpholin ring has an almost ideal chair conformation with QT = 0.6400 (17)Å, θ = 178.37 (15)° and φ = 244 (4)°. In the crystal structure, the molecules are linked by C—H···O intermolecular hydrogen bonds into chains along the b axis (Table 1, Fig.2). In addition an intramolecular O—H···N, hydrogen bond is also observed.

S2. Experimental

The mixture of 9*α*-hydoxypartenolide (9*α*-hydroxy-4,8-dimethyl- 12-methylene- 3,14-dioxatricyclo[9.3.0.0^2,^{4}]tetradec-7-en-13-one) (1 g, 3.8 mmol) and one equivalent of thiomorpholin in EtOH (20 ml) was stirred for ten hours at room temperature. Then the reaction was stopped by adding water (10 ml) and the solution was extracted with chloroform (3 *x* 20 ml). The combined organic layers were dried over anhydrous $MgSO_4$, filtered and concentrated under vacuum to give 0.9 g(2.5 mmol) of the title compound (yield: 66%). Recrystallization was performed from ethyl acetate solution to yield colourless plates.

S3. Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.96 Å (methyl),0.97 Å (methylene), 0.98 Å (methine) with U_{iso}(H) = 1.2Ueq(methylene, methine and OH) or U_{iso}(H) = 1.5Ueq(methyl). Owing to the presence of S atom, the absolute configuration could be fully confirmed, by refining the Flack parameter (Flack & Bernardinelli (2000) as C1(S), C2(*R*), C4(*R*), C9(S), C11(S) and C2(*R*).

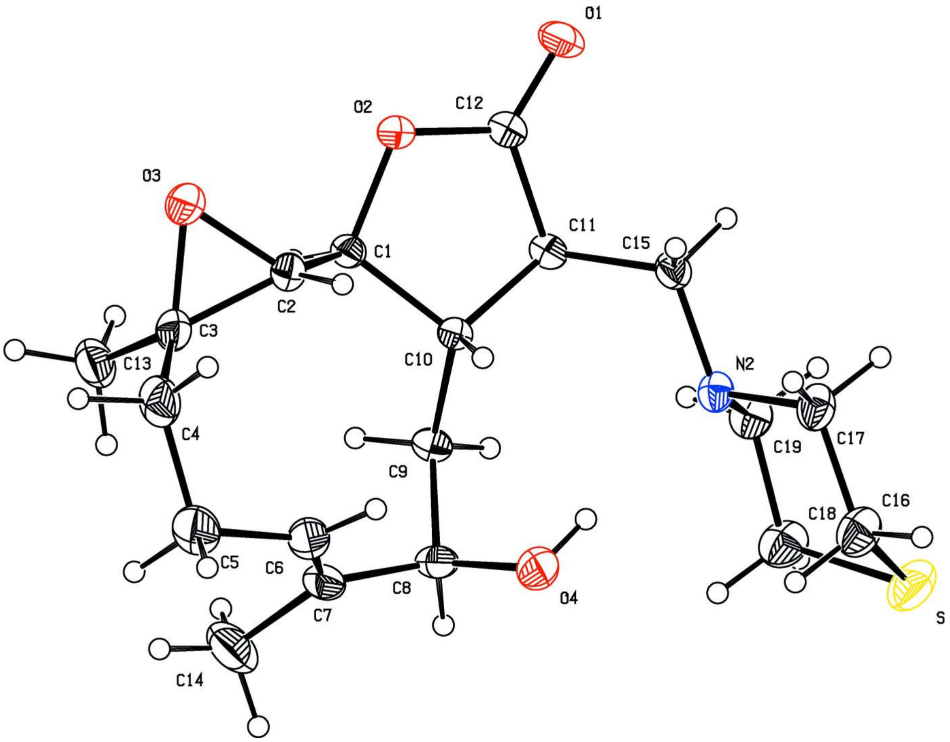

Figure 1

Molecular structure of the title compound with displacement ellipsoids drawn at the 30% probability level.

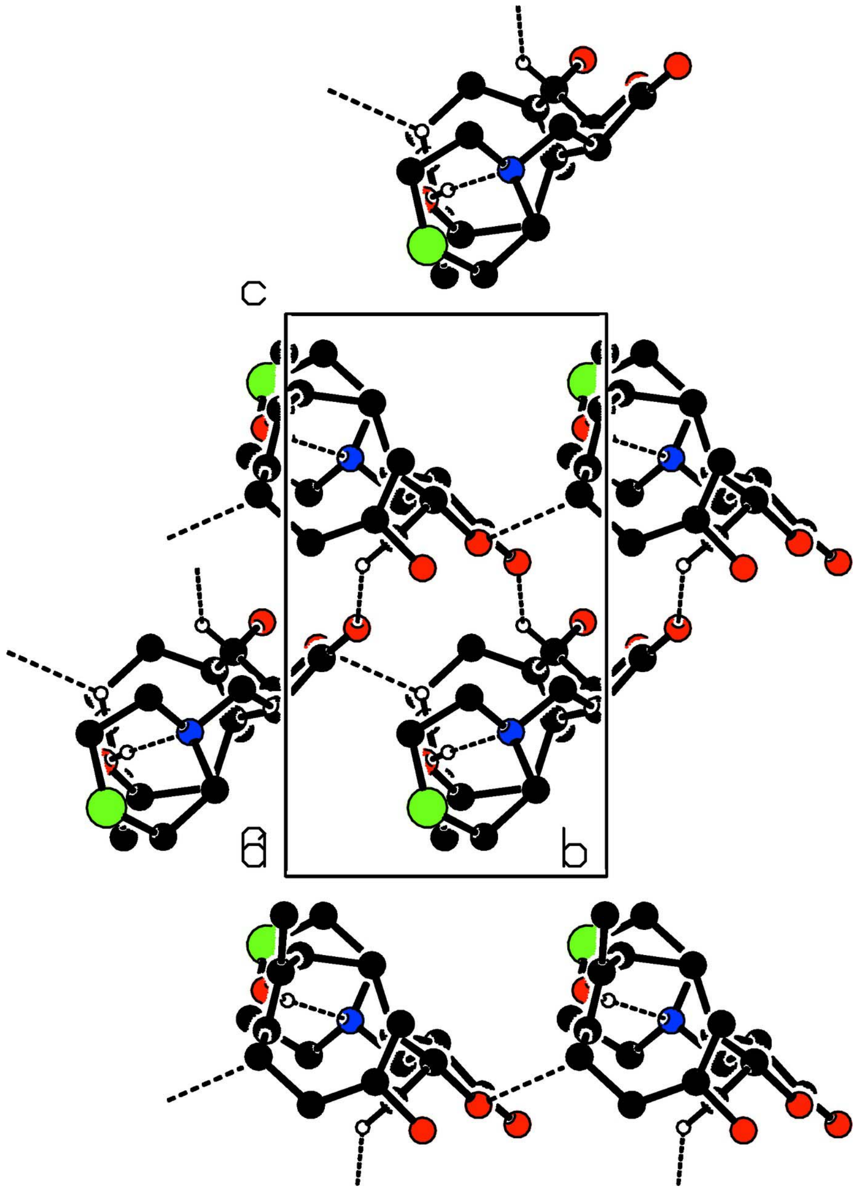

Figure 2

Partial packing view showing the C—H···O interactions (dashed lines) and the formation of a chain parallel to the *b* axis. H atoms not involved in hydrogen bonding have been omitted for clarity. [Symmetry code: (i) *x*, -1 + *y*, *z*.]

(1*S*,2*R*,4*R*,9*S*,11*S*,12*R*)-9*α*-Hydroxy-4,8-dimethyl-12-[(thiomorpholin-4-yl)methyl]-3,14-dioxatricyclo[9.3.0.0^{2,4}]tetradec-7-en-13-one

Crystal data

$C_{19}H_{29}NO_4S$	β = 101.659 (6)°
M_r = 367.49	V = 962.9 (3) Å^3
Monoclinic, $P2_1$	Z = 2
Hall symbol: P 2yb	F(000) = 396
a = 11.920 (2) Å	D_x = 1.268 Mg m^{-3}
b = 6.7919 (13) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
c = 12.144 (3) Å	Cell parameters from 3940 reflections

θ = 2.7–26.4°
μ = 0.19 mm^{-1}
T = 296 K
Platelet, colourless
0.33 × 0.17 × 0.04 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω and φ scans
12288 measured reflections
3940 independent reflections
3749 reflections with $I > 2\sigma(I)$
R_{int} = 0.033
θ_{max} = 26.4°, θ_{min} = 2.7°
h = −14→14
k = −8→8
l = −15→15

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.083
S = 1.03
3940 reflections
229 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0458P)^2 + 0.1195P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ < 0.001
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.18 e Å^{-3}
Absolute structure: Flack & Bernardinelli (2000), 1799 Friedel pairs
Absolute structure parameter: 0.04 (7)

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}
S	0.75906 (4)	0.44169 (9)	0.12214 (4)	0.05970 (15)
C11	0.47717 (11)	0.9710 (2)	0.29664 (11)	0.0287 (3)
H11	0.4771	1.0444	0.2271	0.034*
O2	0.35347 (8)	1.10088 (15)	0.40682 (8)	0.0328 (2)
O4	0.40208 (10)	0.43397 (19)	0.20264 (11)	0.0488 (3)
H4	0.4583	0.5054	0.2205	0.073*
C10	0.37095 (11)	0.8366 (2)	0.28037 (11)	0.0256 (3)
H10	0.3907	0.7195	0.3274	0.031*
O1	0.53049 (10)	1.22191 (19)	0.44127 (10)	0.0458 (3)
C12	0.46117 (12)	1.1124 (2)	0.38823 (12)	0.0317 (3)
O3	0.13476 (9)	0.9267 (2)	0.45208 (10)	0.0465 (3)
C2	0.22047 (12)	0.8371 (2)	0.39912 (12)	0.0325 (3)

H2	0.2684	0.7399	0.4466	0.039*
N2	0.60890 (10)	0.70195 (19)	0.25656 (10)	0.0310 (3)
C15	0.59162 (12)	0.8638 (2)	0.33252 (12)	0.0328 (3)
H15A	0.6533	0.9583	0.3362	0.039*
H15B	0.5962	0.8108	0.4075	0.039*
C9	0.32885 (13)	0.7685 (2)	0.15807 (12)	0.0315 (3)
H9A	0.3845	0.8096	0.1145	0.038*
H9B	0.2576	0.8361	0.1277	0.038*
C3	0.09931 (13)	0.7784 (2)	0.36578 (15)	0.0394 (4)
C1	0.28464 (11)	0.9606 (2)	0.33070 (11)	0.0286 (3)
H1	0.2315	1.0297	0.2711	0.034*
C19	0.64040 (14)	0.7794 (3)	0.15414 (13)	0.0387 (3)
H19A	0.7141	0.8447	0.1737	0.046*
H19B	0.5841	0.8762	0.1201	0.046*
C6	0.18706 (13)	0.4410 (2)	0.27405 (15)	0.0422 (4)
H6	0.2559	0.4241	0.3251	0.051*
C7	0.19578 (13)	0.4844 (2)	0.16970 (15)	0.0415 (4)
C17	0.69958 (13)	0.5726 (3)	0.31802 (13)	0.0399 (4)
H17A	0.6805	0.5356	0.3892	0.048*
H17B	0.7711	0.6451	0.3343	0.048*
C8	0.30887 (14)	0.5453 (2)	0.14119 (14)	0.0377 (3)
H8	0.3042	0.5174	0.0612	0.045*
C4	0.06950 (15)	0.5785 (3)	0.40637 (17)	0.0495 (4)
H4A	−0.0082	0.5813	0.4192	0.059*
H4B	0.1205	0.5488	0.4773	0.059*
C16	0.71589 (16)	0.3889 (3)	0.25369 (16)	0.0496 (4)
H16A	0.6447	0.3153	0.2384	0.059*
H16B	0.7737	0.3070	0.2997	0.059*
C18	0.64687 (16)	0.6178 (3)	0.07026 (14)	0.0498 (4)
H18A	0.6606	0.6759	0.0012	0.060*
H18B	0.5739	0.5497	0.0529	0.060*
C13	0.02051 (14)	0.8583 (3)	0.26257 (18)	0.0547 (5)
H13A	−0.0544	0.8785	0.2784	0.082*
H13B	0.0157	0.7660	0.2019	0.082*
H13C	0.0500	0.9812	0.2416	0.082*
C5	0.07965 (16)	0.4157 (3)	0.32077 (19)	0.0537 (5)
H5A	0.0811	0.2881	0.3570	0.064*
H5B	0.0132	0.4197	0.2597	0.064*
C14	0.09689 (18)	0.4951 (4)	0.06943 (19)	0.0679 (6)
H14A	0.0262	0.4726	0.0938	0.102*
H14B	0.1066	0.3965	0.0155	0.102*
H14C	0.0952	0.6230	0.0355	0.102*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0614 (3)	0.0736 (3)	0.0469 (2)	0.0211 (3)	0.0173 (2)	−0.0094 (3)
C11	0.0308 (7)	0.0301 (7)	0.0252 (6)	−0.0049 (5)	0.0051 (5)	0.0005 (5)

O2	0.0317 (5)	0.0312 (5)	0.0355 (5)	−0.0028 (4)	0.0070 (4)	−0.0086 (4)
O4	0.0411 (6)	0.0332 (6)	0.0703 (8)	0.0046 (5)	0.0068 (5)	−0.0056 (6)
C10	0.0270 (6)	0.0242 (6)	0.0249 (6)	−0.0015 (5)	0.0034 (5)	−0.0009 (5)
O1	0.0422 (6)	0.0481 (7)	0.0460 (6)	−0.0143 (5)	0.0061 (5)	−0.0175 (6)
C12	0.0325 (7)	0.0309 (7)	0.0310 (7)	−0.0039 (6)	0.0044 (5)	−0.0015 (6)
O3	0.0366 (5)	0.0539 (7)	0.0539 (7)	−0.0044 (6)	0.0205 (5)	−0.0164 (6)
C2	0.0283 (7)	0.0357 (7)	0.0339 (8)	0.0011 (6)	0.0072 (6)	−0.0026 (6)
N2	0.0291 (5)	0.0378 (6)	0.0271 (6)	0.0022 (5)	0.0077 (4)	0.0028 (5)
C15	0.0280 (7)	0.0415 (8)	0.0282 (7)	−0.0020 (6)	0.0039 (5)	−0.0038 (6)
C9	0.0353 (7)	0.0320 (7)	0.0259 (7)	−0.0036 (6)	0.0029 (5)	−0.0033 (6)
C3	0.0290 (7)	0.0384 (8)	0.0525 (10)	−0.0007 (6)	0.0123 (6)	−0.0048 (7)
C1	0.0277 (6)	0.0267 (7)	0.0294 (6)	0.0008 (6)	0.0008 (5)	−0.0027 (6)
C19	0.0427 (8)	0.0431 (9)	0.0325 (8)	−0.0002 (7)	0.0130 (6)	0.0051 (7)
C6	0.0388 (8)	0.0243 (7)	0.0621 (10)	−0.0001 (7)	0.0069 (7)	0.0012 (8)
C7	0.0381 (8)	0.0299 (8)	0.0531 (10)	−0.0042 (6)	0.0012 (7)	−0.0095 (7)
C17	0.0355 (8)	0.0521 (10)	0.0322 (8)	0.0092 (7)	0.0067 (6)	0.0046 (7)
C8	0.0419 (8)	0.0321 (8)	0.0373 (8)	−0.0015 (6)	0.0034 (6)	−0.0106 (6)
C4	0.0364 (9)	0.0519 (11)	0.0640 (11)	−0.0091 (8)	0.0191 (8)	0.0040 (9)
C16	0.0498 (9)	0.0484 (11)	0.0504 (10)	0.0141 (8)	0.0097 (8)	0.0056 (8)
C18	0.0586 (11)	0.0602 (11)	0.0312 (8)	0.0090 (9)	0.0107 (7)	−0.0017 (8)
C13	0.0335 (8)	0.0461 (10)	0.0776 (14)	0.0004 (8)	−0.0054 (8)	−0.0018 (9)
C5	0.0491 (9)	0.0350 (9)	0.0782 (13)	−0.0101 (8)	0.0157 (9)	0.0050 (9)
C14	0.0487 (10)	0.0836 (17)	0.0633 (13)	−0.0162 (11)	−0.0080 (9)	−0.0134 (11)

Geometric parameters (Å, º)

S—C18	1.810 (2)	C1—H1	0.9800
S—C16	1.810 (2)	C19—C18	1.510 (3)
C11—C12	1.510 (2)	C19—H19A	0.9700
C11—C15	1.530 (2)	C19—H19B	0.9700
C11—C10	1.5410 (18)	C6—C7	1.325 (3)
C11—H11	0.9800	C6—C5	1.512 (2)
O2—C12	1.3500 (17)	C6—H6	0.9300
O2—C1	1.4592 (16)	C7—C14	1.516 (2)
O4—C8	1.424 (2)	C7—C8	1.516 (2)
O4—H4	0.8200	C17—C16	1.505 (3)
C10—C9	1.5396 (19)	C17—H17A	0.9700
C10—C1	1.5476 (19)	C17—H17B	0.9700
C10—H10	0.9800	C8—H8	0.9800
O1—C12	1.1983 (18)	C4—C5	1.539 (3)
O3—C2	1.4471 (18)	C4—H4A	0.9700
O3—C3	1.454 (2)	C4—H4B	0.9700
C2—C3	1.474 (2)	C16—H16A	0.9700
C2—C1	1.495 (2)	C16—H16B	0.9700
C2—H2	0.9800	C18—H18A	0.9700
N2—C19	1.4674 (19)	C18—H18B	0.9700
N2—C17	1.4735 (19)	C13—H13A	0.9600
N2—C15	1.4758 (19)	C13—H13B	0.9600

C15—H15A	0.9700	C13—H13C	0.9600
C15—H15B	0.9700	C5—H5A	0.9700
C9—C8	1.542 (2)	C5—H5B	0.9700
C9—H9A	0.9700	C14—H14A	0.9600
C9—H9B	0.9700	C14—H14B	0.9600
C3—C13	1.507 (3)	C14—H14C	0.9600
C3—C4	1.511 (3)		

C18—S—C16	96.67 (8)	C18—C19—H19B	109.3
C12—C11—C15	109.33 (11)	H19A—C19—H19B	108.0
C12—C11—C10	104.37 (11)	C7—C6—C5	128.33 (16)
C15—C11—C10	114.62 (12)	C7—C6—H6	115.8
C12—C11—H11	109.4	C5—C6—H6	115.8
C15—C11—H11	109.4	C6—C7—C14	125.47 (17)
C10—C11—H11	109.4	C6—C7—C8	121.62 (15)
C12—O2—C1	111.34 (10)	C14—C7—C8	112.74 (17)
C8—O4—H4	109.5	N2—C17—C16	112.94 (13)
C9—C10—C11	113.63 (12)	N2—C17—H17A	109.0
C9—C10—C1	115.55 (11)	C16—C17—H17A	109.0
C11—C10—C1	102.81 (11)	N2—C17—H17B	109.0
C9—C10—H10	108.2	C16—C17—H17B	109.0
C11—C10—H10	108.2	H17A—C17—H17B	107.8
C1—C10—H10	108.2	O4—C8—C7	111.77 (14)
O1—C12—O2	121.46 (14)	O4—C8—C9	111.68 (13)
O1—C12—C11	127.91 (14)	C7—C8—C9	111.03 (13)
O2—C12—C11	110.62 (11)	O4—C8—H8	107.4
C2—O3—C3	61.08 (10)	C7—C8—H8	107.4
O3—C2—C3	59.68 (10)	C9—C8—H8	107.4
O3—C2—C1	119.82 (13)	C3—C4—C5	111.69 (15)
C3—C2—C1	125.59 (13)	C3—C4—H4A	109.3
O3—C2—H2	113.7	C5—C4—H4A	109.3
C3—C2—H2	113.7	C3—C4—H4B	109.3
C1—C2—H2	113.7	C5—C4—H4B	109.3
C19—N2—C17	110.84 (12)	H4A—C4—H4B	107.9
C19—N2—C15	110.78 (12)	C17—C16—S	112.55 (13)
C17—N2—C15	107.95 (11)	C17—C16—H16A	109.1
N2—C15—C11	113.93 (11)	S—C16—H16A	109.1
N2—C15—H15A	108.8	C17—C16—H16B	109.1
C11—C15—H15A	108.8	S—C16—H16B	109.1
N2—C15—H15B	108.8	H16A—C16—H16B	107.8
C11—C15—H15B	108.8	C19—C18—S	112.36 (12)
H15A—C15—H15B	107.7	C19—C18—H18A	109.1
C10—C9—C8	115.79 (13)	S—C18—H18A	109.1
C10—C9—H9A	108.3	C19—C18—H18B	109.1
C8—C9—H9A	108.3	S—C18—H18B	109.1
C10—C9—H9B	108.3	H18A—C18—H18B	107.9
C8—C9—H9B	108.3	C3—C13—H13A	109.5
H9A—C9—H9B	107.4	C3—C13—H13B	109.5

O3—C3—C2	59.24 (9)	H13A—C13—H13B	109.5
O3—C3—C13	113.05 (14)	C3—C13—H13C	109.5
C2—C3—C13	122.86 (15)	H13A—C13—H13C	109.5
O3—C3—C4	116.16 (15)	H13B—C13—H13C	109.5
C2—C3—C4	115.81 (15)	C6—C5—C4	111.07 (15)
C13—C3—C4	116.52 (15)	C6—C5—H5A	109.4
O2—C1—C2	107.44 (11)	C4—C5—H5A	109.4
O2—C1—C10	105.77 (10)	C6—C5—H5B	109.4
C2—C1—C10	111.70 (12)	C4—C5—H5B	109.4
O2—C1—H1	110.6	H5A—C5—H5B	108.0
C2—C1—H1	110.6	C7—C14—H14A	109.5
C10—C1—H1	110.6	C7—C14—H14B	109.5
N2—C19—C18	111.59 (15)	H14A—C14—H14B	109.5
N2—C19—H19A	109.3	C7—C14—H14C	109.5
C18—C19—H19A	109.3	H14A—C14—H14C	109.5
N2—C19—H19B	109.3	H14B—C14—H14C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4···N2	0.82	2.21	3.0278 (18)	176
C2—H2···O1^{i}	0.98	2.51	3.2948 (19)	137
C6—H6···O2^{ii}	0.93	2.59	3.2526 (19)	129

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