Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = \frac{4}{3}\sum_i\sum_j\beta_{ij}\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S	0.5601 (2)	0.0197 (2)	0.2767 (2)	4.69 (5)
O(1)	0.8645 (7)	0.3186 (5)	0.6945 (5)	5.9 (2)
O(2)	0.7407 (6)	0.3856 (4)	0.4186 (5)	5.2 (2)
O(3)	0.9372 (6)	0.4063 (4)	0.3798 (5)	5.6 (2)
N	0.8686 (8)	−0.0645 (5)	0.5359 (7)	5.6 (2)
C(1)	0.7915 (8)	0.2601 (6)	0.6217 (7)	4.1 (2)
C(2)	0.8430 (7)	0.2051 (6)	0.5368 (6)	3.3 (2)
C(3)	0.7001 (7)	0.1589 (6)	0.4446 (6)	3.1 (1)
C(4)	0.6090 (8)	0.1385 (6)	0.5191 (6)	3.9 (2)
C(5)	0.6404 (9)	0.2278 (7)	0.6008 (8)	4.9 (2)
C(6)	0.9321 (8)	0.2663 (6)	0.4861 (6)	3.6 (2)
C(7)	0.8525 (8)	0.3581 (6)	0.4234 (6)	3.6 (2)
C(8)	0.883 (1)	0.4976 (8)	0.3232 (8)	6.2 (2)
C(9)	0.7300 (8)	0.0690 (6)	0.3820 (6)	3.4 (2)
C(10)	0.8093 (8)	−0.0069 (6)	0.4690 (6)	3.7 (2)
C(11)	0.6263 (8)	−0.0745 (6)	0.2129 (7)	3.8 (2)
C(12)	0.664 (1)	−0.0544 (7)	0.1174 (8)	5.8 (2)
C(13)	0.719 (1)	−0.1300 (9)	0.0719 (9)	9.2 (3)
C(14)	0.738 (1)	−0.2217 (8)	0.121 (1)	8.1 (3)
C(15)	0.701 (1)	−0.2403 (7)	0.214 (1)	7.1 (3)
C(16)	0.644 (1)	−0.1670 (7)	0.2617 (8)	5.4 (3)

Table 2. *Selected bond distances* (Å) *and angles* (°)

S—C(9)	1.812 (7)	C(3)—C(4)	1.54 (1)
S—C(11)	1.769 (9)	C(3)—C(9)	1.54 (1)
O(1)—C(1)	1.208 (9)	C(4)—C(5)	1.53 (1)
O(2)—C(7)	1.16 (1)	C(6)—C(7)	1.53 (1)
O(3)—C(7)	1.34 (1)	C(11)—C(12)	1.38 (1)
O(3)—C(8)	1.43 (1)	C(11)—C(16)	1.39 (1)
N—C(10)	1.13 (1)	C(12)—C(13)	1.39 (2)
C(1)—C(2)	1.53 (1)	C(13)—C(14)	1.38 (2)
C(1)—C(5)	1.50 (1)	C(14)—C(15)	1.36 (2)
C(2)—C(3)	1.566 (9)	C(15)—C(16)	1.39 (2)
C(2)—C(6)	1.52 (1)	C(9)—C(10)	1.48 (1)
C(9)—S—C(11)	100.1 (4)	O(2)—C(7)—C(6)	126.7 (8)
C(7)—O(3)—C(8)	116.3 (7)	O(3)—C(7)—C(6)	108.6 (7)
O(1)—C(1)—C(2)	124.0 (8)	S—C(9)—C(3)	109.8 (5)
O(1)—C(1)—C(5)	127.0 (9)	S—C(9)—C(10)	109.5 (5)
C(2)—C(1)—C(5)	109.0 (6)	C(3)—C(9)—C(10)	111.5 (6)
C(1)—C(2)—C(3)	103.3 (6)	N—C(10)—C(9)	179 (1)
C(1)—C(2)—C(6)	114.2 (6)	S—C(11)—C(12)	119.7 (7)
C(3)—C(2)—C(6)	116.7 (6)	S—C(11)—C(16)	119.3 (8)
C(2)—C(3)—C(4)	103.7 (5)	C(12)—C(11)—C(16)	121.0 (9)
C(2)—C(3)—C(9)	112.3 (5)	C(11)—C(12)—C(13)	117.9 (9)
C(4)—C(3)—C(9)	114.8 (6)	C(12)—C(13)—C(14)	121 (1)
C(3)—C(4)—C(5)	102.6 (7)	C(13)—C(14)—C(15)	120 (1)
C(1)—C(5)—C(4)	106.5 (8)	C(14)—C(15)—C(16)	120 (1)
C(2)—C(6)—C(7)	112.7 (6)	C(11)—C(16)—C(15)	119 (1)
O(2)—C(7)—O(3)	124.5 (7)		

The title compound was prepared by Michael addition of lithiated phenylthioacetonitrile to 2-cyclopentenone followed by methyl bromoacetate alkylation. After the usual treatment of the reaction medium, the residue was dissolved in the minimum amount of ether. After 48 h at room temperature, a solid had precipitated; crystals were grown by slow evaporation of an etheral solution at room temperature; m.p. 382 K.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71450 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1045]

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A Chiral Tetrahydro-γ-pyranonecarboxylate Ester for Asymmetric Nazarov Cyclization

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Abstract

(1*S*,2-*exo*)-1-[(*N*,*N*-Dicyclohexylamino)sulfonylmethyl]-7,7-dimethylbicyclo[2.2.1]heptan-2-yl [2*R*(2α,-3β,5β,6α)]-tetrahydro-2,5,6-trimethyl-4-oxo-2*H*-pyran-3-carboxylate, $C_{31}H_{51}NO_6S$, has an asymmetric unit containing two unique but similarly conformed molecules (*A* and *B*). In molecule *A* the cyclohexyl rings are free to crystallize in either of the approximately coplanar conformations, whereas in molecule *B* only one conformation is allowed. The N atoms in

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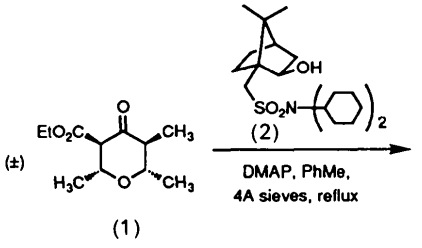
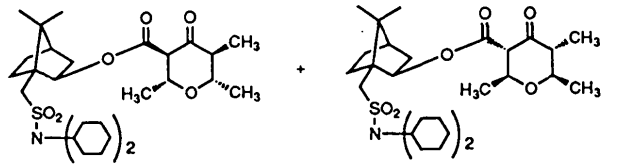
each molecule have approximately planar geometries, as expected from previous related structures. The tetrahydropyranone ring adopts the chair conformation with all substituents equatorial.

Comment

The compound is a precursor for a Nazarov cyclization (Santelli-Rouvier & Santelli, 1983) to give a substituted cyclopentenone (Andrews & Regan, 1991), using a chiral auxiliary attached to the tetrahydr-3-γ-pyranone *via* an ester linkage. In order to produce enantiomerically pure cyclopentenones, this intermediate was prepared from a racemic ester (1) and Oppolzer's enantiomerically pure alcohol (2) (Oppolzer, 1987) yielding two diastereoisomers, (3*a*) and (3*b*). Either isomer could be treated with a suitable Lewis acid causing dehydration to give a divinylketone intermediate, which then undergoes Nazarov's cyclization to a cyclopentenone.





(3*a*) Major diastereoisomer (3*b*) Minor diastereoisomer

The X-ray crystal structure of the major diastereoisomer was determined in order to establish the absolute stereochemical configuration and to examine the conformation of the chiral auxiliary group, following the structure determination of an ester produced from a different chiral auxiliary and the same racemic ester (Andrews, Regan, Wallis & Povey, 1992). The relative stereochemistry of the tetrahydropyranone ring of the major diastereoisomer was the same in both cases.

The two unique molecules have similar conformations in the lattice: the N atoms are both planar [sum of angles at N: molecule *A* 360 (3), molecule *B* 359 (3)°].

In molecule *A* there is freedom for the cyclohexyl rings to crystallize into either approximately coplanar position, while in *B* only one conformation is allowed. This is shown by the apparent planarity of the rings in the plot of *A* (Fig. 1) compared with the obvious puckering of the rings in the plot of *B* (Fig. 2). The extended ellipsoid shape in the *ORTEP* plot (Johnson, 1965) of some of the ring C atoms in molecule *A*, and the apparently larger cyclohexyl

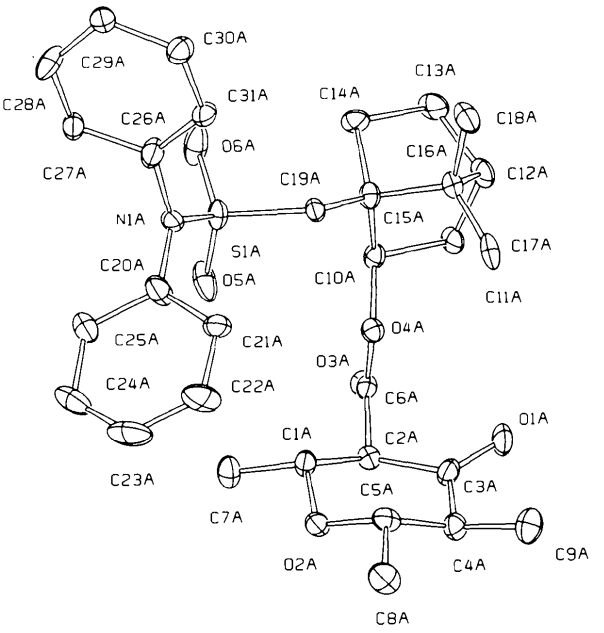


Fig. 1. *ORTEP* drawing of molecule *A* showing the numbering scheme; ellipsoids are represented at the 30% probability level.

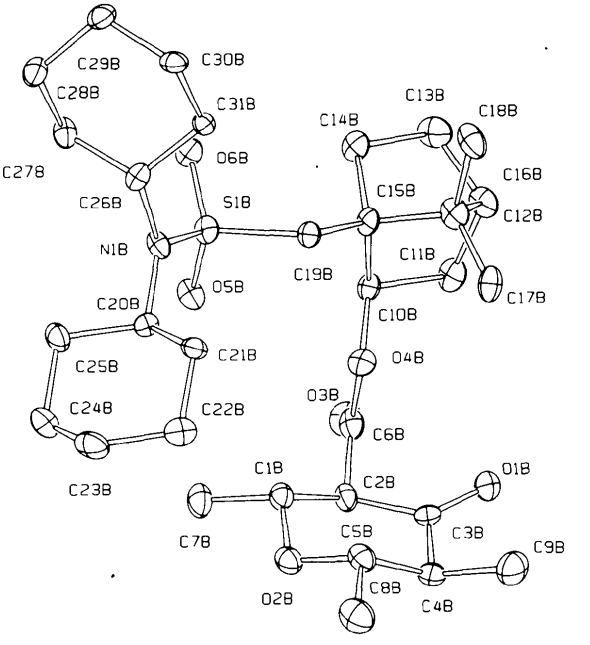


Fig. 2. *ORTEP* drawing of molecule *B* showing the numbering scheme; ellipsoids are represented at the 30% probability level.

bond angles in molecule *A* compared with molecule *B*, confirm this.

The molecules pack in a nose-to-tail fashion with the extra-annular ester carbonyl group extending towards the N atom in the gap between the cyclohexyl rings.

Experimental

Crystal data

$C_{31}H_{51}NO_6S$
M_r = 565.81
Monoclinic
$P2_1$
a = 11.534 (6) Å
b = 22.224 (8) Å
c = 13.295 (9) Å
β = 108.09 (5)°
V = 3239 (3) Å^3
Z = 2
D_x = 1.158 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 20 reflections
θ = 29.5-36.7°
μ = 0.1172 mm^{-1}
T = 294 (1) K
Prism
0.30 × 0.15 × 0.15 mm
Colourless

Data collection

Rigaku AFC-5*R* diffractometer
$\omega/2\theta$ scans
Absorption correction: none
4395 measured reflections
4083 independent reflections
3144 observed reflections [$I > \sigma(I)$]
R_{int} = 0.076
θ_{max} = 60°
$h = -9 \rightarrow 12$
$k = -15 \rightarrow 17$
$l = -11 \rightarrow 13$
3 standard reflections monitored every 150 reflections
intensity variation: −11.8%

Refinement

Refinement on F^2
R = 0.069
wR = 0.080
S = 1.33
3144 reflections
701 parameters
H atoms refined as riding (Beurskens, 1984)
$w = 4F_o^2/\sigma^2(F_o)^2$
$(\Delta/\sigma)_{max} < 0.029$
$\Delta\rho_{max}$ = 0.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.31 e Å^{-3}
Atomic scattering factors from Cromer & Waber (1974)

Program(s) used to solve structure: *TEXSAN* (Molecular Structure Corporation, 1985), *MITHRIL* (Gilmore, 1984) and *SHELXS86* (Sheldrick, 1986). Anomalous-dispersion effects were included in F_c (Ibers & Hamilton, 1964). Molecular graphics: *ORTEP* (Johnson, 1965).

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å^2)

$B_{eq} = (8\pi^2/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
S(1*A*)	0.3599 (2)	−0.0138	0.5993 (2)	6.8 (2)
O(1*A*)	−0.0804 (8)	0.1741 (5)	0.5847 (8)	9.8 (5)
O(2*A*)	0.2611 (7)	0.2394 (4)	0.6622 (6)	6.8 (4)
O(3*A*)	0.0133 (7)	0.1206 (4)	0.3837 (7)	6.7 (4)
O(4*A*)	0.0925 (6)	0.0814 (4)	0.5470 (6)	5.8 (4)
O(5*A*)	0.3450 (7)	0.0380 (6)	0.5343 (8)	11.6 (6)
O(6*A*)	0.3630 (8)	−0.0714 (6)	0.5553 (8)	12.2 (7)
N(1*A*)	0.4863 (7)	−0.0076 (4)	0.6913 (7)	6.0 (4)
C(1*A*)	0.237 (1)	0.1885 (6)	0.597 (1)	6.3 (6)
C(2*A*)	0.097 (1)	0.1839 (5)	0.531 (1)	6.1 (6)
C(3*A*)	0.017 (1)	0.1989 (6)	0.603 (1)	7.1 (7)
C(4*A*)	0.058 (1)	0.2452 (6)	0.684 (1)	7.6 (7)
C(5*A*)	0.199 (1)	0.2391 (5)	0.741 (1)	6.9 (6)
C(6*A*)	0.064 (1)	0.1255 (6)	0.476 (1)	5.9 (6)
C(7*A*)	0.313 (1)	0.1918 (7)	0.522 (1)	8.3 (7)
C(8*A*)	0.253 (1)	0.2885 (7)	0.819 (1)	10.1 (9)
C(9*A*)	−0.012 (2)	0.2484 (8)	0.761 (1)	12.0 (1)
C(10*A*)	0.0596 (8)	0.0213 (5)	0.5046 (9)	5.8 (5)
C(11*A*)	−0.0804 (8)	0.0098 (5)	0.476 (1)	6.6 (6)
C(12*A*)	−0.0887 (9)	−0.0420 (6)	0.549 (1)	7.5 (6)
C(13*A*)	−0.045 (1)	−0.0976 (6)	0.501 (1)	8.7 (7)
C(14*A*)	0.095 (1)	−0.0869 (5)	0.529 (1)	7.2 (6)
C(15*A*)	0.1138 (8)	−0.0259 (5)	0.5892 (8)	5.9 (5)
C(16*A*)	0.019 (1)	−0.0310 (6)	0.649 (1)	7.2 (6)
C(17*A*)	−0.001 (1)	0.0255 (8)	0.707 (1)	8.4 (7)
C(18*A*)	0.042 (1)	−0.0821 (7)	0.732 (1)	9.3 (8)
C(19*A*)	0.2441 (8)	−0.0139 (5)	0.6624 (8)	5.9 (5)
C(20*A*)	0.535 (1)	0.0494 (7)	0.731 (1)	9.6 (9)
C(21*A*)	0.494 (1)	0.0809 (6)	0.812 (1)	7.8 (7)
C(22*A*)	0.547 (2)	0.1393 (8)	0.851 (1)	12.0 (1)
C(23*A*)	0.644 (2)	0.1658 (8)	0.821 (2)	15.0 (1)
C(24*A*)	0.682 (2)	0.1357 (9)	0.740 (2)	12.0 (1)
C(25*A*)	0.633 (1)	0.0763 (7)	0.699 (1)	9.6 (8)
C(26*A*)	0.546 (1)	−0.0610 (7)	0.747 (1)	9.3 (8)
C(27*A*)	0.647 (1)	−0.0851 (6)	0.714 (1)	7.3 (7)
C(28*A*)	0.717 (1)	−0.1353 (8)	0.776 (1)	10.1 (9)
C(29*A*)	0.676 (1)	−0.1670 (7)	0.851 (2)	11.0 (1)
C(30*A*)	0.580 (1)	−0.1398 (6)	0.889 (1)	8.3 (7)
C(31*A*)	0.510 (1)	−0.0891 (6)	0.828 (1)	7.8 (7)
S(1*B*)	0.2568 (2)	0.1241	0.0736 (2)	5.9 (1)
O(1*B*)	0.6768 (8)	−0.0697 (4)	0.3114 (8)	10.1 (5)
O(2*B*)	0.3339 (7)	−0.1303 (4)	0.186 (1)	7.7 (5)
O(3*B*)	0.6056 (7)	−0.0138 (5)	0.0612 (7)	9.0 (5)
O(4*B*)	0.5202 (6)	0.0252 (4)	0.1770 (6)	6.5 (4)
O(5*B*)	0.2842 (6)	0.0768 (4)	0.0134 (6)	7.3 (4)
O(6*B*)	0.2557 (6)	0.1845 (4)	0.0363 (6)	7.0 (4)
N(1*B*)	0.1250 (6)	0.1098 (4)	0.0892 (6)	5.7 (4)
C(1*B*)	0.367 (1)	−0.0800 (6)	0.139 (1)	6.7 (6)
C(2*B*)	0.505 (1)	−0.0785 (6)	0.155 (1)	7.1 (7)
C(3*B*)	0.579 (1)	−0.0931 (5)	0.269 (1)	7.1 (6)
C(4*B*)	0.528 (1)	−0.1402 (6)	0.327 (1)	7.2 (7)
C(5*B*)	0.390 (1)	−0.1298 (6)	0.299 (1)	7.4 (7)
C(6*B*)	0.552 (1)	−0.0202 (7)	0.124 (1)	7.1 (7)
C(7*B*)	0.297 (1)	−0.0818 (7)	0.021 (1)	9.4 (8)
C(8*B*)	0.327 (1)	−0.1764 (8)	0.345 (1)	11.0 (1)
C(9*B*)	0.594 (2)	−0.1368 (8)	0.445 (1)	11.0 (1)
C(10*B*)	0.5597 (8)	0.0848 (6)	0.1554 (9)	5.7 (5)
C(11*B*)	0.699 (1)	0.0955 (7)	0.212 (1)	8.1 (7)
C(12*B*)	0.700 (1)	0.1493 (7)	0.282 (1)	8.2 (7)
C(13*B*)	0.661 (1)	0.2041 (7)	0.208 (1)	9.9 (9)
C(14*B*)	0.525 (1)	0.1918 (6)	0.158 (1)	7.3 (6)
C(15*B*)	0.5001 (8)	0.1322 (5)	0.2084 (9)	5.8 (5)
C(16*B*)	0.589 (1)	0.1377 (6)	0.320 (1)	7.5 (7)
C(17*B*)	0.599 (1)	0.0825 (8)	0.390 (1)	8.8 (8)
C(18*B*)	0.563 (1)	0.1914 (7)	0.382 (1)	9.8 (8)
C(19*B*)	0.3669 (8)	0.1200 (5)	0.2012 (8)	5.6 (5)
C(20*B*)	0.0886 (9)	0.0473 (5)	0.0961 (8)	5.1 (5)
C(21*B*)	0.089 (1)	0.0273 (5)	0.203 (1)	6.3 (6)
C(22*B*)	0.051 (1)	−0.0377 (6)	0.206 (1)	7.9 (7)
C(23*B*)	−0.074 (1)	−0.0489 (6)	0.126 (2)	10.2 (9)
C(24*B*)	−0.072 (1)	−0.0292 (7)	0.019 (1)	9.6 (8)
C(25*B*)	−0.037 (1)	0.0372 (6)	0.015 (1)	7.9 (7)
C(26*B*)	0.0498 (9)	0.1598 (5)	0.1128 (9)	5.6 (5)
C(27*B*)	−0.015 (1)	0.1945 (6)	0.011 (1)	7.0 (6)
C(28*B*)	−0.108 (1)	0.2385 (6)	0.031 (1)	7.6 (6)
C(29*B*)	−0.050 (1)	0.2798 (6)	0.125 (1)	7.8 (6)
C(30*B*)	0.016 (1)	0.2444 (5)	0.225 (1)	6.5 (5)
C(31*B*)	0.1094 (9)	0.1997 (5)	0.2048 (9)	5.2 (5)

Table 2. *Geometric parameters* (Å, °)

S(1*A*)—O(5*A*)	1.42 (1)	S(1*B*)—O(5*B*)	1.416 (7)
S(1*A*)—O(6*A*)	1.41 (1)	S(1*B*)—O(6*B*)	1.429 (8)
S(1*A*)—N(1*A*)	1.591 (8)	S(1*B*)—N(1*B*)	1.628 (7)

S(1A)—C(19A)	1.78 (1)	S(1B)—C(19B)	1.78 (1)
O(1A)—C(3A)	1.21 (1)	O(1B)—C(3B)	1.21 (1)
O(2A)—C(1A)	1.40 (1)	O(2B)—C(1B)	1.40 (1)
O(2A)—C(5A)	1.44 (1)	O(2B)—C(5B)	1.43 (1)
O(3A)—C(6A)	1.19 (1)	O(3B)—C(6B)	1.20 (1)
O(4A)—C(6A)	1.33 (1)	O(4B)—C(6B)	1.34 (1)
O(4A)—C(10A)	1.45 (1)	O(4B)—C(10B)	1.46 (1)
N(1A)—C(20A)	1.42 (2)	N(1B)—C(20B)	1.46 (1)
N(1A)—C(26A)	1.46 (1)	N(1B)—C(26B)	1.50 (1)
C(1A)—C(2A)	1.59 (1)	C(1B)—C(2B)	1.54 (2)
C(1A)—C(7A)	1.52 (2)	C(1B)—C(7B)	1.52 (2)
C(2A)—C(3A)	1.57 (1)	C(2B)—C(3B)	1.53 (2)
C(2A)—C(6A)	1.48 (2)	C(2B)—C(6B)	1.50 (2)
C(3A)—C(4A)	1.45 (2)	C(3B)—C(4B)	1.52 (2)
C(4A)—C(5A)	1.57 (2)	C(4B)—C(5B)	1.54 (2)
C(4A)—C(9A)	1.49 (2)	C(4B)—C(9B)	1.52 (2)
C(5A)—C(8A)	1.51 (2)	C(5B)—C(8B)	1.50 (2)
C(10A)—C(11A)	1.56 (1)	C(10B)—C(11B)	1.57 (1)
C(10A)—C(15A)	1.52 (1)	C(10B)—C(15B)	1.54 (1)
C(11A)—C(12A)	1.52 (1)	C(11B)—C(12B)	1.51 (2)
C(12A)—C(13A)	1.54 (2)	C(12B)—C(13B)	1.54 (2)
C(12A)—C(16A)	1.54 (2)	C(12B)—C(16B)	1.54 (1)
C(13A)—C(14A)	1.56 (2)	C(13B)—C(14B)	1.53 (2)
C(14A)—C(15A)	1.55 (1)	C(14B)—C(15B)	1.55 (2)
C(15A)—C(16A)	1.55 (1)	C(15B)—C(16B)	1.52 (2)
C(15A)—C(19A)	1.54 (1)	C(15B)—C(19B)	1.53 (1)
C(16A)—C(17A)	1.53 (2)	C(16B)—C(17B)	1.52 (2)
C(16A)—C(18A)	1.55 (2)	C(16B)—C(18B)	1.54 (2)
C(20A)—C(21A)	1.47 (2)	C(20B)—C(21B)	1.49 (1)
C(20A)—C(25A)	1.45 (2)	C(20B)—C(25B)	1.53 (2)
C(21A)—C(22A)	1.46 (2)	C(21B)—C(22B)	1.52 (2)
C(22A)—C(23A)	1.42 (3)	C(22B)—C(23B)	1.52 (2)
C(23A)—C(24A)	1.44 (3)	C(23B)—C(24B)	1.50 (2)
C(24A)—C(25A)	1.47 (2)	C(24B)—C(25B)	1.53 (2)
C(26A)—C(27A)	1.47 (2)	C(26B)—C(27B)	1.54 (2)
C(26A)—C(31A)	1.41 (2)	C(26B)—C(31B)	1.49 (1)
C(27A)—C(28A)	1.46 (2)	C(27B)—C(28B)	1.54 (2)
C(28A)—C(29A)	1.42 (2)	C(28B)—C(29B)	1.52 (2)
C(29A)—C(30A)	1.48 (2)	C(29B)—C(30B)	1.53 (2)
C(30A)—C(31A)	1.47 (2)	C(30B)—C(31B)	1.55 (1)

O(5A)—S(1A)—O(6A)	119.8 (7)	O(5B)—S(1B)—O(6B)	118.8 (5)
O(5A)—S(1A)—N(1A)	107.9 (5)	O(5B)—S(1B)—N(1B)	107.9 (4)
O(5A)—S(1A)—C(19A)	108.7 (5)	O(5B)—S(1B)—C(19B)	106.4 (5)
O(6A)—S(1A)—N(1A)	105.2 (5)	O(6B)—S(1B)—N(1B)	108.6 (5)
O(6A)—S(1A)—C(19A)	108.2 (6)	O(6B)—S(1B)—C(19B)	107.8 (5)
N(1A)—S(1A)—C(19A)	106.2 (5)	N(1B)—S(1B)—C(19B)	106.7 (4)
C(1A)—O(2A)—C(5A)	113.5 (8)	C(1B)—O(2B)—C(5B)	112 (1)
C(6A)—O(4A)—C(10A)	115.3 (9)	C(6B)—O(4B)—C(10B)	115.5 (9)
S(1A)—N(1A)—C(20A)	122 (1)	S(1B)—N(1B)—C(20B)	119.4 (6)
S(1A)—N(1A)—C(26A)	119.8 (9)	S(1B)—N(1B)—C(26B)	120.3 (7)
C(20A)—N(1A)—C(26A)	118 (1)	C(20B)—N(1B)—C(26B)	119.6 (7)
O(2A)—C(1A)—C(2A)	111.5 (9)	O(2B)—C(1B)—C(2B)	112 (1)
O(2A)—C(1A)—C(7A)	109 (1)	O(2B)—C(1B)—C(7B)	108 (1)
C(2A)—C(1A)—C(7A)	109 (1)	C(2B)—C(1B)—C(7B)	110 (1)
C(1A)—C(2A)—C(3A)	110 (1)	C(1B)—C(2B)—C(3B)	111 (1)
C(1A)—C(2A)—C(6A)	113 (1)	C(1B)—C(2B)—C(6B)	115 (1)
C(3A)—C(2A)—C(6A)	112 (1)	C(3B)—C(2B)—C(6B)	108 (1)
O(1A)—C(3A)—C(2A)	118 (1)	O(1B)—C(3B)—C(2B)	121 (1)
O(1A)—C(3A)—C(4A)	123 (1)	O(1B)—C(3B)—C(4B)	121 (1)
C(2A)—C(3A)—C(4A)	119 (1)	C(2B)—C(3B)—C(4B)	118 (1)
C(3A)—C(4A)—C(5A)	110 (1)	C(3B)—C(4B)—C(5B)	108 (1)
C(3A)—C(4A)—C(9A)	115 (1)	C(3B)—C(4B)—C(9B)	110 (1)
C(5A)—C(4A)—C(9A)	112 (1)	C(5B)—C(4B)—C(9B)	113 (1)
O(2A)—C(5A)—C(4A)	109 (1)	O(2B)—C(5B)—C(4B)	110 (1)
O(2A)—C(5A)—C(8A)	108 (1)	O(2B)—C(5B)—C(8B)	108 (1)
C(4A)—C(5A)—C(8A)	114 (1)	C(4B)—C(5B)—C(8B)	113 (1)
O(3A)—C(6A)—O(4A)	127 (1)	O(3B)—C(6B)—O(4B)	124 (1)
O(3A)—C(6A)—C(2A)	124 (1)	O(3B)—C(6B)—C(2B)	127 (1)
O(4A)—C(6A)—C(2A)	110 (1)	O(4B)—C(6B)—C(2B)	109 (1)
O(4A)—C(10A)—C(11A)	111.6 (8)	O(4B)—C(10B)—C(11B)	112.4 (9)
O(4A)—C(10A)—C(15A)	110.4 (9)	O(4B)—C(10B)—C(15B)	108.6 (8)
C(11A)—C(10A)—C(15A)	102.9 (9)	C(11B)—C(10B)—C(15B)	102 (1)
C(10A)—C(11A)—C(12A)	103.5 (8)	C(10B)—C(11B)—C(12B)	102.9 (9)
C(11A)—C(12A)—C(13A)	105 (1)	C(11B)—C(12B)—C(13B)	107 (1)
C(11A)—C(12A)—C(16A)	103.8 (9)	C(11B)—C(12B)—C(16B)	103 (1)
C(13A)—C(12A)—C(16A)	102 (1)	C(13B)—C(12B)—C(16B)	103 (1)
C(12A)—C(13A)—C(14A)	103.2 (9)	C(12B)—C(13B)—C(14B)	101 (1)
C(13A)—C(14A)—C(15A)	103 (1)	C(13B)—C(14B)—C(15B)	106 (1)
C(10A)—C(15A)—C(14A)	105.3 (9)	C(10B)—C(15B)—C(14B)	102.7 (8)
C(10A)—C(15A)—C(16A)	103.9 (8)	C(10B)—C(15B)—C(16B)	103.7 (8)
C(10A)—C(15A)—C(19A)	116.4 (9)	C(10B)—C(15B)—C(19B)	115.5 (9)
C(14A)—C(15A)—C(16A)	101 (1)	C(14B)—C(15B)—C(16B)	101 (1)
C(14A)—C(15A)—C(19A)	115.2 (9)	C(14B)—C(15B)—C(19B)	116.1 (8)
C(16A)—C(15A)—C(19A)	113.2 (9)	C(16B)—C(15B)—C(19B)	115.9 (9)
C(12A)—C(16A)—C(15A)	94 (1)	C(12B)—C(16B)—C(15B)	94 (1)
C(12A)—C(16A)—C(17A)	111 (1)	C(12B)—C(16B)—C(17B)	115 (1)
C(12A)—C(16A)—C(18A)	115 (1)	C(12B)—C(16B)—C(18B)	112 (1)
C(15A)—C(16A)—C(17A)	116 (1)	C(15B)—C(16B)—C(17B)	115 (1)
C(15A)—C(16A)—C(18A)	116 (1)	C(15B)—C(16B)—C(18B)	114 (1)
C(17A)—C(16A)—C(18A)	105 (1)	C(17B)—C(16B)—C(18B)	106 (1)
S(1A)—C(19A)—C(15A)	115.5 (7)	S(1B)—C(19B)—C(15B)	117.1 (7)
N(1A)—C(20A)—C(21A)	121 (1)	N(1B)—C(20B)—C(21B)	115.3 (9)
N(1A)—C(20A)—C(25A)	121 (1)	N(1B)—C(20B)—C(25B)	108.9 (9)
C(21A)—C20A)—C(25A)	118 (1)	C(21B)—C(20B)—C(25B)	110 (1)
C(20A)—C(21A)—C(22A)	119 (1)	C(20B)—C(21B)—C(22B)	114 (1)
C(21A)—C(22A)—C(23A)	123 (2)	C(21B)—C(22B)—C(23B)	111 (1)
C(22A)—C(23A)—C(24A)	117 (2)	C(22B)—C(23B)—C(24B)	109 (1)
C(23A)—C(24A)—C(25A)	121 (2)	C(23B)—C(24B)—C(25B)	113 (1)
C(20A)—C(25A)—C(24A)	120 (1)	C(20B)—C(25B)—C(24B)	109 (1)
N(1A)—C(26A)—C(27A)	116 (1)	N(1B)—C(26B)—C(27B)	110.1 (9)
N(1A)—C(26A)—C(31A)	123 (1)	N(1B)—C(26B)—C(31B)	117.5 (8)
C(27A)—C(26A)—C(31A)	121 (1)	C(27B)—C(26B)—C(31B)	113 (1)
C(26A)—C(27A)—C(28A)	117 (1)	C(26B)—C(27B)—C(28B)	110 (1)
C(27A)—C(28A)—C(29A)	122 (1)	C(27B)—C(28B)—C(29B)	112 (1)
C(28A)—C(29A)—C(30A)	119 (1)	C(28B)—C(29B)—C(30B)	112 (1)
C(29A)—C(30A)—C(31A)	118 (1)	C(29B)—C(30B)—C(31B)	111 (1)
C(26A)—C(31A)—C(30A)	121 (1)	C(26B)—C(31B)—C(30B)	110.7 (9)

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