IUCrData

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(1*S*,3*R*,8*R*,9*S*,10*R*)-2,2-Dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodecan-9-yl 4-methylbenzene-1-sulfonate

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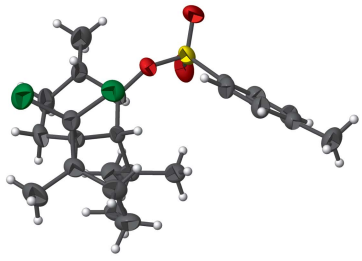
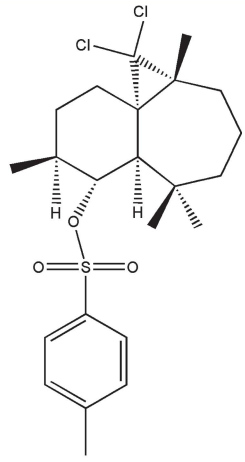
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{23}H_{31}Cl_2O_3S$, was synthesized in three steps from β-himachalene (3,5,5,9-tetramethyl-2,4a,5,6,7,8-hexahydro-1*H*-benzocycloheptene), which was isolated from essential oil of the Atlas cedar (*Cedrus atlantica*). The fused six- and seven-membered rings have boat conformations: the dihedral angle between the mean planes of the rings is 88.03 (12)%. The absolute structure was established unambiguously from anomalous dispersion effects. There are no directional interactions in the crystal.

3D view



Chemical scheme



Structure description

The Atlas cedar (*Cedrus atlantica*), native to Morocco, is the source of essential oils made up mainly (75%) of bicyclic sesquiterpene hydrocarbons, among which is found the compound β-himachalene (El Haib *et al.*, 2011). The reactivity of this sesquiterpene and its derivatives has been studied extensively by our team in order to prepare new products with potential biological properties (El Jamili *et al.*, 2002; Zaki *et al.*, 2014; Benharref *et al.*, 2015). For example, these compounds have been tested for their potential anti-fungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004).

The structure of the title compound was determined as part of our ongoing studies in this area. The molecule is built up from two fused six- and seven-membered rings which is linked to a three-membered ring. An additional toluensulfonic acid group system is attached to the six-membered ring (Fig. 1). The six- and seven- membered rings display boat conformations, as indicated by the total puckering amplitude Q_T = 0.730 (2) Å and

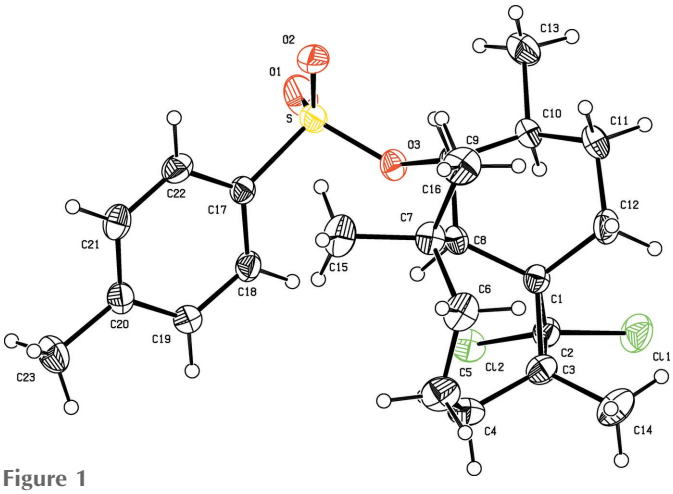


Figure 1
The molecular structure of the title compound, showing the the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

spherical polar angle $\theta = 88.03\ (14)^\circ$ with $\varphi = -170.60\ (14)^\circ$ for the six-membered ring and $Q_T = 1.134\ (2)$ Å, $\theta = 88.22\ (10)$, $\varphi_2 = -51.43\ (10)$ and $\varphi_3 = -62.95\ (3)^\circ$ for the seven-membered ring. Owing to the presence of Cl and S atoms, the absolute configuration was confirmed as C1(1*S*, C3(*R*), C8(*R*), C9(*S*)and C10(*R*). No directional interactions beyond typical van der Waals contacts could be identified in the crystal.

Synthesis and crystallization

Diborane was prepared by addition at 0°C of 2.5 g (17 mmol) of boron trifluoride etherate in 0.5 g (12.6 mmol) of sodium borohydride in 30 ml of diglyme. The diborane formed was driven by a stream of dry nitrogen in 2 g (7 mmol) of (1*S*,3*R*,8*R*)-2,2-dichloro-3,7,7,10-tetramethyltricyclo-[6.4.0.01,3]dodec-9-ene (El Jamili *et al.*, 2002) dissolved in 20 ml of tetrahydrofuran at 273 K. This took about 4 h, then 2 ml of sodium hydroxide 3 *N* was added carefully between 263 K and 273 K in 15 minutes, and then 2 ml of 30% hydrogen peroxide in the vicinity of 298 K. The reaction mixture was then extracted with diethyl ether. The organic phase was washed to neutrality and the solvent was evaporated under vacuum. The residue obtained was chromatographed on a column of silica gel with pentane–ethyl acetate (95/5), which allowed the isolation of pure (1*S*,3*R*,8*R*,9*S*,10*R*)-2,2-dichloro-3,7,7,10-tetramethyltosyltricyclo[6.4.0.01,3]dodecan-9-ol. 1 g (3.3 mmol) of the latter compound was dissolved in pyridine (10 ml). The solution was cooled to 10°C and tosyl chloride (0.6 g, 3.3 mmol) in pyridine (4 ml) was added dropwise. The reaction mixture was stirred overnight and treated with 10 ml of water and extracted with dichlormethane. The organic phase was evaporated and the residue obtained was chromatographed on a column of silica gel with hexane and ethyl acetate (97/3) as eluent to give the sesquiterpene tosylate, with a yield of 87% (1.3 g, 2.8 mmol). The title compound was recrystallized from its ethyl acetate solution.

Table 1
Experimental details.

Crystal data	
Chemical formula	$C_{23}H_{32}Cl_2O_3S$
M_r	459.44
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	298
a, b, c (Å)	9.410 (5), 9.667 (5), 25.285 (5)
V (Å^3)	2300.1 (18)
Z	4
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.40
Crystal size (mm)	0.30 × 0.26 × 0.18
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2003)
T_{min}, T_{max}	0.658, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	23145, 4695, 4050
R_{int}	0.042
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.030, 0.069, 0.97
No. of reflections	4695
No. of parameters	267
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.24, −0.20
Absolute structure	Flack & Bernardinelli (2000), **1527** Friedel pairs
Absolute structure parameter	−0.02 (2)

Computer programs: *APEX2* and *SAINT-Plus* (Bruker, 2009), *SHELXS2014* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *ORTEP-3 for Windows* (Farrugia, 2012) and *publCIF* (Westrip, 2010).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 1.

Acknowledgements

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full crystallographic data

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(1*S*,3*R*,8*R*,9*S*,10*R*)-2,2-Dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]dodecan-9-yl 4-methylbenzene-1-sulfonate

Crystal data

$C_{23}H_{32}Cl_2O_3S$
M_r = 459.44
Orthorhombic, $P2_12_12_1$
a = 9.410 (5) Å
b = 9.667 (5) Å
c = 25.285 (5) Å
V = 2300.1 (18) Å^3
Z = 4
F(000) = 976
D_x = 1.327 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 4695 reflections
θ = 2.3–26.4°
μ = 0.40 mm^{-1}
T = 298 K
Prism, colourless
0.30 × 0.26 × 0.18 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.658, T_{max} = 0.747
23145 measured reflections
4695 independent reflections
4050 reflections with $I > 2\sigma(I)$
R_{int} = 0.042
θ_{max} = 26.4°, θ_{min} = 2.3°
h = −10→11
k = −12→11
l = −31→31

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.069
S = 0.97
4695 reflections
267 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0379P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.24 e Å^{-3}
$\Delta\rho_{min}$ = −0.20 e Å^{-3}
Absolute structure: Flack & Bernardinelli (2000), **1527** Friedel pairs
Absolute structure parameter: −0.02 (2)

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
S	0.30361 (7)	1.09730 (7)	0.32932 (3)	0.04508 (18)
Cl2	0.09152 (7)	1.01208 (7)	0.49466 (2)	0.04986 (19)
Cl1	−0.13706 (9)	1.18643 (9)	0.52767 (3)	0.0642 (2)
O3	0.18685 (16)	1.12166 (18)	0.37229 (6)	0.0404 (4)
O2	0.2495 (2)	1.1256 (2)	0.27804 (8)	0.0676 (7)
O1	0.4254 (2)	1.1701 (2)	0.34718 (10)	0.0734 (7)
C17	0.3410 (2)	0.9205 (3)	0.33419 (9)	0.0352 (5)
C9	0.0330 (2)	1.1402 (3)	0.35953 (9)	0.0361 (6)
H9	0.0216	1.1448	0.3210	0.043*
C7	−0.1280 (3)	0.9254 (3)	0.33748 (9)	0.0413 (6)
C1	−0.1474 (2)	1.0598 (3)	0.42624 (9)	0.0360 (6)
C20	0.4280 (3)	0.6460 (3)	0.34090 (10)	0.0432 (6)
C3	−0.1941 (3)	0.9480 (3)	0.46591 (10)	0.0444 (6)
C8	−0.0504 (2)	1.0149 (3)	0.38083 (8)	0.0331 (5)
H8	0.0207	0.9536	0.3967	0.040*
C6	−0.2455 (3)	0.8346 (3)	0.36231 (11)	0.0554 (8)
H6A	−0.3190	0.8963	0.3753	0.066*
H6B	−0.2874	0.7803	0.3341	0.066*
C19	0.3883 (3)	0.7190 (3)	0.38560 (10)	0.0450 (6)
H19	0.3899	0.6749	0.4183	0.054*
C22	0.3759 (3)	0.8483 (3)	0.28896 (9)	0.0463 (7)
H22	0.3696	0.8909	0.2561	0.056*
C21	0.4200 (3)	0.7126 (3)	0.29281 (10)	0.0474 (7)
H21	0.4451	0.6650	0.2622	0.057*
C18	0.3464 (3)	0.8555 (3)	0.38292 (9)	0.0419 (6)
H18	0.3220	0.9034	0.4135	0.050*
C2	−0.0890 (3)	1.0568 (3)	0.48186 (9)	0.0416 (6)
C12	−0.2463 (3)	1.1782 (3)	0.41175 (11)	0.0497 (7)
H12A	−0.2872	1.2166	0.4437	0.060*
H12B	−0.3234	1.1430	0.3901	0.060*
C15	−0.0172 (3)	0.8332 (3)	0.31048 (11)	0.0590 (8)
H15A	−0.0638	0.7714	0.2864	0.089*
H15B	0.0328	0.7806	0.3368	0.089*
H15C	0.0490	0.8900	0.2914	0.089*
C4	−0.1418 (3)	0.8026 (3)	0.45579 (11)	0.0528 (7)
H4A	−0.1628	0.7458	0.4864	0.063*
H4B	−0.0394	0.8046	0.4514	0.063*
C10	−0.0069 (3)	1.2782 (3)	0.38320 (11)	0.0448 (6)
H10	0.0226	1.2779	0.4204	0.054*

C5	−0.2087 (4)	0.7361 (3)	0.40679 (12)	0.0606 (8)
H5A	−0.1436	0.6669	0.3932	0.073*
H5B	−0.2948	0.6885	0.4175	0.073*
C11	−0.1684 (3)	1.2921 (3)	0.38159 (12)	0.0584 (8)
H11A	−0.1993	1.2908	0.3450	0.070*
H11B	−0.1948	1.3810	0.3964	0.070*
C23	0.4820 (4)	0.5012 (3)	0.34478 (13)	0.0660 (8)
H23A	0.5837	0.5013	0.3416	0.099*
H23B	0.4416	0.4465	0.3169	0.099*
H23C	0.4556	0.4627	0.3784	0.099*
C16	−0.2018 (3)	1.0110 (4)	0.29404 (11)	0.0631 (8)
H16A	−0.2716	1.0705	0.3098	0.095*
H16B	−0.2475	0.9498	0.2694	0.095*
H16C	−0.1325	1.0659	0.2758	0.095*
C13	0.0668 (4)	1.3991 (3)	0.35583 (14)	0.0702 (9)
H13A	0.0357	1.4044	0.3197	0.105*
H13B	0.1679	1.3854	0.3568	0.105*
H13C	0.0434	1.4836	0.3738	0.105*
C14	−0.3432 (3)	0.9518 (4)	0.48932 (12)	0.0682 (9)
H14A	−0.3733	1.0462	0.4931	0.102*
H14B	−0.3428	0.9078	0.5233	0.102*
H14C	−0.4076	0.9039	0.4662	0.102*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
S	0.0336 (3)	0.0400 (4)	0.0617 (4)	0.0032 (3)	0.0093 (3)	0.0097 (3)
Cl2	0.0466 (4)	0.0585 (4)	0.0445 (3)	−0.0001 (3)	−0.0142 (3)	0.0013 (3)
Cl1	0.0760 (5)	0.0677 (5)	0.0489 (4)	0.0035 (4)	0.0103 (3)	−0.0171 (3)
O3	0.0280 (8)	0.0453 (11)	0.0480 (9)	0.0045 (8)	−0.0025 (7)	−0.0001 (8)
O2	0.0667 (14)	0.0767 (16)	0.0595 (12)	0.0271 (12)	0.0190 (10)	0.0305 (12)
O1	0.0382 (11)	0.0457 (13)	0.136 (2)	−0.0081 (10)	0.0087 (12)	−0.0020 (13)
C17	0.0277 (12)	0.0373 (14)	0.0407 (13)	0.0008 (10)	0.0015 (9)	0.0006 (11)
C9	0.0281 (12)	0.0447 (16)	0.0355 (12)	0.0063 (11)	−0.0033 (9)	0.0020 (11)
C7	0.0332 (13)	0.0497 (17)	0.0408 (13)	−0.0021 (12)	−0.0090 (11)	−0.0065 (12)
C1	0.0288 (12)	0.0407 (15)	0.0384 (12)	0.0015 (11)	−0.0024 (10)	−0.0007 (10)
C20	0.0372 (14)	0.0379 (14)	0.0546 (15)	−0.0008 (12)	0.0044 (11)	−0.0060 (12)
C3	0.0401 (14)	0.0509 (16)	0.0422 (13)	−0.0050 (13)	0.0015 (11)	0.0010 (12)
C8	0.0272 (12)	0.0357 (14)	0.0365 (11)	0.0055 (10)	−0.0067 (9)	−0.0017 (10)
C6	0.0416 (15)	0.069 (2)	0.0560 (16)	−0.0130 (15)	−0.0076 (12)	−0.0125 (16)
C19	0.0496 (16)	0.0430 (16)	0.0424 (13)	0.0051 (13)	0.0064 (12)	0.0039 (11)
C22	0.0462 (16)	0.0580 (19)	0.0347 (12)	−0.0003 (14)	0.0031 (11)	0.0024 (12)
C21	0.0446 (15)	0.0531 (18)	0.0444 (14)	−0.0006 (14)	0.0026 (12)	−0.0163 (13)
C18	0.0443 (14)	0.0465 (16)	0.0349 (12)	0.0080 (13)	0.0053 (10)	−0.0051 (11)
C2	0.0412 (14)	0.0460 (15)	0.0375 (12)	0.0042 (12)	0.0017 (11)	−0.0025 (11)
C12	0.0356 (14)	0.0618 (19)	0.0516 (15)	0.0159 (14)	0.0021 (11)	−0.0013 (14)
C15	0.0505 (17)	0.068 (2)	0.0589 (17)	−0.0063 (16)	−0.0041 (14)	−0.0267 (16)
C4	0.0553 (17)	0.0485 (18)	0.0547 (15)	−0.0102 (14)	−0.0010 (14)	0.0124 (13)

C10	0.0448 (15)	0.0375 (15)	0.0519 (15)	0.0108 (12)	0.0048 (12)	0.0042 (12)
C5	0.0636 (19)	0.0540 (19)	0.0643 (18)	−0.0226 (16)	0.0013 (16)	−0.0056 (15)
C11	0.0517 (18)	0.0526 (18)	0.0710 (18)	0.0258 (15)	0.0033 (14)	0.0116 (15)
C23	0.076 (2)	0.0416 (18)	0.081 (2)	0.0097 (16)	0.0080 (17)	−0.0073 (15)
C16	0.0575 (17)	0.086 (2)	0.0461 (14)	−0.0003 (19)	−0.0169 (13)	−0.0009 (16)
C13	0.076 (2)	0.0390 (17)	0.096 (2)	0.0072 (17)	0.0169 (18)	0.0115 (17)
C14	0.0506 (18)	0.090 (3)	0.0639 (18)	−0.0135 (18)	0.0151 (14)	0.0001 (18)

Geometric parameters (Å, º)

S—O1	1.418 (2)	C22—C21	1.379 (4)
S—O2	1.420 (2)	C22—H22	0.9300
S—O3	1.5631 (18)	C21—H21	0.9300
S—C17	1.750 (3)	C18—H18	0.9300
Cl2—C2	1.782 (3)	C12—C11	1.527 (4)
Cl1—C2	1.766 (3)	C12—H12A	0.9700
O3—C9	1.494 (3)	C12—H12B	0.9700
C17—C22	1.379 (3)	C15—H15A	0.9600
C17—C18	1.384 (3)	C15—H15B	0.9600
C9—C10	1.509 (4)	C15—H15C	0.9600
C9—C8	1.541 (3)	C4—C5	1.531 (4)
C9—H9	0.9800	C4—H4A	0.9700
C7—C15	1.532 (4)	C4—H4B	0.9700
C7—C16	1.541 (4)	C10—C13	1.526 (4)
C7—C6	1.544 (4)	C10—C11	1.526 (4)
C7—C8	1.576 (3)	C10—H10	0.9800
C1—C2	1.510 (3)	C5—H5A	0.9700
C1—C12	1.520 (4)	C5—H5B	0.9700
C1—C8	1.530 (3)	C11—H11A	0.9700
C1—C3	1.538 (4)	C11—H11B	0.9700
C20—C21	1.378 (4)	C23—H23A	0.9600
C20—C19	1.384 (4)	C23—H23B	0.9600
C20—C23	1.493 (4)	C23—H23C	0.9600
C3—C2	1.499 (4)	C16—H16A	0.9600
C3—C4	1.511 (4)	C16—H16B	0.9600
C3—C14	1.524 (4)	C16—H16C	0.9600
C8—H8	0.9800	C13—H13A	0.9600
C6—C5	1.514 (4)	C13—H13B	0.9600
C6—H6A	0.9700	C13—H13C	0.9600
C6—H6B	0.9700	C14—H14A	0.9600
C19—C18	1.379 (4)	C14—H14B	0.9600
C19—H19	0.9300	C14—H14C	0.9600
O1—S—O2	119.02 (15)	C3—C2—Cl2	120.5 (2)
O1—S—O3	105.77 (12)	C1—C2—Cl2	121.38 (17)
O2—S—O3	110.71 (11)	Cl1—C2—Cl2	107.28 (13)
O1—S—C17	107.45 (13)	C1—C12—C11	111.7 (2)
O2—S—C17	108.93 (13)	C1—C12—H12A	109.3

O3—S—C17	103.87 (11)	C11—C12—H12A	109.3
C9—O3—S	123.30 (14)	C1—C12—H12B	109.3
C22—C17—C18	120.0 (2)	C11—C12—H12B	109.3
C22—C17—S	118.92 (19)	H12A—C12—H12B	107.9
C18—C17—S	120.87 (19)	C7—C15—H15A	109.5
O3—C9—C10	105.2 (2)	C7—C15—H15B	109.5
O3—C9—C8	108.89 (18)	H15A—C15—H15B	109.5
C10—C9—C8	115.4 (2)	C7—C15—H15C	109.5
O3—C9—H9	109.1	H15A—C15—H15C	109.5
C10—C9—H9	109.1	H15B—C15—H15C	109.5
C8—C9—H9	109.1	C3—C4—C5	113.2 (3)
C15—C7—C16	107.6 (2)	C3—C4—H4A	108.9
C15—C7—C6	109.8 (2)	C5—C4—H4A	108.9
C16—C7—C6	105.8 (2)	C3—C4—H4B	108.9
C15—C7—C8	108.25 (19)	C5—C4—H4B	108.9
C16—C7—C8	114.2 (2)	H4A—C4—H4B	107.8
C6—C7—C8	111.2 (2)	C9—C10—C13	112.6 (2)
C2—C1—C12	117.5 (2)	C9—C10—C11	108.4 (2)
C2—C1—C8	118.4 (2)	C13—C10—C11	111.9 (2)
C12—C1—C8	113.5 (2)	C9—C10—H10	107.9
C2—C1—C3	58.89 (16)	C13—C10—H10	107.9
C12—C1—C3	120.7 (2)	C11—C10—H10	107.9
C8—C1—C3	117.4 (2)	C6—C5—C4	115.5 (3)
C21—C20—C19	117.9 (2)	C6—C5—H5A	108.4
C21—C20—C23	121.0 (2)	C4—C5—H5A	108.4
C19—C20—C23	121.1 (3)	C6—C5—H5B	108.4
C2—C3—C4	118.9 (2)	C4—C5—H5B	108.4
C2—C3—C14	119.1 (2)	H5A—C5—H5B	107.5
C4—C3—C14	112.9 (3)	C10—C11—C12	113.6 (2)
C2—C3—C1	59.63 (16)	C10—C11—H11A	108.8
C4—C3—C1	116.7 (2)	C12—C11—H11A	108.8
C14—C3—C1	120.0 (3)	C10—C11—H11B	108.8
C1—C8—C9	110.1 (2)	C12—C11—H11B	108.8
C1—C8—C7	113.63 (19)	H11A—C11—H11B	107.7
C9—C8—C7	115.13 (19)	C20—C23—H23A	109.5
C1—C8—H8	105.7	C20—C23—H23B	109.5
C9—C8—H8	105.7	H23A—C23—H23B	109.5
C7—C8—H8	105.7	C20—C23—H23C	109.5
C5—C6—C7	119.7 (2)	H23A—C23—H23C	109.5
C5—C6—H6A	107.4	H23B—C23—H23C	109.5
C7—C6—H6A	107.4	C7—C16—H16A	109.5
C5—C6—H6B	107.4	C7—C16—H16B	109.5
C7—C6—H6B	107.4	H16A—C16—H16B	109.5
H6A—C6—H6B	106.9	C7—C16—H16C	109.5
C18—C19—C20	121.7 (2)	H16A—C16—H16C	109.5
C18—C19—H19	119.2	H16B—C16—H16C	109.5
C20—C19—H19	119.2	C10—C13—H13A	109.5
C17—C22—C21	119.6 (2)	C10—C13—H13B	109.5

C17—C22—H22	120.2	H13A—C13—H13B	109.5
C21—C22—H22	120.2	C10—C13—H13C	109.5
C20—C21—C22	121.5 (2)	H13A—C13—H13C	109.5
C20—C21—H21	119.2	H13B—C13—H13C	109.5
C22—C21—H21	119.2	C3—C14—H14A	109.5
C19—C18—C17	119.2 (2)	C3—C14—H14B	109.5
C19—C18—H18	120.4	H14A—C14—H14B	109.5
C17—C18—H18	120.4	C3—C14—H14C	109.5
C3—C2—C1	61.48 (16)	H14A—C14—H14C	109.5
C3—C2—Cl1	120.36 (19)	H14B—C14—H14C	109.5
C1—C2—Cl1	120.26 (18)		