IUCrData

ISSN 2414-3146

1-[(1*R*,2*S*,4*R*,7*S*)-3,3-Dichloro-4,11,11-trimethyltricyclo[5.4.0.0^{2,4}]undecan-7-yl]ethanone

Ahmed Benharref,[a]* Lahcen El Ammari,[b] Mohamed Saadi,[b] Mustapha Ait Elhad,[a] Abdelouahd Oukhrib[a] and Moha Berraho[a]

[a]Laboratoire de Chimie des Substances Naturelles, 'Unité Associé au CNRST (URAC16)', Faculté des Sciences Semlalia, BP 2390 Bd My Abdellah, Université Cadi Ayyad, 40000 Marrakech, Morocco, and [b]Laboratoire de Chimie du Solide Appliquée, Faculté des Sciences, Mohammed V University in Rabat, Avenue Ibn Battouta, BP 1014 Rabat, Morocco. *Correspondence e-mail: benharref@uca.ac.ma

Received 23 January 2017
Accepted 7 February 2017

Edited by C. Rizzoli, Universita degli Studi di Parma, Italy

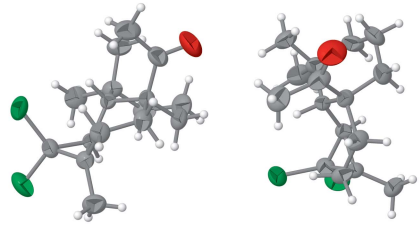
Keywords: crystal structure; β-himachalene; Atlas cedar; *Cedrus atlantica*.

CCDC reference: 1531481

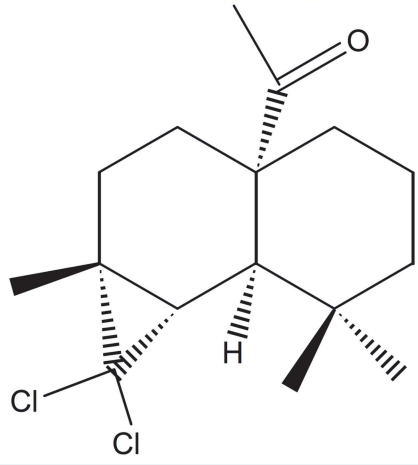
Structural data: full structural data are available from iucrdata.iucr.org

The title compound, $C_{16}H_{24}Cl_2O$, crystallizes with two independent molecules in the asymmetric unit. Each molecule is built up from two fused six-membered rings, one of which is fused to a three-membered ring. The two molecules differ essentially in the orientation of two of the methyl groups. The dihedral angles between the mean planes through the two six-membered rings are 57.98 (13) and 55.29 (13)°. The molecular conformation is stabilized by intramolecular C—H···Cl hydrogen bonds.

3D view



Chemical scheme



Structure description

The bicyclic sesquiterpene β-himachalene is the main constituent of the essential oil of the Atlas cedar (*Cedrus atlantica*) (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 having biological properties (El Jamili *et al.*, 2002; Benharref *et al.*, 2013, 2015, 2016; Zaki *et al.*, 2014). Indeed, these compounds were tested, using the food-poisoning technique, for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2004). In this paper, we present the crystal structure of the title compound.

The asymmetric unit (Fig. 1) includes two crystallographically independent molecules (*A* and *B*). A least-squares overlay of the two molecules (Spek, 2009) is shown in Fig. 2 and reveals that the most important difference between them is the relative orientation of the two acetonic methyl atoms C16 and C29. In both molecules, one of the six-membered rings (C1–C6 and C17–C22) has a chair conformation, as indicated by the total puckering amplitude Q_T = 0.539 (3)/0.542 (3) Å and spherical polar angle θ_2 = 178.2 (3)/175.3 (2)°,

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

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···Cl1	0.98	2.64	3.227 (2)	118
C8—H8B···Cl1	0.97	2.61	3.184 (3)	118
C22—H22···Cl3	0.98	2.61	3.205 (2)	120
C25—H25B···Cl3	0.97	2.63	3.196 (3)	117

with φ_2 = −42 (8)/−22 (4)°, whereas the other six-membered ring (C1/C6–C10 and C17/C22–C26) displays a boat conformation, with Q_T = 0.735 (3)/0.727 (4) Å, θ_2 = 92.13 (18)/93.01 (18)° and φ_2 = −0.6 (2)/63.6 (2)°. In addition, the dihedral angle between the least-squares mean planes through the two six-membered rings is 57.98 (13)° in molecule A and 55.29 (13)° in molecule B. Weak intramolecular C—H···Cl interactions (Table 1) stabilize the molecular conformation. Owing to the presence of Cl atoms, the absolute configuration could be fully confirmed by refining the Flack parameter. The title compound is isostructural with the corresponding dibromo derivative (Zaki *et al.*, 2014).

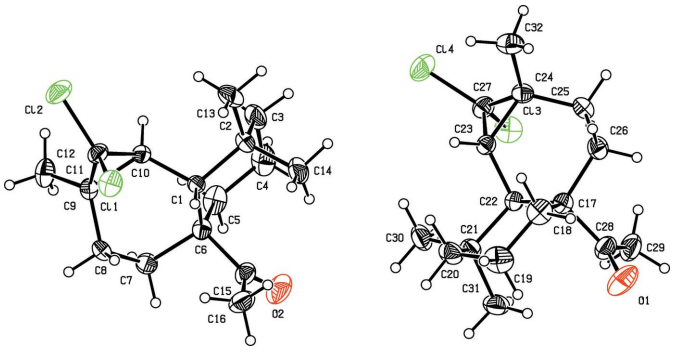


Figure 1
The asymmetric unit of the title compound, with displacement ellipsoids drawn at the 30% probability level. H atoms are represented as small spheres of arbitrary radii.

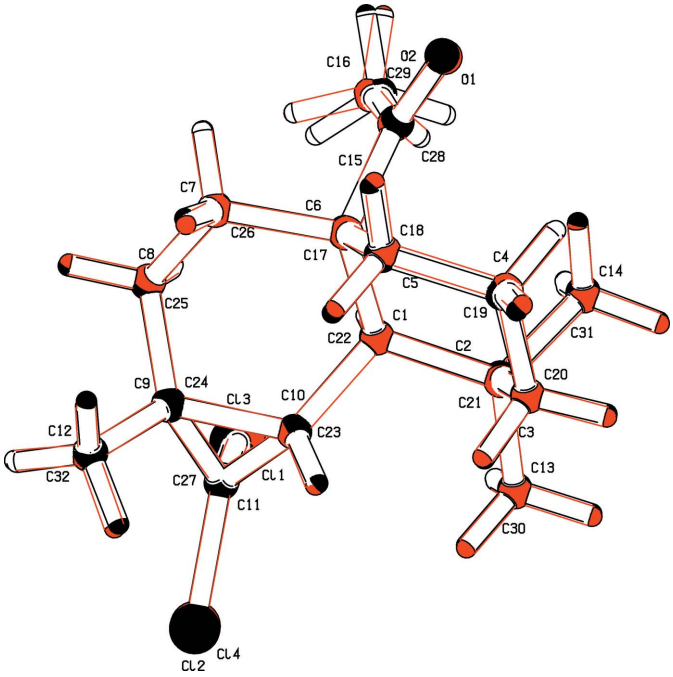


Figure 2
AutoMolFit (*PLATON*; Spek, 2009) of molecule B (red) on molecule A (white).

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{16}H_{24}Cl_2O$
M_r	303.25
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	296
a, b, c (Å)	13.373 (3), 13.996 (3), 17.219 (4)
V (Å^3)	3222.9 (12)
Z	8
Radiation type	Mo $K\alpha$
μ (mm^{-1})	0.39
Crystal size (mm)	0.24 × 0.2 × 0.15
Data collection	
Diffractometer	Bruker X8 APEX
Absorption correction	Multi-scan (*SADABS*; Sheldrick, 2008)
T_{min}, T_{max}	0.679, 0.747
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	42837, 6602, 6117
R_{int}	0.027
(sin $\theta/\lambda)_{max}$ (Å^{-1})	0.625
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.032, 0.084, 1.04
No. of reflections	6602
No. of parameters	351
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.25, −0.15
Absolute structure	Flack x determined using 2562 quotients $[(I^+)-(I^-)]/[(I^+)+(I^-)]$ (Parsons *et al.*, 2013)
Absolute structure parameter	−0.003 (11)

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

Synthesis and crystallization

BF_3–Et_2O (1 ml) was added dropwise to a solution of (1*S*,3*R*,8*S*)-2,2-dichloro-3,7,7,10-tetramethyltricyclo[6.4.0.0^{1,3}]-dodec-9-ene (1 g, 3.3 mmol; Lassaba *et al.*, 1997) in dichloromethane (60 ml) at 195 K under nitrogen. The reaction mixture was stirred for 2 h at a constant temperature of 195 K and then left at ambient temperature for 24 h. Water (60 ml) was added in order to separate the two phases, and the organic phase was dried and concentrated. The residue obtained was chromatographed on silica gel, eluting with hexane–ethyl acetate (97:3 *v*/*v*), which allowed the isolation of the title compound (yield: 34.7 mg, 1 mmol, 30%). Crystals suitable for X-ray analysis were obtained by slow evaporation from a hexane solution.

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. All H atoms were fixed geometrically and treated as riding, with C—H = 0.96 (methyl), 0.97 (methylene) or 0.98 Å (methine), and with U_{iso}(H) = 1.5U_{eq}(C) for methyl H atoms or 1.2U_{eq}(C) otherwise. A rotating model was used for the methyl groups. Two outliers (011 and 101) were omitted in the final cycles of refinement.

Acknowledgements

The authors thank the Unit of Support for Technical and Scientific Research (UATRS, CNRST) for the X-ray measurements.

References

Benharref, A., Elkarroumi, J., El Ammari, L., Saadi, M. & Berraho, M. (2015). *Acta Cryst.* E**71**, o659–o660.
Benharref, A., Oukhrib, A., Ait Elhad, M., ElAmmari, L., Saadi, M. & Berraho, M. (2016). *IUCrData*, **1**, x160703.
Benharref, A., Ourhriss, N., El Ammari, L., Saadi, M. & Berraho, M. (2013). *Acta Cryst.* E**69**, o933–o934.
Bruker (2009). *APEX2* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Daoubi, M., Duran-Patron, R., Hmamouchi, M., Hernandez-Galan, R., Benharref, A. & Isidro, G. C. (2004). *Pest. Manag. Sci.* **60**, 927–932.
El Haib, A., Benharref, A., Parrès-Maynadié, S., Manoury, E., Urrutigoïty, M. & Gouygou, M. (2011). *Tetrahedron Asymmetry*, **22**, 101–108.
El Jamili, H., Auhmani, A., Dakir, M., Lassaba, E., Benharref, A., Pierrot, M., Chiaroni, A. & Riche, C. (2002). *Tetrahedron Lett.* **43**, 6645–6648.
Farrugia, L. J. (2012). *J. Appl. Cryst.* **45**, 849–854.
Lassaba, E., Benharref, A., Giorgi, M. & Pierrot, M. (1997). *Acta Cryst.* C**53**, 1943–1945.
Parsons, S., Flack, H. D. & Wagner, T. (2013). *Acta Cryst.* B**69**, 249–259.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Sheldrick, G. M. (2015*a*). *Acta Cryst.* A**71**, 3–8.
Sheldrick, G. M. (2015*b*). *Acta Cryst.* C**71**, 3–8.
Spek, A. L. (2009). *Acta Cryst.* D**65**, 148–155.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.
Zaki, M., Benharref, A., El Ammari, L., Saadi, M. & Berraho, M. (2014). *Acta Cryst.* E**70**, o444.

full crystallographic data

IUCrData (2017). **2**, x170198 [https://doi.org/10.1107/S2414314617001985]

1-[(1*R*,2*S*,4*R*,7*S*)-3,3-Dichloro-4,11,11-trimethyltricyclo[5.4.0.0^{2,4}]undecan-7-yl]ethanone

Ahmed Benharref, Lahcen El Ammari, Mohamed Saadi, Mustapha Ait Elhad, Abdelouahd Oukhrib and Moha Berraho

1-[(1*R*,2*S*,4*R*,7*S*)-3,3-Dichloro-4,11,11-trimethyltricyclo[5.4.0.0^{2,4}]undecan-7-yl]ethanone

Crystal data

$C_{16}H_{24}Cl_2O$
M_r = 303.25
Orthorhombic, $P2_12_12_1$
a = 13.373 (3) Å
b = 13.996 (3) Å
c = 17.219 (4) Å
V = 3222.9 (12) Å^3
Z = 8
F(000) = 1296
D_x = 1.250 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 6602 reflections
θ = 2.1–26.4°
μ = 0.39 mm^{-1}
T = 296 K
Prismatic, colourless
0.24 × 0.2 × 0.15 mm

Data collection

Bruker X8 APEX diffractometer
Radiation source: fine-focus sealed tube
φ and ω scans
Absorption correction: multi-scan (SADABS; Sheldrick, 2008)
T_{min} = 0.679, T_{max} = 0.747
42837 measured reflections
6602 independent reflections
6117 reflections with $I > 2\sigma(I)$
R_{int} = 0.027
θ_{max} = 26.4°, θ_{min} = 2.1°
h = −16→16
k = −17→17
l = −21→21

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.084
S = 1.04
6602 reflections
351 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0451P)^2 + 0.5607P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.25 e Å^{-3}
$\Delta\rho_{min}$ = −0.15 e Å^{-3}
Absolute structure: Flack x determined using 2562 quotients [(I+)-(I-)]/[(I+)+(I-)] (Parsons *et al.*, 2013)
Absolute structure parameter: −0.003 (11)

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}
Cl1	0.36988 (6)	0.29957 (5)	0.55069 (4)	0.05611 (18)
Cl3	0.95794 (5)	0.53104 (6)	0.79873 (4)	0.0617 (2)
Cl2	0.17235 (7)	0.21684 (5)	0.54209 (6)	0.0714 (2)
Cl4	0.98018 (6)	0.32934 (6)	0.76753 (6)	0.0755 (3)
O2	0.3205 (2)	0.73129 (15)	0.45534 (15)	0.0799 (7)
C1	0.25547 (17)	0.50152 (15)	0.53392 (11)	0.0317 (4)
H1	0.3249	0.4834	0.5447	0.038*
C22	0.72518 (16)	0.49498 (15)	0.76550 (12)	0.0340 (4)
H22	0.7646	0.5487	0.7853	0.041*
C10	0.19975 (17)	0.41104 (16)	0.51128 (13)	0.0356 (5)
H10	0.1278	0.4117	0.5221	0.043*
C9	0.22970 (19)	0.36088 (17)	0.43609 (14)	0.0400 (5)
C23	0.79869 (17)	0.42024 (16)	0.73703 (13)	0.0370 (5)
H23	0.7747	0.3541	0.7387	0.044*
C17	0.66252 (17)	0.53344 (17)	0.69536 (13)	0.0401 (5)
C15	0.3382 (2)	0.64824 (18)	0.46980 (15)	0.0505 (6)
C24	0.86437 (18)	0.44530 (18)	0.66748 (14)	0.0418 (5)
C25	0.8422 (2)	0.54421 (19)	0.63672 (14)	0.0463 (6)
H25A	0.8789	0.5551	0.5889	0.056*
H25B	0.8627	0.5920	0.6743	0.056*
C21	0.6619 (2)	0.45593 (18)	0.83496 (14)	0.0446 (5)
O1	0.5301 (2)	0.6488 (2)	0.69931 (19)	0.0942 (9)
C6	0.25692 (19)	0.57096 (16)	0.46278 (13)	0.0390 (5)
C2	0.2116 (2)	0.54496 (19)	0.60989 (14)	0.0451 (6)
C26	0.7303 (2)	0.5520 (2)	0.62175 (14)	0.0484 (6)
H26A	0.7122	0.5064	0.5817	0.058*
H26B	0.7160	0.6154	0.6019	0.058*
C8	0.3150 (2)	0.41262 (18)	0.39601 (14)	0.0454 (6)
H8A	0.3280	0.3838	0.3458	0.054*
H8B	0.3752	0.4080	0.4271	0.054*
C11	0.24772 (19)	0.31444 (17)	0.51377 (15)	0.0424 (5)
C7	0.2858 (2)	0.51667 (19)	0.38574 (13)	0.0486 (6)
H7A	0.2295	0.5198	0.3503	0.058*
H7B	0.3412	0.5501	0.3615	0.058*
C18	0.5816 (2)	0.4601 (2)	0.67344 (17)	0.0559 (7)
H18A	0.5374	0.4886	0.6352	0.067*
H18B	0.6136	0.4055	0.6493	0.067*
C28	0.6174 (2)	0.6318 (2)	0.71223 (17)	0.0552 (7)
C5	0.1527 (2)	0.6152 (2)	0.45342 (18)	0.0587 (7)

H5A	0.1558	0.6640	0.4135	0.070*
H5B	0.1070	0.5661	0.4356	0.070*
C30	0.7319 (3)	0.4090 (2)	0.89434 (16)	0.0613 (8)
H30A	0.6937	0.3875	0.9382	0.092*
H30B	0.7809	0.4546	0.9112	0.092*
H30C	0.7649	0.3554	0.8708	0.092*
C27	0.90911 (19)	0.43261 (19)	0.74687 (15)	0.0459 (6)
C16	0.4435 (2)	0.6165 (3)	0.4879 (2)	0.0647 (8)
H16A	0.4900	0.6542	0.4585	0.097*
H16B	0.4511	0.5503	0.4744	0.097*
H16C	0.4563	0.6247	0.5424	0.097*
C3	0.1089 (2)	0.5890 (2)	0.59315 (19)	0.0625 (8)
H3A	0.0620	0.5382	0.5810	0.075*
H3B	0.0851	0.6211	0.6395	0.075*
C20	0.5847 (2)	0.3842 (2)	0.80557 (18)	0.0570 (7)
H20A	0.6191	0.3282	0.7859	0.068*
H20B	0.5428	0.3642	0.8486	0.068*
C31	0.6090 (3)	0.5389 (2)	0.87730 (17)	0.0615 (8)
H31A	0.5644	0.5706	0.8420	0.092*
H31B	0.6580	0.5836	0.8958	0.092*
H31C	0.5716	0.5142	0.9205	0.092*
C13	0.2015 (3)	0.4657 (3)	0.67104 (15)	0.0666 (9)
H13A	0.2657	0.4371	0.6800	0.100*
H13B	0.1557	0.4180	0.6527	0.100*
H13C	0.1769	0.4926	0.7187	0.100*
C12	0.1507 (2)	0.3210 (2)	0.38231 (18)	0.0618 (8)
H12A	0.1773	0.2671	0.3548	0.093*
H12B	0.1309	0.3693	0.3458	0.093*
H12C	0.0937	0.3013	0.4122	0.093*
C32	0.8843 (2)	0.3703 (2)	0.60542 (17)	0.0616 (8)
H0A	0.9474	0.3832	0.5809	0.092*
H0B	0.8320	0.3722	0.5673	0.092*
H0C	0.8862	0.3081	0.6290	0.092*
C19	0.5189 (2)	0.4252 (3)	0.7417 (2)	0.0694 (9)
H19A	0.4801	0.4780	0.7623	0.083*
H19B	0.4727	0.3765	0.7239	0.083*
C4	0.1105 (3)	0.6595 (2)	0.5268 (2)	0.0705 (9)
H4A	0.1508	0.7143	0.5413	0.085*
H4B	0.0430	0.6818	0.5169	0.085*
C14	0.2817 (3)	0.6214 (2)	0.64349 (17)	0.0617 (8)
H14A	0.2882	0.6730	0.6071	0.093*
H14B	0.3463	0.5939	0.6530	0.093*
H14C	0.2545	0.6452	0.6913	0.093*
C29	0.6857 (3)	0.7112 (2)	0.7387 (2)	0.0675 (8)
H29A	0.6538	0.7717	0.7300	0.101*
H29B	0.7471	0.7086	0.7098	0.101*
H29C	0.6998	0.7038	0.7930	0.101*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.0571 (4)	0.0460 (3)	0.0652 (4)	0.0108 (3)	−0.0111 (3)	0.0052 (3)
Cl3	0.0468 (4)	0.0750 (5)	0.0635 (4)	−0.0098 (3)	−0.0161 (3)	−0.0089 (4)
Cl2	0.0777 (5)	0.0410 (3)	0.0954 (6)	−0.0158 (3)	0.0135 (4)	0.0114 (4)
Cl4	0.0587 (5)	0.0715 (5)	0.0962 (6)	0.0261 (4)	−0.0045 (4)	0.0146 (4)
O2	0.113 (2)	0.0382 (11)	0.0888 (16)	−0.0112 (12)	−0.0047 (15)	0.0144 (11)
C1	0.0328 (10)	0.0319 (10)	0.0303 (10)	0.0008 (8)	0.0021 (8)	−0.0004 (8)
C22	0.0348 (11)	0.0313 (10)	0.0360 (10)	−0.0029 (9)	−0.0018 (9)	−0.0012 (8)
C10	0.0332 (11)	0.0340 (11)	0.0397 (11)	−0.0009 (9)	0.0043 (9)	−0.0009 (9)
C9	0.0428 (12)	0.0357 (11)	0.0415 (12)	0.0006 (10)	−0.0013 (10)	−0.0064 (9)
C23	0.0362 (11)	0.0341 (11)	0.0408 (12)	−0.0001 (9)	−0.0013 (9)	−0.0010 (9)
C17	0.0354 (11)	0.0423 (12)	0.0425 (11)	−0.0003 (10)	−0.0047 (9)	−0.0001 (10)
C15	0.0699 (18)	0.0381 (13)	0.0435 (13)	−0.0105 (12)	0.0003 (12)	0.0036 (10)
C24	0.0366 (12)	0.0450 (13)	0.0438 (12)	−0.0013 (10)	0.0018 (10)	−0.0038 (10)
C25	0.0473 (14)	0.0528 (14)	0.0387 (12)	−0.0057 (12)	0.0050 (10)	0.0057 (11)
C21	0.0492 (14)	0.0403 (12)	0.0443 (12)	−0.0044 (11)	0.0091 (10)	−0.0006 (10)
O1	0.0635 (15)	0.0844 (18)	0.135 (2)	0.0334 (14)	−0.0193 (16)	−0.0021 (17)
C6	0.0482 (13)	0.0342 (11)	0.0347 (11)	−0.0002 (10)	−0.0057 (10)	0.0033 (9)
C2	0.0487 (14)	0.0489 (14)	0.0378 (12)	0.0015 (12)	0.0080 (10)	−0.0089 (10)
C26	0.0528 (15)	0.0534 (15)	0.0391 (12)	0.0011 (12)	−0.0055 (11)	0.0060 (11)
C8	0.0536 (15)	0.0492 (14)	0.0334 (11)	−0.0016 (12)	0.0092 (10)	−0.0078 (10)
C11	0.0428 (13)	0.0322 (11)	0.0522 (13)	−0.0041 (10)	0.0033 (10)	0.0036 (10)
C7	0.0649 (16)	0.0484 (14)	0.0325 (11)	−0.0077 (13)	0.0022 (11)	0.0043 (10)
C18	0.0465 (14)	0.0605 (17)	0.0608 (16)	−0.0076 (13)	−0.0138 (12)	−0.0071 (14)
C28	0.0565 (16)	0.0521 (15)	0.0572 (15)	0.0159 (13)	−0.0039 (13)	0.0044 (12)
C5	0.0636 (18)	0.0466 (14)	0.0658 (17)	0.0117 (13)	−0.0223 (14)	0.0047 (13)
C30	0.079 (2)	0.0618 (18)	0.0433 (14)	−0.0028 (16)	0.0049 (14)	0.0144 (12)
C27	0.0382 (12)	0.0465 (14)	0.0530 (14)	0.0041 (11)	−0.0052 (10)	0.0002 (11)
C16	0.0553 (17)	0.0665 (19)	0.0724 (19)	−0.0230 (15)	0.0036 (15)	0.0022 (16)
C3	0.0478 (15)	0.0632 (18)	0.0764 (19)	0.0103 (14)	0.0148 (14)	−0.0240 (15)
C20	0.0551 (16)	0.0506 (15)	0.0652 (17)	−0.0151 (13)	0.0176 (14)	−0.0010 (13)
C31	0.0688 (19)	0.0599 (18)	0.0558 (15)	−0.0006 (15)	0.0206 (14)	−0.0106 (14)
C13	0.086 (2)	0.077 (2)	0.0370 (13)	−0.0027 (18)	0.0186 (14)	−0.0001 (13)
C12	0.0664 (19)	0.0543 (16)	0.0646 (17)	−0.0038 (14)	−0.0169 (15)	−0.0172 (13)
C32	0.0594 (17)	0.0658 (18)	0.0597 (16)	0.0008 (15)	0.0126 (14)	−0.0160 (14)
C19	0.0435 (15)	0.077 (2)	0.087 (2)	−0.0209 (15)	−0.0014 (15)	−0.0084 (18)
C4	0.0552 (18)	0.0583 (18)	0.098 (2)	0.0257 (15)	−0.0131 (17)	−0.0148 (17)
C14	0.0718 (19)	0.0671 (18)	0.0463 (14)	−0.0036 (16)	−0.0012 (14)	−0.0212 (13)
C29	0.092 (2)	0.0382 (13)	0.0719 (19)	0.0098 (15)	−0.0013 (17)	0.0010 (13)

Geometric parameters (Å, º)

Cl1—C11	1.765 (3)	C8—H8A	0.9700
Cl3—C27	1.767 (3)	C8—H8B	0.9700
Cl2—C11	1.766 (2)	C7—H7A	0.9700
Cl4—C27	1.766 (3)	C7—H7B	0.9700

O2—C15	1.212 (3)	C18—C19	1.524 (5)
C1—C10	1.520 (3)	C18—H18A	0.9700
C1—C2	1.557 (3)	C18—H18B	0.9700
C1—C6	1.564 (3)	C28—C29	1.508 (5)
C1—H1	0.9800	C5—C4	1.516 (5)
C22—C23	1.517 (3)	C5—H5A	0.9700
C22—C21	1.564 (3)	C5—H5B	0.9700
C22—C17	1.565 (3)	C30—H30A	0.9600
C22—H22	0.9800	C30—H30B	0.9600
C10—C11	1.497 (3)	C30—H30C	0.9600
C10—C9	1.526 (3)	C16—H16A	0.9600
C10—H10	0.9800	C16—H16B	0.9600
C9—C11	1.507 (3)	C16—H16C	0.9600
C9—C12	1.512 (4)	C3—C4	1.510 (5)
C9—C8	1.517 (4)	C3—H3A	0.9700
C23—C27	1.496 (3)	C3—H3B	0.9700
C23—C24	1.526 (3)	C20—C19	1.521 (5)
C23—H23	0.9800	C20—H20A	0.9700
C17—C28	1.531 (4)	C20—H20B	0.9700
C17—C18	1.538 (4)	C31—H31A	0.9600
C17—C26	1.580 (4)	C31—H31B	0.9600
C15—C16	1.508 (5)	C31—H31C	0.9600
C15—C6	1.539 (4)	C13—H13A	0.9600
C24—C27	1.503 (4)	C13—H13B	0.9600
C24—C25	1.512 (4)	C13—H13C	0.9600
C24—C32	1.522 (4)	C12—H12A	0.9600
C25—C26	1.522 (4)	C12—H12B	0.9600
C25—H25A	0.9700	C12—H12C	0.9600
C25—H25B	0.9700	C32—H0A	0.9600
C21—C20	1.526 (4)	C32—H0B	0.9600
C21—C30	1.534 (4)	C32—H0C	0.9600
C21—C31	1.543 (4)	C19—H19A	0.9700
O1—C28	1.213 (4)	C19—H19B	0.9700
C6—C5	1.534 (4)	C4—H4A	0.9700
C6—C7	1.577 (3)	C4—H4B	0.9700
C2—C3	1.533 (4)	C14—H14A	0.9600
C2—C13	1.535 (4)	C14—H14B	0.9600
C2—C14	1.536 (4)	C14—H14C	0.9600
C26—H26A	0.9700	C29—H29A	0.9600
C26—H26B	0.9700	C29—H29B	0.9600
C8—C7	1.518 (4)	C29—H29C	0.9600

C10—C1—C2	110.86 (18)	C19—C18—C17	114.3 (2)
C10—C1—C6	108.84 (17)	C19—C18—H18A	108.7
C2—C1—C6	114.84 (18)	C17—C18—H18A	108.7
C10—C1—H1	107.3	C19—C18—H18B	108.7
C2—C1—H1	107.3	C17—C18—H18B	108.7
C6—C1—H1	107.3	H18A—C18—H18B	107.6

C23—C22—C21	110.91 (18)	O1—C28—C29	119.7 (3)
C23—C22—C17	109.56 (18)	O1—C28—C17	121.4 (3)
C21—C22—C17	114.86 (19)	C29—C28—C17	118.7 (2)
C23—C22—H22	107.0	C4—C5—C6	114.6 (2)
C21—C22—H22	107.0	C4—C5—H5A	108.6
C17—C22—H22	107.0	C6—C5—H5A	108.6
C11—C10—C1	122.34 (19)	C4—C5—H5B	108.6
C11—C10—C9	59.77 (16)	C6—C5—H5B	108.6
C1—C10—C9	118.18 (19)	H5A—C5—H5B	107.6
C11—C10—H10	115.1	C21—C30—H30A	109.5
C1—C10—H10	115.1	C21—C30—H30B	109.5
C9—C10—H10	115.1	H30A—C30—H30B	109.5
C11—C9—C12	119.8 (2)	C21—C30—H30C	109.5
C11—C9—C8	119.3 (2)	H30A—C30—H30C	109.5
C12—C9—C8	115.0 (2)	H30B—C30—H30C	109.5
C11—C9—C10	59.16 (15)	C23—C27—C24	61.17 (16)
C12—C9—C10	120.4 (2)	C23—C27—Cl4	117.33 (19)
C8—C9—C10	111.31 (19)	C24—C27—Cl4	119.62 (19)
C27—C23—C22	121.5 (2)	C23—C27—Cl3	120.81 (19)
C27—C23—C24	59.62 (16)	C24—C27—Cl3	120.98 (19)
C22—C23—C24	117.92 (19)	Cl4—C27—Cl3	109.73 (14)
C27—C23—H23	115.4	C15—C16—H16A	109.5
C22—C23—H23	115.4	C15—C16—H16B	109.5
C24—C23—H23	115.4	H16A—C16—H16B	109.5
C28—C17—C18	111.7 (2)	C15—C16—H16C	109.5
C28—C17—C22	111.9 (2)	H16A—C16—H16C	109.5
C18—C17—C22	109.7 (2)	H16B—C16—H16C	109.5
C28—C17—C26	103.3 (2)	C4—C3—C2	113.1 (3)
C18—C17—C26	108.4 (2)	C4—C3—H3A	109.0
C22—C17—C26	111.60 (18)	C2—C3—H3A	109.0
O2—C15—C16	120.5 (3)	C4—C3—H3B	109.0
O2—C15—C6	121.3 (3)	C2—C3—H3B	109.0
C16—C15—C6	117.9 (2)	H3A—C3—H3B	107.8
C27—C24—C25	120.3 (2)	C19—C20—C21	112.5 (2)
C27—C24—C32	119.2 (2)	C19—C20—H20A	109.1
C25—C24—C32	114.9 (2)	C21—C20—H20A	109.1
C27—C24—C23	59.21 (16)	C19—C20—H20B	109.1
C25—C24—C23	111.9 (2)	C21—C20—H20B	109.1
C32—C24—C23	119.6 (2)	H20A—C20—H20B	107.8
C24—C25—C26	108.5 (2)	C21—C31—H31A	109.5
C24—C25—H25A	110.0	C21—C31—H31B	109.5
C26—C25—H25A	110.0	H31A—C31—H31B	109.5
C24—C25—H25B	110.0	C21—C31—H31C	109.5
C26—C25—H25B	110.0	H31A—C31—H31C	109.5
H25A—C25—H25B	108.4	H31B—C31—H31C	109.5
C20—C21—C30	110.6 (2)	C2—C13—H13A	109.5
C20—C21—C31	110.0 (2)	C2—C13—H13B	109.5
C30—C21—C31	106.7 (2)	H13A—C13—H13B	109.5

C20—C21—C22	110.0 (2)	C2—C13—H13C	109.5
C30—C21—C22	109.2 (2)	H13A—C13—H13C	109.5
C31—C21—C22	110.3 (2)	H13B—C13—H13C	109.5
C5—C6—C15	111.5 (2)	C9—C12—H12A	109.5
C5—C6—C1	108.8 (2)	C9—C12—H12B	109.5
C15—C6—C1	112.59 (19)	H12A—C12—H12B	109.5
C5—C6—C7	109.2 (2)	C9—C12—H12C	109.5
C15—C6—C7	103.4 (2)	H12A—C12—H12C	109.5
C1—C6—C7	111.26 (18)	H12B—C12—H12C	109.5
C3—C2—C13	109.9 (2)	C24—C32—H0A	109.5
C3—C2—C14	109.7 (2)	C24—C32—H0B	109.5
C13—C2—C14	107.4 (2)	H0A—C32—H0B	109.5
C3—C2—C1	109.7 (2)	C24—C32—H0C	109.5
C13—C2—C1	109.1 (2)	H0A—C32—H0C	109.5
C14—C2—C1	111.0 (2)	H0B—C32—H0C	109.5
C25—C26—C17	114.6 (2)	C20—C19—C18	111.1 (2)
C25—C26—H26A	108.6	C20—C19—H19A	109.4
C17—C26—H26A	108.6	C18—C19—H19A	109.4
C25—C26—H26B	108.6	C20—C19—H19B	109.4
C17—C26—H26B	108.6	C18—C19—H19B	109.4
H26A—C26—H26B	107.6	H19A—C19—H19B	108.0
C9—C8—C7	108.5 (2)	C3—C4—C5	111.6 (2)
C9—C8—H8A	110.0	C3—C4—H4A	109.3
C7—C8—H8A	110.0	C5—C4—H4A	109.3
C9—C8—H8B	110.0	C3—C4—H4B	109.3
C7—C8—H8B	110.0	C5—C4—H4B	109.3
H8A—C8—H8B	108.4	H4A—C4—H4B	108.0
C10—C11—C9	61.08 (15)	C2—C14—H14A	109.5
C10—C11—Cl1	120.89 (17)	C2—C14—H14B	109.5
C9—C11—Cl1	121.24 (18)	H14A—C14—H14B	109.5
C10—C11—Cl2	117.51 (17)	C2—C14—H14C	109.5
C9—C11—Cl2	119.17 (18)	H14A—C14—H14C	109.5
Cl1—C11—Cl2	109.73 (13)	H14B—C14—H14C	109.5
C8—C7—C6	115.29 (19)	C28—C29—H29A	109.5
C8—C7—H7A	108.5	C28—C29—H29B	109.5
C6—C7—H7A	108.5	H29A—C29—H29B	109.5
C8—C7—H7B	108.5	C28—C29—H29C	109.5
C6—C7—H7B	108.5	H29A—C29—H29C	109.5
H7A—C7—H7B	107.5	H29B—C29—H29C	109.5

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···Cl1	0.98	2.64	3.227 (2)	118
C8—H8*B*···Cl1	0.97	2.61	3.184 (3)	118
C22—H22···Cl3	0.98	2.61	3.205 (2)	120
C25—H25*B*···Cl3	0.97	2.63	3.196 (3)	117