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(1*S*,3*R*,6*S*)-3-Chloro-*trans*-himachalene

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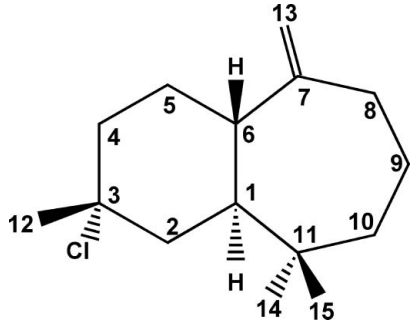
Key indicators: single-crystal X-ray study; T = 180 K; mean σ(C–C) = 0.004 Å; disorder in main residue; R factor = 0.044; wR factor = 0.132; data-to-parameter ratio = 21.2.

The title compound, $C_{15}H_{25}Cl$, was semi-synthesized from natural essential oils of *Cedrus atlantica*. The stereochemistry has been confirmed by single-crystal X-ray diffraction. The asymmetric unit contains two chemically identical molecules. Each is built up from two fused six- and seven-membered rings. In both molecules, the six-membered ring has a perfect chair conformation, whereas the seven-membered ring displays a twist-chair conformation. In one molecule, the seven-membered ring is partially disordered with a site-occupancy ratio of 0.52:0.48.

Related literature

For general background see: El Jamili *et al.* (2002); Dakir *et al.* (2004); Daoubi *et al.* (2005); Kav *et al.* (1992). For a related structure see: Ourhriss *et al.* (2007); Cremer & Pople (1975).



Experimental

Crystal data

$C_{15}H_{25}Cl$
M_r = 240.80
Orthorhombic, $P2_12_12_1$
a = 6.0470 (2) Å
b = 15.7158 (7) Å
c = 29.6845 (12) Å
V = 2821.02 (19) Å^3
Z = 8
Mo $K\alpha$ radiation
μ = 0.25 mm^{-1}
T = 180 (2) K
0.65 × 0.24 × 0.21 mm

Data collection

Oxford Diffraction Xcalibur CCD diffractometer
Absorption correction: multi-scan (*CrysAlis RED*; Oxford Diffraction, 2006)
T_{min} = 0.756, T_{max} = 1.000 (expected range = 0.718–0.950)
23369 measured reflections
6222 independent reflections
3666 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.132
S = 1.01
6222 reflections
294 parameters
15 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.41 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}
Absolute structure: Flack (1983), based on 2652 Friedel pairs
Flack parameter: −0.11 (7)

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2006); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2006); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPIII* (Burnett & Johnson, 1996) and *ORTEP-3 for Windows* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: CS2043).

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supplementary materials

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(1*S*,3*R*,6*S*)-3-Chloro-*trans*-himachalene

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Comment

The isolated sesquiterpenes of *Cedrus atlantica* essential oils were the subject of hemisynthesis in order to prepare chlorinated sesquiterpenoid compounds (El Jamili *et al.*, 2002; Dakir *et al.*, 2004) in good yield. Indeed, these compounds were tested, using the food poisoning technique, for their potential antifungal activity against the phytopathogen *Botrytis cinerea* (Daoubi *et al.*, 2005). Other molecules containing chlorine atom induces stalk-cell differentiation during development of *Dictystelium discoideum* (Kav *et al.*, 1992).

We were interested in the study on the reactivity of compound (A) (Ourhriss *et al.*, 2007) which has two chlorine atoms at different positions, 3 and 7 (Fig. 1). The dehydrohalogenation of (A) gave, after heating at reflux in methanol, the title compound (I) with high chemoselectivity. ^{1}H and ^{13}C NMR spectroscopy did not make it possible to identify the exact structure of this product. Single-crystal X-ray diffraction analysis allowed us to elucidate its configuration and to identify (I) as (1*S*,3*R*,6*S*)-3-chloro-*trans*-himachalene.

The molecule is built up from two fused six-membered and seven-membered rings (Fig. 2). The six membered ring has a perfect chair conformation as indicated by the total puckering amplitude QT= 0.560 (3) Å[0.546 (3) Å] and spherical polar angle θ=168.4 (3)° [174.1 (3)°] with φ= 117 (2)°[121 (3)°] whereas the seven-membered ring in the non disordered molecule displays a twist-chair conformation with QT= 0.807 (7) Å, θ= 36.7 (7)°, φ2= −179.8 (5)° and φ3= 120.8 (3)° (Cremer & Pople, 1975).

Owing to the presence of the Cl atom, the absolute configuration could be fully confirmed to be C1(*S*), C3(*R*) and C6(*S*) (Flack, 1983).

Experimental

1 g (4.16 mmol) of compound (A) was dissolved in 15 ml of methanol. The mixture was heated until total dissolution, and then left resting at 0°C for 1 h. All this allowed us, after filtering under reduced pressure, to prepare (1*S*,3*R*,6*S*)-3-chloro-*trans*-himachalene, (I) in 84% yield. Suitable crystals were obtained by evaporation of a hexane solution at 277 K. m.p. = 325–326 K (hexane); Spectroscopic analysis: ^{1}H NMR (300 MHz, $CDCl_3$) δ (p.p.m.):1.63 (3H12, s), 4.65, 4.67 (H^a-13, H^b-13, 2 s), 0.80, 0.90 (3H14, 3H15, 2 s); ^{13}C NMR (75 MHz, $CDCl_3$) δ (p.p.m.): 47.9 (C-1), 43.0 (C-2), 72.0 (C-3), 42.9 (C-4), 24.4 (C-5), 43.3 (C-6), 155.9 (C-7), 41.5 (C-8), 32.9 (C-9), 31.7 (C-10) 43.4 (C-11), 34.7 (C-12), 110.5 (C-13), 30.2 (C-14), 22.3 (C-15).

Refinement

All H atoms were fixed geometrically and treated as riding with C—H = 0.99 Å (CH_2), 1.0 Å(CH) or 0.98 Å (CH_3) with U_{iso}(H) = 1.2U_{eq}(CH, CH_2) or U_{iso}(H) = 1.5U_{eq}(CH_3).

Figures

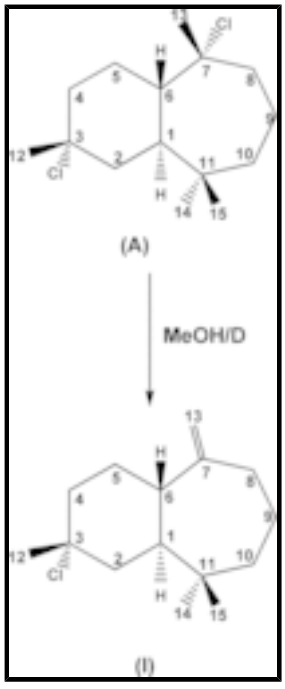

Fig. 1. Scheme showing the transformation of compound (A) to the title compound (I)

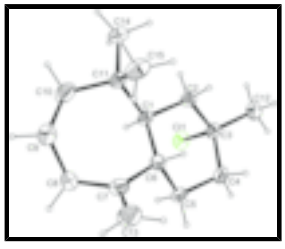

Fig. 2. View of compound I with the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level, H atoms are represented as small spheres of arbitrary radii.

(1*S*,3*R*,6*S*)-3-Chloro-*trans*-himachalene

Crystal data

$C_{15}H_{25}Cl$	F_{000} = 1056
M_r = 240.80	D_x = 1.134 Mg m^{-3}
Orthorhombic, $P2_12_12_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac 2ab	Cell parameters from 3489 reflections
a = 6.0470 (2) Å	θ = 2.7–32.1°
b = 15.7158 (7) Å	μ = 0.25 mm^{-1}
c = 29.6845 (12) Å	T = 180 (2) K
V = 2821.02 (19) Å^3	Prism, colourless
Z = 8	0.65 × 0.24 × 0.21 mm

Data collection

Oxford Diffraction CCD Xcalibur	6222 independent reflections

diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

Detector resolution: 8.2632 pixels mm^{-1}

T = 180(2) K

ω and φ scans

Absorption correction: multi-scan (CrysAlis RED; Oxford Diffraction, 2006)

T_{min} = 0.756, T_{max} = 1.000

23369 measured reflections

3666 reflections with $I > 2\sigma(I)$

R_{int} = 0.047

θ_{max} = 27.1º

θ_{min} = 2.7º

$h = -7 \rightarrow 5$

$k = -20 \rightarrow 20$

$l = -38 \rightarrow 38$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)]$ = 0.044

$wR(F^2)$ = 0.132

S = 1.01

6222 reflections

294 parameters

15 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

$w = 1/[\sigma^2(F_o^2) + (0.0662P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max}$ = 0.001

$\Delta\rho_{max}$ = 0.41 e $Å^{-3}$

$\Delta\rho_{min}$ = −0.39 e $Å^{-3}$

Extinction correction: none

Absolute structure: Flack (1983), based on 2652 Friedel pairs

Flack parameter: −0.11 (7)

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters ($Å^2$)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cl2	0.17012 (12)	−0.00184 (5)	0.82920 (3)	0.0474 (2)	
C1	0.5574 (5)	0.12081 (18)	0.87157 (9)	0.0324 (7)	
H1	0.4003	0.1220	0.8820	0.039*	
C2	0.5564 (5)	0.08395 (17)	0.82356 (9)	0.0340 (7)	
H21	0.4669	0.1220	0.8042	0.041*	
H22	0.7098	0.0852	0.8119	0.041*	
C3	0.4693 (4)	−0.00486 (19)	0.81847 (10)	0.0341 (7)	
C4	0.5669 (5)	−0.06490 (19)	0.85311 (10)	0.0394 (8)	
H41	0.7214	−0.0782	0.8446	0.047*	
H42	0.4822	−0.1188	0.8528	0.047*	
C5	0.5647 (5)	−0.02863 (17)	0.90055 (10)	0.0383 (8)	
H51	0.6399	−0.0686	0.9213	0.046*	
H52	0.4099	−0.0220	0.9108	0.046*	
C6	0.6811 (5)	0.05764 (18)	0.90211 (9)	0.0335 (7)	
H6	0.8301	0.0490	0.8882	0.040*	
C7	0.7212 (6)	0.0838 (2)	0.95016 (11)	0.0456 (9)	
C8	0.5674 (8)	0.1440 (2)	0.97280 (11)	0.0621 (11)	

H81	0.5793	0.1370	1.0059	0.075*	
H82	0.4135	0.1306	0.9639	0.075*	
C9	0.6195 (10)	0.2348 (2)	0.96037 (12)	0.0794 (14)	
H91	0.5466	0.2727	0.9825	0.095*	
H92	0.7810	0.2434	0.9633	0.095*	
C10	0.5521 (7)	0.2611 (2)	0.91505 (12)	0.0606 (11)	
H10A	0.5904	0.3221	0.9119	0.073*	
H10B	0.3889	0.2571	0.9137	0.073*	
C11	0.6439 (5)	0.21461 (19)	0.87306 (10)	0.0369 (7)	
C12	0.4969 (6)	−0.0370 (2)	0.77094 (10)	0.0480 (9)	
H12A	0.4188	0.0010	0.7501	0.072*	
H12B	0.4353	−0.0945	0.7686	0.072*	
H12C	0.6544	−0.0383	0.7632	0.072*	
C13	0.8968 (8)	0.0533 (2)	0.97185 (13)	0.0701 (12)	
H13A	0.9248	0.0700	1.0021	0.084*	
H13B	0.9941	0.0148	0.9571	0.084*	
C14	0.5492 (6)	0.2663 (2)	0.83362 (12)	0.0525 (9)	
H14A	0.5889	0.3264	0.8373	0.079*	
H14B	0.3878	0.2607	0.8332	0.079*	
H14C	0.6104	0.2450	0.8052	0.079*	
C15	0.8931 (5)	0.2188 (2)	0.87064 (13)	0.0588 (10)	
H15A	0.9445	0.1878	0.8440	0.088*	
H15B	0.9565	0.1928	0.8977	0.088*	
H15C	0.9400	0.2783	0.8686	0.088*	
Cl1A	0.61711 (12)	0.71053 (6)	0.79807 (3)	0.0478 (2)	
C2A	1.0131 (5)	0.73751 (18)	0.83943 (9)	0.0317 (7)	
H2A1	0.9427	0.7910	0.8496	0.038*	
H2A2	1.1734	0.7487	0.8361	0.038*	
C3A	0.9197 (4)	0.7146 (2)	0.79321 (9)	0.0367 (7)	
C4A	0.9978 (5)	0.6273 (2)	0.77819 (10)	0.0448 (9)	
H4A1	0.9157	0.6106	0.7507	0.054*	
H4A2	1.1567	0.6305	0.7703	0.054*	
C5A	0.9659 (6)	0.5603 (2)	0.81364 (10)	0.0464 (9)	
H5A1	1.0302	0.5061	0.8029	0.056*	
H5A2	0.8056	0.5513	0.8185	0.056*	
C6A	1.0739 (5)	0.58486 (18)	0.85846 (10)	0.0371 (8)	
H6A	1.2356	0.5927	0.8528	0.044*	
C7A	1.0477 (6)	0.5107 (2)	0.89092 (12)	0.0518 (9)	
C8A	0.8830 (13)	0.5086 (7)	0.9265 (2)	0.0495 (10)	0.52
H8A1	0.8651	0.4490	0.9367	0.059*	0.52
H8A2	0.7396	0.5274	0.9138	0.059*	0.52
C9A	0.9364 (13)	0.5634 (4)	0.9670 (2)	0.0495 (10)	0.52
H9A1	0.8307	0.5491	0.9913	0.059*	0.52
H9A2	1.0861	0.5481	0.9778	0.059*	0.52
C10A	0.9295 (12)	0.6582 (4)	0.9596 (2)	0.0495 (10)	0.52
H10C	0.9698	0.6856	0.9885	0.059*	0.52
H10D	0.7737	0.6736	0.9534	0.059*	0.52
C11A	1.0726 (5)	0.70010 (18)	0.92240 (9)	0.0325 (7)	
C14A	1.3124 (11)	0.6824 (4)	0.9264 (3)	0.0462 (13)	0.52

H14D	1.3924	0.7134	0.9028	0.069*	0.52
H14E	1.3385	0.6212	0.9229	0.069*	0.52
H14F	1.3649	0.7010	0.9560	0.069*	0.52
C15A	1.0291 (11)	0.7947 (5)	0.9304 (3)	0.0462 (13)	0.52
H15D	1.0688	0.8094	0.9614	0.069*	0.52
H15E	0.8720	0.8069	0.9254	0.069*	0.52
H15F	1.1185	0.8285	0.9095	0.069*	0.52
C1A	0.9818 (5)	0.67051 (17)	0.87616 (9)	0.0287 (7)	
H1A	0.8187	0.6627	0.8799	0.034*	
C8B	0.8358 (14)	0.5152 (7)	0.9182 (3)	0.0495 (10)	0.48
H8B1	0.7978	0.4578	0.9296	0.059*	0.48
H8B2	0.7132	0.5349	0.8987	0.059*	0.48
C9B	0.8644 (13)	0.5785 (5)	0.9593 (3)	0.0495 (10)	0.48
H9B1	0.7345	0.6168	0.9598	0.059*	0.48
H9B2	0.8606	0.5445	0.9874	0.059*	0.48
C10B	1.0686 (13)	0.6326 (4)	0.9603 (2)	0.0495 (10)	0.48
H10E	1.2002	0.5956	0.9574	0.059*	0.48
H10F	1.0776	0.6618	0.9898	0.059*	0.48
C14B	1.3206 (12)	0.7264 (5)	0.9206 (3)	0.0462 (13)	0.48
H14G	1.3760	0.7349	0.9513	0.069*	0.48
H14H	1.3356	0.7795	0.9035	0.069*	0.48
H14I	1.4063	0.6815	0.9058	0.069*	0.48
C15B	0.9445 (12)	0.7763 (5)	0.9386 (3)	0.0462 (13)	0.48
H15G	1.0012	0.7942	0.9681	0.069*	0.48
H15H	0.7877	0.7614	0.9414	0.069*	0.48
H15I	0.9611	0.8230	0.9170	0.069*	0.48
C12A	0.9693 (6)	0.7850 (2)	0.75919 (11)	0.0520 (9)	
H12D	1.1293	0.7885	0.7543	0.078*	
H12E	0.9152	0.8395	0.7709	0.078*	
H12F	0.8953	0.7722	0.7306	0.078*	
C13A	1.1925 (9)	0.4474 (2)	0.88913 (15)	0.0889 (16)	
H13C	1.1861	0.4028	0.9107	0.107*	
H13D	1.3026	0.4467	0.8663	0.107*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl2	0.0276 (4)	0.0498 (5)	0.0649 (5)	−0.0004 (4)	0.0030 (4)	−0.0004 (4)
C1	0.0256 (16)	0.0303 (16)	0.0412 (17)	0.0049 (14)	0.0028 (14)	0.0051 (14)
C2	0.0289 (16)	0.0327 (16)	0.0403 (17)	0.0018 (14)	0.0020 (14)	0.0068 (14)
C3	0.0228 (14)	0.0345 (16)	0.0449 (17)	0.0037 (15)	0.0040 (13)	0.0004 (15)
C4	0.0344 (18)	0.0306 (16)	0.0532 (19)	0.0034 (15)	0.0054 (16)	−0.0015 (15)
C5	0.0431 (19)	0.0285 (16)	0.0432 (18)	0.0067 (15)	0.0044 (16)	0.0083 (14)
C6	0.0263 (15)	0.0369 (17)	0.0372 (16)	0.0071 (15)	0.0050 (14)	0.0029 (14)
C7	0.051 (2)	0.0402 (19)	0.046 (2)	0.0000 (17)	−0.0070 (18)	0.0090 (16)
C8	0.094 (3)	0.051 (2)	0.041 (2)	0.010 (2)	0.013 (2)	−0.0008 (17)
C9	0.127 (4)	0.053 (2)	0.059 (3)	0.018 (3)	0.013 (3)	−0.005 (2)
C10	0.077 (3)	0.0359 (19)	0.068 (2)	−0.003 (2)	−0.007 (2)	−0.0039 (18)

C11	0.0292 (16)	0.0320 (16)	0.0495 (18)	0.0028 (15)	0.0000 (15)	0.0026 (15)
C12	0.051 (2)	0.047 (2)	0.0461 (19)	0.0050 (18)	0.0036 (18)	−0.0039 (16)
C13	0.089 (3)	0.060 (2)	0.061 (2)	0.010 (3)	−0.026 (2)	0.002 (2)
C14	0.056 (2)	0.0362 (18)	0.065 (2)	−0.0034 (17)	−0.003 (2)	0.0113 (17)
C15	0.0369 (19)	0.044 (2)	0.096 (3)	−0.0079 (18)	−0.005 (2)	0.011 (2)
Cl1A	0.0273 (4)	0.0734 (6)	0.0428 (4)	0.0024 (4)	−0.0038 (4)	−0.0059 (4)
C2A	0.0239 (15)	0.0372 (16)	0.0340 (16)	−0.0006 (13)	0.0011 (13)	−0.0088 (13)
C3A	0.0222 (14)	0.054 (2)	0.0334 (16)	−0.0012 (16)	0.0013 (13)	−0.0075 (16)
C4A	0.0354 (18)	0.062 (2)	0.0371 (17)	0.0020 (18)	0.0024 (16)	−0.0217 (17)
C5A	0.044 (2)	0.0458 (19)	0.049 (2)	−0.0010 (17)	−0.0041 (16)	−0.0180 (17)
C6A	0.0301 (17)	0.0368 (18)	0.0442 (18)	0.0043 (15)	0.0017 (15)	−0.0124 (15)
C7A	0.062 (2)	0.0309 (18)	0.062 (2)	0.0065 (19)	−0.026 (2)	−0.0100 (17)
C8A	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C9A	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C10A	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C11A	0.0317 (16)	0.0375 (17)	0.0283 (15)	0.0021 (15)	−0.0019 (13)	−0.0042 (13)
C14A	0.037 (2)	0.050 (3)	0.051 (2)	0.011 (3)	−0.0151 (18)	−0.023 (2)
C15A	0.037 (2)	0.050 (3)	0.051 (2)	0.011 (3)	−0.0151 (18)	−0.023 (2)
C1A	0.0210 (14)	0.0341 (16)	0.0310 (15)	0.0010 (13)	0.0014 (13)	−0.0045 (13)
C8B	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C9B	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C10B	0.051 (3)	0.056 (2)	0.0415 (17)	−0.005 (2)	−0.0182 (18)	0.0160 (15)
C14B	0.037 (2)	0.050 (3)	0.051 (2)	0.011 (3)	−0.0151 (18)	−0.023 (2)
C15B	0.037 (2)	0.050 (3)	0.051 (2)	0.011 (3)	−0.0151 (18)	−0.023 (2)
C12A	0.044 (2)	0.071 (2)	0.0409 (19)	0.000 (2)	0.0039 (16)	0.0043 (18)
C13A	0.129 (4)	0.050 (2)	0.087 (3)	0.035 (3)	−0.033 (3)	−0.014 (2)

Geometric parameters (Å, °)

Cl2—C3	1.838 (3)	C4A—H4A2	0.9900
C1—C2	1.538 (4)	C5A—C6A	1.531 (4)
C1—C6	1.538 (4)	C5A—H5A1	0.9900
C1—C11	1.565 (4)	C5A—H5A2	0.9900
C1—H1	1.0000	C6A—C7A	1.521 (4)
C2—C3	1.499 (4)	C6A—C1A	1.549 (4)
C2—H21	0.9900	C6A—H6A	1.0000
C2—H22	0.9900	C7A—C13A	1.326 (5)
C3—C12	1.508 (4)	C7A—C8A	1.451 (9)
C3—C4	1.515 (4)	C7A—C8B	1.518 (10)
C4—C5	1.519 (4)	C8A—C9A	1.515 (8)
C4—H41	0.9900	C8A—H8A1	0.9900
C4—H42	0.9900	C8A—H8A2	0.9900
C5—C6	1.528 (4)	C9A—C10A	1.507 (9)
C5—H51	0.9900	C9A—H9A1	0.9900
C5—H52	0.9900	C9A—H9A2	0.9900
C6—C7	1.504 (4)	C10A—C11A	1.549 (7)
C6—H6	1.0000	C10A—H10C	0.9900
C7—C13	1.331 (5)	C10A—H10D	0.9900
C7—C8	1.488 (5)	C11A—C14A	1.481 (7)

C8—C9	1.507 (5)	C11A—C15B	1.506 (8)
C8—H81	0.9900	C11A—C15A	1.528 (7)
C8—H82	0.9900	C11A—C10B	1.547 (6)
C9—C10	1.465 (5)	C11A—C1A	1.550 (4)
C9—H91	0.9900	C11A—C14B	1.557 (8)
C9—H92	0.9900	C14A—H14D	0.9800
C10—C11	1.548 (4)	C14A—H14E	0.9800
C10—H10A	0.9900	C14A—H14F	0.9800
C10—H10B	0.9900	C15A—H15D	0.9800
C11—C15	1.510 (4)	C15A—H15E	0.9800
C11—C14	1.536 (4)	C15A—H15F	0.9800
C12—H12A	0.9800	C1A—H1A	1.0000
C12—H12B	0.9800	C8B—C9B	1.583 (9)
C12—H12C	0.9800	C8B—H8B1	0.9900
C13—H13A	0.9500	C8B—H8B2	0.9900
C13—H13B	0.9500	C9B—C10B	1.500 (10)
C14—H14A	0.9800	C9B—H9B1	0.9900
C14—H14B	0.9800	C9B—H9B2	0.9900
C14—H14C	0.9800	C10B—H10E	0.9900
C15—H15A	0.9800	C10B—H10F	0.9900
C15—H15B	0.9800	C14B—H14G	0.9800
C15—H15C	0.9800	C14B—H14H	0.9800
Cl1A—C3A	1.837 (3)	C14B—H14I	0.9800
C2A—C3A	1.527 (4)	C15B—H15G	0.9800
C2A—C1A	1.528 (4)	C15B—H15H	0.9800
C2A—H2A1	0.9900	C15B—H15I	0.9800
C2A—H2A2	0.9900	C12A—H12D	0.9800
C3A—C4A	1.518 (4)	C12A—H12E	0.9800
C3A—C12A	1.527 (4)	C12A—H12F	0.9800
C4A—C5A	1.501 (4)	C13A—H13C	0.9500
C4A—H4A1	0.9900	C13A—H13D	0.9500
C2—C1—C6	107.8 (2)	C4A—C5A—H5A2	109.2
C2—C1—C11	112.5 (2)	C6A—C5A—H5A2	109.2
C6—C1—C11	115.4 (2)	H5A1—C5A—H5A2	107.9
C2—C1—H1	106.9	C7A—C6A—C5A	108.3 (2)
C6—C1—H1	106.9	C7A—C6A—C1A	114.4 (2)
C11—C1—H1	106.9	C5A—C6A—C1A	111.1 (2)
C3—C2—C1	116.4 (2)	C7A—C6A—H6A	107.6
C3—C2—H21	108.2	C5A—C6A—H6A	107.6
C1—C2—H21	108.2	C1A—C6A—H6A	107.6
C3—C2—H22	108.2	C13A—C7A—C8A	117.7 (5)
C1—C2—H22	108.2	C13A—C7A—C8B	127.9 (6)
H21—C2—H22	107.3	C8A—C7A—C8B	14.9 (5)
C2—C3—C12	111.5 (3)	C13A—C7A—C6A	118.8 (4)
C2—C3—C4	112.0 (2)	C8A—C7A—C6A	123.3 (5)
C12—C3—C4	112.5 (3)	C8B—C7A—C6A	113.0 (5)
C2—C3—Cl2	107.7 (2)	C7A—C8A—C9A	114.8 (6)
C12—C3—Cl2	106.2 (2)	C7A—C8A—H8A1	108.6
C4—C3—Cl2	106.4 (2)	C9A—C8A—H8A1	108.6

C3—C4—C5	113.1 (2)	C7A—C8A—H8A2	108.6
C3—C4—H41	109.0	C9A—C8A—H8A2	108.6
C5—C4—H41	109.0	H8A1—C8A—H8A2	107.6
C3—C4—H42	109.0	C10A—C9A—C8A	116.1 (6)
C5—C4—H42	109.0	C10A—C9A—H9A1	108.3
H41—C4—H42	107.8	C8A—C9A—H9A1	108.3
C4—C5—C6	110.9 (2)	C10A—C9A—H9A2	108.3
C4—C5—H51	109.5	C8A—C9A—H9A2	108.3
C6—C5—H51	109.5	H9A1—C9A—H9A2	107.4
C4—C5—H52	109.5	C9A—C10A—C11A	120.6 (6)
C6—C5—H52	109.5	C9A—C10A—H10C	107.2
H51—C5—H52	108.0	C11A—C10A—H10C	107.2
C7—C6—C5	110.2 (2)	C9A—C10A—H10D	107.2
C7—C6—C1	117.5 (2)	C11A—C10A—H10D	107.2
C5—C6—C1	109.3 (2)	H10C—C10A—H10D	106.8
C7—C6—H6	106.4	C14A—C11A—C15B	128.8 (5)
C5—C6—H6	106.4	C14A—C11A—C15A	109.8 (4)
C1—C6—H6	106.4	C15B—C11A—C15A	24.2 (4)
C13—C7—C8	120.6 (3)	C14A—C11A—C10B	80.1 (5)
C13—C7—C6	119.3 (3)	C15B—C11A—C10B	107.7 (5)
C8—C7—C6	120.1 (3)	C15A—C11A—C10B	123.4 (5)
C7—C8—C9	111.2 (3)	C14A—C11A—C10A	114.2 (5)
C7—C8—H81	109.4	C15B—C11A—C10A	79.8 (4)
C9—C8—H81	109.4	C15A—C11A—C10A	101.9 (4)
C7—C8—H82	109.4	C10B—C11A—C10A	35.1 (3)
C9—C8—H82	109.4	C14A—C11A—C1A	111.2 (3)
H81—C8—H82	108.0	C15B—C11A—C1A	109.9 (4)
C10—C9—C8	115.7 (4)	C15A—C11A—C1A	111.6 (4)
C10—C9—H91	108.4	C10B—C11A—C1A	115.7 (3)
C8—C9—H91	108.4	C10A—C11A—C1A	107.8 (3)
C10—C9—H92	108.4	C14A—C11A—C14B	27.0 (3)
C8—C9—H92	108.4	C15B—C11A—C14B	107.2 (5)
H91—C9—H92	107.4	C15A—C11A—C14B	85.0 (4)
C9—C10—C11	120.4 (3)	C10B—C11A—C14B	102.9 (4)
C9—C10—H10A	107.2	C10A—C11A—C14B	132.5 (5)
C11—C10—H10A	107.2	C1A—C11A—C14B	113.0 (4)
C9—C10—H10B	107.2	C11A—C14A—H14D	109.5
C11—C10—H10B	107.2	C11A—C14A—H14E	109.5
H10A—C10—H10B	106.9	C11A—C14A—H14F	109.5
C15—C11—C14	108.2 (3)	C11A—C15A—H15D	109.5
C15—C11—C10	112.1 (3)	C11A—C15A—H15E	109.5
C14—C11—C10	103.3 (3)	C11A—C15A—H15F	109.5
C15—C11—C1	111.9 (3)	C2A—C1A—C6A	108.2 (2)
C14—C11—C1	110.6 (2)	C2A—C1A—C11A	112.4 (2)
C10—C11—C1	110.3 (2)	C6A—C1A—C11A	115.7 (2)
C3—C12—H12A	109.5	C2A—C1A—H1A	106.7
C3—C12—H12B	109.5	C6A—C1A—H1A	106.7
H12A—C12—H12B	109.5	C11A—C1A—H1A	106.7
C3—C12—H12C	109.5	C7A—C8B—C9B	110.4 (6)

H12A—C12—H12C	109.5	C7A—C8B—H8B1	109.6
H12B—C12—H12C	109.5	C9B—C8B—H8B1	109.6
C7—C13—H13A	120.0	C7A—C8B—H8B2	109.6
C7—C13—H13B	120.0	C9B—C8B—H8B2	109.6
H13A—C13—H13B	120.0	H8B1—C8B—H8B2	108.1
C11—C14—H14A	109.5	C10B—C9B—C8B	117.5 (7)
C11—C14—H14B	109.5	C10B—C9B—H9B1	107.9
H14A—C14—H14B	109.5	C8B—C9B—H9B1	107.9
C11—C14—H14C	109.5	C10B—C9B—H9B2	107.9
H14A—C14—H14C	109.5	C8B—C9B—H9B2	107.9
H14B—C14—H14C	109.5	H9B1—C9B—H9B2	107.2
C11—C15—H15A	109.5	C9B—C10B—C11A	112.8 (5)
C11—C15—H15B	109.5	C9B—C10B—H10E	109.0
H15A—C15—H15B	109.5	C11A—C10B—H10E	109.0
C11—C15—H15C	109.5	C9B—C10B—H10F	109.0
H15A—C15—H15C	109.5	C11A—C10B—H10F	109.0
H15B—C15—H15C	109.5	H10E—C10B—H10F	107.8
C3A—C2A—C1A	115.7 (2)	C11A—C14B—H14G	109.5
C3A—C2A—H2A1	108.4	C11A—C14B—H14H	109.5
C1A—C2A—H2A1	108.4	H14G—C14B—H14H	109.5
C3A—C2A—H2A2	108.4	C11A—C14B—H14I	109.5
C1A—C2A—H2A2	108.4	H14G—C14B—H14I	109.5
H2A1—C2A—H2A2	107.4	H14H—C14B—H14I	109.5
C4A—C3A—C2A	111.2 (2)	C11A—C15B—H15G	109.5
C4A—C3A—C12A	113.5 (2)	C11A—C15B—H15H	109.5
C2A—C3A—C12A	110.6 (2)	H15G—C15B—H15H	109.5
C4A—C3A—Cl1A	107.5 (2)	C11A—C15B—H15I	109.5
C2A—C3A—Cl1A	107.83 (19)	H15G—C15B—H15I	109.5
C12A—C3A—Cl1A	105.9 (2)	H15H—C15B—H15I	109.5
C5A—C4A—C3A	112.8 (2)	C3A—C12A—H12D	109.5
C5A—C4A—H4A1	109.0	C3A—C12A—H12E	109.5
C3A—C4A—H4A1	109.0	H12D—C12A—H12E	109.5
C5A—C4A—H4A2	109.0	C3A—C12A—H12F	109.5
C3A—C4A—H4A2	109.0	H12D—C12A—H12F	109.5
H4A1—C4A—H4A2	107.8	H12E—C12A—H12F	109.5
C4A—C5A—C6A	112.2 (3)	C7A—C13A—H13C	120.0
C4A—C5A—H5A1	109.2	C7A—C13A—H13D	120.0
C6A—C5A—H5A1	109.2	H13C—C13A—H13D	120.0

Fig. 1

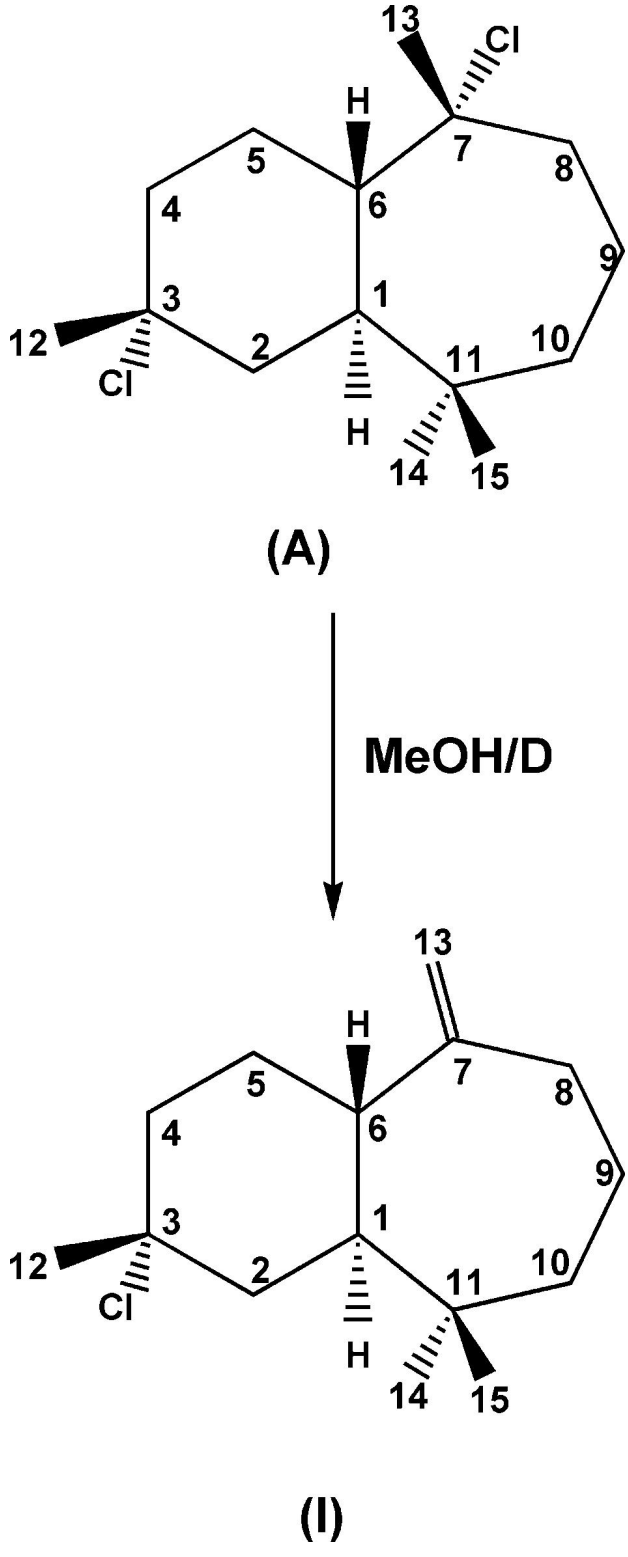
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Fig. 2