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(1*S**,3*R**,5*S**,7*S**)-4,4,8,8-Tetrachloro-1-isopropyl-5-methyltricyclo[5.1.0.0^{3,5}]-octane

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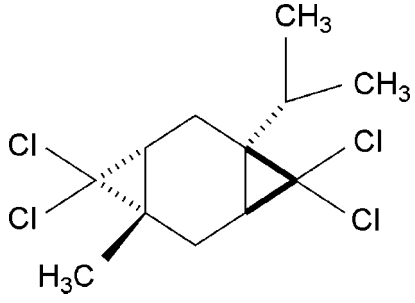
Key indicators: single-crystal X-ray study; T = 150 K; mean σ(C–C) = 0.002 Å; R factor = 0.023; wR factor = 0.054; data-to-parameter ratio = 21.1.

The title compound, $C_{12}H_{16}Cl_4$, is a derivative of the natural product 1-isopropyl-4-methylcyclohexa-1,4-diene, and represents a diastereomer with two *trans*-fused cyclopropane rings. Both enantiomers are present in the non-centrosymmetric polar space group $Pna2_1$. The central cyclohexane ring is planar within 0.02 (1) Å. The C atoms of dichloromethylene groups deviate from this plane by 1.19 (1) and −1.26 (1) Å, whereas the isopropyl and methyl groups are oriented more equatorially, deviating by 0.71 (1) and −0.87 (1) Å, respectively.

Related literature

For the isolation of 1-isopropyl-4-methylcyclohexa-1,4-diene, see: Jamali *et al.* (2013). For the crystal structure of a related compound, see: Lynch *et al.* (1994).



Experimental

Crystal data

$C_{12}H_{16}Cl_4$
M_r = 302.05
Orthorhombic, $Pna2_1$
a = 10.9480 (3) Å
b = 11.8207 (3) Å
c = 10.5027 (4) Å
V = 1359.19 (7) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 0.84 mm^{-1}
T = 150 K
0.30 × 0.26 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.786, T_{max} = 0.988
9762 measured reflections
3130 independent reflections
2973 reflections with $I > 2\sigma(I)$
R_{int} = 0.021

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.054
S = 1.05
3130 reflections
148 parameters
1 restraint
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.23 e Å^{-3}
$\Delta\rho_{min}$ = −0.16 e Å^{-3}
Absolute structure: Flack (1983), 1313 Friedel pairs
Absolute structure parameter: 0.09 (4)

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008).

The authors thank Professor Yurii V. Gatilov (Institute of Organic Chemistry, Novosibirsk, Russia) for the data collection.

Supporting information for this paper is available from the IUCr electronic archives (Reference: LD2121).

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supporting information

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(1*S**,3*R**,5*S**,7*S**)-4,4,8,8-Tetrachloro-1-isopropyl-5-methyltricyclo-[5.1.0.0^{3,5}]octane

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S1. Comment

The molecule of the title compound, (I) (Fig.1) is an enantiomeric pair of diastereomers. The relative configuration at positions 1, 3, 5 and 7 was established as S, R, S, and S, respectively. The cyclohexane ring is approximately planar, the maximum deviation from the mean plane being 0.02 (1) Å. The atoms C4 and C8 of cyclopentane rings deviate from this plane on 1.19 (1) and -1.26 (1) Å, atom C9 of isopropyl and atom C12 of methyl groups deviate on 0.71 (1) and -0.87 (1) Å, respectively.

S2. Experimental

The title compound was synthesized by interaction of /g-terpinene 1-isopropyl-4-methylcyclohexa-1,4-diene, which was isolated from the essential oil of above aerial part of *Juniperus sabina L*., with NaOH in $CHCl_3$ in presence of triethyl-benzylammonium chloride with yield 72% and with melting point 80–84°C.

S3. Refinement

H atoms were positioned geometrically and refined using a riding and rotating model, with C—H = 0.98–1.0 Å and with U_{iso}(H) = 1.2 (1.5 for methyl groups) times the U_{eq}(C). The absolute configurations of the crystal was established by refinement of the Flack parameter.

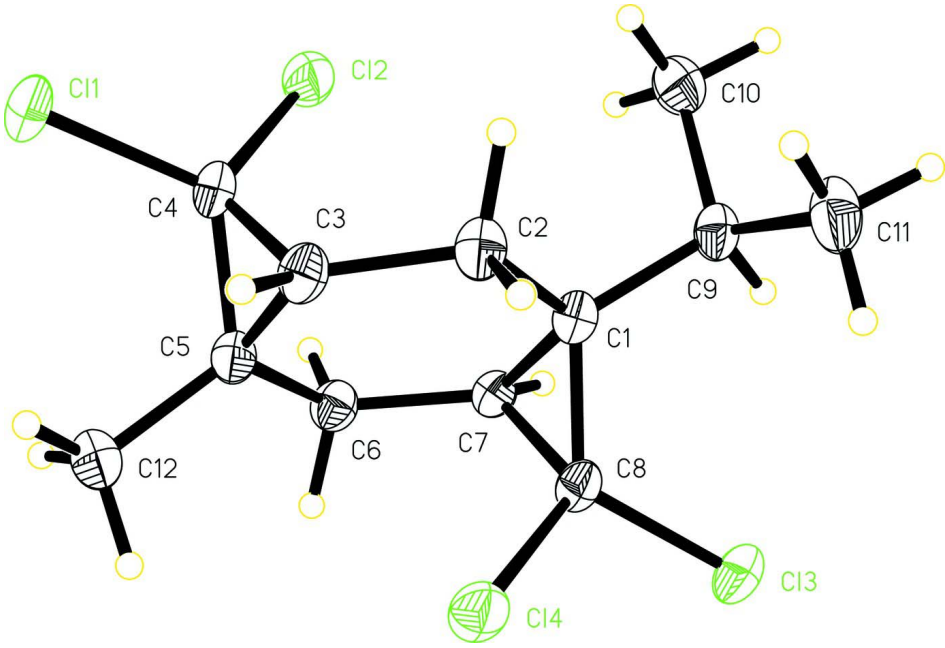

Figure 1

The molecular structure of (I), with atom labels and 50% probability displacement ellipsoids for non-H atoms.

(1*S,3*R**,5*S**,7*S**)-4,4,8,8-Tetrachloro-1-isopropyl-5-methyltricyclo[5.1.0.0^{3,5}]octane**

Crystal data

$C_{12}H_{16}Cl_4$
M_r = 302.05
Orthorhombic, $Pna2_1$
Hall symbol: P 2c -2n
a = 10.9480 (3) Å
b = 11.8207 (3) Å
c = 10.5027 (4) Å
V = 1359.19 (7) Å^3
Z = 4
F(000) = 624
D_x = 1.476 Mg m^{-3}
Melting point = 357–353 K
Mo $K\alpha$ radiation, λ = 0.71073 Å
Cell parameters from 5371 reflections
θ = 2.5–29.2°
μ = 0.84 mm^{-1}
T = 150 K
Irregular, colourless
0.30 × 0.26 × 0.02 mm

Data collection

Bruker APEXII CCD diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 7.11 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2008)
T_{min} = 0.786, T_{max} = 0.988
9762 measured reflections
3130 independent reflections
2973 reflections with $I > 2\sigma(I)$
R_{int} = 0.021
θ_{max} = 29.7°, θ_{min} = 2.6°
h = −13→13
k = −13→15
l = −14→13

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.023
$wR(F^2)$ = 0.054
S = 1.05
3130 reflections
148 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

$w = 1/[\sigma^2(F_o^2) + (0.0259P)^2 + 0.1704P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.23$ e Å^{-3}
$\Delta\rho_{min} = -0.16$ e Å^{-3}
Absolute structure: Flack (1983), 1313 Friedel pairs
Absolute structure parameter: 0.09 (4)

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

	x	*y*	*z*	U_{iso}*/U_{eq}
Cl1	0.57150 (4)	−0.08034 (4)	1.00600 (5)	0.03549 (12)
Cl2	0.38725 (4)	0.05915 (4)	1.12573 (5)	0.03067 (11)
Cl3	−0.09617 (3)	0.02606 (4)	0.84836 (4)	0.02866 (11)
Cl4	0.10624 (4)	−0.10256 (4)	0.74584 (4)	0.02984 (11)
C1	0.15053 (13)	0.08350 (13)	0.90807 (15)	0.0188 (3)
C2	0.27707 (13)	0.08167 (13)	0.84709 (17)	0.0232 (3)
H2A	0.2665	0.0869	0.7537	0.028*
H2B	0.3213	0.1504	0.8748	0.028*
C3	0.35723 (14)	−0.01975 (14)	0.87507 (16)	0.0228 (3)
H3	0.4093	−0.0434	0.8015	0.027*
C4	0.41698 (13)	−0.03625 (14)	1.00155 (18)	0.0240 (3)
C5	0.31894 (14)	−0.11786 (14)	0.96113 (17)	0.0223 (3)
C6	0.19710 (14)	−0.11148 (14)	1.03019 (16)	0.0223 (3)
H6A	0.1526	−0.1831	1.0148	0.027*
H6B	0.2136	−0.1068	1.1227	0.027*
C7	0.11359 (13)	−0.01439 (13)	0.99450 (16)	0.0186 (3)
H7	0.0555	0.0077	1.0636	0.022*
C8	0.06109 (13)	−0.00524 (14)	0.86426 (16)	0.0204 (3)
C9	0.10022 (14)	0.20144 (15)	0.93809 (17)	0.0230 (4)
H9	0.0144	0.1915	0.9687	0.028*
C10	0.17182 (18)	0.25947 (16)	1.04511 (19)	0.0333 (4)
H10A	0.2567	0.2704	1.0183	0.050*
H10B	0.1698	0.2120	1.1216	0.050*
H10C	0.1348	0.3331	1.0639	0.050*
C11	0.09512 (18)	0.27444 (16)	0.8189 (2)	0.0338 (4)
H11A	0.0558	0.3467	0.8390	0.051*
H11B	0.0481	0.2353	0.7529	0.051*
H11C	0.1782	0.2883	0.7880	0.051*
C12	0.35564 (17)	−0.23654 (15)	0.92405 (19)	0.0303 (4)
H12A	0.4327	−0.2342	0.8766	0.045*
H12B	0.2918	−0.2700	0.8706	0.045*
H12C	0.3662	−0.2824	1.0010	0.045*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cl1	0.01859 (18)	0.0338 (2)	0.0541 (3)	0.00717 (17)	−0.0056 (2)	0.0017 (2)

Cl2	0.0291 (2)	0.0298 (2)	0.0332 (2)	0.00478 (17)	−0.00942 (19)	−0.00751 (19)
Cl3	0.01775 (18)	0.0357 (3)	0.0325 (2)	0.00250 (16)	−0.00432 (19)	0.00241 (19)
Cl4	0.0297 (2)	0.0359 (3)	0.0239 (2)	0.00577 (17)	−0.00508 (17)	−0.00837 (18)
C1	0.0177 (7)	0.0225 (8)	0.0161 (8)	0.0025 (6)	0.0012 (6)	0.0021 (6)
C2	0.0202 (8)	0.0252 (8)	0.0241 (9)	0.0021 (6)	0.0043 (7)	0.0065 (7)
C3	0.0183 (7)	0.0276 (9)	0.0227 (9)	0.0034 (6)	0.0030 (6)	0.0003 (7)
C4	0.0166 (7)	0.0250 (9)	0.0304 (10)	0.0056 (6)	−0.0019 (7)	−0.0004 (8)
C5	0.0203 (7)	0.0220 (8)	0.0247 (8)	0.0039 (6)	−0.0024 (6)	−0.0004 (7)
C6	0.0209 (7)	0.0241 (9)	0.0220 (8)	0.0006 (6)	−0.0001 (6)	0.0028 (7)
C7	0.0171 (7)	0.0214 (8)	0.0173 (8)	0.0004 (5)	0.0001 (6)	0.0008 (6)
C8	0.0166 (7)	0.0249 (8)	0.0198 (9)	0.0030 (6)	−0.0007 (6)	−0.0003 (7)
C9	0.0197 (7)	0.0236 (9)	0.0256 (9)	0.0049 (7)	0.0014 (6)	0.0022 (7)
C10	0.0332 (10)	0.0255 (9)	0.0411 (12)	0.0068 (8)	−0.0033 (8)	−0.0080 (8)
C11	0.0317 (10)	0.0299 (10)	0.0399 (12)	0.0072 (8)	0.0031 (7)	0.0124 (9)
C12	0.0295 (9)	0.0254 (10)	0.0359 (11)	0.0061 (7)	−0.0030 (8)	−0.0046 (8)

Geometric parameters (Å, º)

Cl1—C4	1.7707 (15)	C6—C7	1.514 (2)
Cl2—C4	1.7547 (19)	C6—H6A	0.9900
Cl3—C8	1.7689 (14)	C6—H6B	0.9900
Cl4—C8	1.7648 (17)	C7—C8	1.488 (2)
C1—C8	1.507 (2)	C7—H7	1.0000
C1—C7	1.525 (2)	C9—C11	1.522 (2)
C1—C2	1.526 (2)	C9—C10	1.532 (3)
C1—C9	1.532 (2)	C9—H9	1.0000
C2—C3	1.515 (2)	C10—H10A	0.9800
C2—H2A	0.9900	C10—H10B	0.9800
C2—H2B	0.9900	C10—H10C	0.9800
C3—C4	1.493 (2)	C11—H11A	0.9800
C3—C5	1.529 (2)	C11—H11B	0.9800
C3—H3	1.0000	C11—H11C	0.9800
C4—C5	1.504 (2)	C12—H12A	0.9800
C5—C12	1.510 (2)	C12—H12B	0.9800
C5—C6	1.520 (2)	C12—H12C	0.9800
C8—C1—C7	58.76 (10)	C8—C7—C6	121.06 (14)
C8—C1—C2	116.86 (14)	C8—C7—C1	60.00 (11)
C7—C1—C2	118.66 (13)	C6—C7—C1	124.20 (13)
C8—C1—C9	117.56 (13)	C8—C7—H7	113.7
C7—C1—C9	118.21 (13)	C6—C7—H7	113.7
C2—C1—C9	115.19 (13)	C1—C7—H7	113.7
C3—C2—C1	117.11 (13)	C7—C8—C1	61.24 (11)
C3—C2—H2A	108.0	C7—C8—Cl4	119.49 (11)
C1—C2—H2A	108.0	C1—C8—Cl4	119.14 (11)
C3—C2—H2B	108.0	C7—C8—Cl3	118.56 (11)
C1—C2—H2B	108.0	C1—C8—Cl3	121.04 (11)
H2A—C2—H2B	107.3	Cl4—C8—Cl3	110.02 (9)

C4—C3—C2	121.99 (14)	C11—C9—C1	111.10 (14)
C4—C3—C5	59.69 (11)	C11—C9—C10	111.62 (16)
C2—C3—C5	123.80 (13)	C1—C9—C10	111.99 (13)
C4—C3—H3	113.7	C11—C9—H9	107.3
C2—C3—H3	113.7	C1—C9—H9	107.3
C5—C3—H3	113.7	C10—C9—H9	107.3
C3—C4—C5	61.33 (11)	C9—C10—H10A	109.5
C3—C4—Cl2	119.73 (11)	C9—C10—H10B	109.5
C5—C4—Cl2	119.31 (12)	H10A—C10—H10B	109.5
C3—C4—Cl1	118.71 (12)	C9—C10—H10C	109.5
C5—C4—Cl1	120.02 (12)	H10A—C10—H10C	109.5
Cl2—C4—Cl1	110.29 (9)	H10B—C10—H10C	109.5
C4—C5—C12	118.59 (14)	C9—C11—H11A	109.5
C4—C5—C6	117.36 (14)	C9—C11—H11B	109.5
C12—C5—C6	113.74 (14)	H11A—C11—H11B	109.5
C4—C5—C3	58.98 (11)	C9—C11—H11C	109.5
C12—C5—C3	118.63 (15)	H11A—C11—H11C	109.5
C6—C5—C3	119.01 (13)	H11B—C11—H11C	109.5
C7—C6—C5	116.70 (14)	C5—C12—H12A	109.5
C7—C6—H6A	108.1	C5—C12—H12B	109.5
C5—C6—H6A	108.1	H12A—C12—H12B	109.5
C7—C6—H6B	108.1	C5—C12—H12C	109.5
C5—C6—H6B	108.1	H12A—C12—H12C	109.5
H6A—C6—H6B	107.3	H12B—C12—H12C	109.5

C8—C1—C2—C3	−66.2 (2)	C5—C6—C7—C8	64.6 (2)
C7—C1—C2—C3	1.2 (2)	C5—C6—C7—C1	−8.1 (2)
C9—C1—C2—C3	149.62 (15)	C2—C1—C7—C8	−105.70 (16)
C1—C2—C3—C4	−73.4 (2)	C9—C1—C7—C8	106.78 (15)
C1—C2—C3—C5	−0.8 (2)	C8—C1—C7—C6	109.15 (17)
C2—C3—C4—C5	113.23 (16)	C2—C1—C7—C6	3.4 (2)
C2—C3—C4—Cl2	3.9 (2)	C9—C1—C7—C6	−144.07 (16)
C5—C3—C4—Cl2	−109.28 (14)	C6—C7—C8—C1	−114.21 (16)
C2—C3—C4—Cl1	−136.23 (13)	C6—C7—C8—Cl4	−5.1 (2)
C5—C3—C4—Cl1	110.53 (15)	C1—C7—C8—Cl4	109.13 (13)
C3—C4—C5—C12	108.00 (18)	C6—C7—C8—Cl3	134.01 (13)
Cl2—C4—C5—C12	−142.04 (15)	C1—C7—C8—Cl3	−111.78 (14)
Cl1—C4—C5—C12	−0.4 (2)	C2—C1—C8—C7	108.76 (15)
C3—C4—C5—C6	−109.03 (16)	C9—C1—C8—C7	−107.88 (16)
Cl2—C4—C5—C6	0.9 (2)	C7—C1—C8—Cl4	−109.69 (14)
Cl1—C4—C5—C6	142.53 (14)	C2—C1—C8—Cl4	−0.9 (2)
Cl2—C4—C5—C3	109.95 (14)	C9—C1—C8—Cl4	142.43 (13)
Cl1—C4—C5—C3	−108.44 (15)	C7—C1—C8—Cl3	107.83 (14)
C2—C3—C5—C4	−110.31 (18)	C2—C1—C8—Cl3	−143.41 (13)
C4—C3—C5—C12	−107.94 (17)	C9—C1—C8—Cl3	0.0 (2)
C2—C3—C5—C12	141.75 (17)	C8—C1—C9—C11	−86.75 (17)
C4—C3—C5—C6	106.25 (17)	C7—C1—C9—C11	−154.17 (14)
C2—C3—C5—C6	−4.1 (2)	C2—C1—C9—C11	57.21 (19)

C4—C5—C6—C7	76.0 (2)	C8—C1—C9—C10	147.68 (16)
C12—C5—C6—C7	−139.28 (15)	C7—C1—C9—C10	80.26 (18)
C3—C5—C6—C7	8.1 (2)	C2—C1—C9—C10	−68.36 (19)