IUCrData

ISSN 2414-3146

rac,*endo*-9-Hydroxy-9-methyltricyclo[5.2.2.0^{2,6}]-undeca-4,10-dien-8-one

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Received 24 August 2026
Accepted 25 August 2026

Edited by M. Bolte, Goethe-Universität Frankfurt, Germany

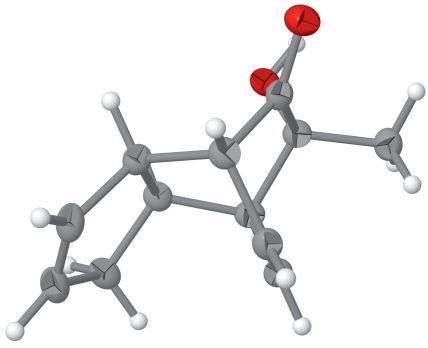
Keywords: crystal structure; hydrogen bridges; ketol.

CCDC reference: 2583168

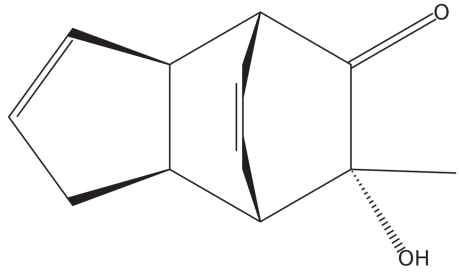
Structural data: full structural data are available from iucrdata.iucr.org

The tricyclic molecule of the title compound, $C_{12}H_{14}O_2$, is composed of four nearly planar subunits. Hydrogen bonds connect the ketol units to chains along the *a*-axis direction. The structure is further consolidated by two C—H···O hydrogen bonds, one to the carbonyl and one to the hydroxy group.

3D view



Chemical scheme



Structure description

The title compound, $C_{12}H_{14}O_2$ (Fig. 1), was prepared in a project on tricyclic frameworks (Detert & Schollmeyer, 2020; Schollmeyer & Detert, 2025, 2026). The synthesis was performed in a Diels–Alder cycloaddition of *o*-quinolacetate and cyclopentadiene followed by hydrolysis. The tricyclic molecule is composed of a [2.2.2] propellane with an annulated cyclopentene. The nearly C_3-symmetrical carbon framework of the propellane is composed of three C_2 bridges, these are essentially coplanar with the bridgehead carbons C2, C5. Subunit C2–C1–C6–C5 is planar within 0.0284 (9) Å and subtends a dihedral angle of 58.54 (14)° with the unit C2–C10–C11–C5. The latter is planar within 0.006 (2) Å and the dihedral angle to the planar unit C2–C3–C4–C5 (max. deviation: 0.0032 Å) amounts to 58.09 (15)°. The third angle is slightly larger, 63.38 (14)°. The *endo*-configuration is proven by the dihedral angle between the planar cyclopentene [max. deviation 0.010 (2) Å] and the vinylene bridge C2–C10–C11–C5 of 63.14 (14)°. Four molecules fill the unit cell: they are symmetrically connected *via* a twofold screw axis parallel to the *a*-axis and physically *via* hydrogen bonds. The O1—H1*O*···O2 hydrogen bonds (Table 1, Fig. 2) connect the molecules into chains along the *a*-axis direction, and additional C—H···O interactions link the chains into ribbons.

Synthesis and crystallization

The synthesis was performed *via* Diels–Alder reaction of 1.10 g (6.62 mmol) 2-methyl-*o*-quinolacetate (Wessely & Sinwel, 1950) in 10 ml xylenes by dropwise addition of 0.46 g

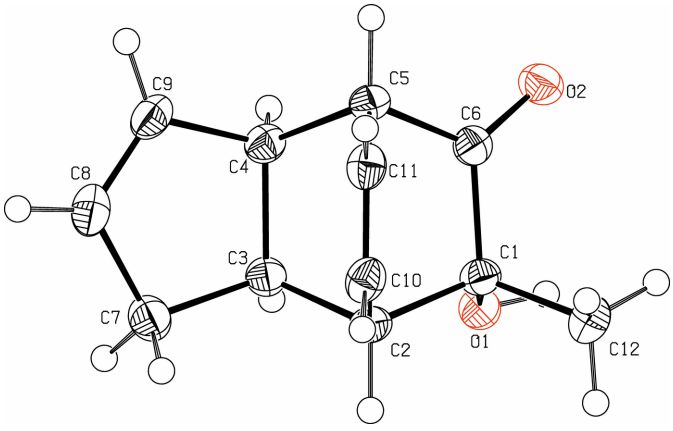


Figure 1
View of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

(1.05 eq) cyclopentadiene in 1.5 ml xylenes. The mixture was refluxed for 18 h while the color changed from yellow to orange and crystals separated. The solvent was distilled off, the residue was added to a mixture of 10 ml 1 *M* sodium hydroxide in water and 1 ml of methanol. After 16 h, the mixture was heated to 333 K until a brown compound separated. The mixture was acidulated, the precipitate was again dissolved in sodium hydroxide/methanol and reprecipitated by addition of hydrochloric acid. Single crystals were grown by slow evaporation of a solution of 60 mg of the precipitate in ether. After filtration through cotton, petroleum ether was added until the solution became turbid and again washed with ether. The now clear solution slowly evaporated and single crystals were collected mechanically from the partially coloured mixture. Yield 22 mg of colourless crystals with m.p. 399-402 K. Literature: 68% yield, m.p. 399-401 K. (Metlesics *et al.*, 1958). IR (ATR): ñ (cm^{-1}): 3403 *s*, 3048*w*, 2983*w*, 2965*w*, 2935*w*, 2918*w*, 2895*w*, 2876*m*, 2845*w*, 1717*vs*, 1619*w*, 1441, 1386*m*, 1364*m*, 1355*m*, 1301*s*, 1264*vw*, 1234*w*, 1200*w*, 1151*s*, 1138*s*, 1112*s*, 1090*m*, 1050, 1014*s*, 975*m*, 948*m*, 925*w*, 892*s*, 853*m*, 790*m*, 778*m*, 750*s*, 731*m*, 708*vs*, 675*s*, 650*w*, 582*vs*, 499*s*; ^{1}H-NMR (400 MHz, $CDCl_3$): 6.37-6.24 (*m*, 1H, H8), 6.13-6.01 (*m*, 1H, H-11), 5.70 (*dq*, ^3J = 5.7 Hz, ^4J = 2.2 Hz, 1H, H-4), 5.44 (*ddd*, ^3J = 5.8 Hz, ^3J = 3.2 Hz, ^4J = 1.5 Hz, 1H, H-5), 3.30-3.15 (*m*, 3H, H-2,-6,-7,), 3.00 (*dt*, ^3J = 6.7 Hz, ^4J = 2.2 Hz, 1H, H-1), 2.63-2.50 (*m*, 2H), 2.06-1.93 (*m*, 1H), 1.28 (*s*, 3H, CH_3). Annotation of NMR signals follows IUPAC nomenclature.

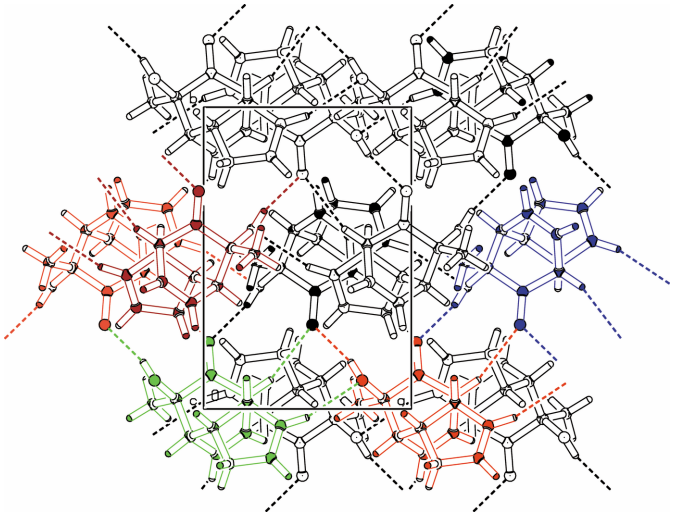


Figure 2
Part of the packing diagram. Hydrogen bonds are shown as dashed lines. View along the *c*-axis direction. Hydrogen atoms not involved in hydrogen bonds are omitted for clarity. Symmetry-related molecules are shown in different colors.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O2^{i}	0.85 (3)	1.99 (3)	2.809 (2)	162 (3)
C5—H5···O2^{ii}	1.00	2.52	3.407 (3)	148
C9—H9···O1^{iii}	0.95	2.55	3.450 (3)	159

Symmetry codes: (i) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (ii) $x+\frac{1}{2}, -y+\frac{1}{2}, -z+1$; (iii) $x+1, y, z$.

Table 2
Experimental details.

Crystal data	
Chemical formula	$C_{12}H_{14}O_2$
M_r	190.23
Crystal system, space group	Orthorhombic, $P2_12_12_1$
Temperature (K)	120
a, *b*, *c* (Å)	7.3829 (3), 10.7318 (3), 12.2961 (4)
V (Å^3)	974.24 (6)
Z	4
Radiation type	Cu *Kα*
μ (mm^{-1})	0.70
Crystal size (mm)	0.56 × 0.25 × 0.24
Data collection	
Diffractometer	Stoe Stadivari
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	5863, 1798, 1754
R_{int}	0.019
$(\sin\theta/\lambda)_{max}$ (Å^{-1})	0.607
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, *S*	0.033, 0.091, 1.12
No. of reflections	1798
No. of parameters	134
H-atom treatment	H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	0.30, −0.14
Absolute structure	Refined as an inversion twin
Absolute structure parameter	0.5 (3)

Computer programs: *X-AREA WinXpose*, *X-AREA Recipe* and *X-AREA Integrate* (Stoe & Cie, 2020), *SHELXT2014* (Sheldrick, 2015*a*), *SHELXL2019/2* (Sheldrick, 2015*b*) and *PLATON* (Spek, 2009).

Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. The structure was refined as a racemic twin.

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full crystallographic data

IUCrData (2026). **11**, x260882 [https://doi.org/10.1107/S2414314626008825]

rac,endo-9-Hydroxy-9-methyltricyclo[5.2.2.0^{2,6}]undeca-4,10-dien-8-one

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rac,endo-9-Hydroxy-9-methyltricyclo[5.2.2.0^{2,6}]undeca-4,10-dien-8-one

Crystal data

$C_{12}H_{14}O_2$
M_r = 190.23
Orthorhombic, $P2_12_12_1$
a = 7.3829 (3) Å
b = 10.7318 (3) Å
c = 12.2961 (4) Å
V = 974.24 (6) Å^3
Z = 4
F(000) = 408
D_x = 1.297 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 11043 reflections
θ = 3.6–69.9°
μ = 0.70 mm^{-1}
T = 120 K
Block, colorless
0.56 × 0.25 × 0.24 mm

Data collection

Stoe Stadivari diffractometer
Radiation source: microfocus tube
Detector resolution: 13.33 pixels mm^{-1}
rotation method, ω scans
5863 measured reflections
1798 independent reflections
1754 reflections with $I > 2\sigma(I)$
R_{int} = 0.019
θ_{max} = 69.5°, θ_{min} = 5.5°
h = −8→8
k = −13→11
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.091
S = 1.12
1798 reflections
134 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: mixed
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0498P)^2 + 0.2628P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max}$ = 0.30 e Å^{-3}
$\Delta\rho_{min}$ = −0.14 e Å^{-3}
Extinction correction: SHELXL2019/2 (Sheldrick, 2015b), $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.027 (2)
Absolute structure: Refined as an inversion twin
Absolute structure parameter: 0.5 (3)

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.

Refinement. Refined as a 2-component inversion twin. The hydroxyl H atom was freely refined. Hydrogen atoms attached to carbons were placed at calculated positions and were refined in the riding-model approximation with $C_{tertiary}$–H = 1.00 Å, $C_{secondary}$–H = 0.99 Å, C_{methyl}–H = 0.98 Å, C_{sp^2}–H = 0.95 Å, and with U_{iso}(H) = 1.5 U_{eq}(C_{methyl}) or with U_{iso}(H) = 1.2 U_{eq}(C) for the remaining H atoms. The methyl group was allowed to rotate but not to tip.

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

	x	*y*	*z*	U_{iso}*/U_{eq}
O1	0.2635 (2)	0.40578 (15)	0.39744 (12)	0.0250 (4)
H1O	0.210 (4)	0.346 (3)	0.429 (2)	0.044 (9)*
O2	0.5241 (2)	0.27907 (14)	0.52851 (13)	0.0278 (4)
C1	0.3673 (3)	0.46715 (19)	0.47879 (17)	0.0224 (5)
C2	0.4350 (3)	0.5909 (2)	0.43046 (17)	0.0240 (5)
H2	0.331230	0.647713	0.414050	0.029*
C3	0.5447 (3)	0.56246 (19)	0.32649 (17)	0.0232 (5)
H3	0.466378	0.516511	0.273651	0.028*
C4	0.7112 (3)	0.4804 (2)	0.35644 (16)	0.0230 (5)
H4	0.706449	0.399843	0.315708	0.028*
C5	0.7129 (3)	0.45495 (18)	0.48239 (16)	0.0227 (5)
H5	0.820256	0.404831	0.505697	0.027*
C6	0.5363 (3)	0.38798 (19)	0.50245 (15)	0.0217 (5)
C7	0.6217 (3)	0.6810 (2)	0.27121 (19)	0.0291 (5)
H7A	0.578751	0.687741	0.195158	0.035*
H7B	0.585296	0.756903	0.311409	0.035*
C8	0.8233 (3)	0.6633 (2)	0.27516 (18)	0.0294 (5)
H8	0.907499	0.722808	0.248544	0.035*
C9	0.8712 (3)	0.5560 (2)	0.31960 (18)	0.0280 (5)
H9	0.993364	0.529570	0.327363	0.034*
C10	0.5610 (3)	0.64894 (19)	0.51272 (18)	0.0252 (5)
H10	0.540961	0.729378	0.542666	0.030*
C11	0.7019 (3)	0.5787 (2)	0.53996 (17)	0.0248 (5)
H11	0.789473	0.604630	0.591891	0.030*
C12	0.2556 (3)	0.4843 (2)	0.58199 (18)	0.0295 (5)
H12A	0.144834	0.530609	0.564830	0.044*
H12B	0.326631	0.530835	0.635710	0.044*
H12C	0.223618	0.402526	0.611783	0.044*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
O1	0.0209 (8)	0.0261 (8)	0.0279 (8)	−0.0035 (6)	−0.0034 (6)	0.0023 (6)
O2	0.0274 (8)	0.0234 (8)	0.0325 (8)	0.0008 (6)	−0.0017 (7)	0.0033 (6)
C1	0.0192 (10)	0.0224 (10)	0.0255 (10)	0.0013 (8)	−0.0007 (8)	0.0000 (8)
C2	0.0202 (10)	0.0216 (10)	0.0302 (11)	0.0021 (8)	−0.0001 (8)	−0.0002 (9)
C3	0.0201 (10)	0.0262 (11)	0.0231 (10)	−0.0008 (8)	−0.0030 (8)	0.0018 (8)
C4	0.0217 (10)	0.0251 (10)	0.0223 (10)	0.0015 (9)	0.0004 (8)	−0.0020 (8)
C5	0.0183 (10)	0.0246 (10)	0.0252 (10)	0.0019 (8)	−0.0016 (8)	0.0013 (8)
C6	0.0224 (10)	0.0252 (11)	0.0174 (9)	0.0005 (9)	−0.0010 (8)	−0.0010 (8)

C7	0.0291 (12)	0.0288 (11)	0.0294 (11)	−0.0026 (9)	−0.0015 (9)	0.0048 (9)
C8	0.0268 (12)	0.0379 (13)	0.0236 (10)	−0.0081 (10)	0.0019 (9)	0.0009 (9)
C9	0.0213 (11)	0.0389 (13)	0.0237 (10)	0.0015 (9)	0.0037 (9)	−0.0026 (9)
C10	0.0250 (10)	0.0242 (10)	0.0265 (11)	−0.0030 (9)	0.0042 (8)	−0.0037 (8)
C11	0.0241 (10)	0.0279 (11)	0.0223 (10)	−0.0054 (8)	0.0000 (8)	−0.0014 (8)
C12	0.0260 (11)	0.0326 (12)	0.0301 (11)	−0.0018 (9)	0.0058 (9)	−0.0019 (9)

Geometric parameters (Å, º)

O1—C1	1.422 (3)	C5—C11	1.508 (3)
O1—H1O	0.85 (3)	C5—C6	1.509 (3)
O2—C6	1.215 (3)	C5—H5	1.0000
C1—C12	1.524 (3)	C7—C8	1.501 (3)
C1—C6	1.538 (3)	C7—H7A	0.9900
C1—C2	1.538 (3)	C7—H7B	0.9900
C2—C10	1.509 (3)	C8—C9	1.323 (3)
C2—C3	1.544 (3)	C8—H8	0.9500
C2—H2	1.0000	C9—H9	0.9500
C3—C7	1.550 (3)	C10—C11	1.328 (3)
C3—C4	1.556 (3)	C10—H10	0.9500
C3—H3	1.0000	C11—H11	0.9500
C4—C9	1.503 (3)	C12—H12A	0.9800
C4—C5	1.573 (3)	C12—H12B	0.9800
C4—H4	1.0000	C12—H12C	0.9800
C1—O1—H1O	106 (2)	C6—C5—H5	112.5
O1—C1—C12	110.50 (17)	C4—C5—H5	112.5
O1—C1—C6	108.33 (16)	O2—C6—C5	124.45 (19)
C12—C1—C6	110.38 (17)	O2—C6—C1	121.39 (19)
O1—C1—C2	107.65 (16)	C5—C6—C1	114.02 (16)
C12—C1—C2	113.15 (17)	C8—C7—C3	104.18 (18)
C6—C1—C2	106.63 (16)	C8—C7—H7A	110.9
C10—C2—C1	107.33 (17)	C3—C7—H7A	110.9
C10—C2—C3	108.26 (18)	C8—C7—H7B	110.9
C1—C2—C3	108.67 (17)	C3—C7—H7B	110.9
C10—C2—H2	110.8	H7A—C7—H7B	108.9
C1—C2—H2	110.8	C9—C8—C7	112.9 (2)
C3—C2—H2	110.8	C9—C8—H8	123.5
C2—C3—C7	113.16 (18)	C7—C8—H8	123.5
C2—C3—C4	109.28 (17)	C8—C9—C4	112.6 (2)
C7—C3—C4	106.19 (18)	C8—C9—H9	123.7
C2—C3—H3	109.4	C4—C9—H9	123.7
C7—C3—H3	109.4	C11—C10—C2	114.70 (19)
C4—C3—H3	109.4	C11—C10—H10	122.7
C9—C4—C3	104.15 (17)	C2—C10—H10	122.7
C9—C4—C5	112.61 (18)	C10—C11—C5	115.05 (19)
C3—C4—C5	109.73 (17)	C10—C11—H11	122.5
C9—C4—H4	110.1	C5—C11—H11	122.5

C3—C4—H4	110.1	C1—C12—H12A	109.5
C5—C4—H4	110.1	C1—C12—H12B	109.5
C11—C5—C6	107.27 (16)	H12A—C12—H12B	109.5
C11—C5—C4	107.98 (16)	C1—C12—H12C	109.5
C6—C5—C4	103.71 (16)	H12A—C12—H12C	109.5
C11—C5—H5	112.5	H12B—C12—H12C	109.5
O1—C1—C2—C10	−174.12 (16)	C11—C5—C6—C1	49.8 (2)
C12—C1—C2—C10	63.5 (2)	C4—C5—C6—C1	−64.3 (2)
C6—C1—C2—C10	−58.0 (2)	O1—C1—C6—O2	−55.1 (2)
O1—C1—C2—C3	−57.2 (2)	C12—C1—C6—O2	66.0 (2)
C12—C1—C2—C3	−179.65 (18)	C2—C1—C6—O2	−170.69 (18)
C6—C1—C2—C3	58.8 (2)	O1—C1—C6—C5	120.74 (18)
C10—C2—C3—C7	−63.2 (2)	C12—C1—C6—C5	−118.15 (19)
C1—C2—C3—C7	−179.44 (17)	C2—C1—C6—C5	5.1 (2)
C10—C2—C3—C4	54.9 (2)	C2—C3—C7—C8	118.3 (2)
C1—C2—C3—C4	−61.4 (2)	C4—C3—C7—C8	−1.6 (2)
C2—C3—C4—C9	−121.26 (19)	C3—C7—C8—C9	1.6 (3)
C7—C3—C4—C9	1.1 (2)	C7—C8—C9—C4	−1.0 (3)
C2—C3—C4—C5	−0.5 (2)	C3—C4—C9—C8	−0.1 (2)
C7—C3—C4—C5	121.89 (18)	C5—C4—C9—C8	−119.0 (2)
C9—C4—C5—C11	62.1 (2)	C1—C2—C10—C11	58.2 (2)
C3—C4—C5—C11	−53.4 (2)	C3—C2—C10—C11	−58.9 (2)
C9—C4—C5—C6	175.74 (17)	C2—C10—C11—C5	1.1 (3)
C3—C4—C5—C6	60.2 (2)	C6—C5—C11—C10	−55.4 (2)
C11—C5—C6—O2	−134.5 (2)	C4—C5—C11—C10	55.8 (2)
C4—C5—C6—O2	111.4 (2)		

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H1*O*···O2^{i}	0.85 (3)	1.99 (3)	2.809 (2)	162 (3)
C5—H5···O2^{ii}	1.00	2.52	3.407 (3)	148
C9—H9···O1^{iii}	0.95	2.55	3.450 (3)	159

Symmetry codes: (i) *x*−1/2, −*y*+1/2, −*z*+1; (ii) *x*+1/2, −*y*+1/2, −*z*+1; (iii) *x*+1, *y*, *z*.