The title racemic triquinane, \( \text{C}_{14}\text{H}_{14}\text{Cl}_{2}\text{O}_{2} \), is composed of four five-membered rings, one of which is a tetrahydrofuran ring to which an allyl group on one side and a hydroxyl group on the other side are attached. The core of the triquinane unit has a \( \text{cis-syn-cis} \) configuration. In the crystal, the molecules are linked by pairwise O—H⋯O hydrogen bonds, generating inversion dimers featuring \( \text{R}_2^2(8) \) loops.

Structure description

Compounds with three fused five-membered rings, known as triquinanes, have gained considerable importance because this core is found in several biologically active compounds (Qiu et al., 2018; Kotha et al., 2020). Therefore, convenient methods to prepare and functionalize triquinanes and the study of their stereochemistry are useful exercises (Mehta & Rao, 1985). Our group has prepared triquinanes from cage compounds in a simplified manner using microwave irradiation (Kotha et al., 2019). Thereafter, we attempted to functionalize the triquinanes and observed a transannular attack at the keto centre (O1—C1—O2) leading to the formation of the title compound, \( \mathbf{1} \).

Compound \( \mathbf{1} \) has three carbo cyclic rings (C1/C2/C3/C4/C5, C4/C5/C6/C7/C8 and C6/C7/C9/C10/C11) and a tetrahydrofuran ring (O1/C1/C5/C6/C11). The allyl group is unsymmetrically substituted at C11 and the hydroxy group is attached to C1 (Fig. 1a). There are six stereogenic centres in \( \mathbf{1} \): in the arbitrarily chosen asymmetric molecule, the configurations are \( \text{C1} \text{R}, \text{C4} \text{R}, \text{C5} \text{S}, \text{C6} \text{R}, \text{C7} \text{S} \) and \( \text{C11} \text{S} \) but crystal symmetry generates a racemic mixture.
The triquinane ring system consists of a cis–syn–cis configuration, i.e., the hydrogen atoms at the ring junction are all above the plane and the first and the third rings are below the plane (Fig. 1b). The chlorine atoms are attached to the unsaturated bonds C2—C3 and C9—C10 in anti-manner with respect to the H atoms of the ring junction. The middle cyclopentyl ring adopts an envelope conformation and the side rings are almost planar.

In the crystal, the molecules are linked by O—H···O hydrogen bonds, generating inversion dimers featuring $R_2^2(8)$ loops (Table 1, Fig. 2) but no intramolecular hydrogen bonds are present.

**Synthesis and crystallization**

The synthesis scheme is shown in Fig. 3. Indium ingots (51 mg, 2.7 eq) were cut into small pieces and transferred to a two-neck round-bottomed flask. Tetrahydrofuran (3 ml) was transferred to the flask under nitrogen at room temperature. Allyl iodide (0.5 ml) was added to this solution via a syringe. After one h, the starting material 2 (40 mg) and trimethylchlorosilane (3 drops) was added to the reaction mixture. On completion of the reaction (TLC monitoring) after 1 h, water was added to the reaction mixture. The aqueous layer was extracted with diethyl ether (Lee et al. 2001). The compound was purified with column chromatography and silica gel (100–200 mesh) was used. Ethyl acetate:petroleum ether (8% of ethyl acetate in total in 100 ml of solution) was used an eluent.

### Table 1

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|------|-------|-------|---------|
| O2—H2···O1$^i$ | 0.84  | 2.06  | 2.893 (3) | 173     |

Symmetry code: $^i$ $\bar{x}$, $\bar{y}$, $\bar{z}$. 

### Table 2

**Experimental details.**

| Crystal data | Chemical formula | $M_r$ |
|--------------|------------------|-------|
|              | C$_{14}$H$_{14}$Cl$_2$O$_2$ | 285.15 |
| Crystal system, space group | Triclinic, $P\bar{T}$ | |
| Temperature (K) | 150 | |
| $a$, $b$, $c$ (Å) | 7.2687 (10), 8.3648 (11), 11.7460 (18) | |
| $\alpha$, $\beta$, $\gamma$ (°) | 80.448 (4), 83.441 (4), 65.285 (4) | |
| $V$ (Å$^3$) | 638.96 (16) | |
| $Z$ | 2 | |
| Radiation type | Mo $K\alpha$ | |
| $\mu$ (mm$^{-1}$) | 0.50 | |
| Crystal size (mm) | 0.32 $\times$ 0.29 $\times$ 0.09 | |

**Data collection**

| Diffractometer | Bruker APEXII CCD |
|----------------|-------------------|
| Absorption correction | Multi-scan (SADABS; Bruker, 2016) |
| $T_{\text{min}}$, $T_{\text{max}}$ | 0.655, 0.746 |
| No. of measured, independent and observed [I > 2σ(I)] reflections | 19764, 2241, 1662 |
| $R_{\text{int}}$ | 0.106 |
| (sin $\theta$/λ)$_{\text{max}}$ (Å$^{-1}$) | 0.594 |

**Refinement**

| $R[F^2 > 2σ(F^2)]$, $wR(F^2)$, $S$ | 0.048, 0.107, 1.09 |
| No. of reflections | 2241 |
| No. of parameters | 164 |
| H-atom treatment | H-atom parameters constrained |
| $\Delta f_{\text{max}}$, $\Delta f_{\text{min}}$ (e Å$^{-3}$) | 0.27, −0.33 |

Computer programs: APEX2 and SAINT (Bruker, 2016), SHELXT (Sheldrick, 2015a), SHELXL (Sheldrick, 2015b) and OLEX2 (Dolomanov et al., 2009).
After that, the crystals suitable for X-ray crystallographic analysis were grown in air in a glass vial using ethyl acetate as solvent (Fig. 3).

Characterization: colourless crystalline solid; m.p. 120–122 °C; $^1$H NMR (500 MHz, CDCl$_3$): δ = 5.73–5.62 (m, 3H), 5.20–5.14 (m, 2H), 3.39–3.30 (m, 2H), 3.23–3.20 (m, 1H), 3.02–2.98 (m, 1H), 2.63 (dd, $J$ = 13.8, 7.0 Hz, 1H), 2.55 (dd, $J$ = 13.8, 7.0 Hz, 1H), 1.95–1.87 (m, 1H), 1.78 (d, $J$ = 13.9 Hz, 1H) p.p.m.; $^{13}$C NMR (125 MHz, CDCl$_3$): δ = 134.1, 133.2, 133.1, 133.0, 132.5, 119.1, 115.5, 97.7, 58.8, 54.8, 47.7, 46.4, 40.4, 35.2 p.p.m.; HRMS (ESI): m/z calculated for C$_{14}$H$_{14}$Cl$_2$NaO$_2$ [M + Na]$^+$: 307.0262; found: 307.0263.

Refinement
Crystal data, data collection and structure refinement details are summarized in Table 2.

Acknowledgements
We thank Darshan S. Mhatre for his help in collecting the X-ray data and with the structure refinement.

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Sheldrick, G. M. (2015b). Acta Cryst. C71, 3–8.
**full crystallographic data**

**IUCrData** (2021). *6*, x211260  [https://doi.org/10.1107/S2414314621012608]

rac-(2aS,2a1R,3aR,3a1S,5aS,6aR)-2a-Allyl-2,4-dichloro-2a,2a1,3a1,5a,6,6a-hexahydro-3aH-3-oxadicyclopenta[cd,gh]pentalen-3a-ol

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rac-(2aS,2a1R,3aR,3a1S,5aS,6aR)-2a-Allyl-2,4-dichloro-2a,2a1,3a1,5a,6,6a-hexahydro-3aH-3-oxadicyclopenta[cd,gh]pentalen-3a-ol

**Crystal data**

\[
\begin{align*}
\text{C}_{14}\text{H}_{14}\text{Cl}_2\text{O}_2 & \quad Z = 2 \\
M_r & = 285.15 \\
\text{Triclinic, } P\bar{1} & \quad F(000) = 296 \\
a = 7.2687 (10) \text{ Å} & \quad D_r = 1.482 \text{ Mg m}^{-3} \\
b = 8.3648 (11) \text{ Å} & \quad \text{Mo } K\alpha \text{ radiation, } \lambda = 0.71073 \text{ Å} \\
c = 11.7460 (18) \text{ Å} & \quad \text{Cell parameters from 3239 reflections} \\
\alpha = 80.448 (4)^\circ & \quad \theta = 2.7–25.0^\circ \\
\beta = 83.441 (4)^\circ & \quad \mu = 0.50 \text{ mm}^{-1} \\
\gamma = 65.285 (4)^\circ & \quad T = 150 \text{ K} \\
V = 638.96 (16) \text{ Å}^3 & \quad \text{Plate, clear light colourless} \\
\end{align*}
\]

**Data collection**

Bruker APEXII CCD
diffractometer
φ and ω scans
Absorption correction: multi-scan
(SADABS; Bruker, 2016)
\[T_{\text{min}} = 0.655, T_{\text{max}} = 0.746\]
19764 measured reflections

**Refinement**

Refinement on \(F^2\)
Least-squares matrix: full
\[R(F^2) = 0.048, \quad wR(F^2) = 0.107\]
\[S = 1.09\]
2241 reflections
164 parameters
0 restraints
Primary atom site location: dual
Hydrogen site location: inferred from neighbour sites
H-atom parameters constrained
\[w = 1/[\sigma(F_c^2) + (0.0235P)^2 + 1.1376P]\]
where \(P = (F_c^2 + 2F_c^2)/3\)
\[(\Delta/\sigma)_{\text{max}} < 0.001\]
\[\Delta\rho_{\text{max}} = 0.27 \text{ e Å}^{-3}\]
\[\Delta\rho_{\text{min}} = -0.33 \text{ e Å}^{-3}\]

**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      | $U_{iso}$ or $U_{eq}$ |
|-----|--------|--------|--------|-----------------------|
| Cl1 | 0.06076 (13) | 0.17026 (12) | 0.42861 (8) | 0.0269 (3) |
| Cl2 | −0.06759 (13) | 0.53279 (13) | 0.15245 (8) | 0.0328 (3) |
| O1  | 0.1461 (3) | 0.5253 (3) | 0.37821 (19) | 0.0191 (5) |
| O2  | 0.2783 (3) | 0.3694 (3) | 0.55085 (19) | 0.0221 (6) |
| H2  | 0.157708 | 0.391949 | 0.574798 | 0.033* |
| C1  | 0.2966 (5) | 0.3622 (4) | 0.4322 (3) | 0.0185 (8) |
| C11 | 0.2225 (5) | 0.5758 (4) | 0.2653 (3) | 0.0192 (8) |
| C3  | 0.4551 (5) | 0.0894 (4) | 0.3551 (3) | 0.0209 (8) |
| H3  | 0.474728 | −0.022512 | 0.335419 | 0.025* |
| C10 | 0.1821 (5) | 0.4890 (4) | 0.1739 (3) | 0.0194 (8) |
| C6  | 0.4571 (5) | 0.4848 (4) | 0.2673 (3) | 0.0179 (7) |
| H6  | 0.518199 | 0.572267 | 0.266037 | 0.021* |
| C2  | 0.2846 (5) | 0.1995 (4) | 0.4009 (3) | 0.0198 (8) |
| C9  | 0.3446 (5) | 0.3828 (4) | 0.1197 (3) | 0.0211 (8) |
| H9  | 0.342144 | 0.317963 | 0.061141 | 0.025* |
| C4  | 0.6154 (5) | 0.1623 (4) | 0.3377 (3) | 0.0203 (8) |
| H4  | 0.732882 | 0.086130 | 0.386498 | 0.024* |
| C7  | 0.5363 (5) | 0.3772 (4) | 0.1626 (3) | 0.0206 (8) |
| H7  | 0.604311 | 0.435233 | 0.101481 | 0.025* |
| C8  | 0.6874 (5) | 0.1927 (5) | 0.2120 (3) | 0.0240 (8) |
| H8A | 0.688215 | 0.100747 | 0.168389 | 0.029* |
| H8B | 0.826068 | 0.188696 | 0.207430 | 0.029* |
| C5  | 0.5042 (5) | 0.3485 (4) | 0.3779 (3) | 0.0195 (8) |
| H5  | 0.584893 | 0.371253 | 0.431830 | 0.023* |
| C13 | 0.1802 (5) | 0.8510 (5) | 0.1251 (3) | 0.0270 (9) |
| H13 | 0.152010 | 0.810452 | 0.061014 | 0.032* |
| C12 | 0.1281 (5) | 0.7775 (4) | 0.2438 (3) | 0.0253 (8) |
| H12A| −0.021149 | 0.820022 | 0.254500 | 0.030* |
| H12B| 0.174476 | 0.823785 | 0.301924 | 0.030* |
| C14 | 0.2611 (6) | 0.9658 (5) | 0.1035 (4) | 0.0399 (11) |
| H14A| 0.291514 | 1.009520 | 0.165285 | 0.048* |
| H14B| 0.289666 | 1.006095 | 0.025767 | 0.048* |

### Atomic displacement parameters (Å²)

|     | $U_{11}$ | $U_{22}$ | $U_{33}$ | $U_{12}$ | $U_{13}$ | $U_{23}$ |
|-----|----------|----------|----------|----------|----------|----------|
| Cl1 | 0.0202 (5) | 0.0279 (5) | 0.0360 (6) | −0.0142 (4) | −0.0005 (4) | −0.0023 (4) |
| Cl2 | 0.0145 (5) | 0.0479 (6) | 0.0353 (6) | −0.0122 (4) | −0.0051 (4) | −0.0026 (5) |
| O1  | 0.0093 (12) | 0.0173 (12) | 0.0240 (14) | 0.0000 (10) | 0.0021 (10) | −0.0013 (10) |
| O2  | 0.0154 (13) | 0.0278 (14) | 0.0207 (14) | −0.0065 (11) | 0.0014 (10) | −0.0045 (11) |
| C1  | 0.0109 (17) | 0.0171 (18) | 0.023 (2) | −0.0020 (14) | 0.0003 (14) | −0.0012 (15) |
| C11 | 0.0128 (18) | 0.0183 (18) | 0.022 (2) | −0.0031 (15) | 0.0037 (14) | −0.0039 (15) |
| C3  | 0.0184 (19) | 0.0157 (18) | 0.027 (2) | −0.0049 (15) | −0.0040 (15) | −0.0026 (15) |
| C10 | 0.0112 (18) | 0.0185 (18) | 0.028 (2) | −0.0057 (15) | −0.0039 (15) | 0.0008 (15) |
| C6  | 0.0095 (17) | 0.0166 (18) | 0.026 (2) | −0.0038 (14) | 0.0024 (14) | −0.0049 (15) |
|   |       |       |       |       |       |       |
|---|-------|-------|-------|-------|-------|-------|
| C2 | 0.0189 (19) | 0.0156 (18) | 0.024 (2) | -0.0074 (16) | -0.0053 (15) | 0.0039 (15) |
| C9 | 0.023 (2) | 0.0166 (18) | 0.022 (2) | -0.0073 (16) | -0.0035 (16) | -0.0008 (15) |
| C4 | 0.0108 (18) | 0.0173 (18) | 0.026 (2) | 0.0015 (15) | -0.0040 (14) | -0.0026 (15) |
| C7 | 0.0134 (18) | 0.0202 (18) | 0.025 (2) | -0.0049 (15) | 0.0038 (14) | -0.0021 (15) |
| C8 | 0.0125 (18) | 0.024 (2) | 0.030 (2) | -0.0022 (16) | -0.0001 (15) | -0.0045 (16) |
| C5 | 0.0116 (17) | 0.0228 (19) | 0.025 (2) | -0.0075 (15) | -0.0002 (14) | -0.0042 (15) |
| C13 | 0.026 (2) | 0.0174 (19) | 0.032 (2) | -0.0030 (16) | -0.0035 (16) | -0.0018 (16) |
| C12 | 0.019 (2) | 0.0178 (19) | 0.031 (2) | -0.0016 (16) | 0.0050 (16) | -0.0032 (16) |
| C14 | 0.056 (3) | 0.043 (3) | 0.032 (2) | -0.033 (2) | -0.002 (2) | -0.0005 (19) |

Geometric parameters (Å, °)

|   |       |       |       |       |       |       |
|---|-------|-------|-------|-------|-------|-------|
| Cl1—C2 | 1.731 (3) | Cl2—C10 | 1.734 (3) | O1—C1 | 1.444 (4) | O1—C11 | 1.445 (4) |
| Cl2—C10 | 1.734 (3) | O1—C11 | 1.445 (4) | O2—C1 | 1.394 (4) | C1—C2 | 1.506 (5) |
| O1—C1 | 1.444 (4) | C3—C4 | 1.526 (5) | C1—C5 | 1.536 (5) | C11—C10 | 1.508 (5) |
| O1—C11 | 1.445 (4) | C6—C7 | 1.552 (5) | C11—C6 | 1.550 (4) | C11—C12 | 1.520 (5) |
| O2—C1 | 1.394 (4) | C6—C5 | 1.545 (5) | C3—C2 | 1.316 (5) | C1—O1 | 110.0 (2) |
| C1—C2 | 1.506 (5) | C9—C7 | 1.515 (5) | C11—C10 | 1.508 (5) | O1—C11 | 111.4 (3) |
| C1—C5 | 1.536 (5) | C10—Cl2 | 118.3 (2) | C10—C11 | 112.9 (3) | O1—C12 | 111.4 (3) |
| C7—C8 | 1.534 (5) | C11—C12 | 113.3 (3) | O1—C11 | 111.4 (3) | O1—C11 | 111.4 (3) |
| C3—C2 | 1.316 (5) | C11—C10 | 111.3 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C5—C6 | 105.6 (5) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C6—C7 | 106.9 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C7—C8 | 110.0 (2) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C1—C2 | 112.9 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C1—C5 | 107.0 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C3—C2 | 111.8 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C5—C6 | 108.4 (2) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C6—C7 | 106.8 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C9—Cl2 | 126.5 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C9—C10 | 115.1 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |
| C11—C6 | 107.5 (3) | C10—C11 | 115.4 (3) | C2—C3 | 114.6 (3) | O1—C11 | 111.4 (3) |

Hydrogen-bond geometry (Å, °)

| D—H···A | D—H | H···A | D···A | D—H···A |
|---------|-----|------|------|---------|
|         |     |      |      |         |
| O2—H2···O1ᵢ          | 0.84 | 2.06 | 2.893 (3) | 173 |
|----------------------|------|------|-----------|-----|

Symmetry code: (i) −x, −y+1, −z+1.