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Key indicators

Single-crystal X-ray study

 $T = 293$ KMean $\sigma(\text{C}-\text{C}) = 0.007$ Å R factor = 0.060 wR factor = 0.160

Data-to-parameter ratio = 9.2

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.4-Chloro-2-[(*E*)-[(1*R*,4*aS*,10*aR*)-7-isopropyl-1,4*a*-dimethyl-1,2,3,4,4*a*,9,10,10*a*-octahydrophenanthren-1-yl]methyliminomethyl]phenol

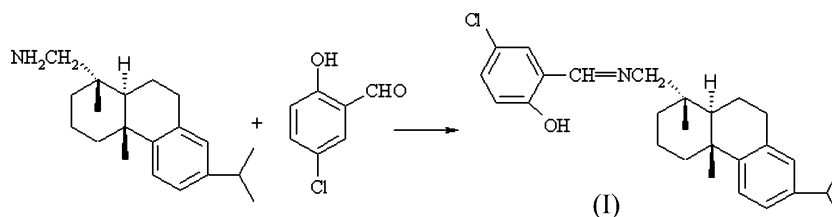
The title compound, $\text{C}_{27}\text{H}_{34}\text{ClNO}$, was synthesized by a direct condensation reaction. The molecule has three chiral centres, which exhibit *R*, *S* and *R* absolute configurations, respectively. Two six-membered rings with different conformations coexist in the structure and they form a *trans* ring junction with two methyl groups in axial positions.

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Comment

Biomass resources have attracted great interest in both industry and fundamental research (San *et al.*, 1993), not only because of the abundance and biodegradation ability of this kind of compound, but also due to their special structures that contain several chiral centres. Dehydroabietylamine is an important chiral diterpenic amine with the hydrophanthrene structure (Gottstein & Cheney, 1965). It has been widely used to resolve racemic mixtures and as a starting material for the design and synthesis of biological compounds (Wilkerson *et al.*, 1993). Pine resin acid is the only available resource of dehydroabietylamine (Buchbauer & Kolbe, 1985). Some of the derivatives of pine resin acid have been prepared during recent decades. They exhibit a special half-chair conformation as well as intermolecular hydrogen bonds (Hamodrakas *et al.*, 1978). The crystal structures of dehydroabietylamine derivatives have not been reported to date. Chiral Schiff bases can be used in the field of asymmetric synthesis (Iglesias *et al.*, 2004), so their synthesis and characterization attracted our attention. In this work, we describe the synthesis and crystal structure of the title compound, (I).



As shown in Figs. 1 and 2, (I) contains four crystallographically distinct six-membered rings. Rings *B* or *C* are not planar. Their internal torsion angles (Table 1) show that these rings exhibit a chair and a half-chair conformation, respectively (Fig. 2). They form a *trans* ring junction with two methyl groups in axial positions. However, the two rings *A* and *D* are planar; the dihedral angle between them is $74.7(16)^\circ$. The hydroxyl group, the Cl atom and ring *D* are almost coplanar. The title compound has three chiral centres; they exhibit *R*, *S* and *R* absolute configurations, respectively (Fig. 1). There is an intramolecular $\text{O}-\text{H}\cdots\text{N}$ hydrogen bond;

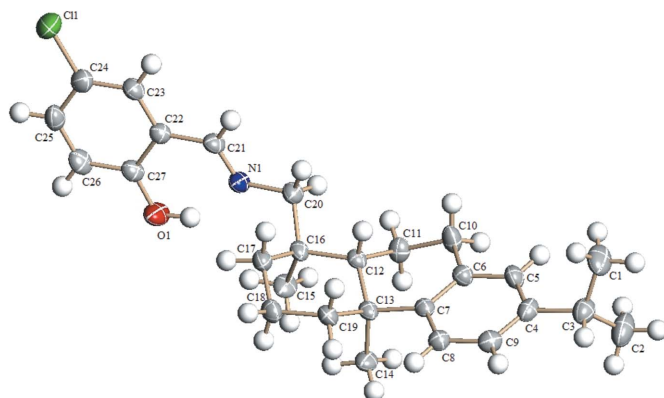


Figure 1
The molecular structure of the title compound, with H atoms represented by small spheres of arbitrary radius and displacement ellipsoids at the 30% probability level.

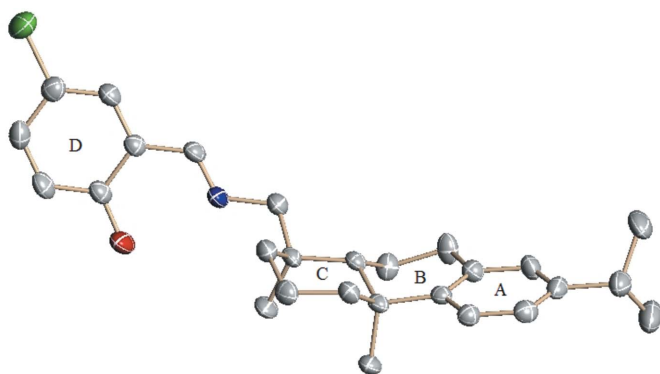


Figure 2
The ring conformations, with displacement ellipsoids at the 30% probability level (H atoms omitted for clarity).

the O—H, H···N and O···N distances and O—H···N angle are 0.91 Å, 1.80 Å and 2.576 Å, 142° respectively.

Experimental

All chemicals purchased were of reagent grade and used without further purification. A mixture of dehydroabietylamine (10 mmol, 2.85 g), 5-chlorosalicylidene (10 mmol, 1.8 g) and ethanol (200 ml) was refluxed for 3 h, then cooled to room temperature. The precipitate was collected and dried under vacuum. The product was then dissolved in ethanol and the solution was left to stand undisturbed. Upon slow evaporation at room temperature for several days, yellow single crystals of the title compound were collected (m.p. 450.8 K).

Crystal data

$C_{27}H_{34}ClNO$
 $M_r = 424.00$
Monoclinic, $P2_1$
 $a = 11.249$ (2) Å
 $b = 6.1790$ (12) Å
 $c = 17.276$ (4) Å
 $\beta = 103.04$ (3)°
 $V = 1169.8$ (4) Å³

$Z = 2$
 $D_x = 1.204$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.18$ mm⁻¹
 $T = 293$ (2) K
Block, yellow
0.40 × 0.30 × 0.10 mm

Data collection

Siemens SMART 1000
diffractometer
 φ and ω scans
Absorption correction: multi-scan
(*SADABS*; Sheldrick, 1996)
 $T_{\min} = 0.931$, $T_{\max} = 0.982$

2657 measured reflections
2526 independent reflections
1639 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.028$
 $\theta_{\text{max}} = 26.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.061$
 $wR(F^2) = 0.160$
 $S = 1.06$
2526 reflections
275 parameters
H atoms treated by a mixture of
independent and constrained
refinement

$w = 1/[\sigma^2(F_o^2) + (0.08P)^2 + 0.0497P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.025$
 $\Delta\rho_{\text{max}} = 0.19$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.24$ e Å⁻³
Absolute structure: Flack (1983)
Flack parameter: 0.07 (18)

Table 1

Selected torsion angles (°).

C10—C6—C7—C8	178.8 (5)	C16—C17—C18—C19	55.2 (6)
C10—C11—C12—C16	159.2 (4)	N—C21—C22—C23	-176.2 (5)

All H atoms were initially located in a difference Fourier map. The methyl H atoms were then constrained to an ideal geometry, with C—H distances of 0.96 Å and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$, but each group was allowed to rotate freely about its C—C bond. The position of the hydroxyl H atom was refined freely along with an isotropic displacement parameter [O—H = 0.91 (6) Å]. All other H atoms were placed in geometrically idealized positions and constrained to ride on their parent atoms, with C—H distances in the range 0.93–0.98 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXL97*; software used to prepare material for publication: *SHELXTL* (Sheldrick, 1997b).

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