Synthesis and crystal structure of 1-((3R,10S,13R,17S)-10,13-dimethyl-3-(phenylamino)hexadecahydro-1H-cyclopenta[α]phenanthen-17-yl)ethan-1-one, C\textsubscript{27}H\textsubscript{39}NO

The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

A mixture of 5α-pregnane-3,20-dione (1.6 mmol) and aniline (1.3 mmol), and one drop of HOAc in MeOH (10 mL) was stirred at room temperature for 24 h. Subsequently, sodium borohydride (3.9 mmol) was added and kept stirring. After 3 h, the reaction was terminated with sodium hydroxide and extracted with ethyl acetate. The combined organic layer was dried over anhydrous Na\textsubscript{2}SO\textsubscript{4}, filtered and evaporated, and the obtained solid was recrystallized with methanol to obtain the colorless needle crystal of the compound. 67% yield.

Experimental details

Comment

Pregnan-type steroidal alkaloids were the main chemical components of the genus Sarcococca (Buxaceae), and...
possessed a range of bioactivities (e.g., cholinesterase inhibiting, antitumor, antibacterial, anti-inflammatory) [5–9]. Our previous study resulted in the discovery of some new steroidal alkaloids from Sarcococca roacifolia and Sarcococca hookeriana which showed significant anti-tumor activity [10–13]. However, the natural alkaloids from Sarcococca have low structural diversity. In order to enriching structural diversity of steroidal alkaloids and finding efficient and low toxicity derivatives. Epiandrosterone was used as the start reagent, the allyl group at C17 compound through Wittig reaction, the hydroxyl at C20 compounds through reduction reaction, and the ketone at C3 and C20 position through Corey reaction, and a series of pregnane-type steroidal alkaloids were obtained by reductive amination of carbonyl compounds. The title compound 1-(3R,10S,13R,17S)-10,13-dimethyl-3-(phenylamino)hexadecahydro-1H-cyclopenta[a]phenanthren-17-yl)ethan-one was obtained by 5α-pregnane-3,20-dione reductive amination.

The title compound is shown in the figure. The bond lengths and angles which were derived from the title structure are within normal ranges. In the molecule, the phenylamino group is planar and the carbonyl group was confirmed by the distances d(C20—O1) = 1.21(4) Å. The compound contains three six-membered rings and one five-membered ring. The structure is consolidated by intermolecular non-classical hydrogen bonding interactions of the type C—H⋯O involving carbonyl O atoms and methyl, methylene and methylidene groups.

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References


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