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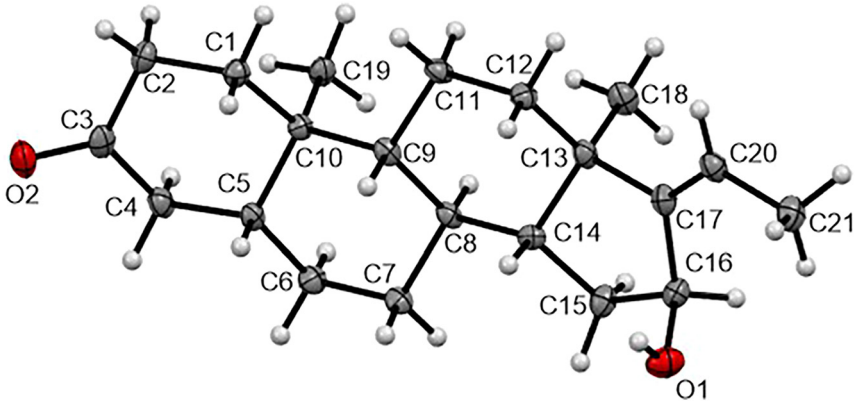
Synthesis and crystal structure of-(10*S*,13*S*,16*R*,*Z*) −17-ethylidene-16-hydroxy-10,13-dimethylhexadecahydro-3 *H*-cyclopenta[*α*] phenanthren-3-one, $C_{21}H_{32}O_2$



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Abstract

$C_{21}H_{32}O_2$, monoclinic, $P2_1$, a = 15.2215(7) Å, b = 7.2235(4) Å, c = 16.3052(7) Å, β = 92.754(4)°, V = 1790.73(15) Å^3, Z = 4, $R_{gt}(F)$ = 0.0503, $wR_{ref}(F^2)$ = 0.1345, T = 297(2) K.

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The molecular structure is shown in the figure. Table 1 contains the crystallographic data. The list of the atoms including atomic coordinates and displacement parameters can be found in the cif- file attached to this article.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size	0.23 × 0.21 × 0.18 mm
Wavelength:	Mo *k*α radiation (0.71073 Å)
μ:	0.07 mm^{-1}
Diffractometer, scan mode:	Bruker diffractometer, φ and ω scans
θ_{max}, completeness:	25.0°, 100 %
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	11,952, 4,924, 0.064
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 4,346
$N(param)_{refined}$:	423
Programs:	Olex2,[1] Bruker,[2] SHELX[3]

1 Source of material

An amount of 0.5 mmol SeO_2 was dissolved in 10 mL anhydrous CH_2Cl_2, 1 mmol t–BuOOH was added and stirred at room temperature for 0.5 h. The temperature was controlled to keep the system at 0 °C, and (10*S*,13*S*, *Z*)-17-ethylidene-10,13-dimethylhexadecahydro-3*H*-cyclopenta[*α*]phenanthren-3-one 1 mmol was dissolved in 5 mL CH_2Cl_2, by slow dropping, the system was light purple, stirring for 5h, TLC monitoring reaction was complete. Add CH_2Cl_2 and $NaHCO_3$ as a saturated solution, then combine with organic phase, dry with anhydrous Na_2SO_4 and concentrate under reduced pressure to obtain the crude yield. The raw product was purified by silica gel chromatography, the mobile phase was petroleum PE:PA, the colorless nubbly crystals were obtained. 80 % yield.

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2 Comment

Plants of the genus *Sarcococca* of Buxaceae are rich in pregnane alkaloids.[4–6] The previous research carried out systematic chemical studies on the *Sarcococca ruscifolia* and *Sarcococca hookeriana* and obtained a series of pregnane alkaloids.[7–17] In order to obtain such compounds with high efficiency and low toxicity, we used the steroid epiandrosterone as the raw material to obtain the C17 vinylidene compound by Wittig reaction, and the C3 ketone compound was obtained by PCC oxidation. The ketone compound was subjected to a Riley reaction to obtain the target compound.

The title compound consists of three methyl groups, three six-membered rings, one five-membered ring, one ketone group, one hydroxyl group and one carbon-carbon double bond, and the substituents on positions C-10, C-13 are β configurations, C-16 is α configuration, d (O1–C16) = 1.437(4) Å, d(O2–C3) = 1.225(3) Å, d(C17–C20) = 1.328(4) Å, d(C20–C21) = 1.497(4) Å. The five membered ring and three six membered rings show intermolecular interactions by van der Waals forces only.

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References

1. Dolomanov, O. V.; Bourhis, L. J.; Gildea, R. J.; Howard, J. A. K.; Puschmann, H. Olex2: a Complete Structure Solution, Refinement and Analysis Program. *J. Appl. Crystallogr.* **2009**, *42*, 339–341.
2. Bruker *Apex2, Saint and Sadabs*; Bruker AXS Inc.: Madison, Wisconsin, USA, 2012.
3. Sheldrick, G. M. Crystal Structure Refinement with *SHELXL*. *Acta Crystallogr.* **2015**, *C71*, 3–8.
4. Vohra, M. I. Antiinflammatory Steroidal Alkaloids from *Sarcococca wallichii* of Nepalese Origin. *Nat. Pro. Commun.* **2015**, *10*, 1533–1536.
5. Devkota, K. P.; Wansi, J. D.; Lenta, B. N. Bioactive Steroidal Alkaloids from *Sarcococca hookeriana*. *Planta Med*. **2010**, *76*, 1022–1025.
6. Khan, M. T. H. Putative Molecular Interactions Involving Naturally Occurring Steroidal Alkaloids from *Sarcococca hookeriana* against Acetyl- and Butyryl-Cholinesterase. *Curr. Bioinform.* **2013**, *8*, 416–428.
7. He, K.; Zou, J.; Wang, J. X.; Du, J. Crystal Structure of (*Z*)- *N*-(17-(1-(dimethylamino)ethyl)-10,13-dimethyl-4-oxo-4,5,6,7,8,9,10,11,12,13,14,15,16,17-Tetradecahydro-1*H*-cyclopenta -[*α*] phenanthren-3-yl)-3-methylbut-2-enimidic acid dichloromethane monosolvate, $C_{29}H_{45}Cl_2N_2O_2$. *Z. Kristallogr. NCS.* **2013**, *228*, 327–328.
8. Huo, S. J.; Wu, J. C.; He, X. C.; Pan, L. T.; Du, J. Two New Cytotoxic Steroidal Alkaloids from *Sarcococca hookeriana*. *Molecules* **2018**, *24*, 11.
9. He, K.; Wang, J. X.; Zou, J.; Wu, J. C.; Huo, S. J.; Du, J. Three New Cytotoxic Steroidal Alkaloids from *Sarcococca hookeriana*. *Molecules* **2018**, *23*, 1181.
10. Zhang, W. W.; Ye, J. H.; Zhou, J.; He, K. Synthesis and Crystal Structure of 1-((3*R*,10*S*,13*R*,17*S*)-10,13- Dimethyl-3-(phenylamino)hexadecahydro-1*H*-cyclopenta [*α*] phenanthren-17-yl)ethan-1-one, $C_{27}H_{39}N_O$. *Z. Kristallogr. NCS.* **2022**, *237*, 815–817.
11. Huang, H.; Zhang, W. W.; Ye, J. H.; Zhou, J.; He, K. Synthesis and Crystal Structure of1-((3*R*,10*S*,13*R*,17*S*)-10,13- Dimethyl-3-(*p*-tolylamino) Hexadecahydro-1*H*-Cyclopenta [*α*] phenanthren-17-yl)ethan-1-One, $C_{28}H_{41}N_O$. *Z. Kristallogr. NCS.* **2023**, *238*, 31–33.
12. Zhang, W. W.; Ye, J. H.; Huang, H.; Zhou, J.; He, K. Synthesis and Crystal Structure of 1-((3*R*,10*S*,13*R*,17*S*)-3- ((4-Methoxyphenyl)amino)-10,13-dimethylhexadecahydro-1*H*- cyclopenta [*α*]-phenanthren-17-yl)ethan-1-One, $C_{28}H_{41}NO_2$. *Z. Kristallogr. NCS.* **2023**, *238*, 189–191.
13. Zhao, J. F.; Zhang, W. W.; Ye, J. H.; He, K.; Zou, J. Synthesis and Crystal Structure of 1-((3*R*,10*S*,13*S*,17*S*) -10,13-Dimethyl-3-(m-tolylamino) hexadecahydro-1*H*-cyclopenta[*α*] phenanthren-17-yl)ethan-1-one, $C_{28}H_{41}N_O$. *Z. Kristallogr. NCS.* **2023**, *238*, 579–581.
14. Yue, L.; Wei, P.; Ye, J. H.; He, K.; Zou, J. Synthesis and Crystal Structure of(3*E*,5*S*,10*S*,13*S*,14*S*, 17*Z*)-17-Ethylidene-10,13-dimethylhexadecahydro-3*H*-cyclopenta[*α*]phenanthren-3-one *O*-(4-fluorobenzoyl) Oxime, $C_{28}H_{36}FNO_2$. *Z. Kristallogr. NCS.* **2023**, *238*, 941–943.
15. Wei, P.; Zhang, J. F.; Zou, J.; Ye, J. H.; He, K.; Xu, H. Synthesis and Crystal Structure of (3*E*,5*S*,10*S*,13*S*,14*S*, 17*Z*)-17-Ethylidene-10,13-dimethylhexadecahydro-3*H*- cyclopenta[*α*]phenanthren-3-one *O*-(methacryloyl) Oxime, $C_{50}H_{74}N_2O_4$. *Z. Kristallogr. NCS.* **2023**, *238*, 1219–1222.
16. Yang, M.; Wei, P.; Sun, Y. C.; Ye, J. H.; Zou, J.; He, K. Synthesis and Crystal Structure of (3*S*,10*S*,13*S*,17*S*)–*N*- (2-Methoxyphenyl)-10,13-dimethyl-17-((*R*)-1-(*p*-tolylamino) ethyl)hexadecahydro-1*H*-cyclopenta[*α*] phenanthren-3-amine, $C_{35}H_{50}N_2O$. *Z. Kristallogr. NCS.* **2025**, *240*, 221–223.
17. Li, L. L.; Wei, P.; Ye, J. H.; Zou, J.; He, K. Synthesis and Crystal Structure Of-(3*S*,10*S*,13*S*,17*S*)–*N*-(2-Methoxyphenyl) -10,13-dimethyl-17-((*R*)-1-(phenylamino)ethyl)hexadecahydro-1*H*-cyclopenta[*α*]phenanthren-3-amine, $C_{34}H_{48}N_2O$. *Z. Kristallogr. NCS.* **2025**, *240*, 195–197.