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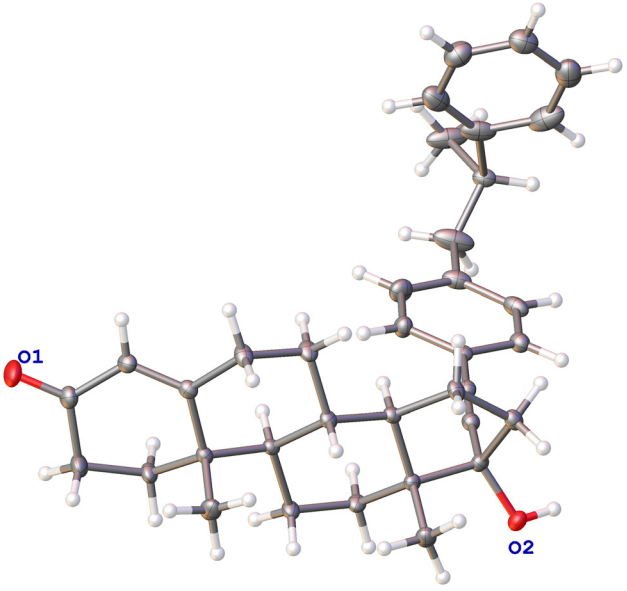
# Crystal structure of (8*R*,9*S*,10*R*,13*S*,14*S*,17*S*)-17-hydroxy-10,13-dimethyl-17-((4-(2-phenylpropyl)phenyl)ethynyl)-1,2,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-3*H*-cyclopenta[*a*]phenanthren-3-one, $C_{36}H_{42}O_2$

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**Abstract**

$C_{36}H_{42}O_2$, monoclinic, $P2_1/n$ (no. 4), $a$ = 9.0092(8) Å, $b$ = 9.4264(11) Å, $c$ = 17.2712(16) Å, $\beta$ = 96.156(8)°, $V$ = 1458.3(3) Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0509, $wR_{ref}(F^2)$ = 0.1272, T = 150.00(10) K.

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**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.15 × 0.12 × 0.10 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 0.07 mm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova, $\omega$ |
| $\theta_{max}$, completeness: | 29.6°, >99 % |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 7892, 5103, 0.032 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs} > 2\sigma(I_{obs})$, 4133 |
| $N(param)_{refined}$: | 358 |
| Programs: | Bruker [1], Olex2 [2], SHELX [3] |

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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.

## 1 Source of materials

The compound was synthesized according to the procedure in our previous work [4]. A mixture of 1-bromo-4-(2-phenylpropyl)benzene (137.0 mg, 0.5 mmol), ethisterone (234.2 mg, 0.75 mmol), bis(triphenylphosphine)palladium(II) chloride (35.1 mg, 0.05 mmol), CuI (19.0 mg, 0.1 mmol), and triethylamine (1.0 mL) in anhydrous MeCN (1.5 mL) was added to a reaction bottle (30 mL). The solution was stirred under nitrogen at 70 °C. After 8 h, the mixture was poured into water and extracted with ethyl acetate (20.0 × 3). The combined organic layer was washed with brine (10 mL), dried over $MgSO_4$, filtered, and concentrated. The crude products were purified by flash column chromatography using petroleum ether/ethyl acetate (10/1, *v*/*v*) to give the title compound

**Table 2:** Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$).

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| O1 | 1.2032 (3) | 1.2494 (3) | 0.81003 (18) | 0.0711 (9) |
| O2 | 0.2507 (2) | 0.5433 (3) | 0.81200 (14) | 0.0449(6) |
| H2 | 0.219124 | 0.459772 | 0.806092 | 0.067* |
| C1 | 0.9622 (3) | 1.0029 (3) | 0.88912 (16) | 0.0348 (7) |
| C2 | 1.0859 (3) | 1.0670 (4) | 0.87196 (18) | 0.0390 (7) |
| H2A | 1.178939 | 1.032478 | 0.895626 | 0.047* |
| C3 | 1.0872 (4) | 1.1864 (4) | 0.8194 (2) | 0.0455(8) |
| C4 | 0.9397 (4) | 1.2357 (4) | 0.7810 (2) | 0.0537 (9) |
| H4A | 0.953343 | 1.276495 | 0.729406 | 0.064* |
| H4B | 0.900589 | 1.311460 | 0.812955 | 0.064* |
| C5 | 0.8264 (3) | 1.1148 (4) | 0.77064 (19) | 0.0429(8) |
| H5A | 0.858381 | 1.046535 | 0.732152 | 0.051* |
| H5B | 0.728358 | 1.153777 | 0.749456 | 0.051* |
| C6 | 0.8087 (3) | 1.0362 (3) | 0.84652 (17) | 0.0324 (6) |
| C7 | 0.7246 (3) | 0.8941 (3) | 0.82665 (15) | 0.0285 (6) |
| H7 | 0.786208 | 0.842067 | 0.791044 | 0.034* |
| C8 | 0.5699 (3) | 0.9158 (4) | 0.77934 (18) | 0.0400 (8) |
| H8A | 0.509288 | 0.979704 | 0.808837 | 0.048* |
| H8B | 0.584289 | 0.963091 | 0.729520 | 0.048* |
| C9 | 0.4842 (3) | 0.7785 (4) | 0.76198 (17) | 0.0372 (7) |
| H9A | 0.535819 | 0.720419 | 0.725348 | 0.045* |
| H9B | 0.382830 | 0.800532 | 0.736822 | 0.045* |
| C10 | 0.4721 (3) | 0.6947 (3) | 0.83653 (16) | 0.0311 (7) |
| C11 | 0.6315 (3) | 0.6634 (3) | 0.87498 (16) | 0.0307 (6) |
| H11 | 0.685280 | 0.614215 | 0.834911 | 0.037* |
| C12 | 0.7194 (3) | 0.7970 (3) | 0.89798 (16) | 0.0312 (6) |
| H12 | 0.668505 | 0.848820 | 0.938240 | 0.037* |
| C13 | 0.8798 (3) | 0.7617 (4) | 0.93202 (18) | 0.0441(8) |
| H13A | 0.877177 | 0.701516 | 0.978805 | 0.053* |
| H13B | 0.930036 | 0.707492 | 0.893215 | 0.053* |
| C14 | 0.9686 (4) | 0.8962 (4) | 0.95394 (18) | 0.0470 (9) |
| H14A | 0.929420 | 0.940264 | 0.999630 | 0.056* |
| H14B | 1.074170 | 0.870207 | 0.969297 | 0.056* |
| C15 | 0.6085 (3) | 0.5531 (4) | 0.93776 (17) | 0.0396 (7) |
| H15A | 0.593959 | 0.600034 | 0.987677 | 0.048* |
| H15B | 0.695621 | 0.488804 | 0.946174 | 0.048* |
| C16 | 0.4669 (3) | 0.4704 (4) | 0.90588 (17) | 0.0388 (7) |
| H16A | 0.390049 | 0.476272 | 0.942648 | 0.047* |
| H16B | 0.490925 | 0.369278 | 0.897994 | 0.047* |
| C17 | 0.4096 (3) | 0.5419 (3) | 0.82644 (16) | 0.0329 (7) |
| C18 | 0.4712 (3) | 0.4659 (3) | 0.76245 (17) | 0.0346 (7) |
| C19 | 0.5143 (3) | 0.4001 (4) | 0.71026 (17) | 0.0362 (7) |
| C20 | 0.5606 (3) | 0.3207 (3) | 0.64661 (16) | 0.0334 (7) |
| C21 | 0.4932 (3) | 0.1919 (4) | 0.62434 (19) | 0.0425 (8) |
| H21 | 0.414510 | 0.156534 | 0.651396 | 0.051* |
| C22 | 0.5396 (4) | 0.1153 (4) | 0.5635 (2) | 0.0514 (9) |
| H22 | 0.491481 | 0.028079 | 0.549073 | 0.062* |
| C23[a] | 0.8488 (7) | 0.0259 (9) | 0.4648 (3) | 0.045 (2) |
| H23[a] | 0.920186 | 0.106665 | 0.462584 | 0.054* |
| C24 | 0.6744 (3) | 0.3682 (4) | 0.6054 (2) | 0.0444 (8) |
| H24 | 0.722190 | 0.455946 | 0.618990 | 0.053* |
| C25 | 0.7191 (4) | 0.2885 (5) | 0.5444 (2) | 0.0544 (10) |
| H25 | 0.797351 | 0.323029 | 0.516805 | 0.065* |
| C26 | 0.7034 (6) | 0.0762 (6) | 0.4571 (2) | 0.0873 (17) |
| H26A[b] | 0.613865 | 0.061642 | 0.419443 | 0.105* |
| H26B[b] | 0.771532 | 0.138041 | 0.430974 | 0.105* |
| H26C[a] | 0.635732 | −0.006365 | 0.448355 | 0.105* |
| H26D[a] | 0.689500 | 0.135339 | 0.409526 | 0.105* |
| C27 | 0.6533 (4) | 0.1614 (4) | 0.52299 (19) | 0.0528 (10) |
| C28[b] | 0.7743 (11) | −0.0566 (11) | 0.4667(4) | 0.039 (3) |
| H28[b] | 0.697844 | −0.132982 | 0.468943 | 0.047* |
| C29 | 0.8691 (4) | −0.0847 (7) | 0.3959 (2) | 0.0860 (18) |
| H29D[a] | 0.974420 | −0.088345 | 0.386580 | 0.129* |
| H29E[a] | 0.808635 | −0.054081 | 0.348265 | 0.129* |
| H29F[a] | 0.836566 | −0.179096 | 0.410822 | 0.129* |
| H29A[b] | 0.974613 | −0.064832 | 0.412125 | 0.129* |
| H29B[b] | 0.833468 | −0.022602 | 0.352306 | 0.129* |
| H29C[b] | 0.857695 | −0.183992 | 0.379524 | 0.129* |
| C30 | 0.8918 (5) | −0.0647 (6) | 0.5410 (2) | 0.0713 (14) |
| C31 | 0.8332 (5) | −0.1826 (7) | 0.5761 (3) | 0.0878 (18) |
| H31 | 0.742351 | −0.223130 | 0.553244 | 0.105* |
| C32 | 0.9059(5) | −0.2426 (6) | 0.6445 (3) | 0.0761 (13) |
| H32 | 0.864226 | −0.322464 | 0.667876 | 0.091* |
| C33 | 1.0349 (5) | −0.1856 (5) | 0.6764 (2) | 0.0685 (12) |
| H33 | 1.086561 | −0.227249 | 0.721576 | 0.082* |
| C34 | 1.0910 (4) | −0.0705 (6) | 0.6448 (3) | 0.0722 (13) |
| H34 | 1.180608 | −0.029244 | 0.668749 | 0.087* |
| C35 | 1.0200 (5) | −0.0108 (5) | 0.5775 (2) | 0.0721 (12) |
| H35 | 1.062634 | 0.070698 | 0.556198 | 0.086* |
| C36 | 0.7239 (4) | 1.1293 (4) | 0.9002 (2) | 0.0537 (10) |
| H36A | 0.628570 | 1.159113 | 0.872269 | 0.081* |
| H36B | 0.783923 | 1.213229 | 0.915914 | 0.081* |
| H36C | 0.705281 | 1.074919 | 0.946498 | 0.081* |
| C37 | 0.3762 (3) | 0.7752 (4) | 0.8905 (2) | 0.0473 (8) |
| H37A | 0.376189 | 0.723601 | 0.939716 | 0.071* |
| H37B | 0.273679 | 0.782953 | 0.865353 | 0.071* |
| H37C | 0.417549 | 0.870345 | 0.900665 | 0.071* |

[a]Occupancy: 0.590(12), [b]Occupancy: 0.410(12).

(197.5 mg, 0.39 mmol). The solid product was recrystallized from a mixture of ethyl acetate and petroleum ether (*v*/*v*, 1/20, 20 mL). Colorless block crystals were obtained after 3 days.

## 2 Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

## 3 Comment

Chiral compounds are important substances in the construction of living organisms and are widely found in natural products, active compounds and drugs [5]. Despite no differences in physical and chemical properties, several

enantiomers have been reported to differ significantly in pharmacological activity, metabolic processes, metabolic rates, and toxicity [6, 7]. Progestrel is a progesterone, which can transform the proliferative endometrium into secretory phase and promote mammary gland development, and its importance has been well known to the public. As part of our current research interest in progesterone compounds, we report here the molecular structure of a novel chiral acetylgestrel derivative.

The asymmetric unit of the title compound contains (8*R*,9*S*, 10*R*,13*S*,14*S*,17*S*)-17-hydroxy-10,13-dimethyl-17-((4-(2-phenylpropyl)phenyl)ethynyl)-1,2,6,7,8,9,10,11,12,13,14,15,16,17-tetradecahydro-3*H*-cyclopenta[*a*]phenanthren-3-one. The single-crystal structure is shown in the figure. All bond lengths and angles of this molecule are in the expected ranges [8, 9].

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