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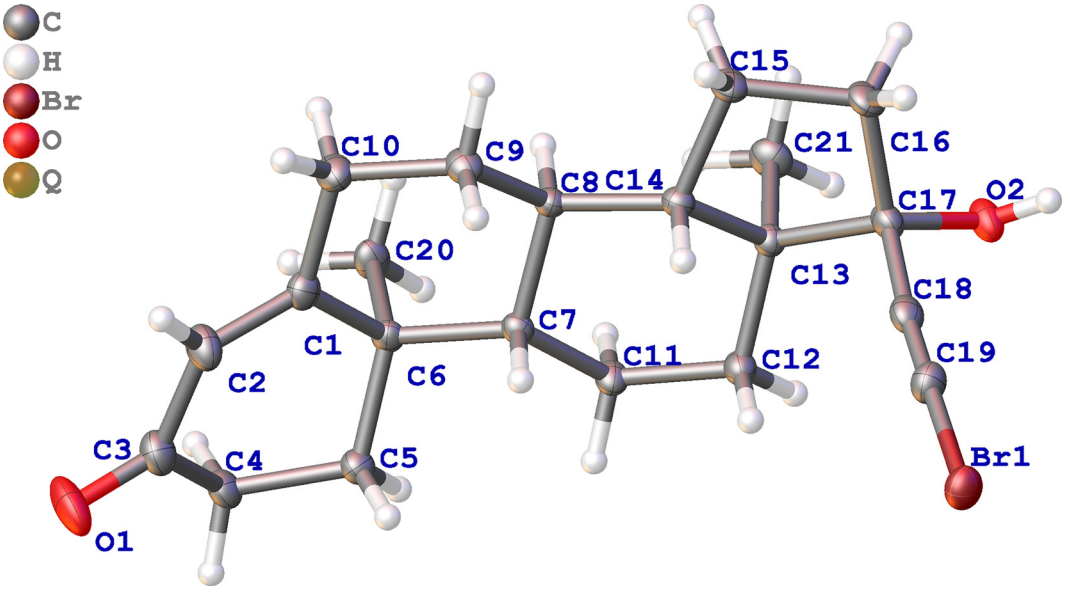
The crystal structure of 17-(bromoethynyl)-17-hydroxy-10, 13-dimethyl- 1,2,6,7,8,9,10,11,12, 13,14,15,16,17-tetradecahydro-3*H*-cyclopenta[*a*] phenanthren-3-one, $C_{21}H_{27}BrO_2$



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Abstract

$C_{21}H_{27}BrO_2$, monoclinic, $P2_1$ (no. 4), a = 6.6220(14) Å, b = 11.7640(2) Å, c = 11.8485(2) Å, β = 95.0757(19)° V = 919.39(3) Å^3, Z = 2, $R_{gt}(F)$ = 0.0264, $wR_{ref}(F^2)$ = 0.0695, T = 150 K, Flack-parameter = 0.007(13).

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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.

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.13 × 0.12 × 0.11 mm
Wavelength:	Cu *K*α radiation (1.54184 Å)
μ:	3.11 mm^{-1}
Diffractometer, scan mode:	SuperNova, ω
θ_{max}, completeness:	73.8°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	5032, 3136, 0.025
Criterion for I_{obs}, $N(hkl)_{gt}$:	I_{obs} > 2 σ(I_{obs}), 3117
$N(param)_{refined}$:	223
Programs:	CrysAlis^{PRO} [1], SHELX [2, 3], Olex2 [4]

Source of material

A mixture of ethisterone (2.0 mmol) was dissolved in acetone (15 mL), and then NBS (2.4 mmol), and silver nitrate (0.12 mmol) was added. The reaction mixture was reacted at room temperature and stirred for 8 h. Afterwards, the mixture was filtered and concentrated under vacuum. The residue was purified by flash chromatography (petroleum ether:acetate = 5:1, v/v) on silica gel to afford the title compound as a white solid.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.57735 (4)	0.73015 (4)	0.02963 (2)	0.02312 (11)
O1	0.8707 (5)	0.4905 (3)	0.8885 (3)	0.0375 (8)
O2	0.1317 (4)	0.3783 (2)	0.0460 (2)	0.0226 (5)
H2	0.080 (10)	0.407 (7)	−0.013 (6)	0.08 (2)*
C1	0.4423 (5)	0.4721 (3)	0.6846 (3)	0.0182 (6)
C2	0.5656 (6)	0.5079 (3)	0.7731 (3)	0.0239 (7)
H2A	0.527705	0.571802	0.812338	0.029*
C3	0.7573 (6)	0.4518 (3)	0.8114 (3)	0.0244 (7)
C4	0.8025 (5)	0.3438 (3)	0.7510 (3)	0.0205 (6)
H4A	0.737225	0.280412	0.785528	0.025*
H4B	0.947568	0.330182	0.758283	0.025*
C5	0.7265 (5)	0.3516 (3)	0.6258 (3)	0.0186 (6)
H5A	0.754962	0.280463	0.589050	0.022*
H5B	0.800738	0.411148	0.590759	0.022*
C6	0.4980 (5)	0.3767 (3)	0.6057 (3)	0.0155 (6)
C7	0.4464 (4)	0.4166 (3)	0.4810 (2)	0.0145 (6)
H7	0.530774	0.483716	0.471017	0.017*
C8	0.2240 (4)	0.4567 (3)	0.4577 (2)	0.0147 (6)
H8	0.133400	0.393500	0.472173	0.018*
C9	0.1848 (5)	0.5550 (3)	0.5373 (3)	0.0190 (6)
H9A	0.266286	0.619795	0.518928	0.023*
H9B	0.043319	0.577037	0.525444	0.023*
C10	0.2346 (5)	0.5238 (3)	0.6614 (3)	0.0220 (7)
H10A	0.226384	0.591657	0.707314	0.026*
H10B	0.134116	0.470364	0.683863	0.026*
C11	0.5056 (5)	0.3284 (3)	0.3925 (3)	0.0173 (6)
H11A	0.650469	0.314959	0.403711	0.021*
H11B	0.437713	0.257121	0.405577	0.021*
C12	0.4510 (5)	0.3653 (3)	0.2695 (3)	0.0182 (6)
H12A	0.533830	0.429992	0.252332	0.022*
H12B	0.480128	0.303731	0.219012	0.022*
C13	0.2272 (5)	0.3974 (2)	0.2495 (2)	0.0148 (6)
C14	0.1858 (4)	0.4924 (3)	0.3343 (2)	0.0151 (6)
H14	0.281437	0.553885	0.322401	0.018*
C15	−0.0264 (5)	0.5366 (3)	0.2923 (3)	0.0204 (6)
H15A	−0.040006	0.616465	0.310348	0.025*
H15B	−0.130904	0.494089	0.326430	0.025*
C16	−0.0415 (5)	0.5180 (3)	0.1622 (3)	0.0217 (7)
H16A	−0.056540	0.590186	0.122805	0.026*
H16B	−0.157113	0.470440	0.138385	0.026*
C17	0.1595 (5)	0.4586 (3)	0.1360 (3)	0.0186 (7)
C18	0.3076 (5)	0.5458 (3)	0.1060 (3)	0.0198 (6)
C19	0.4139 (5)	0.6188 (3)	0.0764 (3)	0.0217 (7)
C20	0.3767 (5)	0.2695 (3)	0.6323 (3)	0.0198 (6)
H20A	0.405844	0.249520	0.710645	0.030*
H20B	0.414356	0.207779	0.585503	0.030*
H20C	0.234277	0.284421	0.617410	0.030*
C21	0.0920 (5)	0.2920 (3)	0.2597 (3)	0.0226 (7)
H21A	−0.047996	0.313918	0.248361	0.034*
H21B	0.118667	0.259260	0.333754	0.034*
H21C	0.121021	0.237146	0.203402	0.034*

Experimental details

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

Comment

Bromoalkynols is a high value synthon which is able to provide multiple reaction by activation of bromine atoms at the ethinyl moiety [5, 6]. Therefore, chemists have long been committed to the efficient conversion of bromoalkynols [7, 8]. However, the crystal structure of the title compound has not been reported before. To the best of our knowledge, only a small amount examples of organic molecules containing bromoalkynols or fused ring alkynol derivatives have been reported so far [9–11].

There is one target molecule in the asymmetric unit of the title compound (see the figure). The so-called Flack parameter (Table 1) verifies the chirality at six carbon atoms (See the figure; C6: *R*; C7: *S*; C8: *R*; C13: *S*; C14: *S*; C17: *S*). Bond lengths and bond angles are all in the expected ranges [12].

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