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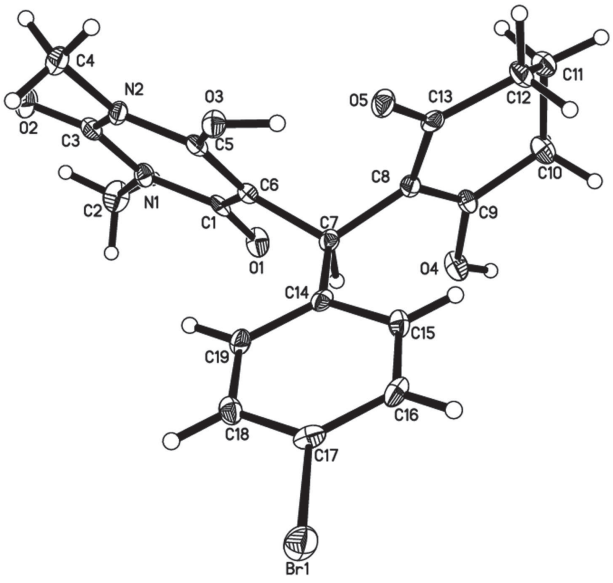
Crystal structure of 5-((4-bromophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl)-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione, $C_{19}H_{19}BrN_2O_5$



DOI 10.1515/ncrs-2015-0288
Received December 18, 2015; accepted March 22, 2016; available online May 16, 2016

Abstract

$C_{19}H_{19}BrN_2O_5$, monoclinic, $P2_1/c$ (no. 14), $a = 10.2634(5)$ Å, $b = 10.7951(4)$ Å, $c = 17.0558(8)$ Å, $\beta = 104.474(2)°$, $V = 1829.71(14)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0360$, $wR_{ref}(F^2) = 0.0772$, $T = 100$ K.

CCDC no.: 1443066

The measurement method and a list of the atoms including atomic coordinates and displacement parameters summarized in Tables 1–3.

Table 1: Data collection and handling.

Crystal:	Block, Colourless, size 0.090×0.124×0.417 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	22.81 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II, φ and ω scans
$2\theta_{max}$:	49.99°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	16043, 3222
$N(param)_{refined}$:	254
Programs:	SAINT [12], SHELX [13]

Source of material

The synthesis of 5-((4-bromophenyl)(2-hydroxy-6-oxocyclohex-1-en-1-yl)methyl)-6-hydroxy-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione prepared according to reported method by Barakat et al. [1]. The precursors used in this study are commercially available. A mixture of 4-bromobenzaldehyde (1.5 mmol), cyclohexane-1,3-dione (1.5 mmol), 1,3-dimethylpyrimidine-2,4,6(1*H*,3*H*,5*H*)-trione (1.5 mmol), and Et_2NH (1.5 mmol, 155 µL) in H_2O (3 mL, degassed). The reaction mixture was further stirred for 24 h at rt (Monitored by TLC). The desired compound was precipitated and filtered off, then recrystallized from a mixture of CH_2Cl_2/EtOH/Et_2O over 2 days and get single cube whitless crystal and to obtain the pure product.

Yield: 90%; m.p. 140 °C; IR (KBr, cm^{-1}) $\nu_{max} = 3055$, 2952, 2661, 1692, 1600, 1483, 1459, 1397, 1242; [Anal. Calcd. for $C_{19}H_{19}BrN_2O_5$ (%): C, 52.43; H, 4.40; Br, 18.36; N, 6.44;

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	4*e*	1.0107	0.5352	0.8708	0.034
H(2B)	4*e*	0.9145	0.4213	0.8778	0.034
H(2C)	4*e*	0.9359	0.5313	0.9427	0.034
H(4A)	4*e*	0.8282	0.8167	0.6687	0.030
H(4B)	4*e*	0.6688	0.8300	0.6344	0.030
H(4C)	4*e*	0.7463	0.7154	0.6073	0.030
H(7A)	4*e*	0.4527	0.5004	0.8402	0.014
H(10A)	4*e*	0.3426	0.7298	1.0251	0.023
H(10B)	4*e*	0.1948	0.7055	0.9689	0.023
H(11A)	4*e*	0.3751	0.8987	0.9495	0.024
H(11B)	4*e*	0.2261	0.9157	0.9616	0.024
H(12A)	4*e*	0.1315	0.8324	0.8318	0.023
H(12B)	4*e*	0.2266	0.9465	0.8229	0.023
H(15A)	4*e*	0.1631	0.5697	0.7466	0.017
H(16A)	4*e*	0.0240	0.4696	0.6360	0.019
H(18A)	4*e*	0.3514	0.3453	0.5679	0.018
H(19A)	4*e*	0.4898	0.4484	0.6777	0.017
H(1O4)	4*e*	0.363(4)	0.520(4)	1.000(3)	0.06(1)
H(1O3)	4*e*	0.427(4)	0.750(4)	0.695(2)	0.05(1)

Found(%): C, 52.44; H, 4.42; Br, 18.36; N, 6.47; LC/MS (ESI, *m/z*): 435.27[M^+].

Experimental details

The X-ray data for the synthesized compound were carried out by Bruker SAINT [12]. The chemical structure were solved by SHELX program (AFIX 13, 23, 43 or 137 option) [13].

Discussion

Barbituric acid is the key pharmacophore of several medicinal targets. For example bucolome, veronal, phenobarbital, seconal, and sodium pentothal representatives have clinical use as anti-inflammatory and hypnotic drugs which are derived from barbituric acid scaffold [2–4]. On the other hand, this scaffold possessing antitumor, anti-hypertensive, sedative, anticonvulsant, anti-oxidant, and anti-diabetic potentials [5–11]. We have described in this text the synthesis of a new pyrimidines derivatives *via* one pot Aldol-Michael reactions in aqueous diethylamine medium. The desired compound was characterized by spectroscopic techniques and by X-ray single crystal analysis.

The structure of the title molecule is shown in the figure. In the target compound molecules were stabilized by the two intermolecular hydrogen bonds and one intramolecular

Table 3: Fractional coordinates and atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4*e*	0.06106(3)	0.31594(3)	0.50239(2)	0.0184(2)	0.0311(2)	0.0184(2)	−0.0069(2)	0.0032(1)	−0.0062(2)
O(1)	4*e*	0.6812(2)	0.4943(2)	0.9091(1)	0.020(1)	0.023(1)	0.012(1)	0.006(1)	0.0049(9)	0.005(1)
O(2)	4*e*	0.9476(2)	0.6634(2)	0.7615(1)	0.013(1)	0.027(1)	0.032(1)	0.001(1)	0.012(1)	0.001(1)
O(3)	4*e*	0.5000(2)	0.7351(2)	0.6747(1)	0.015(1)	0.017(1)	0.015(1)	0.0035(9)	0.005(1)	0.004(1)
O(4)	4*e*	0.3734(2)	0.5296(2)	0.9503(1)	0.025(1)	0.020(1)	0.014(1)	0.008(1)	0.008(1)	0.005(1)
O(5)	4*e*	0.3120(2)	0.8040(2)	0.7331(1)	0.016(1)	0.016(1)	0.019(1)	0.0027(9)	0.0078(9)	0.006(1)
N(1)	4*e*	0.8129(2)	0.5772(2)	0.8344(2)	0.010(1)	0.016(1)	0.014(1)	0.003(1)	0.002(1)	−0.000(1)
N(2)	4*e*	0.7224(2)	0.6990(2)	0.7205(2)	0.013(1)	0.014(1)	0.013(1)	0.001(1)	0.007(1)	0.001(1)
C(1)	4*e*	0.6861(3)	0.5578(3)	0.8493(2)	0.016(2)	0.012(2)	0.012(2)	0.002(1)	0.004(1)	−0.006(1)
C(2)	4*e*	0.9281(3)	0.5107(3)	0.8857(2)	0.016(2)	0.031(2)	0.020(2)	0.004(2)	0.001(1)	0.003(2)
C(3)	4*e*	0.8357(3)	0.6479(3)	0.7717(2)	0.017(2)	0.013(2)	0.017(2)	0.000(1)	0.006(1)	−0.005(1)
C(4)	4*e*	0.7432(3)	0.7712(3)	0.6521(2)	0.022(2)	0.018(2)	0.024(2)	0.000(1)	0.015(1)	0.004(2)
C(5)	4*e*	0.5943(3)	0.6804(3)	0.7309(2)	0.014(2)	0.010(2)	0.015(2)	0.001(1)	0.004(1)	−0.004(1)
C(6)	4*e*	0.5724(3)	0.6097(3)	0.7927(2)	0.012(2)	0.010(2)	0.010(2)	0.002(1)	0.003(1)	−0.001(1)
C(7)	4*e*	0.4341(3)	0.5721(3)	0.8018(2)	0.013(2)	0.011(2)	0.013(2)	0.002(1)	0.005(1)	0.004(1)
C(8)	4*e*	0.3651(3)	0.6667(3)	0.8443(2)	0.011(1)	0.012(2)	0.014(2)	−0.000(1)	0.004(1)	0.000(1)
C(9)	4*e*	0.3442(3)	0.6417(3)	0.9188(2)	0.010(2)	0.013(2)	0.016(2)	0.002(1)	0.004(1)	0.001(1)
C(10)	4*e*	0.2872(3)	0.7316(3)	0.9686(2)	0.021(2)	0.020(2)	0.016(2)	0.002(1)	0.005(1)	−0.004(1)
C(11)	4*e*	0.2832(3)	0.8633(3)	0.9362(2)	0.019(2)	0.019(2)	0.024(2)	−0.001(1)	0.008(2)	−0.008(2)
C(12)	4*e*	0.2260(3)	0.8613(3)	0.8444(2)	0.020(2)	0.012(2)	0.027(2)	0.006(1)	0.011(2)	0.003(1)
C(13)	4*e*	0.3064(3)	0.7777(3)	0.8035(2)	0.008(2)	0.014(2)	0.021(2)	−0.005(1)	0.003(1)	−0.001(1)
C(14)	4*e*	0.3416(3)	0.5180(3)	0.7247(2)	0.011(2)	0.010(2)	0.015(2)	0.001(1)	0.004(1)	0.006(1)
C(15)	4*e*	0.2019(3)	0.5240(3)	0.7106(2)	0.016(2)	0.017(2)	0.011(2)	0.002(1)	0.007(1)	0.001(1)
C(16)	4*e*	0.1190(3)	0.4645(3)	0.6450(2)	0.009(2)	0.020(2)	0.020(2)	−0.002(1)	0.004(1)	0.004(2)
C(17)	4*e*	0.1754(3)	0.3975(3)	0.5925(2)	0.017(2)	0.015(2)	0.013(2)	−0.006(1)	0.001(1)	0.002(1)
C(18)	4*e*	0.3132(3)	0.3909(3)	0.6042(2)	0.019(2)	0.016(2)	0.013(2)	0.003(1)	0.007(1)	0.002(1)
C(19)	4*e*	0.3950(3)	0.4519(3)	0.6699(2)	0.009(2)	0.016(2)	0.019(2)	−0.000(1)	0.006(1)	0.001(1)

one. All geometric paramters of the title molecule are in the expected ranges.

Acknowledgements: The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at King Saud University for its funding this Research group NO (RG -044–1435-1436).

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