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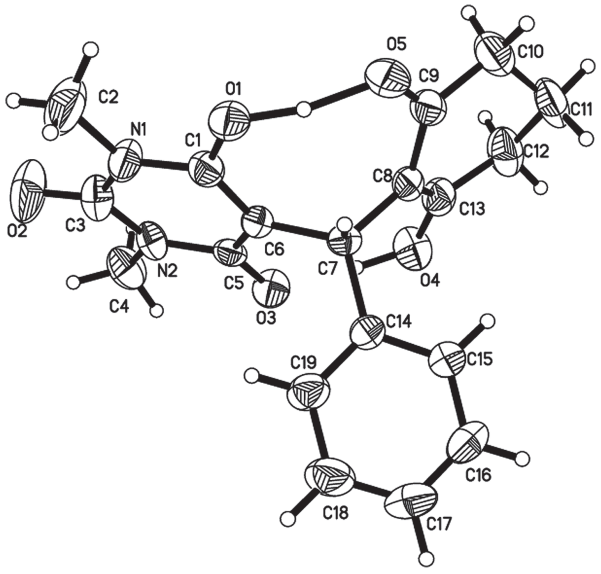
Crystal structure of 6-hydroxy-5-((2-hydroxy-6-oxocyclohex-1-en-1-yl)(phenyl)methyl)-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione, $C_{19}H_{20}N_2O_5$



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Abstract

$C_{19}H_{20}N_2O_5$, orthorhombic, $P2_12_12_1$ (no. 19), $a = 10.3920(6)$ Å, $b = 10.7709(8)$ Å, $c = 14.8810(11)$ Å, $V = 1665.6(2)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0504$, $wR_{ref}(F^2) = 0.1068$, $T = 100$ K.

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The measurement method details and a list of the atoms including atomic coordinates and displacementparameters are listed in Tables 1–3.

Table 1: Data collection and handling.

Crystal:	Block, Colourless, size 0.16×0.18×0.32 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.04 cm^{-1}
Diffractometer, scan mode:	Bruker APEX-II D8 venture, φ and ω scans
$2\theta_{max}$:	50°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	11913, 2923
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1959
$N(param)_{refined}$:	245
Programs:	Bruker programs [12], SHELX [13]

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

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2A)	4*a*	0.0068	0.6348	0.6802	0.118
H(2B)	4*a*	0.0993	0.6552	0.5955	0.118
H(2C)	4*a*	0.0813	0.7637	0.6679	0.118
H(4A)	4*a*	0.3173	0.4102	0.9435	0.078
H(4B)	4*a*	0.2366	0.3283	0.8737	0.078
H(4C)	4*a*	0.1663	0.4339	0.9312	0.078
H(7A)	4*a*	0.5388	0.7374	0.6773	0.035
H(10A)	4*a*	0.7197	0.5783	0.4412	0.068
H(10B)	4*a*	0.6484	0.4529	0.4702	0.068
H(11A)	4*a*	0.8792	0.5488	0.5484	0.072
H(11B)	4*a*	0.8671	0.4223	0.4923	0.072
H(12A)	4*a*	0.7550	0.3256	0.6061	0.070
H(12B)	4*a*	0.8747	0.3918	0.6539	0.070
H(15A)	4*a*	0.8120	0.6679	0.7554	0.054
H(16A)	4*a*	0.9209	0.7698	0.8692	0.068
H(17A)	4*a*	0.8122	0.8770	0.9779	0.067
H(18A)	4*a*	0.5888	0.8800	0.9767	0.071
H(19A)	4*a*	0.4756	0.7801	0.8617	0.055
H(1O4)	4*a*	0.615(4)	0.462(4)	0.799(3)	0.10(2)
H(1O1)	4*a*	0.428(4)	0.702(4)	0.578(3)	0.10(2)

Source of material

The synthesis of 6-hydroxy-5-((2-hydroxy-6-oxocyclohex-1-en-1-yl) (phenyl)methyl)-1,3-dimethylpyrimidine-2,4(1*H*,3*H*)-dione follows a reported protocol [1].

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}
O(1)	4*a*	0.3274(2)	0.7153(2)	0.6108(2)	0.042(1)	0.056(2)	0.034(1)	0.000(1)	−0.004(1)	0.009(1)
O(2)	4*a*	0.0577(2)	0.5267(3)	0.8077(2)	0.036(2)	0.106(2)	0.097(2)	−0.002(2)	0.014(1)	0.034(2)
O(3)	4*a*	0.4884(2)	0.4766(2)	0.8516(1)	0.043(1)	0.044(1)	0.040(1)	0.002(1)	−0.003(1)	0.011(1)
O(4)	4*a*	0.6984(2)	0.4336(2)	0.7634(2)	0.053(2)	0.048(2)	0.051(2)	0.008(1)	0.007(1)	0.010(1)
O(5)	4*a*	0.5352(2)	0.6699(2)	0.5255(1)	0.055(2)	0.064(2)	0.032(1)	−0.003(1)	−0.001(1)	0.006(1)
N(1)	4*a*	0.1953(2)	0.6246(3)	0.7111(2)	0.028(2)	0.059(2)	0.045(2)	−0.003(2)	−0.001(1)	0.011(2)
N(2)	4*a*	0.2731(2)	0.5010(2)	0.8298(2)	0.036(2)	0.037(2)	0.038(2)	0.001(1)	0.012(1)	0.003(1)
C(1)	4*a*	0.3182(3)	0.6521(3)	0.6858(2)	0.037(2)	0.037(2)	0.036(2)	−0.002(2)	0.003(2)	−0.005(2)
C(2)	4*a*	0.0867(3)	0.6736(5)	0.6594(3)	0.037(2)	0.123(4)	0.076(3)	0.009(2)	−0.010(2)	0.026(3)
C(3)	4*a*	0.1679(3)	0.5495(3)	0.7844(2)	0.034(2)	0.053(2)	0.059(2)	0.001(2)	0.010(2)	0.001(2)
C(4)	4*a*	0.2461(3)	0.4110(3)	0.9003(2)	0.061(2)	0.046(2)	0.048(2)	−0.001(2)	0.017(2)	0.007(2)
C(5)	4*a*	0.4003(3)	0.5296(3)	0.8072(2)	0.037(2)	0.032(2)	0.025(2)	−0.003(2)	0.002(2)	−0.007(2)
C(6)	4*a*	0.4212(3)	0.6150(3)	0.7373(2)	0.030(2)	0.030(2)	0.029(2)	0.000(2)	0.000(1)	−0.000(2)
C(7)	4*a*	0.5557(3)	0.6622(3)	0.7149(2)	0.033(2)	0.028(2)	0.026(2)	−0.003(2)	−0.001(1)	0.001(2)
C(8)	4*a*	0.6322(3)	0.5781(3)	0.6523(2)	0.031(2)	0.027(2)	0.035(2)	−0.009(2)	0.005(1)	−0.002(2)
C(9)	4*a*	0.6184(3)	0.5957(3)	0.5584(2)	0.041(2)	0.042(2)	0.035(2)	−0.014(2)	0.006(2)	−0.003(2)
C(10)	4*a*	0.6986(3)	0.5244(4)	0.4930(2)	0.061(2)	0.060(2)	0.049(2)	−0.013(2)	0.020(2)	−0.010(2)
C(11)	4*a*	0.8220(4)	0.4777(4)	0.5351(2)	0.058(2)	0.060(3)	0.063(2)	−0.006(2)	0.034(2)	−0.014(2)
C(12)	4*a*	0.7932(3)	0.4071(3)	0.6213(3)	0.049(2)	0.051(2)	0.074(3)	0.007(2)	0.020(2)	−0.007(2)
C(13)	4*a*	0.7025(3)	0.4770(3)	0.6813(2)	0.037(2)	0.039(2)	0.049(2)	−0.009(2)	0.006(2)	−0.004(2)
C(14)	4*a*	0.6313(3)	0.7137(3)	0.7953(2)	0.033(2)	0.027(2)	0.035(2)	−0.003(2)	−0.001(2)	0.000(2)
C(15)	4*a*	0.7646(3)	0.7117(3)	0.7999(2)	0.039(2)	0.054(2)	0.042(2)	−0.008(2)	0.001(2)	−0.010(2)
C(16)	4*a*	0.8296(4)	0.7723(4)	0.8678(3)	0.040(2)	0.065(3)	0.066(3)	−0.012(2)	−0.010(2)	−0.010(2)
C(17)	4*a*	0.7661(4)	0.8348(3)	0.9320(2)	0.064(3)	0.047(2)	0.056(2)	−0.003(2)	−0.020(2)	−0.013(2)
C(18)	4*a*	0.6344(4)	0.8373(4)	0.9308(3)	0.065(3)	0.056(3)	0.058(3)	0.012(2)	−0.007(2)	−0.023(2)
C(19)	4*a*	0.5669(3)	0.7772(3)	0.8623(2)	0.044(2)	0.040(2)	0.053(2)	0.003(2)	−0.009(2)	−0.012(2)

In a round bottom flask (25 mL), a mixture of benzaldehyde (1.5 mmol), cyclohexane-1,3-dione (1.5 mmol), 1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione (1.5 mmol), and Et_2NH (1.5 mmol, 155 µL) in H_2O (3 mL). The resulting reaction mixture was continuing stirring at r.t for 24 h (TLC n.hexane/Ethyl acetate: 8:2). The resulting precipitated product was filtered, dried and recrystallized from $EtOH/CH_2Cl_2/Et_2O$ over 2 days to get cuboid-shaped crystals as the pure product. Yield: 85%; m.p. 127°C; IR (KBr, cm^{-1}) $\nu_{max} = 3050$, 2950, 2661, 1678, 1588, 1450, 1340, 1226; LC/MS (ESI, *m/z*): 356.38 [M^+]; [Anal. calcd. for $C_{19}H_{20}N_2O_5$ (%): C, 64.04; H, 5.66; N, 7.86; found (%): C, 64.03; H, 5.66; N, 7.85].

Experimental details

Cell refinement and data reduction were collected by Bruker SAINT [12]. The carbon bonded hydrogen atoms were placed on calculated positions with the help of the SHELX program (AFIX 13, 23, 43 or 137 option) [13]. The oxygen bonded hydrogen atoms were refined freely.

Discussion

Barbituric acid represents the key pharmacophore of several pharmaceutically agents. Thus, several substituted barbituric acid for example bucolome, veronal, phenobarbital, seconal, and sodium pentothal have been used as hypnotic and anti-inflammatory clinical drugs [2–4]. Furthermore, this class of compounds exhibit anti-hypertensive, antitumor, sedative, anticonvulsant, anti-oxidant, anti-diabetic potentials [5–11]. We have synthesized a new compound derived from barbituric acid derivative via Aldol-Michael reactions in the presence of $NHEt_2$ in water. The desired compound was characterized by spectroscopic techniques and by X-ray single crystal analysis.

In the target compound, molecules contain no strong intermolecular hydrogen bonds. Two intramolecular hydrogen bonds between O4, and O1 as donors and O3 and O5 as acceptor are present (figure). According to the short distances these hyrogen bonds must be classified as strong.

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