Mezna saleh Altowyan, M. Ali, Obaid S. AlRuqi, Abdullah Mohammed Al-Majid and Assem Barakat*

Crystal structure of 5,5'-((3-hydroxy-4-methoxyphenyl)methylene)bis(1,3-diethyl-6-hydroxy-2-thioxo-2,3-dihydropyrimidin-4(1H)-one),

$C_{24}H_{30}N_4O_6S_2$

Table 1: Data collection and handling.

Crystal:	Orange plate
Size:	0.32 × 0.19 × 0.08 mm
Wavelength:	Mo K α radiation (0.71073 Å)
μ :	0.26 mm ⁻¹
Diffraction, scan mode:	Bruker D8 APEX-II, φ and ω
$\theta_{\max}$, completeness:	26.5°, >99%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	40815, 5252, 0.099
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3096
$N(\text{param})_{\text{refined}}$:	338
Programs:	SHELX [9], Bruker [10]

The asymmetric unit of the crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

<https://doi.org/10.1515/ncrs-2018-0105>

Received March 18, 2018; accepted May 8, 2018; available online June 13, 2018

Abstract

$C_{24}H_{30}N_4O_6S_2$, triclinic, $P\bar{1}$ with $a = 9.2557(4)$ Å, $b = 10.2606(5)$ Å, $c = 14.0972(7)$ Å, $\alpha = 71.949(2)^\circ$, $\beta = 89.305(2)^\circ$, $\gamma = 86.061(2)^\circ$, $V = 1269.84(10)$ Å³, $Z = 2$, $R_{\text{gt}}(F) = 0.058$, $wR_{\text{ref}}(F^2) = 0.142$, $T = 293(2)$ K.

CCDC no.: 1829303

***Corresponding author: Assem Barakat**, Department of Chemistry, College of Science, King Saud University, P. O. Box 2455, Riyadh 11451, Saudi Arabia; and Department of Chemistry, Faculty of Science, Alexandria University, P. O. Box 426, Ibrahimia, Alexandria 21321, Egypt, e-mail: ambarakat@ksu.edu.sa

Mezna saleh Altowyan: Department of Chemistry, College of Science, Princess Nourah Bint Abdulrahman University, Riyadh, Saudi Arabia

M. Ali and Abdullah Mohammed Al-Majid: Department of Chemistry, College of Science, King Saud University, P. O. Box 2455, Riyadh 11451, Saudi Arabia

Obaid S. AlRuqi: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P. O. Box 2457, Riyadh 11451, Saudi Arabia

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
S1	0.55104(10)	0.09379(10)	0.16087(9)	0.0652(3)
S2	0.50367(12)	0.71591(13)	0.58722(7)	0.0745(4)
O1	1.1212(3)	0.6487(3)	−0.00279(19)	0.0556(7)
O2	1.2113(2)	0.8589(2)	0.04294(17)	0.0511(6)
O3	0.8617(2)	0.3871(2)	0.27282(15)	0.0414(5)
O4	0.4152(2)	0.5806(2)	0.12145(16)	0.0446(5)
O5	0.3931(2)	0.7236(2)	0.24519(16)	0.0481(6)
O6	0.8530(2)	0.5461(2)	0.38043(15)	0.0480(6)
H6A	0.8521	0.4958	0.3452	0.072*
N1	0.7160(2)	0.2655(2)	0.20960(17)	0.0321(5)
N2	0.4912(2)	0.3595(2)	0.13843(17)	0.0339(6)
N3	0.4543(2)	0.7114(3)	0.40297(17)	0.0409(6)
N4	0.6851(3)	0.6284(2)	0.46854(18)	0.0387(6)
C1	0.9120(3)	0.6428(3)	0.0971(2)	0.0309(6)
H1A	0.8825	0.5691	0.0787	0.037*
C2	1.0393(3)	0.7007(3)	0.0605(2)	0.0332(7)
C3	1.0854(3)	0.8115(3)	0.0863(2)	0.0355(7)
C4	1.0033(3)	0.8624(3)	0.1498(2)	0.0437(8)
H4A	1.0336	0.9357	0.1683	0.052*
C5	0.8742(3)	0.8042(3)	0.1869(2)	0.0416(7)
H5A	0.8186	0.8400	0.2296	0.050*
C6	0.8276(3)	0.6943(3)	0.1613(2)	0.0282(6)
C7	0.6788(3)	0.6417(3)	0.19588(19)	0.0290(6)
H7A	0.6097	0.7064	0.1494	0.035*

Table 2 (continued)

Atom	x	y	z	U _{iso} [*] /U _{eq}
C8	0.6506(3)	0.5030(3)	0.18640(19)	0.0283(6)
C9	0.7450(3)	0.3880(3)	0.2243(2)	0.0316(6)
C10	0.5870(3)	0.2456(3)	0.1696(2)	0.0367(7)
C11	0.5178(3)	0.4854(3)	0.1484(2)	0.0334(7)
C12	0.8292(3)	0.1510(3)	0.2406(2)	0.0471(8)
H12A	0.9236	0.1885	0.2321	0.057*
H12B	0.8241	0.0940	0.1974	0.057*
C13	0.8148(4)	0.0642(4)	0.3460(3)	0.0737(12)
H13A	0.8913	−0.0074	0.3623	0.111*
H13B	0.7229	0.0240	0.3544	0.111*
H13C	0.8208	0.1198	0.3893	0.111*
C14	0.3493(3)	0.3477(3)	0.0953(2)	0.0440(8)
H14A	0.3594	0.2776	0.0622	0.053*
H14B	0.3199	0.4343	0.0454	0.053*
C15	0.2330(3)	0.3116(4)	0.1732(3)	0.0663(11)
H15A	0.1453	0.2978	0.1429	0.099*
H15B	0.2156	0.3851	0.2015	0.099*
H15C	0.2640	0.2288	0.2249	0.099*
C16	0.6353(3)	0.6508(3)	0.2978(2)	0.0299(6)
C17	0.4941(3)	0.6953(3)	0.3119(2)	0.0358(7)
C18	0.5496(3)	0.6848(3)	0.4821(2)	0.0431(8)
C19	0.7267(3)	0.6063(3)	0.3799(2)	0.0358(7)
C20	0.3026(3)	0.7646(4)	0.4134(3)	0.0543(9)
H20A	0.2384	0.7338	0.3720	0.065*
H20B	0.2726	0.7274	0.4821	0.065*
C21	0.2896(4)	0.9174(4)	0.3835(3)	0.0765(12)
H21A	0.1929	0.9481	0.3960	0.115*
H21B	0.3102	0.9544	0.3138	0.115*
H21C	0.3573	0.9483	0.4216	0.115*
C22	0.7948(4)	0.5965(3)	0.5503(2)	0.0504(9)
H22A	0.7467	0.5698	0.6143	0.060*
H22B	0.8602	0.5200	0.5470	0.060*
C23	0.8788(4)	0.7179(4)	0.5417(3)	0.0727(12)
H23A	0.9470	0.6965	0.5960	0.109*
H23B	0.8138	0.7940	0.5439	0.109*
H23C	0.9298	0.7418	0.4796	0.109*
C24	1.2599(4)	0.9794(3)	0.0579(3)	0.0565(9)
H24A	1.3476	1.0031	0.0216	0.085*
H24B	1.2778	0.9629	0.1277	0.085*
H24C	1.1870	1.0535	0.0346	0.085*
H101	1.197(5)	0.685(4)	−0.013(3)	0.093(16)*
H104	0.401(4)	0.651(5)	0.176(3)	0.119(16)*

Source of material

The mixture of 1,3-diethyl-2-thioxodihydropyrimidine-4,6(1*H*, 5*H*)-dione (2 mmol), and 3-hydroxy-4-methoxybenzaldehyde (1 mmol) in 10 mL of degassed EtOH was stirred at room temperature for 3–5 h until TLC showed complete disappearance of the reactants. The product precipitated and the mixture was filtered and the white solid portion was recrystallized from EtOH.

(92% yield); M.p. 185–187 °C; ¹H NMR (Joel400; DMSO-*d*₆, 400 MHz) δ: 8.33 (brs, 3H, OH), 6.89 (d, 1H, *J* = 7.32 Hz,

C₆H₃), 6.81 (s, 1H, C₆H₃), 6.75 (d, 1H, *J* = 7.32 Hz, C₆H₃), 5.94 (s, 1H, CHC₆H₃), 4.21 (q, 8H, *J* = 12.33 Hz, 4*CH₂ CH₃), 3.90 (s, 3H, OCH₃), 1.20 (m, 12H, 4*CH₂CH₃); ¹³C NMR (DMSO-*d*₆, 100 MHz) δ: 174.5, 61.7, 134.5, 127.6, 113.3, 96.5, 55.8, 42.0, 31.7, 12.5; IR (KBr, cm^{−1}) ν_{max} = 3455, 2978, 2923, 2870, 1720, 1668, 1590, 1455, 1252; Anal. Calcd. for C₂₄H₃₀N₄O₆S₂: C, 53.92; H, 5.66; N, 10.48; S, 11.99; Found: C, 53.90; H, 5.67; N, 10.51; S, 12.01; LC/MS (ESI, *m/z*): [M + H]⁺, found 535.05, C₂₄H₃₀N₄O₆S₂ for 534.05.

Comment

The barbiturates and thiobarbiturates have been processed for biological applications including sedation and anaesthesia. They were also shown to be used as anxiolytics, hypnotics, anti-depressants, anticonvulsants, and to have anti-diabetic activity [1–8]. We describe the reactivity of thiobarbituric acid derivatives and 3-hydroxy-4-methoxybenzaldehyde in EtOH to give the title compound.

The molecular structure of the title compound is built up by thioxodihydropyrimidine rings (C8/C9/N1/C10/N2/C11) and (C16/C17/N3/C18/N4/C19) and a phenyl ring (C1–C6) which are connected together through the benzyl carbon C7. The dihedral angles between these rings are 60.24°, 64.91° and 65.95°, respectively. The bond lengths of the double bonds C8–C9 and C16–C17 are 1.386(4) and 1.387(4) Å, respectively.

Acknowledgements: The authors would like to extend their sincere appreciation to the Deanship of Scientific Research at Princess Nourah Bint Abdulrahman University for providing funding to the research.

References

- Hardman, J. G.; Limbird, L. E., eds.: Goodman and Gilman's The Pharmacological Basis of Therapeutics, 10th ed. Mc-Graw Hill Co., New York, NY, USA (2001).
- Moussier, N.; Bruché, L.; Viani, F.; Zanda, M.: Fluorinated barbituric acid derivatives: Synthesis and bio-activity. *Curr. Org. Chem.* **7** (2003) 1071–1080.
- Al-Majid, A. M.; Barakat, A.; Al-Najjar, H. J.; Mabkhot, Y. N.; Ghabbour, H. A.; Fun, H. K.: Tandem Aldol-Michael reactions in aqueous diethylamine medium: a greener and efficient approach to bis-pyrimidine derivatives. *Int. J. Mol. Sci.* **14** (2013) 23762–23773.
- Bojarski, J. T.; Mokrosz, J. L.; Bartoń, H. J.; Paluchowska, M. H.: Recent progress in barbituric acid chemistry. *Adv. Heterocycl. Chem.* **38** (1985) 229–297.
- Taylor, J. B.: Modern Medical Chemistry, Prentice Hall, New York, NY, USA (1994).
- Barakat, A.; Al-Majid, A. M.; Al-Najjar, H. J.; Mabkhot, Y. N.; Javaid, S.; Yousuf, S.; Choudhary, M. I.: Zwitterionic pyrimidinium adducts as antioxidants with therapeutic

- potential as nitric oxide scavenger. *Eur. J. Med. Chem.* **84** (2014) 146–154.
7. Barakat, A.; Soliman, S. M.; Al-Majid, A. M.; Lotfy, G.; Ghabbour, H. A.; Fun, H. K.; Yousuf, S.; Choudhary, M. I.; Wadood, A.: Synthesis and structure investigation of novel pyrimidine-2,4,6-trione derivatives of highly potential biological activity as anti-diabetic agent. *J. Mol. Struct.* **1098** (2015) 365–376.
 8. Barakat, A.; Ali, M.; Al Majid, A. M.; Yousuf, S.; Choudhary, M. I.: Diethylammonium salts of phenyl-substituted thiobarbituric acid as anti-diabetic agents. U.S. Patent 9,527,820 (2016).
 9. Sheldrick, G. M.: A short history of SHELX. *Acta Cryst.* **A64** (2008) 112–122.
 10. Bruker. SAINT, APEX2 and SADABS. Bruker AXS Inc., Madison, WI, USA (2009).