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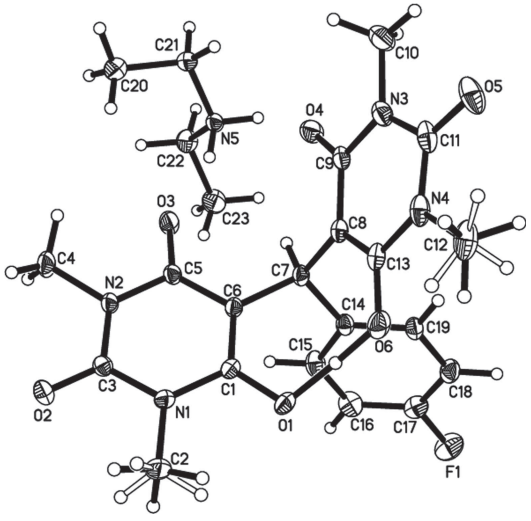
Assem Barakat*, Hazem A. Ghabbour, Abdullah Mohammed Al-Majid, El Sayed H. El Ashry, Ismail Warad and Hoong-Kun Fun

Crystal structure of diethylammonium 5-((4-fluorophenyl)(6-hydroxy-1,3-dimethyl-2,4-dioxo-1,2,3,4-tetrahydropyrimidin-5-yl)methyl)-1,3-dimethyl-2,6-dioxo-1,2,3,6-tetrahydropyrimidin-4-olate, $C_{23}H_{30}FN_5O_6$



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

Hazem A. Ghabbour: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P. O. Box 2457, Riaydh 11451, Saudi Arabia; and Department of Medicinal Chemistry, Faculty of Pharmacy, University of Mansoura, Mansoura 35516, Egypt

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

El Sayed H. El Ashry: Department of Chemistry, Faculty of Science, Alexandria University, P. O. Box 426, Ibrahimia, Alexandria 21321, Egypt

Ismail Warad: Department of Chemistry, Science College, Al-Najah National University, P. O. Box 7, Nablus, Palestine

Hoong-Kun Fun: Department of Pharmaceutical Chemistry, College of Pharmacy, King Saud University, P. O. Box 2457, Riaydh 11451, Saudi Arabia; and X-Ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, Penang 11800, Malaysia

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Abstract

$C_{23}H_{30}FN_5O_6$, monoclinic, $P2_1/n$ (no. 14), $a = 9.5772(3)$ Å, $b = 15.9466(5)$ Å, $c = 15.8380(5)$ Å, $\beta = 103.951(1)°$, $V = 2347.49(13)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0423$, $wR(F^2) = 0.192$, $T = 150$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Violet, block, size 0.417×0.512×0.74 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.07 cm^{-1}
Diffractometer, scan mode:	D8 Venture area detector, φ and ω scans
$2\theta_{max}$:	60°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	44751, 6815
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 6046
$N(param)_{refined}$:	359
Programs:	BRUKER programs [11], SHELXL [12]

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

Atom	Site	x	y	z	U_{iso}
H(2A)[a]	4*e*	0.6870	0.5790	0.8747	0.053
H(2B)[a]	4*e*	0.7986	0.6473	0.9254	0.053
H(2C)[a]	4*e*	0.8557	0.5588	0.8999	0.053
H(2Y)[b]	4*e*	0.860(4)	0.627(2)	0.928(2)	0.029(9)
H(2X)[b]	4*e*	0.696(4)	0.617(2)	0.893(2)	0.026(8)
H(2Z)[b]	4*e*	0.806(4)	0.540(2)	0.885(2)	0.029(9)

Table 2 (continued)

Atom	Site	x	y	z	U_{iso}
H(4A)	4e	1.0721	0.6542	0.6219	0.052
H(4B)	4e	1.1332	0.6096	0.7139	0.052
H(4C)	4e	1.1431	0.7093	0.7057	0.052
H(7A)	4e	0.6059	0.7580	0.5462	0.022
H(10A)	4e	0.5181	0.5761	0.2935	0.052
H(10B)	4e	0.3956	0.6460	0.2660	0.052
H(10C)	4e	0.3530	0.5488	0.2615	0.052
H(12A)[a]	4e	0.2069	0.4972	0.5837	0.059
H(12B)[a]	4e	0.2503	0.4263	0.5232	0.059
H(12C)[a]	4e	0.1157	0.4862	0.4854	0.059
H(12Y)[b]	4e	0.132(4)	0.514(2)	0.534(2)	0.027(9)
H(12X)[b]	4e	0.195(4)	0.435(2)	0.482(2)	0.032(9)
H(12Z)[b]	4e	0.267(4)	0.465(2)	0.589(2)	0.030(9)
H(15A)	4e	0.6364	0.8539	0.6914	0.034
H(16A)	4e	0.5036	0.9575	0.7416	0.037
H(18A)	4e	0.1349	0.8464	0.6100	0.033
H(19A)	4e	0.2697	0.7443	0.5578	0.028
H(20A)	4e	1.0304	0.6543	0.3725	0.053
H(20B)	4e	0.9952	0.6782	0.4633	0.053
H(20C)	4e	1.0781	0.7436	0.4162	0.053
H(21A)	4e	0.8735	0.7656	0.3055	0.030
H(21B)	4e	0.7904	0.6913	0.3406	0.030
H(22A)	4e	0.8827	0.9086	0.3783	0.034
H(22B)	4e	0.9873	0.8649	0.4609	0.034
H(23A)	4e	0.8761	0.9848	0.5044	0.053
H(23B)	4e	0.8295	0.9023	0.5483	0.053
H(23C)	4e	0.7214	0.9438	0.4659	0.053
H(2N5)	4e	0.799(2)	0.773(1)	0.466(1)	0.041(4)
H(1N5)	4e	0.710(2)	0.813(1)	0.385(1)	0.029(4)
H(1O1)	4e	0.456(3)	0.623(2)	0.690(2)	0.075(7)

[a]Disordered, occupancy factor: 0.54; [b]Disordered, occupancy factor: 0.46.

Source of material

The chemical reagents and solvents used in this study are commercially available. The synthesis of the title compound follows a known procedure [1].

Yield: 93%; m.p. 148 °C; ^{1}H-NMR (400 MHz, $CDCl_3$): δ 17.65 (1*H*, s, OH), 7.33 (2*H*,d, $J = 8.8$ Hz, C_6H_4), 7.01 (2*H*, d, $J = 8.8$ Hz, C_6H_4), 5.80(1*H*, s, C_6H_4CH), 3.35 (12*H*, s, $4CH_3$), 3.08 (4*H*,q, $J = 7.3$ Hz, CH_2CH_3), 1.30 (6*H*, t, $J = 7.3$ Hz, CH_2CH_3); ^{13}C-NMR (100 MHz, $CDCl_3$): $\delta = 165.4, 164.5, 151.6, 141.0, 131.2, 128.5, 119.5, 91.6, 42.0, 34.3, 29.2, 29.1, 12.0$.

Experimental details

All hydrogen atoms were placed geometrically on calculated positions using a riding model with the help of the SHELXL program (AFIX 137 option for methyl groups, AFIX 43 option for aromatic H atoms, AFIX 13 for tertiary and AFIX 23 for secondary H atoms) [12]. The hydrogen atoms of two methyl groups (bonded to C2 and C12) are disordered.

Discussion

1,3-Dimethylbarbituric acid skeleton represents the key pharmacophore of several pharmaceutically agents. Thus, several substituted 1,3-dimethylbarbituric acid were reported to exhibit marked chemotherapeutic activities such as antioxidant, anti-inflammatory, HIV-1 and HIV-2 protease inhibitors, anticancer, anticonvulsant and sedative-hypnotic [2–5]. In continues of our research program we report the crystal structure of a target molecule in this class of compounds. This contribution is part of our continuing interest in pyrimidin-based compounds [6–10]. The title compound comprise of a pyrimidine dione anion and a diethylammonium cation.

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
F(1)	4e	0.23009(9)	0.96747(5)	0.71192(6)	0.0476(5)	0.0315(4)	0.0527(5)	0.0133(3)	0.0212(4)	−0.0017(3)
O(1)	4e	0.55322(8)	0.64718(5)	0.75387(5)	0.0192(3)	0.0384(4)	0.0268(4)	0.0014(3)	0.0117(3)	0.0041(3)
O(2)	4e	1.03810(8)	0.61625(5)	0.84023(5)	0.0210(3)	0.0275(4)	0.0240(4)	0.0004(3)	−0.0006(3)	0.0034(3)
O(3)	4e	0.85762(8)	0.72953(6)	0.57420(5)	0.0184(3)	0.0486(5)	0.0236(4)	−0.0038(3)	0.0055(3)	0.0123(3)
O(4)	4e	0.56414(9)	0.70153(6)	0.40686(5)	0.0275(4)	0.0395(5)	0.0249(4)	−0.0107(3)	0.0063(3)	−0.0015(3)
O(5)	4e	0.2233(1)	0.49798(6)	0.35577(7)	0.0320(4)	0.0265(4)	0.0509(6)	−0.0054(3)	−0.0099(4)	−0.0060(4)
O(6)	4e	0.37235(9)	0.59100(5)	0.63272(6)	0.0218(4)	0.0325(4)	0.0358(4)	−0.0052(3)	0.0099(3)	0.0060(3)
N(1)	4e	0.79666(9)	0.63785(6)	0.79821(5)	0.0209(4)	0.0266(4)	0.0173(4)	0.0028(3)	0.0066(3)	0.0024(3)
N(2)	4e	0.94376(8)	0.66817(6)	0.70476(5)	0.0134(3)	0.0263(4)	0.0193(4)	−0.0019(3)	0.0029(3)	0.0015(3)
N(3)	4e	0.4043(1)	0.59500(6)	0.38363(6)	0.0217(4)	0.0249(4)	0.0282(4)	−0.0004(3)	−0.0006(3)	−0.0045(3)
N(4)	4e	0.3074(1)	0.54311(6)	0.49507(7)	0.0166(4)	0.0218(4)	0.0431(5)	−0.0046(3)	0.0028(4)	−0.0001(4)
C(1)	4e	0.6742(1)	0.65966(6)	0.73433(6)	0.0175(4)	0.0224(4)	0.0211(4)	0.0010(3)	0.0070(3)	−0.0001(3)
C(2)	4e	0.7834(1)	0.6029(1)	0.88144(8)	0.0329(6)	0.0541(8)	0.0219(5)	0.0074(5)	0.0121(4)	0.0121(5)
C(3)	4e	0.9325(1)	0.63983(6)	0.78432(6)	0.0189(4)	0.0187(4)	0.0193(4)	−0.0010(3)	0.0031(3)	−0.0008(3)
C(4)	4e	1.0847(1)	0.6596(1)	0.68490(8)	0.0144(4)	0.0602(8)	0.0300(5)	0.0017(5)	0.0061(4)	0.0129(5)
C(5)	4e	0.8283(1)	0.69950(6)	0.64071(6)	0.0155(4)	0.0230(4)	0.0186(4)	−0.0030(3)	0.0026(3)	0.0010(3)
C(6)	4e	0.6893(1)	0.69390(6)	0.65639(6)	0.0145(4)	0.0208(4)	0.0195(4)	−0.0006(3)	0.0038(3)	0.0014(3)
C(7)	4e	0.5613(1)	0.72419(6)	0.58597(6)	0.0135(4)	0.0208(4)	0.0210(4)	−0.0024(3)	0.0030(3)	0.0019(3)

Table 3 (continued)

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(8)	4*e*	0.4779(1)	0.65625(6)	0.52746(7)	0.0139(4)	0.0211(4)	0.0255(4)	−0.0021(3)	0.0034(3)	−0.0003(3)
C(9)	4*e*	0.4879(1)	0.65460(7)	0.43886(7)	0.0151(4)	0.0247(5)	0.0256(5)	−0.0007(3)	0.0005(3)	−0.0014(4)
C(10)	4*e*	0.4190(1)	0.59115(8)	0.29371(8)	0.0354(6)	0.0370(6)	0.0271(5)	0.0014(5)	−0.0026(4)	−0.0060(5)
C(11)	4*e*	0.3061(1)	0.54267(7)	0.40778(8)	0.0186(4)	0.0196(4)	0.0418(6)	0.0014(3)	−0.0036(4)	−0.0023(4)
C(12)	4*e*	0.2121(1)	0.48317(8)	0.5243(1)	0.0229(5)	0.0276(6)	0.0662(9)	−0.0098(4)	0.0093(5)	0.0028(6)
C(13)	4*e*	0.3905(1)	0.59852(6)	0.55434(7)	0.0137(4)	0.0210(4)	0.0337(5)	−0.0002(3)	0.0041(4)	0.0017(4)
C(14)	4*e*	0.4675(1)	0.78744(6)	0.61996(6)	0.0174(4)	0.0198(4)	0.0221(4)	0.0000(3)	0.0031(3)	0.0036(3)
C(15)	4*e*	0.5343(1)	0.85209(7)	0.67452(8)	0.0219(5)	0.0219(5)	0.0373(6)	−0.0011(4)	0.0020(4)	−0.0012(4)
C(16)	4*e*	0.4564(1)	0.91374(7)	0.70495(8)	0.0350(6)	0.0203(5)	0.0354(6)	0.0008(4)	0.0045(5)	−0.0012(4)
C(17)	4*e*	0.3085(1)	0.90934(7)	0.68026(8)	0.0339(6)	0.0224(5)	0.0312(5)	0.0087(4)	0.0114(4)	0.0059(4)
C(18)	4*e*	0.2371(1)	0.84770(7)	0.62630(8)	0.0216(5)	0.0301(5)	0.0309(5)	0.0065(4)	0.0056(4)	0.0050(4)
C(19)	4*e*	0.3179(1)	0.78688(7)	0.59581(7)	0.0172(4)	0.0261(5)	0.0243(5)	0.0011(3)	0.0017(3)	0.0020(4)
N(5)	4*e*	0.80259(9)	0.79860(6)	0.41309(6)	0.0164(4)	0.0259(4)	0.0186(4)	−0.0030(3)	0.0036(3)	0.0004(3)
C(20)	4*e*	1.0038(1)	0.70002(9)	0.40696(8)	0.0319(6)	0.0446(7)	0.0289(5)	0.0094(5)	0.0073(4)	−0.0020(5)
C(21)	4*e*	0.8609(1)	0.73713(7)	0.35873(7)	0.0228(5)	0.0331(5)	0.0208(4)	−0.0011(4)	0.0062(4)	−0.0029(4)
C(22)	4*e*	0.8856(1)	0.87782(7)	0.43296(8)	0.0254(5)	0.0272(5)	0.0326(5)	−0.0073(4)	0.0076(4)	−0.0007(4)
C(23)	4*e*	0.8226(1)	0.93196(8)	0.49320(9)	0.0340(6)	0.0317(6)	0.0394(6)	−0.0046(5)	0.0059(5)	−0.0103(5)

All rings in the anion (N1/N2/C1/C3/C5/C6, N3/N4/C8—C9/C11/C13 and C14—C19) are planar. The dihedral angle between two pyridmidine rings was found to be 68.34(2)°. The phenyl ring appears to be twisted with an angles of 77.15(3)° and 65.75(2)° with respect to the planes of two pyrimidine rings (N1/N2/C1/C3/C5/C6 and N3/N4/C8—C9/C11/C13), respectively. Cations and anions are connected by NH···O hydrogen bonds to form a chain.

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