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Crystal structure of diethylammonium 1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-ide, $C_{10}H_{19}N_3O_3$

Tables 1 and 2 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:	Pink, blocks
Wavelength:	Size $0.55 \times 0.41 \times 0.16$ mm
μ :	Mo K_{α} radiation (0.71073 Å)
Diffractometer, scan mode:	1.0 cm^{-1}
$2\theta_{\text{max}}$, completeness:	Broker APEX-II, φ and ω
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	66.4°, >99%
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	25648, 4436, 0.055
$N(\text{param})_{\text{refined}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 3160
Programs:	157
	SHELX [13], Bruker programs [14]

DOI 10.1515/ncrs-2016-0051

Received February 10, 2016; accepted June 7, 2016; available online June 30, 2016

Abstract

$C_{10}H_{19}N_3O_3$, monoclinic, $P2_1/n$, $a = 4.4714(2)$ Å, $b = 17.8690(7)$ Å, $c = 14.6323(6)$ Å, $\beta = 95.272(1)^\circ$, $V = 1164.17(8)$ Å³, $Z = 4$, $R_{\text{gt}}(F) = 0.0518$, $wR_{\text{ref}}(F^2) = 0.1370$, $T = 100$ K.

CCDC no.: 1452473

Source of material

A mixture of (1.5 mmol) of 1,3-dimethylpyrimidine-2,4,6(1H,3H,5H)-trione and (1.5 mmol, 155 μL) of Et_2NH in H_2O (3 mL) was stirred at r.t. for 30 mins. The target compound was precipitated and filtered. The solid portion was recrystallized from $\text{CH}_2\text{Cl}_2/\text{EtOH}/\text{Et}_2\text{O}$ over 24 h, to get cuboid colourless crystals of the pure product.

Experimental details

Carbon-bound H atoms were placed in calculated positions and were included in the refinement in the riding model approximation. The H atoms of the methyl groups were allowed to rotate with a fixed angle around the C—C bond to best fit the experimental electron density [1].

Discussion

Barbituric acid consists of active methylene group and can act as excellent nucleophile. In the meanwhile, barbituric acid has been found to be associated with important biological activities including anti-hypertensive, anti-inflammatory, antitumor, anticonvulsant and hypnotic drugs [1–12]. We have succeeded to separate the active species which can act as nucleophile generated by aqueous diethylamine medium.

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

Atom	x	y	z	U _{iso} [*] /U _{eq}
O1	0.5693(2)	0.63954(5)	0.55115(6)	0.01686(18)
O2	1.01732(19)	0.65228(5)	0.85558(6)	0.01701(19)
O3	0.3361(2)	0.47036(5)	0.76703(6)	0.0207(2)
N1	0.6862(2)	0.55934(5)	0.80938(6)	0.01324(19)
N2	0.4494(2)	0.55573(5)	0.65952(6)	0.01191(18)
N3	0.2275(2)	0.74202(6)	0.45099(7)	0.01223(19)
C1	0.6092(2)	0.61877(6)	0.63375(8)	0.0120(2)
C2	0.8023(3)	0.65257(6)	0.70195(8)	0.0137(2)
H2A	0.9080	0.6964	0.6872	0.016*
C3	0.8456(2)	0.62417(6)	0.79107(8)	0.0121(2)
C4	0.7332(4)	0.52439(8)	0.89953(9)	0.0250(3)
H4A	0.7562	0.4702	0.8923	0.038*
H4B	0.5599	0.5345	0.9341	0.038*
H4C	0.9149	0.5450	0.9328	0.038*
C5	0.4807(2)	0.52529(6)	0.74650(8)	0.0130(2)
C6	0.2295(3)	0.52080(7)	0.59247(9)	0.0170(2)
H6A	0.0643	0.5001	0.6241	0.025*
H6B	0.3264	0.4805	0.5606	0.025*
H6C	0.1506	0.5584	0.5478	0.025*
C7	0.2712(3)	0.82170(7)	0.58976(9)	0.0209(3)
H7A	0.1579	0.8534	0.6290	0.031*
H7B	0.4233	0.8519	0.5627	0.031*
H7C	0.3699	0.7814	0.6266	0.031*
H8A	−0.0921	0.7567	0.5411	0.019*
H8B	−0.0490	0.8289	0.4787	0.019*
C9	0.0283(3)	0.69953(7)	0.38160(8)	0.0158(2)
H9A	−0.0979	0.7351	0.3431	0.019*
H9B	−0.1066	0.6665	0.4134	0.019*
C10	0.2127(3)	0.65281(7)	0.32121(9)	0.0188(2)
H10A	0.0782	0.6232	0.2787	0.028*
H10B	0.3439	0.6192	0.3596	0.028*
H10C	0.3353	0.6858	0.2863	0.028*
H2N3	0.345(3)	0.7742(9)	0.4209(10)	0.016(4)*
H1N3	0.347(4)	0.7072(10)	0.4851(12)	0.030(5)*

The asymmetric unit is composed of one diethylammonium cation and one 1,3-dimethyl-2,4,6-trioxohexahydropyrimidin-5-ide anion (*cf.* the figure). The molecules packing in the crystal structure is stabilized by intermolecular hydrogen bonds, in which O1, and O2 work act as hydrogen bond acceptors and N3 as hydrogen bond donor. The distances of the interactions between N3—H1N3...O1 and N3—H2N3...O2ⁱ are 1.838 Å and 1.882 Å. (Symmetry code: (i) $x-1/2, -y+3/2, z-1/2$).

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 (RGP-257-1435-1436).

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