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

Abstract

C_{10}H_{19}N_{3}O_{3}, monoclinic, P2_1/n, a = 4.4714(2) Å, b = 17.8690(7) Å, c = 14.6323(6) Å, β = 95.272(1)^o, V = 1164.17(8) Å^3, Z = 4, R_{gt}(F) = 0.0518, wR_{ref}(F^2) = 0.1370, T = 100 K.

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Table 1: Data collection and handling.

| Crystal: | Pink, blocks |
| Wavelength: | Mo Kα radiation (0.71073 Å) |
| μ: | 1.0 cm\(^{-1}\) |
| Diffractometer, scan mode: | Bruker APEX-II, φ and ω |
| 2θ_{max}, completeness: | 66.4°, >99% |
| N(hkl) measured, N(hkl) unique, R_{int}: | 25648, 4436, 0.055 |
| Criterion for I_{obs}, N(hkl)gt: | I_{obs} > 2 σ(I_{obs}), 3160 |
| N(param)refined: | 157 |
| Programs: | SHELX [13], Bruker programs [14] |

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 Et₂NH in H₂O (3 mL) was stirred at r.t. for 30 mins. The target compound was precipitated and filtered. The solid portion was recrystallized from CH₂Cl₂/EtOH/Et₂O 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.
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 as hydrogen bond acceptors and N3 as hydrogen bond donor. The distances of the interactions between N3—H1N3···O1 and N3—H2N3···O2 are 1.838 Å and 1.882 Å. (Symmetry code: (i) x−1/2, −y+3/2, z−1/2).

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References:


