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Crystal structure of dimethyl (2*E*)-2-[(*tert*-butylamino)(1,3-dimethyl-2,4,6-trioxotetrahydropyrimidin-5(2*H*)-ylidene)methyl]but-2-enedioate, $C_{17}H_{23}N_3O_7$

A. Zonouzi*[I], H. Rahmani[II] and H. Samareh Afsari[I]

[I] University of Tehran, Department of Chemistry, P.O. Box 14155-6455, Tehran, Iran
[II] Iranian Research Organization for Science and Technology (IROST), Institute of Chemical Technologies, P.O. Box 15815-3538, Tehran, Iran

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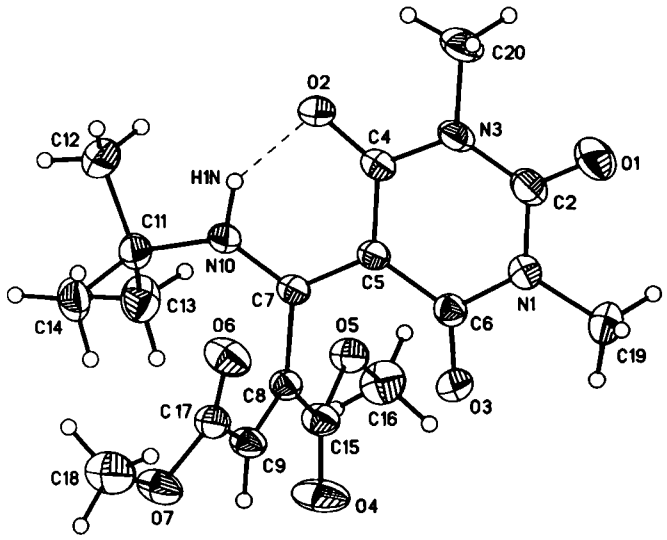

Abstract

$C_{17}H_{23}N_3O_7$, monoclinic, $P12_1/c1$ (no. 14), a = 15.231(3) Å, b = 15.641(3) Å, c = 7.800(2) Å, β = 97.53(3)°, V = 1842.2 Å^3, Z = 4, $R_{gt}(F)$ = 0.040, $wR_{ref}(F^2)$ = 0.115, T = 193 K.

Source of material

The title compound was obtained from the reaction of 1,3-dimethylpyrimidine-2,4,6-trione, *t*-butyl isocyanide and dimethyl acetylene dicarboxylate in cold dichloromethane. The yellow solution was refluxed and purified by column chromatography on alumina using a mixture of ethyl acetate and hexane (20:80, *v*/*v*) as eluent. The colorless solution was allowed to concentrate slowly at room temperature. Recrystallization from methanol gave crystals suitable for X-ray structure analysis.

Discussion

A large number of pyrimidine derivatives are reported to exhibit antimycobacterial, antitumor, antiviral, anticancer, anti-inflammatory, analgesic, antifolate, antimicrobial, anti-fungal, antiproliferative and antihistaminic activities [1]. There are some new reports on crystal structures of pyrimidine derivatives [2-4]. The crystal structure analysis of the title compound was performed in view of its good stability.

In the crystal structure, the molecule is stabilized by one intramolecular N–H···O hydrogen bond. Details of the N10–H1N···O2 bond are: d(N10···O2) = 2.577 Å, d(N10—H1N) = 0.91 Å, d(H1N···O2) = 1.79 Å, ∠N10–H1N···O2 = 142°. The molecules in unit cell are interlinked by van der Waals forces.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.20 × 0.20 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.08 cm^{-1}
Diffractometer, scan mode:	Rebuild Syntex P2$_1$/Siemens P3, $\omega/2\theta$
$2\theta_{max}$:	56°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4756, 4438
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 3088
$N(param)_{refined}$:	251
Programs:	SHELXS-97 [5], SHELXL-97 [6]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1N)	4*e*	0.2093	0.6393	0.3700	0.035
H(9A)	4*e*	0.4408	0.4524	0.2048	0.034
H(12A)	4*e*	0.2052	0.6461	0.6646	0.070
H(12B)	4*e*	0.2568	0.7230	0.5963	0.070
H(12C)	4*e*	0.2954	0.6760	0.7672	0.070
H(13A)	4*e*	0.2661	0.4953	0.6429	0.065
H(13B)	4*e*	0.3511	0.5293	0.7578	0.065
H(13C)	4*e*	0.3594	0.4842	0.5811	0.065
H(14A)	4*e*	0.3939	0.6947	0.4537	0.062
H(14B)	4*e*	0.4366	0.6034	0.4733	0.062
H(14C)	4*e*	0.4331	0.6609	0.6370	0.062
H(16A)	4*e*	0.1465	0.3014	0.4513	0.062
H(16B)	4*e*	0.2470	0.2846	0.5141	0.062
H(16C)	4*e*	0.2066	0.2603	0.3252	0.062
H(18A)	4*e*	0.6190	0.6534	0.1011	0.078
H(18B)	4*e*	0.5489	0.7024	0.1942	0.078
H(18C)	4*e*	0.5294	0.6831	−0.0044	0.078
H(19A)	4*e*	0.0988	0.4665	−0.4068	0.065
H(19B)	4*e*	0.0307	0.4170	−0.3097	0.065
H(19C)	4*e*	0.1315	0.3931	−0.2770	0.065
H(20A)	4*e*	−0.0688	0.6763	−0.0595	0.062
H(20B)	4*e*	−0.0023	0.7327	−0.1476	0.062
H(20C)	4*e*	0.0014	0.7327	0.0542	0.062

* Correspondence author (e-mail: zonouzi@khayam.ut.ac.ir)

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(1)	4e	−0.01022(8)	0.57960(9)	−0.2971(2)	0.0399(6)	0.0775(9)	0.0411(7)	0.0172(6)	−0.0136(5)	−0.0074(6)
O(2)	4e	0.11606(6)	0.67540(6)	0.2230(1)	0.0298(5)	0.0296(5)	0.0402(6)	0.0099(4)	−0.0008(4)	−0.0051(4)
O(3)	4e	0.21808(7)	0.42464(6)	−0.0243(1)	0.0346(5)	0.0290(5)	0.0396(6)	0.0059(4)	0.0017(4)	−0.0069(4)
O(4)	4e	0.35496(7)	0.33344(7)	0.2937(2)	0.0396(6)	0.0240(5)	0.0880(9)	0.0101(5)	0.0235(6)	0.0146(6)
O(5)	4e	0.22622(6)	0.38482(6)	0.3584(1)	0.0272(5)	0.0231(5)	0.0425(6)	−0.0037(4)	0.0094(4)	0.0023(4)
O(6)	4e	0.38104(7)	0.63886(6)	0.0847(2)	0.0317(5)	0.0286(5)	0.0610(7)	0.0017(4)	0.0053(5)	0.0141(5)
N(1)	4e	0.10315(8)	0.50131(8)	−0.1591(2)	0.0261(6)	0.0373(7)	0.0316(6)	0.0011(5)	0.0008(5)	−0.0055(5)
C(2)	4e	0.04542(9)	0.5702(1)	−0.1724(2)	0.0252(7)	0.0464(9)	0.0328(8)	0.0027(6)	0.0013(6)	0.0006(7)
N(3)	4e	0.05359(7)	0.62701(8)	−0.0368(2)	0.0224(5)	0.0366(6)	0.0317(6)	0.0084(5)	0.0005(5)	0.0027(5)
C(4)	4e	0.11719(8)	0.62122(8)	0.1074(2)	0.0205(6)	0.0266(7)	0.0310(7)	0.0012(5)	0.0035(5)	0.0029(6)
C(5)	4e	0.18040(8)	0.55290(8)	0.1108(2)	0.0201(6)	0.0212(6)	0.0297(7)	0.0006(5)	0.0037(5)	0.0005(5)
C(6)	4e	0.17141(9)	0.48854(8)	−0.0226(2)	0.0232(6)	0.0273(6)	0.0292(7)	−0.0013(5)	0.0043(5)	0.0003(5)
O(7)	4e	0.51661(6)	0.58127(6)	0.1423(2)	0.0236(5)	0.0255(5)	0.0741(8)	−0.0037(4)	0.0109(5)	0.0029(5)
C(7)	4e	0.25000(8)	0.54701(8)	0.2489(2)	0.0198(6)	0.0174(5)	0.0309(7)	−0.0005(5)	0.0041(5)	0.0029(5)
C(8)	4e	0.32100(8)	0.48092(8)	0.2448(2)	0.0224(6)	0.0197(6)	0.0301(7)	0.0009(5)	0.0024(5)	0.0004(5)
C(9)	4e	0.40014(9)	0.49706(8)	0.1977(2)	0.0227(6)	0.0206(6)	0.0407(8)	0.0020(5)	0.0039(5)	0.0012(6)
N(10)	4e	0.25392(7)	0.60005(7)	0.3812(2)	0.0236(5)	0.0233(5)	0.0319(6)	0.0048(4)	−0.0011(5)	−0.0018(5)
C(11)	4e	0.31244(9)	0.60687(9)	0.5497(2)	0.0303(7)	0.0284(7)	0.0332(7)	0.0018(5)	−0.0042(6)	−0.0036(6)
C(12)	4e	0.2629(1)	0.6687(1)	0.6541(2)	0.049(1)	0.051(1)	0.0395(9)	0.0094(8)	−0.0021(7)	−0.0154(8)
C(13)	4e	0.3233(1)	0.5210(1)	0.6413(2)	0.053(1)	0.0386(8)	0.0351(8)	0.0003(7)	−0.0072(7)	0.0047(7)
C(14)	4e	0.4024(1)	0.6450(1)	0.5262(2)	0.0334(8)	0.0367(8)	0.051(1)	−0.0071(6)	−0.0067(7)	−0.0061(7)
C(15)	4e	0.30383(9)	0.39113(8)	0.2999(2)	0.0261(7)	0.0216(6)	0.0405(8)	0.0009(5)	0.0059(6)	0.0033(6)
C(16)	4e	0.2048(1)	0.30075(9)	0.4172(2)	0.0458(9)	0.0282(7)	0.053(1)	−0.0100(7)	0.0152(7)	0.0043(7)
C(17)	4e	0.42862(9)	0.58045(9)	0.1347(2)	0.0251(6)	0.0235(6)	0.0382(8)	−0.0017(5)	0.0056(6)	0.0000(6)
C(18)	4e	0.5569(1)	0.6618(1)	0.1052(3)	0.0358(9)	0.0295(8)	0.094(2)	−0.0122(7)	0.0209(9)	0.0001(9)
C(19)	4e	0.0899(1)	0.4391(1)	−0.3003(2)	0.0402(9)	0.050(1)	0.0389(9)	−0.0033(7)	−0.0003(7)	−0.0156(8)
C(20)	4e	−0.0095(1)	0.6983(1)	−0.0484(2)	0.0314(8)	0.0441(9)	0.0459(9)	0.0155(7)	−0.0012(7)	0.0048(7)

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