Crystal structure of dimethyl 7-(cyclohexylamino)2,3,4,5-tetrahydro-1,3-dimethyl-2,4-dioxo-1*H*-pyrano[2,3-*d*]pyrimidine-5,6-dicarboxylate, $C_{19}H_{25}N_3O_7$

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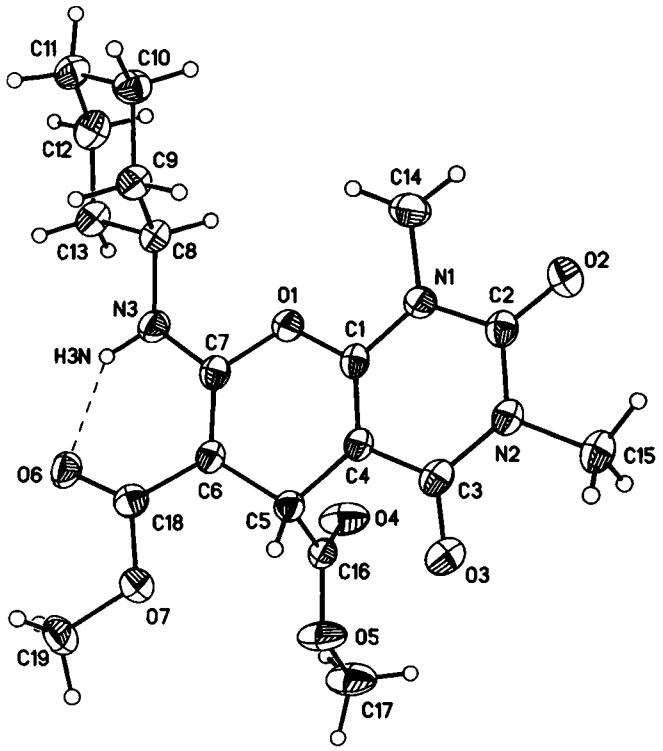

Abstract

$C_{19}H_{25}N_3O_7$, triclinic, $P\bar{1}$ (no. 2), a = 9.304(1) Å, b = 10.844(1) Å, c = 11.033(1) Å, α = 109.574(3)°, β = 102.575(3)°, γ = 102.419(3)°, V = 972.2 Å^3, Z = 2, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.101, T = 120 K.

Source of material

The title compound was obtained from the reaction of 1,3-dimethylpyrimidine-2,4,6-trion, cyclohexyl isocyanide and dimethyl acetylene dicarboxylate in hot toluene, then purified by column chromatography on silica gel using a mixture of ethyl acetate and hexane (40:60, *v*/*v*) as eluent. Recrystallization from ethanol 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 the crystal structures of pyrimidine derivatives [2,3]. Recently we have reported the crystal structure of a pyrimidine derivative [4].

The molecule in the title crystal structure is stabilized by one intramolecular N–H···O type of hydrogen bonding: d(N3—H3N) = 0.90 Å, d(H3N···O6) = 1.97 Å, d(N3···O6) = 2.706 Å, ∠N3–H3N···O6 = 138°. The molecules in the unit cell are interlinked by van der Waals forces.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.10 × 0.15 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.07 cm^{-1}
Diffractometer, scan mode:	Bruker SMART 1000 CCD, φ/ω
$2\theta_{max}$:	52°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7743, 3762
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2250
$N(param)_{refined}$:	266
Program:	SHELXTL [5]

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

Atom	Site	x	y	z	U_{iso}
H(3N)	2*i*	0.7683	−0.0554	0.1433	0.035
H(5A)	2*i*	0.4548	−0.2377	0.3415	0.029
H(8A)	2*i*	1.0130	0.1533	0.3622	0.031
H(9A)	2*i*	0.8174	0.2618	0.3625	0.034
H(9B)	2*i*	0.7874	0.2315	0.2051	0.034
H(10A)	2*i*	1.0682	0.4128	0.4195	0.038
H(10B)	2*i*	0.9467	0.4616	0.3346	0.038
H(11A)	2*i*	1.0119	0.3631	0.1380	0.041
H(11B)	2*i*	1.1691	0.4618	0.2577	0.041
H(12A)	2*i*	1.2259	0.2731	0.2903	0.043
H(12B)	2*i*	1.1944	0.2443	0.1332	0.043
H(13A)	2*i*	0.9417	0.0976	0.0786	0.036
H(13B)	2*i*	1.0636	0.0449	0.1582	0.036
H(14A)	2*i*	0.9178	0.2821	0.6169	0.062
H(14B)	2*i*	1.0426	0.2271	0.6896	0.062
H(14C)	2*i*	0.9600	0.3216	0.7765	0.062
H(15A)	2*i*	0.6421	0.0756	0.9346	0.049
H(15B)	2*i*	0.6391	−0.0818	0.8790	0.049
H(15C)	2*i*	0.4868	−0.0430	0.8291	0.049
H(17A)	2*i*	0.4780	−0.6421	0.3041	0.072
H(17B)	2*i*	0.6064	−0.5428	0.4470	0.072
H(17C)	2*i*	0.6484	−0.5660	0.3095	0.072
H(19A)	2*i*	0.2631	−0.5163	−0.0402	0.064
H(19B)	2*i*	0.4061	−0.4631	−0.0880	0.064
H(19C)	2*i*	0.2821	−0.3817	−0.0728	0.064

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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)	2*i*	0.8234(2)	0.0408(2)	0.4458(2)	0.029(1)	0.0272(9)	0.0252(9)	0.0068(7)	0.0108(8)	0.0111(7)
O(2)	2*i*	0.8417(2)	0.2038(2)	0.8879(2)	0.045(1)	0.034(1)	0.027(1)	0.0100(9)	0.0109(9)	0.0076(8)
O(3)	2*i*	0.4505(2)	−0.1894(2)	0.5936(2)	0.035(1)	0.0320(9)	0.039(1)	0.0114(8)	0.0173(9)	0.0183(8)
O(4)	2*i*	0.7647(2)	−0.3140(2)	0.4174(2)	0.030(1)	0.033(1)	0.075(2)	0.0108(8)	0.010(1)	0.028(1)
O(5)	2*i*	0.5175(2)	−0.4410(2)	0.3436(2)	0.029(1)	0.0245(9)	0.068(1)	0.0054(8)	0.007(1)	0.0226(9)
O(6)	2*i*	0.5882(2)	−0.2208(2)	0.0324(2)	0.053(1)	0.0285(9)	0.027(1)	0.0128(9)	0.0147(9)	0.0120(8)
O(7)	2*i*	0.4291(2)	−0.3501(2)	0.1037(2)	0.037(1)	0.0294(9)	0.027(1)	0.0057(8)	0.0053(8)	0.0096(8)
N(1)	2*i*	0.8213(2)	0.1253(2)	0.6634(2)	0.030(1)	0.023(1)	0.025(1)	0.0079(9)	0.0096(9)	0.0087(9)
N(2)	2*i*	0.6498(2)	0.0033(2)	0.7424(2)	0.029(1)	0.028(1)	0.026(1)	0.0129(9)	0.0116(9)	0.0146(9)
N(3)	2*i*	0.8161(2)	0.0003(2)	0.2314(2)	0.037(1)	0.026(1)	0.026(1)	0.0092(9)	0.014(1)	0.0117(9)
C(1)	2*i*	0.7535(3)	0.0204(2)	0.5373(2)	0.028(1)	0.026(1)	0.022(1)	0.015(1)	0.011(1)	0.012(1)
C(2)	2*i*	0.7749(3)	0.1166(2)	0.7732(2)	0.032(2)	0.028(1)	0.026(1)	0.014(1)	0.011(1)	0.013(1)
C(3)	2*i*	0.5683(3)	−0.0978(2)	0.6127(2)	0.030(1)	0.027(1)	0.031(1)	0.016(1)	0.013(1)	0.017(1)
C(4)	2*i*	0.6327(3)	−0.0891(2)	0.5076(2)	0.025(1)	0.024(1)	0.022(1)	0.010(1)	0.008(1)	0.012(1)
C(5)	2*i*	0.5707(3)	−0.2062(2)	0.3708(2)	0.024(1)	0.026(1)	0.024(1)	0.007(1)	0.007(1)	0.012(1)
C(6)	2*i*	0.6254(3)	−0.1604(2)	0.2691(2)	0.029(1)	0.024(1)	0.023(1)	0.012(1)	0.011(1)	0.012(1)
C(7)	2*i*	0.7496(3)	−0.0447(2)	0.3109(2)	0.028(1)	0.028(1)	0.026(1)	0.014(1)	0.009(1)	0.014(1)
C(8)	2*i*	0.9360(3)	0.1335(2)	0.2738(2)	0.028(1)	0.028(1)	0.027(1)	0.010(1)	0.011(1)	0.014(1)
C(9)	2*i*	0.8676(3)	0.2521(2)	0.2908(2)	0.032(2)	0.028(1)	0.030(1)	0.012(1)	0.014(1)	0.013(1)
C(10)	2*i*	0.9943(3)	0.3874(2)	0.3290(3)	0.036(2)	0.027(1)	0.034(2)	0.011(1)	0.015(1)	0.010(1)
C(11)	2*i*	1.0823(3)	0.3757(3)	0.2256(3)	0.034(2)	0.033(1)	0.039(2)	0.007(1)	0.015(1)	0.018(1)
C(12)	2*i*	1.1453(3)	0.2545(3)	0.2055(3)	0.034(2)	0.045(2)	0.040(2)	0.017(1)	0.021(1)	0.022(1)
C(13)	2*i*	1.0179(3)	0.1206(2)	0.1673(2)	0.032(2)	0.033(1)	0.034(1)	0.017(1)	0.018(1)	0.017(1)
C(14)	2*i*	0.9458(3)	0.2494(2)	0.6887(3)	0.047(2)	0.029(1)	0.039(2)	0.001(1)	0.016(1)	0.007(1)
C(15)	2*i*	0.6003(3)	−0.0128(3)	0.8558(2)	0.039(2)	0.038(1)	0.032(2)	0.019(1)	0.018(1)	0.019(1)
C(16)	2*i*	0.6302(3)	−0.3251(2)	0.3808(2)	0.027(1)	0.024(1)	0.023(1)	0.008(1)	0.009(1)	0.011(1)
C(17)	2*i*	0.5666(3)	−0.5575(2)	0.3517(3)	0.044(2)	0.024(1)	0.074(2)	0.010(1)	0.009(2)	0.024(1)
C(18)	2*i*	0.5511(3)	−0.2421(2)	0.1262(2)	0.033(2)	0.023(1)	0.031(1)	0.014(1)	0.009(1)	0.012(1)
C(19)	2*i*	0.3378(3)	−0.4346(3)	−0.0354(3)	0.049(2)	0.034(2)	0.031(2)	0.006(1)	0.002(1)	0.005(1)

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