Crystal structure of 2-acetonyl-5-benzyl-4,6-dimethoxy-5-nitro-2,5-dihydropyrimidine, $C_{16}H_{19}N_3O_5$

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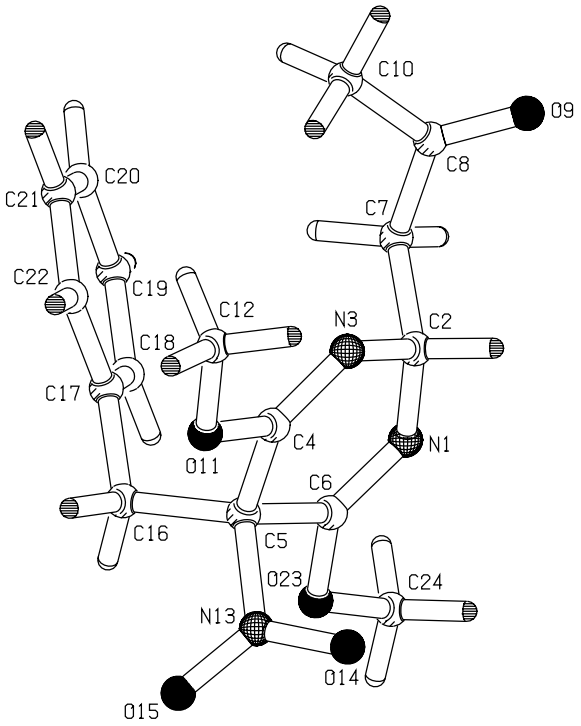

Abstract

$C_{16}H_{19}N_3O_5$, monoclinic, $P12_1/c1$ (No. 14), a = 12.010(1) Å, b = 11.877(1) Å, c = 12.110(1) Å, β = 95.83(2)°, V = 1718.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.044, $wR_{ref}(F^2)$ = 0.129, T = 289 K.

Source of material

The compound was synthesised and crystallised by G. Y. Remennikov [1].

Experimental details

Hydrogen atoms were refined in the riding mode with a fixed isotropic temperature factor 1.2 times *Ueq* of the parent atom.

Discussion

Dihydropyrimidines, already know for more than 100 years, are of major interest because they exhibit a diverse range of biological activities. Some derivatives such as monastrol, block mitosis by inhibiting the motor activity of the mitotic kinesin Eg_5, required for chromosome migration and are used as anticancer drug [2]. Some 5-nitrodihydropyrimidines also show interesting antimicrobial and antiviral activity, others are highly reactive compounds [1]. The activity and reactivity of dihydropyrimidines is strongly related to the stereochemical and conformational structure of the molecule. The inactive, but stable derivative 2-acetonyl-5-benzyl-4,6-dimethoxy-5-nitro-2,5-dihydropyrimidine crystallises with four molecules in the unit cell. On the basis of the bond distances, the 5-nitro-4,5-dihydropyrimidine can be considered as a cyclic imine showing no conjugation. The dihydropyrimidine ring is almost planar with a maximum deviation for C5 of 0.070(2) Å to the best plane through the six-membered ring. This plane makes an angle of 54.1(1)° with the phenyl ring and of 91.4(1)° with the nitro group. The orientation of the nitro group is further described by torsion angle C16–C5–N13–O15 (2.6(3)°). Both methoxy groups are situated close to the plane of the dihydropyrimidine ring (maximum deviation of –0.223(2) Å for C24) resulting in minimal interactions with the 5-benzyl and 5-nitro groups. The distance between the centroids of both six-membered rings is 3.790(2) Å.

Table 1. Data collection and handling.

Crystal:	yellow block, size 0.20 × 0.25 × 0.35 mm
Wavelength:	Cu K_α radiation (1.54178 Å)
μ:	8.12 cm^{-1}
Diffractometer, scan mode:	Siemens P4-PC, $\theta/2\theta$
$2\theta_{max}$:	100.86°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3560, 1785
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1523
$N(param)_{refined}$:	221
Programs:	SHELXS-97 [3], SHELXL-97 [4], PLATON [5]

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

Atom	Site	x	y	z	U_{iso}
H(2A)	4*e*	0.6298	–0.1940	0.6181	0.050
H(7A)	4*e*	0.5297	–0.1739	0.7753	0.060
H(7B)	4*e*	0.5977	–0.0637	0.8067	0.060
H(10A)	4*e*	0.4325	0.1100	0.6195	0.108
H(10B)	4*e*	0.5631	0.1011	0.6408	0.108
H(10C)	4*e*	0.4908	0.1091	0.7413	0.108
H(12A)	4*e*	0.8821	0.1763	0.5487	0.067
H(12B)	4*e*	0.7788	0.1670	0.6176	0.067
H(12C)	4*e*	0.7866	0.0896	0.5138	0.067
H(16A)	4*e*	0.9781	–0.1015	0.9192	0.064
H(16B)	4*e*	0.9807	0.0104	0.8506	0.064
H(18A)	4*e*	0.8166	–0.1416	1.0220	0.073
H(19A)	4*e*	0.6674	–0.0725	1.1063	0.098
H(20A)	4*e*	0.5912	0.0992	1.0559	0.099
H(21A)	4*e*	0.6713	0.2091	0.9282	0.089
H(22A)	4*e*	0.8242	0.1427	0.8464	0.071
H(24A)	4*e*	0.8946	–0.4440	0.9163	0.074
H(24B)	4*e*	0.8095	–0.4352	0.8095	0.074
H(24C)	4*e*	0.7811	–0.3797	0.9206	0.074

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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}
N(1)	4*e*	0.7263(2)	–0.2386(2)	0.7519(2)	0.032(1)	0.040(1)	0.056(1)	0.0003(9)	0.0052(9)	–0.0010(9)
C(2)	4*e*	0.6641(2)	–0.1544(2)	0.6839(2)	0.027(1)	0.044(1)	0.053(1)	–0.002(1)	–0.004(1)	–0.004(1)
N(3)	4*e*	0.7238(2)	–0.0594(2)	0.6437(1)	0.027(1)	0.046(1)	0.047(1)	0.0001(9)	–0.0009(9)	0.0002(9)
C(4)	4*e*	0.8249(2)	–0.0450(2)	0.6777(2)	0.030(2)	0.042(1)	0.045(1)	0.000(1)	0.007(1)	–0.001(1)
C(5)	4*e*	0.8942(2)	–0.1171(2)	0.7614(2)	0.021(1)	0.049(1)	0.051(1)	0.002(1)	0.002(1)	0.004(1)
C(6)	4*e*	0.8279(2)	–0.2210(2)	0.7835(2)	0.030(2)	0.041(1)	0.046(1)	0.005(1)	0.002(1)	0.002(1)
C(7)	4*e*	0.5679(2)	–0.1107(2)	0.7451(2)	0.030(1)	0.052(2)	0.069(2)	–0.002(1)	0.008(1)	0.002(1)
C(8)	4*e*	0.4852(2)	–0.0436(2)	0.6701(3)	0.028(2)	0.062(2)	0.099(2)	0.002(1)	0.003(2)	–0.002(2)
O(9)	4*e*	0.4160(2)	–0.0920(2)	0.6102(3)	0.081(2)	0.098(2)	0.193(3)	–0.013(2)	–0.071(2)	0.009(2)
C(10)	4*e*	0.4936(3)	0.0799(3)	0.6677(3)	0.064(2)	0.073(2)	0.133(3)	0.028(2)	0.011(2)	0.006(2)
O(11)	4*e*	0.8869(1)	0.0414(1)	0.6457(1)	0.0321(9)	0.049(1)	0.069(1)	–0.0045(8)	0.0000(8)	0.0165(8)
C(12)	4*e*	0.8287(2)	0.1257(2)	0.5756(2)	0.051(2)	0.046(2)	0.069(2)	–0.001(1)	0.004(1)	0.015(1)
N(13)	4*e*	0.9959(2)	–0.1592(2)	0.7049(2)	0.033(1)	0.059(2)	0.073(2)	0.005(1)	0.008(1)	0.015(1)
O(14)	4*e*	0.9746(2)	–0.2093(2)	0.6181(2)	0.054(1)	0.106(2)	0.080(1)	0.017(1)	0.020(1)	–0.009(1)
O(15)	4*e*	1.0892(2)	–0.1419(2)	0.7493(2)	0.027(1)	0.097(2)	0.120(2)	0.003(1)	0.001(1)	0.014(1)
C(16)	4*e*	0.9336(2)	–0.0520(2)	0.8686(2)	0.037(2)	0.056(2)	0.063(2)	–0.007(1)	–0.013(1)	0.003(1)
C(17)	4*e*	0.8359(2)	–0.0073(2)	0.9249(2)	0.044(2)	0.050(2)	0.049(1)	–0.009(1)	–0.010(1)	–0.008(1)
C(18)	4*e*	0.7880(2)	–0.0707(2)	1.0029(2)	0.072(2)	0.056(2)	0.054(2)	–0.003(2)	0.001(2)	0.005(1)
C(19)	4*e*	0.6980(3)	–0.0297(3)	1.0526(2)	0.099(3)	0.082(2)	0.066(2)	–0.003(2)	0.025(2)	–0.005(2)
C(20)	4*e*	0.6534(3)	0.0732(3)	1.0237(3)	0.084(2)	0.091(3)	0.076(2)	0.007(2)	0.024(2)	–0.020(2)
C(21)	4*e*	0.7006(3)	0.1384(2)	0.9470(3)	0.088(2)	0.054(2)	0.078(2)	0.011(2)	–0.003(2)	–0.022(2)
C(22)	4*e*	0.7920(2)	0.0984(2)	0.8979(2)	0.069(2)	0.047(2)	0.060(2)	–0.010(1)	–0.005(1)	–0.007(1)
O(23)	4*e*	0.8925(1)	–0.2942(1)	0.8454(1)	0.0361(9)	0.049(1)	0.070(1)	0.0031(8)	0.0023(8)	0.0177(8)
C(24)	4*e*	0.8401(2)	–0.3968(2)	0.8754(2)	0.056(2)	0.052(2)	0.078(2)	0.005(1)	0.012(1)	0.023(1)

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