Crystal structure of 1,3,5-triacetyl-2,4-dioxohexahydro-1,3,5-triazine, $(CH_3CO)_3C_3H_2N_3O_2$

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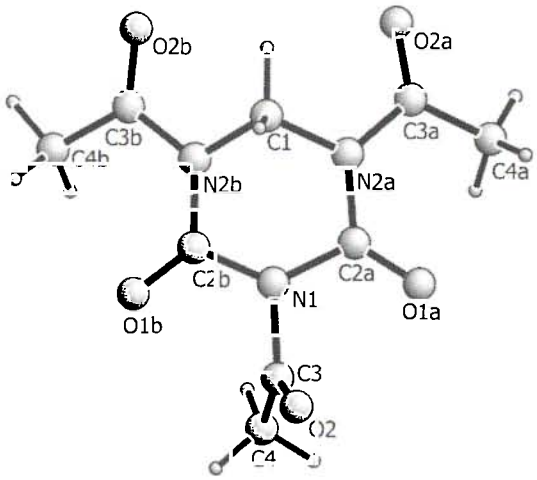

Abstract

$C_9H_{11}N_3O_5$, monoclinic, $P12_1/c1$ (No. 14), $a = 9.135(3)$ Å, $b = 14.269(1)$ Å, $c = 9.318(4)$ Å, $\beta = 118.84(2)°$, $V = 1064.0$ Å^3, $Z = 4$, $R_{gt}(F) = 0.035$, $wR(F) = 0.033$, $T = 294$ K.

Source of material

1,3,5-triacetyl-2,4-dioxohexahydro-1,3,5-triazine can be synthesized by reacting 1,5-diacetyl-2,4-dioxohexahydro-1,3,5-triazine in with ketene in acetanhydride [1].

Discussion

The 2,4-dioxohexahydro-1,3,5-triazine ring adopts a boat conformation with C1 and N1 offset from the basal plane by 0.591(2) Å and 0.218(2) Å, respectively. The V-shape of the molecule is determined by the planes N2a–(C2a)–C3a–(O2a)–C4a and N2b–(C2b)–C3b–(O2b)–C4b with C1 as their common intersection point (dihedral angle 53.44(5)°). The two planes intersect the basal plane asymmetrically with small dihedral angles of 30.80(5)° and 22.66(5)°, whereas the plane defined by C4–C3–(O2)–N1 is almost perpendicular to it (dihedral angle 70.8(1)°). In the crystal structure the molecules are piled above another along [001]. The resulting columns are arranged in a distorted hexagonal rod packing.

Table 1. Data collection and handling.

Crystal:	colourless, prismatic, size 0.35 × 0.40 × 0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	1.17 cm^{-1}
Diffractometer, scan mode:	Enraf-Nonius CAD4, θ/2θ
$2\theta_{max}$:	51.92°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2094, 1968
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 3\ \sigma(I_{obs})$, 1281
$N(param)_{refined}$:	162
Programs:	MolEN [2], ATOMS [3]

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

Atom	Site	x	y	z	U_{iso}
H(41)	4e	0.6754	0.1810	0.7507	0.10(1)
H(42)	4e	0.7557	0.2467	0.6753	0.10(2)
H(43)	4e	0.7601	0.1390	0.6555	0.10(2)
H(4a1)	4e	0.1427	0.4671	0.6836	0.101(9)
H(4a2)	4e	0.1663	0.4663	0.5322	0.088(9)
H(4a3)	4e	0.3067	0.4244	0.6938	0.101(9)
H(4b1)	4e	0.2344	–0.0719	0.8403	0.065(8)
H(4b2)	4e	0.3026	–0.0787	0.7164	0.063(8)
H(4b3)	4e	0.3922	–0.0068	0.8563	0.067(8)
H(11)	4e	–0.0100	0.1832	0.5331	0.051(7)
H(12)	4e	0.0517	0.1627	0.3878	0.039(6)

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}
C(1)	4e	0.0871(2)	0.1799(2)	0.5005(3)	0.034(1)	0.040(1)	0.040(1)	–0.002(1)	0.0169(7)	0.000(1)
C(2a)	4e	0.3086(2)	0.2775(2)	0.5162(2)	0.034(1)	0.042(1)	0.0292(9)	0.003(1)	0.0144(7)	0.005(1)
C(2b)	4e	0.3560(3)	0.1084(2)	0.5921(3)	0.040(1)	0.040(1)	0.045(1)	0.001(1)	0.0248(7)	0.001(1)
O(1a)	4e	0.3686(2)	0.3478(1)	0.4978(2)	0.0508(7)	0.040(1)	0.0622(8)	–0.0014(8)	0.0354(5)	0.0110(8)
O(1b)	4e	0.4551(2)	0.0450(1)	0.6336(2)	0.0581(8)	0.047(1)	0.104(1)	0.0168(8)	0.0551(6)	0.022(1)
N(1)	4e	0.3809(2)	0.1901(1)	0.5233(2)	0.0339(7)	0.037(1)	0.0403(8)	0.0009(8)	0.0247(5)	0.0022(8)
N(2a)	4e	0.1662(2)	0.2709(1)	0.5324(2)	0.0290(7)	0.037(1)	0.0406(8)	0.0013(8)	0.0192(5)	0.0027(9)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
N(2b)	4*e*	0.2092(2)	0.1092(1)	0.5996(2)	0.0303(7)	0.037(1)	0.0401(8)	–0.0016(8)	0.0217(5)	0.0009(9)
C(3)	4*e*	0.5348(2)	0.1943(2)	0.5073(3)	0.042(1)	0.040(1)	0.046(1)	–0.004(1)	0.0304(7)	–0.004(1)
C(3a)	4*e*	0.0997(3)	0.3441(2)	0.5836(3)	0.038(1)	0.047(2)	0.037(1)	0.006(1)	0.0196(7)	0.005(1)
C(3b)	4*e*	0.1631(3)	0.0423(2)	0.6823(2)	0.043(1)	0.037(1)	0.039(1)	–0.009(1)	0.0248(7)	–0.007(1)
O(2)	4*e*	0.5168(2)	0.2044(2)	0.3738(2)	0.0624(8)	0.099(2)	0.0480(7)	–0.013(1)	0.0396(5)	–0.003(1)
O(2a)	4*e*	–0.0254(2)	0.3289(1)	0.5917(2)	0.0529(7)	0.065(1)	0.0849(9)	–0.0035(8)	0.0485(5)	–0.0133(9)
O(2b)	4*e*	0.0268(2)	0.0498(1)	0.6712(2)	0.0457(7)	0.063(1)	0.0674(8)	–0.0022(8)	0.0380(5)	0.0109(9)
C(4)	4*e*	0.6960(3)	0.1899(2)	0.6609(3)	0.029(1)	0.079(2)	0.058(1)	0.000(1)	0.0177(9)	0.008(1)
C(4a)	4*e*	0.1875(3)	0.4356(2)	0.6295(3)	0.052(1)	0.045(2)	0.064(1)	0.004(1)	0.0317(9)	–0.002(1)
C(4b)	4*e*	0.2817(3)	–0.0328(2)	0.7761(3)	0.059(1)	0.038(1)	0.055(1)	0.002(1)	0.0340(8)	0.006(1)

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