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Crystal structure of hexahydro-3-[(phenylmethyl)amino]-2*H*-azepin-2-one, $C_{13}H_{18}N_2O$

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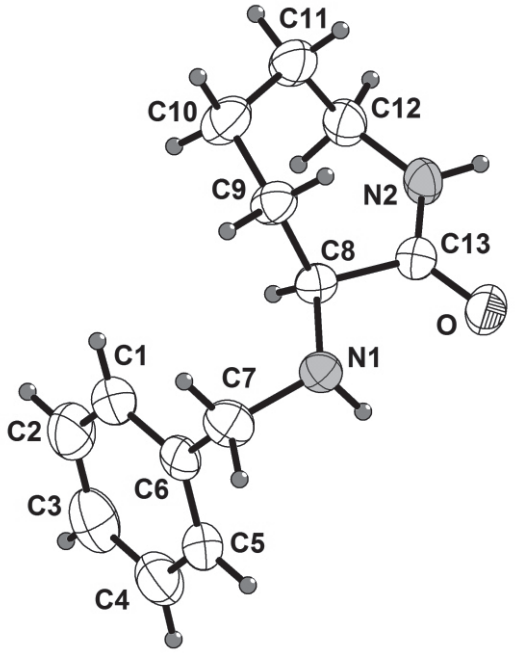

Abstract

$C_{13}H_{18}N_2O$, orthorhombic, *Pbca* (no. 61), a = 9.933(2) Å, b = 9.941(2) Å, c = 23.754(5) Å, V = 2345.6 Å^3, Z = 8, $R_{gt}(F)$ = 0.0680, $wR_{ref}(F^2)$ = 0.1217, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless blocks, size 0.10×0.20×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.79 cm^{-1}
Diffractometer, scan mode:	Nonius cad4, $\omega/2\theta$
$2\theta_{max}$:	50.74°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2162, 2142
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1001
$N(param)_{refined}$:	145
Programs:	DIAMOND [5], SHELX [6]

Source of material

The title compound was obtained from the reduction of the intermedia prepared by 3-aminoazepan-2-one hydrochloride and benzoyl chloride as described in literature [1, 2] and recrystallized from CH_2Cl_2-petroleum ether (bp 40-60 °C) to give the desired crystals suitable for single-crystal X-ray diffraction.

Experimental details

H atoms were positioned geometrically with C–H = 0.93, 0.97 and 0.98 Å for aromatic, methylene and methyne, respectively, and constrained to ride on their parent atoms, with U_{iso}(H) = 1.2 (or 1.5 for methyl groups) times U_{eq}(C).

Discussion

Hexahydro-3-[(phenylmethyl)amino]-2*H*-azepin-2-one is one of 3-(acylamino)azepan-2-ones which are broad spectrum chemokine inhibitors and act as stable, orally available powerful antiinflammatory agents [5, 6]. The asymmetric unit of the title structure consists of one complete molecule. All bond lengths and angles are in the expected ranges. A dihedral angle of 171.4(3)° is found for C12–N2–C13–O. Weak intermolecular N–H···O hydrogen bonds link adjacent molecules pairwise to centrosymmetric dimers.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	8*c*	0.4492	0.0557	0.4474	0.046
H(1B)	8*c*	0.5187	0.1826	0.2923	0.070
H(2A)	8*c*	0.7216	0.3401	0.5176	0.052
H(2B)	8*c*	0.6854	0.0922	0.2364	0.091
H(3A)	8*c*	0.7372	–0.1330	0.2439	0.090
H(4A)	8*c*	0.6233	–0.2675	0.3068	0.080
H(5A)	8*c*	0.4645	–0.1743	0.3652	0.062
H(7A)	8*c*	0.3256	0.1468	0.3530	0.062
H(7B)	8*c*	0.3002	0.0053	0.3795	0.062
H(8A)	8*c*	0.5758	0.2131	0.3956	0.042
H(9A)	8*c*	0.3652	0.3455	0.3873	0.055
H(9B)	8*c*	0.4034	0.4013	0.4468	0.055
H(10A)	8*c*	0.4683	0.5491	0.3753	0.070
H(10B)	8*c*	0.5620	0.4383	0.3497	0.070
H(11A)	8*c*	0.6930	0.5898	0.3994	0.067
H(11B)	8*c*	0.6056	0.5694	0.4536	0.067
H(12A)	8*c*	0.7592	0.3595	0.4084	0.059
H(12B)	8*c*	0.8191	0.4577	0.4533	0.059

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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	8*c*	0.5616(2)	0.1616(2)	0.52009(9)	0.060(2)	0.059(2)	0.042(1)	–0.006(1)	–0.004(1)	0.010(1)
N(1)	8*c*	0.4123(2)	0.1245(2)	0.4283(1)	0.033(2)	0.040(2)	0.042(2)	–0.004(1)	–0.001(1)	0.000(1)
C(1)	8*c*	0.5408(4)	0.0921(4)	0.2959(1)	0.075(3)	0.055(2)	0.044(2)	–0.009(2)	0.000(2)	–0.002(2)
N(2)	8*c*	0.6849(3)	0.3305(3)	0.4850(1)	0.042(2)	0.052(2)	0.036(2)	–0.006(2)	–0.009(1)	0.002(2)
C(2)	8*c*	0.6398(5)	0.0382(4)	0.2621(2)	0.097(4)	0.085(3)	0.046(3)	–0.032(3)	0.015(2)	0.001(3)
C(3)	8*c*	0.6705(4)	–0.0960(5)	0.2666(2)	0.074(3)	0.100(4)	0.052(3)	–0.002(3)	0.006(2)	–0.015(3)
C(4)	8*c*	0.6036(4)	–0.1762(4)	0.3044(2)	0.083(3)	0.063(3)	0.053(2)	0.014(3)	–0.011(2)	–0.013(2)
C(5)	8*c*	0.5077(4)	–0.1205(4)	0.3387(1)	0.063(3)	0.053(2)	0.038(2)	–0.005(2)	–0.007(2)	0.002(2)
C(6)	8*c*	0.4739(3)	0.0137(3)	0.3349(1)	0.036(2)	0.051(2)	0.040(2)	–0.012(2)	–0.012(2)	–0.001(2)
C(7)	8*c*	0.3685(3)	0.0733(3)	0.3730(1)	0.040(2)	0.055(2)	0.060(2)	–0.009(2)	–0.003(2)	–0.003(2)
C(8)	8*c*	0.5116(3)	0.2330(3)	0.4258(1)	0.029(2)	0.038(2)	0.040(2)	–0.003(2)	0.006(2)	0.001(2)
C(9)	8*c*	0.4403(3)	0.3651(3)	0.4121(1)	0.043(2)	0.041(2)	0.054(2)	0.000(2)	–0.009(2)	0.007(2)
C(10)	8*c*	0.5258(3)	0.4735(3)	0.3847(2)	0.059(3)	0.051(2)	0.067(2)	–0.002(2)	–0.011(2)	0.018(2)
C(11)	8*c*	0.6422(3)	0.5240(3)	0.4209(1)	0.058(3)	0.052(2)	0.057(2)	–0.014(2)	–0.010(2)	0.010(2)
C(12)	8*c*	0.7365(3)	0.4158(3)	0.4404(1)	0.046(2)	0.056(2)	0.045(2)	–0.015(2)	–0.007(2)	0.003(2)
C(13)	8*c*	0.5881(3)	0.2400(3)	0.4815(1)	0.034(2)	0.040(2)	0.038(2)	0.003(2)	0.000(2)	0.001(2)

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