Crystal structure of 3-(benzoylamino)azepan-2-one, $C_{13}H_{16}N_2O_2$

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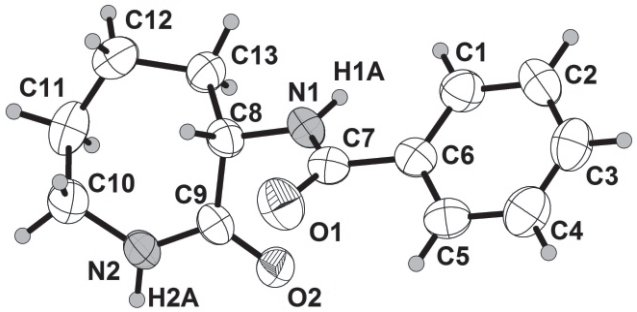

Abstract

$C_{13}H_{16}N_2O_2$, orthorhombic, $P2_12_12_1$ (no. 19), a = 7.488(2) Å, b = 9.633(2) Å, c = 16.933(3) Å, V = 1221.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.0671, $wR_{ref}(F^2)$ = 0.1661, T = 293 K.

Table 1. Data collection and handling.

Crystal:	colourless block, size 0.10×0.20×0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.86 cm^{-1}
Diffractometer, scan mode:	Nonius CAD4, $\omega/2\theta$
$2\theta_{max}$:	50.94°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	2554, 2244
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1413
$N(param)_{refined}$:	154
Programs:	SHELX [4]

Source of material

The title compound was obtained from the reaction of 3-aminoazepan-2-one hydrochloride and benzoyl chloride as described in literature [1] and recrystallized from CH_2Cl_2-petroleum ether (b.p. 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 and 0.97/0.98 Å for aromatic, and aliphatic groups, respectively, and constrained to ride on their parent atoms. U_{iso}(C) for CH_2 and CH groups were set to U_{iso}(H) = 1.2U_{eq}(C) and for methyl groups U_{iso}(H) = 1.5U_{eq}(C).

Discussion

3-(benzoylamino)azepan-2-one belongs to the compound class of 3-(acylamino)azepan-2-ones that are broad spectrum chemokine inhibitors and act as stable, orally available powerful antiinflammatory agents [2, 3]. In the crystal structure, intermolecular N–H···O hydrogen bonds link the molecules into chains, in which may be effective in the stabilization of the structure.

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*a*	–0.2490	0.6943	0.0963	0.052
H(1B)	4*a*	–0.4763	0.7975	0.1455	0.065
H(2A)	4*a*	0.3803	0.6473	0.0259	0.063
H(2B)	4*a*	–0.6990	0.9609	0.1629	0.073
H(3A)	4*a*	–0.6184	1.1877	0.1969	0.076
H(4A)	4*a*	–0.3239	1.2473	0.2124	0.078
H(5A)	4*a*	–0.1014	1.0789	0.1964	0.062
H(8A)	4*a*	0.0464	0.5838	0.1585	0.051
H(10A)	4*a*	0.3232	0.4718	0.1403	0.069
H(10B)	4*a*	0.4612	0.4470	0.0722	0.069
H(11A)	4*a*	0.2067	0.3651	–0.0061	0.074
H(11B)	4*a*	0.2793	0.2640	0.0587	0.074
H(12A)	4*a*	0.0517	0.3319	0.1428	0.071
H(12B)	4*a*	–0.0218	0.2631	0.0657	0.071
H(13A)	4*a*	–0.1973	0.4536	0.0948	0.060
H(13B)	4*a*	–0.0966	0.4832	0.0155	0.060

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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*a*	–0.1475(4)	0.7058(3)	0.1191(2)	0.034(2)	0.056(2)	0.041(2)	0.004(2)	0.000(2)	–0.001(2)
O(1)	4*a*	0.0170(4)	0.8309(3)	0.2049(2)	0.058(2)	0.079(2)	0.057(2)	0.000(2)	–0.017(2)	–0.012(2)
C(1)	4*a*	–0.4461(6)	0.8877(4)	0.1596(3)	0.056(3)	0.049(2)	0.058(3)	–0.003(2)	0.003(2)	–0.006(2)
O(2)	4*a*	0.1132(4)	0.7799(3)	0.0186(2)	0.046(2)	0.048(2)	0.050(2)	0.004(1)	0.002(1)	0.008(1)
N(2)	4*a*	0.2959(5)	0.6055(4)	0.0508(2)	0.035(2)	0.059(2)	0.062(2)	0.005(2)	0.006(2)	0.016(2)
C(2)	4*a*	–0.5796(6)	0.9849(5)	0.1695(3)	0.042(3)	0.076(3)	0.066(3)	0.006(2)	–0.001(2)	0.000(3)
C(3)	4*a*	–0.5304(7)	1.1208(5)	0.1899(3)	0.074(4)	0.054(3)	0.061(3)	0.017(3)	0.003(3)	–0.006(2)
C(4)	4*a*	–0.3546(8)	1.1565(5)	0.1995(3)	0.080(4)	0.056(3)	0.058(3)	0.004(3)	–0.002(3)	–0.003(2)
C(5)	4*a*	–0.2210(6)	1.0555(4)	0.1897(2)	0.058(3)	0.054(3)	0.043(3)	–0.008(2)	0.003(2)	0.000(2)
C(6)	4*a*	–0.2685(6)	0.9215(4)	0.1703(2)	0.047(3)	0.053(3)	0.030(2)	–0.001(2)	0.007(2)	0.000(2)
C(7)	4*a*	–0.1207(6)	0.8170(4)	0.1663(2)	0.045(3)	0.048(2)	0.036(2)	–0.005(2)	0.001(2)	–0.001(2)
C(8)	4*a*	–0.0054(5)	0.6059(4)	0.1069(2)	0.039(2)	0.051(2)	0.038(2)	0.003(2)	0.003(2)	0.004(2)
C(9)	4*a*	0.1417(5)	0.6709(4)	0.0559(2)	0.035(2)	0.052(2)	0.041(2)	0.003(2)	–0.004(2)	–0.004(2)
C(10)	4*a*	0.3373(6)	0.4686(4)	0.0834(3)	0.053(3)	0.060(3)	0.061(3)	0.006(2)	0.003(2)	0.011(2)
C(11)	4*a*	0.2193(7)	0.3520(5)	0.0504(3)	0.081(4)	0.049(3)	0.056(3)	0.018(2)	–0.004(3)	0.000(2)
C(12)	4*a*	0.0385(6)	0.3446(4)	0.0862(3)	0.068(3)	0.047(2)	0.062(3)	–0.006(2)	–0.001(2)	0.005(2)
C(13)	4*a*	–0.0807(5)	0.4711(4)	0.0719(3)	0.048(3)	0.043(2)	0.058(3)	–0.001(2)	–0.006(2)	0.002(2)

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