Crystal structure of 3-cyclohexyl-1,3-oxazinan-2-one, $C_{10}H_{17}NO_2$

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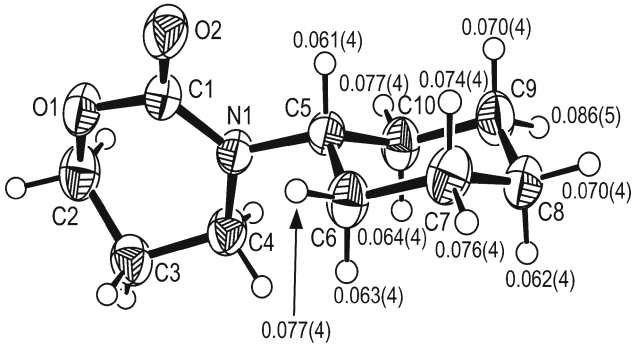

Abstract

$C_{10}H_{17}NO_2$, monoclinic, $P12_1/c1$ (no. 14), a = 10.187(1) Å, b = 9.8426(8) Å, c = 10.766(1) Å, β = 113.589(4)°, V = 989.3 Å^3, Z = 4, $R_{gt}(F)$ = 0.051, $wR_{ref}(F^2)$ = 0.143, T = 295 K.

Source of material

The title compound was synthesized from 1,3-dibromopropane, tetraethylammonium bicarbonate and cyclohexylamine by a one-pot procedure in MeOH as described previously [1]. Suitable single crystals were obtained by slow evaporation of MeOH at room temperature.

Experimental details

All hydrogen atoms were clearly identified in difference Fourier maps and refined including isotropic displacement parameters. In final stages of refinement, soft restraints were applied on C—H bonds grouped according to chemical equivalence.

Discussion

Cyclic urethanes are well known for their biological/pharmacological properties, like the title compound which is of current interest in the syntheses of pharmaceutical compounds and 1,3-amino alcohols [1-4]. Carbamate group π-electron delocalization makes the six-membered urethane ring rather flat (only C3 deviates significantly, by 0.72 Å, from least-squares plane defined by the carbamate group). Within the latter, the bond d(N1—C1) = 1.351(1) Å is much shorter than d(N1—C4) = 1.467(1) Å and d(N1—C5) = 1.474(1) Å, while d(O1—C1) = 1.359(2) Å is considerably shorter than d(O1—C2) = 1.439(2) Å. The cyclohexyl ring shows perfect chair conformation with the urethane substituent in equatorial position and with C1—N1 and C5—H51 bonds synclinal (torsion angle C1–N1–C5–H51 = –25.5(7)°). Molecules within the crystal structure are interconnected *via* hydrogen bonds C2–H21···O2′ (symmetry code: –x+1,y+½,–z+½), running along b axis. The distances of C2···O2′ and H21···O2′ are 3.50 Å and 2.64 Å, respectively, with an angle of ∠C2–H21···O2′ = 146°. Hydrogen atoms within the cyclohexyl moiety show a remarkable feature, i.e. atomic displacement parameters for axial positions are consistently smaller than those in equatorial positions. This is well in accordance with the generally accepted picture that substituents in axial positions are more repulsive. Since the urethane unit is more planar in the title molecule, respective differences among displacement parameters of hydrogen atoms need more careful evaluation. These very subtle details are even more remarkable since they are derived from data routinely collected at ambient conditions.

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.19 × 0.50 × 0.82 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.85 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC-7 with Saturn 724 CCD, profile data from φ-scans
$2\theta_{max}$:	59.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7566, 2846
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 2037
$N(param)_{refined}$:	188
Programs:	SHELXS-97, SHELXL-97 [5], ORTEP-3 [6]

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

Atom	Site	x	y	z	U_{iso}
H(21)	4*e*	0.361(2)	0.261(1)	0.202(1)	0.071(4)
H(22)	4*e*	0.249(1)	0.139(1)	0.117(1)	0.077(4)
H(31)	4*e*	0.337(2)	0.159(1)	–0.053(1)	0.073(4)
H(32)	4*e*	0.269(2)	0.304(1)	–0.032(2)	0.084(5)
H(41)	4*e*	0.498(1)	0.392(1)	0.089(1)	0.067(4)
H(42)	4*e*	0.508(1)	0.319(1)	–0.038(1)	0.070(4)
H(51)	4*e*	0.816(1)	0.192(1)	0.231(1)	0.061(4)
H(61)	4*e*	0.717(1)	0.205(1)	–0.055(1)	0.063(4)
H(62)	4*e*	0.759(2)	0.070(1)	0.031(2)	0.077(4)
H(71)	4*e*	0.999(1)	0.143(1)	0.133(1)	0.074(4)
H(72)	4*e*	0.953(2)	0.143(2)	–0.026(1)	0.076(4)
H(81)	4*e*	0.918(1)	0.382(1)	–0.026(1)	0.062(4)
H(82)	4*e*	1.076(1)	0.349(2)	0.074(1)	0.070(4)
H(91)	4*e*	1.007(1)	0.361(1)	0.259(1)	0.070(4)
H(92)	4*e*	0.967(2)	0.506(1)	0.176(2)	0.086(5)
H(101)	4*e*	0.729(1)	0.431(1)	0.069(1)	0.064(4)
H(102)	4*e*	0.769(2)	0.430(1)	0.226(1)	0.077(4)

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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*	0.60646(9)	0.21303(8)	0.13523(9)	0.0408(4)	0.0422(4)	0.0537(5)	–0.0009(3)	0.0209(4)	0.0068(4)
O(1)	4*e*	0.45090(8)	0.08548(8)	0.19870(9)	0.0519(5)	0.0551(5)	0.0752(6)	–0.0050(3)	0.0337(4)	0.0096(4)
O(2)	4*e*	0.67515(9)	0.02178(9)	0.2630(1)	0.0570(5)	0.0547(5)	0.0850(6)	0.0032(4)	0.0251(5)	0.0249(4)
C(1)	4*e*	0.5839(1)	0.1044(1)	0.2007(1)	0.0480(6)	0.0421(5)	0.0531(6)	–0.0056(4)	0.0232(5)	0.0015(4)
C(2)	4*e*	0.3417(1)	0.1863(1)	0.1370(2)	0.0530(7)	0.0607(7)	0.0870(9)	0.0001(6)	0.0388(7)	0.0022(7)
C(3)	4*e*	0.3477(1)	0.2375(1)	0.0094(2)	0.0443(6)	0.0578(7)	0.0752(8)	0.0024(5)	0.0195(6)	0.0083(6)
C(4)	4*e*	0.4926(1)	0.3021(1)	0.0454(1)	0.0477(6)	0.0494(6)	0.0662(7)	0.0021(5)	0.0220(5)	0.0123(5)
C(5)	4*e*	0.7537(1)	0.2355(1)	0.1461(1)	0.0411(5)	0.0439(5)	0.0483(5)	–0.0019(4)	0.0199(4)	0.0044(4)
C(6)	4*e*	0.7812(1)	0.1669(1)	0.0324(1)	0.0587(7)	0.0442(6)	0.0683(7)	–0.0072(5)	0.0345(6)	–0.0077(5)
C(7)	4*e*	0.9357(1)	0.1874(1)	0.0490(1)	0.0616(7)	0.0550(6)	0.0733(8)	0.0023(5)	0.0410(6)	0.0021(6)
C(8)	4*e*	0.9752(1)	0.3366(1)	0.0602(1)	0.0512(6)	0.0583(7)	0.0654(7)	–0.0057(5)	0.0322(6)	0.0044(5)
C(9)	4*e*	0.9464(1)	0.4050(1)	0.1729(1)	0.0587(7)	0.0650(8)	0.0671(8)	–0.0216(6)	0.0316(6)	–0.0128(6)
C(10)	4*e*	0.7911(1)	0.3865(1)	0.1539(1)	0.0587(7)	0.0477(6)	0.0673(7)	–0.0120(5)	0.0351(6)	–0.0125(5)

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