Crystal structure of 1-(4-chlorobenzyloxy)-3-phenylurea, $C_{14}H_{13}ClN_2O_2$

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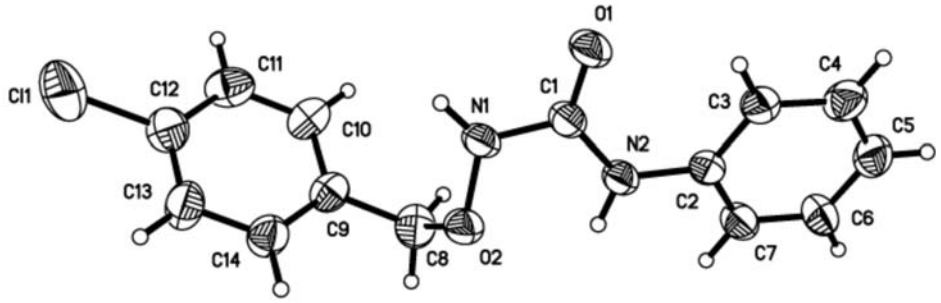

Abstract

$C_{14}H_{13}ClN_2O_2$, monoclinic, $P12_1/c1$ (no. 14), a = 5.3549(4) Å, b = 15.555(1) Å, c = 16.561(2) Å, β = 95.759(1)°, V = 1372.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.049, $wR_{ref}(F^2)$ = 0.149, T = 298 K.

Source of material

Synthesis of the title compound was accomplished by reaction of aniline (6 mmol) with 4-nitrophenyl-4-chlorobenzyloxy-carbamate (6.5 mmol) in anhydrous dichloromethane (40 mL) in the presence of anhydrous triethylamine (0.5 mL). The reaction mixture was stirred for 12 h, then washed with 1 M NaOH (30 mL for three times), 1 M HCl (30 mL) and water (30 mL) [1]. The solution was dried with $MgSO_4$, filtered, and distilled under reduced pressure at 25 °C to gain the desired compound as a yellow solid. The substance was soaked in ether at 0 - 5 °C for 4 h, filtered, then recrystallized from acetic ether/light petroleum, filtered and dried. The structure was confirmed by IR, MS and NMR. Colorless needle-shaped single crystals of the title compound suitable for X-ray diffraction analysis were obtained by slow evaporation of the mixed solvent acetone/cyclohexane (1:2, *v*/*v*) at room temperature for one week (m.p. 130 - 132 °C).

Experimental details

All hydrogen atoms were placed in idealized positions and treated as riding with d(C—H) = 0.93 Å for benzene ring, d(C—H) = 0.97 Å for methylene, d(N—H) = 0.86 Å, and U_{iso}(H) = 1.2 U_{eq}(C,N).

Discussion

In the title crystal structure, the bond length of the carbonyl bond (C=O) is 1.225 Å which is in the normal range of 1.19 - 1.23 Å, similar to that observed in our previous papers [2-4]. There are three significant atomic planes in the structure, two benzene rings and the urea plane (O1, N1, N2, C1). The dihedral angle between the two benzene rings is 68.08(7)°, while the urea group forms dihedral angles of 14.07(2)° and 58.05(7)° with benzene and 4-chlorophenyl rings, respectively. In the crystal structure, molecules are linked into parallel chains along [001] by intermolecular N–H···O hydrogen bonding.

Table 1. Data collection and handling.

Crystal:	colorless needle, size 0.08 × 0.12 × 0.40 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	2.77 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	50.02°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	7122, 2423
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1544
$N(param)_{refined}$:	172
Programs:	SHELXS-97, SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.1094	0.4721	0.4168	0.077
H(2)	4*e*	0.4811	0.6359	0.4145	0.072
H(3)	4*e*	0.0175	0.7523	0.4985	0.080
H(4)	4*e*	0.0053	0.9007	0.5043	0.098
H(5)	4*e*	0.2897	0.9826	0.4433	0.101
H(6)	4*e*	0.5979	0.9158	0.3789	0.097
H(7)	4*e*	0.6163	0.7683	0.3730	0.083
H(8A)	4*e*	0.6602	0.4614	0.3160	0.096
H(8B)	4*e*	0.3972	0.5019	0.2885	0.096
H(10)	4*e*	0.0672	0.4066	0.2453	0.093
H(11)	4*e*	–0.1012	0.2699	0.2314	0.103
H(13)	4*e*	0.4762	0.1781	0.3767	0.105
H(14)	4*e*	0.6436	0.3147	0.3884	0.094

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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}
Cl(1)	4*e*	0.0507(2)	0.10849(6)	0.29104(7)	0.162(1)	0.0943(7)	0.159(1)	–0.0310(6)	0.0019(8)	–0.0437(6)
N(1)	4*e*	0.2208(3)	0.5121(1)	0.4212(1)	0.047(1)	0.057(1)	0.091(1)	–0.0091(8)	0.017(1)	–0.010(1)
N(2)	4*e*	0.3404(3)	0.6549(1)	0.4282(1)	0.051(1)	0.053(1)	0.079(1)	–0.0048(8)	0.0172(9)	–0.0042(9)
O(1)	4*e*	–0.0374(3)	0.6106(1)	0.4663(1)	0.059(1)	0.064(1)	0.123(2)	–0.0066(8)	0.038(1)	–0.006(1)
O(2)	4*e*	0.4739(3)	0.4972(1)	0.4083(1)	0.054(1)	0.065(1)	0.090(1)	–0.0018(7)	0.0169(9)	–0.0069(8)
C(1)	4*e*	0.1653(4)	0.5951(1)	0.4411(2)	0.055(1)	0.056(1)	0.071(2)	–0.008(1)	0.014(1)	–0.004(1)
C(2)	4*e*	0.3181(4)	0.7446(1)	0.4346(1)	0.052(1)	0.055(1)	0.060(1)	–0.004(1)	0.001(1)	–0.002(1)
C(3)	4*e*	0.1350(4)	0.7847(2)	0.4742(2)	0.058(2)	0.063(2)	0.081(2)	0.003(1)	0.010(1)	–0.002(1)
C(4)	4*e*	0.1280(5)	0.8738(2)	0.4773(2)	0.071(2)	0.067(2)	0.107(2)	0.012(1)	0.003(2)	–0.013(2)
C(5)	4*e*	0.2980(5)	0.9229(2)	0.4415(2)	0.082(2)	0.055(2)	0.111(2)	–0.000(1)	–0.011(2)	–0.005(2)
C(6)	4*e*	0.4804(5)	0.8830(2)	0.4029(2)	0.082(2)	0.063(2)	0.097(2)	–0.018(1)	0.006(2)	0.004(1)
C(7)	4*e*	0.4913(5)	0.7947(2)	0.3993(2)	0.064(2)	0.063(2)	0.080(2)	–0.010(1)	0.013(1)	–0.004(1)
C(8)	4*e*	0.4862(5)	0.4635(2)	0.3275(2)	0.086(2)	0.076(2)	0.083(2)	0.006(1)	0.035(2)	0.004(1)
C(9)	4*e*	0.3739(5)	0.3750(2)	0.3180(1)	0.070(2)	0.070(2)	0.058(1)	0.012(1)	0.015(1)	–0.001(1)
C(10)	4*e*	0.1515(5)	0.3607(2)	0.2716(2)	0.078(2)	0.086(2)	0.068(2)	0.020(2)	0.006(1)	0.001(1)
C(11)	4*e*	0.0499(5)	0.2788(2)	0.2631(2)	0.072(2)	0.112(2)	0.070(2)	0.005(2)	–0.007(1)	–0.020(2)
C(12)	4*e*	0.1736(6)	0.2115(2)	0.3018(2)	0.087(2)	0.078(2)	0.078(2)	–0.000(2)	0.002(2)	–0.023(2)
C(13)	4*e*	0.3941(6)	0.2240(2)	0.3495(2)	0.103(2)	0.065(2)	0.091(2)	0.012(2)	–0.011(2)	–0.001(1)
C(14)	4*e*	0.4925(5)	0.3060(2)	0.3566(2)	0.079(2)	0.074(2)	0.077(2)	0.006(1)	–0.011(1)	–0.004(1)

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References

1. Mai, X.; Lu, X.; Xia, H. Y.; Cao, Y.; Liao, Y.; Lv, X.: Synthesis, Antitumor Evaluation and Crystal Structure of Hydroxyurea Derivatives. Chem. Pharm. Bull. **58** (2010) 94-97.
2. Mai, X.; Xia, H. Y.; Cao, Y. S.; Lu, X. S.; Fang, X. N.: 1-(2-fluorobenzyl)-1-(2-fluorobenzyloxy)urea. Acta Crystallogr. **E65** (2009) o442.
3. Mai, X.; Xia, H. Y.; Cao, Y. S.; Lu, X. S.; Liao, Y. J.: Crystal Structure of 1-(benzyloxy)urea, $C_8H_{10}N_2O_2$. Z. Kristallogr. NCS **224** (2009) 547-548.
4. Wei, J.-J.; Mai, X.; Xia, H.-Y.; Tong, W.; Lin, L.: Crystal structure of ethyl 2-(3-(2-fluorobenzyloxy)ureido)acetate, $C_{12}H_{15}FN_2O_4$. Z. Kristallogr. NCS **225** (2010) 553-554.
5. Sheldrick, G. M.: A short history of SHELX. Acta Crystallogr. **A64** (2008) 112-122.