Crystal structure of 3-(4-bromophenyl)-4-(4-chlorophenylamino)furan-2(5*H*)-one, $C_{16}H_{11}BrClNO_2$

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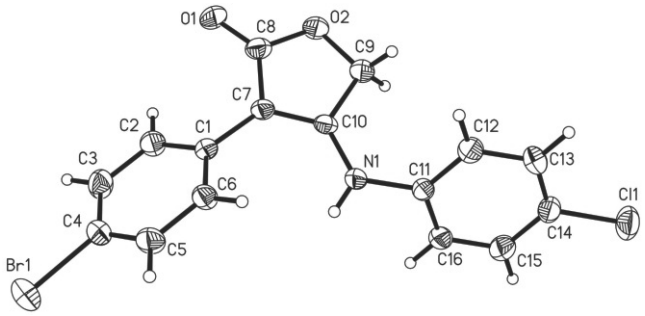

Abstract

$C_{16}H_{11}BrClNO_2$, orthorhombic, *Fdd*2 (no. 43), a = 25.301(1)Å, b = 51.746(2) Å, c = 4.5806(2) Å, V = 5997.0 Å^3, Z = 16, $R_{gt}(F)$ = 0.031, $wR_{ref}(F^2)$ = 0.066, T = 296 K.

Source of material

3-(4-Bromophenyl)-4-hydroxyfuran-2(5*H*)-one (127 mg, 0.5 mmol) was prepared according to [1], and was added into a mixture of 4-chloroaniline (76 mg, 0.6 mmol) and *p*-toluene sulphonic acid (3.4 mg, 0.02 mmol). The resulting mixture was heated to 363 K for 10 min. 5 mL of toluene were then added and refluxed for 4 h. After toluene was removed under reduced pressure, the residue was purified by column chromatography on silica gel, eluting with EtOAc/petroleum ether (v/v = 1/1).

Experimental details

The H atom bonded to N1 was located in difference Fourier map. The configuration of N1 could help to explain why *d*(C10-N1) is shorter than *d*(C11-N1). The Flack parameter is 0.016(8).

Discussion

Many compounds of γ-butyrolactone-core (furanone) show diverse biological activities. For example, some derivatives of γ-butyrolactone (5-alkoxy-4-aminofuran-2(5*H*)-one) showing good antibacterial activity against multiresistant *Staphylococcus aureus* were reported [2]. 4-Alkylamino or 4-arylamino derivatives of 3-arylfuran-2(5*H*)-one are potent inhibitors against tyrosyl-tRNA synthetase (TyrRS) [1,3], one of the aminoacyl-tRNA synthetases (aaRSs).

In the title compound, the bond C7—C10 (1.375(4) Å) was assigned as a double bond [4,5], and the title compound was therefore identified as a furan-2(5*H*)-one, not a furan-2(3*H*)-one. Butyrolactone moiety and the attached amino group (H1, N1, C10 and C11) are nearly co-planar with a dihedral angle of 5.2(2)°, indicating the conjugation of *p* orbital of N1 with the π molecular orbital of C7—C10, which shortens the bond distance of C10—N1. Additionally, *d*(C10—N1) is further shortened to 1.342(3) Å by the strong electron withdrawing effect of the carbonyl group (C8—O1), which is intermediated by π molecular orbital of C7—C10. However, C11—N1 is a typical single bond with the bond length of 1.433(3) Å. The dihedral angle of amino group and 4-chlorobenzene ring is quite large (58.5(2)°). The significant tilting of the amino group therefore breaks the conjugation with its attached 4-chlorobenzene ring. The bond distance *d*(C7—C10) = 1.355(3) Å is much shorter than *d*(C7—C8) = 1.450(4) Å. This significantly increases the steric strain between 4-(4-chlorophenylamino) group and 3-(4-bromophenyl) group, which leads to an increase in angle ∠C1–C7–C10 = 129.4(2)° in comparison with ∠C1–C7–C8 = 123.3(2)°. In the packing, the three independent hydrogen bonds generate a two-dimensional layer running parallel to (010), in which two kinds of hydrogen bonding patterns could be observed with $R_4^3(22)$ and $R_4^4(21)$ rings built from C–H···O and N–H···O hydrogen bonds. On the one hand, each molecule utilizes the two O atoms of lactone moiety as acceptors and is hydrogen-bonded to three others. On the other hand, the same molecule utilizes C5, C16 and N1 as donors and forms hydrogen bonding interactions with another three molecules. The above mentioned layers are stacked by van der Waals forces.

Table 1. Data collection and handling.

Crystal:	colorless block, size 0.20 × 0.20 × 0.30 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	29.22 cm^{-1}
Diffractometer, scan mode:	Bruker APEX, φ/ω
$2\theta_{max}$:	52.98°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	8799, 3097
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2169
$N(param)_{refined}$:	194
Programs:	SHELXS-97, SHELXL-97, SHELXTL [6]

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

Atom	Site	x	y	z	U_{iso}
H(2)	16*b*	0.3210	0.0732	0.1328	0.064
H(3)	16*b*	0.3730	0.0412	0.3390	0.070
H(5)	16*b*	0.4499	0.0942	0.8058	0.066
H(6)	16*b*	0.3978	0.1261	0.6061	0.054
H(9A)	16*b*	0.2841	0.1757	0.0377	0.065
H(9B)	16*b*	0.3095	0.1653	–0.2542	0.065
H(12)	16*b*	0.3411	0.2048	0.2196	0.066
H(13)	16*b*	0.3600	0.2441	0.0031	0.072
H(15)	16*b*	0.4834	0.2123	–0.4066	0.068
H(16)	16*b*	0.4629	0.1727	–0.2006	0.059
H(1)	16*b*	0.413(1)	0.1516(5)	0.201(8)	0.06(1)

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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}
Br(1)	16*b*	0.46186(1)	0.038684(7)	0.7370(1)	0.0758(2)	0.0717(2)	0.1112(4)	0.0184(2)	–0.0157(2)	0.0281(2)
C(1)	16*b*	0.35502(9)	0.10353(5)	0.3388(6)	0.033(1)	0.038(2)	0.047(2)	–0.003(1)	–0.002(1)	0.001(1)
C(2)	16*b*	0.3473(1)	0.07764(5)	0.2657(8)	0.049(2)	0.049(2)	0.062(2)	–0.007(1)	–0.018(2)	–0.000(2)
C(3)	16*b*	0.3784(1)	0.05842(5)	0.3892(8)	0.066(2)	0.043(2)	0.067(2)	–0.008(1)	–0.010(2)	0.001(2)
C(4)	16*b*	0.4168(1)	0.06489(5)	0.5842(7)	0.047(2)	0.050(2)	0.059(2)	0.003(1)	–0.009(2)	0.011(2)
C(5)	16*b*	0.4242(1)	0.09000(5)	0.6684(8)	0.049(2)	0.059(2)	0.057(2)	–0.003(1)	–0.019(2)	0.000(2)
C(6)	16*b*	0.3931(1)	0.10905(5)	0.5467(7)	0.045(2)	0.041(2)	0.051(2)	0.000(1)	–0.010(2)	–0.004(1)
C(7)	16*b*	0.32505(9)	0.12386(5)	0.1915(6)	0.031(1)	0.042(2)	0.043(2)	–0.002(1)	–0.006(1)	–0.001(1)
C(8)	16*b*	0.2727(1)	0.11991(6)	0.0708(7)	0.035(2)	0.057(2)	0.056(2)	–0.003(1)	–0.003(1)	0.000(2)
C(9)	16*b*	0.29719(9)	0.16038(5)	–0.0614(8)	0.040(2)	0.051(2)	0.072(3)	–0.002(1)	–0.015(2)	0.008(2)
C(10)	16*b*	0.34064(9)	0.14788(5)	0.1109(6)	0.028(1)	0.040(2)	0.044(2)	0.004(1)	–0.004(1)	–0.005(1)
C(11)	16*b*	0.39936(9)	0.18475(5)	0.0239(7)	0.037(1)	0.035(2)	0.051(2)	–0.001(1)	–0.007(2)	–0.002(1)
C(12)	16*b*	0.3693(1)	0.20622(5)	0.0900(8)	0.048(2)	0.045(2)	0.072(3)	0.001(1)	0.012(2)	–0.005(2)
C(13)	16*b*	0.3810(1)	0.22976(5)	–0.0361(9)	0.059(2)	0.040(2)	0.081(3)	0.010(1)	0.002(2)	–0.003(2)
C(14)	16*b*	0.4232(1)	0.23190(5)	–0.2178(7)	0.053(2)	0.042(2)	0.056(2)	–0.006(1)	–0.004(2)	0.004(2)
C(15)	16*b*	0.4545(1)	0.21067(5)	–0.2823(9)	0.051(2)	0.058(2)	0.061(2)	–0.005(1)	0.008(2)	0.003(2)
C(16)	16*b*	0.4422(1)	0.18709(5)	–0.1594(7)	0.040(2)	0.040(2)	0.068(2)	0.003(1)	–0.001(2)	–0.008(2)
Cl(1)	16*b*	0.43916(4)	0.26167(2)	–0.3737(2)	0.0893(6)	0.0470(4)	0.0891(7)	–0.0080(4)	0.0078(5)	0.0145(4)
N(1)	16*b*	0.38700(9)	0.16005(4)	0.1479(6)	0.034(1)	0.035(1)	0.071(2)	–0.000(1)	–0.011(1)	0.004(1)
O(1)	16*b*	0.24331(7)	0.10145(4)	0.0880(5)	0.038(1)	0.061(1)	0.088(2)	–0.0173(9)	–0.017(1)	0.007(1)
O(2)	16*b*	0.25712(6)	0.14109(4)	–0.0799(5)	0.037(1)	0.064(1)	0.081(2)	–0.0039(9)	–0.022(1)	0.017(1)

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