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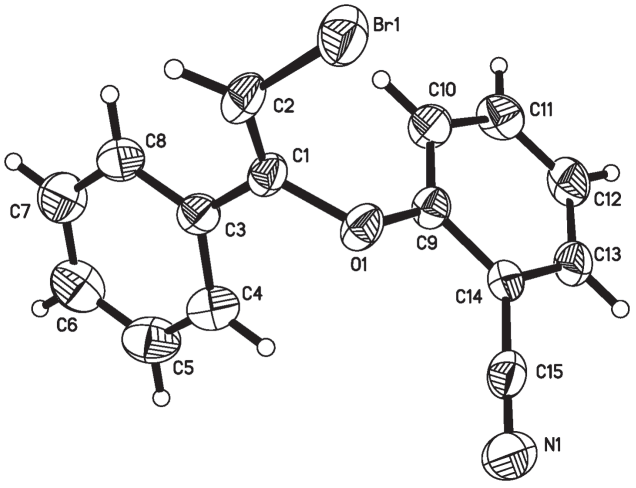
Da-Bin Shi*, Chao Meng and Yong-Zheng Chen

Crystal structure of (*Z*)-2-((2-bromo-1-phenyl-vinyl)oxy)benzonitrile, $C_{15}H_{10}BrNO$



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Abstract

$C_{15}H_{10}BrNO$, monoclinic, $P2_1/n$, $a = 12.567(3)$ Å, $b = 8.7170(17)$ Å, $c = 12.773(3)$ Å, $\beta = 111.41(3)°$, $V = 1302.7(4)$ Å^3, $Z = 4$, $R_{gt}(F) = 0.0510$, $wR_{ref}(F^2) = 0.1265$, $T = 293$ K.

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The crystal structure is shown in the figure. Tables 1–3 contain details of the measurement method and a list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Colourless, block, size 0.30×0.40×0.50 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	31.41 cm^{-1}
Diffractometer, scan mode:	Rigaku Mercury CCD diffractometer, ω scan
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	12357, 2985
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\ \sigma(I_{obs})$, 1583
$N(param)_{refined}$:	163
Programs:	SHELX [8]

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{iso}
H(2)	4*e*	0.6178	0.9487	0.1042	0.078
H(13)	4*e*	0.6565	0.9776	0.6913	0.077
H(8)	4*e*	0.7902	1.0557	0.1549	0.083
H(12)	4*e*	0.6529	1.2397	0.6633	0.086
H(10)	4*e*	0.6500	1.1730	0.3523	0.074
H(11)	4*e*	0.6505	1.3344	0.4961	0.084
H(4)	4*e*	0.8492	0.8022	0.4323	0.088
H(7)	4*e*	0.9816	1.0578	0.1873	0.102
H(5)	4*e*	1.0410	0.8080	0.4639	0.109
H(6)	4*e*	1.1077	0.9319	0.3405	0.110

Source of material

A mixture of 1-(2-bromoethynyl)benzene (0.905 g, 5 mmol), K_2CO_3 (1.38 g, 10 mmol) 2-hydroxybenzonitrile (0.595 g, 5 mmol) and 20 mL acetonitrile in a 50 mL dry flask affiliated with a water separator was refluxed and stirred for 6 h. Acetonitrile was removed by water separator and the residue was washed with water, filtered, dried, and crystallized three times from ethanol yielding title compound as colorless solid [7]. The crystal suitable for X-ray analysis was obtained by slow evaporation of ethanol at room temperature over a period of ten days, yield: 0.56 g (96.2%). Elemental analysis:–found: C, 59.97%; H, 3.32%; N, 4.65%; calculated for $C_{15}H_{10}BrNO$: C, 60.02%; H, 3.36%; N, 4.67%.

Experimental details

All hydrogen atoms were identified in difference Fourier syntheses. The structure was solved by direct methods [8] and refined on F^2 by full-matrix least-squares technique using

***Corresponding author: Da-Bin Shi,** School of Pharmaceutical Sciences, Zunyi Medical College, Zunyi 563000, People's Republic of China, e-mail: sdb007.student@sina.com
Chao Meng and Yong-Zheng Chen: School of Pharmaceutical Sciences, Zunyi Medical College, Zunyi 563000, People's Republic of China

Table 3: Atomic displacement parameters (Å^2).

Atom	Site	*x*	*y*	*z*	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4*e*	0.43878(4)	0.92018(7)	0.13576(4)	0.0711(4)	0.1052(5)	0.0578(4)	−0.0050(3)	0.0259(3)	−0.0026(2)
C(1)	4*e*	0.6748(4)	0.9170(4)	0.2646(3)	0.072(3)	0.043(2)	0.046(2)	−0.004(2)	0.029(2)	−0.003(2)
O(1)	4*e*	0.6433(3)	0.8712(3)	0.3539(2)	0.086(2)	0.045(2)	0.047(2)	−0.010(1)	0.032(1)	−0.007(1)
C(2)	4*e*	0.5950(4)	0.9315(5)	0.1647(3)	0.082(4)	0.075(3)	0.041(2)	−0.004(2)	0.027(2)	0.001(2)
C(3)	4*e*	0.7980(4)	0.9274(4)	0.2880(3)	0.071(3)	0.039(2)	0.055(2)	−0.003(2)	0.024(2)	−0.004(2)
C(14)	4*e*	0.6537(4)	0.9172(4)	0.5388(3)	0.043(2)	0.053(2)	0.052(2)	0.002(2)	0.021(2)	−0.002(2)
C(9)	4*e*	0.6505(3)	0.9782(4)	0.4352(3)	0.054(3)	0.047(2)	0.046(2)	−0.005(2)	0.021(2)	−0.006(2)
C(13)	4*e*	0.6546(4)	1.0164(5)	0.6228(3)	0.070(3)	0.076(3)	0.051(2)	0.010(3)	0.029(2)	−0.002(2)
C(8)	4*e*	0.8404(5)	1.0042(5)	0.2171(4)	0.079(4)	0.055(3)	0.076(3)	0.001(3)	0.032(2)	0.008(2)
C(15)	4*e*	0.6567(4)	0.7545(6)	0.5547(3)	0.059(3)	0.076(4)	0.048(2)	0.000(2)	0.025(2)	0.000(2)
C(12)	4*e*	0.6529(4)	1.1730(6)	0.6064(4)	0.084(4)	0.075(3)	0.060(3)	0.006(3)	0.030(2)	−0.023(2)
C(10)	4*e*	0.6505(4)	1.1326(5)	0.4199(3)	0.074(3)	0.053(3)	0.061(3)	−0.002(2)	0.027(2)	−0.003(2)
C(11)	4*e*	0.6512(4)	1.2288(5)	0.5067(4)	0.083(4)	0.044(3)	0.084(3)	−0.003(2)	0.031(3)	−0.013(2)
C(4)	4*e*	0.8753(5)	0.8534(5)	0.3825(4)	0.076(4)	0.066(3)	0.070(3)	−0.003(3)	0.015(3)	0.005(2)
N(1)	4*e*	0.6611(4)	0.6247(5)	0.5716(3)	0.101(4)	0.070(3)	0.077(3)	0.006(3)	0.036(2)	0.011(2)
C(7)	4*e*	0.9548(5)	1.0060(6)	0.2365(5)	0.078(4)	0.084(4)	0.103(4)	−0.007(3)	0.043(3)	0.007(3)
C(5)	4*e*	0.9897(5)	0.8569(7)	0.4010(5)	0.079(4)	0.089(4)	0.081(4)	0.006(3)	0.002(3)	0.003(3)
C(6)	4*e*	1.0300(5)	0.9319(6)	0.3278(6)	0.065(4)	0.089(4)	0.115(5)	−0.006(3)	0.026(3)	−0.009(3)

the SHELX-97 program package [9]. The hydrogen atoms were placed in geometrically idealized positions and refined using a riding model.

Discussion

In recent years, bromoalkynes have emerged as versatile and useful building blocks in a variety of synthetic transformations, which can be generally designed as a dual functionalized molecules and different reaction intermediates to synthesize all kinds of new compounds with other organic molecules [1–4]. Bond lengths and bond angles within the molecular system are in agreement with the values reported [5, 6]. The title compound contains two aromatic rings which are linked by a C-O moiety, which generates a conjugated system. The dihedral angle between the planes of two rings is 84.6°. Due to the conjugation of these aromatic rings, the bond lengths of C9–O1 (1.374 Å) and C1–O1 (1.395 Å) are shorter than that of typical C–O bond length (1.45 Å). The bond angle (C1–O1–C9) and (C1–C3–C4) are 117.6(3)° and 119.7(4)°, respectively. Classical hydrogen bonds are not found in the crystal structures. It is evident that there is a face-to-face π–π stacking interaction. The centroid-centroid distance between two adjacent benzonitrile moieties is 3.88 Å.

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