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Crystal structure of 3-allyl-4-(2-bromoethyl)-5-(4-methoxyphenyl)-2-(*p*-tolyl)furan, C₂₃H₂₃BrO₂

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Abstract

C₂₃H₂₃BrO₂, monoclinic, *P*₂₁/*c* (no. 14), *a* = 24.193(4) Å, *b* = 5.8614(10) Å, *c* = 13.873(3) Å, β = 93.168(3)°, *V* = 1964.3(6) Å³, *Z* = 4, *R*_{gt}(*F*) = 0.0316, *wR*_{ref}(*F*²) = 0.0915, *T* = 296(2) K.

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The molecular structure is shown in the figure. Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Source of material

(4-Methoxyphenyl)(1-(*p*-tolylethynyl)cyclopropyl)methanone (0.2 mmol, 1.0 eq.), allyl bromide (0.4 mmol, 2.0 eq.), PdBr₂

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.23 × 0.20 × 0.18 mm
Wavelength:	Mo Kα radiation (0.71073 Å)
μ:	2.11 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II, φ and ω
θ _{max} , completeness:	25.0°, >99%
<i>N</i> (<i>hkl</i>) _{measured} , <i>N</i> (<i>hkl</i>) _{unique} , <i>R</i> _{int} :	11421, 3446, 0.025
Criterion for <i>I</i> _{obs} , <i>N</i> (<i>hkl</i>) _{gt} :	<i>I</i> _{obs} > 2 σ(<i>I</i> _{obs}), 2735
<i>N</i> (<i>param</i>) _{refined} :	237
Programs:	Bruker [1], SHELX [2]

(0.03 eq.) and acetonitrile (5 mL) were added to a screw-capped Schlenk tube containing a magnetic stir bar, which was evacuated and refilled with nitrogen for three times. The resulting mixture was stirred at room temperature for 3 h. After the reaction was complete, the reaction mixture was concentrated under reduced pressure, and the residue was purified with petroleum ether-ethyl acetate (15:1, v/v) as eluent by silica gel chromatography column to obtain a colourless solid. Crystals were obtained by slow evaporation of a tetrahydrofuran solution at room temperature after three days.

Experimental details

All hydrogen atoms were placed in calculated positions (C–H = 0.93–0.97 Å) and refined as riding atoms. The *U*_{iso}

Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
Br1	0.80538 (2)	1.43349 (4)	0.48348 (2)	0.05837 (13)
C1	0.79563 (10)	0.7833 (4)	0.73815 (16)	0.0444 (6)
C2	0.76754 (10)	0.8672 (4)	0.65802 (17)	0.0444 (6)
C3	0.71187 (10)	0.7817 (4)	0.65917 (17)	0.0443 (6)
C4	0.70978 (10)	0.6509 (4)	0.73957 (17)	0.0446 (6)
C5	0.85237 (10)	0.7944 (4)	0.77971 (17)	0.0442 (6)
C6	0.88782 (12)	0.9727 (5)	0.7614 (2)	0.0590 (7)
H6	0.875137	1.090814	0.721215	0.071*
C7	0.94177 (12)	0.9807 (5)	0.8012 (2)	0.0614 (7)
H7	0.964986	1.100834	0.786586	0.074*
C8	0.96056 (11)	0.8094 (5)	0.86247 (19)	0.0586 (7)
C9	0.92545 (12)	0.6305 (5)	0.8828 (2)	0.0609 (7)
H9	0.937952	0.514510	0.924245	0.073*
C10	0.87257 (11)	0.6236 (5)	0.84238 (18)	0.0522 (6)
H10	0.849606	0.502614	0.856966	0.063*
C11	1.04803 (14)	0.9908 (7)	0.8911 (3)	0.0909 (11)
H11A	1.055427	0.999513	0.823958	0.136*
H11B	1.082181	0.970264	0.928745	0.136*
H11C	1.030582	1.129354	0.910380	0.136*
C12	0.66853 (11)	0.5015 (4)	0.77978 (17)	0.0451 (6)
C13	0.61223 (11)	0.5440 (5)	0.7682 (2)	0.0569 (7)
H13	0.599808	0.673532	0.734945	0.068*
C14	0.57443 (12)	0.3972 (5)	0.8053 (2)	0.0625 (8)
H14	0.536855	0.429549	0.796664	0.075*
C15	0.59108 (11)	0.2030 (5)	0.85518 (19)	0.0567 (7)
C16	0.64687 (12)	0.1608 (5)	0.8672 (2)	0.0602 (7)
H16	0.658970	0.031381	0.900903	0.072*
C17	0.68540 (11)	0.3058 (5)	0.83044 (18)	0.0531 (6)
H17	0.722922	0.272755	0.839533	0.064*
C18	0.54915 (14)	0.0422 (6)	0.8964 (3)	0.0786 (10)
H18A	0.564734	−0.108086	0.902175	0.118*
H18B	0.516369	0.037798	0.854175	0.118*
H18C	0.539854	0.095433	0.958928	0.118*
C19	0.66637 (11)	0.8182 (5)	0.58225 (18)	0.0605 (7)
H19A	0.644691	0.679356	0.575172	0.073*
H19B	0.682870	0.846961	0.521274	0.073*
C20	0.62741 (14)	1.0176 (6)	0.6038 (2)	0.0694 (8)
H20	0.643218	1.147781	0.631967	0.083*
C21	0.57506 (16)	1.0176 (7)	0.5859 (3)	0.0918 (11)
H21A	0.557757	0.890465	0.557761	0.110*
H21B	0.554234	1.144762	0.601016	0.110*
C22	0.79033 (11)	1.0073 (4)	0.57863 (18)	0.0466 (6)
H22A	0.774486	0.954221	0.516896	0.056*
H22B	0.830102	0.985830	0.579059	0.056*
C23	0.77790 (12)	1.2574 (4)	0.58950 (19)	0.0578 (7)
H23A	0.794968	1.312255	0.650027	0.069*
H23B	0.738215	1.278718	0.591334	0.069*
O1	0.76068 (7)	0.6490 (3)	0.78880 (11)	0.0459 (4)
O2	1.01226 (8)	0.8026 (4)	0.90646 (15)	0.0781 (6)

values were set to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms.

Comment

In recent years, furans as an important class of heterocycles have always been a hot topic due to their wide use in drug science, material science, and synthetic chemistry [3–9]. Now, we report the synthesis and crystal structure of the title compound (*cf.* Figure).

In the crystal structure, the asymmetric unit consists of one molecule with bonding parameters in the normal ranges [10, 11]. The title compound is a non-planar molecule. The dihedral angles of A/B, A/C, B/B are 21.091(1), 22.889(1), and 30.229(1)°, respectively, in which A, B, C representing aromatic rings [C5/C6/C7/C8/C9/C10], [C12/C13/C14/C15/C16/C17], and furan ring [C1/C2/C3/C4/O1], respectively. The intermolecular and weak C–H···Br hydrogen bonds link molecules into chains.

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