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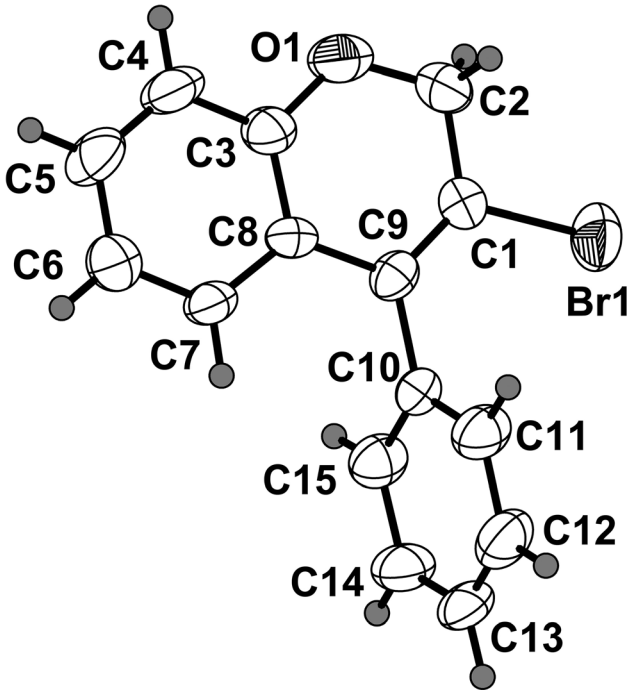
Synthesis and crystal structure of 3-bromo-4-phenyl-2*H*-chromene, $C_{15}H_{11}BrO$



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Abstract

$C_{15}H_{11}BrO$, triclinic, $P\bar{1}$ (no. 2), a = 6.0696(9) Å, b = 10.1112(10) Å, c = 10.9318(13) Å, α = 104.938(10)°, β = 104.246(12)°, γ = 97.021(10)°, V = 615.82(14) Å^3, Z = 2, $R_{gt}(F)$ = 0.0573, $wR_{ref}(F^2)$ = 0.1355, T = 293(2) K.

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Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.34 × 0.20 × 0.11 mm
Wavelength:	Mo *K*α radiation (0.71073 Å)
μ:	3.32 mm^{-1}
Diffractometer, scan mode:	Xcalibur, ω
θ_{max}, completeness:	25.0°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int}:	4320, 2170, 0.054
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\sigma(I_{obs})$, 1383
$N(param)_{refined}$:	154
Programs:	CrysAlis^{PRO} [1], Olex2 [2], SHELX [3]

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.

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

Atom	*x*	*y*	*z*	U_{iso}*/U_{eq}
Br1	0.00958 (12)	0.71427 (7)	0.47674 (6)	0.0700 (3)
O1	0.3547 (10)	0.9778 (5)	0.3253 (5)	0.0957 (18)
C1	0.1773 (9)	0.7723 (6)	0.3685 (5)	0.0462 (15)
C2	0.1967 (10)	0.9235 (6)	0.3821 (6)	0.0569 (17)
H2A	0.0450	0.9396	0.3420	0.068*
H2B	0.2413	0.9748	0.4755	0.068*
C3	0.4405 (10)	0.8910 (6)	0.2364 (5)	0.0498 (15)
C4	0.5733 (12)	0.9558 (6)	0.1743 (6)	0.0644 (18)
H4	0.5947	1.0521	0.1905	0.077*
C5	0.6732 (11)	0.8780 (7)	0.0890 (6)	0.0618 (18)
H5	0.7627	0.9212	0.0470	0.074*
C6	0.6410 (11)	0.7368 (7)	0.0661 (6)	0.0612 (18)
H6	0.7085	0.6837	0.0080	0.073*
C7	0.5082 (10)	0.6720 (6)	0.1288 (5)	0.0487 (15)
H7	0.4899	0.5758	0.1128	0.058*
C8	0.4013 (9)	0.7476 (5)	0.2152 (5)	0.0372 (13)
C9	0.2643 (9)	0.6858 (6)	0.2875 (5)	0.0395 (13)
C10	0.2237 (9)	0.5307 (5)	0.2651 (5)	0.0388 (13)
C11	0.0128 (10)	0.4473 (7)	0.1839 (6)	0.0578 (16)
H11	−0.1027	0.4879	0.1436	0.069*
C12	−0.0271 (12)	0.3039 (7)	0.1624 (6)	0.0640 (18)
H12	−0.1685	0.2488	0.1073	0.077*
C13	0.1411 (12)	0.2436 (6)	0.2220 (6)	0.0549 (17)
H13	0.1127	0.1477	0.2088	0.066*
C14	0.3492 (12)	0.3230 (6)	0.3001 (6)	0.0578 (17)
H14	0.4633	0.2812	0.3398	0.069*
C15	0.3934 (11)	0.4663 (6)	0.3213 (6)	0.0527 (15)
H15	0.5379	0.5195	0.3735	0.063*

Source of material

To a suspension of (3-phenoxyprop-1-yn-1-yl)benzene (41.6 mg, 0.2 mmol) and AgOTf (5.1 mg, 0.01 mmol) in DCE (4 mL) was added NBS (39.1 mg, 0.22 mmol) at 80 °C and stirred for 8 h. After the completion of the reaction, the reaction mixture was washed with saturated sodium thiosulfate solution, saturated $NaHCO_3$ solution, saturated saline solution, respectively. After extraction with ethyl acetate, and distillation off the solvent under reduced pressure, the remaining crude product was purifed by silica gel (petroleum ether) column chromatography to obtain products.

Experimental details

Comment

2*H*-chromenes are an important class of oxygen-containing heterocycles, which are widely found in many bioactive natural products and pharmaceutical molecules, providing key function of regulating various biopolymers [4]. Therefore, more and more chemists are interested in the development of new methods for synthesizing and modifying 2*H*-chromene derivatives [5].

The title compound contains one 2*H*-chromene and one bromine substituent. The bond lengths and angles derived from the title structure are within normal ranges and consistent with those reported previously in similar structures [6]. The C=C bond was determined at the distance of 1.331(6) Å (C1–C9). The aryl ether bond was confirmed by the distance of 1.381(6) Å (O1–C3), and the alkyl ether bonds was identified by the distance of 1.395(7) Å (O1–C2), respectively. The bromine atom was determined at the distance of 1.894(5) Å (Br1–C1). The phenylenyl bond was confirmed by the distance of 1.503(7) Å (C9–C10).

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Conflict of interest statement: The authors declare no conflicts of interest regarding this article.

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