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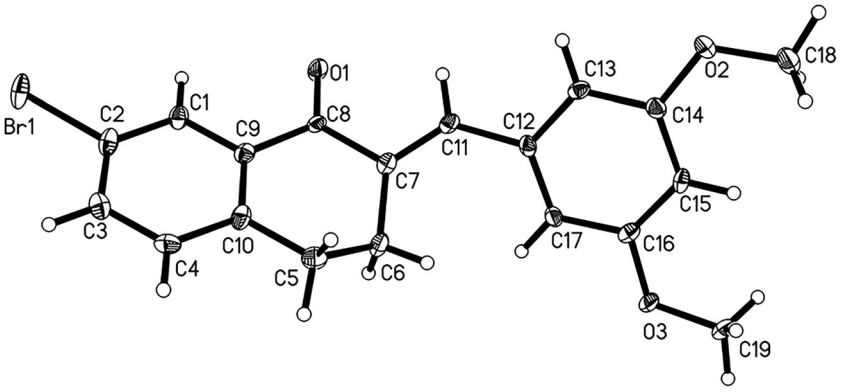
# Crystal structure of (*E*)-7-bromo-2-(3,5-dimethoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one, $C_{19}H_{17}BrO_3$




https://doi.org/10.1515/ncrs-2022-0316
Received June 18, 2022; accepted July 15, 2022;
published online August 3, 2022

**Abstract**

$C_{19}H_{17}BrO_3$, triclinic, $P\bar{1}$ (no. 2), $a$ = 8.2887(7) Å, $b$ = 8.4190(12) Å, $c$ = 13.1164(15) Å, $\alpha$ = 93.634(11)°, $\beta$ = 90.609(9)°, $\gamma$ = 118.201(11)°, $V$ = 804.17(18) Å$^3$, $Z$ = 2, $R_{gt}(F)$ = 0.0738, $wR_{ref}(F^2)$ = 0.1614, T = 150 K.

**CCDC no.:** 2161858

The molecular structure is shown in the figure. Displacement ellipsoids are drawn at the 40% probability level.

Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

**Table 1:** Data collection and handling.

| | |
|---|---|
| Crystal: | Colourless block |
| Size: | 0.15 × 0.12 × 0.10 mm |
| Wavelength: | Mo *K*α radiation (0.71073 Å) |
| $\mu$: | 2.57 mm$^{-1}$ |
| Diffractometer, scan mode: | SuperNova |
| $\theta_{max}$, completeness: | 25.5°, >99% |
| $N(hkl)_{measured}$, $N(hkl)_{unique}$, $R_{int}$: | 5429, 2985, 0.069 |
| Criterion for $I_{obs}$, $N(hkl)_{gt}$: | $I_{obs}$ > 2 σ($I_{obs}$), 2546 |
| $N(param)_{refined}$: | 211 |
| Programs: | CrysAlis$^{PRO}$ [1], SHELX [2, 3] |

## Source of material

The preparation of the intermediate 7-bromo-3,4-dihydro naphthalen-1(2*H*)-one was similar to the previous reported method [4, 5], which was used to synthesize the aromatic tetralones in the next step. 7-Bromo-3,4-dihydronaphthal en-1(2*H*)-one (0.60 g, 2.7 mmol) and 3,5-dimethoxybenzaldehyde (0.67 g, 4.0 mmol) were dissolved in 5 mL of methanol. A NaOH solution (25%, 2 mL) was dropped to the above solution through a constant pressure dropping funnel. The mixture was stirred for 4 h at 298 K, and the progress of the reaction was traced by thin layer chromatography (TLC). After the completion of the reaction, the upper solution was poured off and the remaining solid was washed by the cold methanol and dried. The pure product of the title compound was separated by 200–300 mesh silica gel column (petroleum ether/ethyl acetate/methanol = 10:10:1, v/v/v).

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

| Atom | *x* | *y* | *z* | $U_{iso}$*/$U_{eq}$ |
|---|---|---|---|---|
| Br1 | 1.06665 (8) | 0.74238 (10) | 0.21958 (5) | 0.0303 (3) |
| C1 | 0.8426 (8) | 0.5624 (8) | 0.3815 (4) | 0.0195 (13) |
| H1 | 0.771558 | 0.474543 | 0.330170 | 0.023* |
| C2 | 0.9975 (8) | 0.7158 (8) | 0.3582 (4) | 0.0206 (13) |
| C3 | 1.1045 (8) | 0.8522 (8) | 0.4327 (5) | 0.0231 (13) |
| H3 | 1.207276 | 0.955778 | 0.415461 | 0.028* |
| C4 | 1.0539 (8) | 0.8295 (8) | 0.5334 (5) | 0.0217 (13) |
| H4 | 1.124545 | 0.919343 | 0.584029 | 0.026* |
| C5 | 0.8549 (8) | 0.6535 (9) | 0.6715 (5) | 0.0244 (14) |
| H5A | 0.887366 | 0.771045 | 0.705500 | 0.029* |
| H5B | 0.928711 | 0.607489 | 0.703575 | 0.029* |
| C6 | 0.6522 (8) | 0.5254 (8) | 0.6865 (4) | 0.0204 (13) |
| H6A | 0.636370 | 0.496944 | 0.757402 | 0.024* |
| H6B | 0.580705 | 0.585603 | 0.671510 | 0.024* |
| C7 | 0.5816 (7) | 0.3522 (8) | 0.6186 (4) | 0.0172 (12) |
| C8 | 0.6324 (8) | 0.3708 (8) | 0.5091 (4) | 0.0153 (12) |
| C9 | 0.7936 (8) | 0.5406 (8) | 0.4838 (4) | 0.0169 (12) |
| C10 | 0.9000 (8) | 0.6756 (8) | 0.5605 (4) | 0.0198 (13) |
| C11 | 0.4872 (7) | 0.1826 (8) | 0.6471 (4) | 0.0164 (12) |
| H11 | 0.462205 | 0.091826 | 0.595573 | 0.020* |
| C12 | 0.4174 (7) | 0.1188 (7) | 0.7476 (4) | 0.0158 (12) |
| C13 | 0.4095 (7) | −0.0439 (8) | 0.7722 (4) | 0.0168 (12) |
| H13 | 0.445886 | −0.107472 | 0.725625 | 0.020* |
| C14 | 0.3474 (7) | −0.1101 (8) | 0.8657 (4) | 0.0171 (12) |
| C15 | 0.2896 (8) | −0.0191 (8) | 0.9355 (4) | 0.0179 (12) |
| H15 | 0.249605 | −0.063302 | 0.998698 | 0.021* |
| C16 | 0.2926 (8) | 0.1385 (8) | 0.9093 (4) | 0.0187 (12) |
| C17 | 0.3555 (8) | 0.2097 (8) | 0.8166 (4) | 0.0171 (12) |
| H17 | 0.356464 | 0.316094 | 0.800407 | 0.021* |
| C18 | 0.2627 (9) | −0.3543 (9) | 0.9729 (5) | 0.0281 (15) |
| H18A | 0.331427 | −0.277078 | 1.031810 | 0.042* |
| H18B | 0.262500 | −0.468327 | 0.974168 | 0.042* |
| H18C | 0.139025 | −0.373505 | 0.973577 | 0.042* |
| C19 | 0.2004 (8) | 0.1908 (8) | 1.0759 (4) | 0.0214 (13) |
| H19A | 0.102114 | 0.069609 | 1.077040 | 0.032* |
| H19B | 0.167062 | 0.273034 | 1.112066 | 0.032* |
| H19C | 0.308928 | 0.199309 | 1.108233 | 0.032* |
| O1 | 0.5450 (6) | 0.2496 (6) | 0.4420 (3) | 0.0240 (10) |
| O2 | 0.3450 (6) | −0.2711 (6) | 0.8822 (3) | 0.0263 (10) |
| O3 | 0.2345 (6) | 0.2361 (6) | 0.9728 (3) | 0.0252 (10) |

## Experimental details

The H atoms were placed in idealized positions and treated as riding on their parent atoms, with *d* (C–H) = 0.96 Å (methyl), $U_{iso}$(H) = 1.5$U_{eq}$(C), and *d*(C–H) = 0.97 Å (methylene), $U_{iso}$(H) = 1.2$U_{eq}$(C), and *d*(C–H) = 0.93 Å (aromatic), $U_{iso}$(H) = 1.2$U_{eq}$(C). The analysis of the $F_o/F_c$ data and the two difference density peaks point to a twinning with an expected ratio smaller than 4:1. As the constitution of the target molecules is obtained without serious distorsions, a twin refinement was not undertaken.

## Comment

Curcumin is a yellow phenolic pigment found mainly in the rhizome of turmeric, a plant of the ginger family. It was found to possess multiple anti-inflammatory, anti-tumour and anti-rheumatic effects without toxicity to humans at higher doses [6]. However, the poor bioavailability and unstable structure limited its clinical application [7]. In order to overcome the shortcoming of curcumin derivatives, a set of curcumin analogues were designed and synthesized in recent years. For example, halogenated bis(methoxybenzyl)-4-piperidone displays significant anticancer activity effects [8]. Boc-piperidone chalcones were novel cytotoxic drugs against highly metastatic cancer cells [9]. These results shows that curcumin derivatives have good bioactivity, which can be regarded as anti-inflammatory and anti-tumour agents [10, 11]. Based on the above studies, the target product of (*E*)-7-bromo-2-(3,5-dimethoxybenzylidene)-3,4-dihydronaphthalen-1(2*H*)-one was synthesized by the Claisen–Schmidt condensation reaction.

Single-crystal structure analysis reveals that there is one molecule in the asymmetric unit. In the molecule, the bond length of C7=C11 is 1.344(8) Å, other bond lengths and bond angles are similar to the values reported by related articles [12–15]. As shown in the figure, the methoxybenzene ring and carbonyl group are arranged around the double bond with the torsion of C8–C7–C11–C12 being 180.0(5)°. Thus the title compound adopts the *E* stereochemistry. The C4 atom is deviated from the least-squares plane of the cyclohexanone ring with the value of 0.480(17) Å, which makes the cyclohexyl ring display an envelope conformation. The two benzene rings are not coplanar with the dihedral angle of 59.70(15)°. Through the further observation, it was found that adjacent molecules were linked to a chain structure along the *b* axis by the weak C18–H18B···O3 hydrogen bond. The chains were further interacted to form the bc plane by C18–H18A···Br1 interaction. The neighbouring chains further interact with each other *via* weak C3–H3···O1 hydrogen bond to form a 3D architecture.

Considering the bioactive property of aromatic-tetralones, –Br and –OMe substitutes were selected to modify the structure of curcumin analogue, which can act as the hydrogen-bonding acceptor and enhance interactions with proteins [16].

**Author contributions:** All the authors have accepted responsibility for the entire content of this submitted manuscript and approved submission.
**Research funding:** Shandong Provincial Natural Science Foundation (No. ZR2019MB032), College Youth Innovation Science and Technology Support Programme of Shandong Province (No. 2020KJK003), Key R&D Programme of Shandong Province (No. 2019JZZY011104), Shandong New Drug Loading & Release Technology and Preparation Engineering Laboratory.
**Conflict of interest statement:** The authors declare no conflicts of interest regarding this article.

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