

Hong Dae Choi,^a Pil Ja Seo,^a
Byung Wha Son^b and Uk Lee^{b*}^aDepartment of Chemistry, Dongeui University, San 24 Kaya-dong Busanjin-ku, Busan 614-714, Republic of Korea, and ^bDepartment of Chemistry, Pukyong National University, 599-1 Daeyeon 3-dong Nam-ku, Busan 608-737, Republic of Korea

Correspondence e-mail: uklee@pknu.ac.kr

Key indicators

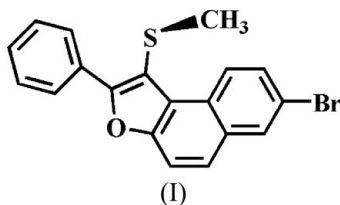
Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C}) = 0.003 \text{ \AA}$
R factor = 0.020
wR factor = 0.048
Data-to-parameter ratio = 14.4For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.7-Bromo-1-methylsulfanyl-2-phenylnaphtho-
[2,1-*b*]furan

The title compound, $\text{C}_{19}\text{H}_{13}\text{BrOS}$, was prepared by the Lewis acid-catalysed reaction of 6-bromo-2-naphthol with 2-chloro-2-(methylsulfanyl)acetophenone. The naphthofuran ring system is nearly planar. The crystal structure is stabilized by a $\text{C}-\text{H} \cdots \pi$ interaction.

Received 15 November 2006
Accepted 23 November 2006

Comment

This work follows on from our previous reports on isomeric naphtho[2,1-*b*]furans (Choi, Woo *et al.*, 2006; Choi, Seo *et al.*, 2006). The title compound, (I), was obtained by the one-pot reaction of 6-bromo-2-naphthol with 2-chloro-2-(methylsulfanyl)acetophenone in the presence of zinc chloride. We report here its crystal structure (Fig. 1).



The bond lengths and angles in (I) are as expected for this type of compound (Choi, Woo *et al.*, 2006; Choi, Seo *et al.*, 2006). The naphtho[2,1-*b*]furan unit is again nearly planar, with a mean deviation of 0.026 Å from the least-squares plane defined by the 13 constituent atoms. The dihedral angle between the mean plane of the naphtho[2,1-*b*]furan group and the phenyl ring is 18.73 (8)°. In the crystal structure, no aromatic $\pi-\pi$ stacking interaction is observed between adjacent molecules. A $\text{C}-\text{H} \cdots \pi$ interaction is, however, found (Fig. 2) [$\text{C}2-\text{H}2 \cdots \text{C}g^i = 2.85 \text{ \AA}$, $\text{C}2 \cdots \text{C}g^i = 3.748 (3) \text{ \AA}$ and $\text{C}2-\text{H}2 \cdots \text{C}g^i = 145^\circ$; symmetry code: (i) $x + \frac{1}{2}, -y - 1, z$; *Cg* is the centroid of the phenyl ring].

Experimental

The title compound was prepared according to the method described for substituted naphtho[2,1-*b*]furans (Choi *et al.*, 1999). Zinc chloride (341 mg, 2.5 mmol) was added at room temperature to a stirred solution of 6-bromo-2-naphthol (558 mg, 2.5 mmol) and 2-chloro-2-(methylsulfanyl)acetophenone (501 mg, 2.5 mmol) in dichloromethane (30 ml). The mixture was stirred for 40 min and quenched with water. The organic layer was separated, dried over magnesium sulfate, filtered and concentrated in vacuum. The residue was purified by column chromatography (CCl_4) to afford (I) as a pale-yellow solid. Crystals suitable for X-ray analysis were obtained by slow evaporation of a chloroform solution [yield 67%, m.p. 417–418 K; $R_F = 0.79$ (CCl_4)].

Crystal data

$C_{19}H_{13}BrOS$
 $M_r = 369.26$
 Orthorhombic, $Pca2_1$
 $a = 17.700 (1) \text{ \AA}$
 $b = 5.2952 (3) \text{ \AA}$
 $c = 15.8343 (9) \text{ \AA}$
 $V = 1484.07 (15) \text{ \AA}^3$

$Z = 4$
 $D_x = 1.653 \text{ Mg m}^{-3}$
 Mo $K\alpha$ radiation
 $\mu = 2.91 \text{ mm}^{-1}$
 $T = 173 (2) \text{ K}$
 Plate, pale yellow
 $0.35 \times 0.20 \times 0.02 \text{ mm}$

Data collection

Bruker SMART CCD
 diffractometer
 φ and ω scans
 Absorption correction: multi-scan
 (SADABS; Sheldrick, 1999)
 $T_{\min} = 0.512$, $T_{\max} = 0.951$

7803 measured reflections
 2856 independent reflections
 2689 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.018$
 $\theta_{\text{max}} = 27.0^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.020$
 $wR(F^2) = 0.048$
 $S = 1.05$
 2856 reflections
 199 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0244P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} < 0.001$
 $\Delta\rho_{\text{max}} = 0.24 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.33 \text{ e \AA}^{-3}$
 Absolute structure: Flack (1983),
 1198 Friedel pairs
 Flack parameter: 0.013 (6)

H atoms were placed in geometrically idealized positions (C–H = 0.93–0.96 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Data collection: SMART (Bruker, 1997); cell refinement: SAINT (Bruker, 1997); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 (Farrugia, 1997) and DIAMOND (Brandenburg, 1998); software used to prepare material for publication: SHELXL97.

References

Brandenburg, K. (1998). DIAMOND (Version 2.1). Crystal Impact GbR, Bonn, Germany.
 Bruker (1997). SMART (Version 5.0) and SAINT (Version 5.0). Bruker AXS Inc., Madison, Wisconsin, USA.

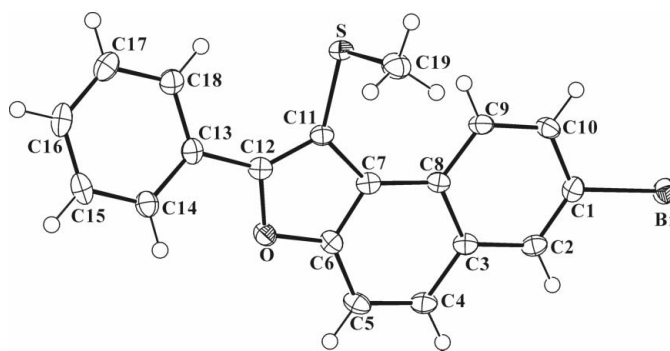


Figure 1

The molecular structure of (I), showing displacement ellipsoids drawn at the 50% probability level.

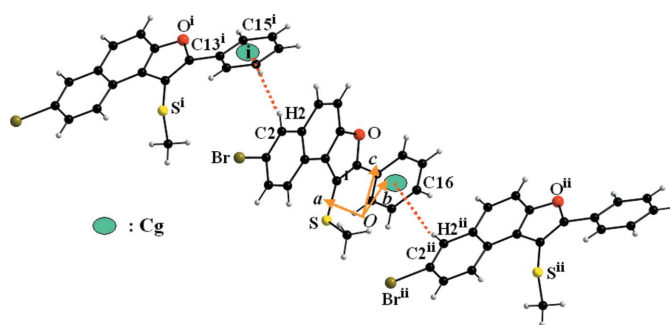


Figure 2

C–H... π interactions in (dotted lines) (I). Cg denotes ring centroids. [Symmetry codes: (i) $x + \frac{1}{2}, -y - 1, z$; (ii) $x - \frac{1}{2}, -y - 1, z$].

- Choi, H. D., Seo, P. J., Kang, B. W., Son, B. W. & Lee, U. (2006). *Acta Cryst. E* **62**, o5625–o5626.
 Choi, H. D., Seo, P. J. & Son, B. W. (1999). *J. Korean Chem. Soc.* **43**, 237–240.
 Choi, H. D., Woo, H. M., Seo, P. J., Son, B. W. & Lee, U. (2006). *Acta Cryst. E* **62**, o3881–o3882.
 Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
 Flack, H. D. (1983). *Acta Cryst. A* **39**, 876–881.
 Sheldrick, G. M. (1997). SHELXS97 and SHELXL97. University of Göttingen, Germany.
 Sheldrick, G. M. (1999). SADABS. University of Göttingen, Germany.