

Bin Chen and Yu-quan Shi*

Department of Chemistry, College of Sciences,
Tianjin University, Tianjin 300072, People's
Republic of China

Correspondence e-mail: qshi4@hotmail.com

Key indicators

Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.005$ Å
Disorder in main residue
 R factor = 0.051
 wR factor = 0.144
Data-to-parameter ratio = 14.1For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.4-Bromo-*N*-(3-thienylmethyl)-1,8-naphthalimide

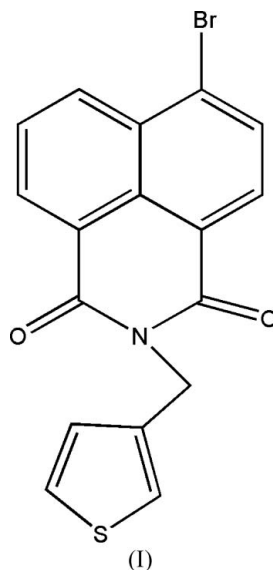
The title compound, $\text{C}_{17}\text{H}_{10}\text{BrNO}_2\text{S}$, is a new fluorescent 1,8-naphthalimide derivative. The thiophene ring is disordered over two positions. The dihedral angle between the major disorder component of the thiophene ring and the plane of the naphthalimide is $76.1(4)^\circ$. π - π Stacking interactions stabilize the crystal structure.

Received 30 May 2006

Accepted 3 July 2006

Comment

In the course of our investigation of 1,8-naphthalimide derivatives, we have prepared the title compound, (I). The head group of the naphthalimide comprises a thiophene ring is linked to the N atom of the dicarboximide ring through a methylene group. The molecular structure is shown in Fig. 1.



In the crystal structure, a π - π stacking interaction is observed between the C2-C7 aromatic ring and its symmetry-related counterpart at $(x, y - 1, z)$, with a centroid separation of 3.854 Å (Fig. 2).

Experimental

4-Bromo-1,8-naphthalimide (2.76 g, 10 mmol) and anhydrous potassium carbonate (1.38 g, 10 mmol) were added to 50 ml of dry DMF. The mixture was stirred at room temperature for 30 min and freshly distilled 3-thienyl bromide (0.88 g, 5 mmol) was added under nitrogen. The solution was light refluxed for 10 h. After cooling, the mixture was poured into water. The resulting precipitate was separated by filtration, washed with water, vacuum dried and chromatographed on silica gel, eluting with dichloromethane to give the title compound. Recrystallization from ethanol afforded pure (I) as

colourless crystals (yield: 78%, m.p. 468 K). A single crystal suitable for X-ray structure analysis was obtained by slow evaporation of an acetone and petroleum ether (2:1) solution at room temperature over 4 d. Analysis calculated for $C_{17}H_{10}BrNO_2S$: C 54.85, H 2.71, N 3.76, found: C 54.84, H 2.70, N 3.77%.

Crystal data

$C_{17}H_{10}BrNO_2S$
 $M_r = 372.23$
 Monoclinic, $P2_1/c$
 $a = 11.4000$ (16) Å
 $b = 7.2252$ (12) Å
 $c = 18.211$ (2) Å
 $\beta = 103.721$ (6)°
 $V = 1457.2$ (4) Å³

$Z = 4$
 $D_x = 1.697$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 2.97$ mm⁻¹
 $T = 294$ (2) K
 Block, colourless
 $0.24 \times 0.16 \times 0.10$ mm

Data collection

Rigaku Saturn diffractometer
 ω scans
 Absorption correction: multi-scan
 (REQAB; Jacobson, 1998)
 $T_{\min} = 0.536$, $T_{\max} = 0.756$

13117 measured reflections
 3445 independent reflections
 1897 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.047$
 $\theta_{\text{max}} = 27.8^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.051$
 $wR(F^2) = 0.144$
 $S = 0.99$
 3445 reflections
 245 parameters

H-atom parameters constrained
 $w = 1/[\sigma^2(F_o^2) + (0.0725P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$
 $\Delta\rho_{\text{max}} = 1.00$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.49$ e Å⁻³

The atoms of the thiophene ring are disordered over two positions; site-occupancy factors were refined and converged to 0.594 (6) and 0.406 (6). The C–S and C=C distances were restrained to C–S = 1.70 (1) Å and C=C = 1.34 (1) Å. All H atoms were initially located in a difference Fourier map and then constrained to an ideal geometry using a riding model, with C–H = 0.93–0.96 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$.

Data collection: *CrystalClear* (Rigaku, 1999); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *CrystalStructure* (Rigaku/MS, 2005).

We are indebted to the Natural Science Foundation of the People's Republic of China (grant No. 60071027) and the Natural Science Foundation of Tianjin City, People's Republic of China (grant No. 023603811) for financial support.

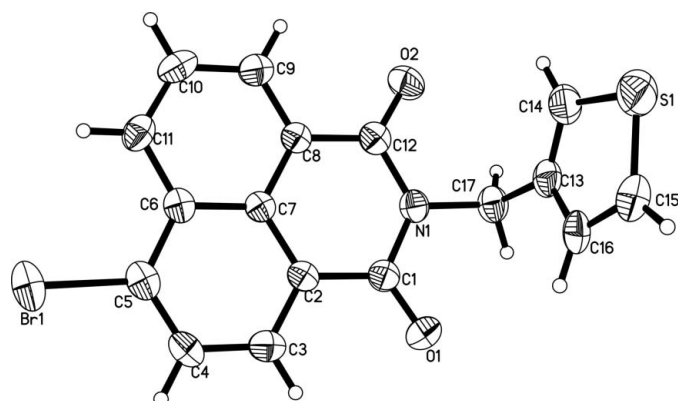


Figure 1

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. Only the major component of the disordered thiophene ring is shown.

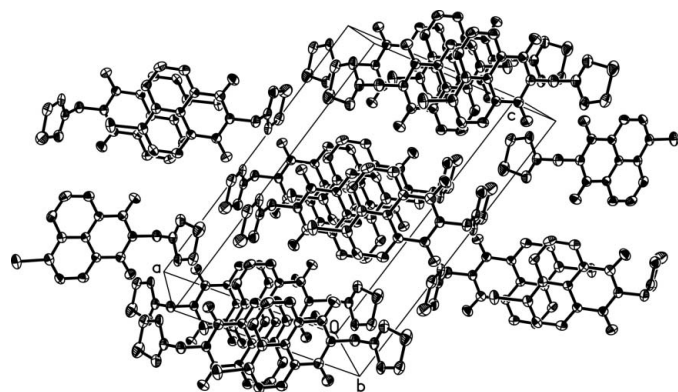


Figure 2

The molecular packing of (I) viewed along the a axis. H atoms have been omitted. Only the major disorder component is shown.

References

- Bruker (1997). *SMART, SAINT and SHELXTL* (Versions 5.10). Bruker AXS Inc., Madison, Wisconsin, USA.
 Jacobson, R. (1998). *REQAB*. Private communication to Rigaku Corporation, Tokyo, Japan.
 Rigaku (1999). *CrystalClear*. Rigaku Corporation, Tokyo, Japan.
 Rigaku/MS (2005). *CrystalStructure*. Version 3.7.0. Rigaku/MS, The Woodlands, Texas, USA.
 Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.