{2-[(2-Bromo-5-methoxybenzylidene)amino]-4,5,6,7-tetrahydrobenzo[b]thiophen-3-yl}(phenyl)methanone

Manpreet Kaur, Jerry P. Jasinski, Thammarse S. Yamuna, H. S. Yathirajan and K. Byrappa


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In the title compound, \( \text{C}_{23}\text{H}_{20}\text{BrNO}_{2}\text{S} \), disorder was modeled for the outer two C atoms of the cyclohexene ring over two sets of sites with an occupancy ratio of 0.580 (11):0.420 (11). Both rings have a half-chair conformation. The dihedral angles between the mean plane of the thiophene ring and the benzene and phenyl rings are 9.2 (2) and 66.1 (2)°, respectively. The benzene and phenyl rings are inclined to each other by 74.8 (8)°. In the crystal, molecules are linked by pairs of \( \text{C}---\text{H}--\text{O} \) hydrogen bonds, forming inversion dimers.

**Related literature**

For applications of 2-aminothiophene derivatives, see: Sabnis et al. (1999); Puterová et al. (2010). For the biological and industrial importance of Schiff bases, see: Desai et al. (2001); Karia & Parsania (1999); Samadhiya & Halve (2001); Singh & Dash (1988); Aydogan et al. (2001); Taggi et al. (2002). For a related structure, see: Kubicki et al. (2012). For puckering parameters, see: Cremer & Pople (1975). For standard bond lengths, see: Allen et al. (1987).
supplementary materials


{2-[(2-Bromo-5-methoxybenzylidene)amino]-4,5,6,7-tetrahydrobenzo[b]thiophen-3-yl}(phenyl)methanone

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1. Comment

2-Aminothiophene derivatives have been used in a number of applications in pesticides, dyes and pharmaceuticals (Sabnis et al. 1999; Puterová et al. 2010). Schiff base compounds show biological activities including antibacterial, antifungal, anticancer and herbicidal activities (Desai et al., 2001; Karia & Parsania, 1999; Samadhiya & Halve, 2001; Singh & Dash, 1988) and have been used as starting materials in the synthesis of compounds of industrial (Aydogan et al., 2001) and biological interest such as β-lactams (Taggi et al., 2002). In continuation of our work on the Schiff base derivatives of 2-aminothiophenes (Kubicki et al., 2012), we report herein on the crystal structure of the title compound.

In the title compound, Fig. 1, disorder was modeled for atoms C5 and C6 of the cyclohexene ring over two sites (A and B) with an occupancy ratio of 0.580 (11):0.420 (11). Both rings have half-chair conformations with puckering parameters (Cremer & Pople, 1975) Q, θ, and ϕ being = 0.520 (6) Å, 49.9 (4) ° and 154.8 (6) °, respectively, for ring A and being = 0.527 (8) Å, 130.1 (5) ° and 322.0 (7) °, respectively, for ring B. The dihedral angles between the mean plane of the thiophene ring and the benzene and phenyl rings are 9.2 (2) ° and 66.1 (2) °, respectively. The benzene and phenyl rings are twisted with respect to each other by 74.8 (8)°. Bond lengths are in normal ranges (Allen et al., 1987).

In the crystal, molecules are linked by pairs of C-H···O hydrogen bonds forming inversion dimers (Table 1 and Fig. 2).

2. Experimental

To a solution of (2-amino-4,5,6,7-tetrahydro-benzo[b]thiophen-3-yl)-phenyl-methanone (200 mg, 0.79 mmol) in 10 ml of methanol an equimolar amount of 2-bromo-5-methoxybenzaldehyde (170 mg, 0.79 mmol) was added with constant stirring. The mixture was then refluxed for 6 hours and a yellow precipitate was obtained. The reaction completion was confirmed by thin layer chromatography. The precipitate was filtered and dried at room temperature overnight. Slow evaporation of a solution in CH₂Cl₂ gave yellow block-like crystals of the title compound.

3. Refinement

All H atoms were placed in calculated positions and refined as riding atoms: C—H = 0.95 - 0.99 Å with Uiso(H) = 1.5Ueq(C-methyl) and = 1.2Ueq(C) for other H atoms. Atoms C5 and C6, of the tetrahydrobenzothiophenyl ring, are disordered over two sites (A and B) and were refined with an occupancy ratio of 0.574 (11):0.426 (11).
**Figure 1**
A view of the molecular structure of the title molecule, with atom labelling. Displacement ellipsoids are drawn at the 30% probability level (the minor component atoms C5B and C6B are not shown).

**Figure 2**
A view along the $b$ axis of the crystal packing of the title compound. The C—H···O hydrogen bonds are shown as dashed lines (see Table 1 for details; H atoms not involved in hydrogen bonding have been omitted for clarity; the minor component atoms C5B and C6B are not shown).
supplementary materials

| {2-[(2-Bromo-5-methoxybenzylidene)amino]-4,5,6,7-tetrahydrobenzo[b]thiophen-3-yl}(phenyl)methanone |

Crystal data

C_{23}H_{20}BrNO_2S  
Formula

\[ M_r = 454.37 \]

\[ D_r = 1.508 \text{ Mg m}^{-3} \]

\[ \text{Monoclinic, } P_{2_1}/n \]

\[ a = 8.84813 \ (17) \ \text{Å} \]

\[ b = 12.5563 \ (2) \ \text{Å} \]

\[ c = 18.4384 \ (4) \ \text{Å} \]

\[ \beta = 102.363 \ (2)° \]

\[ V = 2001.00 \ (7) \ \text{Å}^3 \]

\[ Z = 4 \]

\[ F(000) = 928 \]

\[ D_x = 1.508 \text{ Mg m}^{-3} \]

\[ \text{Cell parameters from 5917 reflections} \]

\[ \theta = 4.3–71.4° \]

\[ \mu = 3.92 \text{ mm}^{-1} \]

\[ T = 173 \text{ K} \]

Irregular, yellow

\[ 0.26 \times 0.22 \times 0.14 \text{ mm} \]

Data collection

Agilent Eos Gemini

diffractometer

Radiation source: Enhance (Cu) X-ray Source

\[ \omega \text{ scans} \]

Absorption correction: multi-scan

\[ \text{Cu } K\alpha \text{ radiation, } \lambda = 1.54184 \ \text{Å} \]

\[ \text{Cell parameters from } 5917 \text{ reflections} \]

\[ \theta_{\text{min}} = 0.725, \ \theta_{\text{max}} = 1.000 \]

12404 measured reflections

3853 independent reflections

3440 reflections with \( I > 2\sigma(I) \)

\[ R_{\text{int}} = 0.034 \]

\[ \theta_{\text{max}} = 71.3°, \ \theta_{\text{min}} = 4.3° \]

h = −10→10

k = −15→13

l = −22→22

Refinement

Refinement on \( F^2 \)

Least-squares matrix: full

\[ R(F^2) = 0.032 \]

\[ wR(F^2) = 0.087 \]

S = 1.05

3853 reflections

273 parameters

0 restraints

Hydrogen site location: inferred from

neighbouring sites

H-atom parameters constrained

\[ \Delta \rho_{\text{max}} = 0.56 \text{ e Å}^{-3} \]

\[ \Delta \rho_{\text{min}} = -0.31 \text{ e Å}^{-3} \]

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance

matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles;

correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate

(isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

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<th>( U_{\text{iso}} )</th>
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### Atomic displacement parameters (Å²)

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supplementary materials

C3—C4—C5B—C6B  $-53.2$ (8)  
C4—C3—C8—S1  $-179.33$ (15)  
C4—C3—C8—C7  $-0.1$ (3)  
C4—C5A—C6A—C7  $-67.6$ (8)  
C4—C5B—C6B—C7  $65.5$ (9)  
C5A—C4—C5B—C6B  $43.5$ (7)  
C5A—C6A—C7—C6B  $-51.1$ (7)  
C5A—C6A—C7—C8  $49.3$ (7)  
C6A—C7—C8—S1  $161.2$ (4)  
C6A—C7—C8—C3  $-17.9$ (4)  
C5B—C4—C5A—C6A  $-48.4$ (6)  
C5B—C6B—C7—C6A  $46.2$ (7)  

C16—C17—C18—C19  $-176.87$ (19)  
C16—C17—C22—C21  $178.74$ (18)  
C17—C18—C22—C21  $-2.4$ (3)  
C18—C17—C22—C21  $-0.9$ (3)  
C18—C19—C20—C21  $0.2$ (3)  
C19—C18—C20—C21  $177.5$ (2)  
C18—C19—C20—C21  $2.7$ (3)  
C19—C20—C21—C22  $1.6$ (3)  
C19—C20—C21—C22  $1.6$ (3)  
C20—C21—C22—C17  $-1.3$ (3)  
C20—C21—C22—C17  $176.77$ (14)  
C20—C21—C22—C17  $-176.77$ (14)  
C20—C21—C22—C17  $2.7$ (3)  
C20—C21—C22—C17  $166.49$ (19)  
C20—C21—C22—C17  $-12.6$ (3)  

Hydrogen-bond geometry ($\text{Å}$, $^\circ$)

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Symmetry code: (i) $-x$, $-y+1$, $-z+1$. 