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Key indicators

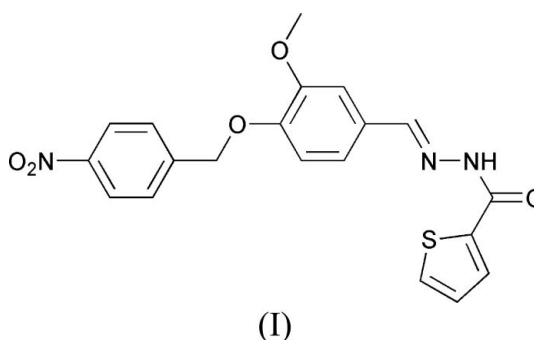
Single-crystal X-ray study
 $T = 294$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.047
 wR factor = 0.125
Data-to-parameter ratio = 11.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.**(E)-N'-[3-Methoxy-4-(4-nitrobenzyloxy)benzylidene]thiophene-2-carbohydrazide**

In the title compound, $\text{C}_{20}\text{H}_{17}\text{N}_3\text{O}_5\text{S}$, the vanillin group makes dihedral angles of 72.33 (7) and 3.72 (14) $^\circ$ with the nitrobenzene ring and the thiophene mean plane, respectively. The packing is stabilized by intermolecular $\text{N}-\text{H}\cdots\text{O}$ hydrogen bonds that form inversion-related dimers.

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Comment

For the background to this study, see a previous paper (Zhang *et al.*, 2006). We report here the synthesis and structure of the title compound, (I).



In the molecule of compound (I) (Fig. 1), the vanillin group (C8–C13/C15/O3/O4) is nearly planar, with an r.m.s. deviation for fitted atoms of 0.0378 Å. This plane makes dihedral angles of 3.72 (14) and 72.33 (7) $^\circ$ with the thiophene ring (C17–C20/S1) and the terminal benzene ring (C1–C6), respectively. The dihedral angle between the thiophene and benzene rings is 70.44 (9) $^\circ$. All bond lengths and angles are within normal ranges (Allen *et al.*, 1987).

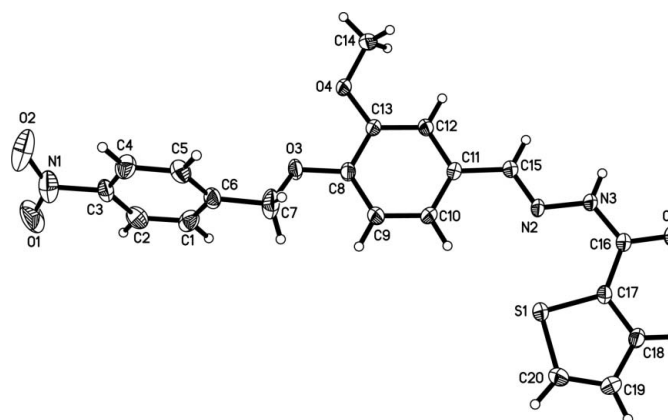


Figure 1

The molecular structure of (I), with displacement ellipsoids drawn at the 30% probability level.

The crystal packing of (I) is stabilized by intermolecular N—H...O=C hydrogen bonds that form inversion-related dimers (Table 1 and Fig. 2).

Experimental

An anhydrous ethanol solution (50 ml) of 4-(4-nitrobenzyloxy)-3-methoxybenzaldehyde (2.87 g, 10 mmol) was added to an anhydrous ethanol solution (50 ml) of thiophene-2-carbohydrazide (1.42 g, 10 mmol) and the mixture was stirred at 350 K for 5 h under nitrogen, giving a yellow precipitate. The product was isolated, recrystallized from acetonitrile and then dried in a vacuum to give the pure compound in 82% yield. Yellow single crystals of (I) suitable for X-ray analysis were obtained by slow evaporation of an acetonitrile solution.

Crystal data

| | |
|-------------------------------|---|
| $C_{20}H_{17}N_3O_5S$ | $V = 933.0 (3) \text{ \AA}^3$ |
| $M_r = 411.44$ | $Z = 2$ |
| Triclinic, $P\bar{1}$ | $D_x = 1.464 \text{ Mg m}^{-3}$ |
| $a = 8.3628 (17) \text{ \AA}$ | Mo $K\alpha$ radiation |
| $b = 9.6991 (19) \text{ \AA}$ | $\mu = 0.21 \text{ mm}^{-1}$ |
| $c = 11.635 (2) \text{ \AA}$ | $T = 294 (2) \text{ K}$ |
| $\alpha = 90.89 (3)^\circ$ | Block, yellow |
| $\beta = 98.02 (3)^\circ$ | $0.14 \times 0.12 \times 0.08 \text{ mm}$ |
| $\gamma = 92.96 (3)^\circ$ | |

Data collection

| | |
|---|--|
| Bruker SMART APEX CCD area-detector diffractometer | 5786 measured reflections |
| φ and ω scans | 3272 independent reflections |
| Absorption correction: multi-scan (SADABS; Sheldrick, 1996) | 2220 reflections with $I > 2\sigma(I)$ |
| $T_{\min} = 0.952, T_{\max} = 0.983$ | $R_{\text{int}} = 0.038$ |
| | $\theta_{\text{max}} = 25.0^\circ$ |

Refinement

| | |
|---------------------------------|--|
| Refinement on F^2 | H-atom parameters constrained |
| $R[F^2 > 2\sigma(F^2)] = 0.048$ | $w = 1/[\sigma^2(F_o^2) + (0.07P)^2]$ |
| $wR(F^2) = 0.125$ | where $P = (F_o^2 + 2F_c^2)/3$ |
| $S = 1.02$ | $(\Delta/\sigma)_{\text{max}} < 0.001$ |
| 3272 reflections | $\Delta\rho_{\text{max}} = 0.26 \text{ e \AA}^{-3}$ |
| 281 parameters | $\Delta\rho_{\text{min}} = -0.24 \text{ e \AA}^{-3}$ |

Table 1

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

| $D-H\cdots A$ | $D-H$ | $H\cdots A$ | $D\cdots A$ | $D-H\cdots A$ |
|--------------------|-------|-------------|-------------|---------------|
| $N3-H3\cdots O5^i$ | 0.86 | 1.95 | 2.812 (3) | 179 |

Symmetry code: (i) $-x + 2, -y, -z + 1$.

H atoms were included in calculated positions and refined using a riding-model approximation, with C—H = 0.93 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for Csp^2 H atoms, C—H = 0.97 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for methylene H atoms, C—H = 0.96 \AA and $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for

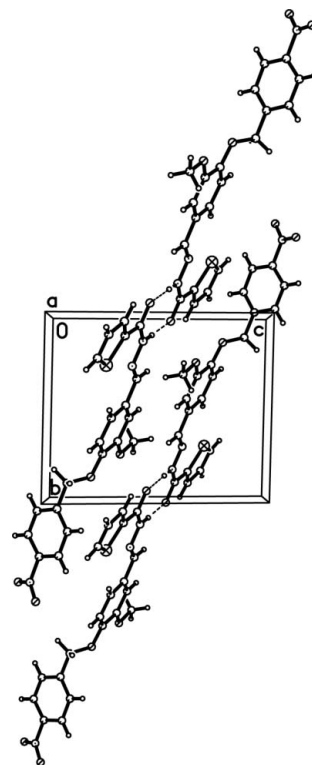


Figure 2

A packing diagram for (I), with hydrogen bonds drawn as dashed lines.

methyl H atoms and N—H = 0.86 \AA and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{N})$ for imino H atoms.

The two O atoms (O1 and O2) of the nitro group (O1/N1/O2) are disordered over two different positions, O1' and O2', with occupancies constrained to be 0.70 and 0.30.

Data collection: SMART (Bruker, 1999); cell refinement: SAINT (Bruker, 1999); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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