

1-(4-Nitrophenyl)-3-(2-thienyl)prop-2-en-1-one

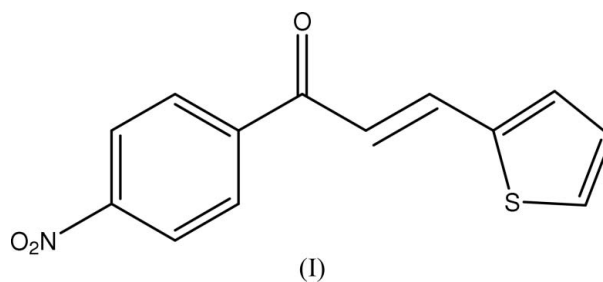
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Key indicators

Single-crystal X-ray study
T = 100 K
Mean $\sigma(\text{C}-\text{C}) = 0.002 \text{ \AA}$
Disorder in main residue
R factor = 0.052
wR factor = 0.152
Data-to-parameter ratio = 32.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.The thiophene ring of the title compound, $\text{C}_{13}\text{H}_9\text{NO}_3\text{S}$, is disordered. An approximate 180° rotation of the thiophene ring with respect to the central C—C single bond is observed in the molecule.Received 7 July 2006
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Comment

The synthesis and study of molecular compounds with nonlinear optical (NLO) properties has attracted much attention, because such materials hold promise for applications in optoelectronic and photonic devices (Bosshard *et al.*, 1995; Nalwa & Miyata, 1997). In order to create efficient quadratic (second-order) NLO materials, both the molecular and bulk properties must be optimized. The majority of promising compounds constitute dipolar donor– π –acceptor ($D-\pi-A$) molecules and these must be arranged non-centrosymmetrically in order to afford macroscopic structures capable of showing bulk quadratic NLO effects, such as frequency doubling (second-harmonic generation, SHG).With appropriate substituents, chalcones are a class of NLO materials (Fichou *et al.*, 1988; Kitaoka *et al.*, 1990; Uchida *et al.*, 1998; Goto *et al.*, 1991; Patil, Ng *et al.*, 2006; Patil, Teh *et al.*, 2006a,b; Zhang *et al.*, 1990; Zhao *et al.*, 2000). In this work we present the single-crystal X-ray structural study of the title compound, (I). Crystals of (I) do not exhibit second-order NLO properties as it crystallizes in a centrosymmetric space group.In (I) (Fig. 1), the thiophene ring is disordered over two sites (atoms of the minor occupancy component are labelled with the suffix *X*). This corresponds to an approximate 180° rotation of the thiophene ring with respect to C9—C10. Bond lengths and angles have normal values (Allen *et al.*, 1987) and are comparable to those observed in related structures (Teh *et al.*, 2006a,b; Patil, Ng *et al.*, 2006; Patil, Teh *et al.*, 2006a,b), except for some parameters of the thiophene ring, which are probably a consequence of the disorder. The nitro group at C3 is almost coplanar with the C1—C6 benzene ring, with O2—N1—C3—C4 and O3—N1—C3—C2 torsion angles of 0.3 (2)

and 0.9 (2)^o, respectively. No hydrogen bond or short contact is found in the crystal structure.

Experimental

2-Thiophenecarboxyaldehyde (0.1 mol) and 4-nitroacetophenone (0.1 mol) were stirred in 100 ml of ethanol at 298 K. An aqueous NaOH solution (10 g, 10%) was added and the mixture was stirred for 2 h. The precipitate that formed was filtered off, washed with water and dried. The resulting crude product was recrystallized twice with acetone. The purity of the compound was checked by thin-layer chromatography. Crystals suitable for single-crystal X-ray diffraction experiments were grown by slow evaporation of an acetone solution of (I).

Crystal data

C ₁₃ H ₉ NO ₃ S	Z = 4
M _r = 259.27	D _x = 1.514 Mg m ⁻³
Monoclinic, P2 ₁ /c	Mo K α radiation
a = 5.9056 (1) Å	μ = 0.28 mm ⁻¹
b = 28.7358 (5) Å	T = 100.0 (1) K
c = 6.9265 (2) Å	Block, yellow
β = 104.568 (1) ^o	0.30 × 0.25 × 0.11 mm
V = 1137.65 (4) Å ³	

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	22840 measured reflections
ω scans	5833 independent reflections
Absorption correction: multi-scan (SADABS; Bruker, 2005)	3745 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.920$, $T_{\max} = 0.970$	$R_{\text{int}} = 0.054$
	$\theta_{\text{max}} = 37.5^{\circ}$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0697P)^2 + 0.0132P]$
$R[F^2 > 2\sigma(F^2)] = 0.052$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.152$	$(\Delta/\sigma)_{\text{max}} < 0.001$
S = 1.08	$\Delta\rho_{\text{max}} = 0.43 \text{ e } \text{Å}^{-3}$
5833 reflections	$\Delta\rho_{\text{min}} = -0.32 \text{ e } \text{Å}^{-3}$
179 parameters	
H-atom parameters constrained	

H atoms were placed in calculated positions, with C–H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$. The ratio of the refined occupancies for the major and minor components of the disordered thiophene ring is 0.926 (3):0.074 (3). Similarity and rigid-bond restraints were applied to the disordered atoms.

Data collection: APEX2 (Bruker, 2005); cell refinement: APEX2; data reduction: SAINT (Bruker, 2005); program(s) used to solve structure: SHELXTL (Sheldrick, 1998); program(s) used to refine structure: SHELXTL; molecular graphics: SHELXTL; software used to prepare material for publication: SHELXTL and PLATON (Spek, 2003).

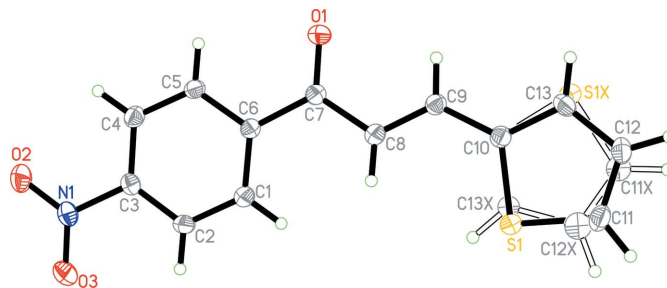


Figure 1

The molecular structure of (I), showing 50% probability displacement ellipsoids and the atomic numbering. Open bonds indicate the minor disorder component.

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