

Xian-Qiang Huang,^{a*} Hui Wang,^b Jian-Min Dou,^a Da-Qi Wang^a and Jin-Xian Wang^c^aDepartment of Chemistry, Liaocheng University, Liaocheng 252059, People's Republic of China, ^bCollege of Life Sciences, Shandong University, Jinan 250100, People's Republic of China, and ^cDepartment of Chemistry, Northwest Normal University, Lanzhou 730070, People's Republic of China

Correspondence e-mail: hxqqxh2008@163.com

Key indicators

Single-crystal X-ray study
 $T = 298$ K
Mean $\sigma(C-C) = 0.006$ Å
Disorder in main residue
 R factor = 0.061
 wR factor = 0.200
Data-to-parameter ratio = 13.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

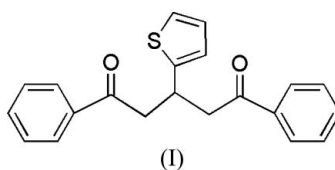
1,5-Diphenyl-3-(2-thienyl)pentane-1,5-dione

The title compound, $C_{21}H_{18}O_2S$, was synthesized by the reaction of thiophene-2-carbaldehyde with acetophenone and NaOH. All bond lengths and angles show normal values. The crystal packing is stabilized by weak intermolecular C—H \cdots O hydrogen bonds and van der Waals forces.

Received 4 August 2006
Accepted 15 August 2006

Comment

The drive towards clean technology has encouraged the application of solvent-free conditions in industrial chemistry (Dittmer, 1997). A move away from the use of solvents in organic synthesis has led in some cases to improved results and more benign synthetic procedures (Kidwai *et al.*, 2001). Adopting the principles of 'green chemistry', we have established that using solvent-free conditions for condensation and Michael reactions results in a dramatic improvement in yields. Our approach reduces the use of organic solvents, which are potentially toxic and hazardous (Anastas & Varner, 1998), and uses simple and mild conditions, with inherently lower costs (Tanaka & Toda, 2000). In view of the current emphasis on 'green chemistry', we set out to develop a solvent-free preparation of coumarins using an inexpensive and non-polluting catalyst.



The synthesis of substituted thiophenes has attracted a great deal of interest over the years due to their presence in

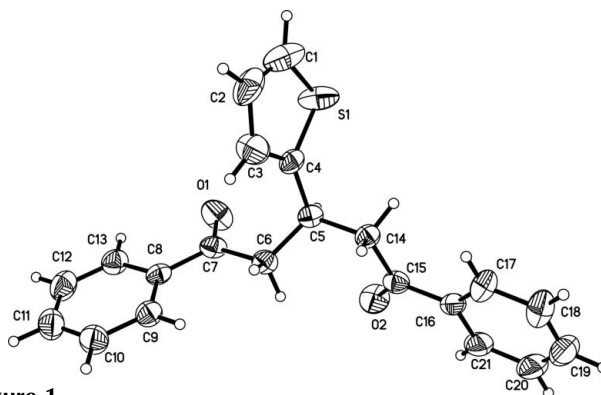


Figure 1
View of (I), showing the atomic labelling and displacement ellipsoids drawn at the 30% probability level. Only major components of the disordered S1 and C3 atoms are shown.

natural products (Koike *et al.*, 1999). 1,5-Diketones are important synthetic intermediates and starting materials in the synthesis of many heterocyclic compounds (Hirsch & Bailey, 1978; Krohnke, 1976). In a continuation of previous work on the synthesis of 1,5-diketones (Constable *et al.*, 1998; Fuchigami *et al.*, 1986), we present a new title compound, (I), synthesized under solvent-free conditions.

In (I) (Fig. 1), the bond lengths and angles are normal and similar to those observed in 1,3,5-triphenylpentane-1,5-dione (Das *et al.*, 1994). Weak intermolecular C—H...O hydrogen bonds (Table 1) link the molecules into chains extending along the *c* axis. The crystal packing (Fig. 2) is further stabilized by van der Waals forces.

Experimental

Acetophenone (0.75 g, 6.25 mmol), freshly distilled thiophene-2-carbaldehyde (0.35 g, 3.125 mmol) and NaOH (0.25 g, 6.25 mmol) were aggregated with a glass paddle in an open flask. The resulting mixture was washed with water several times to remove NaOH and was recrystallized from ethanol, affording the title compound as a crystalline solid.

Crystal data

$C_{21}H_{18}O_2S$	$Z = 4$
$M_r = 334.41$	$D_x = 1.272 \text{ Mg m}^{-3}$
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation
$a = 13.460 (2) \text{ \AA}$	$\mu = 0.20 \text{ mm}^{-1}$
$b = 12.094 (3) \text{ \AA}$	$T = 298 (2) \text{ K}$
$c = 10.762 (2) \text{ \AA}$	Block, colourless
$\beta = 94.456 (3)^\circ$	$0.58 \times 0.55 \times 0.53 \text{ mm}$
$V = 1746.6 (6) \text{ \AA}^3$	

Data collection

Bruker SMART CCD area-detector diffractometer	8511 measured reflections
φ and ω scans	3061 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	1620 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.896$, $T_{\max} = 0.904$	$R_{\text{int}} = 0.060$
	$\theta_{\text{max}} = 25.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0999P)^2 + 0.3043P]$
$R[F^2 > 2\sigma(F^2)] = 0.061$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.200$	$(\Delta/\sigma)_{\text{max}} < 0.001$
$S = 1.00$	$\Delta\rho_{\text{max}} = 0.23 \text{ e \AA}^{-3}$
3061 reflections	$\Delta\rho_{\text{min}} = -0.31 \text{ e \AA}^{-3}$
224 parameters	
H-atom parameters constrained	

Table 1

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C6-H6B\cdots O1^i$	0.97	2.47	3.361 (7)	153

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

All H atoms were positioned geometrically and refined using a riding model, with C—H = 0.93–0.98 \AA and $U_{\text{iso}}(\text{H}) = 1.2\text{--}1.5U_{\text{eq}}(\text{C})$. Atoms S1, C3 and H3 were treated as disordered between two positions, with refined occupancies of 0.735 (5) and 0.265 (5).

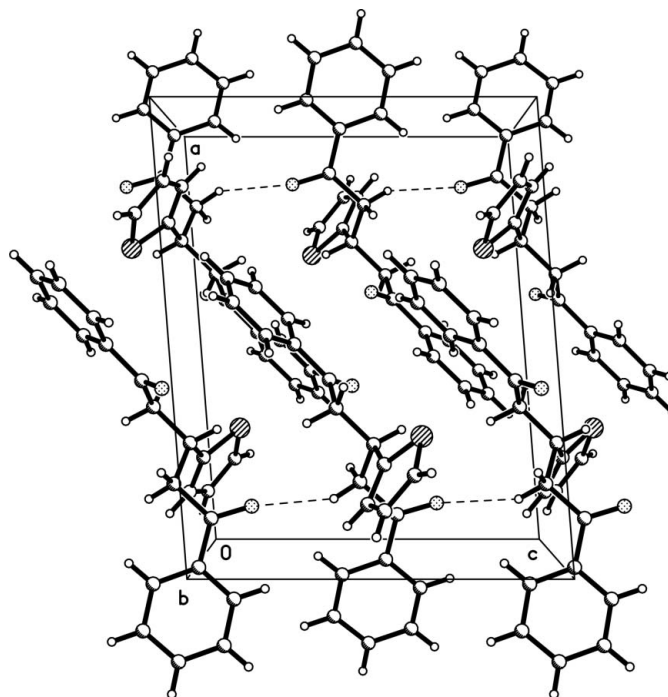


Figure 2

The crystal packing of (I), viewed along the *b* axis. The dashed lines denote C—H...O hydrogen bonds. Only the major components of the disordered S1, C3 and H3 atoms are shown for each molecule.

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); 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.

The authors acknowledge financial support from the National Natural Science Foundation of Liao Cheng University (grant No. X051040).

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