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Key indicators

Single-crystal X-ray study

T = 293 K

Mean $\sigma(\text{C}-\text{C}) = 0.004 \text{ \AA}$

R factor = 0.043

wR factor = 0.112

Data-to-parameter ratio = 12.1

For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

2-Acetylphenyl 5-methylthiophene-2-carboxylate

The non-H atoms of the molecule of the title compound, $\text{C}_{14}\text{H}_{12}\text{O}_3\text{S}$, form three essentially planar fragments, *viz.* the thiophene ring with the methyl C atom and carboxylate group (maximum deviation 0.041 Å; fragment *A*), the oxyphenyl ring with the central acetyl C atom (0.029 Å; *B*), and the acetyl group together with the phenyl C atom bonded to it (<0.001 Å; *C*). These planes form dihedral angles of 88.33 (7)° for *A/B* and 71.9 (2)° for *B/C*. The crystal structure is stabilized by intermolecular interactions of the C—H...O type [$\text{C}\cdots\text{O} = 3.492 (5) \text{ \AA}$].

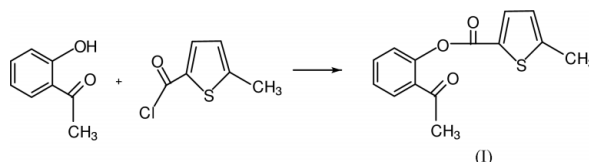
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Comment

The title compound, (I), is a precursor used for the synthesis of 5-(4*H*-4-oxo-1-benzopyran-2-yl)-2-thiophenecarboxaldehyde (Göker *et al.*, 2000). The synthesis of (I) was performed by the esterification of 2'-hydroxyacetophenone with 5-methyl-2-thiophenecarboxylic acid chloride in pyridine, as is shown in the Scheme. The structure of (I) was assigned based on the NMR, mass spectroscopy and elemental analysis. Here we report the results of the X-ray diffraction study.



An *ORTEP*III (Burnett & Johnson, 1996) plot of (I) is shown in Fig. 1. The molecule of (I) is composed of three essentially planar fragments: the thiophene ring with the adjacent methyl and carboxylate groups is planar within 0.041 Å, the oxyphenyl group with the central acetyl atom shows a maximum deviation of 0.029 Å, and the acetyl group with the phenyl C atom, bonded to it, is planar within the precision of the experiment (maximum deviation less than 0.001 Å). The planes of the first two fragments are orthogonal to each other [dihedral angle is equal to 88.33 (7)°]; the *ortho*-acetyl group plane is also almost normal to the benzene ring [dihedral angle 71.9 (2)°].

The crystal structure is stabilized by intermolecular interactions of the C—H...O type [$\text{C14}\cdots\text{O2}^i$ 3.492 (5) Å, $\text{C14}-\text{H143}\cdots\text{O2}^i$ 0.97 (4) Å and $\text{C14}-\text{H143}\cdots\text{O2}^i$ 154 (3)°; symmetry code: (i) $x - 1, y, z$].

Experimental

5-Methyl-2-thiophenecarboxylic acid (8 g, 56.3 mmol) in 50 ml of SOCl_2 was refluxed for 2 h; the excess of the reagent was then

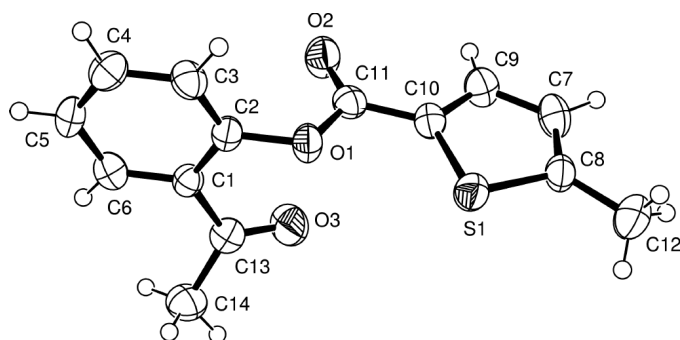


Figure 1

An ORTEPIII drawing of the title compound with the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level; H atoms are shown as small spheres of arbitrary radius.

evaporated under reduced pressure. *o*-Hydroxyacetophenone (7.65 g, 56.3 mmol) and 20 ml of pyridine were added to the residue and the mixture was heated for 0.5 h at 353 K. The mixture was then cooled and poured into ice water, acidified with HCl, and the resulting precipitate was filtered and washed with water. Crystallization from EtOH gave (I) (12.7 g, 86.8%) as colourless crystals, m.p. 383–385 K. Analysis calculated for $C_{14}H_{12}O_3S$: C 64.60, H 4.65, S 12.32%; found: C 64.55, H 4.64, S 12.16%.

Crystal data

$C_{14}H_{12}O_3S$
 $M_r = 260.30$
 Orthorhombic, *Pbca*
 $a = 8.0088$ (11) Å
 $b = 14.3163$ (16) Å
 $c = 22.103$ (3) Å
 $V = 2534.2$ (5) Å³
 $Z = 8$
 $D_x = 1.364$ Mg m⁻³

Mo $K\alpha$ radiation
 Cell parameters from 25 reflections
 $\theta = 2.6$ – 26.3°
 $\mu = 0.25$ mm⁻¹
 $T = 293$ (2) K
 Prism, colourless
 $0.30 \times 0.25 \times 0.20$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan (North *et al.*, 1968)
 $T_{\min} = 0.928$, $T_{\max} = 0.951$
 2551 measured reflections
 2551 independent reflections

1275 reflections with $I > 2\sigma(I)$
 $\theta_{\max} = 26.3^\circ$
 $h = 0 \rightarrow 9$
 $k = 0 \rightarrow 17$
 $l = 0 \rightarrow 27$
 3 standard reflections
 frequency: 120 min
 intensity decay: 1%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.043$
 $wR(F^2) = 0.112$
 $S = 0.98$
 2551 reflections
 211 parameters

All H-atom parameters refined
 $w = 1/[\sigma^2(F_o^2) + (0.0431P)^2]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.20$ e Å⁻³
 $\Delta\rho_{\min} = -0.24$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

S1—C8	1.718 (3)	O2—C11	1.203 (3)
S1—C10	1.717 (3)	O3—C13	1.214 (3)
O1—C2	1.407 (3)	C8—C12	1.492 (5)
O1—C11	1.359 (3)	C10—C11	1.459 (4)
C10—S1—C8	91.83 (14)	C2—C1—C13	122.0 (3)
C11—O1—C2	115.4 (2)	C3—C2—C1	121.8 (3)
C6—C1—C2	117.4 (3)	C3—C2—O1	118.1 (3)
C6—C1—C13	120.6 (3)		

The H atoms were located in a difference map and refined isotropically. The C—H bond distances range from 0.89 (3) to 0.99 (5) Å, while U_{iso} values for H atoms are in the range 0.036–0.109 Å².

Data collection: CAD-4 EXPRESS (Enraf–Nonius, 1994); cell refinement: CAD-4 EXPRESS; data reduction: XCAD4 (Harms & Wocadlo, 1995); program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: ORTEP-3 for Windows (Farrugia, 1997); software used to prepare material for publication: WinGX (Farrugia, 1999).

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