2-Acetylphenyl 5-methylthiophene-2-carboxylate

The non-H atoms of the molecule of the title compound, C14H12O3S, 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 [C—O = 3.492 (5) Å].

Comment

The title compound, (I), is a precursor used for the synthesis of 5-(4H-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.

Experimental

5-Methyl-2-thiophenecarboxylic acid (8 g, 56.3 mmol) in 50 ml of SOCl2 was refluxed for 2 h; the excess of the reagent was then
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 C14H12O3S: C 64.60, H 4.65, S 12.32%; found: C 64.55, H 4.64, S 12.16%.

Crystal data

C14H12O3S
Mr = 260.30
Orthorhombic, Pbcn
a = 8.0088 (11) Å
b = 14.3163 (16) Å
c = 22.103 (3) Å
V = 2534.2 (5) Å³
Z = 8
D₀ = 1.364 Mg m⁻³

Data collection

Enraf-Nonius CAD-4
diffractometer 
ω/2θ scans
Absorption correction: ψ scan
(North et al., 1968)
Tmin = 0.928, Tmax = 0.951
2551 measured reflections
2551 independent reflections
Mo Kα radiation
Cell parameters from 25 reflections
θ = 2.6–26.3°
μ = 0.25 mm⁻¹
T = 293 (2) K
Prism, colourless
0.30 × 0.25 × 0.20 mm
1275 reflections with I > 2σ(I)
θmax = 26.3°
h = 0 → 9
k = 0 → 17
l = 0 → 27
3 standard reflections
frequency: 120 min
intensity decay: 1%

Reﬁnement

Reﬁnement on F²
R(F²) = 0.043
wR(F²) = 0.112
S = 0.98
2551 reﬂections
211 parameters
All H-atom parameters reﬁned
w = 1/[σ²(F⁰²) + (0.0431P)²]
where P = (F⁰² + 2Fc²)/3
(Δρ)max < 0.001
Δρmax = 0.20 e Å⁻³
Δρmin = −0.24 e Å⁻³

Table 1

Selected geometric parameters (Å, °).

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<tbody>
<tr>
<td>S1—C8</td>
<td>1.718 (3)</td>
<td>O2—C11</td>
<td>1.203 (3)</td>
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<tr>
<td>S1—C10</td>
<td>1.717 (3)</td>
<td>O3—C13</td>
<td>1.214 (3)</td>
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<td>O1—C2</td>
<td>1.407 (3)</td>
<td>C8—C12</td>
<td>1.492 (5)</td>
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<tr>
<td>O1—C11</td>
<td>1.359 (3)</td>
<td>C10—C11</td>
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<tr>
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<td>91.83 (14)</td>
<td>C2—C1—C13</td>
<td>122.0 (3)</td>
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<td>115.4 (2)</td>
<td>C3—C2—C1</td>
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<td>C6—C1—C13</td>
<td>117.4 (3)</td>
<td>C3—C2—O1</td>
<td>118.1 (3)</td>
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<tr>
<td>C6—C1—C13</td>
<td>120.6 (3)</td>
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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 Uiso 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).

References