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Key indicators

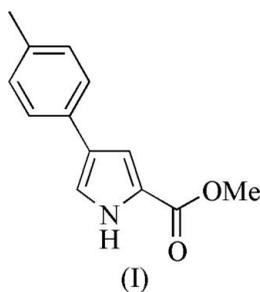
Single-crystal X-ray study
 $T = 193$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
Disorder in main residue
 R factor = 0.044
 wR factor = 0.135
Data-to-parameter ratio = 13.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Methyl 4-*p*-tolyl-1*H*-pyrrole-2-carboxylate

The molecules of the title compound, $\text{C}_{13}\text{H}_{13}\text{NO}_2$, are close to planar [maximum deviation of 0.0753 (15) Å from the least-squares plane defined by all non-H atoms in the molecule]. Molecules form centrosymmetric dimers through N—H...O hydrogen bonding. Molecules further associate through edge-to-face π -stacking between each face of the *p*-tolyl substituent and *ortho* H atoms of the *p*-tolyl units of adjacent molecules.

Received 18 December 2006
Accepted 19 December 2006

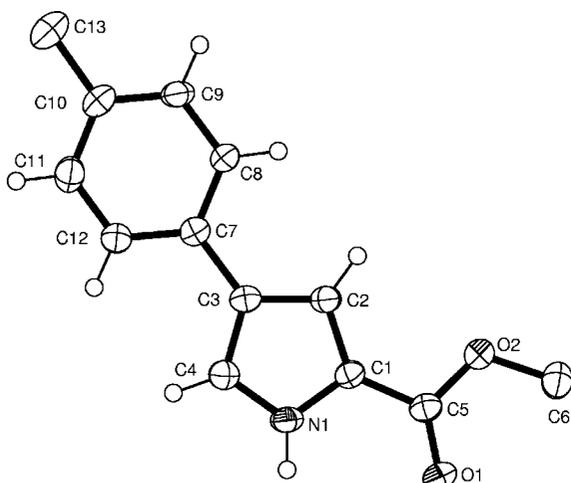
Comment

Aryl pyrroles are a common structural motif in many natural products and there has been much research towards synthetic methods for their preparation (Banwell *et al.*, 2006). Recently, we exploited phosphine-free conditions for the Suzuki–Miyaura arylation of iodopyrroles (Smith *et al.*, 2006). Here, we report the synthesis, NMR spectroscopic characterization and X-ray crystal structure of the previously unreported 4-aryl derivative methyl 4-*p*-tolyl-1*H*-pyrrole-2-carboxylate, (I).

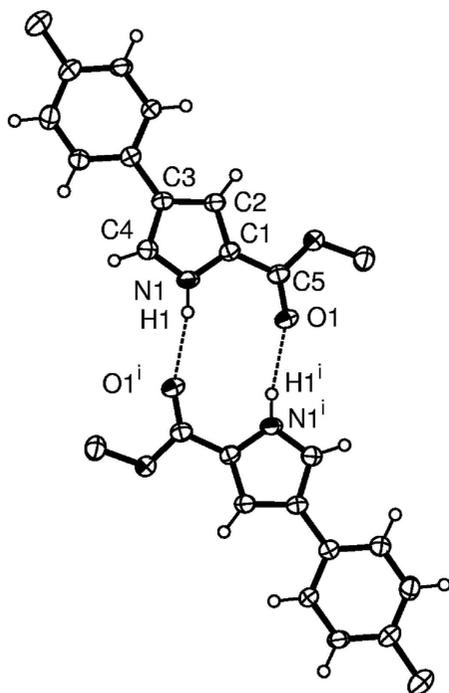


The molecular structure of (I) is depicted in Fig. 1, confirming the ring position of the *p*-tolyl substituent. The *p*-tolyl and carboxylate substituents lie close to the pyrrolyl ring plane, with the dihedral angles between adjacent rings being 4.30 (12) and 4.52 (12)°, respectively. As a result, the molecule of (I) is close to planar, with a maximum deviation of 0.0753 (15) Å for atom N1 from the least-squares plane defined by the C, N, O atoms.

Molecules of (I) form centrosymmetric dimers in the solid state through N—H...O hydrogen bonding, involving the pyrrolyl N—H and carbonyl O atom of the ester functionality, giving rise to ten-membered $\text{C}_4\text{N}_2\text{O}_2\text{H}_2$ rings (Fig. 2 and Table 1). The molecular planes of the constituent molecules are somewhat offset, with the CHNO centres of the hydrogen-bonded ring of each molecule lying out of the related partial molecular plane of the other molecule by between 0.581 (5) and 0.618 (5) Å. In addition, π -stacking between the 4-*p*-tolyl substituents is apparent, such that *ortho* H atoms of the *p*-tolyl units of two adjacent molecules interact with each face of the 4-*p*-tolyl substituent. This gives rise to an infinite three-dimensional arrangement of molecules in the solid state.

**Figure 1**

The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Disordered methyl H atoms have been omitted for clarity.

**Figure 2**

A view of the hydrogen-bonding interactions between molecules of (I), shown as dashed lines. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Disordered methyl H atoms have been omitted for clarity. [Symmetry code: (i) $2 - x, 2 - y, -z$.]

Experimental

Compound (I) was prepared according to our literature method (Smith *et al.*, 2006) as colourless crystals in 55% yield. Crystals suitable for X-ray analysis were obtained by recrystallization from diethyl ether–hexane (slow evaporation of the solution in air following filtration of a dilute solution prepared by the addition of diethyl ether to solubilise the suspension of the compound in hexane) (m.p. 448–450 K).

Crystal data

$C_{13}H_{13}NO_2$
 $M_r = 215.24$
 Monoclinic, $P2_1/n$
 $a = 7.7119$ (15) Å
 $b = 5.4554$ (11) Å
 $c = 26.385$ (5) Å
 $\beta = 94.61$ (3)°
 $V = 1106.5$ (4) Å³

$Z = 4$
 $D_x = 1.292$ Mg m⁻³
 Mo $K\alpha$ radiation
 $\mu = 0.09$ mm⁻¹
 $T = 193$ (2) K
 Block, colourless
 $0.55 \times 0.55 \times 0.45$ mm

Data collection

Enraf–Nonius TurboCAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: none
 1975 measured reflections
 1931 independent reflections

1648 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.020$
 $\theta_{max} = 25.0^\circ$
 3 standard reflections
 frequency: 60 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.044$
 $wR(F^2) = 0.135$
 $S = 1.08$
 1931 reflections
 146 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0637P)^2 + 0.8352P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.25$ e Å⁻³
 $\Delta\rho_{min} = -0.27$ e Å⁻³
 Extinction correction: *SHELXL97*
 (Sheldrick, 1997)
 Extinction coefficient: 0.028 (4)

Table 1

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$N1-H1\cdots O1^i$	0.88	2.12	2.955 (2)	159

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

All H atoms were placed in calculated positions and refined using a riding model, with C–H distances in the range 0.88–0.98 Å and with $U_{iso}(H) = 1.2U_{eq}(C)$, except for methyl H atoms, where $U_{iso}(H) = 1.5U_{eq}(C)$. Disordered methyl H atoms were apparent on both the methyl ester and the *p*-tolyl substituent in Fourier difference maps; these were modelled over two sites with occupancies fixed at 0.5.

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: *X-SEED* (Barbour, 2001) and *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors acknowledge the support of the School of Chemistry in funding this project.

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