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Methyl 4-*p*-tolyl-1*H*-pyrrole-2-carboxylate

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Key indicators

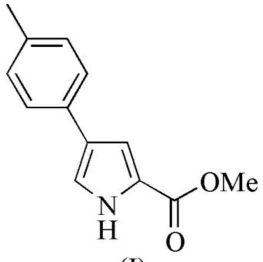
Single-crystal X-ray study
T = 193 K
Mean σ(C–C) = 0.003 Å
Disorder in main residue
R factor = 0.044
wR factor = 0.135
Data-to-parameter ratio = 13.2

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

The molecules of the title compound, $C_{13}H_{13}NO_2$, 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.

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).



(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 $C_4N_2O_2H_2$ 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.

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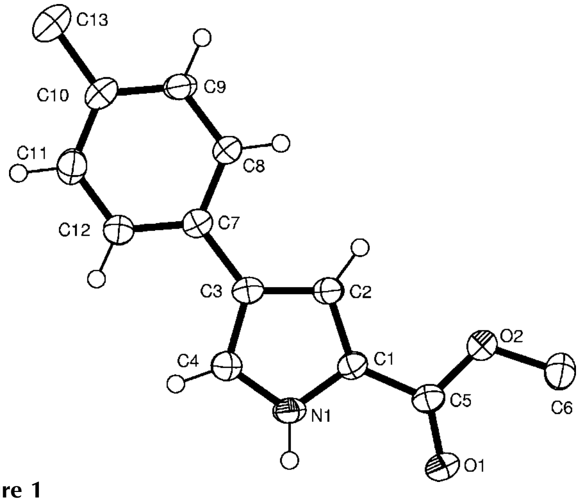

Figure 1
The molecular structure of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Disordered methyl H atoms have been omitted for clarity.

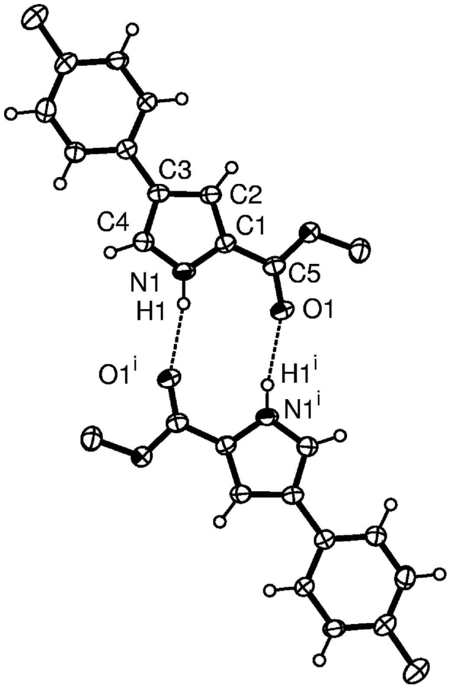

Figure 2
A view of the hydrogen-bonding interactions between molecules of (I), shown as dashed lines. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Disordered methyl H atoms have been omitted for clarity. [Symmetry code: (i) $2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

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) Å^3
$Z = 4$
$D_x = 1.292$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.09$ mm^{-1}
$T = 193$ (2) K
Block, colourless
0.55 × 0.55 × 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 $I > 2\sigma(I)$
$R_{\rm int} = 0.020$
$\theta_{\rm max} = 25.0°$
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 $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$
$\Delta\rho_{\rm max} = 0.25$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.27$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 1997)
Extinction coefficient: 0.028 (4)

Table 1
Hydrogen-bond geometry (Å, °).

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1⋯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 $U_{\rm iso}$(H) = $1.2U_{\rm eq}$(C), except for methyl H atoms, where $U_{\rm iso}$(H) = $1.5U_{\rm eq}$(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).

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