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Key indicators

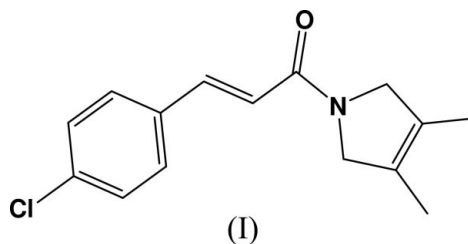
Single-crystal X-ray study
 $T = 298\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$
 R factor = 0.073
 wR factor = 0.186
Data-to-parameter ratio = 16.0For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.3-(4-Chlorophenyl)-1-(3,4-dimethyl-2,5-dihydro-1H-
pyrrol-1-yl)prop-2-enone

The title compound, $\text{C}_{15}\text{H}_{16}\text{ClNO}$, was produced by the reaction of *N,N*-diallylacrylamide with 1-bromo-4-chlorobenzene in the presence of palladium(II) acetate *via* an intramolecular and intermolecular C–C coupling reaction. In the crystal structure, a single weak intermolecular C–H \cdots O interaction gives rise to extended one-dimensional chains.

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Comment

The palladium-catalysed Heck reaction is a powerful tool for constructing nitrogen-containing heterocyclic rings (Tsuji, 1995). Pyrrole derivatives, which have physiological activity, are effective intermediates in the synthesis of many complex natural products (Poli & Giambastiani, 2002; Eriksson *et al.*, 2003). We have reported some novel palladium-catalysed Heck intramolecular and intermolecular reactions of aryl halides with nitrogen-containing olefins (Hu, Zhou, Long *et al.*, 2003; Hu, Zhou, Lian *et al.*, 2003). The reaction of 1-bromo-4-chlorobenzene with *N,N*-diallylacrylamide, in the presence of palladium(II) acetate and triphenylphosphine, in DMF at 383 K for 20 h, gave the unexpected title product containing a dihydropyrrole ring.



In the title molecular structure (Fig. 1), the bond lengths and angles have normal values (Allen *et al.*, 1987). The dihedral angle between the benzene ring (C10–C15) and the dihydropyrrole ring (C3/C4/N/C5/C6) is $2.17(4)^\circ$. In the crystal structure, a single weak intermolecular C–H \cdots O interaction (Table 2) links molecules into one-dimensional chains, with molecules lying parallel to (101) (Fig. 2).

Experimental

An oven-dried Schlenk flask was evacuated, filled with nitrogen, and then charged with *N,N*-diallyl-acrylamide (1.51 g, 10 mmol), 1-bromo-4-chlorobenzene (2.15 g, 11 mmol), tributylamine (3 ml), PPh_3 (52.5 mg, 0.2 mmol), $\text{Pd}(\text{OAc})_2$ (24 mg, 0.1 mol) and DMF (10 ml) to give a yellow solution. The reaction mixture was heated at 383 K with stirring. The reaction mixture was cooled to room temperature after 20 h and the resulting yellow–orange mixture was diluted with Et_2O (10 ml). The mixture was washed with water

(15 ml) and the aqueous layer was extracted with Et₂O (20 ml). The combined organic layers were dried (MgSO₄), filtered and concentrated *in vacuo*. The crude material was purified by flash column chromatography on silica gel (petroleum ether/EtOAc, 10:1) and recrystallized from EtOAc (yield 0.99 g, 38%). Colorless crystals suitable for X-ray diffraction were obtained by recrystallization from a solution of the title compound from ethyl acetate over a period of one week.

Crystal data

C₁₅H₁₆ClNO
M_r = 261.74
 Monoclinic, *P*2₁/*n*
a = 8.8360 (18) Å
b = 15.394 (3) Å
c = 9.988 (2) Å
 β = 94.66 (3)°
V = 1354.1 (5) Å³

Z = 4
D_x = 1.284 Mg m⁻³
 Mo *K*α radiation
 μ = 0.27 mm⁻¹
T = 298 (2) K
 Block, colorless
 0.30 × 0.10 × 0.10 mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 $\omega/2\theta$ scans
 Absorption correction: ψ scan
 (North *et al.*, 1968)
T_{min} = 0.96, *T_{max}* = 0.97
 2812 measured reflections

2640 independent reflections
 1358 reflections with *I* > 2σ(*I*)
R_{int} = 0.036
 θ_{\max} = 26.0°
 3 standard reflections
 every 100 reflections
 intensity decay: none

Refinement

Refinement on *F*²
R [*F*² > 2σ(*F*²)] = 0.073
wR (*F*²) = 0.186
S = 1.02
 2640 reflections
 165 parameters
 H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0687P)^2 + 0.0751P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 (Δ/σ)_{max} < 0.001
 $\Delta\rho_{\max} = 0.24 \text{ e } \text{Å}^{-3}$
 $\Delta\rho_{\min} = -0.26 \text{ e } \text{Å}^{-3}$

Table 1

Selected geometric parameters (Å, °).

Cl–C13	1.745 (4)	N–C4	1.457 (5)
O–C7	1.229 (4)	N–C5	1.461 (5)
N–C7	1.349 (5)		
C7–N–C4	128.2 (3)	N–C4–C3	102.7 (3)
C7–N–C5	120.1 (3)	O–C7–N	121.3 (4)
C4–N–C5	111.6 (3)	O–C7–C8	122.4 (4)
C6–C3–C1	128.4 (4)	N–C7–C8	116.3 (4)
C6–C3–C4	111.6 (4)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
C14–H14A···O ⁱ	0.93	2.44	3.237 (5)	144

Symmetry code: (i) $x - \frac{1}{2}, -y + \frac{3}{2}, z - \frac{1}{2}$.

All H atoms were placed in calculated positions with C–H distances 0.93–0.97 Å and included in the refinement in riding-model approximation with *U*_{iso} = 1.2*U*_{eq}(C) or 1.5*U*_{eq}(C) for methyl H atoms.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms &

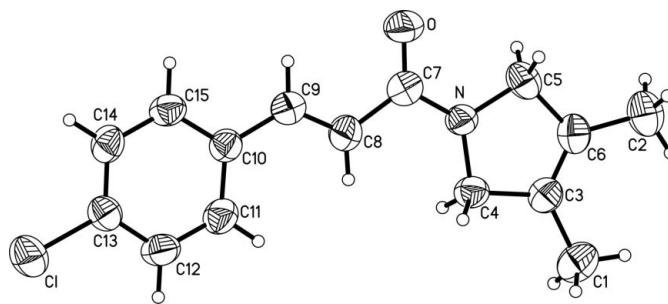


Figure 1

The molecular structure of (I), showing 30% probability displacement ellipsoids and the atom numbering scheme.

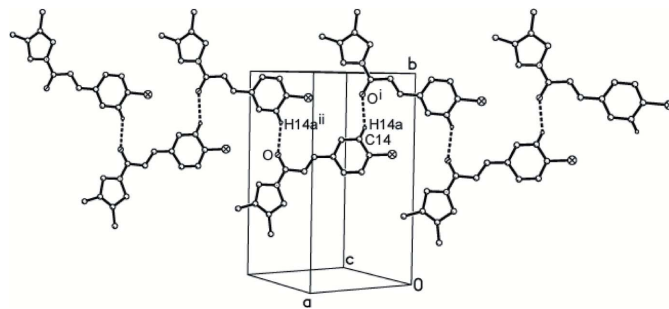


Figure 2

View of the chain structure of the title compound, in which molecules lie parallel to (101). The broken lines show weak C–H···O interactions. [Symmetry codes: (i) $-\frac{1}{2} + x, \frac{3}{2} - y, -\frac{1}{2} + z$; (ii) $\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$.] H atoms not involved in the weak interactions have been omitted.

Wocadlo, 1995); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Sheldrick, 1998); software used to prepare material for publication: *SHELXL97*.

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