

Methyl {2-[4-ethyl-1-(tolylsulfonyl)-2,3-dihydropyrrol-3-yl]ethyl}benzoate

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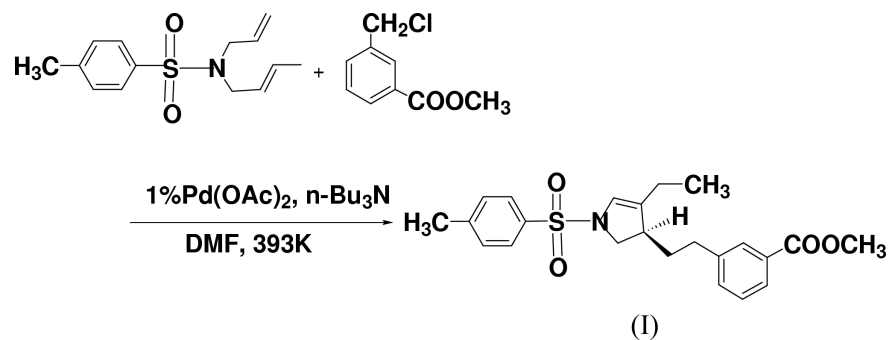
Key indicators

Single-crystal X-ray study
 $T = 293\text{ K}$
Mean $\sigma(\text{C}-\text{C}) = 0.003\text{ \AA}$
 R factor = 0.040
 wR factor = 0.115
Data-to-parameter ratio = 13.9For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

The title compound, $\text{C}_{23}\text{H}_{27}\text{NO}_4\text{S}$, is a novel kind of pyrrole derivative. The molecules are connected by weak $\text{C}-\text{H}\cdots\text{O}$ interactions, forming one-dimensional chains along the b axis.

Comment

Heterocyclic nuclei are present in many pharmacologically active compounds (Tsuji, 1995). Many synthetic methods for the construction of cyclic compounds have been developed (Schuster & Blechert, 1997). Palladium-catalysed annulation technology has become one of the most powerful tools in the construction of heterocyclic compounds (Beletskaya & Cheprakov, 2000). Aryl or olefinic halides have been used as starting materials in various cases, but a few examples of palladium-catalysed annulation reactions of alkyl halides are also known. In our continuing research on palladium-catalysed Heck reactions of benzyl halides with olefins (Hu, Zhou, Long *et al.*, 2003; Hu, Zhou, Lian *et al.*, 2003), a novel palladium-catalysed reaction of benzyl halides with a diene is reported here.



The molecular structure of the title compound, (I), is shown in Fig. 1. In the ethylene link between two rings, the $\text{C}3-\text{C}7$ and $\text{C}7-\text{C}8$ bond lengths are 1.580 (3) and 1.442 (3) \AA , respectively. These values deviate markedly from the typical $\text{Csp}^3-\text{Csp}^3$ bond distance (1.54 \AA). This may be an artifact due to dynamic disorder, created by pedal motion as observed in stilbenes and azobenzenes (Harada & Ogawa, 2001).

There is a chiral carbon atom, C3, in the molecule, but the title compound is racemic. The five-membered ring, comprising atoms $\text{C}1-\text{C}4/\text{N}1$, is nearly planar, the r.m.s. deviation being 0.025 \AA . Atoms $\text{C}1$, $\text{C}4$, $\text{S}1$ and $\text{N}1$ are not coplanar, suggesting sp^3 hybridization of atom $\text{N}1$. There are weak intermolecular $\text{C}-\text{H}\cdots\text{O}$ interactions (Table 2 and Fig. 2), which lead to one-dimensional molecular chains along the b axis.

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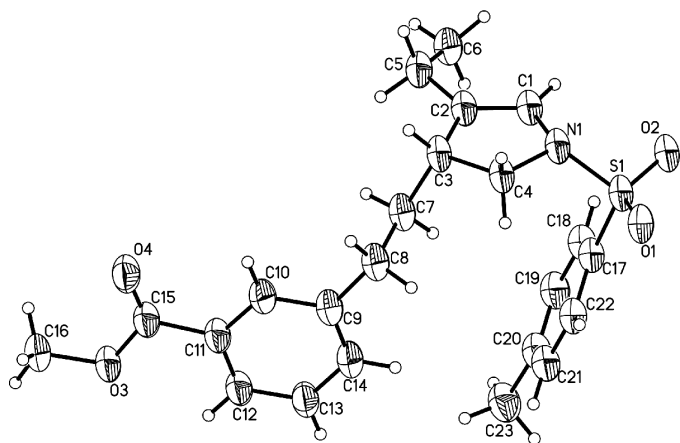


Figure 1
A view of the molecule of (I), showing 50% probability displacement ellipsoids.

Experimental

The title compound, (I), was synthesized according to the following procedure: an oven-dried Schlenk flask was evacuated and filled with nitrogen, then charged with *N*-allyl-*N*-(2-butenyl)-*p*-toluenesulfonamide (1.325 g, 5 mmol), 3-methoxycarbonylbenzyl chloride (1.562 g, 5.5 mmol), tributylamine (1.5 ml, 6 mmol), Pd(OAc)₂ (12 mg, 0.05 mmol) and DMF (2 ml), giving a yellow solution. The reaction mixture was heated at 393 K with stirring. The reaction mixture was cooled to room temperature after 15 h and the resultant yellow–orange mixture was diluted with Et₂O (10 ml). The mixture was washed with H₂O (15 ml) and the aqueous layer was extracted with Et₂O (10 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, 6:1) and recrystallized from MeCN/Et₂O; yield 0.97 g (47%). Colourless crystals of (I) suitable for X-ray diffraction were obtained by diffusion of ethyl acetate into an MeCN solution over a period of one week.

Crystal data

C ₂₃ H ₂₇ NO ₄ S	<i>Z</i> = 2
<i>M_r</i> = 413.52	<i>D_x</i> = 1.283 Mg m ⁻³
Triclinic, <i>P</i> 1̄	Mo <i>K</i> α radiation
<i>a</i> = 8.412 (2) Å	Cell parameters from 620 reflections
<i>b</i> = 10.931 (2) Å	<i>θ</i> = 2.7–20.8°
<i>c</i> = 11.989 (2) Å	<i>μ</i> = 0.18 mm ⁻¹
<i>α</i> = 83.73 (1)°	<i>T</i> = 293 (2) K
<i>β</i> = 81.30 (1)°	Prism, colourless
<i>γ</i> = 80.32 (1)°	0.3 × 0.2 × 0.2 mm
<i>V</i> = 1070.2 (4) Å ³	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	<i>R</i> _{int} = 0.017
<i>φ</i> and <i>ω</i> scans	<i>θ</i> _{max} = 25.0°
5318 measured reflections	<i>h</i> = -9 → 9
3680 independent reflections	<i>k</i> = -12 → 12
3009 reflections with <i>I</i> > 2σ(<i>I</i>)	<i>l</i> = -14 → 10

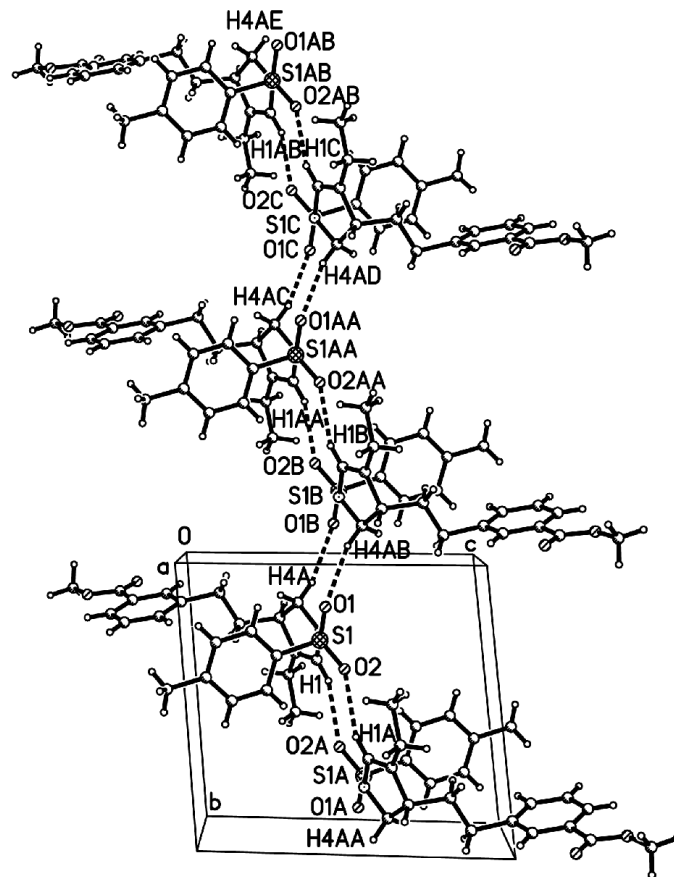


Figure 2
The crystal packing of (I), viewed down the *a* axis, showing, as dashed lines, the C–H...O short contact. [Symmetry codes: (A) 2 - *x*, 1 - *y*, 1 - *z*; (B) 2 - *x*, -*y*, 1 - *z*; (C) 2 - *x*, -1 - *y*, 1 - *z*.]

Refinement

Refinement on <i>F</i> ²	$w = 1/[\sigma^2(F_o^2) + (0.065P)^2 + 0.0934P]$
$R[F^2 > 2\sigma(F^2)] = 0.040$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.115$	$(\Delta/\sigma)_{\max} < 0.001$
<i>S</i> = 1.06	$\Delta\rho_{\max} = 0.41 \text{ e \AA}^{-3}$
3680 reflections	$\Delta\rho_{\min} = -0.41 \text{ e \AA}^{-3}$
265 parameters	H-atom parameters constrained

Table 1

Selected geometric parameters (Å, °).

S1–O1	1.4218 (15)	C1–C2	1.322 (3)
S1–O2	1.4249 (14)	C2–C3	1.503 (3)
S1–N1	1.6321 (18)	C3–C4	1.533 (3)
S1–C17	1.757 (2)	C3–C7	1.580 (3)
N1–C1	1.418 (3)	C7–C8	1.442 (3)
N1–C4	1.492 (2)		
C1–N1–C4	107.12 (14)	C8–C7–C3	114.56 (18)
C1–N1–S1	118.77 (13)	C7–C8–C9	108.61 (18)
C4–N1–S1	118.38 (14)	C10–C9–C14	118.25 (19)
C2–C3–C4	104.18 (15)	C10–C9–C8	116.2 (2)
C2–C3–C7	106.42 (17)	C14–C9–C8	125.50 (19)
C4–C3–C7	114.10 (18)		

Table 2
Hydrogen-bonding geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
C4–H4A \cdots O1 ⁱ	0.97	2.54	3.488 (2)	166
C1–H1 \cdots O2 ⁱⁱ	0.93	2.65	3.523 (2)	157

Symmetry codes: (i) $2-x, -y, 1-z$; (ii) $2-x, 1-y, 1-z$.

All H atoms were positioned geometrically and refined as riding, with $U_{\text{iso}}(\text{H})$ values 1.2 to 1.5 times U_{eq} of the parent atom. The distances to the H atoms were in the range 0.93–0.98 Å.

Data collection: *SMART* (Bruker, 2000); cell refinement: *SMART*; data reduction: *SAINTE* (Bruker, 2000); program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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