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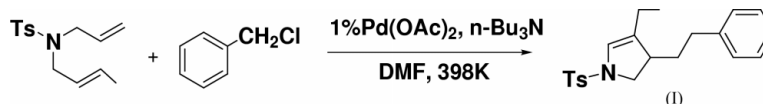
## Key indicators

Single-crystal X-ray study  
 $T = 293\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.005\text{ \AA}$   
 $R$  factor = 0.056  
 $wR$  factor = 0.160  
Data-to-parameter ratio = 14.7For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

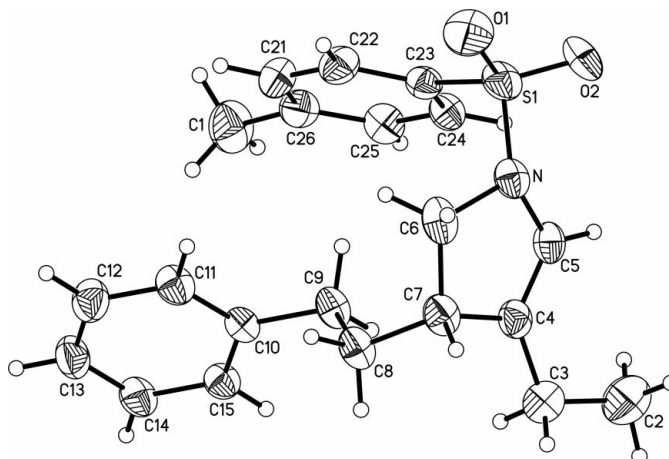
## 3-Ethyl-1-(4-methylphenylsulfonyl)-4-phenethyl-4,5-dihydropyrrole

The title compound,  $\text{C}_{21}\text{H}_{25}\text{NO}_2\text{S}$ , crystallizes in a centrosymmetric triclinic unit cell. In the molecule, the bond lengths and angles are normal, and the dihydropyrrole five-membered ring is almost planar.Received 16 September 2003  
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## Comment

Palladium-catalysed C–C bond formation is one of the most powerful tools of contemporary organic synthesis (Nicolaou & Sorensen, 1996). One of the common features of the newly developed reactions is their application in the construction of cyclic compounds (Negish *et al.*, 1996). Aryl or olefinic halides are often used as starting materials in various cases, but few examples of palladium-catalyzed annulation reactions of alkyl halides are known (Wang *et al.*, 2000). In our continuing research on palladium-catalyzed Heck reactions of benzyl halides with olefins (Hu, Zhou, Lian *et al.*, 2003; Hu, Zhou, Long *et al.*, 2003), a novel palladium-catalyzed reaction of benzyl halides with a diene has been investigated. Benzyl chloride reacted with *N*-allyl-*N*-(2-butenyl)-*p*-toluenesulfonamide, in the presence of palladium acetate in DMF at 388 K for 16 h, to give a novel cyclic product, (I), having a dihydropyrrole ring.

The title compound (Fig. 1) has normal bond lengths and angles. There is a chiral C atom (C7) in the molecule, but (I) is a racemic compound. The dihydropyrrole five-membered ring

**Figure 1**  
View of (I), with the atom labelling and 30% probability displacement ellipsoids.

(N/C4/C5/C6/C7) is almost planar, with an r.m.s. deviation of 0.0152 Å. The crystal packing is shown in Fig. 2.

## Experimental

An oven-dried Schlenk flask was evacuated and filled with nitrogen. It was then charged with *N*-allyl-*N*-(2-butenyl)-*p*-toluenesulfonamide (1.325 g, 5 mmol), benzyl chloride (0.696 g, 5.5 mmol), tributylamine (1.5 ml), Pd(OAc)<sub>2</sub> (12 mg, 0.05 mmol), and DMF (15 ml), giving a yellow solution. The reaction mixture was heated at 388 K with stirring. The reaction mixture was then cooled to room temperature after 16 h and the resultant yellow–orange mixture was diluted with Et<sub>2</sub>O (10 ml). The mixture was washed with H<sub>2</sub>O (15 ml) and the aqueous layer was extracted with Et<sub>2</sub>O (10 ml). The combined organic layers were dried (MgSO<sub>4</sub>), 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/Et<sub>2</sub>O (yield 1.35 g, 76%). Colorless triclinic crystals of the title compound were obtained by diffusion of ethyl acetate into an MeCN solution over a period of one week.

### Crystal data

C <sub>21</sub> H <sub>25</sub> NO <sub>2</sub> S	<i>Z</i> = 2
<i>M<sub>r</sub></i> = 355.48	<i>D<sub>x</sub></i> = 1.248 Mg m <sup>-3</sup>
Triclinic, <i>P</i> $\bar{1}$	Mo <i>K</i> α radiation
<i>a</i> = 8.250 (2) Å	Cell parameters from 25 reflections
<i>b</i> = 10.740 (2) Å	$\theta$ = 2.1–26.7°
<i>c</i> = 12.020 (2) Å	$\mu$ = 0.19 mm <sup>-1</sup>
$\alpha$ = 66.05 (3)°	<i>T</i> = 293 (2) K
$\beta$ = 76.82 (3)°	Block, colourless
$\gamma$ = 81.95 (3)°	0.30 × 0.25 × 0.20 mm
<i>V</i> = 946.3 (4) Å <sup>3</sup>	

### Data collection

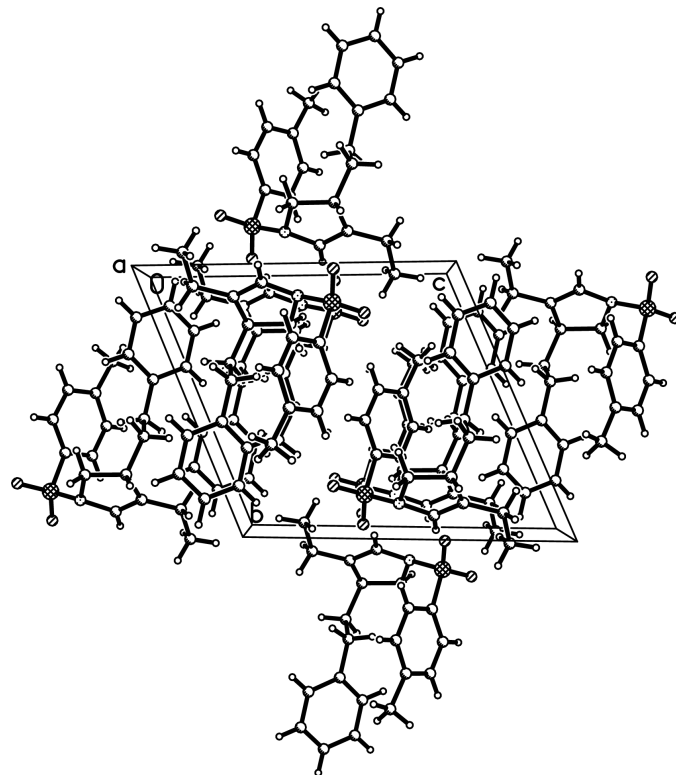
Bruker P4 diffractometer	<i>R</i> <sub>int</sub> = 0.041
$\omega$ scans	$\theta$ <sub>max</sub> = 25.0°
Absorption correction: $\psi$ scan ( <i>SHELXTL</i> ; Bruker, 2000)	<i>h</i> = 0 → 9
<i>T</i> <sub>min</sub> = 0.95, <i>T</i> <sub>max</sub> = 0.96	<i>k</i> = -12 → 12
3583 measured reflections	<i>l</i> = -13 → 14
3330 independent reflections	3 standard reflections every 97 reflections
1852 reflections with <i>I</i> > 2σ( <i>I</i> )	intensity decay: none

### Refinement

Refinement on <i>F</i> <sup>2</sup>	$w = 1/[\sigma^2(F_o^2) + (0.0753P)^2 + 0.1421P]$
$R[F^2 > 2\sigma(F^2)] = 0.056$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.160$	( $\Delta/\sigma$ ) <sub>max</sub> = 0.001
<i>S</i> = 1.03	$\Delta\rho$ <sub>max</sub> = 0.19 e Å <sup>-3</sup>
3330 reflections	$\Delta\rho$ <sub>min</sub> = -0.31 e Å <sup>-3</sup>
226 parameters	
H-atom parameters constrained	

All H atoms were positioned geometrically and refined as riding with isotropic displacement parameters 1.2 to 1.5 times *U*<sub>eq</sub> of the parent atom. The C–H atoms were in the range 0.93–0.98 Å.

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXTL* (Bruker, 2000); program(s) used to refine



**Figure 2**  
The crystal packing of (I), viewed along the *a* axis.

structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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