

(E)-5-Methyl-2-(2-methylstyryl)pyrrole

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Key indicators

Single-crystal X-ray study

$T = 293\text{ K}$

Mean $\sigma(\text{C}-\text{C}) = 0.006\text{ \AA}$

R factor = 0.054

wR factor = 0.186

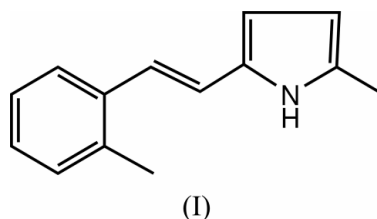
Data-to-parameter ratio = 16.7

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

Evaporation of a dichloromethane solution of the title compound, $\text{C}_{14}\text{H}_{15}\text{N}$, yielded a tar from which single crystals were obtained by sublimation. The title compound consists of two essentially planar moieties that are oriented in a synperiplanar fashion with respect to each other. The geometrical parameters are in accordance with literature values.

Comment

Stereochemical changes in 2-styrylpyrroles might affect the electronic structure and influence their photophysical and photochemical properties (Šindler-Kulyk *et al.*, 1995; Basarić *et al.*, 2000, 2001). The synthesis and conformational analysis, based on the UV photoelectron spectra, of the title compound, (I), have been reported previously (Rademacher *et al.*, 2002). This paper deals with the structure of (I) in the solid state, and is to be regarded as a contribution to the overall understanding of the conformational behaviour of the title compound.



Evaporation of a dichloromethane solution of (I) resulted in the formation of a tarry material, which was recrystallized by sublimation at 277 K. Yellow plates were obtained. The bond lengths and angles are in accordance with the literature values for related compounds (Allen & Kennard, 1993). The average bond distance in a toluene ring is 1.377 (6) Å. The molecule consists of two planar moieties: the toluene ring and the pyrrole group with the central double bond C7=C8 (Fig. 1). The N1–C2–C7–C8 torsion angle [$\varphi = 6.5(6)^\circ$] (Rademacher *et al.*, 2002) suggests an almost planar arrangement of the pyrrole group and the central double bond. The deviations from the least-squares plane drawn through atoms N1/C2–C8 range from $-0.001(4)$ Å for C2 to $0.041(4)$ Å for C7. The relevant torsion angle (φ) defines the synperiplanar orientation of the pyrrole ring with respect to the double bond C7=C8. The toluene ring is twisted with respect to the plane of the central double bond by $29.7(5)^\circ$ (torsion angle C7–C8–C9–C10, τ) (Rademacher *et al.*, 2002). This value defines the synperiplanar orientation of the toluene ring with respect to the central double bond. The dihedral angle between the

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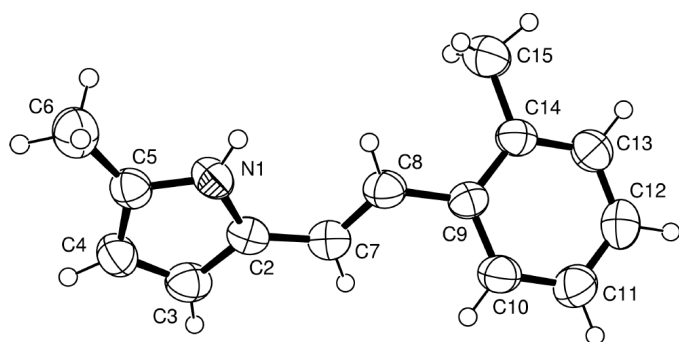


Figure 1
An ORTEPII (Johnson, 1976) drawing of (I), with the atomic numbering scheme. Displacement ellipsoids of non-H atoms are drawn at the 50% probability level.

plane of the toluene ring and the least-squares plane through atoms N1/C2–C8 is $36.90(16)^\circ$, and suggests the borderline synperiplanar orientation of the toluene ring with respect to the remaining (essentially planar) part of the molecule, hence the overall synperiplanar conformation. The results are in accordance with *ab initio* calculations (Rademacher *et al.*, 2002).

The crystal packing of (I) is governed only by weak van der Waals forces.

Experimental

Compound (I) was prepared according to the literature procedure of Rademacher *et al.* (2002).

Crystal data

$C_{14}H_{15}N$	$D_x = 1.156 \text{ Mg m}^{-3}$
$M_r = 197.27$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 15 reflections
$a = 12.056(2) \text{ \AA}$	$\theta = 3.2\text{--}19.7^\circ$
$b = 7.545(1) \text{ \AA}$	$\mu = 0.07 \text{ mm}^{-1}$
$c = 12.62(2) \text{ \AA}$	$T = 293(2) \text{ K}$
$\beta = 99.04(4)^\circ$	Plate, yellow
$V = 1133.7(18) \text{ \AA}^3$	$0.20 \times 0.15 \times 0.06 \text{ mm}$
$Z = 4$	

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.053$
$\omega/2\theta$ scans	$\theta_{\text{max}} = 26.3^\circ$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$h = -15 \rightarrow 14$
$T_{\text{min}} = 0.893$, $T_{\text{max}} = 0.935$	$k = 0 \rightarrow 9$
2404 measured reflections	$l = 0 \rightarrow 15$
2299 independent reflections	3 standard reflections
774 reflections with $I > 2\sigma(I)$	frequency: 120 min
	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.054$	$w = 1/[\sigma^2(F_o^2) + (0.08P)^2]$
$wR(F^2) = 0.186$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 0.90$	$(\Delta/\sigma)_{\text{max}} < 0.001$
2299 reflections	$\Delta\rho_{\text{max}} = 0.20 \text{ e \AA}^{-3}$
138 parameters	$\Delta\rho_{\text{min}} = -0.17 \text{ e \AA}^{-3}$

Table 1

Selected geometric parameters (\AA , $^\circ$).

N1–C2	1.372 (5)	N1–C5	1.368 (5)
C2–N1–C5	111.5 (3)	N1–C5–C6	121.9 (3)
N1–C2–C7	123.6 (3)	N1–C5–C4	105.9 (3)
N1–C2–C3	105.1 (3)		

H-atom coordinates were calculated geometrically and refined using the *SHELXL97* riding model. The methyl groups were allowed to rotate, but not to tip.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *SET4* and *CELDIM* in *CAD-4 Software*; data reduction: *HELENA* (Spek, 1997); program(s) used to solve structure: *SIR97* (Altomare *et al.*, 1999); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *PLATON* (Spek, 1990).

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References

- Allen, F. H. & Kennard, O. (1993). *Chem. Des. Autom. News*, **8**, 1, 31–37.
- Altomare, A., Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Guagliardi, A., Moliterni, A. G. G., Polidori, G. & Spagna, R. (1999). *J. Appl. Cryst.* **32**, 115–119.
- Basarić, N., Marinić, Ž. & Šindler-Kulyk, M. (2001). *Tetrahedron Lett.* **42**, 3641–3643.
- Basarić, N., Tomšić, S., Marinić, Ž. & Šindler-Kulyk, M. (2000). *Tetrahedron*, **56**, 1587–1593.
- Enraf–Nonius (1989). *CAD-4 Software*. Version 5.0. Enraf–Nonius, Delft, The Netherlands.
- Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* **A24**, 351–359.
- Rademacher, P., Basarić, N., Kowski, K. & Šindler-Kulyk, M. (2002). *Eur. J. Org. Chem.* pp. 551–556.
- Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.
- Spek, A. L. (1990). *Acta Cryst.* **A46**, C-34.
- Spek, A. L. (1997). *HELENA*. Utrecht University, The Netherlands.
- Šindler-Kulyk, M., Tomšić, S., Marinić, Ž. & Metelko, B. (1995). *Recl. Trav. Chim. Pay-Bas*, **114**, 476–479.