

Table 1. Selected geometric parameters (Å, °)

C1—N1	1.407 (3)	C7—N1	1.290 (3)
C3—C11	1.738 (3)	C9—O1	1.323 (3)
C5—C12	1.735 (3)		
C2—C1—N1	116.6 (2)	C4—C5—C12	118.3 (2)
C6—C1—N1	124.5 (2)	N1—C7—C8	122.8 (2)
C2—C3—C11	119.9 (2)	O1—C9—C10	116.6 (2)
C4—C3—C11	117.9 (2)	O1—C9—C8	122.5 (3)
C6—C5—C12	119.2 (2)	C7—N1—C1	122.6 (2)

The structure was solved by direct phase determination. It was possible to refine the parameters of the complete structure by full-matrix anisotropic least squares. All phenyl rings were refined without constraints. All H-atom positions (except that of the hydroxyl H atom) were calculated using a riding model and were considered with fixed isotropic *U*'s in all refinements. The H1O atom was located in the difference Fourier maps calculated at the end of the refinement process as a small positive electron density and was not refined.

Data collection: *Diffraction Control Software* (Nonius, 1993). Cell refinement: *Diffraction Control Software*. Data reduction: *REDU4* (Stoe & Cie, 1991). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: HA1210). Services for accessing these data are described at the back of the journal.

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Racemic 1-Acetyl-5-phenyl-3-styryl-2-pyrazoline

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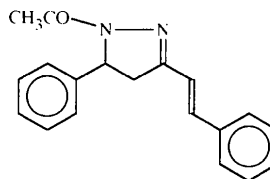
(Received 26 March 1997; accepted 6 January 1998)

Abstract

The asymmetric unit of the racemic title compound, C₁₉H₁₈N₂O, contains two enantiomeric pyrazoline molecules, differing in the configuration at C3. All the rings in the molecules are essentially planar.

Comment

The syntheses of the title compound and its derivatives (Vijayabaskar, 1997) are important as it is known that pyrazolines display various biological properties, such as pesticidal, fungicidal, insecticidal and antiviral activities, and have industrial applications, such as their use as polymer intermediates (Wang *et al.*, 1995; El-Hashash *et al.*, 1995). In order to deduce the configuration (*vis. s-cis* or *s-trans*) of the title pyrazoline, (I), the X-ray structure analysis was undertaken.



(I)

The asymmetric unit consists of two enantiomeric molecules (Fig. 1). The relatively short C1—C6 and C1'—C6' bond distances of 1.444 (4) and 1.443 (4) Å, respectively, in the two molecules are ascribable to the resonance between the C1=N2 and C6=C7 bonds. This resonance is supported by the fact that the five-membered dinitrogen heterocyclic ring, the ethenyl C6 and C7 atoms, and the aryl ring of the styryl group lie in the same plane. Molecular-orbital calculations and spectral measurements are underway to determine whether the diene (N2=C1—C6=C7) prefers the *s-cis* or *s-trans* configuration in solution. The torsion angles about the dienes in the two molecules are found to be $-174.8(3)$ and $171.5(3)^\circ$, indicating that they each exist in the *s-trans* configuration.

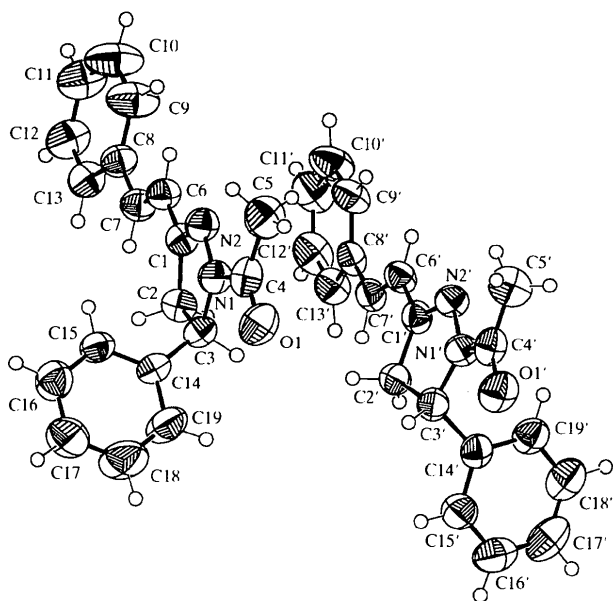


Fig. 1. An ORTEP (Johnson, 1976) diagram of the title compound showing the asymmetric unit with 50% probability ellipsoids.

There is a significant difference in the orientation of the phenyl ring attached to the C3 atom of the heterocyclic ring between the two molecules (see Table 1). The dihedral angle between the heterocyclic ring and the phenyl ring attached to it is found to be $86.5(2)^\circ$ in one molecule (unprimed atom labels) and $95.5(1)^\circ$ in the other (primed atom labels). A packing diagram is available as part of the supplementary material.

Experimental

Details of the synthesis have been described by Vijayabaskar (1997). Analysis found: C 78.51, H 6.18, N 9.60% (M^+ 290); calculated for $C_{19}H_{18}N_2O$: C 78.59, H 6.25, N 9.65% (M^+ 290).

Crystal data

$C_{19}H_{18}N_2O$
 $M_r = 290.35$
 Monoclinic
 $P2_1/a$
 $a = 12.704(2) \text{ \AA}$
 $b = 15.788(4) \text{ \AA}$
 $c = 16.676(2) \text{ \AA}$
 $\beta = 107.70(1)^\circ$
 $V = 3186.4(10) \text{ \AA}^3$
 $Z = 8$
 $D_x = 1.211 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 25 reflections
 $\theta = 7.92\text{--}13.52^\circ$
 $\mu = 0.076 \text{ mm}^{-1}$
 $T = 293(2) \text{ K}$
 Square plate
 $0.4 \times 0.2 \times 0.2 \text{ mm}$
 Pale yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
 ω - 2θ scans
 Absorption correction: none
 5855 measured reflections
 5581 independent reflections
 3399 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.022$
 $\theta_{\text{max}} = 24.98^\circ$
 $h = 0 \rightarrow 15$
 $k = 0 \rightarrow 18$
 $l = -19 \rightarrow 18$
 3 standard reflections every 200 reflections
 intensity decay: 0.9%

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.070$
 $wR(F^2) = 0.148$
 $S = 1.284$
 5581 reflections
 542 parameters
 H atoms refined isotropically
 $w = 1/[\sigma^2(F_o^2) + (0.0284P)^2 + 1.4452P]$
 where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\text{max}} = 0.001$

$\Delta\rho_{\text{max}} = 0.125 \text{ e \AA}^{-3}$
 $\Delta\rho_{\text{min}} = -0.149 \text{ e \AA}^{-3}$
 Extinction correction: SHELXL93 (Sheldrick, 1993)
 Extinction coefficient: 0.0029 (3)
 Scattering factors from International Tables for Crystallography (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

O1—C4	1.227 (4)	O1'—C4'	1.230 (4)
N1—C4	1.355 (4)	N1'—C4'	1.360 (4)
N1—N2	1.388 (3)	N1'—N2'	1.400 (3)
N1—C3	1.485 (4)	N1'—C3'	1.475 (4)
N2—C1	1.289 (4)	N2'—C1'	1.287 (4)
C1—C6	1.444 (4)	C1'—C6'	1.443 (4)
C1—C2	1.495 (4)	C1'—C2'	1.506 (4)
C2—C3	1.540 (4)	C2'—C3'	1.537 (4)
C3—C14	1.511 (4)	C3'—C14'	1.503 (4)
C4—C5	1.507 (6)	C4'—C5'	1.497 (5)
C6—C7	1.327 (4)	C6'—C7'	1.331 (4)
C7—C8	1.465 (4)	C7'—C8'	1.468 (4)
C8—C13	1.387 (5)	C8'—C13'	1.384 (4)
C8—C9	1.386 (5)	C8'—C9'	1.386 (4)
C9—C10	1.378 (5)	C9'—C10'	1.378 (5)
C10—C11	1.366 (6)	C10'—C11'	1.375 (6)
C11—C12	1.366 (6)	C11'—C12'	1.367 (5)
C12—C13	1.380 (5)	C12'—C13'	1.381 (5)
C14—C15	1.367 (4)	C14'—C15'	1.383 (5)
C14—C19	1.377 (4)	C14'—C19'	1.385 (4)
C15—C16	1.376 (5)	C15'—C16'	1.385 (6)
C16—C17	1.372 (6)	C16'—C17'	1.363 (6)
C17—C18	1.352 (6)	C17'—C18'	1.369 (6)
C18—C19	1.380 (6)	C18'—C19'	1.373 (5)
C4—N1—N2	122.2 (3)	C4'—N1'—N2'	122.6 (3)
C4—N1—C3	123.4 (3)	C4'—N1'—C3'	124.2 (3)

N2—N1—C3	113.7 (2)	N2'—N1'—C3'	113.0 (2)
C1—N2—N1	107.6 (3)	C1'—N2'—N1'	107.9 (3)
N2—C1—C6	120.5 (3)	N2'—C1'—C6'	122.2 (3)
N2—C1—C2	114.5 (3)	N2'—C1'—C2'	113.8 (3)
C6—C1—C2	125.0 (3)	C6'—C1'—C2'	124.0 (3)
C1—C2—C3	103.3 (3)	C1'—C2'—C3'	102.8 (3)
N1—C3—C14	112.3 (3)	N1'—C3'—C14'	114.5 (3)
N1—C3—C2	100.7 (3)	N1'—C3'—C2'	101.2 (2)
C14—C3—C2	114.1 (3)	C14'—C3'—C2'	111.1 (3)
O1—C4—N1	119.2 (3)	O1'—C4'—N1'	118.8 (3)
O1—C4—C5	123.3 (4)	O1'—C4'—C5'	123.2 (3)
N1—C4—C5	117.5 (4)	N1'—C4'—C5'	118.0 (3)
C7—C6—C1	124.8 (3)	C7'—C6'—C1'	122.9 (3)
C6—C7—C8	127.2 (3)	C6'—C7'—C8'	127.6 (3)
C9—C8—C13	116.9 (3)	C13'—C8'—C9'	118.3 (3)
C9—C8—C7	123.5 (3)	C13'—C8'—C7'	119.2 (3)
C13—C8—C7	119.6 (3)	C9'—C8'—C7'	122.5 (3)
C10—C9—C8	121.3 (4)	C10'—C9'—C8'	120.6 (4)
C11—C10—C9	120.8 (4)	C11'—C10'—C9'	120.3 (4)
C12—C11—C10	118.9 (4)	C12'—C11'—C10'	119.8 (4)
C11—C12—C13	120.7 (4)	C11'—C12'—C13'	120.1 (4)
C12—C13—C8	121.3 (4)	C12'—C13'—C8'	120.8 (4)
C15—C14—C19	117.9 (3)	C15'—C14'—C19'	117.9 (4)
C15—C14—C3	121.3 (3)	C15'—C14'—C3'	119.7 (3)
C19—C14—C3	120.8 (3)	C19'—C14'—C3'	122.0 (3)
C14—C15—C16	121.7 (3)	C14'—C15'—C16'	120.9 (4)
C17—C16—C15	119.3 (4)	C17'—C16'—C15'	120.0 (5)
C18—C17—C16	120.1 (5)	C16'—C17'—C18'	119.9 (5)
C17—C18—C19	120.2 (4)	C17'—C18'—C19'	120.4 (5)
C14—C19—C18	120.8 (4)	C18'—C19'—C14'	120.8 (4)
N1—C3—C14—C15	62.8 (4)	N1'—C3'—C14'—C15'	155.9 (3)
C2—C3—C14—C15	-50.9 (4)	C2'—C3'—C14'—C15'	-90.1 (4)
N1—C3—C14—C19	-117.9 (4)	N1'—C3'—C14'—C19'	-31.5 (4)
C2—C3—C14—C19	128.3 (4)	C2'—C3'—C14'—C19'	82.4 (4)

All H atoms were located experimentally from the final difference Fourier map and refined isotropically.

Data collection: *CAD-4 Software* (Enraf-Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *SDP* (Enraf-Nonius, 1985). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1976). Software used to prepare material for publication: *SHELXL93*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: DE1064). Services for accessing these data are described at the back of the journal.

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7-Methoxy-2,2,4-trimethyl-1,2-dihydroquinoline

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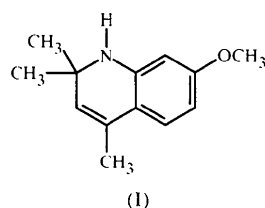
(Received 10 October 1997; accepted 26 January 1998)

Abstract

The title compound, C₁₃H₁₇NO, was prepared using a new class of activator, alkyl bromides. The heterocyclic ring adopts a non-planar conformation, which contrasts with the conformations found for related compounds.

Comment

The exact structure of substituted dihydroquinolines has been the subject of many previous studies. Originally, the structures of such compounds were incorrectly described as simple Schiff bases of acetone (Knoevenagel, 1921). The currently accepted structures were proposed independently on chemical grounds by Reddelien & Thurm (1932), Cliffe (1933) and Murray *et al.* (1933), but it was not until 1965 that the title compound, (I), was first reported (Rosowsky & Modest, 1965). Starting with *m*-anisidine and acetone, with iodine as an activator, yielded (I) as a single product. Although two isomers are possible, the conformation of the isomer produced was identified from NMR shift data.



We produced (I) whilst attempting to synthesize alkylated anisidine by refluxing 6-bromohexan-1-ol with *m*-anisidine in petroleum ether and acetone. Compound (I) was isolated as the only major product. Alkyl bromides have not been reported previously as activators in this reaction. Confirmation that this was the previously reported isomer was obtained by crystal structure analysis.

The molecular structure, shown in Fig. 1, contains a bicyclic nucleus with typical shortenings of the C3—C4 and N1—C5 distances due to conjugation effects [C3—C4 1.469(3), N1—C5 1.381(2) and N1—C1 1.459(3) Å]. Similarly substituted heterocyclic fragments adopt one of two conformations. In 6-ethoxy-