

Table 2. Bond distances (Å) and angles (°) of possible hydrogen bonds (B...H—A)

These possible hydrogen bonds are shown in Fig. 2 with broken lines.

B...H—A	B...A	B...H	H—A	B...H—A
O(1A)...H1(N3A) <sup>(i)</sup> —N(3A)	3.132 (4)	2.24 (5)	1.01 (5)	147 (4)
O(1B)...H1(N3B) <sup>(ii)</sup> —N(3B)	3.152 (4)	2.36 (4)	0.88 (4)	149 (4)
N(1A)...H2(N3B) <sup>(iii)</sup> —N(3B)	2.994 (4)	2.01 (4)	0.99 (4)	168 (3)
N(1B)...H2(N3A)—N(3A)	2.982 (4)	2.08 (4)	0.92 (4)	167 (4)

Symmetry code: none *x*, *y*, *z*; (i) 2 - *x*, 1 - *y*, - *z*; (ii) 1 - *x*, 1 - *y*, - 1 - *z*; (iii) 1 + *x*, *y*, *z*

**Discussion.** The atomic parameters are given in Table 1.\* The unit cell contains two crystallographically independent molecules (labeled *A* and *B* in Fig. 2). *A* and *B* molecules have almost the same geometry, as seen in Fig. 1. All molecules are linked firmly to each other by hydrogen bonds in a zigzag manner (listed in Table 2 and shown with broken lines in Fig. 2). The molecular structure with atom numbering is shown in Fig. 1. The molecule adopts a bridged dihydropyridine skeleton with enamine form at C(5) rather than imine: two H atoms are bonded to N(3), and the short C(5)—C(6) length indicates its double-bond character. This compound loses about 30% of its weight at 410 K probably owing to release of 2-methylpropene, C<sub>4</sub>H<sub>8</sub>.

\* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms and interatomic distances and angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43590 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. Selected bond distances (Å) of molecules *A* and *B*

	(A)	(B)
N(1)—C(3)	1.254 (5)	1.259 (4)
C(5)—N(3)	1.341 (5)	1.343 (5)
C(5)—C(6)	1.340 (5)	1.349 (5)
C(4)—C(8)	1.609 (5)	1.597 (5)
C(7)—C(1)	1.548 (4)	1.530 (5)
C(7)—C(8)	1.539 (5)	1.545 (5)

through retro-Diels—Alder reaction and yields 4-amino-2-methoxy-6-methylpyridine-3,5-dicarbonitrile. It is worth noting that C(4A)—C(8A) and C(4B)—C(8B), 1.609 (5) and 1.597 (5) Å, are significantly long and might be broken easily in the degradation process. The N(1)—C(3) bonds are shorter than the normal C=N double bond (Sandorfy, 1970) by 0.3 or 0.4 Å: they are 1.254 (5) Å in molecule *A* and 1.259 (4) Å in *B* (Table 3).

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## Structure of *cis*-1,2-Diphenyl-2-tolylvinyl Acetate

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**Abstract.** C<sub>23</sub>H<sub>20</sub>O<sub>2</sub>, *M<sub>r</sub>* = 328.41, orthorhombic, *Pbca*, *a* = 13.121 (2), *b* = 30.181 (4), *c* = 9.291 (2) Å, *V* = 3679.3 Å<sup>3</sup>, *Z* = 8, *D<sub>x</sub>* = 1.19 g cm<sup>-3</sup>, λ(Mo *K*α) = 0.71069 Å, μ = 0.69 cm<sup>-1</sup>, *T* = 293 K, *F*(000) = 1392, final *R* = 0.043 for 2426 unique reflections. The acetate group is in an anticlinal antiperiplanar conformation.

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The molecule as a whole has a propeller conformation. The bond lengths and angles are similar to those in other triarylvinyli-X systems. Relative to the *trans* isomer, the *cis* isomer of 1,2-diphenyl-2-tolylvinyl acetate has the higher melting point and tolyl-CH<sub>3</sub> <sup>1</sup>H NMR absorption at higher field.

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**Introduction.** The title compound has been prepared and its structure determined as part of the study of the solvolytic rearrangement of  $^{13}\text{C}$  or  $^{14}\text{C}$  labeled *trans*- and *cis*-1,2-diphenyl-2-tolylvinyl bromides. The two isomers were separated by fractional crystallization, one of which gave crystals suitable for diffraction studies. This isomer melted at 438–440 K; the other melted at 364–366 K. Also the tolyl- $\text{CH}_3$   $^1\text{H}$  NMR absorption for the isomer which gave the better crystals was at  $\delta$  2.27 p.p.m., compared with  $\delta$  2.35 p.p.m. for the other. Based on the conventional assumption that the *trans* isomer melts at higher temperature and shows tolyl- $\text{CH}_3$   $^1\text{H}$  NMR absorption at higher field, this isomer would be expected to be the *trans* isomer.

**Experimental.** The material was obtained from the reaction of *cis*- and *trans*-1,2-diphenyl-2-tolylvinyl bromide with acetic acid in the presence of silver acetate. The *cis* and *trans* mixture of acetates was separated by fractional crystallization from methanol. A transparent crystal in the shape of a rectangular parallelepiped with dimensions  $0.10 \times 0.15 \times 0.30$  mm was chosen for study.  $D_m$  was not measured. A modified Picker four-circle diffractometer was used for data collection, with graphite-monochromated Mo  $K\alpha$  radiation,  $\theta/2\theta$  scan technique. Lattice parameters were obtained from measurement of 58 reflections,  $31 \leq 2\theta < 46^\circ$ . Three standard reflections were monitored after every 47 reflections and did not show significant deviations from mean intensities. A total of 3230 unique reflections were measured in the range  $3 \leq 2\theta \leq 50^\circ$

Table 1. Fractional atomic coordinates with *e.s.d.*'s in parentheses and equivalent isotropic thermal parameters ( $\text{\AA}^2 \times 10^2$ )

$$U_{\text{eq}} = \frac{1}{3} \sum_i a_i^* a_i^* a_i \cdot a_i$$

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{eq}}$
O(1)	0.2538 (1)	0.15406 (4)	0.6310 (1)	4.21 (8)
O(2)	0.1750 (1)	0.21954 (5)	0.6029 (2)	6.5 (1)
C(1)	0.2496 (2)	0.14632 (7)	0.4797 (2)	4.0 (1)
C(2)	0.1848 (2)	0.11517 (6)	0.4353 (2)	3.9 (1)
C(3)	0.2146 (2)	0.19305 (8)	0.6788 (2)	4.9 (2)
C(4)	0.2301 (4)	0.1965 (1)	0.8394 (3)	8.3 (3)
C(5)	0.3259 (2)	0.17233 (6)	0.3985 (2)	4.0 (1)
C(6)	0.4186 (2)	0.18339 (8)	0.4597 (3)	5.1 (2)
C(7)	0.4904 (2)	0.20736 (9)	0.3853 (3)	6.4 (2)
C(8)	0.4709 (2)	0.22094 (9)	0.2460 (3)	6.6 (2)
C(9)	0.3791 (2)	0.21090 (9)	0.1852 (3)	6.7 (2)
C(10)	0.3068 (2)	0.18730 (8)	0.2588 (3)	5.5 (2)
C(11)	0.1865 (2)	0.09832 (7)	0.2838 (2)	4.3 (1)
C(12)	0.2746 (2)	0.08162 (8)	0.2228 (3)	5.6 (2)
C(13)	0.2751 (3)	0.06813 (9)	0.0797 (3)	7.2 (2)
C(14)	0.1879 (4)	0.0717 (1)	-0.0010 (3)	8.5 (3)
C(15)	0.0996 (3)	0.0872 (1)	0.0592 (3)	8.0 (2)
C(16)	0.0982 (2)	0.10024 (8)	0.2025 (3)	6.0 (2)
C(17)	0.1079 (2)	0.09519 (7)	0.5343 (2)	4.0 (1)
C(18)	0.1085 (2)	0.05027 (8)	0.5629 (2)	4.9 (2)
C(19)	0.0380 (2)	0.03145 (9)	0.6554 (3)	5.5 (2)
C(20)	-0.0366 (2)	0.05676 (8)	0.7200 (2)	5.1 (2)
C(21)	-0.0385 (2)	0.10128 (9)	0.6895 (3)	5.4 (2)
C(22)	0.0327 (2)	0.10267 (8)	0.5986 (2)	4.9 (2)
C(23)	-0.1144 (3)	0.0361 (2)	0.8197 (4)	8.2 (2)

Table 2. Bond lengths ( $\text{\AA}$ ) and angles ( $^\circ$ ) with *e.s.d.*'s in parentheses

C(1)–C(2)	1.333 (3)	C(1)–O(1)	1.426 (2)
C(1)–C(5)	1.480 (3)	C(2)–C(11)	1.497 (3)
C(2)–C(17)	1.493 (3)	C(3)–O(1)	1.359 (3)
C(3)–O(2)	1.186 (3)	C(3)–C(4)	1.509 (4)
C(5)–C(6)	1.383 (3)	C(5)–C(10)	1.397 (3)
C(6)–C(7)	1.374 (4)	C(7)–C(8)	1.381 (4)
C(8)–C(9)	1.365 (4)	C(9)–C(10)	1.369 (4)
C(11)–C(12)	1.384 (3)	C(11)–C(16)	1.383 (4)
C(12)–C(13)	1.390 (4)	C(13)–C(14)	1.373 (5)
C(14)–C(15)	1.370 (6)	C(15)–C(16)	1.389 (4)
C(17)–C(18)	1.381 (3)	C(17)–C(22)	1.387 (3)
C(18)–C(19)	1.385 (3)	C(19)–C(20)	1.380 (4)
C(20)–C(21)	1.373 (4)	C(20)–C(23)	1.513 (5)
C(21)–C(22)	1.389 (3)		
C(2)–C(1)–O(1)	116.4 (2)	C(2)–C(1)–C(5)	130.4 (2)
O(1)–C(1)–C(5)	112.9 (2)	C(1)–C(2)–C(11)	121.4 (2)
C(1)–C(2)–C(17)	121.7 (2)	C(11)–C(2)–C(17)	116.9 (2)
C(1)–O(1)–C(3)	116.7 (2)	O(1)–C(3)–O(2)	123.8 (2)
O(1)–C(3)–C(4)	109.4 (2)	O(2)–C(3)–C(4)	126.9 (3)
C(1)–C(5)–C(6)	120.9 (2)	C(1)–C(5)–C(10)	121.6 (2)
C(6)–C(5)–C(10)	117.5 (2)	C(5)–C(6)–C(7)	121.5 (2)
C(6)–C(7)–C(8)	120.1 (3)	C(7)–C(8)–C(9)	119.0 (3)
C(8)–C(9)–C(10)	121.4 (3)	C(5)–C(10)–C(9)	120.5 (2)
C(2)–C(11)–C(12)	121.4 (2)	C(2)–C(11)–C(16)	119.2 (2)
C(12)–C(11)–C(16)	119.4 (2)	C(11)–C(12)–C(13)	120.2 (3)
C(12)–C(13)–C(14)	119.7 (3)	C(13)–C(14)–C(15)	120.6 (3)
C(14)–C(15)–C(16)	119.9 (3)	C(11)–C(16)–C(15)	120.1 (3)
C(2)–C(17)–C(18)	120.7 (2)	C(2)–C(17)–C(22)	121.5 (2)
C(18)–C(17)–C(22)	117.7 (2)	C(17)–C(18)–C(19)	121.2 (2)
C(18)–C(19)–C(20)	121.2 (2)	C(19)–C(20)–C(21)	117.7 (2)
C(19)–C(20)–C(23)	121.2 (3)	C(21)–C(20)–C(23)	121.1 (3)
C(20)–C(21)–C(22)	121.7 (2)	C(17)–C(22)–C(21)	120.5 (2)

with  $0 \leq h \leq 15$ ,  $0 \leq k \leq 35$ ,  $0 \leq l \leq 11$  at  $293 \pm 2$  K; of these 2426 reflections were considered observed with  $I \geq 2\sigma(I)$ . Lorentz and polarization corrections were calculated. Absorption corrections were not applied. The structure was solved by direct methods and Fourier syntheses and refined by full-matrix least squares with  $\sigma^2(F)$  weights using the *XTAL* system (Stewart & Hall, 1984). The  $\sigma(F)$  were determined from counting statistics plus a contribution calculated from the excess scatter of the standard reflections. The *E* map obtained from direct methods gave all non-hydrogen atoms. After several cycles of full-matrix least-squares anisotropic refinement with all non-hydrogen atoms, difference Fourier syntheses gave 19 of the 20 hydrogen atoms. The position of the last H atom was calculated with  $[\text{C}–\text{H}] = 1.00$  Å. Hydrogen atoms were refined isotropically and non-hydrogen atoms were refined anisotropically.  $R = 0.043$  and  $wR = 0.037$ .  $(\Delta/\sigma) = 0.011$  in the final least-squares cycle,  $\Delta\rho_{\text{max}} = 0.20 \text{ e } \text{\AA}^{-3}$  in the final difference Fourier map. Atomic scattering factors were from Cromer & Mann (1968). All computations were carried out with a VAX8600 computer and the *XTAL* system of crystallographic programs.

**Discussion.** The atomic coordinates of the non-hydrogen atoms and equivalent isotropic temperature factors are listed in Table 1. Bond lengths and angles are presented in Table 2. A stereoscopic view of the

molecule and the numbering scheme are shown in Fig. 1.\*

A widely used criterion for differentiating between geometric isomers is based on the assumption of a higher melting point for the more symmetrical isomer (Eliel, 1962). For triarylvinyl derivatives with two identical aryl groups, the *trans* isomer would be more symmetrical and should show a higher melting point. From observations of the chemical shifts in the <sup>1</sup>H NMR spectra of *cis* and *trans* pairs of triarylvinyl derivatives, it has also been suggested that for the *cis* isomer the methoxy protons of an anisyl group should appear at higher field and the methyl protons of a tolyl group at lower field than for the corresponding *trans* isomer (Rappoport & Apeloig, 1969; Rappoport & Houminer, 1973).

A crystallographic confirmation of the above generalizations was obtained in the case of *cis*- and *trans*-1,2-dianisyl-2-tolylvinyl bromides (Wanigasekera, Lee, Houminer, Aviv & Rappoport, 1984). The crystal structure showed the lower melting isomer to be the *cis* isomer, and its methoxy absorptions at  $\delta$  3.67 and  $\delta$  3.77 p.p.m. appear at higher field than the corresponding absorptions at  $\delta$  3.70 and  $\delta$  3.79 p.p.m. for the *trans* isomer. Its methyl absorption at  $\delta$  2.33 p.p.m. occurred at lower field than that at  $\delta$  2.17 p.p.m. for the *trans* isomer. From the trifluoroethanolysis of *cis*- and *trans*-1,2-diphenyl-2-tolylvinyl bromides, only one of the products was isolated in pure crystalline form (Lee & Wanigasekera, 1986), and crystallographic analysis showed that this was *cis*-

1,2-diphenyl-2-tolylvinyl 2,2,2-trifluoroethyl ether (Luo, Barton, Robertson, Lee & Wanigasekera, 1986). The tolyl-CH<sub>3</sub> absorption for this *cis* isomer at  $\delta$  2.33 p.p.m. appeared at a lower field than that for the *trans* isomer at  $\delta$  2.20 p.p.m. The melting point and chemical shift data for both of the foregoing compounds are consistent with the suggested criteria for assigning configuration of triarylvinyl derivatives.

However, crystallographic analysis of the *cis*- and *trans*-1,2-diphenyl-2-tolylvinyl bromides showed that the lower melting isomer was the *trans* isomer while the higher melting one was *cis* (Luo, Barton, Robertson, Lee & Wanigasekera, 1987). Moreover, the tolyl-CH<sub>3</sub> absorption for the *cis* isomer appeared at  $\delta$  2.20 p.p.m., a higher field than that for the *trans* isomer at  $\delta$  2.39 p.p.m. As is evident from Fig. 1, the structure reported here is that of *cis*-1,2-diphenyl-2-tolylvinyl acetate. Neither of these latter compounds (the 1,2-diphenyl-2-tolylvinyl bromides and the 1,2-diphenyl-2-tolylvinyl acetates) are consistent with the aforementioned criteria involving melting point and chemical shifts for the identification of the *trans* and *cis* isomers. It would seem that the only reliable method for assigning the configuration of these compounds is to determine the structure by X-ray diffraction.

The structure of the title compound is that of a vinyl propeller. The vinylic C=C double bond is slightly twisted with an angle of 9.8(2)° between the planes defined by C(5)C(1)O(1) and C(11)C(2)C(17). The three aryl rings are planar with a maximum deviation of 0.015 Å. They are twisted at torsional angles  $\alpha = 31.3(2)$ ,  $\beta = 56.0(2)$ ,  $\beta' = 60.7(2)$ ° where  $\alpha$  is defined as the angle between the  $\alpha$  phenyl ring and the C(5)C(1)O(1) plane,  $\beta$  as the angle between the  $\beta$  phenyl ring and the C(11)C(2)C(17) plane and  $\beta'$  as the angle between the  $\beta'$  tolyl ring and the C(11)C(2)C(17) plane. In related triarylvinyl-*X* compounds the torsion angle formed by the largest group is often the largest. Kaftory, Biali & Rappoport (1985) have attributed this to steric effects. In the title compound the *para*-methyl group would seem too remote to affect directly the  $\beta'$  angle. Nevertheless  $\beta'$  remains the largest torsion angle.

The acetate group is planar, within the e.s.d.'s of the constituent atoms. Biali & Rappoport (1984) have discussed the conformation of acetate groups in trimesitylvinyl acetate compounds, in terms of the C(2)–C(1)–O(1)–C(3) and C(1)–O(1)–C(3)–C(4) torsion angles. In the title compound their values are 109.8(2) and 177.3(3)° respectively. Therefore the conformation is anticlinal, antiperiplanar. The C(1)–C(2) bond length of 1.333(3) Å, the C(1)–O(1) bond length of 1.426(2) Å and other bond lengths and angles are similar to those in other triarylvinyl-*X* systems (Biali & Rappoport, 1984; Luo, Barton, Robertson, Lee & Wanigasekera, 1986, 1987; Kaftory, Apeloig & Rappoport, 1985).

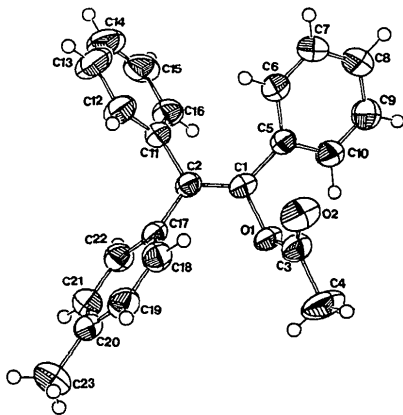


Fig. 1. ORTEP view of the compound with 50% probability ellipsoids.

\* Lists of structure factors, anisotropic temperature factors and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43511 (22 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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## Structure of the Z Isomer of 5-[(4-Methoxyphenyl)methylene]imidazolidine-2,4-dione

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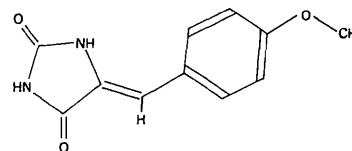
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**Abstract.**  $C_{11}H_{10}N_2O_3$ ,  $M_r = 218.22$ , triclinic,  $P\bar{1}$ ,  $a = 5.350$  (7),  $b = 10.327$  (7),  $c = 10.073$  (7) Å,  $\alpha = 78.7$  (2),  $\beta = 78.0$  (2),  $\gamma = 105.6$  (3)°,  $U = 505.4$  Å<sup>3</sup>,  $Z = 2$ ,  $D_m = 1.40$  (3),  $D_x = 1.43$  g cm<sup>-3</sup>,  $\lambda(\text{Mo K}\alpha) = 0.7107$  Å,  $\mu = 0.66$  cm<sup>-1</sup>,  $F(000) = 228$ , room temperature, final  $R = 0.078$  for 1217 [ $I > 3\sigma(I)$ ] independent observed reflections. The molecule is planar to within 0.30 (1) Å apart from two of the H atoms of the methoxy group. The bond lengths and angles are as expected except for the C–O bond between the methoxy group and the phenyl ring at 1.353 (5) Å, which is shorter than a normal single bond. The two O atoms of the imidazolidine ring are involved in intermolecular hydrogen bonding [O...H–N 2.86 (1) and 2.89 (1) Å].

**Introduction.** Substituted compounds of imidazolidine-2,4-dione (also known as hydantoin) are well documented (*Kirk-Othmer Encyclopedia of Chemical Technology*, 1980). Many have physiological activities and are proprietary preparations, especially those disubstituted at the 5-position, a well-known class of which is the spiro-5-hydantoin. Compounds with a methylene linkage at the 5-position are relatively uncommon and no structure determination has so far

been reported. The title compound is a member of this class.



**Experimental.** Compound prepared as reported elsewhere (Tan, Ang & Fong, 1986). Density measured by flotation in  $CCl_4$ /ligroin. Precession photograph established preliminary cell constants. Crystal of approximate size 0.2 × 0.2 × 0.4 mm mounted on Stoe Stadi-2 diffractometer to rotate about the  $a$  axis. Cell dimensions by measurement of about 20 high-angle axial reflections ( $2\theta$  range 30 to 40°). Intensity data collected via variable-width  $\omega$  scan, background counts every 20 s, step-scan rate 0.033° s<sup>-1</sup>, width  $(1.5 + \sin\mu/\tan\theta)$ . Absorption and extinction corrections not applied. Standard reflections  $h2l$  measured every 20 measurements for each layer: no significant variation in intensity. 1737 unique data measured with  $2\theta_{\text{max}} 50^\circ$  ( $0 \leq h \leq 6$ ,  $-12 \leq k \leq 11$ ,  $-11 \leq l \leq 12$ ). 1217 data