

KAY, M. I. & BEHRENDT, D. R. (1963). *Acta Cryst.* **16**, 157–162.
 LEHMANN, M. & LARSEN, F. K. (1974). *Acta Cryst.* **A30**, 580–584.
 MASLEN, E. N. (1968). *Acta Cryst.* **A24**, 434–437.
 NATKANIEC, I., BIELUSHKIN, A. V. & WASIUTYNSKI, T. (1981).
Phys. Status Solidi B, **105**, 413–423.
 PLAKIDA, N. M., BIELUSHKIN, A. V., NATKANIEC, I. &
 WASIUTYNSKI, T. (1983). *Phys. Status Solidi B*, **118**, 129–133.
 RAICH, J. C. & BERNSTEIN, E. R. (1984). *Mol. Phys.* **53**, 597–614.

RIETVELD, H. M., MASLEN, E. N. & CLEWS, C. J. B. (1970). *Acta Cryst.* **B26**, 693–706.
 TAKEUCHI, H., SUZUKI, S., DIANOUX, A. J. & ALLEN, G. (1981).
Chem. Phys. **55**, 153–162.
 TOUDIC, B., CAILLEAU, H., LECHNER, R. E. & PETRY, W. (1986).
Phys. Rev. Lett. **56**, 347–350.
 TOUDIC, B., GALLIER, J., RIVET, P. & DÉLUGEARD, Y. (1985).
Chem. Phys. **99**, 275–283.

Acta Cryst. (1986). **C42**, 1217–1220

The Structure of Jateorin

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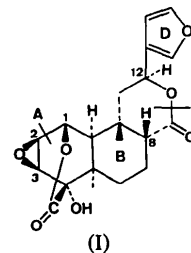
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Abstract. 9-(3-Furyl)decahydro-4-hydroxy-4a,10a-dimethyl-11,12-epoxy-1,4-ethano-3*H*,7*H*-benzo[1,2-*c*:3,4-*c'*]dipyran-3,7-dione, $C_{20}H_{22}O_7$, $M_r = 374.4$, orthorhombic, $P2_12_12_1$, $a = 9.197$ (1), $b = 13.850$ (1), $c = 26.962$ (1) Å, $V = 3434.6$ Å³, $Z = 8$, $D_x = 1.45$ g cm⁻³, Cu $K\alpha$ radiation, Ni filter, $\lambda = 1.5418$ Å, $\mu = 8.73$ cm⁻¹, $F(000) = 1584$, $T = 291$ K, final $R = 0.047$ for 3777 unique observed reflections. The structure and stereochemistry of jateorin proposed by Overton, Weir & Wylie [*J. Chem. Soc. C* (1966), pp. 1482–1490] has been verified.

Introduction. The bitter principle, columbin (Barton & Elad, 1956), is easily epimerized at C(8) by mild treatment with alkali to isocolumbin (Overton, Weir & Wylie, 1961), whose stereochemistry has been established by a crystal structure analysis of its 1-*p*-iodophenyl-3-phenylpyrazoline adduct (Cheung, Melville, Overton, Robertson & Sim, 1966). Here we describe the structure of the 2,3-epoxycolumbin, jateorin, (I), which was isolated as the major product from the alcoholic extract of fresh stems of the plant *Tinospora cordifolia* Miers (Khuda, Khaleque & Roy, 1964). This work was undertaken in order to characterize the compound. The structure confirms that the 1,2-epoxide group adopts a β -configuration in accord with chemical (Barton, Overton & Wylie, 1962; Balasubramanian, Barton & Jackman, 1962) and structural evidence for palmarin (Ferguson & Islam, 1969).



Experimental. Crystals of (I) grow as colourless rectangular prisms; crystal chosen for data collection $0.10 \times 0.08 \times 0.05$ mm; unit-cell parameters obtained by a least-squares fit to the θ values of 75 automatically centred reflections ($27.9 \leq \theta \leq 48.0^\circ$); $1.6 \leq \theta \leq 76.6^\circ$; Enraf–Nonius CAD-4 diffractometer with Cu $K\alpha$ radiation by a θ - 2θ scan technique in 48 steps, time spent measuring the background half that taken to measure the peak; intensity of a reflection and its e.s.d. were calculated from $I = INT - 2(BGL + BGR)$ and $\sigma(I) = [INT + 4(BGL + BGR)]^{0.5}$, where INT, BGL and BGR are the peak intensity, left, and right background counts, respectively. The horizontal detector aperture and the ω -scan range varied as $3.2 + 1.25 \tan\theta$ mm and $0.8 + 0.35 \tan\theta^\circ$. Three standard reflections, remeasured every 100 reflections, no significant variation; data corrected for Lorentz and polarization effects, but not for absorption. $\sigma(F)$ calculated from $\sigma(F) = [\sigma(I)^2 + (Ik)^2]^{0.5}/2F$, where $k = 0.02$. Of a total of 4067 ($+h, +k, +l$) measured intensities ($0 \leq h \leq 11, 0 \leq k \leq 17, 0 \leq l \leq 34$), 3777

satisfied the criterion $I \geq 2.0\sigma(I)$, and only these were used in the solution and refinement of the structure. Computer programs used in this investigation include modified versions of *DATA*P data reduction program (Coppens, Leiserowitz & Rabinovich, 1965), *TRACER* (Jacobson & Lawton, 1965) for cell reduction, *SHELX* (Sheldrick, 1974) for crystal-structure solution and refinement, Weizmann Institute's *BDSL* least-squares program for blocked-diagonal refinement (Busing, Martin & Levy, 1962), *DAESD* program for distances and angles (Davis & Harris, 1970), *XANADU* molecular geometry program (Roberts & Sheldrick, 1976), and *ORTEP* thermal ellipsoid plotting program (Johnson, 1976). No extinction corrections were applied.

Structure solved by direct methods by symbolic addition and subsequent tangent formula expansion. Refinement by blocked least-squares methods, where the function minimized was $\sum w(\Delta F)^2$ with $w = 1/\sigma^2(F)$ and $\Delta F = |F_o| - |F_c|$. Refinement of the non-hydrogen-atom positional and isotropic thermal parameters resulted in $R = 0.15$. Inclusion of anisotropic thermal motion for all non-hydrogen atoms lowered R to 0.074. The positions of all the H atoms except for those bonded to O atoms were included in the refinement at calculated positions (C—H 0.95 Å, $U_H = 0.065 \text{ \AA}^2$). Refinement converged at $R = 0.047$ ($wR = 0.071$) for the 487 variables and 3777 reflections, and the value of S was 2.64, indicating a slight underestimation of the error of an observation of unit weight. In the final refinement cycle the maximum shift-to-error ratio was 0.08. A correction for the effects of anomalous dispersion for O ($f' 0.047$, $f'' 0.032$) was included in the structure-factor calculations, and refinement of the enantiomorphic structure gave no improvement in the weighted R value. The configuration was therefore chosen to correspond to that described by Overton, Weir & Wylie (1966). Atomic scattering curves were taken from *International Tables for X-ray Crystallography* (1974). In the final difference Fourier synthesis, peaks of $\pm 0.41 \text{ e \AA}^{-3}$ were observed.*

Discussion. The results of the X-ray analysis are summarized in Tables 1 and 2, which give the final atomic coordinates and selected interatomic distances and angles. Fig. 1 shows the molecular structure of (I). There are two crystallographically independent molecules in the unit cell and these differ only in the orientation of the furan ring about the C(12)—C(13) bond (Table 2).

* Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and a table of bond distances and angles have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42960 (28 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2)*

The atomic numbering scheme is shown in Fig. 1.

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
O(11)	-0.6433 (5)	0.4446 (3)	0.1912 (2)	0.151
O(21)	-0.7923 (2)	0.5956 (2)	0.2753 (1)	0.061
O(31)	0.1964 (3)	0.2945 (2)	0.3847 (1)	0.063
O(41)	-0.2021 (3)	0.4309 (1)	0.3920 (1)	0.052
O(51)	-0.6249 (4)	0.6915 (2)	0.2070 (1)	0.089
O(61)	-0.2922 (4)	0.5359 (2)	0.4431 (1)	0.080
O(71)	-0.4526 (3)	0.5802 (3)	0.2095 (1)	0.087
C(11)	-0.4141 (4)	0.4898 (3)	0.2339 (1)	0.071
C(21)	-0.5363 (6)	0.4179 (3)	0.2268 (2)	0.097
C(31)	-0.6715 (5)	0.4565 (3)	0.2427 (2)	0.078
C(41)	-0.6544 (3)	0.5584 (2)	0.2637 (1)	0.044
C(51)	-0.5461 (3)	0.5531 (2)	0.3088 (1)	0.031
C(61)	-0.5202 (3)	0.6550 (2)	0.3279 (1)	0.046
C(71)	-0.4048 (5)	0.6590 (3)	0.3683 (2)	0.083
C(81)	-0.2724 (3)	0.5921 (2)	0.3604 (1)	0.044
C(91)	-0.2578 (3)	0.5538 (2)	0.3067 (1)	0.036
C(101)	-0.4022 (3)	0.5058 (2)	0.2902 (1)	0.032
C(111)	-0.1323 (3)	0.4800 (2)	0.3074 (1)	0.041
C(121)	-0.1537 (3)	0.3979 (2)	0.3435 (1)	0.038
C(131)	-0.0150 (3)	0.3441 (2)	0.3524 (1)	0.038
C(141)	0.0532 (5)	0.2781 (3)	0.3201 (1)	0.066
C(151)	0.1741 (4)	0.2480 (2)	0.3413 (1)	0.064
C(161)	0.0807 (3)	0.3539 (2)	0.3908 (1)	0.050
C(171)	-0.5815 (4)	0.6169 (3)	0.2239 (1)	0.059
C(181)	-0.6159 (4)	0.4909 (3)	0.3492 (1)	0.061
C(191)	-0.2064 (4)	0.6371 (3)	0.2736 (2)	0.074
C(201)	-0.2586 (3)	0.5177 (2)	0.4010 (1)	0.047
O(12)	0.1067 (2)	0.1465 (2)	0.8850 (1)	0.071
O(22)	0.3894 (3)	0.3083 (1)	0.8904 (1)	0.052
O(32)	0.5799 (4)	-0.3711 (2)	1.0463 (1)	0.070
O(42)	0.6289 (3)	-0.0621 (1)	1.0281 (1)	0.056
O(52)	0.3731 (3)	0.1994 (2)	0.8068 (1)	0.062
O(62)	0.7725 (4)	0.0628 (2)	1.0323 (1)	0.074
O(72)	0.3383 (2)	0.0642 (1)	0.8487 (1)	0.046
C(12)	0.3081 (3)	0.0285 (2)	0.8981 (1)	0.039
C(22)	0.1826 (3)	0.0850 (2)	0.9193 (1)	0.053
C(32)	0.2110 (3)	0.1873 (2)	0.9184 (1)	0.049
C(42)	0.3605 (3)	0.2090 (2)	0.8967 (1)	0.036
C(52)	0.4784 (3)	0.1563 (2)	0.9293 (1)	0.030
C(62)	0.6289 (3)	0.1731 (2)	0.9064 (1)	0.039
C(72)	0.7521 (3)	0.1151 (2)	0.9309 (1)	0.047
C(82)	0.7057 (3)	0.0139 (2)	0.9493 (1)	0.037
C(92)	0.5675 (3)	-0.0254 (2)	0.9240 (1)	0.029
C(102)	0.4393 (2)	0.0460 (2)	0.9322 (1)	0.027
C(112)	0.5361 (3)	-0.1240 (2)	0.9479 (1)	0.035
C(122)	0.5185 (3)	-0.1209 (2)	1.0034 (1)	0.036
C(132)	0.5257 (3)	-0.2184 (2)	1.0280 (1)	0.039
C(142)	0.4666 (4)	-0.2426 (2)	1.0751 (1)	0.047
C(152)	0.5025 (4)	-0.3341 (2)	1.0841 (1)	0.050
C(162)	0.5942 (5)	-0.2984 (2)	1.0117 (1)	0.059
C(172)	0.3581 (3)	0.1613 (2)	0.8461 (1)	0.040
C(182)	0.4735 (4)	0.2011 (2)	0.9813 (1)	0.048
C(192)	0.6016 (4)	-0.0460 (2)	0.8689 (1)	0.046
C(202)	0.7025 (3)	0.0094 (2)	1.0061 (1)	0.045

Distances and angles correspond closely with those reported for isocolumbin (Cheung, Melville, Overton, Robertson & Sim, 1966), the essential differences resulting from the opposite configuration of C(8) (Table 2). The groups defined by the atoms [C(12), C(13), C(14), C(15), C(16), O(3)], [C(8), C(12), C(20), O(4), O(6)] and [C(1), C(4), C(17), O(5), O(7)] are all coplanar to within 0.04 Å [molecule (1); 0.12 Å molecule (2)]. The epoxide ring is found to be in the β -configuration as was observed for palmarin (Ferguson & Islam, 1969) and proposed by Overton, Weir & Wylie (1966) for jateorin.

Table 2. Selected interatomic distances (Å) and angles (°), with estimated standard deviations in parentheses

	Molecule (1)	Molecule (2)
C(1)—C(2)	1.515 (6)	1.508 (4)
C(1)—O(7)	1.458 (5)	1.447 (3)
C(1)—C(10)	1.536 (4)	1.537 (3)
C(2)—O(1)	1.424 (7)	1.438 (4)
C(2)—C(3)	1.421 (7)	1.442 (4)
C(3)—O(1)	1.424 (6)	1.432 (4)
C(3)—C(4)	1.531 (5)	1.525 (4)
C(4)—C(17)	1.504 (5)	1.517 (4)
C(4)—O(2)	1.404 (4)	1.411 (3)
C(4)—C(5)	1.573 (4)	1.576 (3)
C(5)—C(10)	1.560 (3)	1.571 (3)
C(5)—C(18)	1.531 (4)	1.535 (4)
C(5)—C(6)	1.522 (4)	1.533 (4)
C(6)—C(7)	1.521 (5)	1.539 (4)
C(7)—C(8)	1.546 (5)	1.546 (4)
C(8)—C(20)	1.508 (4)	1.535 (4)
C(8)—C(9)	1.548 (4)	1.542 (4)
C(9)—C(19)	1.535 (5)	1.545 (3)
C(9)—C(10)	1.551 (3)	1.556 (3)
C(9)—C(11)	1.542 (4)	1.538 (3)
C(11)—C(12)	1.510 (4)	1.506 (4)
C(12)—O(4)	1.455 (3)	1.462 (3)
C(12)—C(13)	1.497 (4)	1.506 (4)
C(13)—C(16)	1.365 (4)	1.349 (4)
C(13)—C(14)	1.411 (4)	1.423 (4)
C(14)—C(15)	1.320 (6)	1.332 (4)
C(15)—O(3)	1.352 (4)	1.344 (4)
C(16)—O(3)	1.355 (4)	1.380 (4)
C(17)—O(5)	1.199 (5)	1.191 (3)
C(17)—O(7)	1.348 (5)	1.359 (3)
C(20)—O(4)	1.332 (4)	1.337 (4)
C(20)—O(6)	1.205 (4)	1.209 (4)
C(2)—C(1)—C(10)	105.9 (3)	107.0 (2)
C(2)—C(1)—O(7)	109.1 (3)	108.5 (2)
C(10)—C(1)—O(7)	109.9 (3)	110.2 (2)
C(1)—C(2)—C(3)	111.3 (4)	111.4 (2)
C(1)—C(2)—O(1)	115.4 (4)	115.8 (3)
C(3)—C(2)—O(1)	60.1 (3)	59.6 (2)
C(2)—C(3)—C(4)	111.7 (3)	111.3 (2)
C(2)—C(3)—O(1)	60.1 (3)	60.0 (2)
C(4)—C(3)—O(1)	116.7 (3)	116.2 (3)
C(3)—C(4)—C(5)	107.9 (3)	108.4 (2)
C(3)—C(4)—C(17)	106.2 (3)	104.3 (2)
C(3)—C(4)—O(2)	109.1 (3)	114.1 (2)
C(5)—C(4)—C(17)	107.2 (2)	108.1 (2)
C(5)—C(4)—O(2)	114.7 (2)	112.9 (2)
C(17)—C(4)—O(2)	111.4 (3)	108.7 (2)
C(4)—C(5)—C(6)	108.5 (2)	109.0 (2)
C(4)—C(5)—C(10)	108.0 (2)	108.7 (2)
C(4)—C(5)—C(18)	108.1 (2)	107.6 (2)
C(6)—C(5)—C(10)	111.5 (2)	112.0 (2)
C(6)—C(5)—C(18)	110.3 (2)	109.5 (2)
C(10)—C(5)—C(18)	110.4 (2)	109.9 (2)
C(5)—C(6)—C(7)	112.7 (2)	114.4 (2)
C(6)—C(7)—C(8)	115.5 (3)	114.0 (2)
C(7)—C(8)—C(9)	113.7 (3)	114.0 (2)
C(7)—C(8)—C(20)	112.1 (3)	111.3 (2)
C(9)—C(8)—C(20)	115.9 (2)	114.3 (2)
C(8)—C(9)—C(10)	110.0 (2)	109.7 (2)
C(8)—C(9)—C(11)	106.3 (2)	106.5 (2)
C(8)—C(9)—C(19)	108.3 (2)	108.8 (2)
C(10)—C(9)—C(11)	111.2 (2)	111.3 (2)
C(10)—C(9)—C(19)	114.7 (2)	114.0 (2)
C(11)—C(9)—C(19)	106.0 (2)	106.1 (2)
C(1)—C(10)—C(5)	108.5 (2)	107.7 (2)
C(1)—C(10)—C(9)	114.0 (2)	114.2 (2)
C(5)—C(10)—C(9)	117.0 (2)	116.0 (2)
C(9)—C(11)—C(12)	114.2 (2)	114.3 (2)
C(11)—C(12)—C(13)	111.6 (2)	113.9 (2)
C(11)—C(12)—O(4)	112.4 (2)	113.1 (2)
C(13)—C(12)—O(4)	105.8 (2)	105.6 (2)
C(12)—C(13)—C(14)	127.0 (3)	126.0 (2)
C(12)—C(13)—C(16)	128.5 (2)	127.9 (3)
C(14)—C(13)—C(16)	104.2 (3)	106.0 (2)
C(13)—C(14)—C(15)	108.1 (3)	106.9 (3)
C(14)—C(15)—O(3)	110.7 (3)	111.0 (3)
C(13)—C(16)—O(3)	110.8 (3)	109.6 (3)
C(4)—C(17)—O(5)	126.0 (3)	127.3 (3)
C(4)—C(17)—O(7)	113.3 (3)	112.7 (2)
O(5)—C(17)—O(7)	120.6 (3)	120.1 (3)
C(8)—C(20)—O(4)	121.2 (2)	118.8 (2)
C(8)—C(20)—O(6)	121.3 (3)	123.2 (3)
O(4)—C(20)—O(6)	117.4 (3)	117.7 (3)
C(2)—O(1)—C(3)	59.9 (3)	60.3 (2)

Table 2(cont.)

	Molecule (1)	Molecule (2)
C(15)—O(3)—C(16)	105.9 (3)	106.5 (2)
C(12)—O(4)—C(20)	124.5 (5)	124.3 (2)
C(1)—O(7)—C(17)	114.1 (3)	114.4 (2)
C(5)—C(6)—C(7)—C(8)	39.2	34.8
C(6)—C(7)—C(8)—C(9)	16.1	21.1
C(6)—C(7)—C(8)—C(20)	-117.8	-109.9
C(7)—C(8)—C(9)—C(10)	-53.1	-57.7
C(7)—C(8)—C(9)—C(11)	-173.5	-178.3
C(7)—C(8)—C(9)—C(19)	73.0	67.7
C(20)—C(8)—C(9)—C(10)	79.0	71.8
C(20)—C(8)—C(9)—C(11)	-41.4	-48.8
C(20)—C(8)—C(9)—C(19)	-155.0	-162.8
C(11)—C(12)—C(13)—C(14)	-75.2	-157.6
C(11)—C(12)—C(13)—C(16)	97.9	26.0
O(4)—C(12)—C(13)—C(14)	162.3	77.6
O(4)—C(12)—C(13)—C(16)	-24.7	-98.7
O(11)...C(192 ⁱ)	3.180 (5)	
O(21)...O(52 ⁱⁱ)	3.056 (4)	
O(22)...O(62 ⁱⁱⁱ)	2.948 (3)	

Superscripts i, ii, iii refer to the symmetry operations: $[x - \frac{3}{2}, \frac{1}{2} - y, 1 - z]$; $[-\frac{1}{2} - x, 1 - y, z - \frac{1}{2}]$ and $[x - \frac{1}{2}, \frac{1}{2} - y, 2 - z]$.

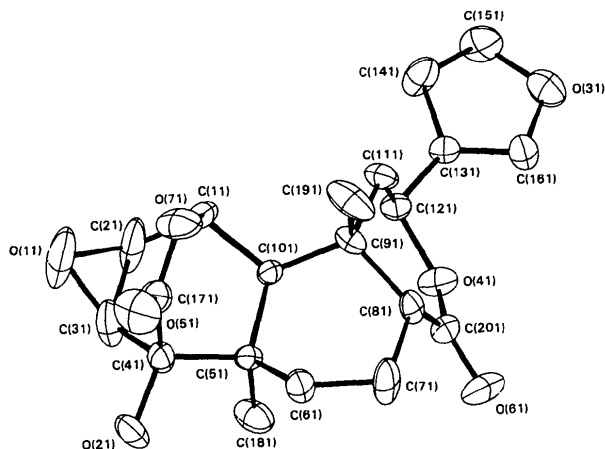


Fig. 1. The molecular structure of 2,3-epoxycolumbin. (1 at the end of the atom number is used to designate molecule 1.)

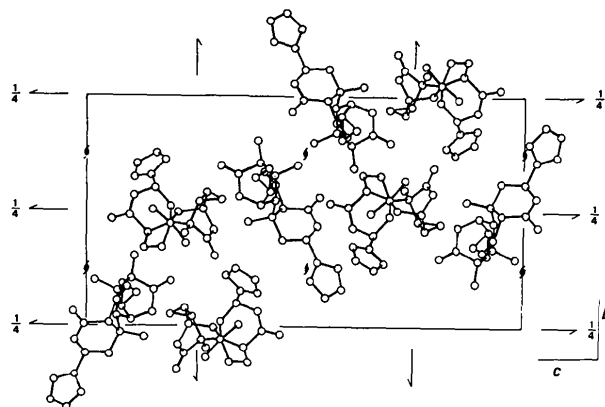


Fig. 2. The contents of the unit cell of columbin-2-epoxide, viewed down a looking towards the origin.

The packing of the molecules in the unit cell is shown in Fig. 2. Intermolecular contacts less than 3.2 Å are given in Table 2.

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References

- BALASUBRAMANIAN, S. K., BARTON, D. H. R. & JACKMAN, L. M. (1962). *J. Chem. Soc.* pp. 4816–4820.
- BARTON, D. H. R. & ELAD, D. (1956). *J. Chem. Soc.* pp. 2085–2090; 2090–2095.
- BARTON, D. H. R., OVERTON, K. H. & WYLIE, A. (1962). *J. Chem. Soc.* pp. 4809–4815.
- BUSING, W. R., MARTIN, K. O. & LEVY, H. A. (1962). *ORFLS*. Report ORNL-TM-305. Oak Ridge National Laboratory, Tennessee, USA.
- CHEUNG, K. K., MELVILLE, D., OVERTON, K. H., ROBERTSON, J. M. & SIM, G. A. (1966). *J. Chem. Soc. B*, pp. 853–861.
- COPPENS, P., LEISEROWITZ, L. & RABINOVICH, D. (1965). *Acta Cryst.* **18**, 1035–1038.
- DAVIS, R. E. & HARRIS, D. R. (1970). *DAESD*. Roswell Park Mem. Inst., USA.
- FERGUSON, G. & ISLAM, K. M. S. (1969). *J. Chem. Soc. B*, pp. 162–170.
- International Tables for X-ray Crystallography* (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JACOBSON, R. A. & LAWTON, S. L. (1965). *TRACER*. United States Energy Commission, Report TID-4500. Iowa State Univ., USA.
- JOHNSON, C. K. (1976). *ORTEP*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
- KHUDA, M. Q., KHALEQUE, A. & ROY, N. (1964). *Scientific Res. (Dacca, Bangladesh)*, **1**, 177.
- OVERTON, K. H., WEIR, N. G. & WYLIE, A. (1961). *Proc. Chem. Soc.* pp. 211–212.
- OVERTON, K. H., WEIR, N. G. & WYLIE, A. (1966). *J. Chem. Soc. C*, pp. 1482–1490.
- ROBERTS, P. & SHELDRIK, G. M. (1976). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
- SHELDRIK, G. M. (1974). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of (Z)-1,2-Diphenyl-2-p-tolylvinyl 2,2,2-Trifluoroethyl Ether

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Abstract. $C_{23}H_{19}F_3O$, $M_r = 368.40$, monoclinic, $P2_1$, $a = 10.590$ (2), $b = 9.634$ (2), $c = 9.582$ (2) Å, $\beta = 95.57$ (2)°, $V = 972.98$ Å³, $Z = 2$, $D_x = 1.26$ g cm⁻³, $D_m = 1.23$ g cm⁻³ by flotation, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo}) = 0.90$ cm⁻¹, $F(000) = 384$, $T = 291$ K, $R = 0.061$ for 1745 observed reflections. The configuration of the molecule is the (Z) isomer. The bond lengths and angles are in agreement with those of similar compounds. The molecule has a propeller conformation.

Introduction. In order to study the solvolytic rearrangement of ¹³C or ¹⁴C labeled (E)- and (Z)-1,2-diphenyl-2-tolylvinyl bromide, the structure of the title compound has been determined to verify the configuration assignment based on ¹H NMR spectra.

Experimental. Crystals of the title compound obtained from the reaction of 1,2-diphenyl-2-tolylvinyl bromide in trifluoroethanol and 2,6-lutidine, and recrystallized from 90% ethanol (Lee & Wanigasekera, 1986). A pale yellow crystal of dimensions 0.1 × 0.3 × 0.3 mm

chosen for study. Intensity data collected with a modified Picker four-circle diffractometer, $\theta/2\theta$ scan technique, graphite-monochromated Mo $K\alpha$ radiation. Three standard reflections monitored after every 47 reflections. The intensity of the standard reflections did not change significantly. Two equivalent sets of data collected in the 2θ range 3 to 53° at 291 ± 2 K in octants (+ $h, +k, +l$) and (- $h, -k, -l$) with $0 \leq h \leq 13$, $0 \leq k \leq 12$ and $0 \leq l \leq 12$. After averaging, 2128 independent reflections used in the structure determination, of which 1745 reflections were considered as observed with $I > 2\sigma(I)$. Lorentz and polarization corrections. Absorption corrections not applied. The E map obtained from direct methods using the *XTAL* system of crystallographic programs (Stewart & Hall, 1984) gave 23 of 27 non-hydrogen atoms. A subsequent difference Fourier synthesis gave all non-hydrogen atoms. After several cycles of full-matrix least-squares anisotropic refinement with all non-hydrogen atoms, difference Fourier syntheses gave only six of the 19 H atoms; remaining H atoms were placed at idealized positions with C–H = 1.00 Å. Tem-