

Fig. 1. Molecular structure and atomic numbering.

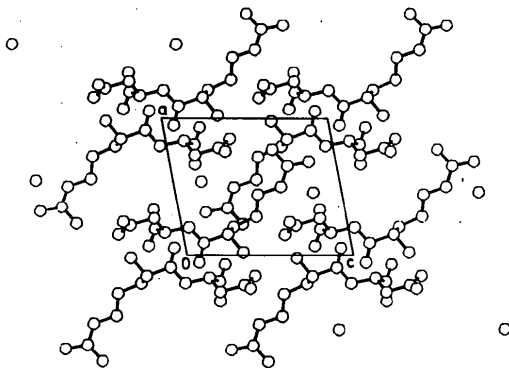


Fig. 2. View of crystal packing along the *b* axis.

**Related literature.** The guanidino group N atoms N4 and N5 interact directly with the peptide backbone O atoms O1 and O2 in contrast with the crystal structure

of the dihydrated form of the peptide (Ramakrishnan & Viswamitra, 1988). The side-chain conformation of arginine has  $\chi^1 = -64.2^\circ$  and  $\chi^2 = -71.4^\circ$ , predicted as a possible geometry (Ponnuswamy & Sasisekharan, 1971) but not seen in earlier arginine crystal structures (Pandit, Seshadri & Viswamitra, 1983; Bhat & Vijayan, 1977).

We thank Dr T. P. Seshadri for his help during data collection and DST and DBT for financial support.

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*Acta Cryst.* (1989). C45, 824–826

## The Structure of 1-Benzylidene-2,3-diphenylindene

BY CHRISTINE R. A. MUCHMORE

*The Upjohn Company, Kalamazoo, Michigan 49001, USA*

AND MARY JANE HEEG\*

*Department of Chemistry, Wayne State University, Detroit, Michigan 48202, USA*

(Received 8 July 1988; accepted 8 November 1988)

**Abstract.**  $C_{28}H_{20}$ ,  $M_r = 356.47$ ,  $P2_1/c$ ,  $a = 10.948$  (2),  $b = 19.856$  (6),  $c = 10.173$  (1) Å,  $\beta = 117.40$  (1)°,  $V = 1963.3$  (7) Å<sup>3</sup>,  $Z = 4$ ,  $D_x = 1.206$  g cm<sup>-3</sup>,  $\lambda(\text{Mo } K\alpha) = 0.71073$  Å,  $\mu = 0.34$  cm<sup>-1</sup>,  $F(000) = 752$ ,  $T = 298$  (2) K,  $R = 0.043$  for 2705 unique observed reflections. The indene portion of the molecule exhibits a small fold of 2.6 (1)° between the fused rings. Lengths and angles within the indene fragment are normal and vary according to hybridization. The five-membered ring is planar (r.m.s.d. = 0.01 Å) as are all six-membered

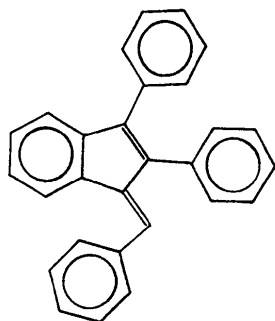
rings. The C=C double bond linking the benzyl group to the five-membered ring is clearly defined by bond length [1.345 (2) Å] and geometry. The benzyl group is related *cis* to the C atom at the fused ring juncture and oriented away from the adjacent phenyl group. Conjugation does not extend beyond the indene fragment to include the phenyl groups. All three pendent phenyl rings are canted 47–57° with respect to the five-membered ring in a paddle-wheel arrangement.

**Experimental.** The title compound was isolated as an unexpected side product from the reaction of elemental

\* To whom all correspondence should be addressed.

bromine with hexaphenylstannole in tetrahydrofuran at 195 K under a nitrogen atmosphere, with a yield of 30%. The reaction mechanism for the formation of this product is unknown. The compound was recrystallized and showed a sharp melting point of 459 K.

Multifaceted orange crystals, 0.4 × 0.5 × 0.6 mm; Nonius CAD-4 diffractometer, monochromated Mo K $\alpha$ ;  $\theta/2\theta$  scans;  $3 \leq 2\theta \leq 52^\circ$ ; lattice parameters from 25 high angle reflections ( $2\theta > 20^\circ$ ); Gaussian integration absorption corrections applied with transmission coefficients 0.987–0.979;  $0 \leq h \leq 13$ ,  $0 \leq k \leq 24$ ,  $-12 \leq l \leq 12$ ; three standard reflections fluctuated 2%; 3966 total reflections, 3842 unique, 2705 observed with  $I_o \geq 2.5\sigma(I)$ . Direct methods; full-matrix refinement via SHELX76 (Sheldrick, 1976) on  $F^*$  minimizing  $\sum w(|F_o| - |F_c|)^2$ ; all C atoms anisotropic; H atoms were placed in a combination of observed and calculated positions and all isotropic H temperature factors were tied to a single variable which refined to 0.077 Å<sup>2</sup>. Number of parameters varied was 255. For observed reflections  $R = 0.043$ ,  $wR = 0.054$ ,  $S = 1.83$ ,  $w = (\sigma_F)^{-2}$ . In a final cycle:  $(\Delta/\sigma)_{\max} < 0.01$ ,  $(\Delta\rho)_{\max} = 0.16$ ,  $(\Delta\rho)_{\min} = -0.16 \text{ e \AA}^{-3}$ . An empirical correction for extinction was applied:  $F_{\text{corr}} = F(1 - 0.001xF^2/\sin\theta)$  where  $x$  refined to 0.00759. Neutral-atom scattering factors from *International Tables for X-ray Crystallography* (1974). Table 1\* gives the atomic coordinates and Table 2 lists bond distances and angles. Fig. 1 illustrates the geometry and labeling scheme. A diagram of the title molecule is shown below.



**Related literature.** Other structurally similar substituted indene molecules have been studied by Gieren & Hahn (1979), Barnes, Paton & Nicholls (1982) and Korp, Bernal, Watkins & Fronczek (1982). It is interesting that the related indene, 2,3-diphenylindene, was isolated as an unexpected side product in the reaction of 1,2,3-triphenylcyclopropene with  $(\text{C}_5\text{H}_5)\text{Co}(\text{CO})_2$  (Jens & Weiss, 1984).

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51586 (17 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

	x	y	z	$U_{\text{eq}}^\dagger (\text{\AA}^2)$
C1	1.1261 (2)	0.39576 (8)	0.4432 (2)	0.0386 (7)
C2	0.9830 (2)	0.41494 (8)	0.3369 (2)	0.0368 (7)
C3	0.8934 (2)	0.36918 (8)	0.3433 (2)	0.0385 (7)
C4	0.9716 (2)	0.31953 (8)	0.4587 (2)	0.0409 (7)
C5	1.1128 (2)	0.33471 (8)	0.5204 (2)	0.0402 (7)
C6	1.2086 (2)	0.29408 (9)	0.6309 (2)	0.0509 (8)
C7	1.1620 (2)	0.24048 (9)	0.6844 (2)	0.059 (1)
C8	1.0222 (2)	0.2276 (1)	0.6275 (2)	0.059 (1)
C9	0.9257 (2)	0.26633 (9)	0.5133 (2)	0.0515 (9)
C10	1.2369 (2)	0.42596 (8)	0.4452 (2)	0.0437 (8)
C11	1.3837 (2)	0.41142 (8)	0.5387 (2)	0.0448 (8)
C12	1.4711 (2)	0.4068 (1)	0.4740 (2)	0.060 (1)
C13	1.6101 (2)	0.3929 (1)	0.5598 (3)	0.069 (1)
C14	1.6644 (2)	0.3851 (1)	0.7104 (3)	0.068 (1)
C15	1.5797 (2)	0.3916 (1)	0.7766 (2)	0.0616 (9)
C16	1.4415 (2)	0.40500 (9)	0.6920 (2)	0.0508 (8)
C17	0.9494 (2)	0.47317 (8)	0.2356 (2)	0.0373 (7)
C18	0.8759 (2)	0.46398 (9)	0.0830 (2)	0.0452 (8)
C19	0.8422 (2)	0.5184 (1)	-0.0119 (2)	0.0563 (9)
C20	0.8823 (2)	0.5825 (1)	0.0435 (3)	0.059 (1)
C21	0.9554 (2)	0.59272 (9)	0.1941 (3)	0.060 (1)
C22	0.9894 (2)	0.53839 (9)	0.2907 (2)	0.0510 (9)
C23	0.7428 (2)	0.36568 (9)	0.2481 (2)	0.0429 (7)
C24	0.6589 (2)	0.4224 (1)	0.2183 (2)	0.0544 (9)
C25	0.5183 (2)	0.4177 (1)	0.1261 (3)	0.067 (1)
C26	0.4597 (2)	0.3564 (1)	0.0636 (3)	0.075 (1)
C27	0.5415 (2)	0.3001 (1)	0.0926 (3)	0.071 (1)
C28	0.6818 (2)	0.3043 (1)	0.1843 (2)	0.0573 (9)

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

Table 2. Bond lengths (Å) and angles (°)

C1–C2	1.486 (2)	C12–C13	1.390 (3)
C1–C5	1.488 (2)	C13–C14	1.373 (2)
C1–C10	1.345 (2)	C14–C15	1.380 (2)
C2–C3	1.361 (2)	C15–C16	1.379 (3)
C2–C17	1.478 (2)	C17–C18	1.394 (2)
C3–C4	1.469 (2)	C17–C22	1.399 (2)
C3–C23	1.480 (2)	C18–C19	1.381 (2)
C4–C5	1.408 (2)	C19–C20	1.380 (3)
C4–C9	1.391 (2)	C20–C21	1.378 (2)
C5–C6	1.389 (2)	C21–C22	1.390 (2)
C6–C7	1.394 (2)	C23–C24	1.396 (3)
C7–C8	1.389 (3)	C23–C28	1.397 (3)
C8–C9	1.388 (3)	C24–C25	1.389 (3)
C10–C11	1.471 (2)	C25–C26	1.386 (3)
C11–C12	1.391 (2)	C26–C27	1.377 (3)
C11–C16	1.393 (2)	C27–C28	1.385 (3)
C1–C2–C3	109.6 (1)	C6–C7–C8	120.6 (2)
C1–C2–C17	123.3 (1)	C7–C8–C9	121.0 (2)
C1–C5–C4	107.0 (1)	C10–C11–C12	119.4 (1)
C1–C5–C6	132.9 (2)	C10–C11–C16	122.7 (1)
C1–C10–C11	129.2 (1)	C11–C12–C13	120.5 (1)
C2–C1–C5	105.5 (1)	C11–C16–C15	121.3 (1)
C2–C1–C10	122.6 (1)	C12–C11–C16	117.8 (2)
C2–C3–C4	108.6 (1)	C12–C13–C14	120.7 (2)
C2–C3–C23	127.7 (1)	C13–C14–C15	119.4 (2)
C2–C17–C18	120.4 (1)	C14–C15–C16	120.2 (1)
C2–C17–C22	120.8 (1)	C17–C18–C19	120.5 (2)
C3–C2–C17	127.0 (1)	C17–C22–C21	120.1 (1)
C3–C4–C5	109.2 (1)	C18–C17–C22	118.8 (1)
C3–C4–C9	130.1 (2)	C18–C19–C20	120.3 (1)
C3–C23–C24	121.9 (2)	C19–C20–C21	120.2 (2)
C3–C23–C28	119.7 (2)	C20–C21–C22	120.1 (2)
C4–C3–C23	123.7 (1)	C23–C24–C25	120.4 (2)
C4–C5–C6	120.1 (1)	C23–C28–C27	120.7 (2)
C4–C9–C8	118.6 (2)	C24–C23–C28	118.5 (2)
C5–C1–C10	131.4 (2)	C24–C25–C26	120.3 (2)
C5–C4–C9	120.6 (1)	C25–C26–C27	119.8 (2)
C5–C6–C7	118.9 (2)	C26–C27–C28	120.3 (2)

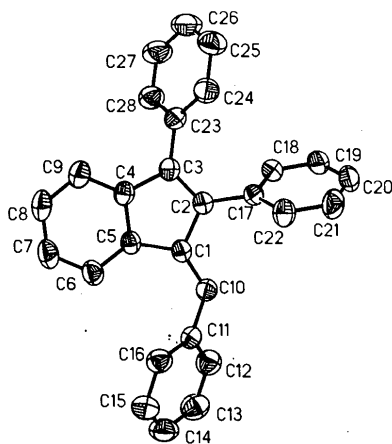


Fig. 1. ORTEP drawing (at 50% probability) of 1-benzylidene-2,3-diphenylindene showing the adopted labeling.

This compound was prepared under the auspices of the late Professor J. J. Zuckerman, formerly of the Department of Chemistry, University of Oklahoma, Norman, OK 73109.

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*Acta Cryst.* (1989). **C45**, 826–827

### Ethyl 2,3-Bis(trifluoromethyl)-2,3-dihydroindolizine-1-carboxylate

BY R. G. PRITCHARD, R. E. BANKS AND S. MOHIALDIN

*Department of Chemistry, University of Manchester Institute of Science and Technology, PO Box 88, Manchester M60 1QD, England*

(Received 30 September 1988; accepted 10 November 1988)

**Abstract.**  $C_{13}H_{11}F_6NO_2$ ,  $M_r = 327.2$ , monoclinic,  $P2_1/c$ ,  $a = 15.144$  (2),  $b = 14.036$  (2),  $c = 14.101$  (2) Å,  $\beta = 110.99$  (1)°,  $V = 2798.4$  Å<sup>3</sup>,  $Z = 8$ ,  $D_x = 1.55$  Mg m<sup>-3</sup>,  $F(000) = 1328$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069$  Å,  $\mu = 0.110$  mm<sup>-1</sup>,  $T = 293$  K,  $R = 0.054$  for 2533 unique reflexions [ $F \geq 3\sigma(F)$ ]. The asymmetric unit contains two molecules distinguished only by a minor disorder of one of the ethyl groups. With the exception of the disordered region, bond lengths agree within  $1\sigma$ , bond angles within  $2\sigma$ , and torsion angles within  $3\sigma$ . A short C–C bond [1.421 (5), 1.425 (6) Å] links the carboxy and indolizine  $\pi$  systems, thereby forming a dipole, whose poles are situated at the ring N and carboxy O.

**Experimental.** The sample was prepared by 1,3-dipolar cycloaddition of *N*-2,2,2-trifluoroethylpyridinium-triflate to perfluorobut-2-yne in the presence of triethylamine and chloroform solvent. Purification by column chromatography followed by recrystallization from methylene chloride and methanol yielded crystals suitable for X-ray work.

Crystal dimensions 0.5 × 0.3 × 0.2 mm, Enraf–Nonius CAD-4 diffractometer, graphite-monochromated Mo  $K\alpha$  radiation, unit-cell dimensions from

setting angles of 25 accurately centred reflexions ( $7.5 \leq \theta \leq 8.7^\circ$ ),  $\omega$ - $2\theta$  scan mode,  $\omega$  scan width  $0.70 + 0.35 \tan \theta$  (°) and scan speed ranging from 0.6 to 5° min<sup>-1</sup> according to the intensity gathered in a pre-scan,  $-17 \leq h \leq 15$ ,  $0 \leq k \leq 16$ ,  $0 \leq l \leq 16$ ,  $0 \leq \theta \leq 25^\circ$ , 5404 reflexions measured, 3691 unique ( $R_{\text{int}} = 0.011$ ), 2533 observed [ $F \geq 3\sigma(F)$ ], intensity standards (602, 053, 115) measured every 2.5 h, decay 12%, decay and Lp corrections applied but absorption ignored, *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980) used to solve the phase problem, all non-hydrogen atoms except minor component of disordered atom C(14B) [C(15B) 8 (2)% occupancy] found in Fourier map, remaining atoms, including H, from  $\Delta F$  synthesis, full-matrix least-squares refinement based on  $F$  using *SHELX76* (Sheldrick, 1976) with bond lengths C(13B)–C(15B) and C(13B)–C(14B) tied together, final  $R = 0.054$ ,  $wR = 0.045$ ,  $w = 1.9898/[\sigma^2(F) + 0.0003F^2]$ , anisotropic thermal parameters for heavier atoms, isotropic for H and C(15B). Maximum fluctuation in final  $\Delta F$  map in range  $-0.22$  to  $0.24$  e Å<sup>-3</sup>, maximum  $\Delta/\sigma$  0.060 [ $x$ , H(131B)]. Scattering factors from *International Tables for X-ray Crystallography* (1974), computation carried out on the joint CDC7600/