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Structure of 2,3-Diphenyl-1-(*p*-tolyl)-1-aza-1,3-pentadiene

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Abstract. $C_{23}H_{21}N$, $M_r = 311{\cdot}4$, triclinic, $P\bar{1}$, $a = 9{\cdot}927\,(4)$, $b = 10{\cdot}143\,(9)$, $c = 10{\cdot}487\,(3)$ Å, $\alpha = 63{\cdot}29\,(2)$, $\beta = 96{\cdot}26\,(5)$, $\gamma = 108{\cdot}97\,(2)°$, $V = 891{\cdot}2\,(9)$ Å^3, $Z = 2$, $D_x = 1{\cdot}16$ g cm^{-3}, $\lambda(\text{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}621$ cm^{-1}, $F(000) = 332$, $T = 290$ K, $R = 0{\cdot}056$ for 2095 observed reflexions. The two double bonds are oriented synclinal with respect to each other, the N=C—C=C torsion angle being −69·3 (5)°; bond lengths are N=C, 1·280 (4); C—C, 1·486 (4); C=C, 1·327 (6) Å.

Introduction. The title compound has been synthesized and studied by spectroscopy by Arjona, Plumet, Ossorio & Santesmases (1985).

We have determined the crystal structure of this compound in order to establish the stereochemistry around the double-bond system, which was difficult to resolve by conventional techniques such as ^{1}H NMR and ^{13}C NMR spectroscopy.

Experimental. Yellow, prismatic crystal 0·3 × 0·5 × 0·6 mm. Enraf–Nonius CAD-4*F* automatic diffractometer; cell dimensions refined by least-squares fitting of θ values of 25 reflexions; intensities of 3887 unique reflexions, $1 < \theta < 27°$, *hkl* −12,−13,0 to 12,13,13, monochromatic Mo $K\alpha$ radiation, $\omega/2\theta$ scans; three reflexions monitored periodically during data collection showed no crystal decomposition; intensities corrected for Lorentz and polarization effects; 2095 considered observed $[I > 2\sigma(I)]$. Scattering factors for neutral atoms from *International Tables for X-ray Crystallography* (1974). Structure solved with *MULTAN*77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977); best *E* map revealed all non-H atoms. An empirical absorption correction (Walker & Stuart, 1983) was applied at the end of the isotropic refinement; anisotropic full-matrix least-squares refinement, $\sum w(|F_o| - |F_c|)^2$ minimized; unit weights; a difference synthesis calculated with reflexions having $\sin\theta/\lambda < 0{\cdot}5$ Å^{-1} showed all H atoms as highest peaks; final refinement with fixed isotropic temperature factors for H atoms led to $R = 0{\cdot}056$; max. and average Δ/σ in final LS cycle 0·53 and 0·032; final difference synthesis had no electron density $> 0{\cdot}24$ or $< -0{\cdot}22$ e Å^{-3}. No correction for secondary extinction. Most of the calculations performed with *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976). Least-squares planes computed with *PARST*5 (Nardelli, Musatti, Domiano & Andreetti, 1965).

Discussion. Table 1† contains the final atomic parameters. Fig. 1 (Johnson, 1965) shows the geometry of the structure and the atom labelling. Bond lengths and angles together with their e.s.d.'s are in Table 2.

Table 1. *Coordinates and equivalent isotropic thermal parameters* (Å^2 × 10^3)

$$U_{\text{eq}} = \tfrac{1}{3}\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j\cos(a_i,a_j).$$

	x	y	z	U_{eq}
N	−0·0597 (3)	−0·2580 (3)	0·9665 (3)	53 (1)
C(1)	0·0676 (3)	−0·1896 (3)	0·9117 (3)	47 (1)
C(2)	0·1058 (3)	−0·0972 (3)	0·7563 (3)	45 (1)
C(3)	0·0811 (3)	−0·1667 (4)	0·6711 (4)	58 (2)
C(4)	0·0220 (4)	−0·3368 (5)	0·7165 (5)	78 (2)
C(5)	−0·1809 (3)	−0·2430 (4)	0·8833 (3)	51 (2)
C(6)	−0·2051 (3)	−0·1018 (4)	0·8031 (4)	58 (2)
C(7)	−0·3312 (4)	−0·0965 (4)	0·7334 (4)	63 (2)
C(8)	−0·4344 (3)	−0·2292 (4)	0·7391 (4)	60 (2)
C(9)	−0·4094 (4)	−0·3694 (4)	0·8216 (4)	64 (2)
C(10)	−0·2866 (4)	−0·3765 (4)	0·8934 (4)	61 (2)
C(11)	−0·5680 (4)	−0·2209 (5)	0·6572 (5)	87 (3)
C(12)	0·1716 (3)	0·0734 (3)	0·7037 (3)	45 (1)
C(13)	0·1701 (3)	0·1452 (4)	0·7892 (3)	52 (2)
C(14)	0·2309 (4)	0·3039 (4)	0·7426 (4)	58 (2)
C(15)	0·2952 (4)	0·3946 (4)	0·6072 (4)	62 (2)
C(16)	0·2987 (4)	0·3257 (4)	0·5216 (4)	66 (2)
C(17)	0·2392 (4)	0·1679 (4)	0·5683 (4)	61 (2)
C(18)	0·1862 (3)	−0·2086 (3)	1·0118 (3)	49 (2)
C(19)	0·1586 (4)	−0·2618 (4)	1·1572 (4)	61 (2)
C(20)	0·2689 (5)	−0·2797 (5)	1·2503 (4)	75 (2)
C(21)	0·4032 (5)	−0·2468 (5)	1·2011 (5)	80 (2)
C(22)	0·4312 (4)	−0·1955 (5)	1·0585 (5)	79 (2)
C(23)	0·3230 (4)	−0·1747 (4)	0·9641 (4)	64 (2)

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† Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and bond lengths and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42426 (30 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 2. *Bond lengths* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

N—C(1)	1·280 (4)	C(9)—C(10)	1·366 (5)
N—C(5)	1·415 (4)	C(12)—C(13)	1·390 (6)
C(1)—C(2)	1·486 (4)	C(12)—C(17)	1·397 (4)
C(1)—C(18)	1·490 (5)	C(13)—C(14)	1·386 (5)
C(2)—C(3)	1·327 (6)	C(14)—C(15)	1·383 (5)
C(2)—C(12)	1·487 (5)	C(15)—C(16)	1·372 (7)
C(3)—C(4)	1·487 (6)	C(16)—C(17)	1·376 (5)
C(5)—C(6)	1·383 (5)	C(18)—C(19)	1·395 (5)
C(5)—C(10)	1·385 (5)	C(18)—C(23)	1·379 (5)
C(6)—C(7)	1·383 (5)	C(19)—C(20)	1·386 (6)
C(7)—C(8)	1·382 (5)	C(20)—C(21)	1·364 (7)
C(8)—C(9)	1·382 (6)	C(21)—C(22)	1·373 (7)
C(8)—C(11)	1·504 (5)	C(22)—C(23)	1·381 (6)
C(1)—N—C(5)	122·4 (3)	C(2)—C(12)—C(17)	121·9 (3)
N—C(1)—C(18)	117·2 (3)	C(2)—C(12)—C(13)	120·9 (3)
N—C(1)—C(2)	125·1 (3)	C(13)—C(12)—C(17)	117·1 (4)
C(2)—C(1)—C(18)	117·7 (3)	C(12)—C(13)—C(14)	122·0 (3)
C(1)—C(2)—C(12)	116·8 (3)	C(13)—C(14)—C(15)	119·5 (4)
C(1)—C(2)—C(3)	120·4 (3)	C(14)—C(15)—C(16)	119·4 (4)
C(3)—C(2)—C(12)	122·9 (3)	C(15)—C(16)—C(17)	121·1 (3)
C(2)—C(3)—C(4)	126·0 (3)	C(12)—C(17)—C(16)	120·9 (4)
N—C(5)—C(10)	118·6 (3)	C(1)—C(18)—C(23)	121·5 (3)
N—C(5)—C(6)	122·8 (4)	C(1)—C(18)—C(19)	119·4 (4)
C(6)—C(5)—C(10)	118·3 (4)	C(19)—C(18)—C(23)	119·1 (3)
C(5)—C(6)—C(7)	119·9 (4)	C(18)—C(19)—C(20)	119·4 (4)
C(6)—C(7)—C(8)	121·8 (4)	C(19)—C(20)—C(21)	120·7 (4)
C(7)—C(8)—C(11)	121·2 (4)	C(20)—C(21)—C(22)	120·4 (5)
C(7)—C(8)—C(9)	117·5 (4)	C(21)—C(22)—C(23)	119·6 (4)
C(8)—C(9)—C(10)	121·3 (4)	C(18)—C(23)—C(22)	120·8 (4)
C(5)—C(10)—C(9)	121·2 (4)		

The two phenyl groups are synclinal with respect to the C(1)—C(2) bond, torsion angle C(18)—C(1)—C(2)—C(12), −71·4 (4)°.

In the double-bond system, the two double bonds are oriented synclinal about C(1)—C(2); the torsion angle N=C(1)—C(2)=C(3) is −69·3 (5)°. The deviations of the atoms from the best least-squares plane through N,C(1),C(2),C(3) are 0·095 (3), −0·261 (4), 0·229 (4) and −0·124 (4) Å, respectively.

The packing of the crystal is determined solely by van der Waals interactions.

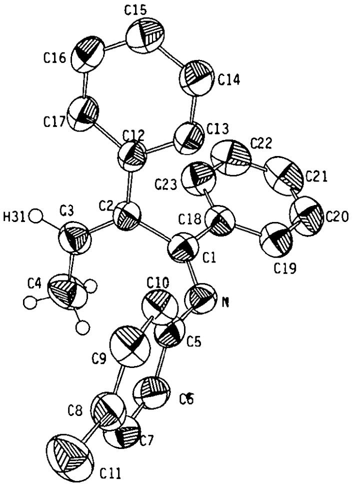

Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule showing the atom numbering.

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Structure of 2-Benzoyl-1,2-dihydro-1-isoquinolinecarbonitrile

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Abstract. $C_{17}H_{12}N_2O$, $M_r = 260{\cdot}30$, monoclinic, $P2_1/c$, $a = 7{\cdot}2105\ (9)$, $b = 12{\cdot}628\ (2)$, $c = 15{\cdot}953\ (2)$ Å, $\beta = 112{\cdot}26\ (1)°$, $V = 1344{\cdot}3\ (3)$ Å^3, $D_m = 1{\cdot}285$, $D_x = 1{\cdot}286$ Mg m^{-3}, $Z = 4$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}54178$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 0{\cdot}661$ mm^{-1}, $F(000) = 554$, room temperature, $R = 0{\cdot}040$ for 1579 observed reflexions. The

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