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Structures of 1,1-Diphenyl-2-aza-1,3-butadienes. III. 3-Cyano-4-(4-cyanophenyl)-1,1-diphenyl-2-aza-1,3-butadiene

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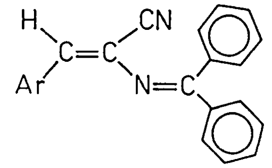
Abstract

The title structure, 4CNPD [3-(4-cyanophenyl)-2-(diphenylmethyleneamino)propenonitrile], consists of discrete *Z* diastereomeric molecules packed by van der Waals forces. The azabutadiene fragment, C1=N1—C2=C3, is twisted around the central single bond at 130.1 (7)°. The non-substituted phenyl rings are tilted with respect to the N1=C1(C1*a*)—C1*b* plane at 26.6 (2) and 55.8 (3)°. The dihedral angle between these rings is 73.7 (2)°. The cyano-substituted phenyl ring is almost coplanar with the N1—C2(C4)=C3—C1*c* plane. The phenyl rings are not plane-to-plane coupled. There are no intermolecular contacts between non-H atoms shorter than 3.3 Å.

Comment

2-Aza-1,3-dienes (I) have attracted considerable attention in recent years because of their application in the synthesis of heterocyclic compounds (Boger & Weinreb, 1987; Barluenda, Joglar, Gonzalez & Fustero, 1990; Barluenda, Aznar, Fustero & Tomas, 1990; Barluenda, Carlon, Pelaez, Joglar & Lopez Ortiz, 1992). Following our interest in the synthesis (Dryanska, 1990), chemistry (Dryanska, 1992) and structure (Angelova, Macíček & Dryanska, 1993; Macíček, Angelova & Dryanska, 1993) of functionalized 2-aza-1,3-butadienes, we present here the structural data for the title compound.



(I)

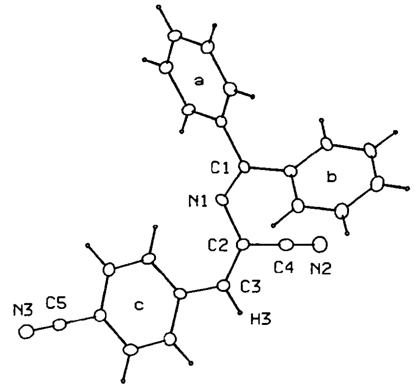


Fig. 1. *ORTEP* (Johnson, 1976) drawing of the 4CNPD molecule with the atom-numbering scheme and 30% probability thermal ellipsoids. H-atom spheres are arbitrarily reduced.

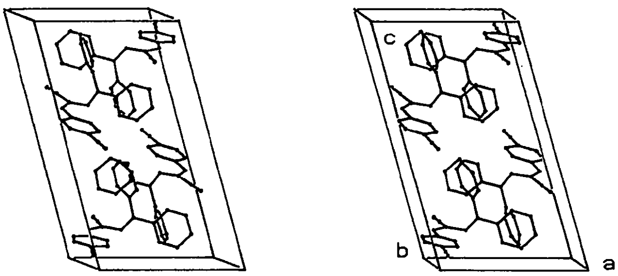


Fig. 2. Stereoscopic view of the molecular packing.

Experimental

Crystal data

$C_{23}H_{15}N_3$

$M_r = 333.39$

Mo $K\alpha$ radiation

$\lambda = 0.71073$ Å

Monoclinic
$P2_1/c$
$a = 10.591$ (1) Å
$b = 9.724$ (1) Å
$c = 18.324$ (3) Å
$\beta = 106.26$ (1)°
$V = 1811.8$ (7) Å^3
$Z = 4$
$D_x = 1.222$ Mg m^{-3}

Cell parameters from 22 reflections
$\theta = 17.98$-19.14°
$\mu = 0.06$ mm^{-1}
$T = 292$ K
Prismatic
0.23 × 0.23 × 0.20 mm
Yellow

Data collection

Enraf-Nonius CAD-4 diffractometer
Continuous-scan profiles
Absorption correction: none
3736 measured reflections
3541 independent reflections
958 observed reflections $[I > 2.5\sigma(I)]$

$R_{\rm int} = 0.018$
$\theta_{\rm max} = 26$°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 11$
$l = -22 \rightarrow 21$
3 standard reflections
frequency: 120 min
intensity variation: 1.3%

Refinement

Refinement on F
Final $R = 0.069$
$wR = 0.058$
$S = 1.190$
958 reflections
235 parameters
H-atom parameters refined as riding

$w = 1/[\sigma^2(F) + (0.026F)^2]$
$(\Delta/\sigma)_{\rm max} = 0.030$
$\Delta\rho_{\rm max} = 0.451$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.37$ e Å^{-3}
Atomic scattering factors from *SDP/PDP* (Enraf-Nonius, 1985)

Data collection: CAD-4 software (Enraf-Nonius, 1988). Data reduction: *SDP/PDP* (Enraf-Nonius, 1985). Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP/PDP*. Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *KAPPA* (Maciček, 1992).

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

$$U_{\rm eq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm eq}$
N1	0.2675 (4)	0.2385 (5)	0.1469 (3)	0.046 (2)
N2	0.0588 (5)	0.0279 (6)	0.2110 (3)	0.084 (2)
N3	0.1700 (5)	0.9913 (6)	0.0102 (3)	0.074 (2)
C1	0.3694 (6)	0.2021 (6)	0.2000 (3)	0.041 (2)
C2	0.1438 (5)	0.2489 (7)	0.1602 (3)	0.052 (2)
C3	0.0634 (6)	0.3572 (7)	0.1420 (3)	0.050 (2)
C4	0.0964 (6)	0.1259 (7)	0.1894 (4)	0.059 (2)
C5	0.1500 (6)	0.8847 (7)	0.0315 (4)	0.057 (2)
C1*a*	0.4923 (5)	0.1795 (6)	0.1781 (3)	0.039 (2)
C2*a*	0.4874 (5)	0.1463 (6)	0.1038 (3)	0.045 (2)
C3*a*	0.6014 (6)	0.1306 (7)	0.0823 (4)	0.056 (2)
C4*a*	0.7243 (6)	0.1494 (7)	0.1341 (3)	0.061 (2)
C5*a*	0.7295 (6)	0.1823 (7)	0.2085 (4)	0.065 (3)
C6*a*	0.6164 (6)	0.1976 (6)	0.2303 (4)	0.053 (2)
C1*b*	0.3731 (6)	0.1787 (6)	0.2806 (3)	0.043 (2)
C2*b*	0.4123 (6)	0.0557 (7)	0.3166 (3)	0.056 (2)
C3*b*	0.4061 (6)	0.0344 (7)	0.3905 (4)	0.066 (3)
C4*b*	0.3645 (6)	0.1370 (7)	0.4290 (4)	0.067 (3)
C5*b*	0.3283 (6)	0.2616 (8)	0.3951 (3)	0.061 (2)
C6*b*	0.3304 (6)	0.2840 (7)	0.3209 (3)	0.051 (2)
C1*c*	0.0899 (5)	0.4902 (7)	0.1117 (3)	0.048 (2)
C2*c*	−0.0038 (6)	0.5925 (7)	0.1049 (4)	0.059 (2)
C3*c*	0.0141 (6)	0.7211 (7)	0.0786 (4)	0.067 (2)
C4*c*	0.1249 (6)	0.7506 (7)	0.0571 (3)	0.051 (2)
C5*c*	0.2202 (6)	0.6508 (7)	0.0623 (3)	0.057 (2)
C6*c*	0.2035 (5)	0.5227 (7)	0.0898 (3)	0.053 (2)

Table 2. *Geometric parameters* (Å, °)

N1—C1	1.285 (7)	C1—C1*b*	1.482 (9)
N1—C2	1.402 (8)	C2—C3	1.336 (9)
N2—C4	1.145 (9)	C2—C4	1.46 (1)
N3—C5	1.148 (9)	C3—C1*c*	1.465 (9)
C1—C1*a*	1.483 (9)	C5—C4*c*	1.44 (1)
C1—N1—C2	121.0 (5)	N1—C2—C4	116.1 (5)
N1—C1—C1*a*	116.8 (5)	C3—C2—C4	118.6 (6)
N1—C1—C1*b*	125.4 (6)	C2—C3—C1*c*	127.9 (6)
C1*a*—C1—C1*b*	117.8 (5)	N2—C4—C2	178.6 (7)
N1—C2—C3	125.0 (6)	N3—C5—C4*c*	179.2 (7)
C2—N1—C1—C1*a*	173.5 (5)	N1—C1—C1*b*—C2*b*	122.8 (7)
C2—N1—C1—C1*b*	−5.3 (9)	N1—C2—C3—C1*c*	−6 (1)
C1—N1—C2—C3	130.1 (7)	C4—C2—C3—C1*c*	−179.8 (6)
C1—N1—C2—C4	−56.1 (8)	C2—C3—C1*c*—C2*c*	−173.1 (6)
N1—C1—C1*a*—C2*a*	−24.4 (8)		

3-Cyano-4-(4-cyanophenyl)-1,1-diphenyl-2-aza-1,3-butadiene was prepared according to a literature method (Dryanska, 1990) from *N*-diphenylmethyleneaminoacetonitrile and 4-cyanobenzaldehyde. M.p. 388-389 K (from ethanol); ^{1}H NMR (80 MHz, CDCl$_3$): δ 6.56 (*s*, 1H), 7.00-7.90 (*m*, 14H, aromatic H); elemental analysis, found (calc.): C 83.09 (82.86), H 4.60 (4.54), N 12.76 (12.60)%.

This work has been stimulated by the participation of JM and OA in a Grant-in-Aid Project between the International Centre for Diffraction Data and the Institute of Applied Mineralogy.

Lists of structure factors, anisotropic thermal parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71220 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: KA1037]

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Structure of a 2-Acylindan-1-one

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Abstract

2-(1-Hydroxyethylidene)indan-1-one exists as the enol form and is nearly planar with a maximum deviation from the least-squares plane of all non-H atoms of 0.045 (2) Å. The molecule has a *Z* configuration about the enol double bond. The hydroxyl H atom forms an intramolecular hydrogen bond to the carbonyl O atom, having an O···O distance of 2.610 (2) Å and an angle about the H atom of 150 (2)°. The C(=O)—C=C—O torsion angle about the enol double bond has a magnitude of 1.9 (2)°. The C=O and C=C distances are 1.257 (2) and 1.359 (2) Å, respectively.

Comment

The synthesis of some derivatives of atipamezol (Garcia & Enas, 1992), required in our laboratory, involved the preparation of (*Z*)-2-acetylindan-1-one as an intermediate. It was prepared by condensing phthalaldehyde with acetone in aqueous potassium hydroxide (Thiele & Falk, 1906). Bright yellow crystals were grown by slow evaporation from an ethanol–tetrahydrofuran mixture (1:2); m.p. 337–340 K. Studies performed by Forsén, Merényi & Nilsson (1964) suggested that 2-acylindan-1,3-diones exist exclusively in the enol form, which had been partially confirmed by the X-ray diffraction study of Csöregh & Norrestam (1976). A more precise study by Korp, Bernal & Lemke (1980) confirmed the enol form with an intramolecular hydrogen bond, H···O 1.56 (5) Å. This result is similar to that found for (*Z*)-2-acetylindan-1-one; H···O 1.69 (2) Å. The occurrence of the title compound in the enol form agrees with similar results for the parent compounds 2-pivaloylindan-1,3-dione and 2-acetylindan-1,3-dione (Korp, Bernal & Lemke, 1980). Structural data for 2-pivaloylindan-1,3-dione and 2-acetylindan-1,3-dione (Korp, Bernal & Lemke, 1980) are in agreement with those of the title compound. The configuration at the C9=C10 double bond has been determined to be *Z* according to the Cahn–Ingold–Prelog convention (Cahn, Ingold & Prelog, 1956).

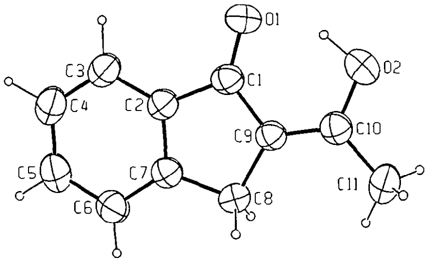

Fig. 1. *ORTEP* drawing (Johnson, 1965) of the molecule, representing heavy atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radii.

Experimental

Crystal data

$C_{11}H_{10}O_2$
$M_r = 174.2$
Monoclinic
$P2_1/n$
$a = 7.2980\ (3)$ Å
$b = 16.0475\ (8)$ Å
$c = 7.9701\ (3)$ Å
$\beta = 108.289\ (4)°$
$V = 886.3\ (2)$ Å^3
$Z = 4$
$D_x = 1.305$ Mg m^{-3}

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 10\text{–}12°$
$\mu = 0.08$ mm^{-1}
$T = 298$ K
Needle fragment
0.50 × 0.37 × 0.28 mm
Yellow

Data collection

Enraf–Nonius CAD-4 diffractometer
ω-2θ scans
Absorption correction: none
2667 measured reflections
2347 independent reflections
1490 observed reflections [$I > 3\sigma(I)$]

$R_{\rm int} = 0.014$
$\theta_{\rm max} = 29°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 21$
$l = -10 \rightarrow 10$
3 standard reflections
frequency: 167 min
intensity variation: <2%

Refinement

Refinement on F
Final $R = 0.039$
$wR = 0.047$
$S = 2.261$
1490 reflections
159 parameters

$\Delta\rho_{\rm max} = 0.17$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.11$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $g = 2.7\ (2) \times 10^{-6}$