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cis-1,1′-Dimethyl-3,3′-diphenyl-2,2′-biimidazolidinylidene

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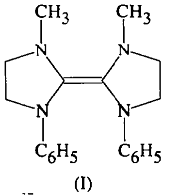
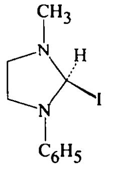
Abstract

The structure of the air-sensitive title compound consists of $C_{20}H_{24}N_4$ molecules connected through a hydrogen bond. Neither the molecule as a whole nor the individual five-membered rings have a planar geometry. The dihedral angle between the best planes of the two phenyl rings is 50.87 (14)°. Weak intermolecular hydrogen bonding is observed between a C atom and an imidazolidine N atom.

Comment

Olefins with four electron-donating substituents are powerful reducing agents (Hoffmann, 1968; Çetinkaya, King, Krishnamurthy, Lappert & Pedly, 1971; Hocker & Merten, 1972). Although structural studies have been reported on the symmetrical olefins (Hitchcock, 1979; Çetinkaya, Hitchcock, Jasim, Lappert & Spyropulous, 1992; Çetinkaya, Hitchcock, Küçükbay, Lappert & Al-Juaid, 1994), little is known about the unsymmetrical ones. In this paper, we describe the synthesis of the title compound, (I), obtained from 2-iodo-1-methyl-3-phenylimidazolidine, (II), and its crystal structure.


(I)


(II)

Least-squares planes calculations show that the imidazolidine rings are not planar, as can be seen from Fig. 1. To make sure that the molecule is really asymmetric about the double bond, three sets of data were collected from three different crystals in sealed capillaries with different scanning speeds. They all gave the molecular structure presented here. The imidazolidine rings have envelope conformations with the N1 and N2 atoms lying 0.416 (4) and −0.540 (5) Å from the C1, N3, C6, C7 and C2, N4, C3, C4 planes, respectively. The folding of the envelope cover for the two imidazolidine rings is in opposite directions, as indicated by the signs of the distances. The degree of the folding for the two envelope configurations is different, however. The dihedral angles between the respective planes of the envelope configurations are 29.2 (4) and 35.4 (2)°. The two ylidene groups are also in *trans* positions with respect to one another. The distances of the C8 and C5 atoms from the planes through C7, C1, N1 and C2, C4, N2 are −0.964 (5) and −1.217 (7) Å, respectively. Imidazolidine N atoms are in a distorted pyramidal position, with N1 0.384 (3) and N2 0.525 (3) Å from the C1, C7, C8 and C2, C4, C5 planes, respectively. Within the phenyl rings, C—C lengths vary from 1.364 (6) to 1.398 (5) Å. The average bond lengths around the phenyl rings show slightly longer C—C distances next to the N atom compared with the remaining bonds, as observed in similar compounds (Hitchcock, 1979). There is an intermolecular hydrogen bond between atoms C4 and N1(2 − *x*, 1 − *y*, 1 − *z*) of the imidazolidine groups, with a *D*—*A* distance of 3.481 (4) Å and a *D*—H···*A* angle of 131.1 (2)°. The closest intermolecular contacts are observed between atoms C6 and C17(2 − *x*, −*y*, −*z*) [3.623 (5) Å], and between C7 and C7(2 − *x*, 1 − *y*, −*z*) [3.730 (6) Å]. These intermolecular interactions may provide an explanation for the observed asymmetry.

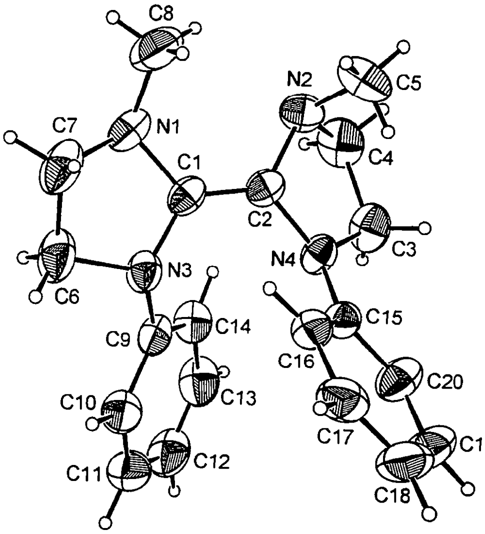


Fig. 1. *ORTEP* (Johnson, 1965) drawing of (I) with the atomic numbering scheme. The displacement ellipsoids are drawn at the 50% probability level. H atoms are shown as small circles of arbitrary radii.

Experimental

The starting material, 2-iodo-1-methyl-3-phenyl-2-imidazolidine, (II), was synthesized according to the literature procedure of Çetinkaya *et al.* (1992) and converted into (I) by reaction with NaH.I in tetrahydrofuran. Colourless crystals suitable for X-ray studies were obtained by cooling a solution of (I) in toluene/hexane and were sealed into a capillary tube for protection.

Crystal data

$C_{20}H_{24}N_4$
$M_r = 320.441$
Triclinic
$P\bar{1}$
$a = 8.4511\,(13)$ Å
$b = 9.8471\,(16)$ Å
$c = 12.2407\,(18)$ Å
$\alpha = 111.352\,(12)^\circ$
$\beta = 91.970\,(14)^\circ$
$\gamma = 108.781\,(11)^\circ$
$V = 884.9\,(6)$ Å^3
$Z = 2$
$D_x = 1.202$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 16.48–18.07^\circ$
$\mu = 0.068$ mm^{-1}
$T = 295$ K
Irregular
$0.50 \times 0.30 \times 0.25$ mm
Colourless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: none
3481 measured reflections
3147 independent reflections
2020 reflections with $I > 3\sigma(I)$

$R_{\text{int}} = 0.009$
$\theta_{\max} = 26.32^\circ$
$h = -10 \rightarrow 10$
$k = -11 \rightarrow 11$
$l = -14 \rightarrow 0$
3 standard reflections frequency: 120 min intensity decay: none

Refinement

Refinement on F
$R = 0.046$
$wR = 0.057$
$S = 1.49$
2020 reflections
217 parameters
H atoms: see below
Weighting scheme: see below

$(\Delta/\sigma)_{\max} = 0.0001$
$\Delta\rho_{\max} = 0.27$ e Å^{-3}
$\Delta\rho_{\min} = -0.35$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

N1—C1	1.415 (4)	N3—C9	1.420 (4)
N1—C7	1.455 (5)	N4—C2	1.430 (4)
N1—C8	1.452 (5)	N4—C3	1.459 (4)
N2—C2	1.440 (4)	N4—C15	1.388 (4)
N2—C4	1.475 (4)	C1—C2	1.341 (4)
N2—C5	1.484 (4)	C3—C4	1.533 (5)
N3—C1	1.412 (4)	C6—C7	1.497 (5)
N3—C6	1.472 (4)		
C1—N1—C7	105.5 (3)	C2—N4—C15	125.7 (3)
C1—N1—C8	120.2 (3)	C3—N4—C15	121.3 (3)
C7—N1—C8	113.6 (3)	N1—C1—N3	107.6 (3)
C2—N2—C4	100.4 (3)	N1—C1—C2	127.1 (3)
C2—N2—C5	111.4 (3)	N3—C1—C2	125.0 (3)
C4—N2—C5	111.6 (3)	N2—C2—N4	108.5 (3)
C1—N3—C6	109.0 (3)	N2—C2—C1	123.7 (3)
C1—N3—C9	123.3 (3)	N4—C2—C1	125.1 (3)
C6—N3—C9	115.9 (3)	N4—C3—C4	102.5 (3)
C2—N4—C3	108.1 (3)	N4—C15—C16	121.8 (3)

The weighting scheme used was $w = (\text{threshold}/F^2)$ if $F \geq$ threshold or $w = 0$ if $F^2 <$ cutoff.σF^2 (threshold = 93.03 and cutoff = 4). All non-H atoms were refined with anisotropic displacement parameters. H atoms were placed geometrically 0.95 Å from their parent atoms. After refining the methyl H atoms for a few cycles, a riding model was used for all H atoms, with $B_{\text{iso}}(\text{H}) = 1.3B_{\text{eq}}(\text{C})$.

Data collection: *CAD-4 Express* (Enraf–Nonius, 1993). Data reduction: *MolEN* (Fair, 1990). Program(s) used to solve structure: *SIR* (Burla *et al.*, 1989) in *MolEN*. Program(s) used to refine structure: *LSFM* in *MolEN*. Molecular graphics: *ORTEP* (Johnson, 1965) in *MolEN*.

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Lists of structure factors, anisotropic displacement parameters, atomic coordinates and complete geometry have been deposited with the IUCr (Reference: BM1050). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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9,10-Bis{2-[4-(*N*,*N*-diphenylamino)phenyl]ethynyl}anthracene

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Abstract

The crystal structure of the title compound, $C_{54}H_{36}N_2$, shows that the anthracene rings in the unit cell do not stack in a face-to-face manner. Instead, the anthracene