Table 3. *Ring-puckering and asymmetry parameters*

Ring	Q (Å)	φ (°)	θ (°)	C_s (°)	C_2 (°)	
B	0.512 (8)	172 (1)	54.0 (9)	8.6 (C5)	19.4 (C5—C10)	E_4
B*	0.552	166	52.3	14.3 (C5)	13.7 (C5—C10)	$E_4 \leftrightarrow {}^3H_4$
C	0.572 (8)	238 (6)	7.0 (8)	6.9 (C8)	4.7 (C8—C9)	${}^1C_4$
				0.4 (C9)	5.4 (C9—C11)	${}^1C_4$
				7.2 (C11)	10.0 (C11—C12)	${}^1C_4$
C*	0.568	249	10.5	10.6 (C8)	8.8 (C8—C9)	${}^1C_4$
				2.2 (C9)	5.0 (C9—C11)	${}^1C_4$
				8.5 (C11)	13.4 (C11—C12)	${}^1C_4$
D	0.503 (8)	194 (1)		15.2 (C13)	5.8 (C16)	${}^2H_1 \rightarrow E_1$
D*	0.483	193		14.2 (C13)	6.0 (C16)	${}^2H_1 \rightarrow E_1$

* Values resulting from molecular-mechanics calculations.

H atoms were generated and refined as riding groups (overall isotropic displacement parameters were refined for different CH types), except those attached to the O atoms, whose positions were found in the ΔF map and refined isotropically.

Program used to solve the structure: *SHELXS*86 (Sheldrick, 1985). Program used to refine the structure: *SHELX*76 (Sheldrick, 1976). Software to prepare material for publication: *CSU* (Vicković, 1988). Software for performing molecular-mechanics calculations: *PCMODEL* (Serena Software, 1989).

Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the IUCr (Reference: KA1093). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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5-Imino-1,4,4-triphenylimidazolidin-2-one: an Imidazolidine with an Exocyclic Imino Group

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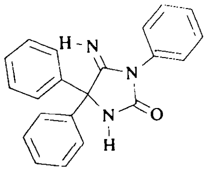
Abstract

The conformation of the imidazolidine ring in the title compound, $C_{21}H_{17}N_3O$, is almost flat with a maximum deviation of ±0.031 (8) Å from the least-squares plane through the ring. The structure is stabilized by an intermolecular hydrogen bond. This type of compound can be used in addition reactions with isocyanates.

Comment

In the past, 5-iminoimidazolidin-2-ones were only reported as intermediates of hydantoins, without further investigation of their structure. In order to characterize one such reaction product unambiguously, an X-ray structure determination of the title compound, (I), has been undertaken.



(I)

Heterocycles with an imino group next to the ring N atom can undergo Dimroth rearrangement (Dimroth, 1909). During this rearrangement the group bound to the ring N atom moves to the exocyclic N atom (Brown & Ienega, 1974). The resulting product no longer has a free imino function. Furthermore, the only published NMR shift for the imino group of a 4,4-disubstituted 5-imidazolidin-2-one differs by about 1.4 p.p.m. from

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our results (Menéndez & Söllhuber, 1991). Therefore, independent proof of the imino function was required.

The exocyclic imino group shows a rather short C5═N51 double bond of 1.267 (2) Å. Furthermore, the C5═N51—H51 angle is reduced to 110 (2)°, probably as a result of the lone pair at N51.

The structure is stabilized by an intermolecular hydrogen bond, N3—H3···N51. Details of this hydrogen bond are given in Table 2. The atom H51, bound to atom N51, is not involved in any hydrogen bonding, although N51 could act as a donor atom for hydrogen bonding.

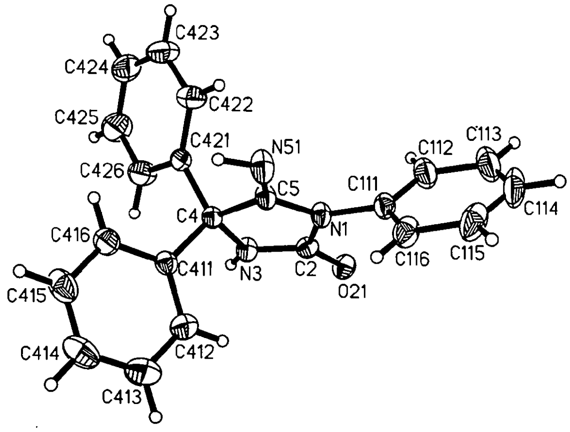

Fig. 1. *ORTEP*II (Johnson, 1976) drawing, as implemented in *SHELXTL-Plus* (Sheldrick, 1992), of the title compound with displacement ellipsoids at 30% probability and atomic numbering scheme.

Experimental

The title compound was synthesized by cyclization of 1-[1-cyano-1,1-(diphenyl)methyl]-3-phenylurea in hot absolute ethanol in the presence of a catalytic amount of potassium *tert*-butylate. Suitable crystals melting at 487–488 K were obtained by recrystallization from ethanol.

Crystal data

$C_{21}H_{17}N_3O$
$M_r = 327.38$
Orthorhombic
$Pca2_1$
$a = 12.759\,(1)$ Å
$b = 10.027\,(1)$ Å
$c = 14.000\,(1)$ Å
$V = 1791.1\,(3)$ Å^3
$Z = 4$
$D_x = 1.214$ Mg m^{-3}

Cu $K\alpha$ radiation
$\lambda = 1.54178$ Å
Cell parameters from 24 reflections
$\theta = 43.5–48.5°$
$\mu = 0.608$ mm^{-1}
$T = 293\,(1)$ K
Transparent block
0.5 × 0.5 × 0.4 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$2\theta/\omega$ scans
Absorption correction: none

$R_{\rm int} = 0.0174$
$\theta_{\rm max} = 76.5°$
$h = 0 \rightarrow 16$
$k = 0 \rightarrow 12$
$l = -17 \rightarrow 0$

1950 measured reflections
1923 independent reflections
1913 observed reflections [$I > 2\sigma(I)$]

3 standard reflections
frequency: 120 min
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.0328$
$wR(F^2) = 0.0919$
$S = 1.031$
1923 reflections
250 parameters
All H-atom parameters refined for H3 and H51; riding model for all other H atoms
$w = 1/[\sigma^2(F_o^2) + (0.0624P)^2 + 0.1610P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} = -0.177$

$\Delta\rho_{\rm max} = 0.207$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.157$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0342 (14)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)
Absolute configuration: Flack (1983), $\chi = 0.1\,(3)$

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

$U_{\rm iso}$ for H atoms; $U_{\rm eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$ for others.

	x	y	z	$U_{\rm iso}/U_{\rm eq}$
N1	0.1306 (1)	0.6395 (1)	0.2438 (1)	0.0409 (3)
C111	0.1952 (1)	0.7554 (1)	0.2387 (2)	0.0460 (4)
C112	0.1900 (2)	0.8491 (2)	0.3105 (2)	0.0623 (6)
C113	0.2532 (2)	0.9620 (2)	0.3040 (3)	0.0848 (9)
C114	0.3191 (2)	0.9800 (2)	0.2287 (3)	0.0914 (12)
C115	0.3239 (2)	0.8865 (3)	0.1584 (3)	0.0827 (9)
C116	0.2610 (2)	0.7733 (2)	0.1615 (2)	0.0589 (5)
C2	0.0200 (1)	0.6453 (1)	0.2452 (1)	0.0365 (3)
O21	−0.0308 (1)	0.7472 (1)	0.2476 (1)	0.0468 (3)
N3	−0.0145 (1)	0.5194 (1)	0.2443 (1)	0.0407 (3)
H3	−0.079 (2)	0.501 (2)	0.248 (2)	0.049 (5)
C4	0.0685 (1)	0.4201 (1)	0.2466 (1)	0.0351 (3)
C411	0.0659 (1)	0.3296 (2)	0.1587 (1)	0.0387 (4)
C412	0.0346 (2)	0.3818 (2)	0.0720 (2)	0.0680 (6)
C413	0.0379 (3)	0.3044 (3)	−0.0097 (2)	0.0903 (9)
C414	0.0725 (2)	0.1763 (3)	−0.0061 (2)	0.0780 (8)
C415	0.1034 (2)	0.1230 (2)	0.0791 (2)	0.0697 (6)
C416	0.1008 (2)	0.2000 (2)	0.1618 (2)	0.0522 (4)
C421	0.0634 (1)	0.3444 (2)	0.3418 (1)	0.0411 (4)
C422	0.1415 (2)	0.3448 (2)	0.4098 (2)	0.0652 (6)
C423	0.1274 (3)	0.2768 (3)	0.4960 (2)	0.0838 (8)
C424	0.0368 (3)	0.2105 (3)	0.5151 (2)	0.0799 (8)
C425	−0.0417 (3)	0.2112 (3)	0.4489 (2)	0.0820 (8)
C426	−0.0287 (2)	0.2759 (2)	0.3625 (2)	0.0642 (6)
C5	0.1656 (1)	0.5097 (1)	0.2398 (2)	0.0388 (3)
N51	0.2607 (1)	0.4755 (1)	0.2320 (2)	0.0555 (5)
H51	0.266 (2)	0.384 (2)	0.229 (2)	0.054 (6)

Table 2. *Selected geometric parameters* (Å, °)

N1—C5	1.376 (2)	N3—H3	0.85 (2)
N1—C2	1.412 (2)	C4—C411	1.528 (2)
N1—C111	1.427 (2)	C4—C5	1.533 (2)
C2—O21	1.211 (2)	C4—C421	1.536 (2)
C2—N3	1.337 (2)	C5—N51	1.267 (2)
N3—C4	1.454 (2)	N51—H51	0.92 (2)
C5—N1—C2	111.3 (1)	C411—C4—C5	108.4 (1)
C5—N1—C111	125.5 (1)	N3—C4—C421	109.1 (1)
C2—N1—C111	123.0 (1)	C411—C4—C421	113.9 (1)
C112—C111—N1	119.5 (2)	C5—C4—C421	112.2 (1)
C116—C111—N1	119.8 (2)	C416—C411—C4	121.9 (2)

O21—C2—N3	128.4 (1)	C412—C411—C4	119.3 (2)
O21—C2—N1	124.8 (1)	C422—C421—C4	124.6 (2)
N3—C2—N1	106.9 (1)	C426—C421—C4	117.3 (2)
C2—N3—C4	114.0 (1)	N51—C5—N1	124.7 (1)
C2—N3—H3	122.0 (13)	N51—C5—C4	128.4 (1)
C4—N3—H3	123.7 (13)	N1—C5—C4	106.9 (1)
N3—C4—C411	111.9 (1)	C5—N51—H51	110.3 (15)
N3—C4—C5	100.7 (1)		

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3···N51^{i}	0.84 (2)	2.07 (2)	2.873 (2)	158 (2)

Symmetry code: (i) $x - \frac{1}{2}, 1 - y, z$.

From the systematic absences ($0kl$: $k = 2n$ and $h0l$: $l = 2n$) the possible space groups *Pbcm* (No. 57) and *Pca*2$_1$ (No. 29) were deduced. With Z = 4, space group *Pbcm* is only possible if the molecule lies on the mirror plane. For steric reasons, this is very unlikely. Together with $|E^*E-1| = 0.825$, the structure could be solved easily in *Pca*2$_1$ (No. 29), while several computer runs in *Pbcm* did not produce any reasonable solutions (Sheldrick, 1990).

The positions of H3 and H51 were taken from a difference Fourier map and refined without any restrictions; all other H atoms were refined with distance restraints for the C—H distances (Sheldrick, 1993). All distances and angles are generally as expected. Some Friedel opposites have been collected. These Friedel pairs are regarded as symmetry independent and have not been merged.

Since the title compound crystallizes in a non-centrosymmetric space group, polar axis restraints were applied by the method of Flack & Schwarzenbach (1988).

Data collection: Enraf–Nonius CAD-4 diffractometer software. Cell refinement: Enraf–Nonius CAD-4 diffractometer software. Data reduction: *CADSHEL* (Kopf, 1987). Program(s) used to solve structure: *SHELXS*90 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976) in *SHELXTL-Plus* (Sheldrick, 1992); *PLATON*92 (Spek, 1992). Software used to prepare material for publication: *CIF2TEX* (Kopf, 1992*a*); *FCF2FOC* (Kopf, 1992*b*).

This work was supported by the Fond der Chemischen Industrie.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: AB1252). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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A Derivative of 1,3,4-Oxadiazole

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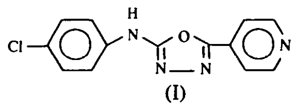
Abstract

The molecules of title compound, 2-(4-chloroanilino)-5-(4-pyridyl)-1,3,4-oxadiazole, $C_{13}H_9ClN_4O$, are nearly planar with a maximum deviation of 0.094 (2) Å from the plane. Intermolecular N(1)—H···N(4′) hydrogen bonds link the molecules into chains [N(1)···N(4′) = 2.902 (3) Å, N(1)—H···N(4′) = 178 (2)°].

Comment

It has been reported that compounds containing the heterocyclic 1,3,4-oxadiazole ring may have biological activity (Vansdadia, Vikani & Hansa Rarekh, 1988). In order to determine the structure of such a ring system we have synthesized the title compound, (I), following the method of Zhang, Yang & Zeng (1988).



(I)

The molecule is composed of three coplanar rings. The torsion angles C(8)—O—C(7)—N(1), C(2)—C(3)—