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Acta Cryst. (1993). C**49**, 1536–1537

Structure of 3-(3,5-Dimethylpiperidino)-*N*-(*p*-chlorophenyl)succinimide

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(*Received* 6 *July* 1992; *accepted* 17 *February* 1993)

Abstract

In the title molecule, 3-(3,5-dimethylpiperidino)-1-(4-chlorophenyl)-2,5-pyrrolidinedione (1), the *N*-(*p*-chlorophenyl) substituent is rotated by 68.8° relative to the succinimide plane. The piperidinyl ring has a chair conformation with all substituents in equatorial positions; the conformation around the piperidino–succinimide C—N bond is staggered.

Comment

The title compound was prepared in the framework of our structural and conformational studies (Joseph-Nathan, Mendoza & Garcia G., 1972, 1974; Soriano-García, Toscano, Mendoza, García G., Guzmán, Alemán & Huipe N., 1990; Mendoza, Garcia G., Guzmán, Gutierrez & Chavez, 1991) of some 3-(1-cycloalkylamine)succinimides derived by condensation reactions from maleimides or isomaleimides and cycloalkylamines.

The chemical structure of (1) as well as its molecular stereochemistry are unequivocally established by the present X-ray investigation. The central succinimide moiety is planar within 0.004 Å. Its N atom [N(2)] has a planar-trigonal bond configuration with the *N*-(*p*-chlorophenyl) substituent, rotated by 68.8° around the C(10)—N(2) bond, out of the succinimide plane. The dimethylpiperidine substituent has a chair conformation with both methyl groups and the succinimide moiety in equatorial positions. The conformation around the N(1)—C(8) bond is staggered; all relevant torsion angles are close to 60 and 180°. The piperidine N(1) atom has a trigonal-pyramidal bond configuration; its displacement from the plane of three neighbouring atoms is 0.422 (4) Å.

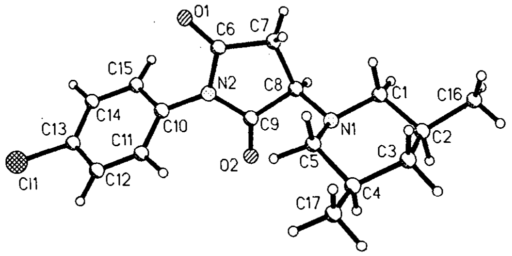

Fig. 1. General view of the title molecule (1).

Experimental

Crystal data

$C_{17}H_{21}ClN_2O_2$
M_r = 320.8
Monoclinic
$P2_1/c$
a = 8.804 (3) Å
b = 6.472 (2) Å
c = 29.075 (12) Å
β = 94.75 (2)°
V = 1651.0 (3) Å^3
Z = 4
D_x = 1.291 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.71073 Å

Cell parameters from 24 reflections
θ = 12–13°
μ = 0.240 mm^{-1}
T = 153 K
Needles
0.4 × 0.2 × 0.1 mm
Colourless
Crystal source: from hexane–ethyl acetate solution (2/3)

Data collection

Siemens *P*3/*PC* diffractometer
$\theta/2\theta$ scans
Absorption correction: none
6095 measured reflections
5773 independent reflections
1176 observed reflections [$F > 6.0\sigma(F)$]

$R_{\rm int}$ = 0.049
$\theta_{\rm max}$ = 30°
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 9$
$l = -42 \rightarrow 43$
2 standard reflections monitored every 98 reflections
intensity variation: ±1.8%

Refinement

Refinement on F
Final R = 0.052
wR = 0.064
S = 2.16
1176 reflections
262 parameters
H atoms refined isotropically with fixed $U_{\rm iso}$ = 0.05 Å^2
$w = 1/\sigma^2(F)$

$(\Delta/\sigma)_{\rm max}$ = 0.333
$\Delta\rho_{\rm max}$ = 0.40 e Å^{-3}
$\Delta\rho_{\rm min}$ = −0.29 e Å^{-3}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV)

Acta Crystallographica Section C
ISSN 0108-2701 ©1993

Table 1. *Fractional atomic coordinates and equivalent isotropic thermal parameters* (Å²)

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

	x	y	z	U_{eq}
Cl(1)	0.2122 (3)	0.2497 (4)	0.0577 (1)	0.043 (1)
O(1)	0.3922 (6)	−0.4067 (9)	0.2281 (2)	0.031 (2)
O(2)	0.0428 (6)	0.0606 (10)	0.2712 (2)	0.037 (2)
N(1)	0.1881 (7)	−0.1111 (10)	0.3580 (2)	0.024 (2)
N(2)	0.2200 (6)	−0.1508 (10)	0.2406 (2)	0.021 (2)
C(1)	0.1974 (9)	−0.2382 (15)	0.3999 (3)	0.025 (2)
C(2)	0.2038 (10)	−0.1125 (14)	0.4431 (3)	0.033 (3)
C(3)	0.3433 (10)	0.0288 (14)	0.4441 (3)	0.033 (3)
C(4)	0.3469 (9)	0.1567 (14)	0.4001 (3)	0.031 (3)
C(5)	0.3275 (9)	0.0162 (14)	0.3577 (3)	0.026 (3)
C(6)	0.3071 (8)	−0.3249 (12)	0.2535 (3)	0.024 (3)
C(7)	0.2746 (10)	−0.3854 (13)	0.3010 (3)	0.029 (3)
C(8)	0.1555 (9)	−0.2278 (15)	0.3166 (3)	0.027 (3)
C(9)	0.1281 (8)	−0.0859 (14)	0.2747 (3)	0.027 (3)
C(10)	0.2162 (9)	−0.0529 (13)	0.1967 (3)	0.024 (3)
C(11)	0.2756 (9)	0.1433 (12)	0.1920 (3)	0.025 (3)
C(12)	0.2724 (8)	0.2387 (15)	0.1511 (3)	0.027 (3)
C(13)	0.2113 (9)	0.1334 (14)	0.1123 (3)	0.031 (3)
C(14)	0.1502 (9)	−0.0647 (13)	0.1148 (3)	0.025 (3)
C(15)	0.1520 (8)	−0.1590 (14)	0.1576 (3)	0.026 (3)
C(16)	0.2134 (12)	−0.2593 (20)	0.4854 (3)	0.046 (3)
C(17)	0.4914 (9)	0.2846 (15)	0.3999 (3)	0.034 (3)

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

Cl(1)—C(13)	1.758 (9)	C(3)—C(4)	1.53 (1)
O(1)—C(6)	1.22 (1)	C(4)—C(5)	1.53 (1)
O(2)—C(9)	1.21 (1)	C(4)—C(17)	1.52 (1)
N(1)—C(1)	1.47 (1)	C(6)—C(7)	1.49 (1)
N(1)—C(5)	1.48 (1)	C(7)—C(8)	1.56 (1)
N(1)—C(8)	1.43 (1)	C(8)—C(9)	1.53 (1)
N(2)—C(6)	1.40 (1)	C(10)—C(15)	1.41 (1)
N(2)—C(9)	1.40 (1)	C(11)—C(12)	1.34 (1)
N(2)—C(10)	1.42 (1)	C(12)—C(13)	1.39 (1)
C(1)—C(2)	1.50 (1)	C(13)—C(14)	1.39 (1)
C(2)—C(3)	1.53 (1)	C(14)—C(15)	1.38 (1)
C(2)—C(16)	1.55 (1)		

C(1)—N(1)—C(5)	109.1 (6)	C(6)—C(7)—C(8)	106.5 (7)
C(1)—N(1)—C(8)	113.3 (7)	N(1)—C(8)—C(7)	119.9 (6)
C(5)—N(1)—C(8)	113.5 (6)	N(1)—C(8)—C(9)	111.1 (7)
C(6)—N(2)—C(9)	112.9 (6)	C(7)—C(8)—C(9)	103.2 (6)
C(6)—N(2)—C(10)	124.9 (7)	O(2)—C(9)—N(2)	124.6 (7)
C(9)—N(2)—C(10)	122.1 (6)	O(2)—C(9)—C(8)	126.6 (7)
N(1)—C(1)—C(2)	112.9 (7)	N(2)—C(9)—C(8)	108.8 (7)
C(1)—C(2)—C(3)	108.4 (7)	N(2)—C(10)—C(11)	121.0 (7)
C(1)—C(2)—C(16)	109.3 (8)	N(2)—C(10)—C(15)	119.1 (7)
C(3)—C(2)—C(16)	111.1 (7)	C(11)—C(10)—C(15)	119.9 (7)
C(2)—C(3)—C(4)	112.3 (7)	C(10)—C(11)—C(12)	121.9 (8)
C(3)—C(4)—C(5)	110.1 (7)	C(11)—C(12)—C(13)	118.3 (9)
C(3)—C(4)—C(17)	112.0 (7)	Cl(1)—C(13)—C(12)	119.6 (7)
C(5)—C(4)—C(17)	111.0 (7)	Cl(1)—C(13)—C(14)	118.2 (6)
N(1)—C(5)—C(4)	111.4 (7)	C(12)—C(13)—C(14)	122.2 (8)
O(1)—C(6)—N(2)	122.8 (7)	C(13)—C(14)—C(15)	118.5 (8)
O(1)—C(6)—C(7)	128.7 (7)	C(10)—C(15)—C(14)	119.0 (8)
N(2)—C(6)—C(7)	108.5 (7)		

All calculations were performed with *SHELXTL-PC* (Sheldrick, 1990) programs on an IBM PC/AT computer

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Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71116 (16 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: VS1003]

Acta Cryst. (1993). C**49**, 1537–1540

Structure of the Partial Cone Conformer of 25,26,27,28-Tetrakis[(2-pyridylmethyl)-oxy]calix[4]arene

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(*Received* 5 *October* 1992; *accepted* 25 *January* 1993)

Abstract

The partial cone conformer of tetrakis[(2-pyridylmethyl)-oxy]pentacyclo[19.3.1.1^{3,7}.1^{9,13}.1^{15,19}]octacosa-1(25),-3,5,7(28),9,11,13(27),15,17,19(26),21,23-dodecaene, (I), adopts a conformation in which the pendant OCH_2py group of the rotated aryl ring is oriented away from the calixarene cavity produced by the other three aryl rings, with its N atom *exo* to the calixarene cup. The orientation of the four aromatic rings is such that two rings are almost parallel to each other and the other two are at an angle of 42°. This conformation precludes any solvent molecule being enclathrated within the small molecular cavity.

Comment

We have recently shown that the conformer distribution in the exhaustive *o*-alkylation of calix[4]arenes with 2-(chloromethyl)pyridine hydrochloride is strongly affected by the base used: NaH induces only cone conformers, while K_2CO_3 or Cs_2CO_3 lead preferentially to partial cone and/or 1,3 alternate conformers (Pappalardo, Giunta, Foti, Ferguson, Gallagher & Kaitner, 1992). The title

Acta Crystallographica Section C
ISSN 0108-2701 ©1993