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

O5—C5	1.236 (3)	C1—C9a	1.506 (4)
O8—C8	1.219 (3)	C2—C3	1.497 (4)
O9a—C9a	1.410 (3)	C4a—C5	1.493 (3)
O9a—C9b	1.435 (4)	C4a—C8a	1.367 (3)
N1—C1a	1.488 (4)	C5—C6	1.445 (3)
N1—C1	1.473 (3)	C6—C6a	1.504 (4)
N1—C2	1.470 (3)	C6—C7	1.372 (3)
N4—C3	1.492 (3)	C7—C8	1.519 (3)
N4—C4a	1.360 (3)	C8a—C8	1.419 (3)
N4—C9a	1.485 (3)	C8a—C9	1.458 (3)
N7—C7	1.357 (3)	C9a—C9	1.530 (3)
C1—C2	1.486 (4)	C9—C10	1.335 (4)
C9a—O9a—C9b	114.5 (2)	C5—C6—C7	119.5 (2)
C1a—N1—C1	113.0 (2)	C6a—C6—C7	122.7 (2)
C1a—N1—C2	113.5 (2)	N7—C7—C6	124.5 (2)
C1—N1—C2	60.7 (2)	N7—C7—C8	112.3 (2)
C3—N4—C4a	125.7 (2)	C6—C7—C8	123.2 (2)
C3—N4—C9a	111.0 (2)	C4a—C8a—C8	120.5 (2)
C4a—N4—C9a	108.6 (2)	C4a—C8a—C9	108.7 (2)
N1—C1—C2	59.5 (2)	C8—C8a—C9	130.6 (2)
N1—C1—C9a	113.7 (2)	O8—C8—C7	118.9 (2)
C2—C1—C9a	107.9 (2)	O8—C8—C8a	124.8 (2)
N1—C2—C1	59.8 (2)	C7—C8—C8a	116.2 (2)
N1—C2—C3	111.3 (2)	O9a—C9a—N4	112.6 (2)
C1—C2—C3	109.7 (2)	O9a—C9a—C1	103.7 (2)
N4—C3—C2	102.5 (2)	O9a—C9a—C9	115.3 (2)
N4—C4a—C5	123.4 (2)	N4—C9a—C1	102.8 (2)
N4—C4a—C8a	112.9 (2)	N4—C9a—C9	103.7 (2)
C5—C4a—C8a	123.7 (2)	C1—C9a—C9	118.3 (2)
O5—C5—C4a	120.6 (2)	C8a—C9—C9a	105.5 (2)
O5—C5—C6	123.0 (2)	C8a—C9—C10	129.6 (3)
C4a—C5—C6	116.4 (2)	C9a—C9—C10	124.8 (2)
C5—C6—C6a	117.8 (2)		
C9b—O9a—C9a—C9	−57.7 (3)	C1a—N1—C2—C3	154.8 (2)
O9a—C9a—C9—C10	−56.0 (4)		

All H atoms were found from difference Fourier maps. All non-H atoms were refined anisotropically, with all H atoms isotropic.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *CAD-4 Software*. Program(s) used to solve structure: *MULTAN*11/82 (Main *et al.*, 1982). Program(s) used to refine structure: *SDP-Plus* (Frenz, 1985). Molecular graphics: *ORTEP*II (Johnson, 1976).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates, complete geometry and torsion angles have been deposited with the IUCr (Reference: TA1079). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3-(*m*-Bromophenyl)-1-(3-methyl-2-pyridyl)pyrrolidin-2,5-dione

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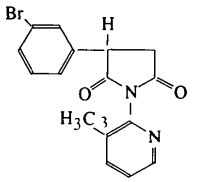
Abstract

The title compound, $C_{16}H_{13}BrN_2O_2$, is one of the 27 phenylsuccinimides for which the correlation between structure and anticonvulsant activity was examined in an earlier study. All of the possible minimum-energy conformations for all of the compounds were found using molecular modelling. For eight of the 27 derivatives the X-ray structures were determined and these were used as a guide to find the correct conformations among the minimum-energy conformations derived from calculations. The results of the present study show that the title compound also has a conformation similar to that calculated previously which confirms that the correct conformation was selected to calculate the structure–activity correlations.

Comment

In our previous paper (Kwiatkowski & Karolak-Wojciechowska, 1993), we studied the correlation between structure and anticonvulsant activity, based on the maximal electrical shock (MES) test, of phenylsuccinimides. The correlation was based on 27 derivatives of which 12 were active, including the title compound (Zejc, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wilczyńska, 1987). Active derivatives could be distinguished from inactive ones based on the sign of the difference between the minima of the molecular electrostatic potential (MEP) at the carbonyl O atoms of the five-membered imide ring. Since this correlation is based on the structural and electronic parameters derived from the geometrical description of the molecule, the three-dimensional conformations of all the investigated compounds had to be determined. We solved the structures of eight derivatives (Kwiatkowski & Karolak-Wojciechowska, 1990, 1991, 1992*a*, 1992*b*; Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990) which served as a guide to finding the correct minimum energy conformations from among all the minimum-energy conformations derived

by molecular modelling (Burkert & Allinger, 1982). The geometry of the correct conformations were further optimized using semi-empirical calculations (*MOPAC* 6.0; QCPE, 1990). Then, the MEP distributions were calculated using an MNDO approximation. The results of molecular modelling showed that for compounds with pyridyl substituents there were two equi-energetic conformers differing only in the rotation of the pyridyl group. Moreover, the MEP distribution was found to depend strongly on the N-atom position at the pyridyl substituent. The structure of (I) (Fig. 1) was solved to confirm the selection of the correct minimum-energy conformation from molecular modelling. The X-ray results fully support the previous selection (Fig. 2): the torsion angle C3—N1—C5—N2 has a value of −74.0° in the optimized conformation, close to the value of −91.2 (2)° found from the structure determination of (I).



(I)

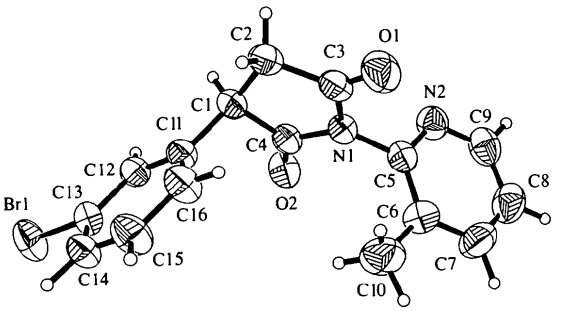


Fig. 1 Molecular structure of (I) showing 50% probability displacement ellipsoids.

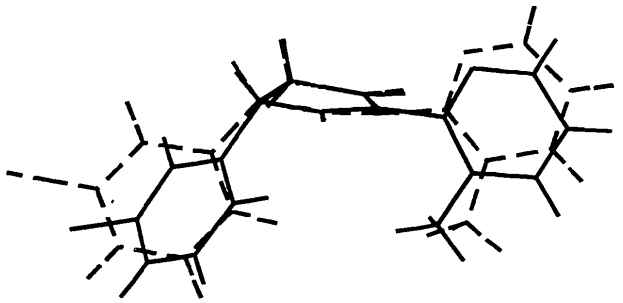


Fig. 2 Superposition of crystallographic (solid lines) and modelled (dashed lines) conformations of (I).

Experimental

The synthesis of compound, (I), is described in the paper by Zejc, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wilczyńska (1987). Crystals suitable for structure analysis were grown from ethanol solution.

Crystal data

$C_{16}H_{13}BrN_2O_2$
M_r = 345.19
Triclinic
$P\bar{1}$
a = 8.6110 (10) Å
b = 9.8480 (10) Å
c = 9.9480 (10) Å
α = 68.960 (15)°
β = 68.300 (15)°
γ = 89.180 (15)°
V = 724.71 (13) Å^3
Z = 2
D_x = 1.582 Mg m^{-3}
D_m not measured
Cu $K\alpha$ radiation
λ = 1.54178 Å
Cell parameters from 25 reflections
θ = 10–55°
μ = 3.919 mm^{-1}
T = 293 (2) K
Needle
0.4 × 0.4 × 0.3 mm
Colourless

Data collection

KUMA KM-4 diffractometer
ω–2θ scans
Absorption correction: empirical *via* ψ scans (North, Phillips & Matthews, 1968)
T_{min} = 0.33, T_{max} = 0.87
3123 measured reflections
2956 independent reflections
2362 observed reflections [$I > 2\sigma(I)$]
R_{int} = 0.0327
θ_{max} = 80.99°
h = −10 → 10
k = −11 → 12
l = 0 → 11
2 standard reflections monitored every 100 reflections
intensity decay: 2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.0567
$wR(F^2)$ = 0.1594
S = 1.147
2956 reflections
191 parameters
H atoms: riding model
$w = 1/[\sigma^2(F_o^2) + (0.059P)^2 + 1.4438P]$ where $P=(F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = −0.21
$\Delta\rho_{max}$ = 0.92 e Å^{-3}
$\Delta\rho_{min}$ = −0.97 e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0036 (4)
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C, Tables 4.2.6.8 and 6.1.1.4)

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

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

	x	y	z	U_{eq}
Br1	0.28296 (5)	0.52064 (5)	0.94544 (4)	0.06972 (12)
N1	0.3552 (3)	0.2473 (2)	1.6834 (3)	0.0432 (6)
O1	0.1286 (3)	0.1851 (3)	1.9173 (3)	0.0670 (7)
O2	0.5327 (3)	0.3474 (3)	1.4255 (2)	0.0576 (6)
C1	0.2535 (4)	0.4234 (3)	1.5159 (3)	0.0469 (7)
C2	0.1357 (4)	0.3875 (4)	1.6845 (4)	0.0566 (8)
C3	0.1964 (4)	0.2619 (3)	1.7811 (3)	0.0502 (7)
C4	0.3998 (4)	0.3397 (3)	1.5291 (3)	0.0442 (7)
C5	0.4634 (4)	0.1487 (3)	1.7373 (3)	0.0436 (7)
C6	0.4504 (4)	0.0080 (3)	1.7418 (4)	0.0531 (8)
C7	0.5630 (5)	−0.0794 (4)	1.7907 (4)	0.0673 (11)
C8	0.6748 (5)	−0.0237 (4)	1.8332 (4)	0.0668 (11)

C9	0.6727 (4)	0.1184 (4)	1.8255 (4)	0.0588 (9)
N2	0.5704 (3)	0.2062 (3)	1.7754 (3)	0.0503 (7)
C10	0.3208 (6)	−0.0491 (4)	1.7010 (5)	0.0825 (12)
C11	0.1836 (3)	0.3779 (3)	1.4161 (3)	0.0433 (6)
C12	0.2497 (4)	0.4554 (3)	1.2570 (3)	0.0440 (7)
C13	0.1896 (4)	0.4143 (3)	1.1638 (3)	0.0455 (7)
C14	0.0629 (4)	0.2993 (3)	1.2267 (4)	0.0541 (8)
C15	−0.0017 (4)	0.2220 (4)	1.3853 (4)	0.0616 (9)
C16	0.0585 (4)	0.2603 (4)	1.4808 (4)	0.0566 (9)

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

N1—C4	1.379 (3)	N1—C5	1.438 (4)
N1—C3	1.395 (4)		
C4—N1—C3	113.0 (2)	C3—N1—C5	123.9 (2)
C4—N1—C5	123.1 (2)		

The H atoms were located from the $\Delta\rho$ map after anisotropic refinement of non-H atoms. The isotropic displacement parameters for all H atoms were held at 1.5 times the respective values for the parent C atom and their positions were refined using a riding model. The highest residual electron density exists in the proximity of the Br atom.

Data collection: KUMA Diffraction software. Cell refinement: KUMA Diffraction software. Data reduction: *DATARED* (Pèpe, 1979); KUMA Diffraction software. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXL*93. Software used to prepare material for publication: *SHELXL*93.

The crystallographic studies were supported by Technical University of Łódź.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BM1056). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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(*Z*)-4-[(*S*)-2,2-Dimethyl-1,3-dioxolan-4-ylmethylidene]-2-phenyl-1,3-oxazol-5(4*H*)-one

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Abstract

In the title compound, $C_{15}H_{15}NO_4$, the whole molecule, except for the dioxolane ring, adopts a nearly planar conformation. The dioxolane ring, which has an envelope conformation, is mainly situated on the *si,si* diastereotopic face of the olefinic bond. In the crystals of the title compound, rows of molecules are held together along the *x* direction by C···O intermolecular hydrogen bonds.

Comment

Azalactones unsaturated in position 4 are easily transformed into *N*-acyl-α,β-didehydroamino acid derivatives, which are powerful synthetic tools (Schmidt, Lieberknecht & Wild, 1988) present in many natural peptides, several of which exhibit biological activity (Schmidt, Häusler, Oehler & Poisel, 1979). Moreover, these compounds have proved to be useful intermediates in the synthesis of amino acids (Badsashah, Khan & Kidwai, 1972; Karpeiskaya, Levitina, Godunova & Klavunovskii, 1986), cycloaliphatic amino acids (Cativiela, Mayoral, Avenoza, González & Roy, 1990; Cativiela, Díaz-de-Villegas, Mayoral, Avenoza & Peregrina, 1993; Cativiela, Díaz-de-Villegas, Avenoza & Peregrina, 1993) and cyclopropylamino acids (Arenal, Bernabé, Fernández-Alvarez & Penadés-Ullate, 1985; Bland, Shah, Bortolusi & Stammer, 1988). In particular, (*Z*)-4-[(*S*)-2,2-dimethyl-1,3-dioxolan-4-ylmethylidene]-2-phenyl-1,3-oxazol-5(4*H*)-one, (I), has been successfully used as a substrate in asymmetric Diels–Alder (Buñuel, Cativiela & Díaz-de-Villegas, 1994, 1995; Buñuel, Cativiela, Díaz-de-Villegas & Garcia, 1994) and 1,3-dipolar cycloaddition (Cativiela, Díaz-de-Villegas, Lahoz & Jiménez, 1994; Cativiela, Díaz-de-Villegas & Jiménez, 1995*a*,*b*,*c*) reactions to afford versatile precursors of interesting cyclic amino acids. We report here the crystal-structure analysis of this chiral (*Z*)-azalactone derived from (*R*)-glyceraldehyde in order to provide conformational data to assist in the interpretation of the stereochemical course of reactions where the title compound is used as a substrate.