

distorted into slight 'boat' conformations (Table 3) with the C(3) and C(6) atoms at the apices, distortions which act to move each nitro group away from the incipient lone pair. There are no especially short intermolecular interactions (Fig. 2).

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Structure and Conformation of *N*-(4-Methyl-2-pyridyl)-*p*-chlorophenylsuccinimide

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Abstract. 3-(3-Chlorophenyl)-1-(4-methyl-2-pyridyl)-2,5-pyrrolidinedione, $C_{16}H_{13}ClN_2O_2$, $M_r = 300.74$, triclinic, $P\bar{1}$, $a = 10.330$ (2), $b = 10.958$ (2), $c = 13.251$ (3) Å, $\alpha = 78.48$ (2), $\beta = 80.86$ (2), $\gamma = 87.04$ (3)°, $V = 1450.8$ (5) Å³, $Z = 4$, $D_x = 1.377$ g cm⁻³, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 2.4$ mm⁻¹, $F(000) = 624$, $T = 293$ K, final $R = 0.065$ for 3257 reflections selected from 4928 unique data according to the criterion $I > 4\sigma(I)$. Two molecules in the independent part of the unit cell are indicated as conformational isomers resulting from phenyl- and pyridyl-ring rotations. Both forms are not too far from the energetically equivalent minima on the conformation map (molecular-mechanics calculation).

Introduction. The present work refers to comprehensive studies on the structure–activity relationship in phenylsuccinimide anticonvulsants (Łucka-Sobstał, Zejc & Obniska, 1977; Lange, Rump, Ilczuk,

Łapszewicz, Rabsztyn & Walczyna, 1977; Lange, Rump, Gałęcka, Ilczuk, Lechowska-Postek, Rabsztyn, Szymańska & Walczyna, 1977; Łapszewicz, Lange, Rump & Walczyna, 1978; Chmielewska, 1983, 1984; Zejc & Obniska, 1984; Zejc, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wiczyńska, 1987). All derivatives from this class of compound subjected to our earlier X-ray structure investigations (Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990; Kwiatkowski & Karolak-Wojciechowska, 1990, 1991, 1992a) reveal pharmacological activity, while the title compound is inactive. It was, therefore, of interest to solve the structure of the inactive derivative and thus gain the three-dimensional data required for comparison with the active succinimides (Kwiatkowski & Karolak-Wojciechowska, 1992b).

Experimental. The title compound recrystallized from ethanol to give colourless crystals. Unit-cell

dimensions of a 0.20 × 0.15 × 0.25 mm crystal, and intensity data were obtained from a KM-4 diffractometer. Final lattice parameters were determined from least-squares refinement of 25 reflections (25 < θ < 50°). No absorption correction was applied to data collected for θ < 80° and h 0/12, k -13/13, l -16/16, using ω -1.7 θ scans and Cu $K\alpha$ radiation at room temperature. Intensities of three standard reflections monitored every 50 reflections showed no significant fluctuations. 6053 reflections were measured of which 4928 were unique, $R_{\text{int}} = 0.033$; 3257 satisfied the $I > 4\sigma(I)$ criterion.

The direct-methods program *SHELXTL-PC* (Sheldrick, 1990) gave the solution of the structure and an E map provided the positions of all non-H atoms. The space group $P\bar{1}$ was chosen from the E -value distribution. All H atoms were located from $\Delta\rho$ maps and refined in a riding model with blocked isotropic thermal parameters taken as 1.5 times the temperature factors for their parent atoms. Refinement by full-matrix least squares on F with anisotropic temperature factors for all non-H atoms converged to $R = 0.065$ and $wR = 0.079$, with $w = 1/[\sigma^2(F) + 0.0085F^2]$ and extinction correction parameter $g = 0.011$; changes in all parameters $\Delta/\sigma < 0.05$; $S = 1.11$. The minimum and maximum peaks in the final $\Delta\rho$ map were -0.35 and 0.79 e Å⁻³ in the position close to the Cl atom. Atomic scattering factors were as given in *SHELXTL-PC*. All calculations were performed using the *SHELXTL-PC* system and the *CSU* program (Vicković, 1988).

Discussion. The two independent molecules of the title compound are presented (together with atom-numbering system) in Fig. 1. The atomic coordinates for non-H atoms of both molecules are given in Table 1,* while the respective bond lengths and angles are collected in Table 2. There are no unexpected values of bond lengths and angles in the structure.

All phenylsuccinimides previously investigated by us crystallized in centrosymmetric space groups (Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990; Kwiatkowski & Karolak-Wojciechowska, 1990, 1991, 1992a). Therefore, for a structure-activity relationship the relative configuration of the chiral centre has been chosen arbitrarily as R , and as a result, all the values of torsion angles were calculated assuming this configuration.

In fact, two independent molecules represent two rotational conformers with different positions of

Table 1. Non-H fractional atomic coordinates ($\times 10^4$) and equivalent isotropic temperature factors ($\text{\AA}^2 \times 10^3$)

$$U_{\text{eq}} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
Molecule (I)				
N(11)	-7120 (3)	5636 (3)	303 (2)	52 (1)
C(41)	-8025 (3)	5121 (3)	-167 (3)	54 (1)
C(11)	-8017 (4)	3716 (3)	252 (3)	57 (1)
C(21)	-6763 (4)	3486 (3)	748 (3)	63 (1)
C(31)	-6358 (3)	4731 (3)	848 (3)	54 (1)
O(11)	-5472 (3)	4975 (3)	1275 (2)	66 (1)
O(21)	-8688 (3)	5696 (3)	-777 (2)	66 (1)
C(111)	-8171 (4)	2957 (3)	-542 (3)	60 (1)
C(121)	-7200 (4)	2913 (4)	-1385 (3)	68 (1)
C(131)	-7375 (5)	2200 (4)	-2114 (4)	81 (1)
C(141)	-8484 (6)	1508 (5)	-1985 (5)	92 (1)
C(151)	-9426 (6)	1543 (5)	-1155 (5)	97 (2)
C(161)	-9295 (4)	2252 (4)	-418 (4)	77 (1)
Cl(11)	-6147 (2)	2155 (2)	-3161 (1)	119 (1)
C(51)	-6936 (3)	6939 (3)	203 (3)	51 (1)
C(61)	-6994 (4)	7434 (4)	1088 (3)	65 (1)
C(71)	-6795 (4)	8706 (4)	967 (4)	73 (1)
C(81)	-6575 (5)	9383 (4)	-53 (5)	84 (1)
C(91)	-6535 (5)	8801 (4)	-867 (4)	81 (1)
N(21)	-6708 (4)	7572 (3)	-765 (3)	66 (1)
C(101)	-6838 (6)	9308 (6)	1895 (5)	110 (2)
Molecule (II)				
N(12)	-6530 (3)	3723 (3)	3591 (2)	60 (1)
C(42)	-5758 (4)	2773 (4)	3246 (3)	58 (1)
C(12)	-4335 (4)	3114 (3)	3116 (3)	58 (1)
C(22)	-4395 (4)	4302 (4)	3565 (4)	71 (1)
C(32)	-5825 (4)	4685 (4)	3745 (4)	70 (1)
O(12)	-6285 (3)	5605 (3)	4011 (4)	101 (1)
O(22)	-6183 (3)	1841 (3)	3057 (2)	71 (1)
C(112)	-3448 (4)	2103 (3)	3578 (3)	56 (1)
C(122)	-2189 (4)	1981 (3)	3051 (3)	58 (1)
C(132)	-1328 (4)	1114 (4)	3502 (4)	68 (1)
C(142)	-1689 (5)	338 (4)	4462 (4)	81 (1)
C(152)	-2950 (5)	450 (5)	4974 (4)	86 (1)
C(162)	-3828 (5)	1348 (4)	4538 (3)	75 (1)
Cl(12)	274 (1)	1012 (1)	2872 (1)	83 (1)
C(52)	-7934 (4)	3789 (4)	3689 (3)	64 (1)
C(62)	-8637 (4)	3287 (4)	4649 (4)	78 (1)
C(72)	-9982 (5)	3391 (5)	4774 (5)	84 (1)
C(82)	-10527 (4)	4014 (6)	3944 (6)	99 (2)
C(92)	-9741 (5)	4486 (6)	2971 (5)	102 (2)
N(22)	-8408 (3)	4380 (4)	2840 (3)	81 (1)
C(102)	-10782 (5)	2870 (6)	5784 (6)	116 (2)

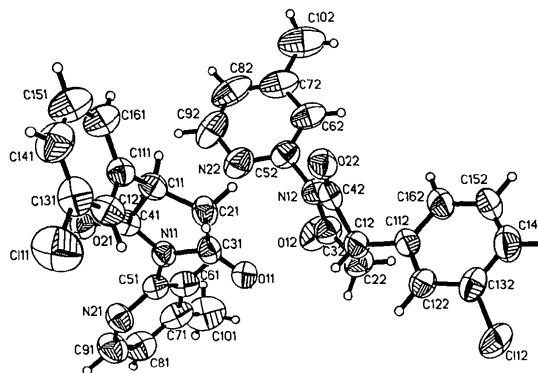


Fig. 1. The molecular structure and atom-numbering system of the title compound.

both aromatic rings (see Fig. 2). The torsion angle $\varphi = \text{C}(4)\text{—C}(1)\text{—C}(11)\text{—C}(12)$ (describing the phenyl-ring position) equals 67.6 (5)° in molecule (I) and -142.6 (4)° in molecule (II), while the torsion angle $\nu = \text{C}(4)\text{—N}(1)\text{—C}(5)\text{—N}(2)$ (pyridyl-ring positions)

* 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 55277 (14 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0364]

Table 2. Bond lengths (Å) and bond angles (°)

Molecule (I)		Molecule (II)	
N(11)—C(41)	1.400 (5)	N(12)—C(42)	1.385 (5)
N(11)—C(31)	1.392 (4)	N(12)—C(32)	1.377 (6)
N(11)—C(51)	1.427 (5)	N(12)—C(52)	1.435 (5)
C(41)—C(11)	1.529 (4)	C(42)—C(12)	1.510 (6)
C(41)—O(21)	1.203 (5)	C(42)—O(22)	1.217 (6)
C(11)—C(21)	1.531 (6)	C(12)—C(22)	1.530 (6)
C(11)—C(111)	1.497 (6)	C(12)—C(122)	1.498 (5)
C(21)—C(21)	1.484 (5)	C(22)—C(3)	1.510 (6)
C(31)—O(11)	1.216 (5)	C(32)—O(12)	1.186 (6)
C(111)—C(121)	1.384 (5)	C(112)—C(122)	1.388 (6)
C(111)—C(161)	1.398 (6)	C(112)—C(162)	1.382 (5)
C(121)—C(131)	1.396 (7)	C(212)—C(132)	1.380 (6)
C(131)—C(141)	1.376 (8)	C(132)—C(142)	1.389 (6)
C(131)—C(11)	1.731 (5)	C(132)—C(112)	1.740 (4)
C(141)—C(151)	1.353 (8)	C(142)—C(152)	1.381 (7)
C(151)—C(161)	1.390 (9)	C(152)—C(162)	1.404 (7)
C(51)—C(61)	1.379 (6)	C(52)—C(62)	1.384 (6)
C(51)—N(21)	1.322 (5)	C(52)—N(22)	1.333 (5)
C(61)—C(71)	1.393 (6)	C(62)—C(72)	1.374 (7)
C(71)—C(81)	1.396 (7)	C(72)—C(82)	1.360 (9)
C(71)—C(101)	1.501 (9)	C(72)—C(102)	1.481 (9)
C(81)—C(91)	1.353 (9)	C(82)—C(92)	1.426 (8)
C(91)—N(21)	1.345 (6)	C(920)—N(22)	1.362 (6)
C(41)—N(11)—C(31)	112.4 (3)	C(42)—N(12)—C(32)	113.9 (3)
C(41)—N(11)—C(51)	124.7 (3)	C(42)—N(12)—C(52)	124.4 (3)
C(31)—N(11)—C(51)	122.8 (3)	C(32)—N(12)—C(52)	121.5 (3)
N(11)—C(41)—C(11)	107.3 (3)	N(12)—C(42)—C(12)	108.7 (3)
N(11)—C(41)—O(21)	125.5 (3)	N(12)—C(42)—O(22)	124.5 (4)
C(11)—C(41)—O(21)	127.2 (3)	C(12)—C(42)—O(22)	126.8 (4)
C(41)—C(11)—C(21)	103.5 (3)	C(42)—C(12)—C(22)	103.3 (3)
C(41)—C(11)—C(111)	113.8 (3)	C(42)—C(12)—C(112)	115.2 (3)
C(21)—C(11)—C(111)	116.7 (3)	C(22)—C(12)—C(112)	114.8 (3)
C(11)—C(21)—C(31)	105.7 (3)	C(12)—C(22)—C(32)	106.3 (4)
N(11)—C(31)—C(21)	108.6 (3)	N(12)—C(32)—C(22)	106.8 (4)
N(11)—C(31)—O(11)	123.3 (3)	N(12)—C(32)—O(12)	125.2 (4)
C(21)—C(31)—O(11)	128.0 (3)	C(22)—C(32)—O(12)	127.9 (4)
C(11)—C(111)—C(121)	121.1 (3)	C(12)—C(112)—C(122)	118.6 (3)
C(11)—C(111)—C(161)	119.7 (4)	C(12)—C(112)—C(162)	121.5 (4)
C(121)—C(111)—C(161)	119.2 (4)	C(122)—C(112)—C(162)	119.8 (4)
C(111)—C(121)—C(131)	119.7 (4)	C(112)—C(122)—C(132)	119.3 (4)
C(121)—C(131)—C(141)	120.6 (5)	C(122)—C(132)—C(142)	122.0 (4)
C(121)—C(131)—C(11)	119.0 (4)	C(122)—C(132)—C(12)	119.3 (3)
C(141)—C(131)—C(11)	120.4 (4)	C(142)—C(132)—C(12)	118.6 (4)
C(131)—C(141)—C(151)	119.5 (6)	C(132)—C(142)—C(152)	118.4 (5)
C(141)—C(151)—C(161)	121.6 (6)	C(142)—C(152)—C(162)	120.3 (5)
C(111)—C(161)—C(151)	119.3 (4)	C(112)—C(162)—C(152)	120.2 (4)
N(11)—C(51)—C(61)	119.4 (3)	N(12)—C(52)—C(62)	117.4 (4)
N(11)—C(51)—N(21)	115.1 (3)	N(12)—C(52)—N(22)	115.1 (4)
C(61)—C(51)—N(21)	125.5 (4)	C(62)—C(52)—N(22)	127.5 (4)
C(51)—C(61)—C(71)	118.1 (4)	C(52)—C(62)—C(72)	118.3 (4)
C(61)—C(71)—C(81)	116.6 (4)	C(62)—C(72)—C(82)	117.1 (5)
C(61)—C(71)—C(101)	121.1 (4)	C(62)—C(72)—C(102)	120.6 (5)
C(81)—C(71)—C(101)	122.3 (5)	C(82)—C(72)—C(102)	122.3 (6)
C(71)—C(81)—C(91)	120.3 (5)	C(72)—C(82)—C(92)	121.4 (6)
C(81)—C(91)—N(21)	123.9 (5)	C(82)—C(92)—N(22)	121.7 (5)
C(51)—N(21)—C(91)	115.6 (4)	C(52)—N(22)—C(92)	113.9 (4)

equals -53.5 (5) and 89.5 (5) $^\circ$ respectively in the two molecules.

In the solution, however, no separate stable conformers with different pyridyl-ring orientations are observed. The evidence was provided by ^{13}C NMR spectra in which only single signals from all characteristic groups [$\delta = 21.45$ (CH_3), 37.61 (CH_2), 46.27 (CH)] were found. This observation prompted us to perform the conformation analysis to decide which of the two crystallographic conformations is closer to the energy minimum. Thus, the molecular-mechanics calculation was performed using the *MMX* program (PC version) (Burkert & Allinger, 1982) taking into account π overlap. We calculated the energies of conformations by clockwise variation of two torsion angles φ and ν described above. The

energy was minimized for each 15° rotation from -180 to 180° of both angles. The results of the calculation are depicted in Fig. 3.

There are four energetically equivalent energy minima visible in Fig. 3. The crystallographically observed conformations of the molecule are marked on the map by black squares with numbers (I) and (II) and are not too far from energy minima (marked by stars) with coordinates $\varphi_1 = 50^\circ$, $\nu_1 = -90^\circ$ and $\varphi_2 = -130^\circ$, $\nu_2 = 90^\circ$, respectively. Because of the lack of a distinct global minimum, the optimization of the geometries of the two crystallographic conformations ($\varphi = 67.6$, -142.6° ; $\nu = -53.5$, 89.5°) and four energy minima conformations ($\varphi = -130$, 50 , -130 , 50° ; $\nu = 90$, 90 , -90 , -90°) were performed using the MNDO method (Dewar & Thiel, 1977). The calculations were carried out with an IBM-486 AT computer using a program adapted from the

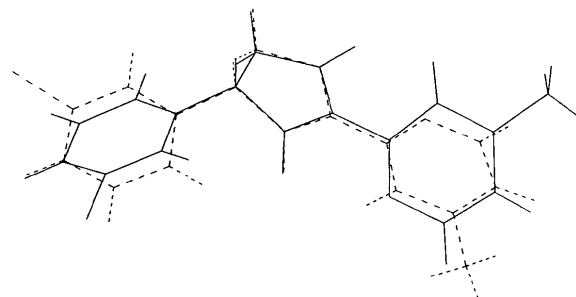


Fig. 2. Fitting of both independent molecules.

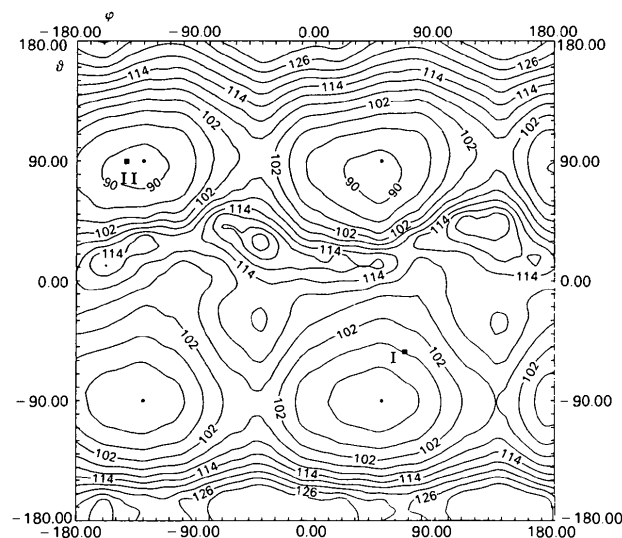


Fig. 3. The strain-energy distribution map showing functions of the torsion angles $\varphi = \text{C}(4)\text{—C}(1)\text{—C}(11)\text{—C}(12)$ and $\nu = \text{C}(4)\text{—N}(1)\text{—C}(5)\text{—N}(2)$. The equienergetic lines are for each 6 kJ mol^{-1} from 90 to 130 kJ mol^{-1} .

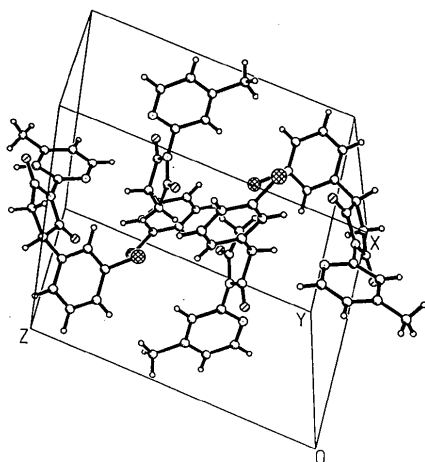


Fig. 4. The unit-cell packing.

standard *MOPAC* program (QCPE No. 464). The energy was minimized with respect to all geometrical variables. The calculations showed that the conformation obtained from $\varphi = -130^\circ$, $\nu = 90^\circ$ has lowest energy, but the differences between the energies for all calculated conformers do not exceed 1 kJ mol^{-1} . Therefore, it is probable that this conformation is the one observed in solution.

The four molecules in the unit cell (Fig. 4) are packed in such a manner that two molecules with conformation (I) are almost seized by two molecules with conformation (II). As is clear from Fig. 4, different conformations of both pairs of the molecules result in a compact packing within the unit cell.

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Structure of a Phenylhydrazone of Diacetone Protected 3-Keto-glucose

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Abstract. (*Z*)-3-Deoxy-1,2:5,6-di-*O*-isopropylidene-3-phenylhydrazone- α -D-ribo-hexofuranose, C₁₈H₂₄N₂O₅, $M_r = 348.40$, monoclinic, $P2_1$, $a = 10.867$ (2),

$b = 5.790$ (1), $c = 14.740$ (2) Å, $\beta = 94.75$ (1)°, $V = 924.3$ (8) Å³, $Z = 2$, $D_x = 1.26 \text{ g cm}^{-3}$, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 0.99 \text{ cm}^{-1}$, $F(000) = 372$, $T = 293 \text{ K}$, $R = 0.068$ for 2597 observed reflections. The title compound possesses the *Z* configuration. The phenyl ring is nearly coplanar with the furanose ring. The *gauche* conformation around the C(14)—C(8) bond

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