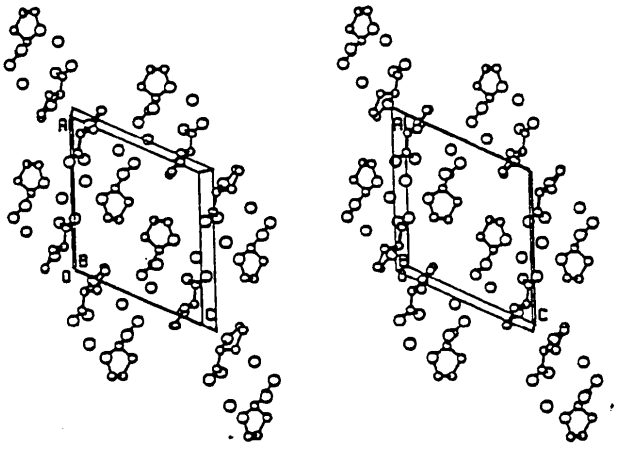

Fig. 2. Stereoscopic *PLUTO* (Motherwell, 1973) drawing of the packing of molecules viewed along the *b* axis.

Bond lengths and angles of the tetrahydrofuran rings of the molecules are similar to those of tetrahydrofuran at 148 K (Luger & Buschmann, 1983). Torsion angles indicate that the twofold ring symmetry of the latter compound no longer exists in the title compound.

The C—O bond lengths of two carboxylic groups, which are not significantly different from each other, are intermediate between single- and double-bond length. This indicates that the groups are deprotonated and electron-delocalized, though asymmetries in the C—C—O angles [C(2)—C(6)—O(7) and C(12)—C(16)—O(17) are larger than C(2)—C(6)—O(8) and C(12)—C(16)—O(18) respectively] suggest that C(6)—O(7) and C(16)—O(17) have double-bond character.

An unusual feature is the coordination of the two water molecules; four H atoms are tetrahedrally coordinated around each water O atom. All the H atoms around the water O(19) atom, and three H atoms around the water O(20) atom seem to be involved in hydrogen bonds, indicating that the water molecules take alternative orientations to form the hydrogen bonds. The packing scheme reveals that two layers of the molecules are held together by the water molecules to form a three-layered sheet. There are no interactions between the sheets except for van der Waals contacts.

References

AGTARAP, A., CHAMBERLIN, J. W., PINKERTON, M. & STEINRAUF, L. (1967). *J. Am. Chem. Soc.* **89**, 5737–5739.

BELANGER, P. C. & WILLIAMS, H. W. R. (1983). *Can. J. Chem.* **61**, 1383–1386.

DEBAERDEMAEKER, T., GERMAIN, G., MAIN, P., TATE, T. & WOOLFSON, M. M. (1987). *MULTAN87. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

ISHIGURO, M., IWATA, H., NAKATSUKA, T., TANAKA, R., MAEDA, Y., NISHIHARA, T., NOGUCHI, T. & NISHINO, T. (1988). *J. Antibiot.* **41**, 1685–1693.

JOHNSON, C. K. (1965). *ORTEP.* Report ORNL-3784. Oak Ridge National Laboratory, Tennessee, USA.

LUGER, P. & BUSCHMANN, J. (1983). *Angew. Chem. Int. Ed. Engl.* **22**, 410.

MOTHERWELL, W. D. S. (1973). *PLUTO.* Program for plotting molecular and crystal structures. Univ. Cambridge, England.

NORTH, A. C. T. PHILLIPS, D. C. & MATHEWS, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Acta Cryst. (1990). C**46**, 108–112

Structures of Three *N*-Pyridyl-2-phenylsuccinimides and Structural Evidence for Substituent Effects on Anticonvulsant Properties

BY WITOLD KWIATKOWSKI AND JANINA KAROLAK-WOJCIECHOWSKA*

Institute of General Chemistry, Technical University, 90–924 Łódź, Zwirki 36, *Poland*

AND JOLANTA OBNISKA AND ALFRED ZEJC

Institute of Pharmaceutical Chemistry, Medical Academy, 31-065 *Kraków, Skaleczna* 10, *Poland*

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Abstract. $T = 295$ K, Mo $K\alpha$ with $\lambda = 0.70930$ Å. Compound (I-6): $C_{15}H_{12}N_2O_2$, $M_r = 252.26$, monoclinic, $P2_1/c$, $a = 8.441$ (3), $b = 15.269$ (1), $c = 9.745$ (2) Å, $\beta = 92.34$ (2)°, $V = 1254.9$ (19) Å^3, $Z = 4$, $D_x = 1.335$ g cm^{-3}, $\mu = 0.85$ cm^{-1}, $F(000) = 528$, $R = 0.0345$ for 1682 observed reflections. Compound (I-10): $C_{16}H_{14}N_2O_2$, $M_r = 266.30$, monoclinic, $P2_1/n$, $a = 11.637$ (1), $b = 5.793$ (1), $c = 20.778$ (2) Å, $\beta =$

* To whom all correspondence should be addressed.

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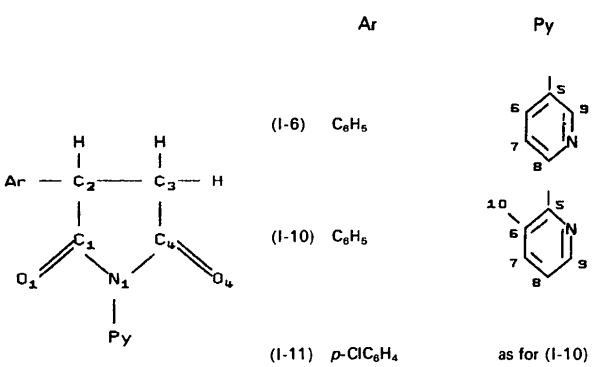
105·26 (1)°, $V = 1351·3$ (25) Å^3, $Z = 4$, $D_x = 1·309$ g cm^{-3}, $\mu = 0·82$ cm^{-1}, $F(000) = 560$, $R = 0·0379$ for 1840 observed reflections. Compound (I-11): $C_{16}H_{13}ClN_2O_2$, $M_r = 300·74$, triclinic, $P\bar{1}$, $a = 9·076$ (3), $b = 9·366$ (1), $c = 10·477$ (3) Å, $\alpha = 118·27$ (2), $\beta = 93·85$ (2), $\gamma = 105·26$ (1)°, $V = 737·2$ (15) Å^3, $Z = 2$, $D_x = 1·350$ g cm^{-3}, $\mu = 2·61$ cm^{-1}, $F(000) = 312$, $R = 0·0528$ for 2018 observed reflections. The three *N*-pyridyl-2-phenylsuccinimides [*N*-(3-methyl-2-pyridyl)-2-*p*-chlorophenylsuccinimide (I-11); *N*-(3-methyl-2-pyridyl)-2-phenylsuccinimide (I-10) and *N*-(3-pyridyl)-2-phenylsuccinimide (I-6)], examined by means of X-ray structure analysis, have been previously subjected to extensive pharmacological screening, with regard to their anticonvulsive activity. Pharmacological properties of the compounds examined are clearly connected with the conformation of the molecules. The conformation of the molecules of biologically active derivatives (I-10) and (I-11) differs from the conformation of the inactive molecule of (I-6). This difference involves relative positioning of the pyridyl ring and the succinimide moiety. The Cl atom in (I-11) has only a minor effect on the conformation and geometry of the molecule in comparison with (I-10).

Introduction. While searching for new compounds with predictable anticonvulsant properties, our attention was drawn to a group of phenylsuccinimides with a pyridyl substituent at the N atom (scheme). This led to the synthesis of numerous new phenylsuccinimides (Łucka-Sobstel Zejc & Obniska 1977; Zejc & Obniska, 1984; Zjec, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wiczyńska, 1987). The preliminary pharmacological screening of 29 derivatives, conducted by Wilimowski & Kędzierska (1979) proved that many were characterized by strong anticonvulsive activity. Two of these compounds, those with the best biological properties, were later subjected to extended pharmacological screenings (Chmielewska, 1983, 1984). The results pointed to the possibility of full acceptance of both these compounds in medical practice.



In the present paper we examine the structures of these two compounds, labelled on the scheme (I-10) [*N*-(3-methyl-2-pyridyl)-2-phenylsuccinimide] and (I-11) [*N*-(3-methyl-2-pyridyl)-2-*p*-clorophenylsuccinimide], as well as of a third compound (I-6) [*N*-(3-pyridyl-2-phenylsuccinimide] which is pharmacologically inactive.

The results of conformational analysis of a number of clinically applied anticonvulsants (Codding, Duke, Dargie & Benedictson, 1986; Wong, Defina & Andrews, 1986), including succinimides, have pointed to the presence of lipophilic aromatic rings in their molecules (connected to the heterocyclic system) as an indispensable condition for the compounds to be biologically active. Moreover, in biologically active molecules there should be centres potentially able to participate in the formation of H bonds, which play an important role in the interaction with the receptor. The three phenylsuccinimides selected for structural analysis satisfy all these conditions.

The aim of the research described in this paper is to investigate the relationships between structure and biological activity of *N*-pyridyl-2-phenylsuccinimides, searching for differences in conformation between active and inactive derivatives, and to determine the influence of phenyl-ring substitution by a Cl atom.

Experimental. Colourless crystals of (I-6), (I-10) and (I-11) suitable for X-ray structure analysis were obtained by recrystallization from ethanol. The data collection was carried out on an Enraf–Nonius CAD-4 diffractometer; cell dimensions of (I-6), (I-10) and (I-11) were from diffractometric measurements, 25 reflections ($6 < \theta < 38°$); crystals used: 0·3 × 0·5 × 0·5 mm for (I-6), 0·6 × 0·5 × 0·5 mm for (I-10) and 0·3 × 0·4 × 0·3 mm for (I-11); intensity data collection: $\theta < 25°$, ω–2θ scan method, monochromated Mo $K\alpha$ radiation, three intensity control reflections indicated no decay; 1968 recorded and 1682 observed reflections with $F > 3\sigma(F)$ for (I-6), 2155 and 1840 for (I-10) and 2353 and 2018 for (I-11), respectively, Lp correction applied. Atomic scattering factors were taken from *SHELX*76 (Sheldrick, 1976).

The structures were solved by direct methods with *SHELXS*86 (Sheldrick, 1986); $R(E) = 0·213$ for (I-6), 0·211 for (I-10) and 0·318 for (I-11); space group $P\bar{1}$ for (I-11) was established on the basis of *E*-value distribution; all three structures were refined on *F* by full-matrix least-squares (*SHELX*76), first isotropically to $R = 0·084$ for (I-6), 0·135 for (I-10) and 0·163 for (I-11); all H atoms from $\Delta\rho$ maps were refined in the riding model; after anisotropic refinement of all non-H atoms, final $R = 0·0345$, $wR = 0·0352$ with $w = 1·8031/[\sigma^2(F_o) + 0·0001(F_o)^2]$ and extinction correction factor $g = 0·00517$ for (I-6); *R*

Table 1. *Positional parameters* ($\times 10^4$) *for the non*-H *atoms and* U_{eq} ($Å^2 \times 10^3$) *for* (I-6)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
O4	4202 (1)	4328 (1)	2071 (1)	55 (1)
O1	−987 (1)	4104 (1)	617 (1)	59 (1)
N1	1708 (2)	4121 (1)	1077 (1)	39 (1)
C1	129 (2)	4234 (1)	1405 (2)	47 (1)
C16	3232 (2)	5753 (1)	4936 (2)	48 (1)
C9	1578 (2)	2953 (1)	−610 (2)	48 (1)
C4	2781 (2)	4363 (1)	2139 (2)	43 (1)
C5	2194 (2)	3753 (1)	−190 (2)	38 (1)
C11	2305 (2)	5598 (1)	3762 (2)	42 (1)
N2	2019 (2)	2546 (1)	−1750 (2)	59 (1)
C7	3771 (2)	3746 (1)	−2140 (2)	53 (1)
C6	3289 (2)	4168 (1)	−968 (2)	45 (1)
C8	3116 (2)	2947 (1)	−2477 (2)	58 (1)
C12	1846 (2)	6307 (1)	2952 (2)	57 (1)
C15	3673 (2)	6596 (1)	5305 (2)	61 (1)
C3	1837 (2)	4672 (1)	3350 (2)	49 (1)
C13	2277 (3)	7146 (1)	3329 (2)	66 (1)
C14	3182 (3)	7291 (1)	4504 (2)	66 (1)
C2	110 (2)	4538 (1)	2873 (2)	63 (1)

Table 2. *Positional parameters* ($\times 10^4$) *for the non*-H *atoms and* U_{eq} ($Å^2 \times 10^3$) *for* (I-10)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
N1	2023 (1)	437 (2)	−78 (1)	44 (1)
O4	3398 (1)	−1822 (2)	638 (1)	69 (1)
N2	1724 (1)	−1943 (3)	−1013 (1)	59 (1)
O1	426 (1)	2758 (3)	−551 (1)	71 (1)
C1	1107 (2)	1972 (3)	−59 (1)	48 (1)
C3	2036 (2)	627 (3)	1056 (1)	46 (1)
C11	2995 (2)	1494 (3)	1654 (1)	42 (1)
C4	2598 (2)	−432 (3)	543 (1)	46 (1)
C13	4196 (2)	908 (4)	2778 (1)	59 (1)
C5	2345 (1)	−213 (3)	−675 (1)	43 (1)
C12	3307 (2)	172 (3)	2230 (1)	50 (1)
C6	3275 (2)	924 (3)	−837 (1)	51 (1)
C15	4471 (2)	4275 (4)	2184 (1)	61 (1)
C16	3595 (2)	3546 (3)	1633 (1)	53 (1)
C14	4777 (2)	2960 (4)	2757 (1)	61 (1)
C7	3569 (2)	140 (4)	−1400 (1)	67 (1)
C9	2027 (2)	−2614 (4)	−1561 (1)	69 (1)
C2	1151 (2)	2399 (3)	659 (1)	53 (1)
C8	2947 (2)	−1647 (4)	−1765 (1)	70 (1)
C10	3925 (2)	2885 (4)	−427 (1)	80 (1)

Table 3. *Positional parameters* ($\times 10^4$) *for the non*-H *atoms and* U_{eq} ($Å^2 \times 10^3$) *for* (I-11)

$U_{eq} = (U_{11} + U_{22} + U_{33})/3.$

	x	y	z	U_{eq}
Cl	4518 (1)	2874 (2)	145 (1)	91 (1)
N1	1084 (3)	−2212 (3)	3501 (3)	45 (1)
O4	1960 (3)	721 (3)	4990 (3)	60 (1)
C11	1151 (4)	153 (4)	1894 (3)	46 (1)
C4	1157 (4)	−560 (4)	3864 (4)	46 (1)
N2	1277 (4)	−2967 (4)	5311 (3)	59 (1)
C16	1435 (4)	1869 (4)	2376 (4)	56 (1)
C15	2452 (5)	2691 (5)	1827 (4)	59 (1)
C5	2000 (4)	−2588 (4)	4395 (3)	46 (1)
C3	121 (4)	−690 (4)	2597 (3)	50 (1)
O1	−268 (3)	−4998 (3)	1689 (3)	72 (1)
C1	−23 (4)	−3483 (5)	2205 (4)	53 (1)
C13	2905 (5)	88 (5)	255 (4)	63 (1)
C14	3175 (4)	1800 (5)	781 (4)	57 (1)
C6	3519 (4)	−2551 (4)	4250 (4)	52 (1)
C12	1874 (5)	−739 (4)	804 (4)	59 (1)
C2	−795 (4)	−2607 (4)	1636 (4)	57 (1)
C8	3584 (5)	−3343 (5)	6094 (4)	70 (1)
C9	2073 (5)	−3355 (5)	6139 (4)	70 (1)
C7	4309 (5)	−2937 (5)	5145 (4)	65 (1)
C10	4210 (5)	−2187 (5)	3124 (5)	73 (1)

$= 0.0379$, $wR = 0.0414$ with $w = 2.007/[\sigma^2(F_o) + 0.0001\ (F_o)^2]$ and $g = 0.00812$ for (I-10); $R = 0.0528$, $wR = 0.0604$ with $w = 5.780/[\sigma^2(F_o) + 0.0002(F_o)^2]$ and $g = 0.0082$ for (I-11). The highest peak in $\Delta\rho$ maps = 0.14 (I-6), 0.17 (I-10) and 0.39 (I-11) e $Å^{-3}$; max. shift/e.s.d. = 0.001 (I-6), 0.001 (I-10) and 0.05 (I-11); calculations were performed on an Amstrad 1512 PC. Tables 1–3 report the atomic coordinates of compounds (I-6), (I-10), (I-11), respectively. The molecules are illustrated by stereoviews in Figs. 1–3. Selected bond distances and angles for all three compounds are given in Table 4.*

Discussion. The succinimide derivatives which have been used as anticonvulsant drugs in medicinal practice (Vida, 1977) may be divided into two basic types: those containing the NH group and those which are N-substituted. The structures of the molecules from these two groups differ not only with regard to the presence of intermolecular H bonds of the O···H···N type, but also with regard to the geometry of the molecules (Crowston, Lobo, Prabhakar, Rzepa & Williams, 1984; Sheldrick, 1981; Sheldrick & Akrigg 1987). In the structures of the three compounds examined no occurrence of H bonds was noticed. So, in the crystalline structures of our compounds all the intermolecular distances are longer than the sum of the van der Waals radii.

Figs. 1–3 show that the conformation of the pharmacologically inactive molecule (I-6) (Fig. 1) differs from the conformation of the molecules with confirmed anticonvulsive activity, (I-10) and (I-11) (Figs. 2, 3). This difference may be expressed as a different orientation of the pyridyl ring with respect to the five-membered succinimide ring. The dihedral angle between the mean planes of the five-membered ring and of the pyridyl ring is 53.6 (1)° in the structure of (I-6), while it is much larger for (I-10) and (I-11) [83.9 (1) and 83.2 (1)°, respectively]. Both types of compounds differ also, though to a much smaller degree, in the slope of the phenyl ring towards the succinimide moiety. Dihedral angles between the planes of the phenyl ring and of the five-membered ring are 83.7 (1)° in (I-6) and 96.1 (1) and 98.1 (1)° in

* Structure factors, anisotropic temperature factors for non-H atoms, H-atom coordinates and full lists of bond distances and bond angles for all structures have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52195 (46 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 4. *Selected bond distances* (Å) *and angles* (°)

	(I-6)	(I-10)	(I-11)
C1—O1	1·208 (2)	1·206 (2)	1·203 (4)
C4—O4	1·204 (2)	1·207 (2)	1·203 (4)
N1—C4	1·397 (2)	1·384 (2)	1·389 (4)
N1—C1	1·393 (2)	1·398 (2)	1·388 (4)
N1—C5	1·433 (2)	1·439 (2)	1·429 (4)
C8—N2	1·337 (2)		
N2—C9	1·339 (2)	1·335 (2)	1·326 (4)
N2—C5		1·324 (2)	1·328 (4)
C9—C5	1·383 (3)		
C6—C10		1·500 (3)	1·506 (5)
C14—Cl			1·744 (3)
Av. C—C distance in phenyl ring	1·380 (3)	1·384 (2)	1·376 (4)
Av. C—C distance in pyridyl ring	1·365 (9)	1·355 (11)	1·355 (12)
C4—N1—C5	122·8 (1)	122·6 (1)	124·1 (3)
C1—N1—C4	113·3 (1)	113·0 (1)	113·0 (3)
C1—N1—C5	123·8 (1)	124·4 (1)	122·9 (2)
N1—C4—O4	124·4 (1)	123·8 (2)	123·9 (3)
O4—C4—C3	127·5 (2)	127·7 (2)	127·9 (3)
N1—C4—C3	108·1 (1)	108·4 (1)	108·2 (3)
C2—C1—O1	128·1 (2)	128·7 (2)	128·8 (3)
C2—C1—N1	107·7 (2)	107·9 (1)	107·5 (3)
O1—C1—N1	124·3 (2)	123·5 (2)	123·7 (3)
C13—C14—C15	119·7 (2)	119·2 (2)	121·1 (3)
N1—C5—C6	121·2 (1)	118·9 (2)	119·9 (3)
N1—C5—C9	119·0 (1)		
N1—C5—N2		115·2 (2)	114·9 (3)
C9—C5—C6	119·8 (1)		
N2—C5—C6		125·9 (2)	125·3 (3)
C5—N2—C9		116·2 (2)	116·9 (3)
C9—N2—C8	116·7 (2)		
N2—C9—C8		123·3 (2)	122·7 (4)
N2—C9—C5	122·8 (2)		
N2—C8—C7	124·1 (2)		
Av. C—C—C angle in phenyl ring	120·0 (4)	120·0 (3)	120·0 (5)
Av. angle in pyridyl ring	119·9 (10)	120·0 (16)	119·8 (16)

(I-10) and (I-11), respectively. The differences in the conformations of the three molecules are shown in Fig. 4. It is obvious that the change of the pyridyl-substituent position, in the case of (I-10) and (I-11), was forced by the presence of an *ortho* methyl group. As a result, a rotation of the whole pyridyl substituent round the N1—C5 bond occurred (about 40°).

In the structure of the molecule of the pharmacologically inactive compound (I-6), we observed relatively short intramolecular distances C9(H9)···O1, and C6(H6)···O4. They are, respectively, 3·070 (3) and 3·041 (3) Å. This is probably the cause of (I-6) being pharmacologically inactive; the only element that makes the formation of H bonds with the receptor possible is the planar —CO—N(py)—CO— group. In (I-6) both carboxyl O atoms are 'blocked' by the pyridyl substituent, which makes formation of these bonds more difficult. In (I-10) and (I–11), the rotation of the pyridyl substituent results in an 'exposure' of both carboxyl groups, allowing the formation of hydrogen bonds C=O···H—*R* (*R* = receptor). This should explain the high anticonvulsive activity of both those compounds with exposed carbonyl groups and the inactivity of compound (I-6) with these groups blocked.

Unlike the phenyl and pyridyl rings, the five-membered rings constituting the succinimide moiety are not exactly planar. As demonstrated by the analysis of the values and signs of torsion angles, in all three cases this ring has the half-chair conformation. It is not, however, identical in all cases. Thus, in (I-6) and (I-10) the twofold symmetry axis bisects the

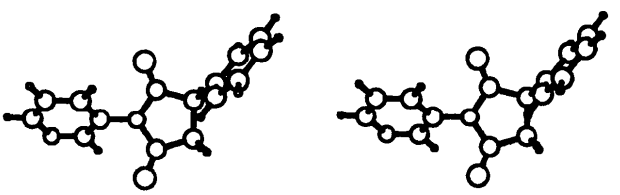


Fig. 1. The molecular structure of *N*-(3-pyridyl)-2-phenylsuccinimide (I-6).

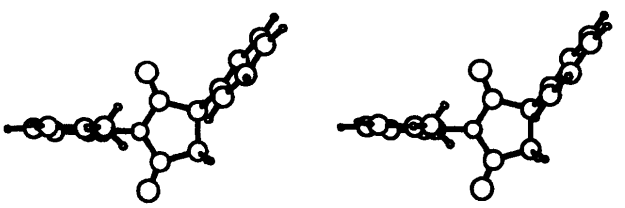


Fig. 2. The molecular structure of *N*-(3-methyl-2-pyridyl)-2-phenylsuccinimide (I-10).

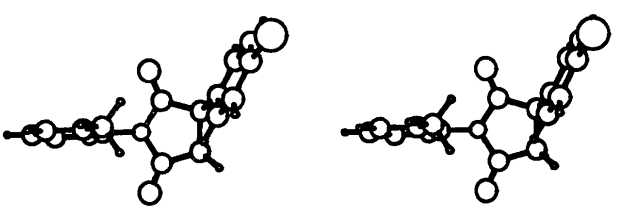


Fig. 3. The molecular structure of *N*-(3-methyl-2-pyridyl)-2-*p*-chlorophenylsuccinimide (I-11).

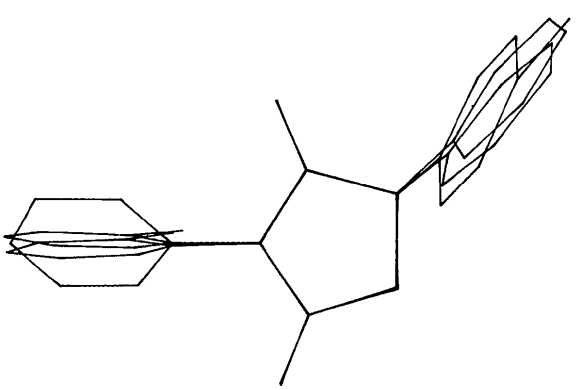


Fig. 4. The projection of three phenylsuccinimide molecules on the five-membered-ring plane.

C4—N1 bond, while in (I-11) it bisects the N1—C1 bond. Asymmetry parameters (Duax & Norton 1975) are, respectively, ΔC_2(C4—N1) = 0·76 for (I-6), 1·2 for (I-10) and ΔC_2(C1—N1) = 0·65 for (I-11). It follows that, from the viewpoint of the structure, the molecules of compounds (I-10) and (I-11) differ only in the symmetry of the five-membered rings. The bond lengths and angles in the three structures have expected values.

In conclusion, it follows from our studies that for the presence of anticonvulsant properties the pyridyl ring must be anticlinal with respect to the succinimide moiety. This position can be achieved by the *ortho* substitution of a methyl group into the pyridyl ring. This, in turn, facilitates the formation of hydrogen bonds with a receptor. The presence of a Cl substituent in the phenyl ring influences for instance the reduction of (I-11) toxicity, but in principle does not change the conformation of the molecule.

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References

CHMIELEWSKA, B. (1983). *Pharmazie*, **38**, 872–873.
CHMIELEWSKA, B. (1984). *Pharmazie*, **39**, 259–262.
CODDING, P. W., DUKE, N. E., DARGIE, R. L. & BENEDICTSON, M. S. (1986). *Pre-Meeting Symposium of Organic Crystallographic Chemistry*, Rydzyna, Poland.
CROWSTON, E. H., LOBO, A. M., PRABHAKAR, S., RZEPA, H. S. & WILLIAMS, D. J. (1984). *J. Chem. Soc. Chem. Commun.* pp. 276–278.
DUAX, W. L. & NORTON, D. A. (1975). *Atlas of Steroid Structure*, Vol. 1. New York: Plenum.
ŁUCKA-SOBSTEL, B., ZEJC, A. & OBNISKA, J. (1977). *Arch. Immunol. Therap. Exp.* **25**, 285–289.
SHELDRICK, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.
SHELDRICK, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Federal Republic of Germany.
SHELDRICK, W. S. (1981). *Acta Cryst.* B**37**, 300–302.
SHELDRICK, W. S. & AKRIGG, D. (1987). *Acta Cryst.* C**43**, 594–595.
VIDA, J. A. (1977). *Anticonvulsants*. New York: Academic Press.
WILIMOWSKI, M. & KĘDZIERSKA, L. (1979). *Arch. Immunol. Therap. Exp.* **27**, 389–396.
WONG, M. G., DEFINA, J. A. & ANDREWS, P. R. (1986). *J. Med. Chem.* **29**, 562–572.
ZEJC, A. & OBNISKA, J. (1984). *Acta Pol. Pharm*, **5**, 529–533.
ZEJC, A., OBNISKA, J., CHOJNACKA-WÓJCIK, E., TATARCZYŃSKA, E. & WICZYŃSKA, B. (1987). *Pol. J. Pharm.* **39**, 91–95.

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Structure of *N,N,N',N'*-Tetrakis(2-fluoro-2,2-dinitroethyl)oxamide by the Consistent Electron Density Approach

BY T. NARAYANA BHAT*

Laboratory of Molecular Biology, National Institutes of Health, Bethesda, Maryland 20892, *USA*

AND HERMAN L. AMMON

Department of Chemistry and Biochemistry and Center for Advanced Research in Biotechnology, University of Maryland, College Park, Maryland 20742, *USA*

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Abstract. $C_{10}H_8F_4N_{10}O_{18}$, $M_r = 632·22$, monoclinic, *Pc*, $a = 7·8885$ (6), $b = 6·7787$ (4), $c = 21·595$ (2) Å, $\beta = 108·21$ (1)°, $V = 1096$ (1) Å^3, $Z = 2$, $D_x = 1·914$ g cm^{-3}, λ(Cu $K\alpha$) = 1·5418 Å, $\mu = 18·7$ cm^{-1}, $F(000) = 636$, $T = 293$ K, 1791 unique data, 1733 with $I > 3\sigma(I)$, $R = 0·047$. The structure was solved by the consistent electron density approach (CEDA), in which a small starting set of random phases was refined and expanded by the application of restraints to the electron density. A refinable preliminary structure was obtained by fitting a model to a 3 Å map, calculated with 27 reflections (all data with $d > 3$ Å, and $|F_o| > 170$) whose phases had been determined by the CEDA. The molecule has an extended, open conformation; the two pairs of fluorodinitroethyl substituents located across the molecular center from each other show *i* and C_2 pseudo-symmetry respectively.

Introduction. The fluorodinitromethyl group is of interest as a substituent in energetic materials such as explosives and propellants. It has been called an 'explosophore', a term that has a meaning similar to that of 'chromophore' for appropriate functionality. At the University of Maryland we have been examin-

*Present address: Département d'immunologie, Institut Pasteur, 75724 Paris CEDEX 15, France.

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