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Structure of *m*-Nitrophenylsuccinimide

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Abstract. $C_{10}H_8N_2O_4$, $M_r = 220.18$, monoclinic, $P2_1/c$, $a = 16.381\,(3)$, $b = 7.042\,(1)$, $c = 18.157\,(4)$ Å, $\beta = 113.02\,(3)°$, $V = 1927.8\,(8)$ Å^3, $Z = 8$, $D_x = 1.517$ g cm^{-3}, $\lambda(\text{Cu } K\alpha) = 1.54178$ Å, $\mu = 0.98$ mm^{-1}, $F(000) = 912$, final $R = 0.038$ for 2902 observed reflections with $I > 4\sigma(I)$ (of 3566 unique data). The two independent molecules differ slightly in the inclination of the phenyl and five-membered rings and the respective C4—C1—C11—C12 torsional angles equal −125.7 (2) and −126.8 (2)° in molecules (I) and (II). The five-membered rings are in the twist conformation [the deviations of C1 and C2 from the plane N1, C4, C3 are −0.196 (1) and 0.144 (1) Å in molecule (I) and −0.022 (1) and 0.091 (1) Å in molecule (II)]. Both molecules are joined by a hydrogen bond N11—H1···O12 = 2.902 (2) Å and form two columns along the [010] direction of the unit cell.

Experimental. The title compound was recrystallized from ethanol and gave light-yellow crystals. Crystal dimensions 0.15 × 0.20 × 0.20 mm. Unit-cell dimensions and intensity measurements were obtained from a KM-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections ($25 < \theta < 50°$); no absorption correction applied; $\theta < 80°$; h: −18/18, k: 0/8, l: 0/21; ω–1.7θ scan technique; Cu $K\alpha$ radiation at room temperature; intensity of two standard reflections monitored every 50 reflections showed no significant fluctuations; 3566 unique reflections measured of which 2902 satisfied the criterion $I > 4\sigma(I)$.

The direct-methods technique using the *SHELXTL-PC* program (Sheldrick, 1989) gave no structure solution. The structure was solved successfully by the *SIR* program (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989) and an *E* map provided the positions of all non-H atoms. All H atoms were located from a $\Delta\rho$ map and refined in the riding model with blocked isotropic thermal parameters taken as 1.5 times the temperature factors for their parent atoms (positional parameters of H atoms bonded to N atoms were refined). Refinement by full-matrix least squares with anisotropic temperature factors for all non-H atoms converged to $R = 0.038$ and $wR = 0.044$ with $w = 1.0/[\sigma(F)^2 + 0.0003F^2]$; empirical extinction correction coefficient $g = 0.0099$; changes

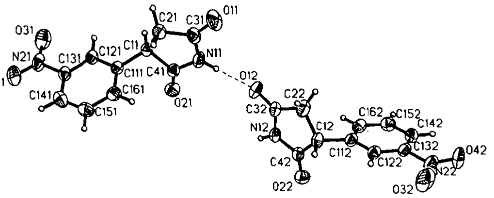

Fig. 1. View of the two independent molecules and atom-numbering system of *m*-nitrophenylsuccinimide.

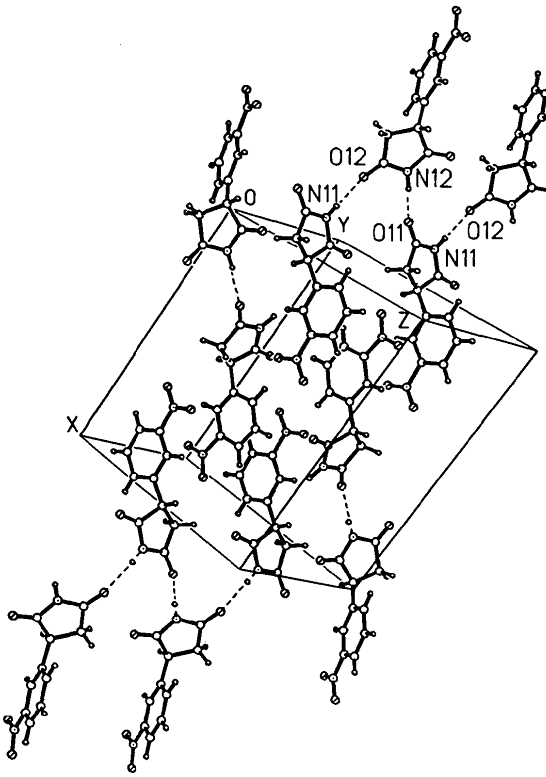

Fig. 2. The unit-cell packing with hydrogen-bonding system marked by dashed lines.

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Table 1. *Non*-H *fractional atomic coordinates* (× 10^4) *and equivalent isotropic temperature factors* (× 10^3 Å^2) *with e.s.d.'s in parentheses*

	x	*y*	*z*	U_{eq}*
Molecule (I)				
N11	1074 (1)	− 2254 (2)	2330 (1)	41 (1)
C41	277 (1)	− 1440 (2)	1866 (1)	33 (1)
C11	− 379 (1)	− 3055 (2)	1491 (1)	31 (1)
C21	232 (1)	− 4774 (2)	1597 (1)	37 (1)
C31	1093 (1)	− 4189 (3)	2251 (1)	40 (1)
O21	128 (1)	241 (2)	1799 (1)	47 (1)
O11	1709 (1)	− 5196 (2)	2641 (1)	63 (1)
C111	− 1023 (1)	− 2645 (2)	653 (1)	29 (1)
C121	− 1926 (1)	− 2795 (2)	463 (1)	32 (1)
C131	− 2505 (1)	− 2464 (2)	− 318 (1)	36 (1)
C141	− 2220 (1)	− 1972 (3)	− 913 (1)	44 (1)
C151	− 1325 (1)	− 1821 (3)	− 721 (1)	46 (1)
C161	− 727 (1)	− 2156 (3)	51 (1)	38 (1)
N21	− 3463 (1)	− 2651 (2)	− 522 (1)	48 (1)
O31	− 3710 (1)	− 3051 (3)	12 (1)	65 (1)
O41	− 3965 (1)	− 2429 (3)	− 1215 (1)	77 (1)
Molecule (II)				
N12	3312 (1)	2785 (2)	3014 (1)	40 (1)
C42	4115 (1)	3514 (2)	3070 (1)	37 (1)
C12	4778 (1)	1879 (3)	3275 (1)	38 (1)
C22	4255 (1)	175 (3)	3398 (1)	50 (1)
C32	3324 (1)	875 (3)	3162 (1)	40 (1)
O22	4241 (1)	5155 (2)	2966 (1)	54 (1)
O12	2677 (1)	− 40 (2)	3109 (1)	60 (1)
C112	5628 (1)	2333 (2)	3984 (1)	34 (1)
C122	6442 (1)	2140 (2)	3923 (1)	35 (1)
C132	7206 (1)	2471 (2)	4601 (1)	37 (1)
C142	7196 (1)	3005 (3)	5328 (1)	43 (1)
C152	6380 (1)	3218 (3)	5373 (1)	46 (1)
C162	5604 (1)	2889 (3)	4709 (1)	41 (1)
N22	8068 (1)	2223 (2)	4537 (1)	50 (1)
O32	8080 (1)	1944 (3)	3886 (1)	81 (1)
O42	8738 (1)	2311 (3)	5148 (1)	79 (1)

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

Table 2. *Bond lengths* (Å) *and bond angles* (°) *with e.s.d.'s in parentheses*

Molecule (I)		Molecule (II)	
N11—C41	1.372 (2)	N12—C42	1.378 (2)
N11—C31	1.372 (3)	N12—C32	1.370 (3)
C41—C11	1.530 (2)	C42—C12	1.526 (2)
C41—O21	1.205 (2)	C42—O22	1.202 (2)
C11—C21	1.534 (2)	C12—C22	1.541 (3)
C11—C111	1.502 (2)	C12—C112	1.515 (2)
C21—C31	1.503 (2)	C22—C32	1.497 (2)
C31—O11	1.210 (2)	C32—O12	1.212 (2)
C111—C121	1.385 (2)	C112—C122	1.387 (3)
C111—C161	1.401 (3)	C112—C162	1.389 (3)
C121—C131	1.383 (2)	C122—C132	1.390 (2)
C131—C141	1.378 (3)	C132—C142	1.379 (3)
C131—N21	1.471 (2)	C132—N22	1.472 (3)
C141—C151	1.372 (2)	C142—C152	1.379 (3)
C151—C161	1.381 (2)	C152—C162	1.387 (2)
N21—O31	1.221 (3)	N22—O32	1.206 (3)
N21—O41	1.214 (2)	N22—O42	1.219 (2)
C41—N11—C31	113.9 (2)	C42—N12—C32	114.3 (2)
N11—C41—C11	107.3 (1)	N12—C42—C12	107.7 (1)
N11—C41—O21	125.4 (2)	N12—C42—O22	124.4 (2)
C11—C41—O21	127.3 (2)	C12—C42—O22	127.9 (2)
C41—C11—C21	102.7 (1)	C42—C12—C22	103.9 (1)
C41—C11—C111	113.6 (1)	C42—C12—C112	112.2 (1)
C21—C11—C111	116.4 (1)	C22—C12—C112	114.8 (2)
C11—C21—C31	104.3 (1)	C12—C22—C32	105.3 (2)
N11—C31—C21	107.6 (2)	N12—C32—C22	108.3 (2)
N11—C31—O11	124.7 (2)	N12—C32—O12	124.2 (2)
C21—C31—O11	127.7 (2)	C22—C32—O12	127.5 (2)
C11—C111—C121	119.8 (1)	C12—C112—C122	120.3 (1)
C11—C111—C161	121.2 (2)	C12—C112—C162	120.5 (2)
C121—C111—C161	119.1 (2)	C122—C112—C162	119.2 (2)
C111—C121—C131	118.7 (2)	C112—C122—C132	118.3 (2)
C121—C131—C141	122.6 (2)	C122—C132—C142	123.4 (2)
C121—C131—N21	118.7 (1)	C122—C132—N22	118.0 (1)
C141—C131—N21	118.7 (2)	C142—C132—N22	118.6 (2)
C131—C141—C151	118.4 (2)	C132—C142—C152	117.5 (2)
C141—C151—C161	120.5 (2)	C142—C152—C162	120.7 (2)
C111—C161—C151	120.6 (2)	C112—C162—C152	121.0 (2)
C131—N21—O31	118.2 (2)	C132—N22—O32	118.7 (2)
C131—N21—O41	118.2 (2)	C132—N22—O42	118.2 (2)
O31—N21—O41	123.5 (2)	O32—N22—O42	123.1 (2)

in all parameters $\Delta/\sigma < 0.002$; $S = 2.52$. The minimum and maximum peaks in the final $\Delta\rho$ map were -0.23 and $0.26\ \text{e Å}^{-3}$; atomic scattering factors as given in the program. All calculations were performed using the *SHELXTL* system (Sheldrick, 1989) on a PC computer and with the *CSU* program (Vicković, 1988). A general view of molecules (I) and (II) together with the atom-numbering system is shown in Fig. 1. The packing in the unit cell and the hydrogen-bonding system are clear from Fig. 2. Atomic coordinates are given in Table 1.* There are no unusual bond lengths or angles in the molecules (see Table 2).

Related literature. The present work relates to comprehensive studies on the structure–activity relationships in five-membered heterocyclic anticonvulsants (Łucka-Sobstal, Zejc & Obniska, 1977; Lange, Rump, Ilczuk, Łapszewicz, Rabsztyn & Walczyna, 1977; Lange, Rump, Gałecka, 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 of phenylsuccinimide subjected to our earlier X-ray structure analyses (Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990; Kwiatkowski & Karolak-Wojciechowska, 1990, 1991, 1992) reveal pharmacological activity while the title compound is inactive. It was, therefore, of interest to solve this structure and thus gain the three-dimensional data required for comparison with the active succinimide.

We are most obliged to Professor Jerzy Lange for his kind offering of the compounds to be used in our researches and for helpful discussions.

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* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54540 (11 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR0340]

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Structure of an Anticonvulsant *N*-Methyl-*m*-bromophenylsuccinimide

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Abstract. 3-(3-Bromophenyl)-1-methyl-2,5-pyrrolidinedione, $C_{11}H_{10}BrNO_2$, $M_r = 268.11$, triclinic, $P\bar{1}$, $a = 11.606$ (2), $b = 11.832$ (2), $c = 10.370$ (2) Å, $\alpha = 101.84$ (1), $\beta = 107.76$ (1), $\gamma = 118.28$ (2)°, $V = 1085.7$ (5) Å^3, $Z = 4$, $D_x = 1.640$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 5.04$ mm^{-1}, $F(000) = 536$, room temperature, final $R = 0.061$ for 2971 observed reflections with $I > 3\sigma(I)$ (of 3743 unique data). The only slight differences in conformation of the two independent molecules are in the inclination of the phenyl and five-membered rings. The torsion angle C4—C1—C11—C12 is −115.4 (4)° in molecule I and −131.6 (4)° in molecule II. The five-membered imide ring has the open-envelope conformation in molecule I [the deviation of C11 from the plane N11—C41—C21—C31 is −0.117 (3) Å] and the twist conformation in molecule II [the deviations of C12 and C22 from the plane N12—C42—C32 are 0.117 (1) and −0.064 (1) respectively].

Experimental. The title compound (Lange, Urbański, Venulet, Desperak-Naciążek & Szmal, 1966) was recrystallized from ethanol and gave colorless block-like crystals. Crystal dimensions 0.30 × 0.15 × 0.30 mm. Unit-cell dimensions and intensity measurements obtained from a KM-4 diffractometer. Final lattice parameters from least-squares refinement of 25 reflections ($25 < \theta < 50°$); no absorption correction applied; $\theta < 80°$; h: 0/−11, k: −12/12, l: −11/11; $\omega - 1.8\theta$ scan technique; Cu $K\alpha$ radiation at room temperature; intensity of two standard reflections monitored every 50 reflections showed no significant fluctuations; 3743 unique reflections measured with 2971 satisfying the criterion $I > 3\sigma(I)$.

The direct methods routine in the *SHELXTL-PC* program (Sheldrick, 1989) gave the solution of the structure in space group $P\bar{1}$ with the E map providing positions of all non-H atoms in two independent molecules. The centrosymmetric space group was indicated by the distribution of E values. All H atoms were located from a $\Delta\rho$ map and refined in the 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 $R = 0.061$ and $wR = 0.090$ with $w = 1/[\sigma^2(F) + 0.0036F^2]$ and extinction correction parameter $g = 0.011$; maximum $\Delta/\sigma = 0.17$; $S = 2.07$. The minimum and maximum peaks in the final $\Delta\rho$ map were −1.18 and 1.34 e Å^{-3} and the peaks were located around the Br-atom positions;

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