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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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atomic scattering factors as given in the program. All calculations were performed using the *SHELXTL* system on a PC computer and the *CSU* program (Vicković, 1988).

A general view of the two molecules together with the atom-numbering system is shown in Fig. 1. The similarity between the conformations of the two molecules is clear from Fig. 2. The atomic coordinates are given in Table 1.* There are no unexpected values between the bond lengths and angles given in Table 2.

Related literature. The present investigation was undertaken as part of our systematic studies on the conformation and structure of phenylsuccinimides with expected anticonvulsant activity, to determine the structure–activity relationships (Łucka-Sobstal, Zejc & Obniska, 1977; Lange, Rump, Ilczuk, Łapiszewicz, Rabsztyn & Walczyna, 1977; Lange, Rump, Gałecka, Ilczuk, Lechowska-Postek, Rabsztyn, Szymańska & Walczyna, 1977; Łapiszewicz, Lange, Rump & Walczyna, 1978; Chmielewska, 1983, 1984; Zejc & Obniska, 1984; Zejc, Obniska, Chojnacka-Wójcik, Tatarczyńska & Wiczyńska, 1987). We have solved the structure of eight derivatives (Kwiatkowski, Karolak-Wojciechowska, Obniska & Zejc, 1990; Kwiatkowski & Karolak-Wojciechowska, 1990, 1991, 1992) from the 27 available compounds. The activity of the title

* 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 54470 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

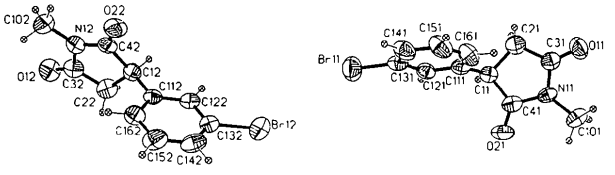


Fig. 1. View of the two independent molecules with the atom-numbering system for *N*-(methyl)-*m*-Br-phenylsuccinimide.

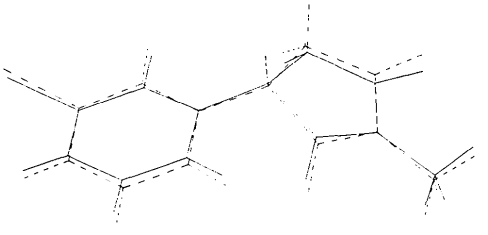


Fig. 2. Comparison of the conformation of the two molecules.

Table 1. *Fractional atomic coordinates* ($\times 10^4$) *and equivalent isotropic temperature factors* ($\times 10^3$ Å^2) *with their e.s.d's in parentheses*

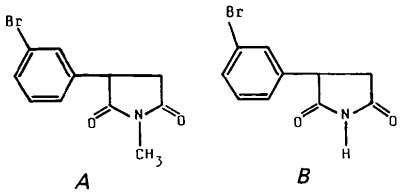
$U_{eq} = \frac{1}{3}\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
Molecule I				
N11	−1239 (3)	704 (3)	3075 (3)	44 (1)
C41	178 (4)	1395 (3)	3333 (3)	42 (1)
C11	649 (4)	2788 (4)	3214 (4)	48 (1)
C21	−780 (4)	2706 (4)	2650 (4)	55 (1)
C31	−1911 (4)	1379 (4)	2678 (4)	56 (1)
O11	−3167 (3)	935 (4)	2422 (4)	75 (1)
O21	925 (3)	945 (3)	3623 (3)	65 (1)
C111	1459 (4)	3070 (3)	2300 (4)	45 (1)
C121	2924 (4)	4198 (4)	2963 (4)	48 (1)
C131	3654 (4)	4474 (4)	2120 (4)	57 (1)
C141	2966 (4)	3664 (5)	621 (4)	65 (1)
C151	1503 (4)	2521 (5)	−43 (4)	74 (1)
C161	769 (4)	2238 (4)	788 (4)	61 (1)
Br11	5673.1 (7)	6046.6 (7)	3072.8 (9)	84 (1)
C101	−2064 (5)	−639 (4)	3145 (4)	62 (1)
Molecule II				
N12	15164 (3)	12310 (3)	2066 (3)	42 (1)
C42	13793 (4)	11146 (4)	1601 (4)	46 (1)
C12	13686 (4)	10859 (4)	2958 (4)	46 (1)
C22	15298 (4)	11894 (4)	4202 (4)	44 (1)
C32	16101 (4)	12840 (4)	3573 (4)	51 (1)
O12	17347 (3)	13902 (3)	4215 (3)	70 (1)
O22	12825 (3)	10470 (3)	342 (3)	66 (1)
C112	12965 (4)	9321 (3)	2638 (4)	45 (1)
C122	11836 (4)	8641 (4)	2999 (4)	44 (1)
C132	11161 (4)	7225 (4)	2685 (4)	49 (1)
C142	11575 (4)	6461 (4)	2009 (4)	56 (1)
C152	12725 (5)	7141 (4)	1692 (4)	65 (1)
C162	13411 (4)	8567 (4)	1992 (4)	53 (1)
Br12	9599.6 (7)	6304.6 (6)	3167.0 (7)	73 (1)
C102	15657 (4)	12818 (4)	1073 (4)	62 (1)

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

Molecule I		Molecule II	
N11—C41	1.346 (5)	N12—C42	1.358 (4)
N11—C31	1.395 (7)	N12—C32	1.390 (4)
N11—C101	1.448 (5)	N12—C102	1.427 (6)
C41—C11	1.517 (6)	C42—C12	1.541 (7)
C42—O21	1.212 (6)	C42—O22	1.197 (4)
C11—C21	1.528 (7)	C12—C22	1.540 (4)
C11—C111	1.506 (7)	C12—C112	1.504 (6)
C21—C31	1.509 (6)	C22—C32	1.493 (6)
C31—O11	1.205 (6)	C32—O12	1.209 (4)
C111—C121	1.380 (4)	C112—C122	1.389 (6)
C111—C161	1.387 (5)	C112—C162	1.384 (7)
C121—C131	1.377 (7)	C122—C132	1.380 (6)
C131—C141	1.371 (5)	C132—C142	1.377 (8)
C131—Br11	1.906 (3)	C132—Br12	1.904 (5)
C141—C151	1.384 (5)	C142—C152	1.377 (7)
C151—C161	1.370 (7)	C152—C162	1.392 (6)
C41—N11—C31	114.0 (3)	C42—N12—C32	113.2 (4)
C41—N11—C101	125.4 (4)	C42—N12—C102	123.2 (4)
C31—N11—C101	120.6 (4)	C32—N12—C102	123.0 (4)
N11—C41—C11	109.0 (3)	N12—C42—C12	108.9 (4)
N11—C41—O21	123.8 (4)	N12—C42—O22	125.2 (4)
C11—C41—O21	127.2 (4)	C12—C42—O22	125.9 (4)
C41—C11—C21	103.9 (3)	C42—C12—C22	102.7 (4)
C41—C11—C111	112.7 (4)	C42—C12—C112	111.8 (4)
C21—C11—C111	116.4 (4)	C22—C12—C112	116.7 (4)
C11—C21—C31	105.4 (4)	C12—C22—C32	105.5 (4)
N11—C31—C21	107.2 (4)	N12—C32—C22	108.5 (4)
N11—C31—O11	123.9 (4)	N12—C32—O12	123.4 (4)
C21—C31—O11	129.0 (4)	C22—C32—O12	128.0 (4)
C11—C111—C121	119.9 (4)	C12—C112—C122	119.7 (4)
C11—C111—C161	121.9 (4)	C12—C112—C162	121.1 (4)
C121—C111—C161	118.2 (4)	C122—C112—C162	119.2 (4)
C111—C121—C131	119.8 (4)	C112—C122—C132	119.7 (4)
C121—C131—C141	122.0 (4)	C122—C132—C142	121.4 (4)
C121—C131—Br11	118.6 (3)	C122—C132—Br12	119.1 (3)
C141—C131—Br11	119.4 (4)	C142—C132—Br12	119.5 (3)
C131—C141—C151	118.3 (5)	C132—C142—C152	119.1 (4)
C141—C151—C161	120.1 (5)	C142—C152—C162	120.2 (4)
C111—C161—C151	121.6 (4)	C112—C162—C152	120.4 (4)

compound (*A*) is comparable to the activity of *m*-Br-phenylsuccinimide (*B*), a clinically used anticonvulsant (LEFADOL) (Lange *et al.*, 1966). This structure was investigated because of the very poor quality of monocrystals obtainable for LEFADOL.



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Structure of 2-Amino-5-methylisophthalonitrile

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Abstract. $C_9H_7N_3$, $M_r = 157{\cdot}2$, monoclinic, $P2_1/c$, $a = 7{\cdot}145\ (8)$, $b = 10{\cdot}866\ (5)$, $c = 11{\cdot}006\ (6)$ Å, $\beta = 107{\cdot}53\ (6)°$, $V = 814{\cdot}8$ Å^3, $Z = 4$, $D_x = 1{\cdot}281$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 0{\cdot}76$ cm^{-1}, $F(000) = 328$, $T = 293$ K, $R = 0{\cdot}048$ for 1023 observed reflections. The reaction of α,β-unsaturated aldehydes with malononitrile is a synthetic method leading to 2-alkoxy-3-cyanopyridines. In this reaction a minor product is also obtained having an isophthalonitrilic structure. Thus, the title compound comes from the reaction between methacrolein and malononitrile. The X-ray determination confirms the chemical structure. The amino group evenly releases electronic charge towards both nitrile groups. Bond distances, as well as NMR and IR data, are in agreement with this assumption.

Experimental. In the context of a study on the reaction between α,β-unsaturated aldehydes with malononitrile, the title compound was obtained in low yield as a side product. After chromatographic separation, single crystals were obtained by slow evaporation from a hexane solution. A suitable prismatic single crystal *ca* 0·5 × 0·4 × 0·4 mm was moun-

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