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N,N'-Biphthalimide

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Abstract. $C_{16}H_8N_2O_4$, monoclinic, space group $P2_1/c$, $a = 8\cdot473$ (1), $b = 13\cdot404$ (1), $c = 11\cdot604$ (1) Å, $\beta = 92\cdot79$ (1)°, $Z = 4$, $D_x = 1\cdot47$ g cm^{-3}. $R = 5\cdot1\%$ for 1390 observed reflexions. The pseudo symmetry of the molecule is 222; the two halves of the dimer are twisted through 78°.

Introduction. The title compound was obtained during attempts to recrystallize an intermediate in the synthesis of tetracycline analogues. It was decided to undertake the determination of the structure by X-ray diffraction methods.

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The intensities were collected from a crystal of dimensions $0\cdot29 \times 0\cdot20 \times 0\cdot20$ mm on a Philips PW 1100 four-circle diffractometer with graphite-monochromated Mo $K\alpha$ radiation and an $\omega/2\theta$ scan. 2310 reflexions were recorded up to $\theta = 25°$. 1390 obeyed the condition $I > 2\sigma(I)$ and were considered observed. The intensities were corrected for Lorentz and polarization factors. Absorption corrections were not applied ($\mu = 1\cdot17$ cm^{-1}). The structure was solved with *MULTAN* 77 (Main, Lessinger, Woolfson, Germain & Declercq, 1977) and refined in the usual way (Foces-Foces, Cano & García-Blanco, 1978). The final R was $5\cdot1\%$. Weights were applied by adjusting curves as functions of $\sin \theta/\lambda$ and F_o. R_w was $5\cdot7\%$. The final difference synthesis showed no electron density $>0\cdot26$ e Å^{-3}.

Table 1. *Final positional parameters with their e.s.d.'s*

	x	y	z		x	y	z
N(1)	0·2916 (4)	0·2173 (2)	0·3979 (3)	N(1')	0·2556 (4)	0·3162 (2)	0·4116 (3)
C(2)	0·4414 (4)	0·1838 (3)	0·3662 (3)	C(2')	0·1725 (4)	0·3726 (3)	0·3272 (3)
C(3)	0·4261 (4)	0·0742 (3)	0·3633 (3)	C(3')	0·1653 (4)	0·4734 (3)	0·3785 (3)
C(4)	0·5389 (5)	0·0028 (3)	0·3430 (3)	C(4')	0·0952 (5)	0·5590 (3)	0·3339 (4)
C(5)	0·4933 (5)	−0·0955 (3)	0·3481 (3)	C(5')	0·1013 (5)	0·6434 (3)	0·4033 (4)
C(6)	0·3398 (5)	−0·1222 (3)	0·3717 (3)	C(6')	0·1794 (5)	0·6430 (3)	0·5099 (4)
C(7)	0·2274 (5)	−0·0504 (3)	0·3922 (3)	C(7')	0·2543 (5)	0·5579 (3)	0·5528 (4)
C(8)	0·2735 (4)	0·0479 (2)	0·3891 (3)	C(8')	0·2432 (4)	0·4727 (3)	0·4865 (3)
C(9)	0·1827 (4)	0·1392 (3)	0·4121 (3)	C(9')	0·3017 (5)	0·3706 (3)	0·5120 (3)
O(10)	0·0483 (3)	0·1507 (2)	0·4363 (3)	O(10')	0·3716 (4)	0·3365 (2)	0·5940 (3)
O(11)	0·5503 (3)	0·2374 (2)	0·3493 (2)	O(11')	0·1244 (4)	0·3417 (2)	0·2356 (3)
H(4)	0·647 (5)	0·024 (3)	0·332 (3)	H(4')	0·036 (5)	0·554 (3)	0·253 (4)
H(5)	0·567 (5)	−0·143 (3)	0·332 (3)	H(5')	0·063 (5)	0·713 (5)	0·366 (4)
H(6)	0·303 (5)	−0·193 (4)	0·371 (4)	H(6')	0·193 (4)	0·708 (4)	0·555 (3)
H(7)	0·121 (6)	−0·067 (3)	0·406 (4)	H(7')	0·311 (6)	0·553 (4)	0·633 (5)

The atomic coordinates are listed in Table 1. Table 2 gives bond distances and angles. Fig. 1 shows an *ORTEP* plot (Johnson, 1971) of the dimer with the numbering of the atoms.*

Discussion. The Winkler & Dunitz (1971) parameters were calculated and showed no significant deformations in the amide groups. As far as the torsion angles are concerned in the five-membered ring, we have calculated the maximum angle of torsion φ_m (Altona, Geise & Romers, 1968) for the title compound and for nine others (Allen & Trotter, 1971; Andreetti, Bocelli, Coghi & Sgarabotto, 1974, 1975; Andreetti, Bocelli & Sgarabotto, 1973, 1977; Glass & Johnson, 1976; Michel & Durant, 1975; Petersen, 1969) and we think that all the rings could be considered planar; O(10) and O(11) deviate just −0·02 and −0·01 Å respectively, from the five-membered-ring plane in the present work. The two halves of the dimer are twisted through 78°.

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* Lists of structure factors, anisotropic thermal parameters and a comparison of bond distances in some phthalimide groups have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 33785 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
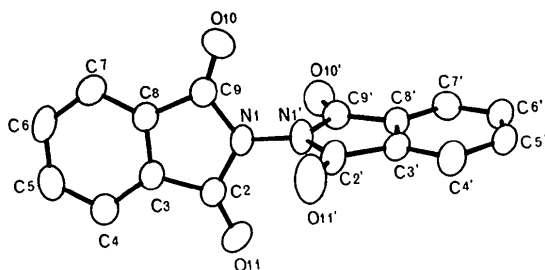

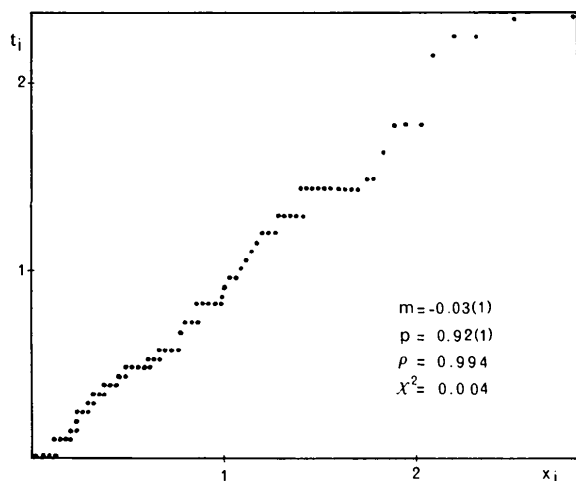Fig. 1. An *ORTEP* drawing of the dimer showing the numbering of the atoms.

m = -0.03(1)
p = 0.92(1)
P = 0.994
X^2 = 0.004

Fig. 2. Half-normal probability plot.

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

N(1)—C(2)	1·412 (5)		N(1')—C(2')	1·400 (5)
N(1)—C(9)	1·410 (5)		N(1')—C(9')	1·414 (5)
C(2)—C(3)	1·475 (5)		C(2')—C(3')	1·478 (5)
C(2)—O(11)	1·194 (4)		C(2')—O(11')	1·194 (5)
C(3)—C(4)	1·381 (5)		C(3')—C(4')	1·382 (5)
C(3)—C(8)	1·387 (5)		C(3')—C(8')	1·387 (5)
C(4)—C(5)	1·376 (6)		C(4')—C(5')	1·388 (6)
C(5)—C(6)	1·389 (6)		C(5')—C(6')	1·374 (7)
C(6)—C(7)	1·382 (6)		C(6')—C(7')	1·387 (6)
C(7)—C(8)	1·375 (5)		C(7')—C(8')	1·378 (5)
C(8)—C(9)	1·477 (5)		C(8')—C(9')	1·480 (5)
C(9)—O(10)	1·196 (5)		C(9')—O(10')	1·187 (5)
N(1)—N(1')	1·372 (4)			

C(2)—N(1)—C(9)	113·3 (3)		C(2')—N(1')—C(9')	114·0 (3)
C(2)—N(1)—N(1')	123·1 (3)		C(2')—N(1')—N(1)	123·2 (3)
C(9)—N(1)—N(1')	123·6 (3)		C(9')—N(1')—N(1)	122·7 (3)
N(1)—C(2)—C(3)	104·1 (3)		N(1')—C(2')—C(3')	104·0 (3)
N(1)—C(2)—O(11)	124·3 (3)		N(1')—C(2')—O(11')	125·0 (3)
C(3)—C(2)—O(11)	131·6 (3)		C(3')—C(2')—O(11')	131·0 (4)
C(2)—C(3)—C(4)	129·3 (3)		C(2')—C(3')—C(4')	129·3 (4)
C(2)—C(3)—C(8)	109·3 (3)		C(2')—C(3')—C(8')	109·3 (3)
C(4)—C(3)—C(8)	121·4 (3)		C(4')—C(3')—C(8')	121·4 (3)
C(3)—C(4)—C(5)	117·3 (4)		C(3')—C(4')—C(5')	117·1 (4)
C(4)—C(5)—C(6)	121·5 (4)		C(4')—C(5')—C(6')	121·5 (4)
C(5)—C(6)—C(7)	121·0 (4)		C(5')—C(6')—C(7')	121·2 (4)
C(6)—C(7)—C(8)	117·6 (4)		C(6')—C(7')—C(8')	117·6 (4)
C(7)—C(8)—C(3)	121·2 (3)		C(7')—C(8')—C(3')	121·1 (3)
C(7)—C(8)—C(9)	129·7 (3)		C(7')—C(8')—C(9')	129·9 (3)
C(9)—C(8)—C(3)	109·1 (3)		C(9')—C(8')—C(3')	109·1 (3)
C(8)—C(9)—N(1)	104·1 (3)		C(8')—C(9')—N(1')	103·6 (3)
C(8)—C(9)—O(10)	131·3 (4)		C(8')—C(9')—O(10')	131·8 (4)
N(1)—C(9)—O(10)	124·6 (4)		N(1')—C(9')—O(10')	124·6 (3)

The dimer has been internally checked by a half-normal probability plot (Fig. 2) (Abrahams & Keve, 1971). We have considered the molecule to be divided into four parts and we have compared the intramolecular distances of one part with each other. The parameters describing the fitting of the plot with respect to a normal variate are shown in Fig. 2, suggesting pseudo 222 symmetry. A table comparing the structure with other compounds containing the phthalimide group has been deposited. The most significant differences are found for the five-membered ring.

The angles C(6)—C(7)—C(8) and C(3)—C(4)—C(5) are reduced to 117·6 and 117·3°. This has been observed in other compounds where the benzene ring is joined to a smaller ring. The mean bond length in the aromatic ring is 1·382 Å, and for the C—H bonds, 1·00 Å.

The packing is determined by van der Waals forces.

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The Structure of Diisopropylammonium Diisopropyldithiocarbamate

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Abstract. $\{[(CH_3)_2CH]_2NH_2\}^+\{[(CH_3)_2CH]_2NCS_2\}^-$, monoclinic, $P2_1/c$, $a = 9.032$ (1), $b = 11.525$ (1), $c = 16.638$ (1) Å, $\beta = 98.53$ (1)°, $Z = 4$, $V = 1712.6$ Å^3, $D_m = 1.078$, $D_x = 1.080$ g cm^{-3}, m.p. 96–97°C. The structure has been refined to $R(F^2) = 0.065$ for 2683 reflexions and 274 parameters. Two cations and two anions are linked to form a centrosymmetric dimer. Each molecular ion participates in three intermolecular H⋯S hydrogen bonds between the ammonium protons and the dithiocarbamate S atoms, at 2.41 (3), 2.52 (3) and 2.85 (3) Å.

Introduction. This investigation closely follows the structural study of dimethylammonium dimethyldithiocarbamate (Wahlberg, 1978), (M).

The compound was synthesized in benzene from diisopropylamine and CS_2 and was recrystallized from CS_2. The plate-shaped transparent crystals were pale yellow. They sublimed markedly from *ca* 20°C below the melting point. The space-group assumption was made from systematically absent reflexions among $h0l$ for odd l and $0k0$ for odd k. The unit-cell parameters were based on 50 lines from a Guinier–Hägg powder photograph. The internal standard was CoP_3 ($a = 7.70778$ Å) and the radiation used was Cr $K\alpha_1$.

A crystal was sealed in a thin-walled glass capillary. Approximate distances between pairs of surfaces were: (100) 0.04 mm, (001) 0.27 mm, (011) 0.22 mm and (01̄1) 0.22 mm. A Stoe–Philips four-circle PDP 8/I computer-controlled diffractometer with graphite-monochromatized Mo $K\alpha$ radiation was used for data collection. A unique set of intensities was measured in one quadrant of reciprocal space, $4.5 < 2\theta < 48°$. The count rate was reduced by a factor 2^n, $0 \le n \le 5$. The reflexions were scanned in the ω–2θ mode. The longest scan time was 160 s. The background was measured on each side of the reflexion for 25 s. Three standard reflexions showed stability of the crystal and the diffractometer system. Corrections were applied for background and for Lorentz, polarization and absorption effects [μ(Mo $K\alpha$) = 2.84 cm^{-1}]. The transmission factor varied from 0.94 to 0.99. 3458 intensities were reduced to 2683 structure factors with $F_m^2 > -2.40\sigma(F^2)$ and $0.060 < (\sin\theta)/\lambda < 0.573$ Å^{-1}.

The structure was solved by direct methods (Long, 1965). The positions of the H atoms were obtained from a difference synthesis. In the last cycle of full-matrix least-squares refinement, 274 independent parameters were varied and all the structure factors were included. The thermal parameters were aniso-