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The Structure of *N*-(2-Methylphenyl)-3,6-dithiacyclohexene-1,2-dicarboximide

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Abstract. $C_{13}H_{11}NO_2S_2$, orthorhombic, *Pbca*, $a = 16{\cdot}360\,(4)$, $b = 19{\cdot}676\,(7)$, $c = 8{\cdot}041\,(3)$ Å, $V = 2588{\cdot}1$ Å^3, $Z = 8$, $D_x = 1{\cdot}42$, $D_m = 1{\cdot}40$ Mg m^{-3}, $F(000) = 1152$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 3{\cdot}55$ mm^{-1}. The structure has been solved by direct methods with the 1506 independent reflections with $I > 1{\cdot}96\sigma(I)$. The refinement by full-matrix least squares with anisotropic temperature factors gave a conventional $R = 0{\cdot}059$. The 3,6-dithiacyclohexene ring has a deformed half-chair conformation with the asymmetry parameter $\Delta C_2(1,2) = 11{\cdot}7°$. The imide ring is not perfectly planar. The dihedral angle between phenyl and imide rings is 75·0 (6)°. The differences between the C(sp^3)–S (1·807 Å av.) and C(sp^3)–S (1·729 Å av.) bond lengths are smaller than normal.

Introduction. This work is part of a series of X-ray diffraction investigations of N-substituted derivatives of 3,6-dithiacyclohexene-1,2-dicarboximide which is directed towards a better understanding of the influence of the N substituent on the spatial shape of the molecule, the conformation of the dithiacyclohexene ring, the hybridization of the N-atom orbitals and the planarity of the central part of the molecule (Bukowska-Strzyżewska & Pniewska, 1979*a*,*b*; Dobrowolska & Bukowska-Strzyżewska, 1980). This paper describes the molecular structure of *N*-(2-methylphenyl)-3,6-dithiacyclohexene-1,2-dicarboximide.

The molecule (I) and its resonance forms (II and III) are shown in Fig. 1. The compound was synthesized by Hahn & Rybczyński (1971, 1976) in order to investigate the influence of the N substituent on the pharmacological activity of this group of heterocyclic compounds.

The crystals were obtained from methanol solution. The space group was determined from Weissenberg photographs. The cell parameters and intensities were measured with a single crystal of approximate dimensions 0·15 × 0·15 × 0·30 mm on a Syntex $P2_1$ single-crystal diffractometer. Intensity data were collected to $\theta = 60°$ by the $\theta/2\theta$ scan method using monochromatized X-rays. The unobserved reflections were omitted from the structure analysis. Correction for absorption was neglected in view of the low μr value for the crystal ($\mu = 3{\cdot}55$ mm^{-1}). The atomic scattering factors were taken from Doyle & Turner (1968).

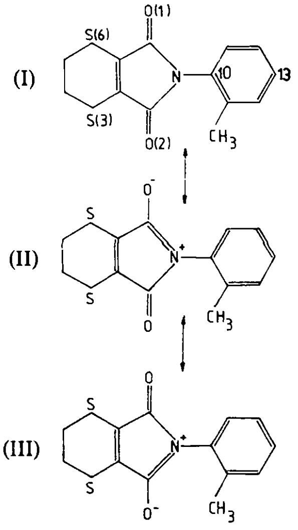

Fig. 1. Molecule (I) and its resonance forms (II and III).

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Table 1. *Atomic parameters for N-*(2-*methylphenyl*)-3,6-*dithiacyclohexene-*1,2-*dicarboximide with e.s.d.'s in parentheses* ($x,y,z \times 10^5$ *for* S, $\times 10^4$ *for* C, N, O *and* $\times 10^3$ *for* H *atoms*)

	x	y	z	$B_{\rm iso}$ (Å^2)
S(3)	35416 (9)	29887 (9)	634 (17)	4·11 (6)
S(6)	50861 (7)	35709 (8)	28506 (18)	3·84 (6)
O(2)	1980 (2)	3292 (2)	2281 (4)	4·0 (2)
O(1)	3955 (2)	4069 (2)	5783 (5)	5·5 (2)
N(9)	2791 (2)	3762 (2)	4318 (4)	2·9 (2)
C(4)	4573 (3)	3263 (3)	−386 (6)	4·3 (2)
C(5)	5191 (3)	3100 (3)	935 (7)	4·3 (3)
C(2)	3455 (3)	3347 (2)	2028 (5)	2·8 (3)
C(1)	4037 (3)	3573 (3)	3040 (6)	3·0 (2)
C(8)	2642 (3)	3452 (2)	2813 (6)	3·0 (5)
C(7)	3634 (3)	3942 (3)	4573 (7)	3·7 (2)
C(10)	2164 (3)	3950 (3)	5509 (6)	3·1 (2)
C(11)	2058 (3)	3539 (3)	6908 (6)	4·1 (2)
C(12)	1464 (4)	3710 (3)	8048 (7)	4·8 (2)
C(13)	982 (3)	4266 (4)	7778 (8)	4·9 (3)
C(14)	1096 (3)	4661 (3)	6398 (7)	4·5 (3)
C(15)	1690 (3)	4513 (3)	5227 (7)	3·7 (3)
C(16)	1801 (4)	4955 (3)	3700 (8)	6·1 (3)
H(41)	457	378	−54	4·3
H(42)	475	306	−148	4·3
H(51)	513	259	121	4·3
H(52)	576	316	49	4·3
H(111)	243	311	708	4·1
H(121)	138	341	908	4·8
H(131)	55	439	865	4·9
H(141)	71	506	621	4·5

The structure was solved by direct methods with the *MULTAN* program (Germain, Main & Woolfson, 1971). The refinement of the structure was carried out by the full-matrix least-squares method. H atoms were revealed in a difference Fourier synthesis. The parameters of the H atoms were not refined. Unit weight was assigned to each reflection. The final $R = 0\cdot059$. The atomic parameters are given in Table 1.*

Discussion. Bond lengths and angles are listed in Table 2.

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

S(3)–C(2)	1·735 (5)	C(8)–N(9)	1·377 (6)
S(3)–C(4)	1·808 (5)	C(8)–O(2)	1·206 (6)
S(6)–C(1)	1·722 (4)	C(10)–C(11)	1·396 (7)
S(6)–C(5)	1·806 (6)	C(10)–C(15)	1·372 (7)
C(1)–C(2)	1·329 (6)	C(11)–C(12)	1·379 (8)
C(1)–C(7)	1·496 (7)	C(12)–C(13)	1·366 (9)
C(2)–C(8)	1·488 (6)	C(13)–C(14)	1·365 (9)
C(4)–C(5)	1·501 (7)	C(14)–C(15)	1·395 (8)
C(7)–N(9)	1·403 (6)	C(15)–C(16)	1·515 (8)
C(7)–O(1)	1·193 (7)	N(9)–C(10)	1·451 (6)

C(2)–S(3)–C(4)	97·8 (2)	C(2)–C(8)–N(9)	106·1 (4)
C(1)–S(6)–C(5)	99·9 (2)	C(8)–N(9)–C(7)	110·6 (4)
C(2)–C(1)–S(6)	131·2 (4)	C(8)–N(9)–C(10)	124·6 (4)
S(6)–C(1)–C(7)	120·9 (4)	C(7)–N(9)–C(10)	124·8 (4)
C(2)–C(1)–C(7)	107·9 (4)	N(9)–C(10)–C(11)	118·2 (4)
S(3)–C(2)–C(1)	129·4 (4)	C(11)–C(10)–C(15)	122·1 (5)
S(3)–C(2)–C(8)	121·0 (3)	N(9)–C(10)–C(15)	119·7 (4)
C(1)–C(2)–C(8)	109·5 (4)	C(10)–C(11)–C(12)	118·8 (5)
S(3)–C(4)–C(5)	115·0 (4)	C(11)–C(12)–C(13)	119·8 (6)
S(6)–C(5)–C(4)	115·4 (4)	C(12)–C(13)–C(14)	120·9 (6)
C(1)–C(7)–O(1)	127·7 (5)	C(13)–C(14)–C(15)	121·2 (5)
C(1)–C(7)–N(9)	105·9 (4)	C(10)–C(15)–C(14)	117·2 (5)
O(1)–C(7)–N(9)	126·4 (5)	C(16)–C(15)–C(14)	120·8 (5)
O(2)–C(8)–N(9)	125·9 (4)	C(10)–C(15)–C(16)	122·0 (5)
C(2)–C(8)–O(2)	128·0 (4)		

The 3,6-dithiacyclohexene ring has an imperfect half-chair conformation. The torsion angles φ, $|\varphi|_{\rm av}$ and asymmetry parameters (Duax & Norton, 1975) are given in Fig. 2. The asymmetry parameter in relation to the twofold axis is $\Delta C_2(1,2) = 11\cdot7°$, and in relation to the 1–4 mirror plane $\Delta C_s(1,4) = 17\cdot5°$. The average S–C($sp^3$) bond length (1·807 Å) is in good agreement with the accepted value of 1·812 Å (Pauling, 1960) and slightly lower than in other 3,6-dithiacyclohexene rings: 1·823 (2) Å (Bukowska-Strzyżewska & Pniewska, 1979*b*), 1·823 (4) Å (Kirfel, Will & Fickentscher, 1975), 1·819 (7) Å (Bukowska-Strzyżewska & Pniewska, 1979*a*). The average bond lengths S–C(sp^2) = 1·729, C(sp^2)–C(sp^2) = 1·329 (6) and C(sp^3)–C(sp^3) = 1·501 (7) Å are in good agreement with values found in the rings of the structures mentioned above.

The imide ring is not perfectly planar. Table 3 gives the equation of its best plane, the deviations of atoms from the plane and χ^2 and p values. It is assumed that if $p < 0\cdot01$ the set of atoms defining the plane is not rigorously planar within experimental error. The three N–C bonds are practically coplanar, but the C(10)–C(13) line of the phenyl ring deviates considerably from the imide ring plane. The C–N bond lengths [1·403 (6) and 1·377 (6) Å] are in good agreement with those in *N*-(2-chlorophenyl)-3,6-dithiacyclohexene-1,2-dicarboximide [average 1·385 (7) Å; Bukowska-Strzyżewska & Pniewska, 1979*a*]. If a C(sp^3)–N(sp^3) single-bond distance is taken as 1·47 Å and a C(sp^2)-N(sp^2) double-bond distance as 1·29 Å (Pauling, 1960), and if a linear relationship between

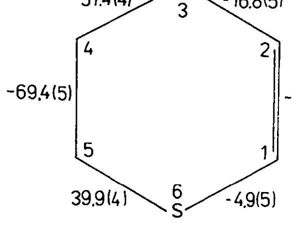

Fig. 2. The torsion angles (°). [$\Delta C_2(1,2) = 11\cdot7°$; $|\varphi|_{\rm av} = 30\cdot9°$.]

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35356 (38 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 3. *Deviations* (Å) *of the atoms from the least-squares planes*

The imide ring

$-0{\cdot}0533X + 0{\cdot}9015Y - 0{\cdot}4295Z = 4{\cdot}9315$

C(1)	0·004 (5)	*O(2)	−0·052 (4)
C(2)	0·003 (5)	*C(4)	0·591 (6)
C(7)	−0·013 (6)	*C(5)	−0·209 (6)
C(8)	−0·010 (5)	*C(10)	−0·016 (5)
N(9)	0·008 (4)	*C(13)	−0·136 (7)
*S(3)	0·039 (2)	$\chi^2 = 13{\cdot}69$	
*S(6)	−0·025 (2)	$p < 0{\cdot}01$	
*O(1)	−0·057 (5)		

The phenyl ring

$-0{\cdot}6554X - 0{\cdot}5650Y - 0{\cdot}5013Z = -8{\cdot}9313$

C(10)	0·000 (5)	C(15)	−0·005 (5)
C(11)	0·005 (5)	*N(9)	0·016 (4)
C(12)	−0·007 (6)		
C(13)	0·000 (6)	$\chi^2 = 4{\cdot}03$	
C(14)	0·006 (6)	$0{\cdot}10 < p < 0{\cdot}50$	

* Not included in the calculation of the plane.

bond length and bond order is assumed, the present C—N bonds possess 35 and 45% double-bond character. This may be due to the different contribution of resonance structures (II) and (III) (Fig. 1). The C—O bond lengths correspond to this scheme, the shorter C(7)—O(1) = 1·193 (7) Å bond is adjacent to the longer C(7)—N(9) = 1·403 (6) Å bond and the longer C(8)—O(2) = 1·206 Å is adjacent to the shorter C(8)—N(9) = 1·377 (6) Å. The phenyl ring is perfectly planar within the observed errors. The dihedral angle between the phenyl and imide rings is 75°. The maximum difference between C—H bond lengths is 0·035 Å, with a mean value of 1·02 Å.

The only noteworthy intermolecular contact is S(6)···O(2) = 3·149 (3) Å between molecules related by the *a* glide.

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Structure of 1,2,2,6,6-Pentamethyl-4-vinyl-4-piperidinol*

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Abstract. $C_{12}H_{23}NO$, orthorhombic, $P2_12_12_1$, $a = 7{\cdot}860$ (2), $b = 10{\cdot}935$ (3), $c = 14{\cdot}407$ (3) Å, $Z = 4$, $V = 1238{\cdot}3$ Å³, $D_x = 1{\cdot}06$ Mg m⁻³, $F(000) = 440$, $\mu r(\mathrm{Cu}\,K\alpha) = 0{\cdot}15$. 1514 independent reflexions were measured on a CAD-4 diffractometer. The structure was solved by direct methods and refined to $R = 0{\cdot}054$ for 999 reflexions with $I > 3\sigma(I)$. The piperidine ring adopts a chair conformation with an axial hydroxyl group. The formation of an intramolecular =C—H···O bond is suggested. Between molecules there are only van der Waals contacts.

Introduction. In molecules with bulky ring substituents, steric interactions strongly influence the conformation of the molecular skeleton. 2,2,6,6-

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* The Conformation of the Piperidine Ring. IV.

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