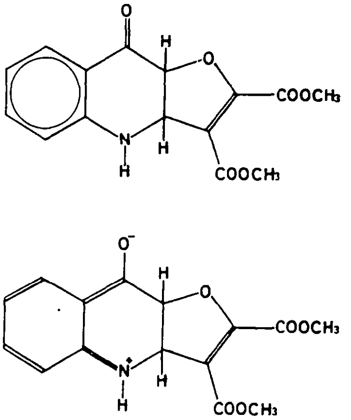

Fig. 4. Possible mesomeric forms. The second form is an intramolecular charge-transfer form.

the case of cyclobutenes (Davis & Neckers, 1978). Further work in this respect is in progress.

References

ANSELL, G. B., MOORE, D. W. & NIELSEN, A. T. (1971). *J. Chem. Soc. B*, **23**, 2376–2382.
CELIKEL, R., GEDDES, A. J. & SHELDRICK, B. (1976). *Cryst. Struct. Commun.* **5**, 43–46.
DAVIS, P. D. & NECKERS, D. C. (1978). *Tetrahedron Lett.* pp. 2979–2982.
GERMAIN, G., MAIN, P. & WOOLFSON, M. M. (1971). *Acta Cryst.* A**27**, 368–376.
International Tables for X-ray Crystallography (1974). Vol. IV, pp. 72–98. Birmingham: Kynoch Press.
ISHIGURO, Y., FUNAKOSHI, K., SAEKI, S., HAMANA, M. & UEDA, I. (1980). *Heterocycles*, **14**, 179–184.
KING, T. J., HASTINGS, J. S. & HELLER, H. G. (1975). *J. Chem. Soc. Perkin Trans.* 1, pp. 1455–1457.
PAULING, L. (1960). *The Nature of the Chemical Bond*, 3rd ed. Ithaca: Cornell Univ. Press.
SAKURAI, T., IWASAKI, H., WATANABE, Y., KOBAYASHI, K., BANDO, Y. & NAKAMICHI, Y. (1974). *Rikagaku Kenkyusho Hokoku*, **50**, 75–91 (in Japanese).

Acta Cryst. (1980). B**36**, 3167–3169

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

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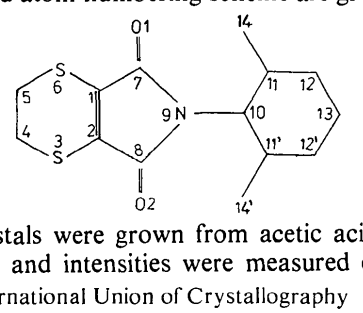
(*Received* 5 *February* 1980; *accepted* 19 *July* 1980)

Abstract. $C_{14}H_{13}NO_2S_2$, orthorhombic, *Pbcm*, $a = 9{\cdot}920\,(3)$, $b = 16{\cdot}380\,(7)$, $c = 8{\cdot}712\,(1)$ Å, $V = 1415{\cdot}6$ Å^3, $Z = 4$, $D_x = 1{\cdot}37$, $D_m = 1{\cdot}35$ Mg m^{-3}, $F(000) = 608$, $\lambda(\mathrm{Cu}\ K\alpha) = 1{\cdot}5418$ Å, $\mu(\mathrm{Cu}\ K\alpha) = 3{\cdot}27$ mm^{-1}. The structure has been solved by direct methods with 799 independent reflections having $I > 1{\cdot}96\ \sigma(I)$ Full-matrix least-squares refinement with anisotropic temperature factors gave a conventional $R = 0{\cdot}070$. The molecules occupy the special position on the mirror plane in the unit cell. The S, C(1) and C(2) atoms and 1,2-dicarboximide ring are on the mirror plane. The C(4) and C(5) atoms of the 3,6-dithiacyclohexene ring deviate statistically from the plane. The phenyl ring is perpendicular to the imide ring.

Introduction. The present structural investigation was undertaken as part of a study of the geometries and conformations of new heterocyclic compounds revealing high pharmacological activity (Bukowska-Strzyżewska, Dobrowolska & Pniewska, 1978; Bukowska-Strzyżewska & Pniewska, 1979*a*,*b*; Dobrowolska & Bukowska-Strzyżewska, 1980*a*,*b*,*c*; Bukowska-Strzyżewska, Dobrowolska & Głowiak, 1981). A series of *N*-substituted derivatives of 3,6-dithiacyclohexene-1,2-dicarboximide were synthesized in the Department of Chemistry of the University of Łódź (Hahn & Rybczyński, 1971, 1976). This paper describes the molecular structure of *N*-(2,6-dimethylphenyl)-3,6-dithiacyclohexene-1,2-dicarboximide. The formula and atom-numbering scheme are given below:



The crystals were grown from acetic acid. The cell parameters and intensities were measured on a single

* Alternative name: *N*-(2,6-dimethylphenyl)-5,6-dihydro-1,4-dithiin-2,3-dicarboximide.

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crystal at room temperature, on an automatic Syntex $P2_1$ single-crystal diffractometer. Intensity data were collected by the θ–2θ scan technique at a scan rate of 2·0–20·0° min^{-1} with monochromatized Cu $K\alpha$ radiation. Absorption corrections were ignored. The atomic scattering factors were taken from Doyle & Turner (1968). The structure was solved by direct methods with *MULTAN* (German, Main & Woolfson, 1971). The refinement of the structure was carried out by the full-least-squares method.

The S, O and disordered C(4) and C(5) atoms show relatively high B_{33} temperature factors (9–17 Å^2). The z coordinates of C(4) and C(5), deviating statistically from the mirror plane, were refined with an occupancy factor of 0·5. The H atoms were not located. The final R value for all observed reflections was 0·070. The final atomic parameters are given in Table 1.*

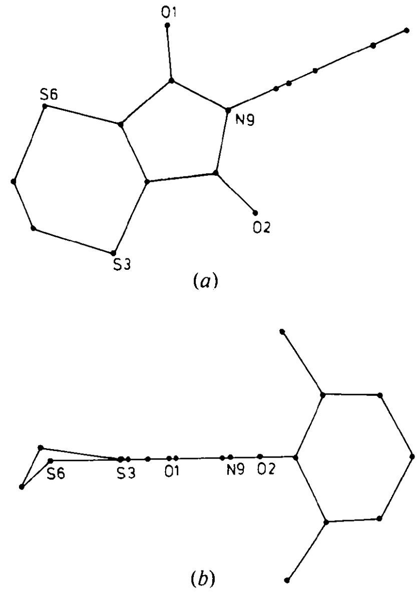

Fig. 1. Views of the molecule projected: (*a*) along the *c* axis, (*b*) along the *a* axis.

Discussion. Views of the molecule projected along the *c* and *a* axes are shown in Fig. 1. Bond lengths and angles with estimated standard deviations are listed in Table 2.

In spite of the nonplanarity of the dithiacyclohexene ring the molecules occupy the special position 4(*d*). C(4) and C(5) deviate statistically from the mirror plane by ±0·350 (32) and ±0·520 (18) Å. The accuracy of the localization of these atoms is considerably low because of the serious correlations between B_{33} and z (the average B_{33} of these atoms is about 12 Å^2 which corresponds to a r.m.s. amplitude of about 0·4 Å parallel to z). The 3,6-dithiacyclohexene ring has the half-chair conformation. The deformation of the ring in relation to the twofold-axis symmetry, calculated as $\Delta C_2 = [\sum_{i=1}^{i=2} (\varphi_i - \varphi'_i)/2]^{1/2}$, is 5·8°. The C($sp^2$)–S bonds lengths are identical [1·724 (8) Å] and agree with the values found in other dithiacyclohexene rings. Also the C(sp^3)–S bonds lengths are almost identical [1·849 (12) and 1·843 (13) Å] and consistent with

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35511 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atomic parameters (fractional coordinates* $\times 10^4$*) for N-(2,6-dimethylphenyl)-3,6-dithiacyclohexene-1,2-dicarboximide with e.s.d.'s in parentheses*

	x	y	z	B_{iso} (Å^2)
S(3)	4235 (2)	2938 (1)	2500	4·80 (6)
S(6)	7467 (3)	2004 (1)	2500	5·34 (6)
O(1)	9285 (6)	3603 (3)	2500	5·21 (21)
O(2)	5159 (6)	4785 (3)	2500	5·40 (23)
C(1)	7021 (8)	3021 (4)	2500	3·62 (24)
C(2)	5825 (8)	3363 (4)	2500	3·32 (23)
C(4)	4712 (13)	1868 (6)	2901 (36)	5·09 (32)
C(5)	5820 (13)	1593 (7)	1903 (21)	5·66 (33)
C(7)	8094 (8)	3669 (4)	2500	3·68 (20)
C(8)	6020 (8)	4266 (5)	2500	3·97 (21)
C(10)	8010 (8)	5209 (4)	2500	3·31 (20)
C(11)	8261 (5)	5568 (3)	1064 (9)	4·42 (20)
C(12)	8801 (6)	6381 (4)	1147 (10)	5·37 (25)
C(13)	9040 (10)	6763 (6)	2500	5·44 (25)
C(14)	7991 (8)	5153 (4)	−434 (8)	6·52 (22)
N(9)	7400 (6)	4404 (3)	2500	3·39 (21)

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

S(3)–C(2)	1·724 (8)	C(2)–S(3)–C(4)	98·5 (7)
S(3)–C(4)	1·849 (12)	C(1)–S(6)–C(5)	97·2 (5)
S(6)–C(1)	1·724 (7)	C(2)–C(1)–S(6)	130·1 (5)
S(6)–C(5)	1·843 (13)	S(6)–C(1)–C(7)	120·1 (4)
C(1)–C(2)	1·312 (11)	C(2)–C(1)–C(7)	109·8 (5)
C(1)–C(7)	1·503 (11)	S(3)–C(2)–C(1)	131·0 (5)
C(2)–C(8)	1·493 (10)	S(3)–C(2)–C(8)	121·2 (5)
C(4)–C(5)	1·473 (26)	C(1)–C(2)–C(8)	107·8 (5)
C(7)–N(9)	1·387 (9)	S(3)–C(4)–C(5)	111·7 (13)
C(7)–O(1)	1·187 (10)	S(6)–C(5)–C(4)	112·5 (1)
C(8)–N(9)	1·387 (10)	C(1)–C(7)–O(1)	129·8 (6)
C(8)–O(2)	1·205 (10)	C(1)–C(7)–N(9)	105·2 (5)
C(10)–C(11)	1·405 (8)	O(1)–C(7)–N(9)	125·0 (6)
C(10)–N(9)	1·451 (9)	O(2)–C(8)–N(9)	125·8 (6)
C(11)–C(12)	1·438 (8)	C(2)–C(8)–O(2)	127·4 (6)
C(11)–C(14)	1·496 (10)	C(2)–C(8)–N(9)	106·8 (5)
C(12)–C(13)	1·355 (9)	C(8)–N(9)–C(7)	110·4 (5)
		C(8)–N(9)–C(10)	124·0 (5)
		C(7)–N(9)–C(10)	125·6 (5)
		N(9)–C(10)–C(11)	117·0 (5)
		C(11)–C(10)–C(11')	125·9 (6)
		C(10)–C(11)–C(12)	114·2 (6)
		C(10)–C(11)–C(14)	123·7 (6)
		C(12)–C(11)–C(14)	122·1 (6)
		C(11)–C(12)–C(13)	122·4 (6)
		C(12)–C(13)–C(12')	120·9 (7)

literature data. The slight shortening of the $C(sp^3)-C(sp^3)$ bond [1·473 (26) Å] is probably due to the large B_{33} values of these atoms.

The imide ring is ideally planar. Typical and identical values of the $N-C(sp^2)$ bond lengths [1·387 (9) Å] and almost identical (within the 2σ range) $C(sp^2)-O$ bond lengths are observed. The symmetrical π delocalization within C=O and C—N bonds is due to the ideal planarity of the ring. The phenyl ring perpendicular to the imide ring is ideally planar. The average C—C bond length [1·399 (8) Å] is typical for the phenyl ring and the average C—C—C bond angle is 120·0 (6)°. The bond angle at C(11) substituted by the methyl group is decreased to the value 114·1 (6)° and that at C(10) substituted by the imide ring is increased to the value 125·9 (6)°.

The molecular packing is comparatively loose. The coefficient of molecular packing expressed according to Kitaigorodsky (1973) as $w = (\sum_i \Delta V_i)/V$ is only 0·69; ($\sum_i \Delta V_i$ is the volume of molecules, limited by the van der Waals radii, contained in the unit cell and V is the volume of the unit cell). Unusually long distances along the z axis are observed between all atoms situated on the mirror planes. All these distances exceed $\frac{1}{2}c =$ 4·356 Å. These long intermolecular distances result from the perpendicular position of the dimethylphenyl ring with respect to the mirror plane. The intramolecular distance C(14)···C(14′) determines the value of c as 8·712 (1) Å and it is this value that determines the long intermolecular distances between atoms on the mirror planes. The shortest intermolecular distance along the z axis is 3·836 (18) Å [between C(2) and C(5′) – the latter deviating statistically from the mirror plane]. The very loose packing of the dithiacyclohexene and imide rings results in strong thermal oscillations of C(4), C(5), S and O along the z axis. Fig. 2 shows the packing.

This work has been financially supported by the Polish Academy of Science, problem MR. I-9.

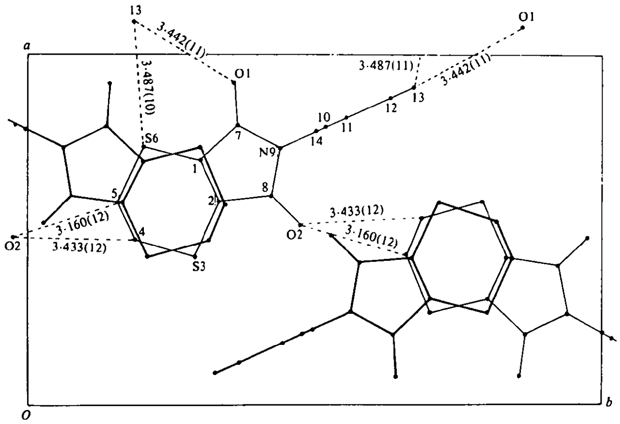

Fig. 2. Molecular packing. (Distances in Å.)

References

Bukowska-Strzyżewska, M., Dobrowolska, W. & Głowiak, T. (1981). *Acta Cryst.* B**37**. To be published.

Bukowska-Strzyżewska, M., Dobrowolska, W. & Pniewska, B. (1978). *Pol. J. Chem.* **52**, 1843–1845.

Bukowska-Strzyżewska, M. & Pniewska, B. (1979*a*). *Acta Cryst.* B**35**, 640–643.

Bukowska-Strzyżewska, M. & Pniewska, B. (1979*b*). *Acta Cryst.* B**35**, 633–639.

Dobrowolska, W. & Bukowska-Strzyżewska, M. (1980*a*). *Acta Cryst.* B**36**, 317–321.

Dobrowolska, W. & Bukowska-Strzyżewska, M. (1980*b*). *Acta Cryst.* B**36**, 462–464.

Dobrowolska, W. & Bukowska-Strzyżewska, M. (1980*c*). *Acta Cryst.* B**36**, 890–893.

Doyle, P. A. & Turner, P. S. (1968). *Acta Cryst.* A**24**, 390–397.

Germain, G., Main, P. & Woolfson, M. M. (1971). *Acta Cryst.* A**27**, 368–376.

Hahn, W. E. & Rybczyński, B. (1971). *Soc. Sci. Lodz. Acta Chim.* **16**, 123–128.

Hahn, W. E. & Rybczyński, B. (1976). *Rocz. Chem.* **50**, 1523–1533.

Kitaigorodsky, A. I. (1973). *Molecular Crystals and Molecules*. London: Academic Press.

Acta Cryst. (1980). B**36**, 3169–3172

The Structure of 2-Oxo-2′-thioxobis(5,5-dimethyl-1,3,2-dioxaphosphorinanyl) Oxide*

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(*Received* 10 *December* 1979; *accepted* 21 *July* 1980)

Abstract. $C_{10}H_{20}O_6P_2S$, orthorhombic, *Pbca*, $a =$ 27·949 (5), $b =$ 9·893 (2), $c =$ 11·519 (3) Å, $V =$ 3185·0 Å^3, $D_x =$ 1·37, $D_m =$ 1·38 Mg m^{-3}, $Z =$ 8, $F(000) =$ 1392, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 3·72 mm^{-1}. The crystals are isostructural with the analogous pyrophosphate [Bukowska-Strzyżewska & Dobrowolska (1978). *Acta Cryst.* B**34**, 1357–1360]. Disorder of the S(2) and O(12) atoms was observed, with the P=S and P=O bonds statistically inter-

* 2-(5,5-Dimethyl-2-thioxo-1,3,2-dioxaphosphorinan-2-yloxy)-5,5-dimethyl-1,3,2-dioxaphosphorinane 2-oxide.

0567-7408/80/123169-04$01.00