indicated in Fig. 3, which shows the central core. The Si atom arrangement is that of possibility (II) in Fig. 1.

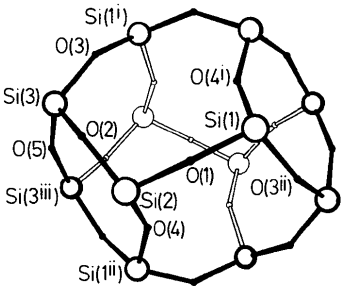

Fig. 3. The $Si_{12}O_{18}$ core, showing the numbering scheme. For symmetry operators, see Table 2.

Bond lengths and angles are similar to those in $(PhSi)_8O_{12}$ (Hossain, Hursthouse & Malik, 1979).

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Structure of Dimethyl 3a,4,9,9a-Tetrahydro-9-oxo-*cis*-furo-[3,2-*b*]quinoline-2,3-dicarboxylate

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Abstract. $C_{15}H_{13}NO_6$, monoclinic, $P2_1/c$, $Z = 4$, $a = 11{\cdot}635$ (4), $b = 10{\cdot}442$ (3), $c = 12{\cdot}347$ (3) Å, $\beta = 108{\cdot}81$ (2)°, $D_m = 1{\cdot}417$, $D_x = 1{\cdot}422$ Mg m^{-3}. The structure was solved by the direct method; block-diagonal least-squares refinement led to a final R of 0·047 for 2198 observed reflexions. Molecules are held together by van der Waals interactions and strong NH···O hydrogen bonds. The furan ring has abnormal bond angles, indicating distortion caused by the cycloaddition. The pyridine ring in the quinoline moiety has a conformation of a slightly creased hinge.

Introduction. During the course of studies on the 1,3-dipolar cycloaddition of quinoline 1-oxides, reaction of 4-methoxyquinoline 1-oxide with dimethyl acetylenedicarboxylate was found to afford the title compound (*A*), though in a small yield of 8·1%, together with the 2-substituted quinoline (*B*) and the furo[3,2-*c*]quinoline (*C*) as shown in Fig. 1 (Ishiguro, Funakoshi, Saeki, Hamana & Ueda, 1980). Although the demethylation of the 4-methoxy group of the quinoline ring is inevitable, the formation of *A* is very noticeable because *A* is the first example of 2,3-dihydroquinolines obtained from the 1,3-dipolar cycloaddition of quinoline 1-oxides. By an X-ray diffraction method the conformation of *A* was determined as given in the title.

The title compound was recrystallized from an ethanol solution as yellow plates. Preliminary Weissenberg photographs showed the systematic absences of the space group $P2_1/c$. The unit-cell parameters were determined by a least-squares procedure using the 2θ values of 15 reflexions measured on a Syntex $P\bar{1}$ four-circle diffractometer. The density was measured by flotation in an aqueous KI solution. A single crystal

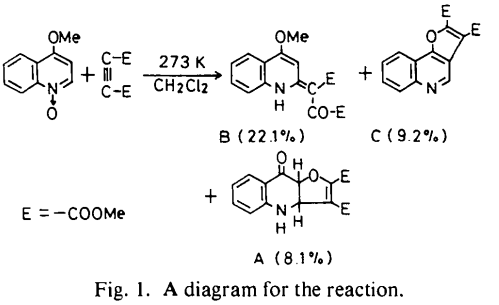

Fig. 1. A diagram for the reaction.

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with approximate dimensions 0·26 × 0·33 × 0·41 mm was used for intensity measurements. 4686 reflexions within a range of $2\theta \leq 60°$ were collected by the θ–2θ scan technique with a variable scan rate in 2θ from 4·8 to 12·0° min^{-1} (monochromated *Mo Kα* radiation). 2198 independent reflexions with $I > 2{\cdot}33\sigma(I)$ were considered to be observed and they were used for the analysis. Corrections for Lorentz and polarization effects and for fluctuation of the monitored intensities were applied, but no absorption corrections were made.

The structure was solved by the direct method using *MULTAN* (Germain, Main & Woolfson, 1971). Among 219 reflexions with $E \geq 1{\cdot}9$, the phases of three reflexions were determined by the $\sum_1$ relation and three reflexions to define the origin were selected by the program. Three additional reflexions were then allowed to assume all possible sign combinations. An E map calculated with the phases which gave the highest value of the figure of merit (1·046) revealed all 22 non-hydrogen atoms as the largest peaks in the asymmetric unit. The R value calculated with this model was 0·25 for all observed reflexions.

Table 1. *Fractional atomic coordinates with their estimated standard deviations in parentheses*

	x	y	z	B_{eq} or B_{iso} (Å^2)
C(1)	0·8560 (2)	0·1783 (2)	0·4607 (2)	3·0
C(2)	0·8281 (2)	0·1014 (2)	0·3496 (2)	3·2
C(3)	0·7345 (2)	0·1546 (2)	0·2429 (2)	3·3
C(4)	0·6710 (2)	0·2700 (2)	0·2532 (2)	3·2
C(5)	0·5908 (2)	0·3291 (3)	0·1550 (2)	4·4
C(6)	0·5295 (3)	0·4378 (3)	0·1637 (3)	5·4
C(7)	0·5458 (3)	0·4899 (3)	0·2714 (3)	5·5
C(8)	0·6215 (3)	0·4346 (3)	0·3688 (2)	4·6
C(9)	0·6868 (2)	0·3234 (2)	0·3617 (2)	3·1
C(10)	0·8614 (2)	0·0718 (2)	0·5451 (2)	2·9
C(11)	0·8138 (2)	−0·0336 (2)	0·4873 (2)	3·2
C(12)	0·7920 (3)	−0·1642 (2)	0·5274 (2)	3·8
C(13)	0·6992 (4)	−0·2774 (3)	0·6411 (4)	7·9
C(14)	0·9079 (2)	0·0926 (2)	0·6688 (2)	3·1
C(15)	0·9637 (3)	−0·0078 (3)	0·8510 (2)	4·8
N	0·7617 (2)	0·2696 (2)	0·4591 (2)	3·9
O(1)	0·7177 (2)	0·0954 (2)	0·1535 (1)	4·8
O(2)	0·7820 (2)	−0·0233 (1)	0·3722 (1)	3·9
O(3)	0·8392 (2)	−0·2574 (2)	0·5064 (2)	6·1
O(4)	0·7164 (2)	−0·1588 (2)	0·5861 (2)	4·8
O(5)	0·9322 (2)	0·1969 (2)	0·7113 (1)	4·4
O(6)	0·9221 (2)	−0·0162 (2)	0·7272 (1)	4·0
H(C1)	0·937 (2)	0·224 (3)	0·476 (2)	2·0 (6)
H(C2)	0·907 (2)	0·084 (3)	0·333 (2)	2·0 (6)
H(C5)	0·580 (3)	0·288 (3)	0·077 (2)	3·1 (7)
H(C6)	0·471 (3)	0·481 (3)	0·094 (3)	3·6 (7)
H(C7)	0·504 (3)	0·570 (3)	0·281 (3)	4·4 (8)
H(C8)	0·633 (3)	0·474 (3)	0·444 (3)	3·4 (7)
H(N)	0·771 (3)	0·312 (3)	0·525 (2)	3·0 (7)
H(1C13)	0·639 (4)	−0·257 (4)	0·677 (4)	7·6 (12)
H(2C13)	0·666 (4)	−0·337 (4)	0·586 (4)	8·3 (12)
H(3C13)	0·773 (4)	−0·305 (5)	0·692 (4)	9·9 (14)
H(1C15)	0·975 (3)	−0·090 (4)	0·873 (3)	5·6 (9)
H(2C15)	1·036 (4)	0·046 (4)	0·874 (3)	6·7 (11)
H(3C15)	0·905 (4)	0·028 (4)	0·875 (3)	6·5 (10)

Block-diagonal least-squares refinement of the atomic coordinates and anisotropic thermal parameters reduced R to 0·076. A difference Fourier synthesis calculated at this stage gave all the H-atom peaks. By introduction of these H atoms into the refinement the final R dropped to 0·047 for the observed reflexions. In the final three cycles of the refinement the weighting scheme was changed from unit weights to $w = 0{\cdot}095/(4{\cdot}80–0{\cdot}225|F_o| + 0{\cdot}0028|F_o|^2)$ for $|F_o| > 55$, $w = 1{\cdot}165$ for $55 \geq |F_o| \geq 25$ and $w = 2{\cdot}964–0{\cdot}571|F_o|$ for $|F_o| < 25$. Final shifts of the positional and thermal parameters of the non-hydrogen atoms were approximately one tenth of the standard deviations. A final difference Fourier map showed no peaks higher than 0·3 e Å^{-3}. The final atomic coordinates are listed in Table 1.*

The atomic scattering factors and the anomalous dispersion factors were taken from *International Tables for X-ray Crystallography* (1974). All the calculations were performed on the FACOM M-190 computer at the Computer Center of Kyushu University with the *UNICS* II program system (Sakurai, Iwasaki, Watanabe, Kobayashi, Bando & Nakamichi, 1974).

Discussion. Fig. 2 shows the crystal structure of a cell projected along the a axis and Fig. 3 the atom-numbering system, bond distances and angles. The molecules are held together by van der Waals forces and strong hydrogen bonds NH···O(1) as shown in Fig. 2 and Table 2.

The molecule consists of three parts: the two methoxycarbonyl groups, the furan ring and the quinoline ring. The furan ring is approximately planar

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 35492 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

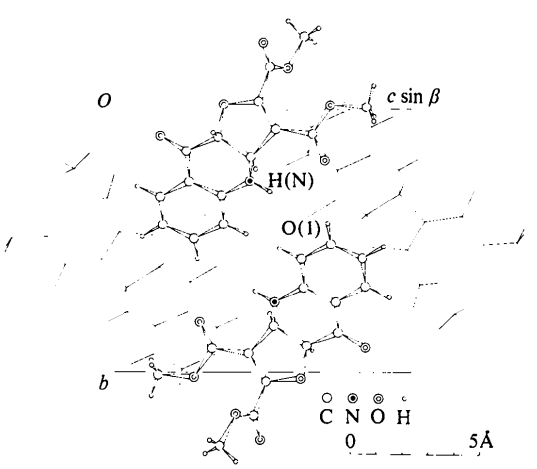
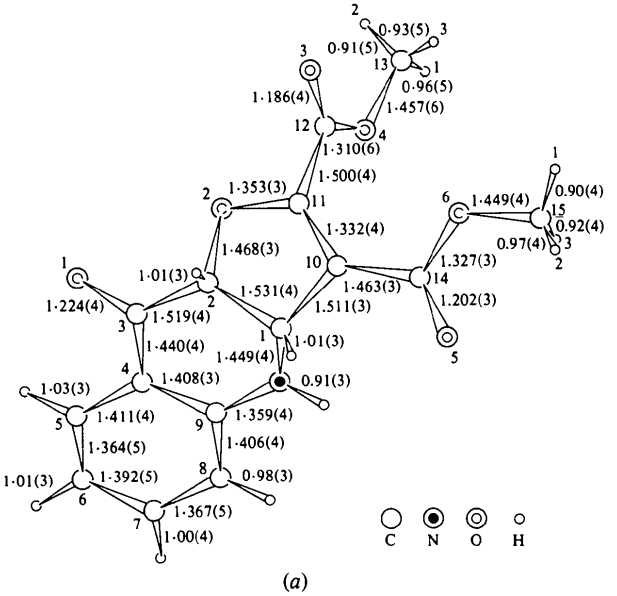
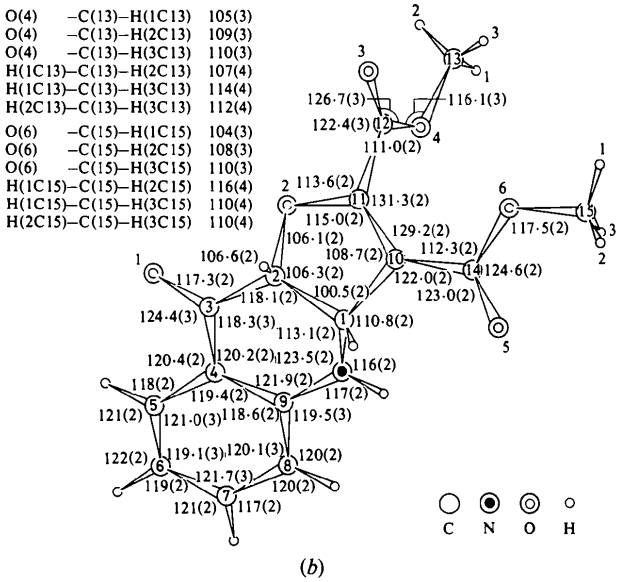


Fig. 2. The crystal structure projected along the a axis. The dashed line indicates the hydrogen bond.



(*a*)

C(2)	–C(1)	–H(C1)	109(2)°
C(10)	–C(1)	–H(C1)	113(2)
N	–C(1)	–H(C1)	110(2)
C(1)	–C(2)	–H(C2)	109(2)
C(3)	–C(2)	–H(C2)	109(2)
O(2)	–C(2)	–H(C2)	107(2)
O(4)	–C(13)	–H(1C13)	105(3)
O(4)	–C(13)	–H(2C13)	109(3)
O(4)	–C(13)	–H(3C13)	110(3)
H(1C13)	–C(13)	–H(2C13)	107(4)
H(1C13)	–C(13)	–H(3C13)	114(4)
H(2C13)	–C(13)	–H(3C13)	112(4)
O(6)	–C(15)	–H(1C15)	104(3)
O(6)	–C(15)	–H(2C15)	108(3)
O(6)	–C(15)	–H(3C15)	110(3)
H(1C15)	–C(15)	–H(2C15)	116(4)
H(1C15)	–C(15)	–H(3C15)	110(4)
H(2C15)	–C(15)	–H(3C15)	110(4)



(*b*)

Fig. 3. (*a*) Bond distances (Å); (*b*) bond angles (°).

within ±0·12 Å as shown in Table 3. The angles C(10)–C(11)–O(2) and C(2)–C(1)–C(10) are abnormally large and small respectively, as shown in Fig. 3. The bond distance O(2)–C(11) is somewhat shorter than O(2)–C(2). These abnormal bond angles and distances may be caused by the sp^2 hybridization of C(11), and by some conjugation between O(2), C(11), C(10), C(14) and O(5). A similar anomaly was also found in the case of benzofurans (Ansell, Moore & Nielsen, 1971; King, Hastings & Heller, 1975; Celikel, Geddes & Sheldrick, 1976). In the quinoline ring, the benzene ring is planar, but the pyridine ring has a conformation of a slightly creased hinge at the N···C(3) axis as shown in Table 3. This hinge form may be attributed to a *gauche* conformation between C(1) and C(2). The benzene and furan rings are tilted to each other at 54°. The two mesomeric forms shown in Fig. 4 can qualitatively explain the observed variations of the bond lengths of the quinoline ring.

The UV spectrum of the compound exhibited peaks at 237·4 nm (log ε = 4·42) and 406·6 nm (log ε = 3·56) in methanol. The latter peak seems to be caused by an intramolecular charge-transfer (CT) absorption. However, it cannot be concluded whether the CT absorption is caused by the intramolecular charge transfer in the enamide system [N,O(1)] or between the C(10)–C(11) double bond and the benzene ring as in

Table 2. *Intermolecular distances shorter than the sum of the van der Waals radii*

The van der Waals radii are taken from Pauling (1960).

Atoms	Distance	Translation	Symmetry*
C(3)–O(5)	2·903 (4) Å	0, 1, 0	(4)
C(3)–H(C7)	2·83 (4)	1, –1, 0	(2)
C(3)–H(N)	2·87 (3)	0, 1, 0	(4)
C(4)–H(C7)	2·85 (3)	1, –1, 0	(2)
C(14)–H(C2)	2·85 (3)	2, 0, 1	(3)
O(1)–H(C8)	2·56 (3)	0, 1, 0	(4)
O(1)–N	2·968 (3)	0, 1, 0	(4)†
O(1)–H(N)	2·11 (3)	0, 1, 0	(4)†
O(3)–H(C1)	2·57 (3)	2, 0, 1	(3)
O(5)–C(3)	2·903 (4)	0, 1, 1	(4)
O(6)–H(C2)	2·49 (3)	2, 0, 1	(3)

* (1) x,y,z; (2) $-x, \frac{1}{2}+y, \frac{1}{2}-z$; (3) $-x,-y,-z$; (4) $x, -\frac{1}{2}-y, -\frac{1}{2}+z$.

† Indicates the hydrogen bond.

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

(*a*) The equation of the benzene ring is $0{\cdot}8258X + 0{\cdot}5578Y - 0{\cdot}3452Z = 6{\cdot}9370$, where X,Y,Z are expressed in Å along the crystallographic cell edges.

C(4)*	0·002 (3)	C(1)	0·362 (3)
C(5)*	–0·006 (3)	C(2)	0·119 (3)
C(6)*	0·003 (4)	C(3)	–0·016 (3)
C(7)*	0·004 (3)	N	–0·006 (3)
C(8)*	–0·007 (3)	O(1)	–0·141 (2)
C(9)*	0·003 (3)		

(*b*) The equation of the furan ring is $0{\cdot}9509X - 0{\cdot}2920Y - 0{\cdot}2089Z = 7{\cdot}8407$.

C(1)*	–0·102 (3)	C(3)	–0·813 (3)
C(2)*	0·110 (3)	N	–1·420 (3)
C(10)*	0·063 (2)	C(12)	0·062 (3)
C(11)*	0·007 (3)	C(14)	0·196 (2)
O(2)*	–0·079 (2)		

* Indicates the atoms involved in the calculation of each least-squares plane.

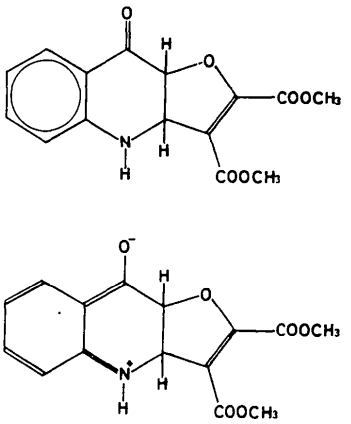


Fig. 4. Possible mesomeric forms. The second form is an intramolecular charge-transfer form.

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

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

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

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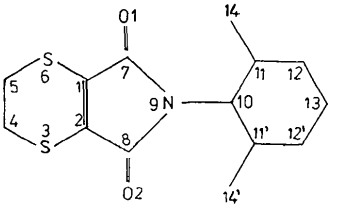
(*Received* 5 *February* 1980; *accepted* 19 *July* 1980)

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}, $F(000) = 608$, λ(Cu $K\alpha$) = 1·5418 Å, μ(Cu $K\alpha$) = 3·27 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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