Main, P., Fiske, S. J., Hull, S. E., Lessinger, L., Germain, G., Declercq, J. P. & Woolfson, M. M. (1980). *MULTAN* 80. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Merck, A.-G., E. (1965). Netherlands Patent Appl. 297,368; *Chem. Abstr.* **64**, No. 3563a.

Merck, A.-G., E. (1966). Netherlands Patent Appl. 6,511,478; *Chem. Abstr.* **65**, No. 10596f.

Meyers, A. I. & Shaw, C. C. (1974). *Tetrahedron Lett.* pp. 717–720.

Singh, P. & Hodgson, D. J. (1976). *Acta Cryst.* B**32**, 2329–2333.

Stewart, J. M., Machin, P. A., Dickinson, C., Ammon, H. L., Heck, H. S. & Flack, H. (1976). The XRAY system–version of 1976. Tech. Rep. TR-446. Computer Science Center, Univ. of Maryland, College Park, Maryland.

Stewart, R. F., Davidson, E. R. & Simpson, W. T. (1965). *J. Chem. Phys.* **42**, 3175–3187.

Wolpert-Defilippes, M. K., Adamson, R. H., Cysyk, R. L. & Jones, D. C. (1975). *Biochem. Pharmacol.* **24**, 751–754.

Wolpert-Defilippes, M. K., Bono, V. H., Dion, R. L. & Johns, D. G. (1975). *Biochem. Pharmacol.* **24**, 1735–1738.

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6-Chloro-1-methyl-4-phenyl-1*H*-2,1,3-benzothiadiazine 2,2-Dioxide, $C_{14}H_{11}ClN_2O_2S$

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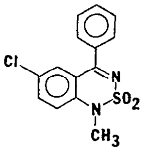
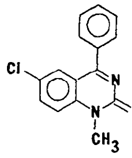
Abstract. M_r = 306·5, $P2_1/c$, a = 6·098 (2), b = 14·685 (3), c = 15·143 (2) Å, β = 95·89 (2)°, V = 1348·9 (6) Å^3, Z = 4, D_m = 1·49 Mg m^{-3}, D_x = 1·51 Mg m^{-3}, λ(Cu $K\alpha$) = 1·54178 Å, μ(Cu $K\alpha$) = 39·9 mm^{-1}, R = 0·066, R_w = 0·099 with 1737 independent non-zero reflections. The S atom is tetrahedral and the phenyl ring makes an angle of 55° to the plane of the heterocyclic ring. There is a close intermolecular approach of a Cl atom to an adjacent fused-ring system.

Introduction. The title compound, (1), was obtained as an unexpected by-product in the synthesis of 6-chloro-1-methyl-4-phenylquinazolin-2(1*H*)-one, (2), (Kamal, Rao & Sattur, 1980) and the X-ray investigation was undertaken to confirm the structure of the compound and to ascertain the geometry about the S atom.



(1)



(2)

Experimental. Light-yellow plate, 0·38 × 0·45 × 0·63 mm, D_m by flotation in CCl_4–toluene, data collected on an Enraf–Nonius CAD-4, lattice parameters from 15 high-angle reflections, systematic absences: $h0l$, $l = 2n + 1$; $0k0$, $k = 2n + 1$, maximum sin θ/λ = 0·58 Å^{-1}, standards (124 and 212) did not vary more than 6%, 2350 reflections measured, 1737 unique non-zero reflections at the 3σ significance level; structure solution with *MULTAN*78 (Main, Woolfson, Lessinger, Germain & Declercq, 1978), least-squares refinement using *F*'s, hydrogens located and refined isotropically, all other atoms refined anisotropically, R = 0·066, wR = 0·099, $w = 33{\cdot}57/\{[\sigma(F_o)]^2 + (0{\cdot}045\,F_o)^2\}$ where $\sigma(F_o)$ is the standard deviation based on counting statistics, maximum least-squares shift-to-error in final refinement cycle 0·017, maximum peak height in final difference Fourier synthesis 0·59e Å^{-3}, $F(000)$ = 632, scattering factors from *International Tables for X-ray Crystallography* (1974), refinements carried out with *SHELX*76 (Sheldrick, 1976).†

Discussion. The molecular structure of the title compound is confirmed. The atomic coordinates are reported in Table 1. Fig. 1 shows the atom-numbering scheme and the intramolecular bond lengths and angles. All these lengths and angles are normal.

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† Lists of structure factors, anisotropic thermal parameters and H atom coordinates have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 38120 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Table 1. *Atomic coordinates in fractional crystal coordinates and equivalent isotropic values of the anisotropic thermal parameters for non-*H *atoms and isotropic thermal parameters for* H ($U_{eq} = \frac{1}{3}$ *trace* $\tilde{\mathbf{U}}$)

Estimated standard deviations are given in parentheses. For labeling of atoms, see Fig. 1.

	x	*y*	*z*	*U*(Å^2)
C(4)	0·7087 (6)	−0·1458 (2)	0·7435 (2)	0·028 (2)
C(5)	0·7186 (7)	−0·0206 (3)	0·6326 (3)	0·031 (2)
C(6)	0·6341 (7)	0·0583 (3)	0·5951 (3)	0·030 (2)
C(7)	0·4471 (8)	0·0966 (3)	0·6237 (3)	0·037 (2)
C(8)	0·3564 (7)	0·0611 (3)	0·6941 (3)	0·042 (2)
C(9)	0·4463 (6)	−0·0169 (3)	0·7379 (3)	0·034 (2)
C(10)	0·6249 (6)	−0·0603 (2)	0·7034 (2)	0·027 (2)
C(11)	0·8553 (6)	−0·2054 (2)	0·6966 (2)	0·041 (2)
C(12)	1·0454 (7)	−0·2402 (3)	0·7429 (3)	0·041 (2)
C(13)	1·1872 (8)	−0·2930 (3)	0·6992 (3)	0·035 (2)
C(14)	1·1381 (8)	−0·3135 (3)	0·6099 (3)	0·034 (2)
C(15)	0·9482 (8)	−0·2815 (3)	0·5650 (3)	0·046 (3)
C(16)	0·8068 (7)	−0·2268 (3)	0·6077 (3)	0·044 (2)
C(17)	0·1754 (9)	−0·0087 (5)	0·8489 (5)	0·055 (3)
N(1)	0·3626 (5)	−0·0520 (2)	0·8124 (2)	0·033 (2)
N(3)	0·6658 (5)	−0·1732 (2)	0·8211 (2)	0·033 (2)
S(2)	0·5390 (2)	−0·1058 (1)	0·8843 (1)	0·058 (2)
O(2)	0·4183 (6)	−0·1602 (2)	0·9375 (2)	0·050 (2)
O(2)′	0·6955 (6)	−0·0434 (2)	0·9272 (2)	0·037 (1)
Cl(6)	0·7617 (2)	0·1102 (1)	0·5109 (1)	0·055 (1)
H(5)	0·819 (7)	−0·047 (3)	0·610 (3)	0·03 (1)
H(7)	0·391 (9)	0·140 (4)	0·594 (4)	0·06 (2)
H(8)	0·235 (9)	0·084 (4)	0·709 (3)	0·06 (2)
H(12)	1·090 (8)	−0·225 (3)	0·801 (3)	0·05 (1)
H(13)	1·310 (9)	−0·315 (3)	0·730 (3)	0·05 (1)
H(14)	1·233 (7)	−0·349 (3)	0·579 (3)	0·04 (1)
H(15)	0·909 (8)	−0·294 (3)	0·504 (3)	0·06 (1)
H(16)	0·675 (8)	−0·207 (3)	0·579 (3)	0·05 (1)
H(17)	0·226 (10)	0·048 (4)	0·871 (4)	0·07 (2)
H(17)′	0·132 (9)	−0·040 (3)	0·877 (3)	0·04 (1)
H(17)″	0·079 (19)	−0·009 (7)	0·793 (7)	0·17 (4)

Table 2. *Torsion angles* (°) *for the heterocyclic ring in the title compound*

N(1)–S(2)–N(3)–C(4)	37 (1)	C(17)–N(1)–S(2)–N(3)	161 (1)
S(2)–N(3)–C(4)–C(10)	−8 (1)	C(17)–N(1)–C(9)–C(10)	−179 (1)
N(3)–C(4)–C(10)–C(9)	−17 (1)	O(2)–S(2)–N(1)–C(9)	−161 (1)
C(4)–C(10)–C(9)–N(1)	4 (1)	O(2)′–S(2)–N(1)–C(9)	68 (1)
C(10)–C(9)–N(1)–S(2)	30 (1)	O(2)–S(2)–N(3)–C(4)	150 (1)
C(9)–N(1)–S(2)–N(3)	−48 (1)	O(2)′–S(2)–N(3)–C(4)	−81 (1)

The phenyl ring is accurately planar with none of the torsion angles around the ring greater than 2·2°. The plane of the C atoms of the fused ring is, however, only nearly planar with the greatest torsion angle being 5·7°. The heterocyclic ring is puckered so that the tetrahedral geometry of the $-SO_2-$ group can be accommodated. A list of torsion angles for the heterocyclic ring is given in Table 2. We note that the conformations of S(2)–O(2) and S(2)–O(2)′ with respect to the ring are about 20 and 75°. The S atom is tetrahedral with the angle formed by the planes N(1), S(2), N(3) and O(2), S(2), O(2)′ being 89°. The configuration about N(1) is pyramidal with N(1) lying at a distance of 0·223 (3) Å from the plane formed by C(9), C(17) and S(2).

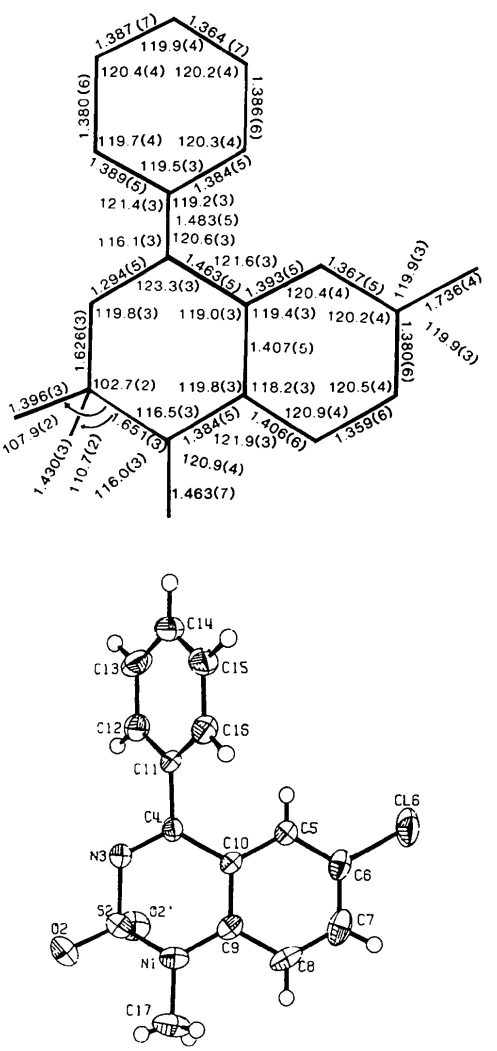

Fig. 1. Bond lengths (Å) and angles (°) and atom-numbering scheme for the title compound. The angles N(3)–S(2)–O(2), N(3)–S(2)–O(2)′ and O(2)–S(2)–O(2)′ are respectively 107·5 (2), 108·8 (2) and 118·1 (2)° and, for clarity, are not indicated.

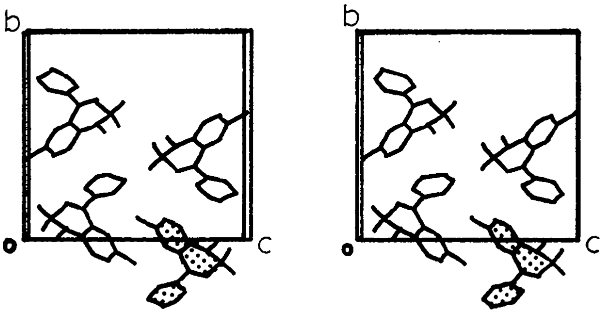

Fig. 2. Stereoscopic view of the crystal structure down the *a* axis showing the four molecules in general positions. The reference molecule is shaded.

A stereoscopic view of the crystal structure is shown in Fig. 2. The Cl atom of the reference molecule is situated at a distance of 3·565 (4) Å from the plane of the fused ring of the molecule (1−*z*, −*y*, 1−*z*).

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References

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 99–102. Birmingham: Kynoch Press.

Kamal, A., Rao, K. R. & Sattur, P. B. (1980). *Synth. Commun.* **10**, 799.

Main, P., Woolfson, M. M., Lessinger, L., Germain, G. & Declercq, J. -P. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data.* Univs. of York, England, and Louvain, Belgium.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

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Structure of 1,3,4,5,6,9-Hexamethyl-8-*exo*-methylenetricyclo[4.4.0.0^{3,9}]dec-4-en-2-one, $C_{17}H_{24}O$

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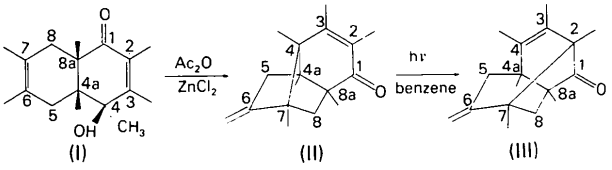
Abstract. $M_r = 244{\cdot}36$, monoclinic, $P2_1/a$, $a = 12{\cdot}374$ (2), $b = 8{\cdot}771$ (1), $c = 13{\cdot}743$ (2) Å, $\beta = 104{\cdot}027$ (6)°, $V = 1447{\cdot}1$ (3) Å^3, $Z = 4$, $D_c = 1{\cdot}222$ g cm^{-3}, λ (Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}626$ cm^{-1}, $T = 295$ K. Final $R = 0{\cdot}037$ for 1369 observed reflections. The structural analysis showed that a deconjugative 1,3-shift occurred on photolysis of the isomeric precursor in solution.

Introduction. Although efforts to relate structure with the solid-state photoreactivity of tetrahydronaphthoquinols have met with much success (Greenhough & Trotter, 1981; Appel, Jiang, Scheffer & Walsh, 1982; Secco & Trotter, 1982), one aspect of the study, that of hydrogen-bonding effects on reactivity, has been more difficult to ascertain. The present compound arose from attempts to acetylate the heptamethyl-4*β*-ol derivative (I) in the hope that by replacing OH with OAc, the reactivity in the absence of hydrogen bonding could have then been studied. Instead, however, the *exo*-methylene ketone (II) was formed as the major product, from which the title compound (III) was subsequently produced. The following crystal analysis shows that a deconjugative 1,3-shift resulted from the photolysis of (II) in solution leading to the isometric *exo*-methylene ketone (III).



Experimental. Generated from 2,3,4α,4a*β*,6,7,8a*β*-heptamethyl-4a*β*,5,8,8a*β*-tetrahydronaphthoquin-4*β*-ol (I) *via* intermediate 1,3,6,7,8,9-hexamethyl-4-*exo*-methylenetricyclo[4.4.0.0^{3,7}]dec-7-en-10-one (II) (Jiang, Scheffer, Secco, Trotter & Wong, 1982); recrystallization of methylene ketone (III) from a hexane/acetone solution yielded translucent crystals whose faces appeared related by a twofold rotation axis parallel to the long axis of the crystals; 0·3 × 0·2 × 0·3 mm, CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$, ω–(5/3)θ scan, $\theta \leq 27{\cdot}5°$, ω-scan width $(0{\cdot}65 + 0{\cdot}35\tan\theta)°$ extended 25% on each side for background measurement, horizontal aperture $(2{\cdot}00 + \tan\theta)$ mm, vertical aperture 4 mm, scan speeds 0·91–10·06 deg min^{-1}, maximum scan time 75 s, cell parameters from least-squares fit of sinθ values for 22 reflections with $11 < \theta < 18°$, Lp corrections, 3297 reflections, 1369 (41·5%) with $I \geq 3\sigma(I)$ where $\sigma^2(I) = S + 2B + [0{\cdot}04(S - B)]^2$, S = scan count, B = time-averaged background count; structure

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