

Lódź for pointing out the interesting features of the molecule and for a sample of the compound. This research was supported by project MR.I-9 from the Polish Academy of Sciences.

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S,S-Dimethyl-N-(2-oxo-1-indoliny)sulfoximide

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Abstract. C₁₀H₁₂O₂N₂S, monoclinic, *P*2₁/*c*, *a* = 12.310 (2), *b* = 10.970 (2), *c* = 7.955 (1) Å, β = 93.93 (1)°, *Z* = 4, *V* = 1071.7 (4) Å³, *M_r* = 266.2, *D_x* = 1.65 g cm⁻³. The structure was solved by Patterson syntheses and refined to *R* = 0.049 for 3186 observed reflections.

Introduction. The title compound was obtained by Dr B. Marco (1975) in the course of the synthesis of 3,4-diketocinnoline derivatives (Lora-Tamayo, Marco &

Navarro, 1974). Organic chemists are interested in the mechanism of formation of these products and want to know the precise conformation, which is not clear from other techniques; thus the present work was undertaken. The intensities were measured from a 0.41 × 0.34 × 0.62 mm crystal on a Philips PW 1100 four-circle diffractometer, with graphite-monochromated Mo K_α radiation (λ = 0.7107 Å). The ω–2θ scan technique was employed. 4716 unique reflections were recorded up to θ = 35° of which 3186 were considered

Table 1. Final positional parameters and their standard deviations, and bond distances (Å) involving the hydrogen atoms

	<i>x</i>	<i>y</i>	<i>z</i>		<i>x</i>	<i>y</i>	<i>z</i>	C–H bond
S(1)	0.33743 (3)	0.55708 (3)	0.25159 (5)	H(5 <i>a</i>)	0.254 (2)	0.091 (2)	0.377 (3)	0.97 (3)
O(4)	0.2877 (1)	0.5786 (1)	0.0839 (2)	H(5 <i>b</i>)	0.307 (3)	0.111 (3)	0.199 (4)	0.99 (3)
N(2)	0.2789 (1)	0.4764 (1)	0.3800 (2)	H(7)	0.068 (3)	0.055 (3)	0.113 (4)	0.96 (4)
N(3)	0.2596 (1)	0.3580 (1)	0.3132 (2)	H(8)	−0.081 (3)	0.172 (4)	0.029 (5)	0.92 (4)
C(4)	0.3227 (1)	0.2588 (1)	0.3563 (2)	H(9)	−0.084 (3)	0.391 (3)	0.060 (4)	1.01 (3)
C(5)	0.2627 (1)	0.1472 (1)	0.2852 (2)	H(10)	0.074 (2)	0.472 (3)	0.193 (4)	0.84 (3)
C(6)	0.1575 (1)	0.1977 (1)	0.2101 (2)	H(12 <i>a</i>)	0.502 (3)	0.568 (4)	0.160 (5)	0.96 (4)
C(7)	0.0674 (1)	0.1419 (2)	0.1312 (2)	H(12 <i>b</i>)	0.461 (3)	0.426 (4)	0.168 (5)	0.98 (4)
C(8)	0.0216 (2)	0.2141 (2)	0.0768 (3)	H(12 <i>c</i>)	0.507 (3)	0.499 (4)	0.334 (5)	0.96 (4)
C(9)	0.0192 (1)	0.3391 (2)	0.1003 (3)	H(13 <i>a</i>)	0.289 (4)	0.726 (5)	0.365 (6)	0.87 (5)
C(10)	0.0720 (1)	0.3967 (2)	0.1770 (2)	H(13 <i>b</i>)	0.396 (4)	0.753 (4)	0.290 (6)	1.04 (5)
C(11)	0.1586 (1)	0.3236 (1)	0.2308 (2)	H(13 <i>c</i>)	0.401 (4)	0.684 (4)	0.458 (6)	0.92 (5)
C(12)	0.4705 (2)	0.5051 (2)	0.2241 (3)					
C(13)	0.3541 (3)	0.6939 (2)	0.3645 (3)					
O(14)	0.4110 (1)	0.2622 (1)	0.4376 (2)					

Table 2. Bond distances (Å), bond angles (°) and torsional angles (°)

S(1)—O(4)	1.448 (1)	C(4)—O(14)	1.226 (2)
S(1)—N(2)	1.565 (1)	C(5)—C(6)	1.495 (2)
S(1)—C(12)	1.762 (2)	C(6)—C(7)	1.380 (2)
S(1)—C(13)	1.754 (2)	C(6)—C(11)	1.391 (2)
N(2)—N(3)	1.417 (2)	C(7)—C(8)	1.396 (3)
N(3)—C(4)	1.367 (2)	C(8)—C(9)	1.384 (3)
N(3)—C(11)	1.416 (2)	C(9)—C(10)	1.393 (3)
C(4)—C(5)	1.519 (2)	C(10)—C(11)	1.379 (2)
O(4)—S(1)—N(2)	120.6 (1)	C(5)—C(4)—O(14)	127.7 (1)
O(4)—S(1)—C(12)	106.0 (1)	C(4)—C(5)—C(6)	103.6 (1)
O(4)—S(1)—C(13)	111.1 (1)	C(5)—C(6)—C(7)	131.7 (1)
N(2)—S(1)—C(12)	111.8 (1)	C(5)—C(6)—C(11)	108.6 (1)
N(2)—S(1)—C(13)	101.1 (1)	C(7)—C(6)—C(11)	119.7 (1)
C(12)—S(1)—C(13)	105.3 (1)	C(6)—C(7)—C(8)	118.6 (2)
S(1)—N(2)—N(3)	110.2 (1)	C(7)—C(8)—C(9)	120.7 (2)
N(2)—N(3)—N(4)	123.9 (1)	C(8)—C(9)—C(10)	121.2 (2)
N(2)—N(3)—C(11)	122.5 (1)	C(9)—C(10)—C(11)	117.1 (2)
C(4)—N(3)—C(11)	111.7 (1)	N(3)—C(11)—C(6)	108.7 (1)
N(3)—C(4)—C(5)	107.1 (1)	N(3)—C(11)—C(10)	128.7 (1)
N(3)—C(4)—O(14)	125.2 (1)	C(6)—C(11)—C(10)	122.6 (1)

O(4)—S(1)—N(2)—N(3)	58.5 (1)
C(13)—S(1)—N(2)—N(3)	-178.7 (1)
C(12)—S(1)—N(2)—N(3)	-67.1 (1)
S(1)—N(2)—N(3)—C(4)	100.5 (1)
C(11)—N(3)—C(4)—C(5)	4.5 (2)
N(3)—C(4)—C(5)—C(6)	-3.7 (1)
C(4)—C(5)—C(6)—C(11)	1.7 (1)
C(5)—C(6)—C(11)—N(3)	0.9 (1)
C(6)—C(11)—N(3)—C(4)	-3.52 (1)

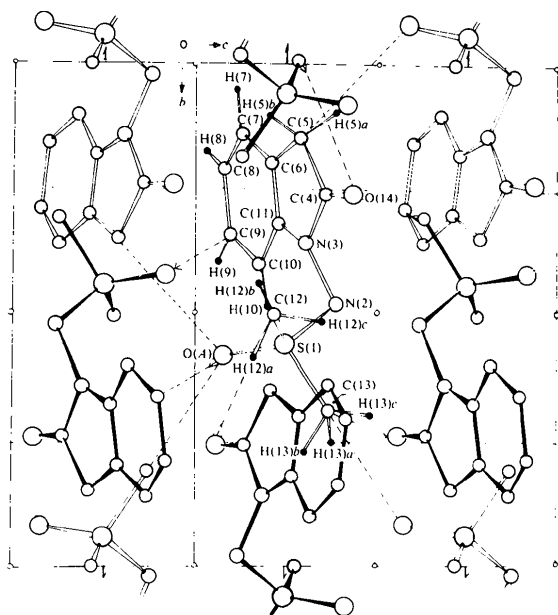


Fig. 1. Projection of the crystal structure along [100], showing the numbering system and the main intermolecular contacts.

Table 3. Main intermolecular interactions involving H atoms

Symmetry code				
	None	x, y, z	(iii)	$x, \frac{1}{2} - y, \frac{1}{2} + z$
(i)	$-x + 1, \frac{1}{2} + y, \frac{1}{2} - z$	(iv)	$-x, -y + 1, -z$	
(ii)	$-x + 1, -y + 1, -z + 1$	(v)	$x, -y + \frac{1}{2}, z + \frac{1}{2}$	
	X	H	Y	
	O(14 ⁱ)	...	H(12a)	—C(12)
	O(14 ⁱⁱ)	...	H(13c)	—C(13)
	O(4 ⁱⁱⁱ)	...	H(5a)	—C(5)
	O(4 ^{iv})	...	H(9)	—C(9)
	O(4 ^v)	...	H(13a)	—C(13)
$X \cdots Y$	$Y-H$	$H \cdots X$	$X-H \cdots Y$	$H-Y \cdots X$
3.463 (3) Å	0.96 (4) Å	2.53 (4) Å	165 (3)°	11 (2)°
3.232 (3)	0.92 (5)	2.48 (5)	140 (4)	29 (3)
3.431 (2)	0.97 (3)	2.50 (3)	162 (2)	13 (2)
3.636 (2)	1.01 (3)	2.70 (3)	153 (2)	20 (2)
3.184 (3)	0.87 (5)	2.77 (5)	111 (4)	54 (3)

as observed by an $I > 2\sigma(I)$ criterion. No decomposition was detected.

The unit-cell dimensions were determined from the 2θ and -2θ values of 61 reflections. Lorentz-polarization corrections were applied, but no account was taken of absorption [$\mu(\text{Mo } K\alpha) = 3.05 \text{ cm}^{-1}$]. The structure was solved by the heavy-atom method and refined by full-matrix least-squares calculations. All H atoms were located in a difference Fourier synthesis and included in the final cycles of refinement. The weighting scheme was: $w = K/|f(F_o)|^2 f(S)$ with $f(F_o) = 0.42 + 0.26F_o - 0.08F_o^2$ for $F_o < 3.45$ and $f(F_o) = 0.38 - 0.02F_o$ otherwise, and $f(S) = 8.67 - 28.89S + 25.86S^2$ for $S < 0.63$, where $S = \sin \theta/\lambda$ and $K = 5.47$, chosen to make the average values of $w(|F_o| - |F_c|)^2$ as equal as possible in the different $|F_o|$ and $\sin \theta$ intervals. The weighted and unweighted agreement indices for the observed reflections were 0.055 and 0.049 respectively; $R_w = (\sum w\Delta^2 / \sum w|F_o|^2)^{1/2}$. There were no peaks larger than $0.39 \text{ e } \text{Å}^{-3}$ in the final difference map. Table 1 lists the fractional coordinates and hydrogen bonds. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974).*

Discussion. The geometrical features of the molecule are detailed in Table 2, with the numbering system given in Fig. 1. All bond lengths and angles in the five-membered ring have values consistent with those of other structures (Chiaroni, Doucerain & Riche, 1976; Karle, Britts & Gum, 1964; Karle, Dragonette &

* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 32883 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 13 White Friars, Chester CH1 1NZ, England.

Brenner, 1965; Riche & Pascard-Billy, 1976). The six C—C bond lengths in the benzene ring average 1.387 Å.

The bonds about the S atom form a distorted tetrahedron as in other sulphone groups (Giuseppetti, Tadini, Bettinetti & Giordano, 1977; Hamodrakas, Filippakis & Sheldrick, 1977; Hamodrakas & Filippakis, 1977). The S—C distance lies within the range of reported values (Calleri, Chiari, Villa, Manfredotti, Guastini & Viterbo, 1976; Tranqui, Vicat & Fillion, 1976). The O(A)—S(1)—N(2) angle is increased to 120.6°, while the N(2)—S(1)—C(13) angle is decreased to 103.0°; dihedral angles C(12)S(1)C(13)—N(2)S(1)O(A), O(A)S(1)C(13)—N(2)S(1)C(12) and O(A)S(1)C(12)—N(2)S(1)C(13) are 84.4, 84.4 and 90.0° respectively. The S(1)—N(2) bond, 1.565 (1) Å, is slightly longer than double-bond values previously observed for compounds containing this group (Tickle & Prout, 1971; Cameron & Duncanson, 1976; Wieggers & Vos, 1963). Following the conformational criterion of Altona, Geise & Romers (1968) we calculated the pseudo-rotation phase angles: $\Delta = -8.51^\circ$, $\varphi_m = 4.54^\circ$. The conformation of the ring can be considered as a distorted half chair. The shorter intermolecular distances are contained in Table 3.

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2-Phenylcyclopropane-1-spiro-4'-(2'-benzylthio-4',5'-dihydro-6'H-1',3'-thiazine)-5'-spiro-2''-oxirane (CTO)

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Abstract. C₂₀H₁₉ONS₂, monoclinic, $P2_1/c$, $Z = 4$, $a = 18.193$ (3), $b = 5.584$ (1), $c = 17.563$ (2) Å, $\beta = 101.55$ (2)°, μ (Mo $K\alpha$) 2.92 cm⁻¹, $\rho_c = 1.34$, $\rho_m = 1.31$ g cm⁻³ (floatation). Final R and R_w values for the observed reflections are 0.054 and 0.049. The heterocyclic six membered ring has an 'overall' half-chair conformation.

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Introduction. The title compound is used as the base in the preparation of synthetic cephalopurines. It presented, when examined by IR and NMR spectroscopy, some structural ambiguities, namely the existence of either a six-membered or a five-membered ring.

A crystal of approximate dimensions 0.2 × 0.2 × 0.4 mm was used. 3089 independent reflections up