

Table 1. Atomic coordinates ( $\times 10^4$ ) and equivalent isotropic temperature factors ( $\text{\AA}^2$ )
$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	x	y	z	$U_{eq}$
O1	3793 (2)	789 (1)	893 (1)	0.050 (1)
C2	4861 (3)	1490 (2)	491 (1)	0.044 (1)
C3	6680 (3)	1449 (2)	686 (1)	0.047 (1)
C4	7621 (3)	691 (2)	1327 (1)	0.046 (1)
C5	7042 (3)	-842 (2)	2417 (1)	0.045 (1)
C6	5817 (4)	-1550 (2)	2726 (2)	0.055 (1)
C7	3972 (4)	-1457 (2)	2423 (2)	0.060 (1)
C8	3302 (3)	-660 (2)	1824 (2)	0.057 (1)
C9	4555 (3)	24 (2)	1500 (1)	0.045 (1)
C10	6417 (3)	-43 (2)	1762 (1)	0.042 (1)
O11	8843 (2)	-847 (1)	2718 (1)	0.055 (1)
C12	9487 (4)	-1565 (3)	3446 (2)	0.065 (1)
O13	6203 (3)	3383 (2)	-268 (1)	0.069 (1)
C14	6989 (5)	4265 (3)	-698 (3)	0.080 (1)
O15	9287 (2)	688 (2)	1478 (1)	0.070 (1)
C1'	3728 (3)	2207 (2)	-149 (1)	0.048 (1)
C2'	4433 (4)	3128 (2)	-538 (2)	0.056 (1)
C3'	3297 (5)	3744 (2)	-1164 (2)	0.070 (1)
C4'	1492 (4)	3479 (2)	-1395 (2)	0.073 (1)
C5'	785 (4)	2597 (2)	-1000 (2)	0.069 (1)
C6'	1891 (4)	1967 (2)	-386 (2)	0.058 (1)

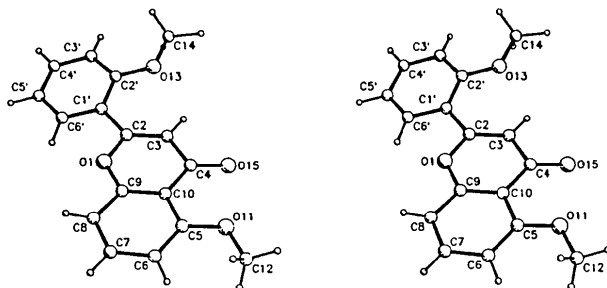


Fig. 1. Stereoscopic view of the molecule with atom numbering.

between the benzopyran and the phenyl planes is  $0^\circ$  while in 2',4',5,7-tetramethoxyflavone it is  $24^\circ$ . In this latter molecule a slight puckering of the benzopyran was observed; in the title compound all the endocyclic torsion angles have values less than  $3(1)^\circ$  and the maximum deviation from the best mean

Table 2. Bond distances ( $\text{\AA}$ ) and bond angles ( $^\circ$ )

C2—O1	1.360 (3)	C9—O1	1.374 (2)
C3—C2	1.341 (3)	C1'—C2	1.477 (3)
C4—C3	1.447 (3)	C10—C4	1.476 (3)
O15—C4	1.225 (3)	C6—C5	1.382 (3)
C10—C5	1.417 (3)	O11—C5	1.351 (3)
C7—C6	1.387 (4)	C8—C7	1.375 (3)
C9—C8	1.387 (3)	C10—C9	1.386 (3)
C12—O11	1.426 (3)	C14—O13	1.426 (3)
C2'—O13	1.357 (3)	C2'—C1'	1.410 (3)
C6'—C1'	1.392 (3)	C3'—C2'	1.393 (3)
C4'—C3'	1.377 (4)	C5'—C4'	1.375 (4)
C6'—C5'	1.381 (3)		
C9—O1—C2	120.7 (2)	C3—C2—O1	120.5 (2)
C1'—C2—O1	110.3 (2)	C1'—C2—C3	129.2 (2)
C4—C3—C2	123.4 (2)	C10—C4—C3	114.6 (2)
O15—C4—C3	120.7 (2)	O15—C4—C10	124.7 (2)
C10—C5—C6	119.9 (2)	O11—C5—C6	123.5 (2)
O11—C5—C10	116.6 (2)	C7—C6—C5	120.1 (2)
C8—C7—C6	121.7 (2)	C9—C8—C7	117.3 (2)
C8—C9—O1	114.2 (2)	C10—C9—O1	122.2 (2)
C10—C9—C8	123.6 (2)	C5—C10—C4	124.3 (2)
C9—C10—C4	118.5 (2)	C9—C10—C5	117.2 (2)
C12—O11—C5	118.0 (2)	C2'—O13—C14	119.0 (2)
C2'—C1'—C2	122.6 (2)	C6'—C1'—C2	119.1 (2)
C6'—C1'—C2'	118.3 (2)	C1'—C2'—O13	117.8 (2)
C3'—C2'—O13	122.8 (2)	C3'—C2'—C1'	119.3 (3)
C4'—C3'—C2'	121.0 (3)	C5'—C4'—C3'	119.9 (2)
C6'—C5'—C4'	120.1 (3)	C5'—C6'—C1'	121.3 (3)

plane through the ten atoms of the heterocycle is  $0.05(1) \text{\AA}$ .

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## 5-Hydroxy-5-methylsydno[3,4-a]indole

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**Abstract.**  $\text{C}_{10}\text{H}_8\text{N}_2\text{O}_3$ ,  $M_r = 204.18$ , monoclinic,  $P2_1/a$ ,  $a = 8.702(4)$ ,  $b = 12.063(2)$ ,  $c = 9.711(4) \text{\AA}$ ,  $\beta = 115.10(2)^\circ$ ,  $V = 923.1(6) \text{\AA}^3$ ,  $Z = 4$ ,  $D_x =$

$1.47 \text{ g cm}^{-3}$ ,  $\lambda(\text{Mo } K\alpha) = 0.71069 \text{\AA}$ ,  $\mu = 1.04 \text{ cm}^{-1}$ ,  $F(000) = 424$ ,  $T = 296 \text{ K}$ , final  $R = 0.047$  for 1843 reflections with  $I > 3\sigma(I)$ . The sydnone ring exhibits

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Table 1. Atomic positional and isotropic displacement parameters for 5-hydroxy-5-methylsydno[3,4-*a*]indole

Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as

$$\frac{1}{3}(U_{11} + U_{22} + U_{33} + U_{12}\cos\gamma + U_{13}\cos\beta + U_{23}\cos\alpha).$$

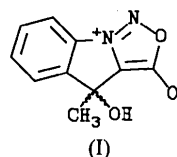
	x	y	z	$U(\text{\AA}^2)$
C(1)	-0.2091 (2)	0.5227 (1)	0.0432 (2)	0.0393 (6)
C(2)	-0.2940 (2)	0.4247 (2)	0.0415 (2)	0.0442 (6)
C(3)	-0.2163 (2)	0.3408 (2)	0.1457 (2)	0.0440 (7)
C(4)	-0.0497 (2)	0.3509 (1)	0.2558 (2)	0.0399 (6)
C(4a)	0.0371 (2)	0.4479 (1)	0.2597 (2)	0.0318 (5)
C(5)	0.2187 (2)	0.4819 (1)	0.3676 (2)	0.0299 (5)
C(5a)	0.2243 (2)	0.5949 (1)	0.3012 (2)	0.0310 (5)
C(6)	0.3253 (2)	0.6887 (1)	0.3179 (2)	0.0333 (5)
O(6)	0.4684 (2)	0.7190 (1)	0.4010 (2)	0.0451 (5)
O(7)	0.2185 (2)	0.7601 (1)	0.2006 (1)	0.0398 (4)
N(8)	0.0599 (2)	0.7134 (1)	0.1182 (2)	0.0389 (5)
N(8a)	0.0749 (2)	0.6181 (1)	0.1843 (2)	0.0309 (4)
C(8b)	-0.0448 (2)	0.5299 (1)	0.1541 (2)	0.0317 (5)
O(9)	0.3381 (2)	0.4096 (1)	0.3505 (1)	0.0401 (4)
C(10)	0.2430 (2)	0.4900 (2)	0.5320 (2)	0.0413 (6)

Table 2. Interatomic bond distances ( $\text{\AA}$ ) and angles ( $^\circ$ ) for 5-hydroxy-5-methylsydno[3,4-*a*]indole

C(1)—C(2)	1.391 (3)	C(5)—O(9)	1.419 (2)
C(1)—C(8b)	1.381 (2)	C(5)—C(10)	1.522 (3)
C(1)—H(1)	.990 (2)	C(5a)—C(6)	1.399 (2)
C(2)—C(3)	1.387 (3)	C(5a)—N(8a)	1.344 (2)
C(2)—H(2)	.965 (2)	C(6)—O(6)	1.219 (2)
C(3)—C(4)	1.395 (2)	C(6)—O(7)	1.416 (2)
C(3)—H(3)	.972 (3)	O(7)—N(8)	1.388 (2)
C(4)—C(4a)	1.385 (2)	N(8)—N(8a)	1.296 (2)
C(4)—H(4)	.985 (2)	N(8a)—C(8b)	1.430 (2)
C(4a)—C(5)	1.536 (2)	O(9)—H(9)	.935 (2)
C(4a)—C(8b)	1.384 (2)	C(10)—H(10a)	.980 (3)
C(5)—C(5a)	1.517 (2)	C(10)—H(10b)	.893 (3)
		C(10)—H(10c)	.954 (2)
C(2)—C(1)—C(8b)	115.6 (2)	C(5)—C(5a)—N(8a)	110.6 (1)
C(2)—C(1)—H(1)	125.4 (2)	C(5)—C(5a)—C(6)	144.0 (1)
C(8b)—C(1)—H(1)	118.8 (2)	C(5)—C(5a)—N(8a)	110.6 (1)
C(1)—C(2)—C(3)	121.3 (1)	C(6)—C(5a)—N(8a)	105.4 (1)
C(1)—C(2)—H(2)	117.0 (2)	C(5a)—C(6)—O(6)	137.3 (1)
C(3)—C(2)—H(2)	121.7 (2)	C(5a)—C(6)—O(7)	103.4 (1)
C(2)—C(3)—C(4)	121.5 (2)	O(6)—C(6)—O(7)	119.2 (1)
C(2)—C(3)—H(3)	116.2 (2)	C(6)—O(7)—N(8)	111.7 (1)
C(4)—C(3)—H(3)	122.3 (2)	O(7)—N(8)—N(8a)	102.0 (1)
C(3)—C(4)—C(4a)	118.2 (2)	C(5a)—N(8a)—N(8)	117.5 (1)
C(3)—C(4)—H(4)	119.0 (2)	C(5a)—N(8a)—C(8b)	112.2 (1)
C(4a)—C(4)—H(4)	122.7 (2)	N(8)—N(8a)—C(8b)	130.3 (1)
C(4)—C(4a)—C(5)	129.5 (1)	C(1)—C(8b)—C(4a)	124.8 (2)
C(4)—C(4a)—C(8b)	118.6 (1)	C(1)—C(8b)—N(8a)	129.1 (1)
C(5)—C(4a)—C(8b)	111.9 (1)	C(4a)—C(8b)—N(8a)	106.1 (1)
C(4a)—C(5)—C(5a)	.99.1 (1)	C(5)—O(9)—H(9)	105.2 (2)
C(4a)—C(5)—O(9)	110.5 (1)	C(5)—C(10)—H(10a)	111.6 (2)
C(4a)—C(5)—C(10)	112.2 (2)	C(5)—C(10)—H(10b)	112.8 (2)
C(5a)—C(5)—O(9)	110.2 (2)	C(5)—C(10)—H(10c)	110.8 (2)
C(5a)—C(5)—C(10)	111.9 (1)	H(10a)—C(10)—H(10b)	102.7 (3)
O(9)—C(5)—C(10)	112.3 (1)	H(10a)—C(10)—H(10c)	106.8 (2)
C(5)—C(5a)—C(6)	144.0 (1)	H(10b)—C(10)—H(10c)	111.8 (3)
C(5)—C(5a)—N(8a)	110.6 (1)		

distances and angles that are in agreement with the generalized features suggested by Hope & Thiessen [*J. Am. Chem. Soc.* (1967), **89**, 5977–5978] and does not appear to be distorted by the five-membered ring fusion to benzene. The complete ring structure is planar with a maximum deviation from planarity of 0.016 (2)  $\text{\AA}$ .

**Experimental.** Synthesis of (I) by heating a methanol solution of 3-(2-acetylphenyl)sydnone (Lowe & Turnbull, 1986) and excess methylamine or ammonia in a pressure vessel at 353 K for 12 h, evaporation, washing with with minimum 2-propanol;



colorless rectangular crystal (0.31 × 0.47 × 0.41 mm), glass fiber mount; Enraf–Nonius CAD-4 diffractometer system,  $\omega/2\theta$  scanning technique, variable scan speed (0.76–5.58° min<sup>-1</sup>), Mo  $K\alpha$  radiation and a graphite monochromator; lattice parameters by least squares utilizing 25 accurately centered reflections ( $\theta = 4.2$  to 15.7°); three reflections (0 $\bar{2}$ 3, 1 $\bar{4}$ 1, 004) measured every 2 h revealed minimal change (< 3.1% over 98 h); 5902 reflections collected ( $1 < \theta < 30^\circ$ ; range of  $hkl$ : -12→12, -16→16, -13→13), 2681 unique, 1843 with  $I > 3\sigma(I)$ ; Lorentz, polarization and numerical absorption (transmission = 94.74–97.07%) corrections applied; equivalent reflections averaged ( $R_{\text{int}} = 2.0\%$ , based on  $I$  for 3095 replicates); solution from direct methods (Hall & Stewart, 1990), 232 reflections ( $E > 1.75$ ), 1991 triplets and 674 quartets, all non-hydrogen atoms from phase set with highest combined figure of merit; hydrogen-atom positions were located by difference Fourier synthesis and additively constrained to the attached non-hydrogen atom,  $U = 0.035 \text{\AA}^2$ ; refinement on  $F$  using full-matrix least squares with weights calculated by the method of Robertson & Wang (1990), 136 variables,  $R = 0.047$ ,  $wR = 0.050$ ,  $S = 0.91$ , maximum shift/e.s.d. in final cycle 0.0003, maximum and minimum peak in final  $\Delta F$  0.36 and -0.30 e  $\text{\AA}^{-3}$ ,  $R$  factor for all reflections is 0.069.

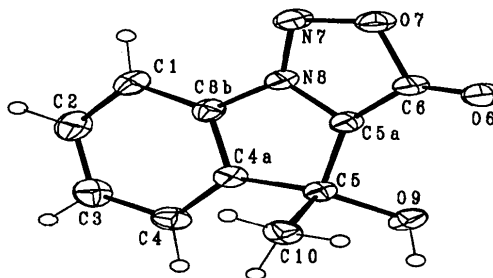


Fig. 1. Molecular structure of 5-hydroxy-5-methylsydno[3,4-*a*]indole. Non-hydrogen atoms are shown at 30% of thermal probability. Hydrogen atoms are drawn artificially reduced in size.

Scattering factors used are those of Cromer & Waber (1974). Investigation used *XTAL/PC* (Hall & Stewart, 1990; Grossie, 1990). Table 1\* presents the final atomic coordinates and equivalent isotropic thermal parameters. Selected derived values in the form of interatomic distances and angles are tabulated in Table 2. A drawing of the molecular structure is shown in Fig. 1.

**Related literature.** The structures of other sydnone have been reported by Barnighausen, Jellinek, Munnik & Vos (1963), Hope & Thiessen (1969) and King, Preston, Suffolk & Turnbull (1979). The sydnone ring in the title compound is virtually identical (within errors) to the other sydnones reported even though there should be considerable structural strain from the ring fusion.

\* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, dihedral angles and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54519 (18 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: ST0533]

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## Structure of the Anti-Malarial Drug Primaquine Diphosphate

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**Abstract.** 8-(4-Amino-1-methylbutylamino)-6-methoxyquinoline bis(dihydrogenphosphate),  $C_{15}H_{23}N_3O_2^+ \cdot 2(H_2PO_4)^-$ ,  $M_r = 455.35$ , triclinic,  $P\bar{1}$ ,  $Z = 2$ ,  $a = 7.389$  (6),  $b = 8.862$  (4),  $c = 16.055$  (10) Å,  $\alpha = 97.57$  (2),  $\beta = 100.21$  (3),  $\gamma = 77.01$  (2)°,  $V = 1003.6$  (5) Å<sup>3</sup>,  $D_m = 1.495$  (by flotation),  $D_x = 1.507$  g cm<sup>-3</sup>,  $\lambda(Cu K\alpha) = 1.5418$  Å,  $\mu(Cu K\alpha) = 24.48$  cm<sup>-1</sup>,  $F(000) = 480$ , room temperature,  $R = 0.068$  for 3448 observed reflections. The above working cell is related to the reduced cell with angles  $\alpha = 82.43$ ,  $\beta = 79.79$  and  $\gamma = 77.01$ ° by the transformation  $(-1\ 0\ 0/0\ -1\ 0/0\ 0\ 1)$ . Primaquine diphosphate

was crystallized in the dicationic form with protonation on the quinoline ring nitrogen atom and on the terminal amino group. One dihydrogenphosphate anion is chelated by the quinoline ring and the butylamino side chain. The other dihydrogenphosphate anion is hydrogen bonded to the terminal amino group. The C(14) atom is nearly in the plane of the quinoline ring with a C(9)—C(8)—N(13)—C(14) torsion angle of 169°. The butyl-diamino side chain is kinked by rotation about the C(14)—C(16) bond with a N(13)—C(14)—C(16)—C(17) torsion angle of -59°. The C(15) methyl substituent is inline with the rest of the butyl chain. The terminal amino group N(19) is hydrogen bonded to three symmetry-related phosphate groups while N(1) and N(13) are 'chelated' to a fourth phosphate group.

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