Instrumental Achievements

Crystal Structure of 2,3-Dihydro-3-ethyl-9-(phenylsulfonyl)carbazole-4(1*H*)-one

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The title compound (Fig. 2) may be considered as a synthetic precursor of tetracyclic indole alkaloids, dasycarpidone and uleine, which have been isolated from Aspidosperma.[1] It was prepared from the reaction of 2,3-dihydro-3-ethyl-carbazole-4(1*H*)-one (1.0 g, 4.7 mmol) and tetrabutylammonium hydrogen sulfate (0.1 g, 0.3 mmol) in 30 ml chloroform by a method of Volker.[2] Later, aqueous sodium hydroxide (50%) was added and stirred for 15 min. Benzene sulfonylchloride (1 ml) was dropped into this mixture and stirred at 298 K for 3 h and then washed with water. The organic layer was dried with

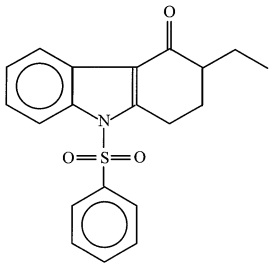

Fig. 1 Chemical diagram.

Table 1 Crystal and experimental data

Formula: $C_{20}H_{19}NO_3S$
Formula weight = 353.44
Crystal system: monoclinic
Space group: $P2_1/c$ $Z = 4$
a = 8.138(1)Å
b = 11.728(1)Å
c = 18.590(1)Å
β = 102.20(1)°
V = 1734.2(3)Å^3
D_x = 1.354 g/cm^3
μ(Cu K$_\alpha$) = 1.77 mm^{-1}
T = 293 K
Color: yellow
Crystal size: 0.20 × 0.25 × 0.30 mm
λ(Cu K$_\alpha$) = 1.54184Å
R = 0.055 wR = 0.066
No. of reflections measured = 3761
No. of reflections used = 2505, [F >3.0 $\sigma(F)$]
No. of parameters = 230
Goodness-of-fit = 1.13
$(\Delta/\sigma)_{max}$ = 0.01
$(\Delta\rho)_{max}$ = 0.40
$(\Delta\rho)_{min}$ = –0.31
$2\theta_{max}$ = 148.7°
Measurements: Enraf-Nonius CAD-4 diffractometer
Program system: CAD-4 EXPRESS Software
Structure determination: MolEN
Treatment of hydrogen atoms: difference synthesis and geometric calculation
Refinement: full-matrix least-squares

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Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	B_{eq}/Å^2
S1	0.4386(1)	0.24388(8)	0.17754(4)	4.70(2)
O1	0.1776(5)	0.5685(2)	-0.0859(1)	8.22(9)
O2	0.5559(3)	0.1568(2)	0.1681(2)	6.28(6)
O3	0.4864(3)	0.3299(2)	0.2317(1)	6.18(6)
C1	0.3474(6)	0.5186(3)	0.1409(2)	5.52(9)
C2	0.2501(8)	0.6191(4)	0.1093(2)	8.4(1)
C3	0.2558(7)	0.6548(3)	0.0343(2)	6.7(1)
C4	0.2307(5)	0.5555(3)	-0.0195(2)	5.31(9)
C4a	0.2816(4)	0.4435(3)	0.0114(2)	3.99(7)
C5	0.2441(5)	0.3030(3)	-0.0997(2)	4.94(8)
C5a	0.2856(4)	0.3362(3)	-0.0259(2)	4.03(7)
C6	0.2628(5)	0.1901(4)	-0.1159(2)	6.0(1)
C7	0.3190(5)	0.1101(4)	-0.0623(2)	6.2(1)
C8	0.3628(5)	0.1413(3)	0.0110(2)	5.50(9)
C8a	0.3464(4)	0.2546(3)	0.0276(2)	4.13(7)
C9a	0.3378(4)	0.4267(3)	0.0852(2)	4.04(7)
N9	0.3809(3)	0.3126(2)	0.0972(1)	4.32(6)
C10	0.1590(6)	0.7587(3)	0.0032(3)	7.0(1)
C11	0.1703(8)	0.8597(4)	0.0518(3)	9.3(2)
C12	0.2525(4)	0.1779(3)	0.1892(2)	3.86(7)
C13	0.1088(4)	0.2429(3)	0.1853(2)	5.03(8)
C14	-0.0349(5)	0.1918(4)	0.1978(2)	5.75(9)
C15	-0.0344(5)	0.0793(4)	0.2139(2)	5.80(9)
C16	0.1074(6)	0.0145(4)	0.2185(2)	6.2(1)
C17	0.2553(5)	0.0634(3)	0.2050(2)	4.93(8)

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

Table 3 Bond distances (Å) and angles (°)

S1-O2	1.432(3)	N9-C8a	1.433(4)
S1-O3	1.420(3)	N9-C9a	1.392(4)
S1-N9	1.674(3)	C4-C4a	1.463(5)
S1-C12	1.754(3)	C4a-C5a	1.439(5)
C5-C5a	1.399(4)	C4a-C9a	1.365(4)
O2-S1-O3	120.1(1)	C5a-C4a-C9a	108.9(3)
O2-S1-N9	107.5(2)	C4a-C5a-C8a	107.1(3)
O2-S1-C12	108.5(2)	C4a-C9a-N9	108.5(3)
O3-S1-N9	106.1(1)	C8a-N9-C9a	108.4(2)
O3-S1-C12	109.8(2)	N9-S1-C12	103.7(1)

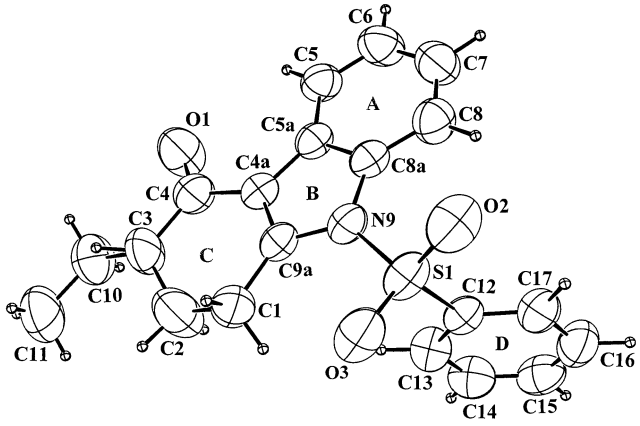

Fig. 2 Molecular structure of the title compound with atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

magnesium sulfate and evaporated. The residue was crystallized from ethanol.

The results of an X-ray structure determination are given in Tables 1 – 3, and the molecular structure in Fig. 2.

Rings A, B and D are planar, while ring C is not planar with a maximum deviation at C2 [–0.238(6)Å]. They are also twisted with respect to each other. The dihedral angles between the least-squares planes are A/C=5.4(5), A/D=91.3(1), B/C=5.3(5), B/D=90.9(1) and C/D=96.0(1)˚.

References

1. J. A. Joule, M. Ohashi, and B. Gilbert, *Tetrahedron*, **1965**, *21*, 1717.
2. O. I. Volker, *Synthesis*, **1979**, 136.