Instrumental Achievements

Crystal Structure of *N*-(1,2,3,4-Tetrahydrocarbazole-1-yl)-2-methoxyacetamide

Tuncer HÖKELEK*† and Süleyman PATIR**

**Hacettepe University, Department of Physics, 06532 Beytepe-Ankara, Turkey*
***Hacettepe University, Department of Science Faculty of Education, 06532 Beytepe-Ankara, Turkey*

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Tetrahydrocarbazole systems are present in the framework of a number of indole-type alkaloids of biological interest. The presence of a 2-methoxyacetamide group on the C1 atom may be used as a key intermediate in the synthesis of the 1,5-methanoazocino(3,4-b)indole skeleton, which is the precursor for the synthesis of strictamine- and akuammiline-type indole alkaloids.[1]

The title compound was prepared from the catalytic reduction of 2,3,4,9-tetrahydrocarbazole-1-one-oxime (2 g, 10 mmol), 3 ml 2-methoxyacetanhydride and 100 mg Pd/C in 20 ml absolute THF, at room temperature under normal atmospheric pressure for 3 days. The resulting mixture was filtered from the catalyst (Pd/C). The organic layer was dried with magnesium sulfate and evaporated. The residue was crystallized from diethyl ether.

The results of X-ray structure determination are given in Tables 1 – 3.

Table 1 Crystal and experimental data

Formula: $C_{15}H_{18}N_2O_2$
Formula weight = 258.32
Crystal system: monoclinic
Space group: $P2_1/n$ $Z = 4$
a = 8.083(1)Å
b = 10.092(1)Å
c = 16.951(2)Å
β = 102.78(2)°
V = 1348.5(3)Å^3
D_x = 1.272 g/cm^3
μ(Cu K$_\alpha$) = 0.687 mm^{-1}
T = 293 K
Colorless
Crystal size: 0.20 × 0.25 × 0.30 mm
λ(Cu K$_\alpha$) = 1.54184 Å
R = 0.045 wR = 0.055
No. of reflections measured = 2747
No. of reflections used = 2472
No. of parameters=232
Goodness-of-fit = 1.19
$(\Delta/\sigma)_{max}$ = 0.01
$(\Delta\rho)_{max}$ = 0.22 eÅ^{-3}
$(\Delta\rho)_{min}$ = –0.35 eÅ^{-3}
$2\theta_{max}$= 148.5°
Measurements: Enraf-Nonius CAD-4 diffractometer
Program system: CAD-4 EXPRESS software
Structure determination: MolEN
Treatment of hydrogen atoms: difference synthesis
Refinement: Full matrix least-squares

† To whom correspondence should be addressed.

The title molecule (Fig. 2) consists of a carbazole skeleton and a methoxyacetamide chain at position 1. The types and the coordination positions of the substituents are dependent on the geometry of the carbazole skeleton.[2-6]

The rings A and B are close to planar, while the ring C is not planar, with a maximum deviation at C2 (0.338(2)Å). They are also twisted with respect to each other. The dihedral angles between the best planes are A/B = 1.0(1), A/C = 5.0(1) and B/C

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters

Atom	x	y	z	B_{eq}/Å^2
O1	0.4095(2)	-0.1363(1)	0.96323(7)	0.0492(3)
O2	0.8475(2)	-0.1781(1)	0.96846(8)	0.0643(3)
N1	0.5715(2)	-0.0877(1)	0.87353(8)	0.0413(3)
N9	0.5660(2)	0.2001(1)	0.86757(7)	0.0406(3)
C1	0.4405(2)	-0.0210(2)	0.81328(9)	0.0387(3)
C2	0.4114(2)	-0.0882(2)	0.7305(1)	0.0469(3)
C3	0.2981(2)	-0.0029(2)	0.6662(1)	0.0496(4)
C4	0.3823(2)	0.1283(2)	0.65313(9)	0.0430(3)
C4a	0.4635(2)	0.1886(2)	0.73290(9)	0.0360(3)
C5	0.5429(2)	0.4340(2)	0.7071(1)	0.0449(3)
C5a	0.5307(2)	0.3191(2)	0.75230(9)	0.0373(3)
C6	0.6142(2)	0.5468(2)	0.7467(1)	0.0517(4)
C7	0.6728(2)	0.5486(2)	0.8308(1)	0.0518(4)
C8	0.6633(2)	0.4377(2)	0.8769(1)	0.0475(4)
C8a	0.5926(2)	0.3233(2)	0.83736(9)	0.0387(3)
C9a	0.4875(2)	0.1206(2)	0.80390(9)	0.0358(3)
C10	0.5474(2)	-0.1350(2)	0.94328(9)	0.0402(3)
C11	0.7034(3)	-0.1886(2)	1.0008(1)	0.0553(4)
C12	0.9995(3)	-0.2027(3)	1.0234(1)	0.0815(6)

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

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

O1-C10	1.235(2)	N1-C10	1.329(2)
O2-C11	1.396(3)	N9-C8a	1.379(2)
O2-C12	1.390(2)	N9-C9a	1.383(2)
N1-C1	1.463(2)		
C11-O2-C12	114.5(2)	O1-C10-N1	124.5(1)
C1-N1-C10	124.1(1)	O1-C10-C11	119.6(2)
C8a-N9-C9a	108.3(1)	N1-C10-C11	115.8(2)

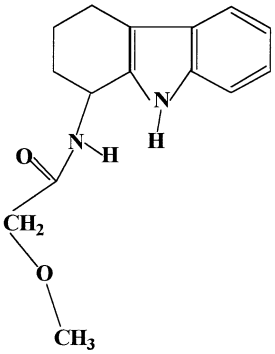

Fig. 1 Chemical diagram.

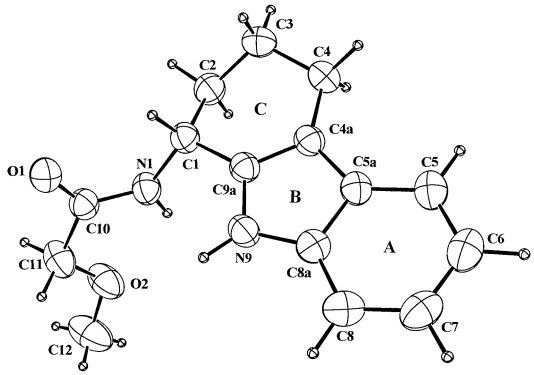

Fig. 2 Molecular structure of the title compound with atom-numbering scheme. The thermal ellipsoids are drawn at the 50% probability level.

= 5.2(1)˚. Ring C has a sofa conformation with a local pseudo-mirror running along the mid-points of C2–C3 and C4a–C9a bonds.

References

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