Instrumental Achievements

Crystal Structure of 2-[(5′,6′,7′,8′-Tetrahydro-5′,5′,8′,8′-tetramethyl)-2′-naphthyl]-1-ethyl-1*H*-benzimidazole-5-carboxylic Acid Ethyl Ester

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The title compound, which is a benzimidazole analogue of retinoids with a tetrahydronaphthalene structure (Fig. 1), was synthesized using an $NaHSO_3$ addition product of 5,6,7,8-tetrahydro-5,5,8,8-tetramethyl-2-naphthalene-carboxaldehyde (compound I) as a starting material, which was prepared as described.[1] The condensation of compound I and 4-ethylamino-3-amino-ethylbenzoate (mp: 73˚C) in DMF for 70 h gave the title compound (recrystallized from hexane/ethyl acetate; mp, 178˚C).

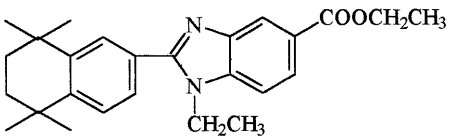

Fig. 1 Chemical structure.

Table 1 Crystal and experimental data

Formula: $C_{26}H_{32}N_2O_2$	
Formula weight = 404.557	
Crystal system: monoclinic	
Space group: *Pc*	
a = 11.853(5)Å	Z =2
b = 6.207(4)Å	β = 108.99(4)˚
c = 16.346(6)Å	
V =1137.3(8)Å^3	
D_c = 1.18 g/cm^3	
μ(Mo K$_\alpha$) = 0.070 mm^{-1}	
T = 295 K	
Color: white, prismatic	
Crystal size: 0.03 × 0.42 × 0.48 mm	
$2\theta_{max}$ = 52.6 with Mo K$_\alpha$	
R = 0.051	
Rw = 0.056	
No. of reflections used = 1531	
No. of parameters = 269	
Goodness-of-fit = 0.98	
$(\Delta/\sigma)_{max}$ = 0.004	
$(\Delta\rho)_{max}$ = 0.14 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.336 eÅ^{-3}	
Measurements: Enraf Nonius CAD-4 diffractometer	
Program system: CAD-4 EXPRESS Software	
Structure determination: MolEN	
Treatment of hydrogen atoms: geometric calculation	
Refinement: full-matrix least-squares (MolEN)	

The structure of the molecule is shown in Fig. 2 (ORTEP-MolEN).[2] Table 1 gives the crystal and relevant X-ray data. The fractional coordinates and equivalent isotropic temperature factors with estimated standard deviations for the non-hydrogen atoms are listed in Table 2; selected geometric parameters are given in Table 3.

The benzimidazole ring system is planar. The exocyclic angles around atom N1 show considerable asymmetry, although

Table 2 Final atomic coordinates and equivalent isotropic thermal parameters for non-hydrogen atoms

Atom	x	y	z	B_{eq}/Å^2
O1	0.6458(3)	0.2306(7)	0.8995(3)	5.72(9)
O2	0.7335(3)	0.5545(7)	0.9196(2)	5.77(9)
N1	0.9962(3)	0.2072(6)	0.6769(2)	3.04(8)
N3	1.0080(3)	0.5367(6)	0.7347(2)	3.22(8)
C2	1.0417(4)	0.4082(7)	0.6823(3)	2.90(1)
C4	0.865	0.4641(8)	0.817	3.14(9)
C5	0.7943(4)	0.3064(9)	0.8336(3)	3.40(9)
C6	0.7909(4)	0.0981(9)	0.8012(3)	3.90(9)
C7	0.8561(5)	0.0406(9)	0.7477(3)	4.10(9)
C8	0.9243(4)	0.2034(8)	0.7295(3)	3.00(9)
C9	0.9322(4)	0.4089(8)	0.7632(2)	2.83(9)
C10	1.0202(4)	0.0185(8)	0.6314(3)	3.80(9)
C11	0.9169(6)	-0.0400(9)	0.5521(4)	5.60(9)
C20	1.1187(4)	0.4824(8)	0.6321(3)	2.93(9)
C21	1.0797(4)	0.4549(9)	0.5423(3)	3.60(9)
C22	1.1432(4)	0.5529(9)	0.4946(3)	4.00(9)
C23	1.2442(4)	0.6720(8)	0.5330(3)	3.20(9)
C24	1.2878(4)	0.6962(8)	0.6233(3)	3.10(9)
C25	1.2193(4)	0.5979(8)	0.6699(3)	2.88(9)
C26	1.3030(4)	0.7900(9)	0.4738(3)	4.30(9)
C27	1.4210(6)	0.8800(9)	0.5253(4)	9.60(9)
C28	1.4437(7)	0.9450(9)	0.6110(4)	9.80(9)
C29	1.4012(4)	0.8110(9)	0.6715(3)	4.10(9)
C30	1.3228(5)	0.6320(9)	0.4084(3)	6.90(9)
C31	1.2193(6)	0.9630(9)	0.4240(4)	7.60(9)
C32	1.3855(6)	0.9610(9)	0.7403(4)	8.30(9)
C33	1.4953(7)	0.6470(9)	0.7165(6)	8.60(9)
C50	0.7163(4)	0.3558(9)	0.8885(3)	3.90(9)
C51	0.6504(6)	0.6400(9)	0.9657(4)	7.30(9)
C52	0.6983(7)	0.5650(9)	1.0530(4)	9.10(9)

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

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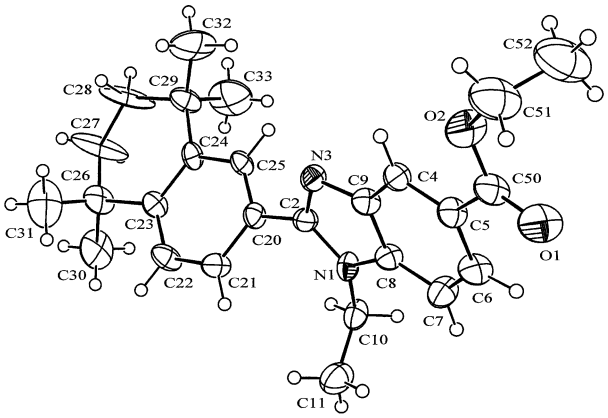

Fig. 2 ORTEP drawing of the title compound with atomic labeling. The displacement ellipsoids are drawn at the 40% probability level.

the sum of the valence angles around N1 is 360˚, indicating no significant pyramidalization of this atom. The observed bond lengths are normal.[3] The 5,5,8,8-tetramethyl-2-naphthyl ring system is planar and makes an angle of 51.62˚ with the benzimidazole ring plane. The COO group and the benzimidazole ring are almost coplanar with a torsion angle, C4–C5–C50–O2, of –4.0(6)˚.

Table 3 Selected geometric parameters (Å, ˚)

N1 - C2	1.351 (6)	C26 – C27	1.486 (8)
N3 – C2	1.323 (6)	C24- C29	1.499 (6)
N1 – C10	1.465 (7)	C10 – C11	1.509 (7)
C2 – C20	1.484 (7)	C5 – C50	1.514 (8)
C2-N1-C10	129.3 (4)	C6-C5-C50	117.7 (5)
C2-N1-C8	106.6 (4)	O2-C51-C52	105.7 (6)
C8-N1-C10	123.9 (4)		
C4-C5-C50-O2	-4.0 (6)	C2-N1-C10-C11	-108.4 (6)
N3-C2-C20-C25	47.3 (7)	C6-C50-C5-O1	-5.9 (7)

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