Instrumental Achievements

Crystal Structure of 2-Dibenzoylmethyl Benzimidazole

Sema ÖZTÜRK,*† Mehmet AKKURT,* Mehmet SÖNMEZ, and Semra İDE*****

**Department of Physics, Faculty of Arts and Sciences, Erciyes University, Kayseri-38039, Turkey*
***Department of Chemistry, Faculty of Arts and Sciences, Yüzüncü Yıl University, Van-65080, Turkey*
****Department of Physics Engineering, Faculty of Engineering, Hacettepe University, Beytepe, Ankara-06532, Turkey*

(Received September 22, 2000; Accepted June 15, 2001)

Many substituted benzimidazoles are currently used in the treatment of various infections caused by fungi, bacteria, nematodes and viruses.[1] The benzimidazole ring is included in biologically active substances and in a variety of clinically useful drugs such as Omeprazole, Astemizole, Emedastine difumarate, and Cyanocobalamine.[2] In particular, during the last decade, the antiallergic and antihistaminic activity of the benzimidazoles has received much attention.[3]

The title compound was prepared by the reaction of dibenzoylacetic acid-*N*-carboxymethylamide and *o*-phenylenediamine. Measured amounts (0.650 g of dibenzoylacetic acid-*N*-carboxymethylamide and 0.220 g of *o*-phenylenediamine) were joined in 30 ml of xylene. This mixture was heated in a reflux for 4 h. After the solvent was removed from the rotovapor, the precipitate so formed was recrystallized from absolute ethanol (Fig. 1).

The product shows a *keto-enol* tautomery. In this study, we describe the single crystal structure analysis of the title compound with a *keto*-form in solid phase. A summary of the key crystallographic information is given in Table 1. The atomic coordinates and equivalent isotropic displacement parameters for non-hydrogen atoms are listed in Table 2, and

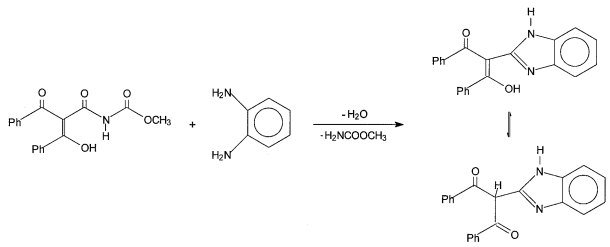

Fig. 1 Synthesis pathway of the title compound.

Table 1 Crystal and experimental data

Formula: $C_{22}H_{16}N_2O_2$	
Formula weight = 340.37	
Crystal system: monoclinic	
Space group: $P2_1/c$ (No. 14); $Z = 4$	
a = 7.7837(6)Å	
b = 6.3635(5)Å	β = 90.490(3)°
c = 33.440(1)Å	
V = 1656.2(2)Å^3	
D_x = 1.365 g/cm^3	
μ(Cu K$_\alpha$) = 7.11 cm^{-1}	
T = 295 K	
F(0 0 0) = 712	
Crystal size = 0.80 × 0.40 × 0.10 mm	
Radiation: Cu K$_\alpha$	
R = 0.073	
Rw = 0.297	
No. of unique data measured = 2810	
No. of observed data with [$I \geq 2\sigma(I)$] = 1620	
No. of parameters = 237	
Goodness-of-fit = 2.6	
$(\Delta\rho)_{max}$ = 0.45 eÅ^{-3}	
$(\Delta\rho)_{min}$ = –0.41 eÅ^{-3}	
Measurements: Enraf Nonius CAD-4 diffractometer	
Structure determination: SIR92	
Treatment of hydrogen atoms: geometric calculation	
Refinement: full matrix least-squares SHELXL93	

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

Atom	x	y	z	U_{eq}
O9	0.408(2)	-0.021(2)	0.4213(3)	0.082(4)
O10	0.475(2)	0.599(2)	0.3479(3)	0.088(4)
N1	0.584(2)	0.280(2)	0.4527(4)	0.077(4)
N2	0.608(2)	0.576(2)	0.4195(3)	0.067(3)
C1	0.677(2)	0.418(2)	0.4777(4)	0.069(4)
C2	0.743(2)	0.395(3)	0.5141(4)	0.072(4)
C3	0.830(2)	0.565(3)	0.5324(5)	0.074(4)
C4	0.849(2)	0.752(3)	0.5110(5)	0.081(5)
C5	0.780(2)	0.772(2)	0.4723(4)	0.069(4)
C6	0.696(2)	0.604(2)	0.4555(4)	0.057(4)
C7	0.541(2)	0.383(2)	0.4178(4)	0.064(4)
C8	0.434(2)	0.434(2)	0.3876(4)	0.058(4)
C9	0.350(2)	0.350(2)	0.3958(4)	0.064(4)
C90	0.168(2)	0.168(2)	0.3793(4)	0.067(4)
C91	0.048(2)	0.048(2)	0.3692(4)	0.069(4)
C92	-0.113(2)	-0.113(2)	0.3594(4)	0.068(4)
C93	-0.167(2)	-0.167(2)	0.3572(4)	0.082(5)
C94	-0.046(2)	-0.046(2)	0.3674(5)	0.081(5)
C95	0.111(2)	0.111(2)	0.3775(4)	0.067(4)
C10	0.422(2)	0.422(2)	0.3490(4)	0.062(4)
C100	0.352(2)	0.352(2)	0.3136(4)	0.069(4)
C101	0.253(2)	0.253(2)	0.2875(5)	0.087(5)
C102	0.192(3)	0.192(3)	0.2506(5)	0.096(6)
C103	0.230(3)	0.230(3)	0.2401(6)	0.108(7)
C104	0.332(3)	0.332(3)	0.2647(5)	0.089(5)
C105	0.391(2)	0.391(2)	0.3017(5)	0.073(4)

$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* (\boldsymbol{a}_i \cdot \boldsymbol{a}_j)$.

† To whom correspondence should be addressed.
E-mail: ozturk@erciyes.edu.tr

Table 3 Bond lengths (Å), bond and torsion angles (˚)

O9 — C9	1.24(2)	C9 — C90	1.55(2)
O10 — C10	1.21(2)	C90 — C95	1.40(2)
N1 — C7	1.37(2)	C90 — C91	1.41(2)
N1 — C1	1.41(2)	C91 — C92	1.33(2)
N2 — C7	1.34(2)	C92 — C93	1.37(2)
N2 — C6	1.39(2)	C93 — C94	1.40(2)
C1 — C2	1.32(2)	C94 — C95	1.29(2)
C1 — C6	1.41(2)	C10 — C100	1.41(2)
C2 — C3	1.41(2)	C100 — C101	1.36(2)
C3 — C4	1.40(2)	C100 — C105	1.49(2)
C4 — C5	1.40(2)	C101 — C102	1.39(2)
C5 — C6	1.37(2)	C102 — C103	1.36(3)
C7 — C8	1.42(2)	C103 — C104	1.38(2)
C8 — C9	1.42(2)	C104 — C105	1.38(2)
C8 — C10	1.52(2)		
C7 — N1 — C1	109(1)	C8 — C9 — C90	121(1)
C7 — N2 — C6	110(1)	C95 — C90 — C91	118(2)
C2 — C1 — N1	132(2)	C95 — C90 — C9	118(1)
C2 — C1 — C6	122(1)	C91 — C90 — C9	124(1)
N1 — C1 — C6	105(1)	C92 — C91 — C90	121(2)
C1 — C2 — C3	120(1)	C91 — C92 — C93	122(2)
C4 — C3 — C2	119(1)	C92 — C93 — C94	116(2)
C3 — C4 — C5	121(2)	C95 — C94 — C93	124(2)
C6 — C5 — C4	119(1)	C94 — C95 — C90	119(2)
C5 — C6 — N2	133(1)	O10 — C10 — C100	118(1)
C5 — C6 — C1	119(1)	O10 — C10 — C8	120(1)
N2 — C6 — C1	107(1)	C100 — C10 — C8	122(1)
N2 — C7 — N1	108(1)	C101 — C100 — C10	123(2)
N2 — C7 — C8	128(1)	C101 — C100 — C105	116(2)
N1 — C7 — C8	124(1)	C10 — C100 — C105	121(1)
C7 — C8 — C9	118(1)	C100 — C101 — C102	125(2)
C7 — C8 — C10	116(1)	C103 — C102 — C101	118(2)
C9 — C8 — C10	126(1)	C102 — C103 — C104	120(2)
O9 — C9 — C8	121(1)	C105 — C104 — C103	124(2)
O9 — C9 — C90	117(1)	C104 — C105 — C100	117(1)

Table 4 Possible hydrogen bond lengths (Å) and angles (˚)

D–H··A	D–H (Å)	H··A (Å)	D··A (Å)	D–H··A (˚)
H2—H2N...O9^{i}	0.86(2)	2.56(2)	3.00(2)	113(1)
N2—H2N...O10	0.86(2)	2.07(2)	2.61(2)	120(1)
C2—H2...O9^{ii}	0.93(3)	2.57(2)	3.43(2)	154(2)

Equivalent positions: [*i=x, 1+y, z; ii=1–x, 1–y, –z*].

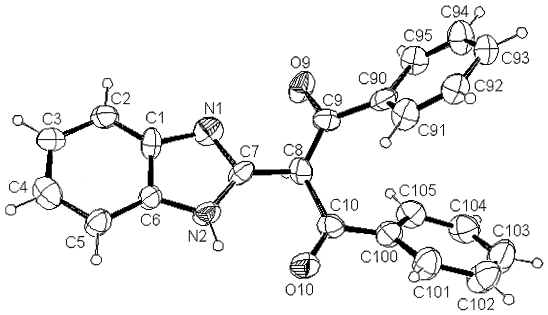

Fig. 2 ORTEP view of the title compound showing the labeling of the non-H atoms. Thermal ellipsoids are shown at the 30% probability level.

bond lengths and bond angles are given in Table 3. Figure 2 represents the molecular structure of the title compound.

In the title compound, N1–C7 and N2–C7 bond lengths are very close to N1–C1 [1.35(1)Å] and N2–C1 [1.32(1)Å] bond lengths in the bis(methyl-3-ethyl-benzimidazolidine-2-ylium) tetrafluoroborate.[4] N1–C7 bond length [1.37(2)Å] is longer than N2–C7 bond length [1.34(2)Å], the corresponding values, in 1-ethyl-3-methylbenzimidazole-2-thione,[5] 1.364(5) and 1.349(5)Å, in 2-(3-methoxy-2-hydroxyphenyl)benzimidazole,[6] are 1.371(4) and 1.325(5)Å; and in 1-(phenylmethyl)-2-(4-methoxyphenylmethyl)-1*H*-benzimidazole-5-carboxylic acid,[7] 1.365(4) and 1.331(4)Å, respectively. The benzimidazole ring system is almost planar, with a dihedral angle of 2.1(5)˚ between the imidazole and benzene ring planes. The angles N2-C7-C8 = 128(1) and N1-C7-C8 = 124(1)˚ are almost symmetric.

The structure is stabilized by one intra and two intermolecular hydrogen bonds, which are given in Table 4. For a structure analysis, U_{eq} normally has values about 0.05 Å^2 at room temperature. The high U_{eq} values for some atoms in Table 2 may indicate a low quality of the investigated crystal.

Acknowledgements

The authors wish to acknowledge the purchase of the CAD-4 diffractometer under Grant DPT/TBAG1 of the Scientific and Technical Research Council of Turkey.

References

1. W. B. Pratt, "*Chemotherapy of Infection*", **1977**, Oxford University Press, New York.
2. H. Göker and U. Abbasoğlu, *Arch. Pharm.* [Weinheim], **1995**, *328*, 425.
3. S. Özbey, E. Kendi, H. Göker, and M. Tunçbilek, *J. Chem. Crsytallogr*., **1998**, *28*, 461.
4. A. Aydın, H. Soylu, B. Güneş, M. Akkurt, F. Ercan, H. Küçükbay, and E. Çetinkaya, *Z. Kristallogr*., **1998**, *213*, 473.
5. Ş. K. İngeç, H. Soylu, H. Küçükbay, F. Ercan, and M. Akkurt, *Anal. Sci*., **1999**, *15*, 927.
6. Y. Elerman and M. Kabak, *Acta Crystallogr*., **1997**, *C53*, 372.
7. S. Özbey, S. İde, and E. Kendi, *J. Mol. Struct*., **1998**, *23*, 442.