International Journal of ChemTech Research
CODEN (USA): IJCRGG ISSN: 0974-4290
Vol.9, No.04 pp 301-305, 2016

Crystal structure analysis of N,2-diphenylacetamide

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Abstract: The crystal structure ofN,2-diphenylacetamide($C_{28}H_{26}N_2O_2$). The compound crystallizes in OrthorhombicPna21space group with unit cell parameters at 296(2) K as follows: a = 9.1034(5) Å, b =10.552(6) Å, c =11.769(8) Å, α = β = γ = 90°.Crystal data were collected using BRUKER SMART APEX II CCD X-ray diffractometer. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures to the final R_1 of 0.060 using SHELXL programs.
Key Words: acetamide and crystal structure.

Introduction

Schiff base compounds are some of the most important stereochemical models in transition metal coordination chemistry,with their ease of preparation and structural variations[1]. In continuation of our works on the synthesisand structural characterization of Schiff base ligands here we report the structure of the title compound.

Experimental

X-ray Structure Determination

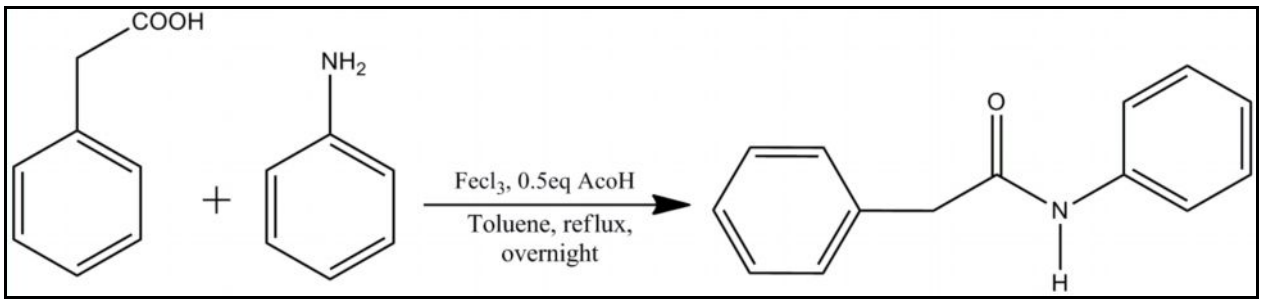
Single crystal of the compound suitable for x-ray diffraction was obtained by slow evaporationmethod. Three dimensional intensity data were collected on a Bruker[2] SMART APEX CCD Diffractometer using graphite monochromatized Mo-Kα radiation (λ= 0.71073 Å) at Department of chemistry, IIT, Chennai, India. The structure was solved by direct methods and refined on F^2 by full-matrix least-squares procedures using the SHELXL programs[3]. All the non-hydrogen atoms were refined using isotropic and later anisotropic thermal parameters. The hydrogen atoms were included in the structure factor calculation at idealized positions by using a riding model, but not refined. Images were created with ORTEP-3[4].The crystallographic data for the compound are listed in Table 1.

Table 1: Crystal data and structure refinement of the titled compound

Compound	**Parameters**
Empirical formula	C_{28} H_{26} N_2 O_2
Formula weight	422.51
Temperature	293(2) K
Wavelength	0.71073 Å
Crystal system, space group	Pna21, Orthorhombic
Unit cell dimensions	a = 9.1034(5) Å alpha = 90°
	b = 10.5529(6) Å beta = 90°
	c = 11.7699(8) Å gamma = 90°
Volume	1130.70(12) $Å^3$
Z, Calculated density	2, 1.241 Mg/m^3
Absorption coefficient	0.078 mm^{-1}
F(000)	448
Crystal size	0.25 x 0.16 x 0.10 mm
Theta range for data collection	2.59 to 24.99 deg.
Limiting indices	-10<=h<=8, -12<=k<=12, -13<=l<=8
Reflections collected / unique	4641 / 1507 [R(int) = 0.0204]
Completeness to theta = 24.99	99.90%
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	1507 / 1 / 145
Goodness-of-fit on F^2	1.083
Final R indices [I>2sigma(I)]	**R1 = 0.0609**, wR2 = 0.1505
R indices (all data)	R1 = 0.0626, wR2 = 0.1541
Largest diff. peak and hole	0.322 and -0.477 $e.Å^{-3}$

Synthesis of the compound

To a solution of 2-phenylacetic acid (1.0 mmol) in toluene (6 mL) 20 mol% $FeCl_3$ and an additional0.5 eq. of AcOH were added and the reaction mixture was stirred at 50°C for 10–15 min. Then an aniline (1.0 mmol)in toluene (4 mL) was added slowly to the above reaction mixture. The reaction mixture was refluxed at 75°C and the reaction was completed overnight monitored by TLC. Upon complete consumption of the starting materials, the reaction mixture was filtered and the filtrate was evaporated under reduced pressure. The crude product was extracted into EtOAc (15 mL). The EtOAc layer was then washed with 5% Na_2CO_3 (5 mL), dil. HCl (5 mL), water (2 X5 mL) and brine (5 mL). The organic layer was dried over anhydrous Na_2SO_4 and the solvent was evaporated in vacuum to afford the crude which was then purified through silica gel column chromatography (EtOAc/hexane). Then the product was dissolved in EtoAc and do warm-up and kept in RT, finally we got yellow colored crystal. The scheme diagram is given below.



Results and Discussion

The Asymmetric unit of the title compound is shown in Fig. 1. The two phenyl rings are essentially coplanar,making a dihedral angle of 23.14°.The carbamate group is twisted slightly from the attached benzene ring, with a C—N—C—O torsion angle of 1.1(5)°.

In the crystal, molecules are linked by pairs of N---H...O hydrogen bonds, forming inversion dimers with an $R^2_2(6)$ ring motif. The dimers are linked by pairs of C---H...O hydrogen bonds, enclosing $R^2_2(6)$ ring motif, forming chains along [010](Fig 2& Table 2). The crystal packing is further stabilized by C---H...***π***and ***π***---***π***intermolecular interactions.The selected bond lengths and angles are listed in table 3 and 4, respectively.

Table 2: Hydrogen-bond geometry [Å]

Symmetry code: i) -1/2+x,1/2-y,z

Distance (Å)				Angle (º)
D—H…A	D—H	H…A	D…A	D—H…A
N1---H1A…O1^{i}	0.86	1.98	2.799(3)	160
C8---H8A…O1^{ii}	0.97	2.59	2.922(3)	100
C8---H8B…O1^{iii}	0.97	2.44	2.922(3)	111

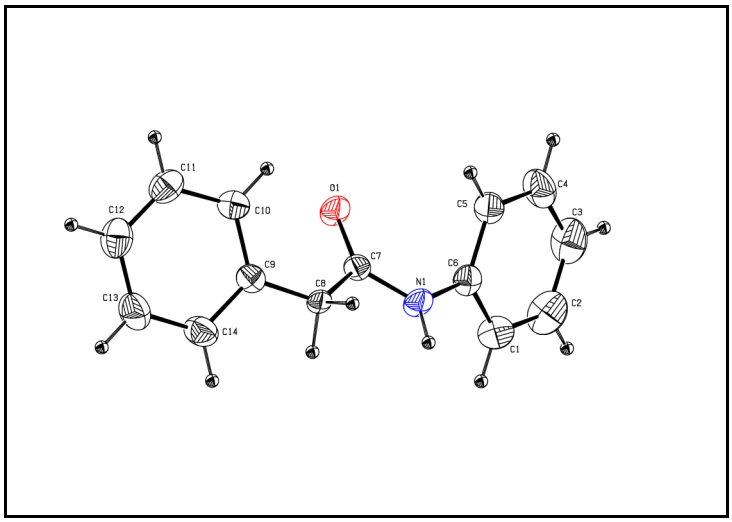


Fig.1.The molecular structure of the title compound, with the atom-numbering scheme. The displacement ellipsoids are drawn at 30% probability level. H atoms are shown as spheres of arbitrary radius.

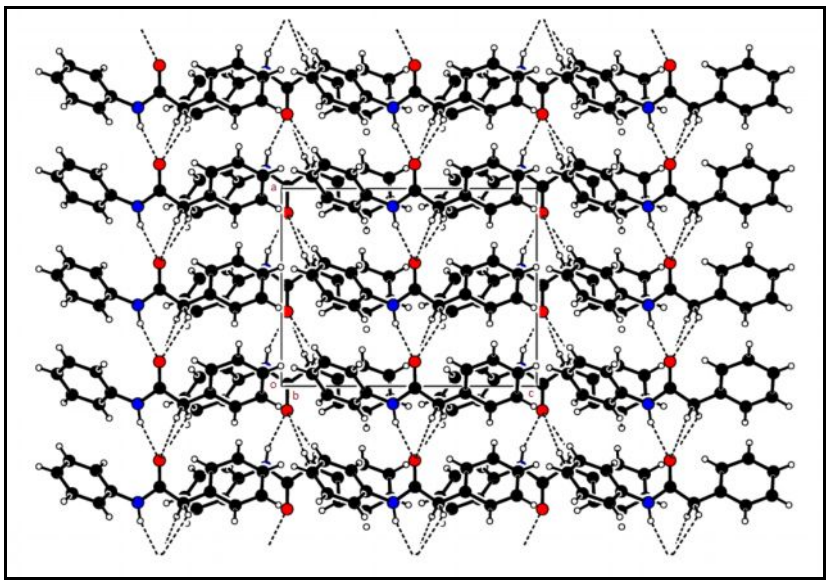


Fig.2. The crystal packing of the title compound, viewed along b axis, showing N---H...O and C---H…O hydrogen bonds.The hydrogen bonds are shown as dashed lines(see Table 2 for details).

Table 3: Selected Bond lengths (Å)Table 4: Selected Bond angles (º)

Bond	Length (Å)
C(1)-C(6)	1.369(5)
C(1)-C(2)	1.394(6)
C(1)-H(1)	0.93
C(2)-C(3)	1.377(7)
C(2)-H(2)	0.93
C(3)-C(4)	1.363(7)
C(3)-H(3)	0.93
C(4)-C(5)	1.399(5)
C(4)-H(4)	0.93
C(5)-C(6)	1.381(5)
C(5)-H(5)	0.93
C(6)-N(1)	1.419(4)
C(7)-O(1)	1.224(3)
C(7)-N(1)	1.337(4)
C(7)-C(8)	1.371(4)
C(8)-C(9)	1.417(4)
C(8)-H(8A)	0.97
C(8)-H(8B)	0.97
C(9)-C(14)	1.376(5)
C(9)-C(10)	1.386(4)
C(10)-C(11)	1.373(5)
C(10)-H(10)	0.93
C(11)-C(12)	1.354(5)
C(11)-H(11)	0.93
C(12)-C(13)	1.381(6)
C(12)-H(12)	0.93
C(13)-C(14)	1.371(6)
C(13)-H(13)	0.93
C(14)-H(14)	0.93
N(1)-H(1A)	0.86

Bond	Angle (º)
C(6)-C(1)-C(2)	119.7(4)
C(6)-C(1)-H(1)	120.1
C(2)-C(1)-H(1)	120.1
C(3)-C(2)-C(1)	120.3(4)
C(3)-C(2)-H(2)	119.9
C(1)-C(2)-H(2)	119.9
C(4)-C(3)-C(2)	119.8(4)
C(4)-C(3)-H(3)	120.1
C(2)-C(3)-H(3)	120.1
C(3)-C(4)-C(5)	120.5(4)
C(3)-C(4)-H(4)	119.8
C(5)-C(4)-H(4)	119.8
C(6)-C(5)-C(4)	119.3(3)
C(6)-C(5)-H(5)	120.3
C(4)-C(5)-H(5)	120.3
C(1)-C(6)-C(5)	120.3(3)
C(1)-C(6)-N(1)	118.5(3)
C(5)-C(6)-N(1)	121.1(3)
O(1)-C(7)-N(1)	124.3(3)
O(1)-C(7)-C(8)	121.8(3)
N(1)-C(7)-C(8)	113.9(2)
C(7)-C(8)-C(9)	125.2(2)
C(7)-C(8)-H(8A)	106
C(9)-C(8)-H(8A)	106
C(7)-C(8)-H(8B)	106
C(9)-C(8)-H(8B)	106
H(8A)-C(8)-H(8B)	106.3
C(14)-C(9)-C(10)	119.5(3)

Conclusion

The crystal structure analysis of a novel N,2-diphenylacetamidecompound was studied using x-ray diffraction method. In the crystal, molecules are linked by pairs of N---H...O hydrogen bonds, forming inversion dimers with an $R^2_2(6)$ ring motif. The dimers are linked by pairs of C---H...O hydrogen bonds, enclosing $R^2_2(6)$ ring motif, forming chains along [010]

Acknowledgments

The authors thank the Department of chemistry, IIT, Chennai, India, for X-ray intensity data collection.

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