



NMR, X-RAY AND IR STUDIES OF (E)-3-(PHENYLIMINO)PETAN-2-ONE {ArN(CH₂(C-Me)₂O} (Ar = C₆H₅)

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ABSTRACT

A range of new Schiff bias compounds such (E)-3-(phenylimino)petan-2-one {ArN(CH₂(C-Me)₂O} (Ar = C₆H₅) contain the chelating imino and oxygen in position α - has been prepared, and structurally characterized in several cases. Well-defined (E)-3-(phenylimino)petan-2-one {ArN(CH₂(C-Me)₂O} (Ar = C₆H₅) was obtained by stoichiometric addition of {ArNH₂} (Ar = C₆H₅) and {CO(CH(CH₃))₂CH₂CO} in n-propanol at reflux temperature. IR, NMR and X-ray studies have been obtained for this compound.

Keywords: Schiff bias compounds, (E)-3-(phenylimino)petan-2-one, chelating imino and oxygen, IR, NMR and X-ray studies.

INTRODUCTION

Schiff bias serial have been focus in the last few years ago, especially after reactions with transition metals to forward complexes. Copolymerization of ethylene and amides are widespread in the literature, but this interest is largely confined to the past ten years. Gibson et al. have reported the synthesis, characterisation, and the formation of coordinated mono-alkyl magnesium complexes by reaction of β -diketimine with dimethylmagnesium in 1:1 molar ratio in diethyl ether.¹ Also Qing and his group reported the synthesis β -diketiminato ligands, which used as ligand with titanium salt to offered bis(β -diketiminato)titanium complexes [ArNC(CR₃)(CHC(CR₃)NAr)]₂TiCl₂ (R= F, Ar = 2,6-diisopropylphenyl) and then were examined as catalysis for polymerisation.² A new 1,4,5,8-naphthalendiimide group and its Pt(II) phosphine complexes are reported by Julia et al.³ A serial of amido-imine bisphenol Schiff-base reacted with manganese salt to offered Manganese amido-imine bisphenol hangman complexes were prepared by Nocera et al.⁴

EXPERIMENTAL

Synthesis of (E)-3-(phenylimino)petan-2-one (3)

To a solution of aniline (2 g, 21 mmol) (1) in n-propanol (30 ml) and then (2.15 g, 21 mmol) of n-bromosecinimide (2) in solution was added wise in room temperature. The reaction mixture was stirred with reflux overnight. The mixture was concentrated in vacuum and then was dried over sodium sulphate anhydrite. Colourless crystals were obtained after crystallisation from Et₂O at room temperature (2.0 g, 11.283 mmol, 50%).

Characterising data for **3**: ¹H NMR (Chloroform-*d*), δ_H 12.7 (s, 1H, HN-Ar), 7.15 (m, 5H, Ar), 5.2 (s, 2H, CH), 2.2 (s, 3H, CH₃), 1.9 (s, 3H, CH₃).

¹³C NMR (chloroform-*d*) δ_C 155.3 (C=O), 140(C(NHAr)), 130, 125, 123 (C₆H₃NH), 90 (NC(Me)CH₂C(Me)CO), 30, 25 (2Me).

Spectroscopic Techniques:

¹H, ¹³C NMR spectra of **3** was acquired at El-Manar university Tunisia on a Bruker Spectrospin AG 300DPX and operating at 300.13 MHz for ¹H measurements, 75.47 MHz for ¹³C measurements.

In solution **3** behaviour as shown in room temperature ^1H NMR spectrum in chloroform- d_1 . The peak arising from the ArNH group are resolvable ($\delta = 12.7$ ppm) suggesting that they are joint to the $\text{C}(\text{Me})\text{CH}_2\text{C}(\text{Me})\text{O}$. Also other important peaks are arising as given above. In meantime carbon 13 showed the pick at 140 which is indicate to $\text{C}(\text{NHAr})$.

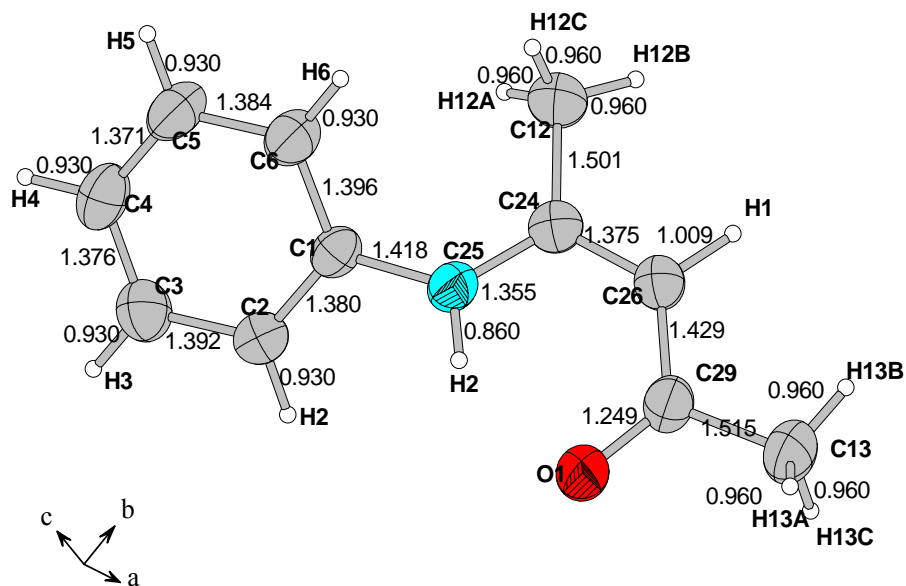


Fig.-2: Molecular structure of **3**.

Table-1: Selected bond lengths and angles in **3**

Bond	Length/(Å)	Fragment	Angle/($^\circ$)
O1—C29	1.252 (2)	C24—C25—C1	131.21 (16)
C25—C24	1.357 (2)	C25—C24—N26	120.07 (17)
C25—C1	1.417 (2)	C25—C24—C12	120.02 (16)
C24—N26	1.373 (3)	N26—C24—C12	119.86 (18)
C24—C12	1.502 (3)	C24—N26—C29	124.74 (18)
N26—C29	1.430 (3)	O1—C29—N26	123.49 (18)
C29—C13	1.512 (3)	O1—C29—C13	118.66 (18)
C1—C2	1.383 (3)	N26—C29—C13	117.84 (18)

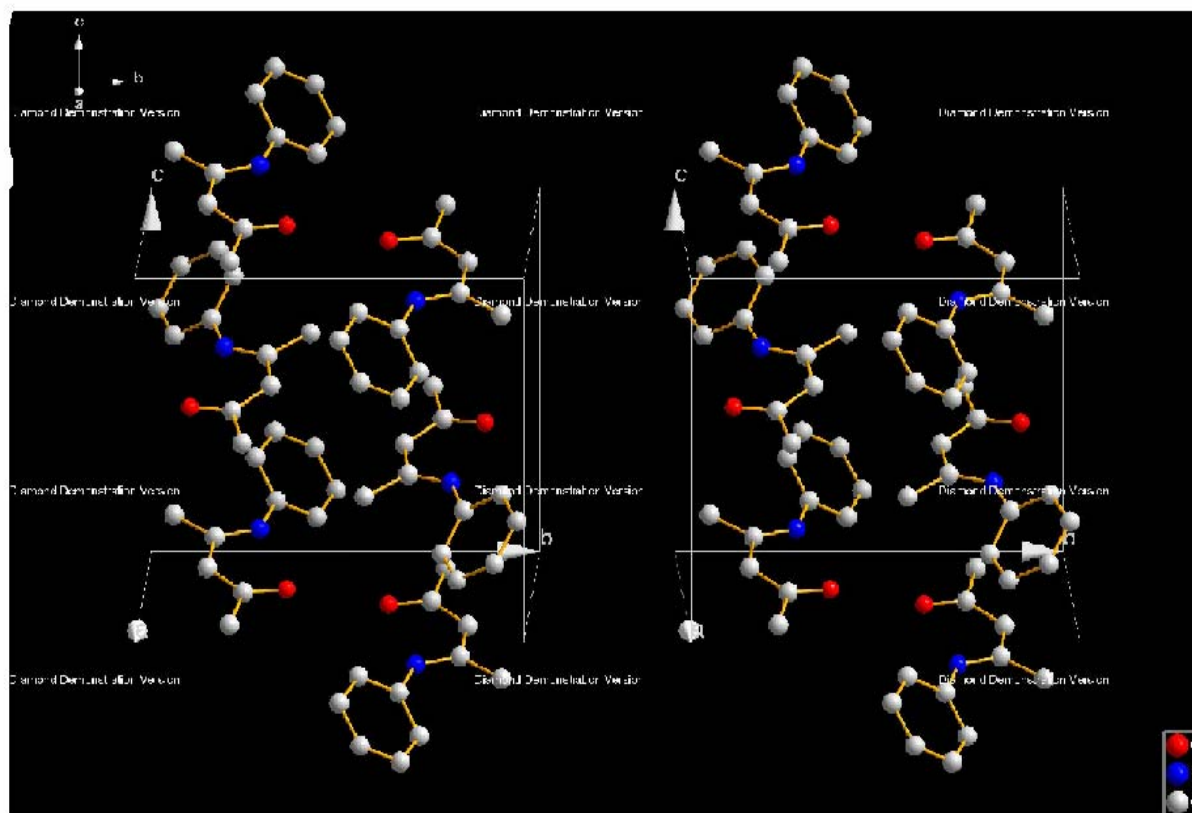


Fig.-3: Molecular packing of **3**.

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