

Synthesis of N – Hydroxy – 3, 3 – Dimethyl – 2, 6 – Diaryl Piperidin – 4 – One Thiosemi Carbazone

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ABSTRACT

N – hydroxyl – 3, 3 – dimethyl – 2, 6 – diaryl piperidin – 4 – one thiosemicarbazone is synthesized by the reaction of *N* – hydroxyl – 3, 3 – dimethyl – 2, 6 – diaryl piperidin – 4 – one oxime & thiosemicarbazide in presence of (SiO₂ & DCC) silicadioxide & dicyclohexyl carbodimide heterogeneous catalyst under microwave irradiation. The structure of the thiosemicarbazone is confirmed by elemental analysis, ¹H NMR and ¹³C NMR spectral studies.

KEYWORDS : *N* – hydroxyl – 3, 3 – dimethyl – 2, 6 – diaryl piperidin – 4 – one thiosemicarbazone, ¹H and ¹³C NMR.

INTRODUCTION

In this method of synthesis of thio semicarbazone by microwave irradiation in presence of heterogeneous catalyst SiO₂ – DCC (Dicyclohexyl Carbodiimide) giving maximum yield of products¹⁻³.

Synthesis and spectral studies of *N* – hydroxyl – 3, 3 – dimethyl – 2, 6 – diarylpiperidin – 4 – one thiosemicarbazones

When *N* – hydroxyl – 3, 3 – dimethyl – 2, 6 – diarylpiperidin – 4 – one is treated with thiosemicarbazide in the presence of heterogeneous catalyst SiO₂ – Dicyclohexylcarbodiimide (DCC) couple in solvent free conditions under microwave irradiation (scheme – 11), thiosemicarbazone are formed.

The products are identified by ¹H and ¹³C NMR spectral analysis. Here the conformation of *N* – hydroxyl – 3, 3 – dimethyl – 2, 6 – diarylpiperidin – 4 – one thiosemicarbazones are discussed. The preferred conformation is proposed by analysis of the coupling constant (Fig. 116a).

Analysis of ¹H NMR spectrum

The ¹H NMR spectrum is displayed in Plate 24. The signal at 2.59 – 2.63 ppm is assigned to H_{3a} (*syn* α). The anti β hydrogen (H_{2a}) any *syn* β hydrogen (H_{6a}) appear at 3.67 and 3.73 ppm respectively. The NH₂ protons appear at 6.38 ppm, the CSNH proton appears at 8.57 ppm.

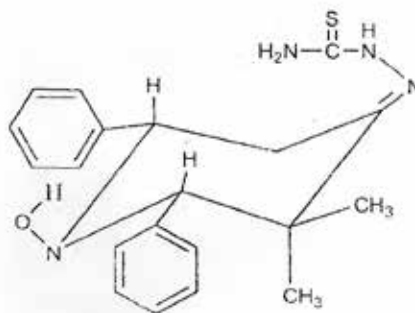
The N-OH proton appears at 4.53 ppm. Two singlets appeared at 0.94 and 1.24 ppm are due to methyl protons at C – 3. The multiplet in the region of 7.26 – 7.50 ppm is assigned to the aromatic protons.

A double doublet is observed at 2.56 – 2.63 ppm, another double doublet at 3.72 – 3.76 ppm and the values are 2.83 and 13.28 Hz and three lines are observed at 2.34 ppm.

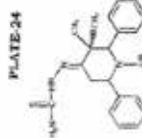
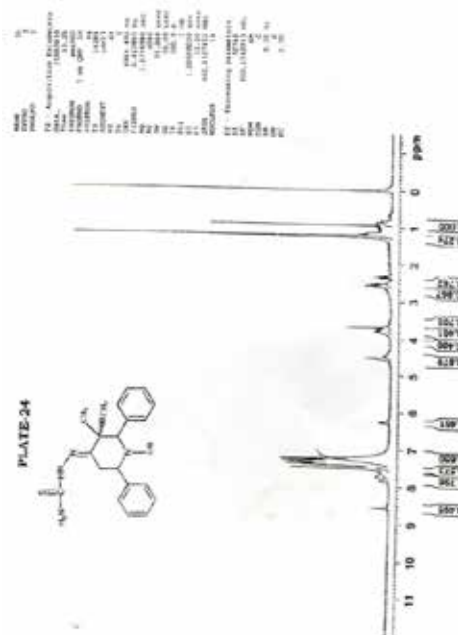
Analysis of ¹³C NMR spectrum

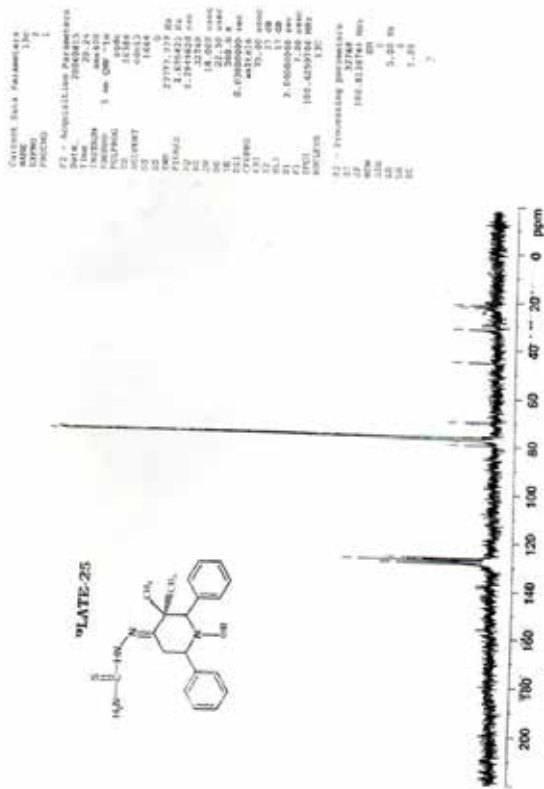
The ¹³C NMR spectrum is displayed in Plate 25. The ¹³C NMR spectrum shows that the C-5 (*syn* α) carbon appears at 31.5 ppm. The *syn* β (C-6) carbon appear at 70.1 ppm. The *anti* α (C-3) and *anti* β (C-2) carbons appear at 45.5 and 79.5 ppm respectively. The signal at 156.1 ppm is for C-4 carbon, the two *ipso* carbons appear at 138.1 and 143.5 ppm respectively.

The signal at 21.4 and 22.4 ppm is due to methyl carbons at C – 3. The aromatic carbons appear in the range of 126.9 – 128.8 ppm. The signal at 179.6 ppm is due to C = S.



116a





CONCLUSION:

The yields of N - bydroxy - 3, 3 - dimethyl - 2, 6 - diphenyl piperidin - 4 - one thiosemicarbazone are more than 85%. The coupling constant values reveal that the compound mainly exists in chair conformation.

REFERENCES

1. Fischer and Tafel, Ann., 1885, 227, 324. | 2. R.C. Elderfield, Heterocyclic Compounds, Vol.V, New York, John Wiley & Sons, INC, 365. | 3. Utzinger and Hoelle, Helv. Chim. Acta, 1952, 35,1370, 2054.