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## Synthesis and Characterisation of new Spiro Heterocycles

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### ABSTRACT

Heterocyclic compounds containing  $\beta$ -lactum structure with different hetero atoms are found to show biological activities<sup>1</sup>. In the present work, it has been synthesized the semicarbozone and thiosemicarbozone derivatives of 3-methyl-2,6-diphenylpiperidin-4-one followed by cyclisation to get the spiro compounds. These spiro compounds. The spiro products have been characterized by C,H,M analysis, IR <sup>1</sup>H-NMR and <sup>13</sup>C-NMR spectral data.

**Key words:** Synthesis, Spiro Heterocycles,  $\beta$ -lactum.

### INTRODUCTION

Owing to their pharmacological and other biological activities heterocyclic compounds gain importance in the chemical research. Alkaloids are pharmacologically important class of compounds because they contain piperidine ring as heterocycle<sup>1</sup>. Likewise, heterocyclic compounds containing  $\beta$ -lactum structure with different hetero atoms are found to show a variety of biodynamic activities against bacteria and virus.

The main objective of this work is to synthesize some new type of heterocyclic compounds containing nitrogen and sulphur in the nucleus with varying nature of substituents as side chain and to characterize their structure. The structures confirmed may be, in a way, helpful in the study of their pharmacological behaviour.

### MATERIAL AND METHODS

Piperidin-4-one was prepared in the laboratory based on the literature method<sup>3-6</sup>. Semicarbazide hydrochloride and thiosemicarbazones were supplied by Himedia, Mumbai. TLC was carried out on a silica gel coated plates. IR spectra were recorded on a Shimadzu IR-Affinity-1 instrument and <sup>1</sup>H- and <sup>13</sup>C-NMR spectra on Bruker 300 MHz spectrometer.

#### Synthesis of N-(1-acetyl-7-9-diphenyl-4-oxa-1,2,8-tri aza spiro [4,5] dec-2-en-3yl) acetamide (d)<sup>2</sup>

15.4 g of 2,6-diphenylpiperidin-4-one semicarbazone in 5ml of pyridine and 1.5 ml of acetic anhydride mixture was heated over a water bath at 100°C with magnetic stirring. The heating was continued for 2 hours. Then the reaction mixture

was poured into ice water. White coloured solid of (1-acetyl-7-9-diphenyl-4-oxa-1,2,8-tri aza spiro<sup>4,5</sup> dec-2-en-3yl) acetamide was separated filtered and dried. Then it was recrystallized from ethanol. Melting point: 128°C.

IR (cm<sup>-1</sup>): 3400 (NH str.), 3065 (Aromatic C-H str.), 1701 (C=O str.), 1409 (C=N str.), 1296 (C-N str.), 700 (Cyclic C-S)

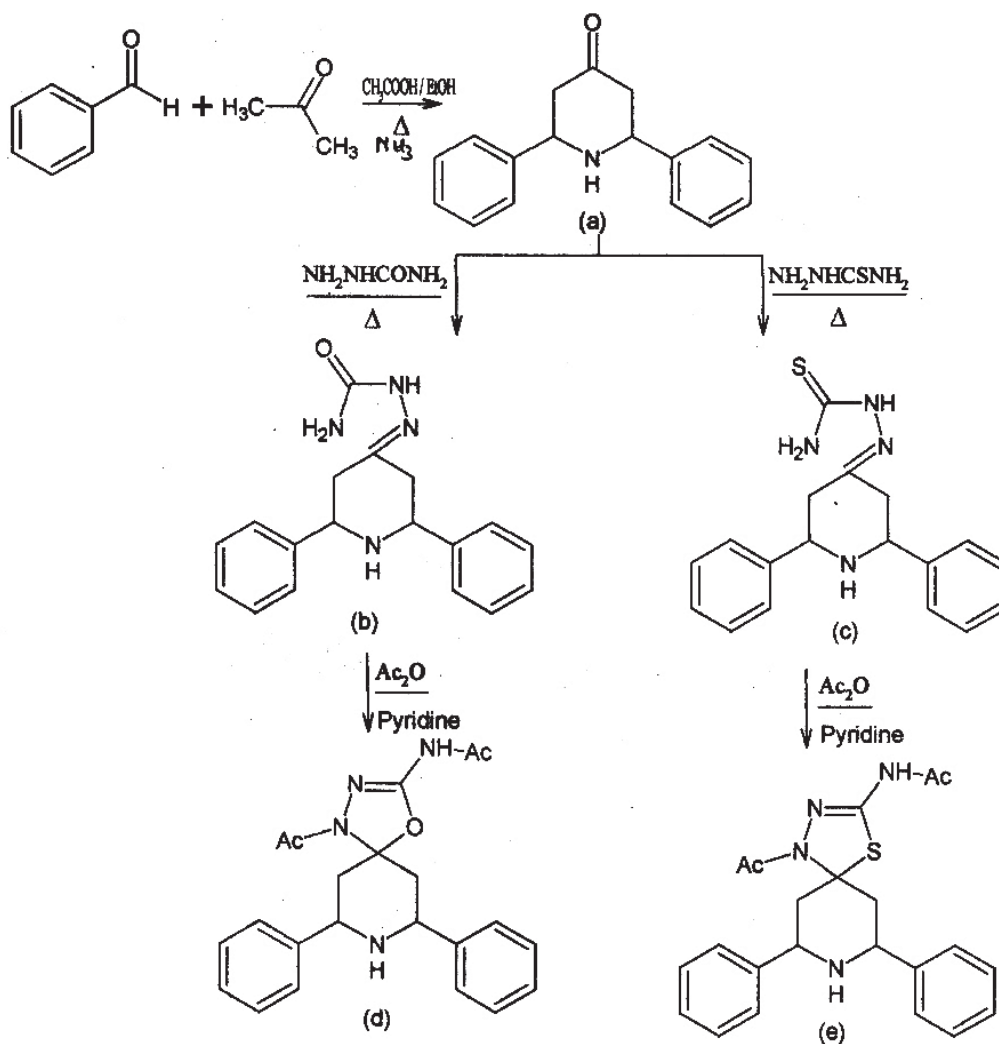
<sup>1</sup>H NMR (ppm): 7.35 - 7.02 (10 H, m, aromatic-H), 6.2 (1H, s, NH-amide), 3.02 (2H, m, benzylic-H of piperidine), 2.08 (4H, m, methylene-

H at C-3 and C-5 of piperidine) 1.7 (1H, s, NH of piperidine), 1.2 (6H, s, acetyl CH<sub>3</sub>).

<sup>13</sup>C NMR (pm): 172 (CO of acetyl group), 128-127 (aromatic-Carbons), 49 (C-3 and C-5 of piperidine), 23.0 (CH<sub>3</sub> of acetyl group).

### Synthesis of N-(1-acetyl-7-9-diphenyl-4-thia-1,2,8-tri aza spiro [4,5] dec-2-en-3yl) acetamide (e)<sup>2</sup>

15.4 g of 2,6-diphenylpiperidin-4-one thiosemicarbazone in 5ml of pyridine and 1.5 ml of acetic anhydride mixture was heated over a water bath at



Scheme

100°C with magnetic stirring. The heating was continued for 2 hours. Then the reaction mixture was poured into ice water, white coloured solid of (1-acetyl-7-9-diphenyl-4-thio-1,2,8-tri aza spiro<sup>4,5</sup> dec-2-en-3yl) acetamide was separated filtered and dried. Then it was recrystallized from ethanol. Melting point: 124°C.

IR (cm<sup>-1</sup>): 3400 (NH str.), 3030 (aromatic C-H), 1602 (C=N str.), 1328 (C-N str.), 1150 (C-CO), 698 (Cyclic C-N str.)

<sup>1</sup>H NMR (ppm): 7.7-7.0 (10 H, m, aromatic-H), 5.9(1H, s, NH-amide), 4.39-3.8 (2H, m, C-2 and C-6 protons of piperidine), 2.49-2.10(4H, m, C-3 and C-5 protons of piperidine) 1.9 (1H, s, NH of piperidine), 1.2 (6H, s, acetyl CH<sub>3</sub>).

<sup>13</sup>C NMR (pm): 176 (CO of acetyl group), 143.0 (C-N), 128-126 (aromatic-c), 60.1 (C-3 and C-5 of piperidine), 23-34 (CH<sub>3</sub> of acetyl group).

## RESULTS AND DISCUSSION

The preliminary laboratory analysis and C,H,N analysis revealed the formation of the compound (d). The IR data showed the characteristics stretching frequencies as follows<sup>7</sup>. 3400 cm<sup>-1</sup> for NH, 3065 cm<sup>-1</sup> for aromatic CH, 1701 cm<sup>-1</sup> for carbonyl group and 1490 cm<sup>-1</sup> for C=N stretching besides other characteristic frequency.

The <sup>1</sup>H NMR data supported the structure of the compounds as predicted (d) in the scheme i.e. δ 7.55-7.02 corresponds to aromatic hydrogens δ 6.2 corresponds to NH of amide group, δ 3.02 and δ 2.08 are assigned to the ring protons of piperidine, δ 1.7 and δ 1.2 are assigned to NH proton of piperidine and acetyl protons of spiro ring substituent respectively. The various peak assignments of <sup>13</sup>C-NMR spectrum also confirm the assigned structure to compound (d).

Similarly the formation of compound (e) also was revealed from preliminary laboratory analysis and C,H,N analysis. The IR data showed the characteristic stretching frequencies as follows<sup>7</sup>. 3400 cm<sup>-1</sup> for 3030 cm<sup>-1</sup> for aromatic CH, 1630 for carbonyl group and 1602 cm<sup>-1</sup> for C=N stretching besides other characteristic frequencies. The <sup>1</sup>H NMR data supported the structure of the compound as predicted (e) in the Scheme i.e. δ 7.7-7 corresponds to aromatic hydrogens δ 5.9 corresponds to NH of amide group. δ 4.32-3.8 are assigned to the methine protons of piperidine, δ 2.49-2.10 corresponds to methylene protons of piperidine, δ 1.19 is assigned to NH protons of piperidine and δ 1.2 corresponds to acetyl protons of spiro ring substituent. The various peak assignments of <sup>13</sup>C-NMR spectrum also confirm the assigned structure to compound (e). The <sup>1</sup>H-NMR and <sup>13</sup>C-NMR data were assigned to the compounds with the help of chem office 2005.

## REFERENCES

- Jayaraman, R.S, Avila, *Chem. Rev.* **81**: 1499 (1981).
- Fazal Mohamed, M.I., Seenii Mubarak, M., *Indian. J. Heterocyclic Chem.* **18**: 19 (2008).
- Noller. C., Baliah. V., *J. Am. Chem. Soc.* **70**: 3853 (1948).
- Baliah V., Govindarajan T.S., *Curr. Sci.* **23**: 91 (1954).
- Baliah V, Gopalakrishanan J., Govindarajan, T.S., *J. Indian. Chem. Soc.* **31**: 832 (1954).
- Baliah, V., Ekambaram, A., Govindarajan, T.S., *Curr. Sci.* **23**: 264 (1954).
- Silverstein, Bessler and Morill, *Spectroscopic Identification of Organic Compounds*, 4<sup>th</sup> Edition, (1981).