

# Contents

|                  |   |                       |
|------------------|---|-----------------------|
| <b>Chapter 1</b> | <b>Theoretical approach and Review work</b>                                       | <b>Page no (1-34)</b> |
|                  | 1,3-dipole  | 2                     |
|                  | HMO calculation   | 2 - 5                 |
|                  | Reactivity profile of 1, 3-dipoles  | 6                     |
|                  | Nomenclature  | 7                     |
|                  | Synthesis of nitrones   | 10 - 13               |
|                  | 1,3-dipolar cycloaddition reaction  | 13 - 17               |
|                  | Stereoselectivity in nitronene cycloaddition                                      | 17                    |
|                  | Current literature survey   | 18 - 31               |
|                  | Reference   | 32 - 34               |
| <b>Chapter 2</b> | <b>Experimental section</b>   | <b>(35 – 91)</b>      |
| <b>I</b>         | General method for Preparation of chlorohydrin and its tautomer                   | 35 - 36<br>36 - 37    |
|                  | General method for Preparation of <i>N</i> -phenyl- $\alpha$ -chloronitrone (1)   |                       |
|                  | <b>Reaction of nitrone 1 with alkynes</b>   |                       |
|                  | 1. Ethyl propiolate cycloadduct   | 37 - 38               |
|                  | 2. Dimethyl acetylene dicarboxylate cycloadduct                                   | 38 - 39               |
|                  | 3. Phenyl methyl propiolate cycloadduct   | 40 - 41               |
|                  | 4. Acetylene dicarboxylic acid cycloadduct  | 41 - 42               |
|                  | <b>Cycloaddition reaction of nitrone (1) with alkenes</b>                         | 42                    |
|                  | <b>Aqueous phase synthesis of</b>   |                       |
|                  | 5. Cyclohexene cycloadduct  | 43 - 44               |
|                  | 6. <i>N</i> -methyl maleimide cycloadduct   | 44 - 45               |
|                  | 7. <i>N</i> -phenyl maleimide cycloadduct   | 45 - 46               |
|                  | 8. <i>N</i> -cyclohexyl maleimide cycloadduct                                     | 47 - 48               |
|                  | 9. <i>p</i> -OMe- <i>N</i> -phenyl maleimide cycloadduct                          | 48 - 49               |
|                  | 10. Acenaphthylene cycloadduct  | 49 - 50               |
|                  | 11. Ethyl acrylate cycloadduct  | 51                    |
|                  | 12. Methyl vinyl ketone cycloadduct   | 52                    |
| <b>II</b>        | <b>General method for preparation of <i>N</i>-cyclohexyl-<math>\alpha</math>-</b> | <b>52</b>             |

|            |   |         |
|------------|---|---------|
|            | <b>amino nitron (2) at elevated temperature</b>   |         |
|            | <b>General method for preparation of nitron (2) at RT</b>   | 53 - 54 |
|            | <b>General method for cycloaddition of nitron (2) at elevated temperature</b>                     | 54      |
|            | <b>General method for cycloaddition of nitron (2) at RT with alkenes (Solvent less condition)</b> | 54      |
|            | Synthesis of  |         |
|            | 13. Cyclohexene cycloadduct   | 55      |
|            | 14. <i>N</i> -Phenyl maleimide cycloadduct  | 55 - 56 |
|            | 15. <i>N</i> -Methyl maleimide cycloadduct  | 56 - 57 |
|            | 16. <i>N</i> -Cyclohexyl maleimide cycloadduct  | 57 - 58 |
|            | 17. <i>P</i> -OMe- <i>N</i> -phenyl maleimide cycloadduct   | 58 - 59 |
|            | 18. Ethyl acrylate cycloadduct  | 59 - 60 |
|            | 19. Methyl vinyl ketone cycloadduct   | 60 - 61 |
|            | 20. Styrene cycloadduct   | 61      |
|            | 21. Acenaphthylene cycloadduct  | 62      |
|            | 22. <i>p</i> -benzoquinone cycloadduct  | 62 - 63 |
|            | <b>Cycloaddition reaction of nitron 2 with alkynes</b>  | 63      |
|            | Aqueous phase synthesis of  |         |
|            | 23. Phenyl methyl propiolate cycloadduct  | 64      |
|            | 24. Dimethyl acetylene dicarboxylate cycloadduct  | 64 - 65 |
|            | 25. Ethyl propiolate cycloadduct  | 65 - 66 |
|            | 26. Propiolic acid cycloadduct  | 66 - 67 |
|            | <b>1,3-dipolar cycloaddition reaction of nitron (2) with alkenes</b>                              | 67 - 68 |
|            | Aqueous phase Synthesis of  |         |
|            | 27. <i>N</i> -Phenyl maleimide cycloadduct  | 68 - 69 |
|            | 28. <i>N</i> -Methyl maleimide cycloadduct  | 69 - 70 |
|            | 29. <i>N</i> -Cyclohexyl maleimide cycloadduct  | 70 - 71 |
|            | 30. Styrene cycloadduct   | 71      |
|            | 31. Ethyl acrylate cycloadduct  | 72      |
|            | 32. Acenaphthylene cycloadduct  | 72 - 73 |
|            | 33. Tetrachloro ethylene cycloadduct  | 73 - 74 |
| <b>III</b> | <b>General method for preparation of <i>N</i>-phenyl-<math>\alpha</math>-amino nitron (3)</b>     | 74 - 75 |

|                  |   |                    |
|------------------|---|--------------------|
|                  | <b>1,3-dipolar cycloaddition reaction of nitron (3) at RT<br/>with alkenes</b>                                  |                    |
|                  | Aqueous phase synthesis of  |                    |
|                  | 34. <i>N</i> -ethyl maleimide cycloadduct   | 75 - 76            |
|                  | 35. <i>p</i> -OMe- <i>N</i> -phenyl maleimide cycloadduct   | 76 - 77            |
|                  | 36. Acenaphthylene cycloadduct  | 77 - 78            |
|                  | 37. Ethyl acrylate cycloadduct  | 78                 |
|                  | 38. Methyl vinyl ketone cycloadduct   | 79                 |
|                  | <b>General method for preparation of nitron (3) at RT<br/>with alkynes</b>                                      | 79 - 80            |
|                  | Synthesis of  |                    |
|                  | 37. Phenyl methyl propiolate cycloadduct  | 80                 |
|                  | 38. Dimethyl acetylene dicarboxylate cycloadduct  | 81                 |
| IV               | <b>Synthesis of aldehyde from alkyl halide using <math>\alpha</math>-<br/>chloro nitron as oxidizing agent.</b> | 81 - 83            |
|                  | <b>Cycloaddition reaction of side product (2) with <i>N</i>-<br/>phenyl-<math>\alpha</math>-chloro nitron</b>   | 83 - 84            |
|                  | <b>aldehyde synthesis from <math>\alpha</math>-amino nitron</b>   | 84 - 86            |
|                  | <b>General procedure for cycloaddition (for regioselective<br/><i>spiro</i> cycloadducts)</b>                   | 86 - 89            |
|                  | Reference   | 90 - 91            |
| <b>Chapter 3</b> | <b><i>Results and Discussion</i></b>  | <b>(92 - 133)</b>  |
|                  | Synthesis of <i>N</i> -phenyl- $\alpha$ -chloro nitron (1) from Pyran   | 92 - 93            |
|                  | Synthesis of <i>N</i> -cyclohexyl- $\alpha$ -amino nitron (2) from DMF  | 93 - 94            |
|                  | Synthesis of <i>N</i> -phenyl- $\alpha$ -amino nitron (3) from DMF  | 94                 |
|                  | Interpretation of the mass spectra for $\alpha$ -chloro nitron  | 108 - 113          |
|                  | Interpretation of the mass spectra for $\alpha$ -amino nitron   | 114 - 120          |
|                  | Interpretation of $^1\text{H}$ NMR spectra  | 121 - 128          |
|                  | Interpretation of $^{13}\text{C}$ NMR Spectra   | 129                |
|                  | Interpretation of other spectra   | 129 - 131          |
|                  | Reference   | 132 - 133          |
| <b>Chapter 4</b> | <b><i>Scope and Objectives</i></b>  | <b>(134 - 140)</b> |
|                  | Antibacterial screening   | 137                |
|                  | Reference   | 139 - 140          |
|                  | <b>ANNEXURE-1 <i>Corrigendum</i></b>  |                    |
|                  | <b>ANNEXURE-2 Published &amp; accepted (in press) papers</b>  |                    |