**Programme: M. Sc. Part-I (Chemistry)** 

**Course Code: OCC-402** 

Title of the Course: Laboratory course in Organic Chemistry

Number of Credits: 02 Effective from AY: 2018-19

|                    |   | 1   |
|--------------------|---|-----|
| Prerequisites      | Students should have exposure to common laboratory practices and techniques   |     |
| for the            | studied at F Y B Sc, S Y B Sc and T Y B Sc chemistry theory / practical       |     |
| course:            | courses.  |     |
| Course             | To translate certain theoretical concepts learnt earlier into experimental    |     |
| <b>Objectives:</b> | knowledge by providing hands on experience of basic laboratory techniques     |     |
|                    | required for organic syntheses.   |     |
|                    |   |     |
| Course             | Students shall gain the understanding of:                                     |     |
| <b>Outcomes:</b>   | 1. Stoichiometric requirements during organic syntheses.                      |     |
|                    | 2. Safe and good laboratory practices, handling laboratory glassware,         |     |
|                    | equipment and chemical reagents.  |     |
|                    | 3. Common laboratory techniques including reflux, distillation, steam         |     |
|                    | distillation, vacuum distillation, aqueous extraction, thin layer             |     |
|                    | chromatography (TLC)  |     |
| Content:           | 1. Introduction to laboratory equipments, apparatus and safety:               | 04  |
|                    | a) Use of common laboratory equipments like fume hoods, vacuum pumps,         | hr  |
|                    | weighing balance etc. to be explained to the students.                        |     |
|                    | b) Introduction to various types of quick fit joints and apparatus to the     |     |
|                    | students.   |     |
|                    | c) Discussion of Safety Techniques: i) Disposal of chemicals, ii) Usage of    |     |
|                    | protective equipments, iii) First aid, iv) Fire extinguishers, types of fire, |     |
|                    | d) Hazards of chemicals.  |     |
|                    | d) Hazards of Chemicais.  | 24  |
|                    | 2. Laboratory Techniques:   | hr  |
|                    | a. Simple distillation (any one): Toluene-dichloromethane mixture using       | 111 |
|                    | water   |     |
|                    | condenser, nitrobenzene and aniline using air condenser.                      |     |
|                    | b) Steam distillation (anyone): Separation of o-and p- nitrophenols,          |     |
|                    | naphthalene   |     |
|                    | from its suspension in water, clove oil from cloves.                          |     |
|                    | c) Crystallisation: Concept of induction of crystallization(any one):         |     |
|                    | i) Crystallisation of phthalic acid from hot water using fluted filter        |     |
|                    | paper and stemless funnel.  |     |
|                    | ii) Acetanilide from boiling water iii) Naphthalene from ethanol.             |     |
|                    | iv) Decolorisation and crystallization of brown sugar (sucrose) with          |     |
|                    | animal charcoal using gravity filtration.                                     |     |
|                    |   |     |
|                    | d) Sublimation: Simple or vacuum sublimation of camphor, naphthalene,         |     |
|                    | anthracene or succinic acid (any one).  |     |
|                    | e) Vacuum distillation (any one): o-dichlorobenzene, diphenyl ether. Also use |     |
|                    | of nomograph should be explained.   |     |
|                    | f) Thin layer Chromatography (any one): Separation of o and p-nitroanilines,  |     |
|                    | Separation of analgesic drugs, Separation of o and p-nitrophenols,            |     |
|                    |   |     |
| 1                  |   | 1   |

|              | 3. Organic synthesis (any four experiments):                                    | 16  |
|--------------|---|-----|
|              | a)  | hr  |
|              | Aliphatic electrophilic substitution: Preparation of iodoform from ethanol      | 111 |
|              | & acetone.  |     |
|              | b) Aromatic electrophilic substitution (anyone): Preparation of p-bromoacet-    |     |
|              | anilide, bromination of acetophenone to phenacyl bromide, nitration of          |     |
|              | napthathalene to 1-nitronaphthalene, nitration of benzaldehyde to 3-            |     |
|              | nitrobenzaldehdye.  |     |
|              | c) Oxidation of: i) Benzoic acid from toluene ii) Cyclohexanone from            |     |
|              | cyclohexanol, iii) isoborneol to camphor using Jones reagent (any one).         |     |
|              | d) Reduction (any one): Reduction of $o$ -nitroaniline to $o$ -phenylenediamine |     |
|              | using Sn/HCl; Reduction of p-nitro benzaldehyde to p-nitrobenzyl alcohol        |     |
|              | using NaBH <sub>4</sub>   |     |
|              | e) Bromination of an alcohol using CBr <sub>4</sub> / triphenylphosphine.       |     |
|              | f) Grignard reaction: Triphenylmethanol from benzoic acid ester or              |     |
|              | benzophenone. g)  |     |
|              | Aldol condensation: Dibenzal acetone from benzaldehyde                          |     |
|              | h) Acetoacetic ester condensation : Preparation of ethyl <i>n</i> -             |     |
|              | butylacetoacetate or ethyl acetoacetate.  |     |
|              | i) Cannizzaro reaction using 4-chlorobenzaldehyde as substrate.                 |     |
|              | j) Friedel Craft's reaction (any one): using toluene and succinic anhydride,    |     |
|              | resorcinol to resacetophenone, benzene and maleic anhydride to                  |     |
|              | $\beta$ -benzoylacrylic acid  |     |
|              | k) Solvent free preparation of coumarin by the Knoevenagel                      |     |
|              | condensation  |     |
|              | under MW irradiation.   |     |
|              | 1) Preparation of oxidizing agent (any one): Pyridinium chlorochromate-silica,  |     |
|              | pyridinium chlorochromate-alumina, MnO <sub>2</sub> .                           |     |
|              | m) Preparation of cuprous chloride.   |     |
|              |   |     |
|              | 3. Isolation from natural sources: (any one)                                    | 4hr |
|              | Caffeine from tea powder, piperine from pepper, cinnamaldehyde from             |     |
|              | cinnamon  |     |
|              |   |     |
| Pedagogy:    | Students should be given suitable pre- and post-lab assignments and             |     |
| G 6.         | explanation revising the theoretical aspects of laboratory experiments prior to |     |
|              | the conduct of each experiment. Each of the experiments should be done          |     |
|              | individually by the students.   |     |
|              |   |     |
| References / | 1. A.I. Vogel, A.R. Tatchell, B. S. Furniss, A.J. Hannaford,                    |     |
| Readings     | Vogel's Textbook of Practical Organic Chemistry, 5th Ed., Prentice Hall;        |     |
|              | 2011.   |     |
|              | 2. D. Pasto, C. Johnson and M. Miller, Experiments and Techniques in            |     |
|              | OrganicChemistry, 1st Ed., Prentice Hall, 1991.                                 |     |
|              | 3. L.F. Fieser, K.L. Williamson "Organic Experiments" 7th edition D. C.         |     |
|              | Heath, 1992.  |     |
|              | 4. K.L. Williamson, K.M. Masters, Macroscale and Microscale Organic             |     |

- Experiments, 6<sup>th</sup> Edition, Cengage Learning, 2010
- 5. R.K. Bansal, *Laboratory Manual in Organic Chemistry*, New Age International, 5<sup>th</sup> Edition, 2016.
- 6. S. Delvin, Green Chemistry, Sarup & Sons, 2005.
- 7. O.R. Rodig, C.E. Bell Jr. and A.K. Clark, *Organic Chemistry Laboratory Standard and Microscale Experiments*, Saunders College Publishing, 3<sup>rd</sup> edition, 2009.
- 8. J. Mohan, Organic Analytical Chemistry, Narosa Publishing House, 2014.