KUT 206/2 – Chemistry Practical I - Organic

Course Objective: 1) To become acquainted with the chemistry of functional groups and the principles of qualitative organic analysis. 2) To introduce the application of fractional distillation, gas chromatography, spectroscopic and extraction techniques in organic chemistry.

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| 1. Thin-Layer Chromatography (TLC) | • Separation of the components of the Analtech dye mixture provided  
• Calculation of the $R_f$ value of each of the dye spots  
• Identification of an unknown mixture of two white compounds | 3                           | • Acquire the basic skill and technique of TLC.  
• Know the principles and applications of TLC.                                                                                   |
| 2. Column Chromatography (CC)     | • Preparation of a chromatographic column  
• Separation of an unknown aromatic hydrocarbon (biphenyl or naphthalene or triphenylmethane) from 9-fluorenone using CC  
• Determination of the melting points of the two components recovered from the column  
• Identification of the aromatic compound from its melting point                                                                 | 3                           | • Acquire the basic skill and technique of CC.  
• Know the principles and applications of CC.  
• Recognize the advantages and disadvantages of CC in comparison with TLC.  
• Acquire the technique of melting point determination. |
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| 3. Qualitative Organic Analysis: Identification of Functional Groups | • Observation of the results/products expected from the classification tests for the detection of aldehydes, ketones, alcohols, phenols and amines  
• Observation of the result of the Hinsberg test on an unknown amine to determine if it is primary, secondary or tertiary  
• Interpretation of the IR spectrum of an unknown sample and comparison of the functional group(s) deduced from the results of the classification tests | 3 | • Know some of the important chemical and physical properties characteristic of many functional groups.  
• Recognize the characteristic functional group stretching frequencies and the advantages of using IR for the identification of functional groups.  
• Identify functional groups of organic compounds. |
| 4. Qualitative Organic Analysis: Identification of An Unknown | • Determination of the boiling point of an unknown compound  
• Determination of its functional group(s) from classification and solubility tests  
• Confirmation of the functional group(s) by IR  
• Small scale preparation of an appropriate derivative and determination of the melting point of the purified product  
• Identification of the unknown from data obtained above  
• Confirmation of its structure from its NMR spectrum | 6 | • Acquire the technique for the determination of the boiling point of a liquid.  
• Acquire the skill for a small scale preparation.  
• Interpret simple NMR spectra.  
• Identify the structure of a simple unknown organic compound. |
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| 5. Fractional Distillation; Gas Chromatography      | • Fractional distillation of a 1:1 mixture of acetone and ethanol  
• Determination of the relative amounts of acetone and ethanol in the initial distillate by GC  
• Evaluation of the efficiency of the fractionation column in this distillation                                                             | 3                           | • Set up a fractional column and carry out the distillation.  
• Know the basic principles of fractional distillation and gas chromatography.  
• Apply the GC peak areas for the quantification of the components in the distillate, taking into consideration the relative response factors.  
• Calculate the enrichment factor from the data on the initial distillate from a simple distillation.  
• Evaluate the number of theoretical plates (and HETP) hence the efficiency of the fractionation column.                              |
| 6. Isolation of Trimyristin from Nutmeg             | • Isolation of trimyristin from nutmeg by solvent extraction  
• Purification of trimyristin by recrystallization                                                                                                                                     | 3                           | • Know one of the techniques in natural product extraction.  
• Acquire the skill in microscale recrystallization.                                                                                                                                 |
| 7. Isolation of Caffeine from Tea                   | • Isolation of caffeine from tea  
• Purification of the product by sublimation                                                                                                                                                   | 3                           | • Be acquainted with one of the techniques for the extraction of an alkaloid from a plant material.  
• Have learnt to carry out a sublimation for the purification of a product.                                                                                                                 |
| 8. Sodium Borohydride Reduction of Camphor         | • Small scale reduction of camphor utilizing sodium borohydride  
• Calculation of the composition of isoborneol and borneol in the product by (i) GC and (ii) $^1$H-NMR utilizing difference in the chemical shifts of the proton attached to the carbon bearing the hydroxyl group | 3                           | • Write the mechanism for the reduction of camphor by sodium borohydride, recognizing that the stereochemical course of the reduction is controlled by steric factor.  
• Know that IR can be used to indicate if all the camphor has been reduced.                                                                                                                   |
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| 9. Selective Oxidation of Indene to Homophthalic Acid Using Aqueous Acidic Potassium Dichromate Solution | • A small scale oxidation of indene by potassium dichromate | 3 | • Handle hazardous chemicals such as concentrated sulphuric acid and potassium dichromate.  
• Balance the equation and calculate the number of moles and grams of potassium dichromate required for the reaction.  
• Understand what corresponds to 10 mol % of potassium dichromate in this reaction. |
| 10. Photoreduction of Benzophenone to Benzopinacol | • A photoreduction of benzophenone using direct sunlight | 3 | • Learn the principles of this photochemical process.  
• Write the mechanism for the photoreduction of benzophenone to benzopinacol.  
• Predict the products of photoreduction of aromatic ketones. |
| 11. Carbocationic Rearrangement of Benzopinacol to Benzopinacolone | • Rearrangement of benzopinacol to benzopinacolone in the presence of glacial acetic acid and iodine. | 3 | • Write the mechanism for the rearrangement of benzopinacol to benzopinacolone clearly indicating the function of the iodine added.  
• Predict the product of a pinacol-pinacolone rearrangement. |
| 12. Conjugate Addition to α,β-Unsaturated Carbonyl | • Preparation of maleanic acid from maleic anhydride and aniline.  
• Conversion of maleanic acid to N-phenylmaleimide.  
• Conjugate addition of aniline to N-phenylmaleimide to give 1-phenyl-3-phenylaminopyrrolidine-2, 5-dione | 3 | • Acquire the skill for a multi-step small scale synthesis.  
• Write the mechanisms for the three reactions.  
• Calculate the overall yield for the synthesis of 1-phenyl-3-phenylaminopyrrolidine-2, 5-dione. |
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| 13. Steam Distillation of Lemon Grass                  | • Identification of the major components of lemon grass by using GC.     | 3                           | • Learn the technique, principles and applications of GC.  
• Acquire the technique of the extraction of essential oil from plants.  
• Know the major component of lemon grass.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |
| 14. Kinetic and Thermodynamic Reaction Conditions      | • Observation of the concepts of the kinetic and thermodynamic reaction conditions | 3                           | • Know the product related to the kinetic and thermodynamic control of reaction.  
• Know the concept of recrystalization.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                           |
| **TOTAL**                                             |                                                                          | **45**                      |                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                             |