

## 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.

Experiment title	Content	Number of laboratory hours	Expected outcome – upon completion of these experiments, the student should be able to:
1. Thin-Layer Chromatography (TLC)	<ul style="list-style-type: none"><li>• Separation of the components of the Analtech dye mixture provided</li><li>• Calculation of the <math>R_f</math> value of each of the dye spots</li><li>• Identification of an unknown mixture of two white compounds</li></ul>	3	<ul style="list-style-type: none"><li>• Acquire the basic skill and technique of TLC.</li><li>• Know the principles and applications of TLC.</li></ul>
2. Column Chromatography (CC)	<ul style="list-style-type: none"><li>• Preparation of a chromatographic column</li><li>• Separation of an unknown aromatic hydrocarbon (biphenyl or naphthalene or triphenylmethane) from 9-fluorenone using CC</li><li>• Determination of the melting points of the two components recovered from the column</li><li>• Identification of the aromatic compound from its melting point</li></ul>	3	<ul style="list-style-type: none"><li>• Acquire the basic skill and technique of CC.</li><li>• Know the principles and applications of CC.</li><li>• Recognize the advantages and disadvantages of CC in comparison with TLC.</li><li>• Acquire the technique of melting point determination.</li></ul>

Experiment title	Content	Number of laboratory hours	Expected outcome – upon completion of these experiments, the student should be able to:
3. Qualitative Organic Analysis: Identification of Functional Groups	<ul style="list-style-type: none"> <li>• Observation of the results/products expected from the classification tests for the detection of aldehydes, ketones, alcohols, phenols and amines</li> <li>• Observation of the result of the Hinsberg test on an unknown amine to determine if it is primary, secondary or tertiary</li> <li>• Interpretation of the IR spectrum of an unknown sample and comparison of the functional group(s) deduced from the results of the classification tests</li> </ul>	3	<ul style="list-style-type: none"> <li>• Know some of the important chemical and physical properties characteristic of many functional groups.</li> <li>• Recognize the characteristic functional group stretching frequencies and the advantages of using IR for the identification of functional groups.</li> <li>• Identify functional groups of organic compounds.</li> </ul>
4. Qualitative Organic Analysis: Identification of An Unknown	<ul style="list-style-type: none"> <li>• Determination of the boiling point of an unknown compound</li> <li>• Determination of its functional group(s) from classification and solubility tests</li> <li>• Confirmation of the functional group(s) by IR</li> <li>• Small scale preparation of an appropriate derivative and determination of the melting point of the purified product</li> <li>• Identification of the unknown from data obtained above</li> <li>• Confirmation of its structure from its NMR spectrum</li> </ul>	6	<ul style="list-style-type: none"> <li>• Acquire the technique for the determination of the boiling point of a liquid.</li> <li>• Acquire the skill for a small scale preparation.</li> <li>• Interpret simple NMR spectra.</li> <li>• Identify the structure of a simple unknown organic compound.</li> </ul>

Experiment title	Content	Number of laboratory hours	Expected outcome – upon completion of these experiments, the student should be able to:
5. Fractional Distillation; Gas Chromatography	<ul style="list-style-type: none"> <li>Fractional distillation of a 1:1 mixture of acetone and ethanol</li> <li>Determination of the relative amounts of acetone and ethanol in the initial distillate by GC</li> <li>Evaluation of the efficiency of the fractionation column in this distillation</li> </ul>	3	<ul style="list-style-type: none"> <li>Set up a fractional column and carry out the distillation.</li> <li>Know the basic principles of fractional distillation and gas chromatography.</li> <li>Apply the GC peak areas for the quantification of the components in the distillate, taking into consideration the relative response factors.</li> <li>Calculate the enrichment factor from the data on the initial distillate from a simple distillation.</li> <li>Evaluate the number of theoretical plates (and HETP) hence the efficiency of the fractionation column.</li> </ul>
6. Isolation of Trimyristin from Nutmeg	<ul style="list-style-type: none"> <li>Isolation of trimyristin from nutmeg by solvent extraction</li> <li>Purification of trimyristin by recrystallization</li> </ul>	3	<ul style="list-style-type: none"> <li>Know one of the techniques in natural product extraction.</li> <li>Acquire the skill in microscale recrystallization.</li> </ul>
7. Isolation of Caffeine from Tea	<ul style="list-style-type: none"> <li>Isolation of caffeine from tea</li> <li>Purification of the product by sublimation</li> </ul>	3	<ul style="list-style-type: none"> <li>Be acquainted with one of the techniques for the extraction of an alkaloid from a plant material.</li> <li>Have learnt to carry out a sublimation for the purification of a product.</li> </ul>
8. Sodium Borohydride Reduction of Camphor	<ul style="list-style-type: none"> <li>Small scale reduction of camphor utilizing sodium borohydride</li> <li>Calculation of the composition of isborneol and borneol in the product by (i) GC and (ii) <sup>1</sup>H-NMR utilizing difference in the chemical shifts of the proton attached to the carbon bearing the hydroxyl group</li> </ul>	3	<ul style="list-style-type: none"> <li>Write the mechanism for the reduction of camphor by sodium borohydride, recognizing that the stereochemical course of the reduction is controlled by steric factor.</li> <li>Know that IR can be used to indicate if all the camphor has been reduced.</li> </ul>

Experiment title	Content	Number of laboratory hours	Expected outcome – upon completion of these experiments, the student should be able to:
9. Selective Oxidation of Indene to Homophthalic Acid Using Aqueous Acidic Potassium Dichromate Solution	<ul style="list-style-type: none"> <li>A small scale oxidation of indene by potassium dichromate</li> </ul>	3	<ul style="list-style-type: none"> <li>Handle hazardous chemicals such as concentrated sulphuric acid and potassium dichromate.</li> <li>Balance the equation and calculate the number of moles and grams of potassium dichromate required for the reaction.</li> <li>Understand what corresponds to 10 mol % of potassium dichromate in this reaction.</li> </ul>
10. Photoreduction of Benzophenone to Benzopinacol	<ul style="list-style-type: none"> <li>A photoreduction of benzophenone using direct sunlight</li> </ul>	3	<ul style="list-style-type: none"> <li>Learn the principles of this photochemical process.</li> <li>Write the mechanism for the photoreduction of benzophenone to benzopinacol.</li> <li>Predict the products of photoreduction of aromatic ketones.</li> </ul>
11. Carbocationic Rearrangement of Benzopinacol to Benzopinacolone	<ul style="list-style-type: none"> <li>Rearrangement of benzopinacol to benzopinacolone in the presence of glacial acetic acid and iodine.</li> </ul>	3	<ul style="list-style-type: none"> <li>Write the mechanism for the rearrangement of benzopinacol to benzopinacolone clearly indicating the function of the iodine added.</li> <li>Predict the product of a pinacol-pinacolone rearrangement.</li> </ul>
12. Conjugate Addition to $\alpha,\beta$ -Unsaturated Carbonyl	<ul style="list-style-type: none"> <li>Preparation of maleanilic acid from maleic anhydride and aniline.</li> <li>Conversion of maleanilic acid to <i>N</i>-phenylmaleimide.</li> <li>Conjugate addition of aniline to <i>N</i>-phenylmaleimide to give 1-phenyl-3-phenylaminopyrrolidine-2, 5-dione</li> </ul>	3	<ul style="list-style-type: none"> <li>Acquire the skill for a multi-step small scale synthesis.</li> <li>Write the mechanisms for the three reactions.</li> <li>Calculate the overall yield for the synthesis of 1-phenyl-3-phenylaminopyrrolidine-2, 5-dione.</li> </ul>

<b>Experiment title</b>	<b>Content</b>	<b>Number of laboratory hours</b>	<b>Expected outcome – upon completion of these experiments, the student should be able to:</b>
13. Steam Distillation of Lemon Grass	<ul style="list-style-type: none"> <li>• Identification of the major components of lemon grass by using GC.</li> </ul>	3	<ul style="list-style-type: none"> <li>• Learn the technique, principles and applications of GC.</li> <li>• Acquire the technique of the extraction of essential oil from plants.</li> <li>• Know the major component of lemon grass.</li> </ul>
14. Kinetic and Thermodynamic Reaction Conditions	<ul style="list-style-type: none"> <li>• Observation of the concepts of the kinetic and thermodynamic reaction conditions</li> </ul>	3	<ul style="list-style-type: none"> <li>• Know the product related to the kinetic and thermodynamic control of reaction.</li> <li>• Know the concept of recrystallization.</li> </ul>
<b>TOTAL</b>		<b>45</b>	