UNIVERSITI TEKNOLOGI MARA

CHM510: ANALYTICAL SEPARATION METHODS

Course Name (English)	ANALYTICAL SEPARATION METHODS APPROVED
Course Code	CHM510
MQF Credit	4
Course Description	This course will interactively engage students cognitively and scientifically in areas of chromatography, electrophoresis, and sample preparation. Students will define concepts, state and explain chromatographic theories, make decision and justification as to the possible outcome of an analysis, perform investigations via laboratory exercises and in writing, discuss results of investigations leading to its relation to the chromatographic principles or theories. The lecture session is used to discuss the instrumentation and application of each separation and sample preparation techniques. The outcomes shall be assessed through a variety of tools which include the final examination, tests, laboratory reports and laboratory skills.
Transferable Skills	Written Report Blended Learning
Teaching Methodologies	Lectures, Lab Work, Discussion
CLO	CLO1 Explain the concepts and theories in separation methods: gas chromatography, liquid chromatography, supercritical fluid chromatography, and electrophoresis. CLO2 Choose the suitable separation technique for a given analysis. CLO3 Conduct scientific experiments in areas of chromatography and sample preparation for chromatographic analysis CLO4 Summarise a written report of scientific experiments on chromatography and sample preparation for chromatographic analysis.
Pre-Requisite Courses	No course recommendations

Topics
1. 1.0 Chromatographic Separations 1.1) 1.1 General Introduction to Chromatography 1.2) Principles and Theory of Chromatography 1.3) Relative Migration Rates of Solutes, Retention Time, Retention and Selectivity Factors. 1.4) 1.2 Zone Broadening and Column Efficiency 1.5) 1.2.1 Theoretical Plates and Plate Height 1.6) 1.2.2 Kinetic Variables Affecting Zone Broadening (van Deemter Equation) 1.7) 1.3 Optimization of Column Performance 1.8) 1.3.1 Column Resolution 1.9) 1.3.2 Variables Affecting Column Performance 1.10) 1.4 Qualitative Application of Chromatography 1.11) 1.5 Quantitative Application of Chromatography 1.12) 1.5.1 External Standard Method 1.13) 1.5.2 Internal Standard Method 1.14) 1.5.3 Multiple Point Calibration
2. 2.0 Gas Chromatography (GC) 2.1) 2.1 Principles of GC 2.2) 2.2 Carrier Gas 2.3) 2.3 GC Injectors 2.4) 2.3.1 Direct/flash Vaporization Injector 2.5) 2.3.2 Split/Splitless Injector 2.6) 2.3.3 Oncolumn Injector 2.7) 2.4 GC Columns 2.8) 2.4.1 Types of Columns

2.9) 2.4.2 Types of Stationary Phases Bonded and Cross-linked Phases Film Thickness
2.10) Retention/Kovats Index
2.11) 2.4.3 Column Temperatures
2.12) Isothermal
2.13) Temperature Programming
2.14) 2.5 GC Detectors
2.15) 2.5.1 Flame Ionization Detectors (FID)
2.16) 2.5.2 Electron Capture Detectors (ECD)
2.17) 2.5.3 Thermal Conductivity Detector (TCD)
2.18) 2.5.4 Other Types of Detectors (Flame Photometric Detector (FPD) and Nitrogen Phosphorous Detector (NPD))
2.19) 2.6 Interfacing GC with Spectroscopic Methods: Mass Spectrometry Detector (GC-MSD) and Fourier Transform Infrared Spectroscopy (GC-FTIR)
2.20) 2.7 Sample Preparation for GC
2.21) 2.7.1 Headspace
2.22) 2.7.2 Pyrolysis
2.23) 2.7.3 Derivatization

3. 3.0 High Performance Liquid Chromatography (HPLC)
3.1) 3.1 Mobile Phase
3.2) 3.1.1 Solvent Treatment
3.3) 3.1.2 Isocratic Elution and Gradient Elution
3.4) 3.2 Pumping Systems: Types of Pumps
3.5) 3.3 Sample Injection Systems
3.6) 3.4 HPLC Columns
3.7) 3.4.1 Analytical Column and Guard Column
3.8) 3.4.2 Column Thermostats
3.9) 3.5 HPLC Detectors
3.10) 3.5.1 Absorbance Detectors (UV and PDA)
3.11) 3.5.2 Refractive Index Detectors
3.12) 3.5.3 Other Types of Detectors (Conductivity, Fluorescence, Mass Spectrometry)
3.13) 3.6 Partition Chromatography
3.14) 3.6.1 Principle of Partition Chromatography
3.15) 3.6.2 Reversed-Phase and Normal Phase
3.16) 3.6.3 Method Development in Partition Chromatography
3.17) 3.6.4 Applications of Partition Chromatography
3.18) 3.6.5 Ion Pair Liquid Chromatography
3.19) 3.7 Adsorption Chromatography
3.20) 3.7.1 Principle of Adsorption Chromatography
3.21) 3.7.2 Applications of Adsorption Chromatography
3.22) 3.8 Ion Exchange Chromatography
3.23) 3.8.1 Principle of Ion Exchange Chromatography
3.24) 3.8.2 Types of Ion Exchange
3.25) 3.8.3 Suppressor Column
3.26) 3.8.4 Applications of Ion-Exchange Chromatography
3.27) 3.9 Size-Exclusion Chromatography
3.28) 3.9.1 Principle of Size-Exclusion Chromatography
3.29) 3.9.2 Applications of Size-Exclusion Chromatography, Gel Filtration and Gel Permeation

4. 4.0 Supercritical Fluid Chromatography (SFC)
4.1) 4.1 Principle of SFC
4.2) 4.2 Instrumentation and Operating Variables
4.3) 4.2.1 Effects of Pressure
4.4) 4.2.2 Stationary Phases
4.5) 4.2.3 Mobile Phases
4.6) 4.2.4 Detectors
4.7) 4.3 Application of SFC

5. 5.0 Capillary Electrophoresis (CE)
5.1) 5.1 Principle of CE
5.2) Migration Rates, Electroosmotic Flow, Electrophoretic Flow
5.3) 5.2 Instrumentation
5.4) 5.2.1 Sample Introduction
5.5) 5.2.2 Detector
5.6) 5.3 Capillary Zone Electrophoresis (CZE)
5.7) Principle of CZE
5.8) 5.4 Capillary Gel Electrophoresis (CGE)
5.9) Principle of CGE
5.10) 5.5 Micellar Electrokinetic Capillary Chromatography (MECC)
5.11) Principle of MECC

6. 6.0 Sample Preparation for Chromatographic Analysis
6.1) 6.1 Extraction for Aqueous Samples
6.2) 6.1.1 Liquid-liquid Extraction (LLE)
6.3) 6.1.2 Solid Phase Extraction (SPE)
6.4) 6.1.3 Solid Phase Microextraction (SPME)
6.5) 6.2 Extraction of Solid Samples
6.6) 6.2.1 Soxhlet Extraction
6.7) 6.2.2 Supercritical Fluid Extraction (SFE)
6.8) 6.2.3 Microwave assisted Extraction (MAE)
6.9) 6.2.4 Pressurized Solvent/Liquid Extraction (Accelerated Solvent
6.10) Extraction) (PSE/PLE/ASE)

Assessment Breakdown	%
Continuous Assessment	60.00%
Final Assessment	40.00%

Details of Continuous Assessment

Assessment Type	Assessment Description	% of Total Mark	CLO
Quiz	Quiz 1 (Topic 1: Chromatographic Separation)	5%	CLO1
Quiz	Quiz 2 (Topic 6: Sample Preparation for Chromatographic Analysis)	5%	CLO2
Test	Test 1 (Topic 2: Gas Chromatography)	10%	CLO2
Test	Test 2 (Topic 3: High-Performance Liquid Chromatography)	10%	CLO3
Test	Test 3 (Topic 4: Supercritical Fluid Chromatography and Topic 5: Capillary Electrophoresis)	10%	CLO3
Written Report	Submission of lab reports for five selected experiments (GC, HPLC, FAME-GC-FID, SPME-GC-MSD, SPE-GC-ECD)	20%	CLO4

Reading List

Recommended Text

- **Douglas A. Skoog,F. James Holler,Stanley R. Crouch 2007, *Principles of Instrumental Analysis*, 6 Ed., Brooks/Cole Cengage Learning [ISBN: 0-495-12570-9]**
- **Nor'ashikin Saim,Ruziyati Tajuddin,Mardiana Saaid 2012, *Analytical Separation Methods Laboratory Guide*, 2 Ed., UiTM Press [ISBN: 9789673634361]**
- **Skoog, D.A., West, D.M., Holler, F.J. 1996, *Fundamental of Analytical Chemistry*, 7 Ed., Saunders**
- **Harris, D.C. 2007, *Quantitative Chemical Analysis*, 7 Ed., Freeman**
- **Dean, J.R. 1998, *Extraction Methods for Environmental Analysis*, John Wiley & Sons**

Article/Paper List: This Course does not have any article/paper resources

Other References: This Course does not have any other resources