Annexe A: New/Revised Course Content in OBTL+ Format

Course Overview

The sections shown on this interface are based on the templates UG OBTL+ or PG OBTL+
If you are revising/duplicating an existing course and do not see the pre-filled contents you expect in the subsequent sections e.g. Course Aims, Intended Learning Outcomes etc. please refer to Data Transformation Status for more information.

Expected Implementation in Academic Year	AY2022-2023
Semester/Trimester/Others (specify approx. Start/End date)	Semester 1
Course Author * Faculty proposing/revising the course	Leong Weng Kee
Course Author Email	chmlwk@ntu.edu.sg
Course Title	STRUCTURAL DETERMINATION
Course Code	CM4012
Academic Units	3
Contact Hours	43
Research Experience Components	Not Applicable

Course Requisites (if applicable)

Pre-requisites	CM3011 and CM3021 or by permission
Co-requisites	
Pre-requisite to	
Mutually exclusive to	CM412S
Replacement course to	
Remarks (if any)	

Course Aims

This course is divided into two parts.

Part I: The aim of this part concerns the use of NMR chemical shifts to elucidate structures of organic molecules. The specific focus is to learn how different (common) functional groups and non-covalent interactions (in organic chemistry) affect the 1H and 13C NMR chemical shifts of molecules.

Part II: The aim of this part is to show how you can obtain structural information on molecular compounds. We will begin with some general considerations in structural determination. This will be followed by more details on two very important techniques – NMR spectroscopy and X-ray crystallography. The former is a powerful method for studying molecules in solution and the latter for structures in the solid state. For the former, we will be looking at some concepts essential to an understanding of NMR spectroscopy, phenomena which can affect the appearance of an NMR spectrum, and some useful NMR techniques that can be used to study molecular structures and dynamics.

Course's Intended Learning Outcomes (ILOs)

Upon the successful completion of this course, you (student) would be able to:

ILO 1	Explain the simplified physics basis of NMR, including nuclear spin states, magnetic field, induced filed, and origin of different chemical shifts for protons present in organic molecules.
ILO 2	List the effects of functional groups on 1H NMR Chemical Shifts, including: (a) how do these functional groups/atoms affect the NMR chemical shifts; what are the other factors that can affect the NMR chemical shifts; and (c) what are the structural information we can get from the NMR chemical shifts. The student should develop understanding on the following specific topics: Electronegativity of neighboring atoms and groups, Magnetic anisotropy, and Electrostatic Interactions
ILO 3	Explain structural equivalence in NMR chemical shift assignments. The students are supposed to figure out how many different sets of proton or carbon chemical shifts can be expected from a molecule. Specific topics include “symmetry of molecules and structure equivalence” and “Chemical Equivalence by Interconversion”
ILO 4	Explain 13C NMR of sp3 carbons, including α-Substituent and Other Effects, β-Substituent Effects, γ-Substituent Effects and other special systems.
ILO 5	Explain 13C NMR of sp2 and sp carbons, including α-Substituent and Other Effects, Effects from other neighboring groups and charges.
ILO 6	Preliminarily integrate the use of NMR for structure determination for modern research especially in the area of synthetic chemistry
ILO 7	Integrate knowledge learned in this course and previous courses to proposed reasonable structures for relatively simple organic molecules, and the ability predict NMR chemical shifts for common functional groups in sophisticated molecules.
ILO 8	List down some techniques commonly used to help in structural determination, particularly, spectroscopic techniques such as IR, NMR, ESR and UV, mass spectrometry and X-ray crystallography, and their following characteristics: the molecular information that can be obtained from them, some of the limitations associated with them,
ILO 9	Demonstrate that different spectroscopic techniques probe different molecular properties and that these correspond to different transition energies.
ILO 10	Describe and explain that the intensity of a spectral line depends on: the population difference between the ground and excited states (Boltzmann distribution), the selection rules, using examples such as the electronic transitions of transition metal coordination complexes, path length of the sample (Beer-Lambert law).

ILO 11	Attribute spectral line widths to Collision broadening Doppler broadening Relaxation time (Heisenberg Uncertainty Principle)
ILO 12	Relate the Uncertainty Principle to: spectroscopic linewidths in terms of the lifetime in the excited state, apparent structure of a molecule in relation to the spectroscopic timescale, using interconversion between two states to illustrate.
ILO 13	State that the Fourier transform relates the time domain to the frequency domain, i.e., that it allows transformation of the free induction decay (FID) into a spectrum.
ILO 14	Describe pulse NMR in terms of the bulk magnetization and in the rotating frame of reference, and in particular the following: the time constant T1 for recovery of the z magnetization and how it can be determined by the inversion recovery experiment, the time constant T2 for loss of xy magnetization and its description with isochromats Prior knowledge of chemical shifts and coupling in NMR are assumed.
ILO 15	Describe and identify: complex spin systems, such as, AB2, ABX and AA'BB', but detailed analyses of these are not required. second order effects. virtual coupling. magnetic vs chemical equivalence. diastereotopic nuclei. coupling to quadrupolar nuclei, including their spin states and relaxation characteristics. the effect of paramagnetism.

ILO 16	Explain the effect that chemical exchange can have on the appearance of an NMR spectrum and that information on the chemical dynamics can be obtained via: variable temperature experiments – what it entails, lineshape analyses to yield rate constant and G^{1} at the coalescence temperature, and parameters from Eyring plots, measurement of T1 for systems undergoing exchange in the fast exchange limit, an EXSY experiment for systems undergoing exchange in the slow exchange limit. Knowledge of pulse sequences and other technical details are not required but should be able to state that the EXSY and NOESY pulse sequences are identical.
ILO 17	Demonstrate knowledge of the following on the Nuclear Overhauser Effect (NOE): state the effect and the definition of the NOE factor ( ). describe how the intensity of resonances can be affected by NOE, including by nuclei with negative gyromagnetic ratios. use of inverse-gated decoupling to minimize the effects of NOE in decoupling experiments. detection of insensitive nuclei can be achieved via NOE, and also polarization transfers. a NOESY spectrum provides information on proximity of nuclei. Knowledge of the origins of NOE and polarization transfer are not required, but knowledge of gyromagnetic ratios is assumed.
ILO 18	Describe how no-D 1H NMR spectra can be acquired, including using a solvent suppression sequence. Knowledge of technical details are not required.
ILO 19	Demonstrate knowledge of the following concerning the diffraction of X-rays by a crystal: X-rays are scattered by the electrons in atoms and molecules, there exist relationships between the observable diffraction pattern and relative intensities on the one hand (Bragg's law and the reciprocal lattice), and the unit cell and its contents on the other (structure factor), solving an X-ray crystal structure involves modelling the observed diffraction pattern, through the structure factor, with the unit cell contents and hence molecular structure. The construction of the reciprocal lattice and details of the structure factor are not required, only an appreciation of the links described above.

ILO 20	Extract the following from the output of a crystallographic study: identify the various items in crystallographic tables, assess the quality of a crystal structure determination from the crystallographic tables and ORTEP plot, make statistically meaningful comparisons of bond parameters,
ILO 21	State the limitations and problems that are associated with crystallographic studies on molecular species.

Course Content

Part I
- Introduction to NMR (a brief review)
- The effects of functional groups on 1H NMR Chemical Shifts
- Structure equivalence
- A brief overview of 13C NMR
- 13C NMR of sp3 carbons
- 13C NMR of sp2 and sp carbons

Part II
- Fundamental concepts in structural methods
- Appearance of NMR spectra
- Fundamentals of X-ray crystallography
- Interpreting X-ray crystallographic data

Reading and References (if applicable)

Books

Title: Modern NMR Techniques for Chemistry Research

Authors: Andrew E Derome

Publisher: Pergamon

Call no.: QD96.N8D437

Title: 200 and More NMR Experiments – A Practical Course

Authors: Stefan Berger and Siermar Braun

Publisher: Wiley

Call no.: QD96.N8B496

Title: NMR Spectroscopy in Inorganic Chemistry

Author: Jonathan A. Iggo

Publisher: OUP

Call no.: QD96.N8I24

Title: X-ray Structure Determination – A Practical Guide

Authors: George H. Stout and Lyle H. Jensen

Publisher: Wiley

Call no.: QD945.S889

Title: Crystal Structure Determination

Author: Werner Massa

Publisher: Springer

Call no.: QD945.M414

Websites

For NMR - https://www.chem.wisc.edu/areas/reich/nmr/

For X-ray crystallography - https://www.iucr.org/education/pamphlets

Planned Schedule

Week or Session	Topics or Themes	ILO	Readings	Delivery Mode	Activities
1	Introduction to NMR (a brief review), The effects of functional groups on 1H NMR Chemical Shifts: Electronegativity of neighboring atoms and groups	1-2,6-7			
2	The effects of functional groups on 1H NMR Chemical Shifts: Magnetic anisotropy	1-2, 6-7			
3	The effects of functional groups on 1H NMR Chemical Shifts: Electrostatic Interactions	1-2, 6-7			
4	Structure equivalence: 3.1: Symmetry of molecules and structure equivalence , 3.2: Chemical Equivalence by Interconversion, A brief overview of 13C NMR	3-7			

Week or Session	Topics or Themes	ILO	Readings	Delivery Mode	Activities
5	13C NMR of sp3 carbons: 5.1:α-Substituent and Other Effects, 5.2:β-Substituent Effects, 5.3:γ-Substituent Effects and other special systems	4,6-7			
6	13C NMR of sp2 and sp carbons: 6.1: α-Substituent and Other Effects, 6.2: Effects from other neighboring groups and charges	5,6-7			
7	Special Topics in NMR, summary, practice questions, and preparation for mid-term.	1-7			
8	Fundamental concepts in structural methods	1-5	Concepts to be learnt: 1. Factors that affect spectral intensities and linewidths 2. Linewidths and excited state lifetimes		Bite-sized presentations: 1. Characteristics of spectroscopic techniques in structural studies 2. Appearance of spectral lines

Week or Session	Topics or Themes	ILO	Readings	Delivery Mode	Activities
9	Appearance of NMR spectra	6-8	Concepts to be learnt: Describing pulse NMR - bulk magnetization and the rotating frame The time constants T1 and T2. Measurement of T1 by inversion recovery Virtual coupling Second order effects Chemical vs magnetic equivalence Quadrupolar nuclei – their spin states and relaxation characteristics Paramagnetic complexes – the effect of unpaired electrons on NMR		Bite-sized presentations: 1. Some NMR basics 2. Pulse NMR and the Fourier transform 3. Describing the bulk magnetization 4. Relaxation of the magnetization vector and relaxation mechanisms 5. Measurement of T1 by inversion recovery 6. Effects related to chemical shifts and coupling constants 7. Effects related to relaxation

Week or Session	Topics or Themes	ILO	Readings	Delivery Mode	Activities
10	Chemical exchange, NoE and other useful NMR experiments	9-11	Concepts to be learnt: Effect of chemical exchange on an NMR spectrum – studying chemical dynamics Variable temperature experiments and kinetic information The EXSY experiment The Nuclear Overhauser Effect (NOE) Effects on intensities - Inverse gated decoupling Internuclear distance information – NOESY and variants Sensitivity enhancement by polarisation transfer, e.g., INEPT, DEPT		Bite-sized presentation: 1.The Nuclear Overhauser Effect (NOE) 2. Chemical exchange

Week or Session	Topics or Themes	ILO	Readings	Delivery Mode	Activities
11	Fundamentals of X-ray crystallography	12	Concepts to be learnt: Objectives of an X-ray structural determination: Relationship between unit cell and diffraction pattern Crystal lattice, unit cell and space group Bragg's Law Reciprocal lattice and Miller indices Relationship between unit cell content and intensity of "reflections" Atomic scattering factors and displacement factors The structure factor		Bite-sized presentations: 1. Generation and characteristics of X-rays for chemical crystallography 2. Crystal system 3. Crystal Class 4. Translational Symmetry 5. The Bragg equation 6. Reciprocal space 7. Atomic scattering factors 8. The structure factor
12	Interpreting X-ray crystallographic data	13-14	The process of structural determination, incl structural solution and refinement Contents of the crystallographic tables, including terms such as structural refinement parameters, R-factors, and residuals Simple statistical analysis of structural parameters Limitation of X-ray crystallography, including things that can go wrong in a structural determination		Bite-sized presentations: X-ray structural solution and refinement Results of a crystallographic determination What can go wrong with an X-ray crystallographic determination

Learning and Teaching Approach

Approach	How does this approach support you in achieving the learning outcomes?
Directed self-learning	This course involves directed self-learning and peer learning. The first component will involve a list of the concepts to be learned on your own each week. The supporting material available include: Bite-sized videos – these are about 5-10 min long each. A list of the currently available ones for each week's lesson is provided, and they have also been set up in a LAMS environment on NTULearn. You can watch any of them at any time and for any number of times. Each presentation is on one or a couple of concepts. They are not meant to cover everything that you can learn about a topic or concept, but as a springboard for you to explore your books and the internet for more. Online quizzes The online quizzes are structured within the LAMS environment. It is intended as a feedback system to guide you on the weekly learning. You are therefore expected to gain 100% competency on it. Internet and ebooks. List of recommended textbooks Concept questions (appendix 3) – these are meant to prompt you on what you will need to learn. We will go through them during the key points sessions.
Key point lectures	During these sessions, we will begin with (a) feedback from the previous reflections session, (b) revision of the key points for the previous week's lesson, together with (c) some clicker-based questions. The concept question for the week will then be discussed, using clicker-based questions. This is followed by the key points to be learned for the week, reinforced with another series of clicker-based questions. Reproductions of the Powerpoint slides for the lectures are already available from NTULearn so that you can focus on learning rather than on copying things down
Tutorials	The tutorial sessions and questions are to test your competence in the concepts to be learned, as well as the ability to connect ideas and concepts. It is meant also to create a peer teaching and learning environment. The questions are to be tackled individually, and as a team. You will have already been provided with a list of tutorial questions (appendix 4), one for each tutorial session. The tutorial class sizes are kept small deliberately, in order that it will not inhibit discussion. All tutorial classes will be facilitated by the lecturer. In each tutorial session, you will self-assemble into groups of up to six to discuss the tutorial question which you already have on hand. This will be followed by a class-level discussion. After that, a second (in-class) tutorial question will be provided to the class for discussion.
Reflections	During these sessions, you will be given a set of questions based primarily on the concepts to be learned for the week. This is to allow you to ponder over the concepts and allow you to determine if you have understood the lesson or not. It is also a chance for you to explore connected ideas, and perhaps come up with new queries, all of which can be jotted down. The lecturer will go through all the reflection papers and any misconception, etc., from the class will be noted and discussed at the next key point lecture session.

Assessment Structure

Assessment Components (includes both continuous and summative assessment)

No.	Component	ILO	Related PLO or Accreditation	Weightage	Team/Individual	Rubrics	Level of Understanding
1	Continuous Assessment (CA): Test/Quiz(Quiz 1)	ILO1-2	Competence, Communication	15	Individual	Holistic	Not Applicable
2	Continuous Assessment (CA): Test/Quiz(Quiz 2)	ILO1-3	Competence, Creativity	15	Individual	Holistic	Not Applicable
3	Continuous Assessment (CA): Test/Quiz(Mini-quiz)	ILO 1-13	Competence	2.5	Individual	Holistic	Not Applicable
4	Continuous Assessment (CA): Others(Pre- and in-class tutorial questions)	1-14	Competence, Communication	7.5	Individual	Holistic	Not Applicable
5	Continuous Assessment (CA): Others(Reflections)	ILO 1-13	Competence, Creativity, Communication	10	Individual	Holistic	Not Applicable
6	Continuous Assessment (CA): Test/Quiz(Mid-term Test I)	ILO1-7	Competence, Creativity,	20	Individual	Analytic	Not Applicable
7	Continuous Assessment (CA): Test/Quiz(Mid-term Test II)	ILO 1-13	Competence, Creativity, Communication	25	Individual	Analytic	Not Applicable
8	Continuous Assessment (CA): Others(Concept question)			5	Individual	Analytic	Not Applicable

Description of Assessment Components (if applicable)

Formative Feedback

Part I

Frequent and flexible office hours will be made available for face-to-face discussions on concepts and problems that are challenging to you. For students with excellent performance and the willing to receive deeper trainings, additional study materials will be made available upon request.

Part II

Formative feedback: The online quizzes will allow you to monitor your learning of the concepts on a weekly basis. During the weekly workshop sessions, there will also be clicker-type questions to allow you to assess your understanding although it will not be formally assessed. Through the weekly reflections, you will be able to test your own learning. The lecturer will have read through your reflections and offer a formative feedback to the class weekly.

Summative Feedback: Summative feedback will be in the form of the mid-terms.

NTU Graduate Attributes/Competency Mapping

This course intends to develop the following graduate attributes and competencies (maximum 5 most relevant)

Attributes/Competency	Level
Collaboration	Intermediate
Learning Agility	Advanced
Critical Thinking	Advanced

Course Policy

Policy (Academic Integrity)

Good academic work depends on honesty and ethical behaviour. The quality of your work as a student relies on adhering to the principles of academic integrity and to the NTU Honour Code, a set of values shared by the whole university community. Truth, Trust and Justice are at the core of NTU's shared values. As a student, it is important that you recognize your responsibilities in understanding and applying the principles of academic integrity in all the work you do at NTU. Not knowing what is involved in maintaining academic integrity does not excuse academic dishonesty. You need to actively equip yourself with strategies to avoid all forms of academic dishonesty, including plagiarism, academic fraud, collusion and cheating. If you are uncertain of the definitions of any of these terms, you should go to the academic integrity website for more information. On the use of technological tools (such as Generative AI tools), different courses / assignments have different intended learning outcomes. Students should refer to the specific assignment instructions on their use and requirements and/or consult your instructors on how you can use these tools to help your learning. Consult your instructor(s) if you need any clarification about the requirements of academic integrity in the course.

Policy (General)

You are expected to complete all assigned pre-class readings and activities, attend all seminar classes punctually and take all scheduled assignments and tests by due dates. You are expected to take responsibility to follow up with course notes, assignments and course related announcements for seminar sessions they have missed. You are expected to participate in all seminar discussions and activities.

Policy (Absenteeism)

Absence from class without a valid reason will affect your overall course grade. Valid reasons include falling sick supported by a medical certificate and participation in NTU's approved activities supported by an excuse letter from the relevant bodies.

If you miss a lesson, you must inform the course instructor via email prior to the start of the class.

Policy (Others, if applicable)

Diversity and Inclusion policy
Integrating a diverse set of experiences is important for a more comprehensive understanding o science and engineering.
It is our goal to create an inclusive and collaborative learning environment that supports a diversity of perspectives and learning experiences. That honours your identities; including ethnicity, gender, socioeconomic status, sexual orientation, religion or ability.
To help accomplish this:

- If you are neuroatypical or neurodiverse, have dyslexia or ADHD (for example), or have a social anxiety disorder or social phobia:
- If you feel your performance in the course is being impacted by your experiences outside of class:
- If something was said in the course (by anyone, including instructor/supervisor) that made you uncomfortable.

Please e-mail to your Associate Chair (Students & Continuing Education) at ac-cceb-stud@ntu.edu.sg about how we can help facilitate your learning experience.
As a participant in course discussions you should also strive to honour the diversity of your classmates. You can

do this by; using preferred pronouns and names; being respectful of others opinions and actively making sure all voices are being heard; and refraining from the use of derogatory or demeaning speech or actions.
All members of the course are expected to strictly adhere to the student code of conduct (https://www.ntu.edu.sg/life-at-ntu/student-life/student-conduct) . If you witness something that goes against this or have ny other concerns, please speak to your instructors or a faculty member.

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