

<b>Academic Year</b>	AY20/21	<b>Semester</b>	1
<b>Course Coordinator</b>	Leong Weng Kee (Assoc Prof), Robin Chi (Prof)		
<b>Course Code</b>	CM4012 / CM412S <sup>1</sup>		
<b>Course Title</b>	Structural Determination		
<b>Pre-requisites</b>	CM3011 and CM3021 or by permission		
<b>Mutually Exclusive</b>	CM412S		
<b>No of AUs</b>	3		
<b>Contact Hours</b>	Lectures/reflections: 32 hours, Tutorials: 5 hours		

### 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 <sup>1</sup>H and <sup>13</sup>C 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.

### Intended Learning Outcomes (ILO)

At the end of the course, you should be able to:

#### Part I:

1. Explain the simplified physics basis of NMR, including nuclear spin states, magnetic field, induced field, and origin of different chemical shifts for protons present in organic molecules.
2. List the effects of functional groups on <sup>1</sup>H 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
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”
4. Explain <sup>13</sup>C NMR of sp<sup>3</sup> carbons, including α-Substituent and Other Effects, β-Substituent Effects, γ-Substituent Effects and other special systems.

<sup>1</sup> CM412S is a self-paced version of the course

5. Explain  $^{13}\text{C}$  NMR of  $\text{sp}^2$  and  $\text{sp}$  carbons, including  $\alpha$ -Substituent and Other Effects, Effects from other neighboring groups and charges.
6. Preliminarily integrate the use of NMR for structure determination for modern research especially in the area of synthetic chemistry
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.

### Part II:

#### **Fundamental concepts in structural methods**

1. 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,
2. Demonstrate that different spectroscopic techniques probe different molecular properties and that these correspond to different transition energies.
3. 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).
4. Attribute spectral line widths to
  - Collision broadening
  - Doppler broadening
  - Relaxation time (Heisenberg Uncertainty Principle)
5. 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.

#### **NMR spectroscopy**

6. 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.
7. Describe pulse NMR in terms of the bulk magnetization and in the rotating frame of reference, and in particular the following:
  - the time constant  $T_1$  for recovery of the  $z$  magnetization and how it can be determined by the inversion recovery experiment,
  - the time constant  $T_2$  for loss of  $xy$  magnetization and its description with isochromatsPrior knowledge of chemical shifts and coupling in NMR are assumed.
8. Describe and identify:
  - complex spin systems, such as,  $\text{AB}_2$ ,  $\text{ABX}$  and  $\text{AA}'\text{BB}'$ , 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.
9. 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  $\Delta G^\ddagger$  at the coalescence temperature, and parameters from Eyring plots,
  - measurement of  $T_{1\rho}$  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.
10. Demonstrate knowledge of the following on the Nuclear Overhauser Effect (NOE):
- state the effect and the definition of the NOE factor ( $\eta$ ).
  - 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 polarisation 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.
11. Describe how no-D  $^1\text{H}$  NMR spectra can be acquired, including using a solvent suppression sequence. Knowledge of technical details are not required.

### **X-ray crystallography**

12. 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 exists 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.
13. 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,
14. State the limitations and problems that are associated with crystallographic studies on molecular species.

**Assessment (includes both continuous and summative assessment)**

Component	Course ILO Tested	Related Programme LO or Graduate Attributes	Weighting	Team/Individual	Assessment rubrics
<b>Part I</b>					
1. quiz 1 (week 4)	1-2	Competence, Creativity, Character Civic-mindedness	15%	Individual	See appendix
2. quiz 2 (week 6)	3	Competence, Creativity, Communication	15%	Individual	See appendix
<b>Part II</b>					
3. Mid-term (week 13)	1-14	Competence, Creativity, Character	30%	Individual	See appendix
<b>Part I &amp; II</b>					
4. Final Examination	ALL	Competence, Creativity, Character	40%	Individual	See appendix
<i>Total</i>			100%		

### 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 summative feedback to the class weekly.  
Summative Feedback: Summative feedback will be in the form of the mid-term and the final examination, the former of which will be reviewed in class.

## Learning and Teaching approach

### Part I

- Main topics will be delivered as lectures in the classroom. All students are encouraged to attend the class, instead of watching the video at home.
- Practice questions will be discussed during lecture time.
- One or two topics will be assigned for self-learning. The content will vary from batch to batch, and details will be announced in due course.
- Frequent and flexible officer 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

### Part II

<p><b>Directed self-learning</b> (~6 hours)</p>	<p>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:</p> <p><b>Bite-sized videos</b> – 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.</p> <p><b>Online quizzes</b> 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.</p> <p><b>Internet and ebooks.</b></p> <p><b>List of recommended textbooks</b></p> <p><b>Concept questions</b> (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.</p>
<p><b>Key point lectures</b> (5 hours)</p>	<p>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.</p>
<p><b>Tutorials</b> (5 hours)</p>	<p>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 (&lt;25) 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.</p>

<b>Reflections</b> (5 hours)	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.
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### Reading and References

#### **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>

### Course Instructors

Instructor	Office Location	Phone	Email
Leong Weng Kee (Assoc Prof)	CBC-06-07	6592 7577	chmlwk@ntu.edu.sg
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## Planned Weekly Schedule

### Part I

Week 1 (first one hour): Introduction to NMR (a brief review)

Course ILO: 1-2,6-7

Week 1-3: The effects of functional groups on  $^1\text{H}$  NMR Chemical Shifts

Course ILO: 1-2, 6-7

2.1: Electronegativity of neighboring atoms and groups

- a. Electronegativity
- b. Charged Molecules
- c. Lone Pair Interactions

2.2: Magnetic anisotropy

- a. Aromatic and "normal"  $\pi$ -systems
- b. Other bonding systems
- c. Steric Compression

2.3: Electrostatic Interactions

- a. Hydrogen bonding
- b. Other electrostatic interactions

Week 4 (first two hours): Structure equivalence

Course ILO: 3, 6-7

3.1: Symmetry of molecules and structure equivalence

3.2: Chemical Equivalence by Interconversion

Week 4 (last hour): A brief overview of  $^{13}\text{C}$  NMR

Course ILO: 4-5,6-7

Week 5:  $^{13}\text{C}$  NMR of  $\text{sp}^3$  carbons

Course ILO: 4,6-7

5.1:  $\alpha$ -Substituent and Other Effects

5.2:  $\beta$ -Substituent Effects

5.3:  $\gamma$ -Substituent Effects and other special systems

Week 6:  $^{13}\text{C}$  NMR of  $\text{sp}^2$  and  $\text{sp}$  carbons

Course ILO: 5,6-7

6.1:  $\alpha$ -Substituent and Other Effects

6.2: Effects from other neighboring groups and charges

Week 7. Special Topics in NMR, summary, practice questions, and preparation for exams.

Course ILO: 1-7

### Part II

**Week 8 – Fundamental concepts in structural methods**

Course ILO: 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 9 – Appearance of NMR spectra**

**Course ILO: 6-8**

### **Concepts to be learnt:**

1. Describing pulse NMR - bulk magnetization and the rotating frame
2. The time constants  $T_1$  and  $T_2$ . Measurement of  $T_1$  by inversion recovery
3. Virtual coupling
4. Second order effects
5. Chemical vs magnetic equivalence
6. Quadrupolar nuclei – their spin states and relaxation characteristics
7. 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  $T_1$  by inversion recovery
6. Effects related to chemical shifts and coupling constants
7. Effects related to relaxation

## **Week 10 – Chemical exchange, NoE and other useful NMR experiments**

**Course ILO: 9-11**

### **Concepts to be learnt:**

1. Effect of chemical exchange on an NMR spectrum – studying chemical dynamics
  - Variable temperature experiments and kinetic information
  - The EXSY experiment
2. The Nuclear Overhauser Effect (NOE)
  - Effects on intensities - Inverse gated decoupling
  - Internuclear distance information – NOESY and variants
3. Sensitivity enhancement by polarisation transfer, e.g., INEPT, DEPT

### **Bite-sized presentations:**

1. The Nuclear Overhauser Effect (NOE)
2. Chemical exchange

## **Week 11 – Fundamentals of X-ray crystallography**

**Course ILO: 12**

### **Concepts to be learnt:**

Objectives of an X-ray structural determination:

1. Relationship between unit cell and diffraction pattern
  - Crystal lattice, unit cell and space group
  - Bragg's Law
  - Reciprocal lattice and Miller indices
2. 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

**Week 12 – Interpreting X-ray crystallographic data**

**Course ILO: 13-14**

**Concepts to be learnt:**

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

## Appendix 1: Assessment Rubrics

### Continual assessments

#### Part I

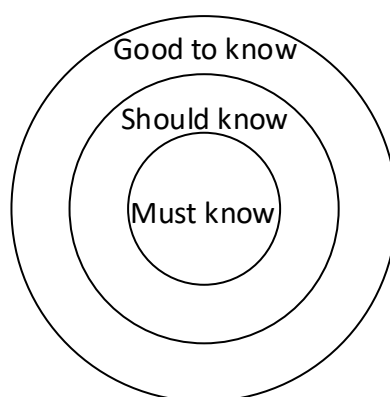
Quiz 1 (15%): this quiz tests your knowledge on how different functional groups and interactions affect the  $^1\text{H}$  NMR chemical shift. Around 70-80% of the questions are direct reflection of the content of the lecture materials; the remaining 20-30% test your integrated understanding of the topic

Quiz 2 (15%): this quiz tests your knowledge on several types of structural equivalence, and the use of structural equivalence to assist you assign the NMR chemical shifts. Around 70-80% of the questions are direct reflection of the content of the lecture materials; the remaining 20-30% test your integrated understanding of the topic.

#### Part II

Mid-term (30%)

The mid-term will be reviewed in class and hence provides formative feedback. In both assessments, you are expected to demonstrate knowledge of the course material, including the ability to utilize and extend them. The components of questions may be thought of as organized into three tiers:



Competency descriptor	Details
Must know	These are questions based primarily on lower-level course contents or expected prior knowledge. A demonstration of complete knowledge of these corresponds to a C grade and is required for progression of assessment beyond.
Should know	These questions will be those based directly on the course contents. Complete competency in them demonstrates mastery of the knowledge expected for the course, i.e., with organometallic chemistry. Demonstration of such competency corresponds to a B grade and is required for progression of the assessment beyond this.
Good to know	These questions require students to think beyond what has been explicitly covered in the course. It requires extension of thought beyond examples that have been encountered and may involve content knowledge of lower-level courses but not entirely new concepts. Those who can reach these will correspond to an A grade.

**Final exam (40%):** The final examination for part I will test your basic understanding of the topics covered (around 60% of the questions) and integrated understanding and applications of NMR chemical shifts for structural determination (around 40% of the questions). That for part II will follow the description above for the mid-term.

## Appendix 2: Intended Affective Outcomes

One primary goal in chemistry is to understand molecular properties and behavior. This hinges on knowing the structure of the molecule under study. This course serves to introduce the main structural determination methods in molecular chemistry. Molecular structures are, however, not static and this course serves to introduce you to the world of molecules and the ever-changing structures. The "big picture" attributes to be gained include:

There is much more to NMR than chemical shifts and coupling constants

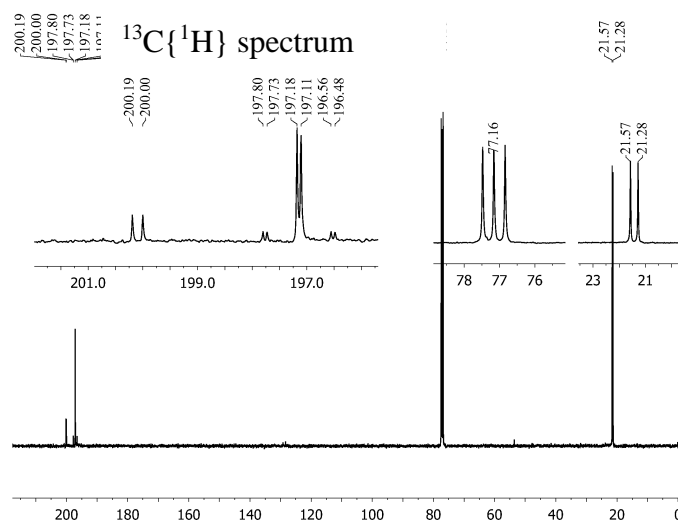
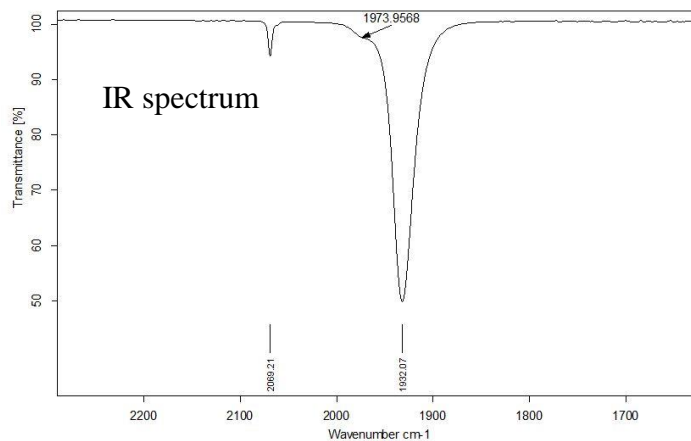
The world of the molecules is more complex and exciting than what you may have been led to believe

Thinking is hard work

## Appendix 3: Concept Questions

### Lesson 1 – Fundamental concepts in structural methods

Why do the spectra of  $W(CO)_5(PMe_3)$  have the appearance shown? Estimate the linewidths of the spectra. Why do they have such different linewidths?

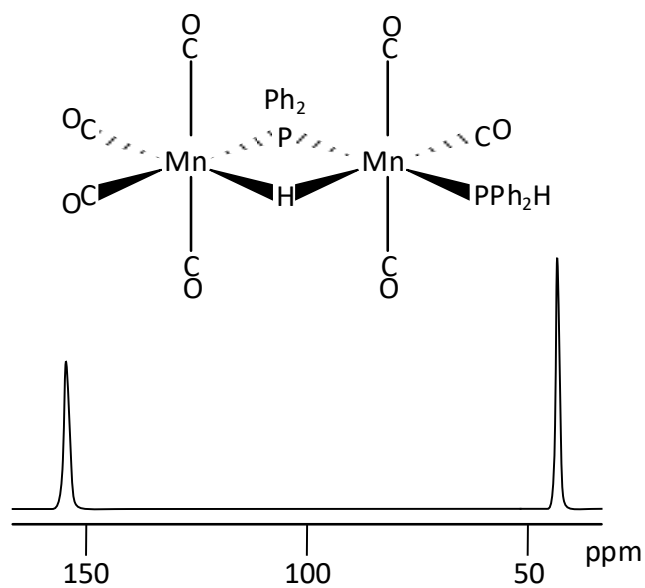


### Lesson 2 – Appearance of NMR spectra

How can we describe the behaviour of an ensemble of nuclear spins (with  $I = \frac{1}{2}$ ) in an “NMR experiment”?

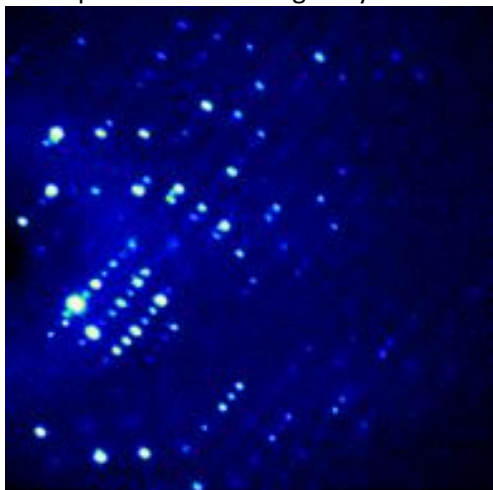
### Lesson 3 – Chemical exchange, NoE and other useful NMR experiments

The  $^{31}\text{P}\{^1\text{H}\}$  NMR spectrum of  $\text{Mn}_2(\mu\text{-H})(\mu\text{-PPh}_2)(\text{CO})_7(\text{PPh}_2\text{H})$  is shown below, together with the molecular structure. Why are the two resonances different in intensity? What can be done to obtain a “better” spectrum?



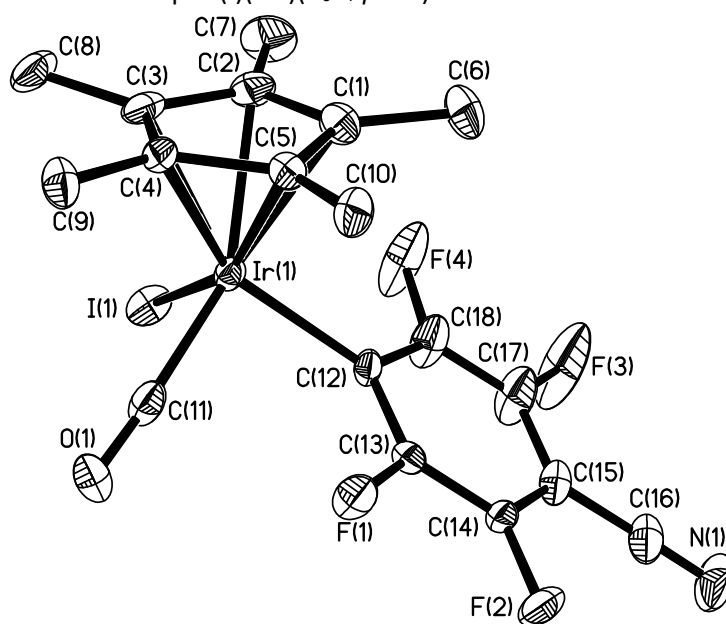
### Lesson 4 – Fundamentals of X-ray crystallography

Shown below is an X-ray diffraction pattern from a single crystal. How does it arise?



## Lesson 5 – Interpreting X-ray crystallographic data

The ORTEP plot of the molecule  $\text{Cp}^*\text{Ir}(\text{I})(\text{CO})(\text{C}_6\text{F}_4\text{-}p\text{-CN})$  is shown below.



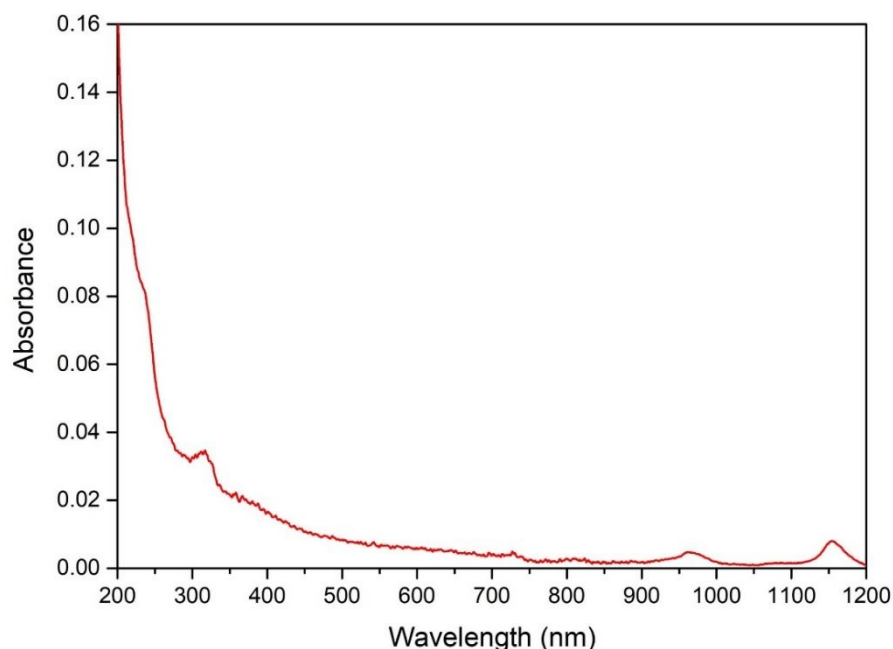
- (a) What is an ORTEP plot? What does it show?
- (b) The C-C bond lengths ( $\text{\AA}$ ) associated with the aromatic ring are:  
C(12)-C(13) = 1.381(8); C(12)-C(18) = 1.392(9); C(13)-C(14) = 1.382(8);  
C(14)-C(15) = 1.381(9); C(15)-C(17) = 1.379(10); C(17)-C(18) = 1.371(10);  
C(15)-C(16) = 1.441(9)  
Are there significant differences?

## Appendix 4: Tutorial Questions

### Lesson 1

Suppose you wish to obtain the electronic and NMR spectra of a sample of  $[\text{Fe}(\text{OH}_2)_6]^{2+}$ . Roughly what amount of sample will be required for each spectrum? Why are the amounts required different?

The electronic spectrum of  $[\text{Fe}(\text{OH}_2)_6]^{2+}$  is shown below. Why are the intensities of the peaks indicated so different?



### Lesson 2

The acquisition of  $^{13}\text{C}\{^1\text{H}\}$  NMR spectra is common for those working with organic and organometallic compounds.

- What does the designation  $^{13}\text{C}\{^1\text{H}\}$  mean?
- What are the problems associated with acquiring a  $^{13}\text{C}$  NMR spectrum, and how can they be overcome?
- Can you acquire a  $^{13}\text{C}$  NMR spectrum without a deuterated solvent? Why?

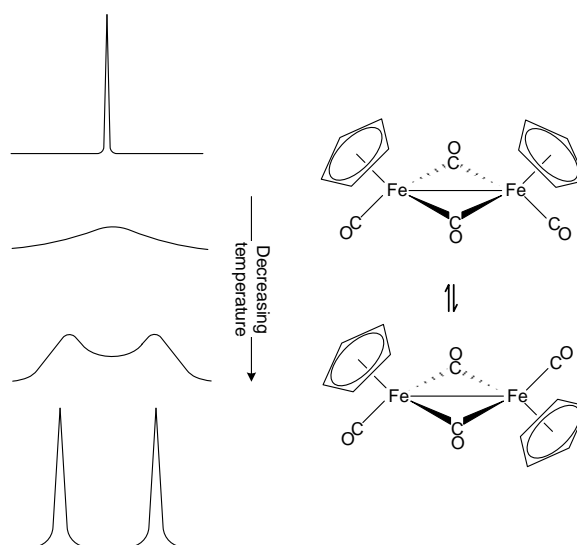
### Lesson 3

The complex  $\{\text{CpFe}(\text{CO})_2\}_2$  exists as a pair of *cis* and *trans* isomers which interconvert in solution. The variable temperature  $^1\text{H}$  NMR spectrum is shown to the right. The  $^{13}\text{C}$  NMR spectrum reveals that at high temperature, all the CO ligands become equivalent.

- Suggest two plausible mechanisms to account for the observations.

**Hint:** Consider concerted vs non-concerted CO and Cp exchanges.

- How may you differentiate between the mechanisms?



#### Lesson 4

Single crystal X-ray structural determination of the complexes  $\text{Os}_3\text{M}(\text{CO})_{12}[\text{C}_3\text{N}_2\text{H}_4(2,6\text{-iPr}_2\text{C}_6\text{H}_3)_2]$  (where M = Pd or Pt) were carried out and the following unit cell parameters were obtained:

M = Pd:  $a = 12.1588(10) \text{ \AA}$ ;  $b = 12.6035(10) \text{ \AA}$ ;  $c = 18.1256(13) \text{ \AA}$ ;  
 $\alpha = 96.546(1)^\circ$ ;  $\beta = 107.720(2)^\circ$ ;  $\gamma = 109.278(2)^\circ$ .

M = Pt:  $a = 12.0228(10) \text{ \AA}$ ;  $b = 12.6612(9) \text{ \AA}$ ;  $c = 18.2751(14) \text{ \AA}$ ;  
 $\alpha = 96.983(3)^\circ$ ;  $\beta = 108.336(2)^\circ$ ;  $\gamma = 108.426(2)^\circ$ .

- To which crystal system do the unit cells belong?
- In what way are the X-ray diffraction patterns for these two crystals likely to differ?

#### Lesson 5

The table given on the next page is typical for a structural determination.

- Identify the various items.
- Something is not quite satisfactory about this crystallographic study; what evidence is there?

Table 1. Crystal data and structure refinement for A.

Identification code	A	
Empirical formula	C17 H14 O11 Os3	
Formula weight	964.88	
Temperature	103(2) K	
Wavelength	0.71073 $\text{ \AA}$	
Crystal system	Triclinic	
Space group	$P \bar{1}$	
Unit cell dimensions	$a = 11.0223(8) \text{ \AA}$	$\alpha = 102.825(2)^\circ$
	$b = 14.4146(10) \text{ \AA}$	$\beta = 104.078(2)^\circ$
	$c = 15.0393(11) \text{ \AA}$	$\gamma = 94.973(2)^\circ$
Volume	2234.7(3) $\text{ \AA}^3$	
Z	4	
Density (calculated)	2.868 $\text{ Mg/m}^3$	
Absorption coefficient	17.069 $\text{ mm}^{-1}$	
F(000)	1728	
Crystal size	0.32 x 0.10 x 0.10 $\text{ mm}^3$	
Theta range for data collection	2.07 to 26.37 $^\circ$ .	
Index ranges	-13 $\leq h \leq$ 13, -18 $\leq k \leq$ 17, 0 $\leq l \leq$ 18	
Reflections collected	13701	
Independent reflections	9343 [R(int) = 0.0806]	
Completeness to theta = 26.37 $^\circ$	99.0 %	
Max. and min. transmission	0.2801 and 0.0735	
Refinement method	Full-matrix least-squares on F <sup>2</sup>	
Data / restraints / parameters	9343 / 132 / 390	
Goodness-of-fit on F <sup>2</sup>	1.069	
Final R indices [I > 2 $\sigma$ (I)]	R1 = 0.0744, wR2 = 0.2108	
R indices (all data)	R1 = 0.0872, wR2 = 0.2255	
Largest diff. peak and hole	3.308 and -5.722 $\text{ e.\AA}^{-3}$	



## **CBC Programme Learning Outcome**

The Division of Chemistry and Biological Chemistry (CBC) offers an undergraduate degree major in Chemistry that satisfies the American Chemical Society (ACS) curricular guidelines and equips students with knowledge relevant to the industry. Graduates of the Division of Chemistry and Biological Chemistry should have the following key attributes:

### **1. Competence**

Graduates should be well-versed in the foundational and advanced concepts of chemical science, be able to evaluate chemistry-related information critically and independently, and be able to use complex reasoning to solve emergent chemical problems.

### **2. Creativity**

Graduates should be able to synthesize and integrate multiple ideas across the curriculum, and propose innovative solutions to emergent chemistry-related problems based on their training in chemistry.

### **3. Communication**

Graduates should be able to demonstrate clarity of thought, independent thinking, and sound scientific analysis and reasoning through written and oral reports to audiences with varying technical backgrounds. They should also be able to effectively engage other professional chemists in collaborative endeavours.

### **4. Character**

Graduates should be able to act in responsible ways and uphold the high ethical standards that the society expects of professional chemists.

### **5. Civic-mindedness**

Graduates should be aware of the impact of chemistry on society, and how chemistry can be applied to benefit mankind. They should also be aware of and uphold the best chemical safety practices.