For this experiment, you are required to prepare a **short report** containing the following:

- An experimental method, briefly outlining the experimental steps that you took in this experiment. Ensure that you use the correct tense and voice, use a sufficient level of detail to enable others to repeat your experiment, but do not include unnecessary information (like glassware).
- A table (with appropriate heading) summarising the main peaks and peak assignments for EITHER your NMR spectrum, OR your IR spectrum.
- A computer-drawn structure of your identified unknown. You should use ChemSketch, ChemDraw, MarvinSketch, or other appropriate software. Include a suitable figure caption, with the name of the compound.
- A comparison of your experimental melting point for your assigned unknown with the literature, and a correctly formatted reference to the literature value.

3. EXPERIMENTS

Experiment 1 – Identification of an unknown organic compound

Identification of unknown samples is essential for many chemists. For example, synthetic chemists must identify unexpected side products of reactions, natural products chemists extract and study components of plants and microorganisms, while environmental chemists need to determine the identity and source of contaminants. Identification of unknown compounds typically involves combination of a range of techniques. In this experiment, you will use four different techniques to determine the identity of an unknown organic compound.

1. LEARNING OUTCOMES

After undertaking this experiment, you will have the following laboratory skills:
- Use of an NMR spectrometer
- Use of an IR spectrometer
- Thin-layer chromatography
- Staining of TLC plates
- Use of a melting point apparatus

After undertaking this experiment and writing the lab report, you will understand:
- The use of melting point, and NMR and IR spectroscopy to characterise an unknown compound.
- How to write an experimental method, and to prepare and present figures and tables.
- How to calculate R_F values, and to use TLC information to identify an unknown compound.
- How to combine information from different analytical techniques to deduce the identity of an unknown organic compound.

2. CHEMICALS USED AND SAFETY DATA

Safety information about TLC stains will be provided at the TLC workstation.
Safety information about unknown samples will be provided on the data sheet.

3.   PROCEDURE

This experiment will be performed individually. In the first part of this lab session, you will rotate in groups around four stations, at which you will collect key data to determine the structure of an unknown organic compound. You can collect your **unknown compound** from the Service Room.

In the second part you will receive instruction in the interpretation of NMR and IR spectra, and a tutorial on aspects of report preparation and presentation. If you need to check or finalise results from part 1, you should have time following part 2.

Part 1: Data Collection for Unknown Identification

Station 1 – Nuclear magnetic resonance (NMR) spectroscopy and melting points

At this station, the demonstrator will instruct you in the collection of an NMR spectrum of your unknown sample. Place a small amount (approx. 10 mg) of sample into a glass vial and add 1 mL deuterated chloroform (refer to the solvent table located in the fumehood to determine which solvent to use for your sample). Your demonstrator will show you how to prepare, collect and export an NMR spectrum of this sample. You should also print out the spectrum and paste it into your lab manual.

Station 2 – Melting Point Apparatus

At this station, you will determine the melting point of your unknown sample and compare it to literature values. Follow the instructions on determination of melting point in Appendix 4. You should take at least 2 readings. Record the data on the data sheet and note down all the possible compounds with melting points in the range of your recorded value (5-6 degrees either side).

Station 3 – Infrared (IR) spectroscopy

At this station, you will collect an IR spectrum of your unknown sample. Your demonstrator will show you how to collect an IR spectrum of your sample and export it electronically. Print out the spectrum and paste it into your lab manual. Write the major peaks in your lab notebook and use Table 2 of Appendix 3 to decide the possible identity of these peaks. You should practice downloading the spectrum from the computer so that you can replot it in Excel – this will be useful for the other experiments.

Station 4 – Thin-layer chromatography (TLC)

The demonstrator will show you how to set up and run a TLC plate, and how to visualise the plate using UV light and stains. For more information, see Appendix 5 and check the online video.

You will begin this station by first testing your compound against a series of chemoselective stains. Weigh out 3-5 mg of your sample into a scintillation vial and dissolve in acetone 3-5 mL (The mass and volume does not have to be accurate for this procedure). Cut one TLC plate into 4 strips. Using a TLC spotter, spot your sample onto the end of each of the TLC strips. Observe one of these strips under the long wave and short wave UV light in the TLC Light box. Record any observations. Next take the strip and stain it with the first of the TLC stains. Follow the instruction provided at the stain. Repeat the staining process with the remaining stains and

TLC strips.   Record any observations and what it represents in terms of which functional groups that may be present OR absent.

Next you will perform two TLC analyses on your unknown. You should spot your own sample on a TLC plate with any appropriate reference samples. If you have already identified possible candidates from the chemoselective staining or from your results from other stations, you should run these as reference samples. Use 10% (v/v) methanol in dichloromethane (5 mL) as your eluent. Whilst you wait for your TLC to run, repeat the steps above to run a second TLC with 100% dichloromethane as the eluent. Once you have run your TLC plates, and appropriate unknowns, take a photo of each. Use an appropriate stain you identified in the first part to visualise your TLC if needed. Sketch the TLC on your results sheet and make sure to record the solvent system that you used. Calculate the R_F value for your unknown. Dispose of your TLCs in the cardboard "contaminated broken glass" waste bins.

Part 2: Data Analysis and Scientific Reporting Tutorials

The second part of this lab session comprises two tutorials.

In one tutorial, you will review the information contained in NMR and IR spectra so that you can analyse and interpret the spectrum of your unknown collected at Stations 1 and 3. This should allow you to finalise the identification of your unknown. If necessary, a demonstrator will assist you to repeat one of the tests from part 1.

In the other tutorial, you will be introduced to acceptable scientific literature sources (books, databases and journals), the correct format for scientific referencing for your reports, and briefly discuss the question "What you need to reference?" (see also Appendix 2)

You should also refresh your knowledge on the correct presentation of figures and figure captions, tables and table headings, and experimental method in a report, oral presentation or poster. You should have covered this already on Canvas. The review material is also available online in the Training folder on Canvas.

4.   RESULTS ENTRY

Once you have collected all the data, you will be able to decide which unknown sample you have. You must enter the identity of this compound (along with the sample number) into the data entry system on the computers **before you leave the lab**.

Your results mark (/10) will be given to you immediately. This will count for 50% of your mark for this experiment. If the identity of your compound is incorrect, you should review your deductions and try again. A mark penalty will apply for each additional attempt down to a minimum of 5/10.