EXPERIMENT CODE

Rapid Determination of Enantiomeric Excess by NMR Spectroscopy

Student Name: (Use block capitals)	**Student ID No. (SRN)**

Group:	
Date of Expt:	
Partner's Name(s):	

Assessment Criteria

This proforma is worth a total of 50 marks.

The first part of this proforma will look at the accuracy of the data you acquired and the way you have interpreted the data to produce calibration curves. Additionally, it will assess your ability to use these calibration curves to determine the *ee* of unknown solutions.

The second part of this proforma will consist of two essay style questions which will assess your ability to critically evaluate the data you have obtained.

The final part of proforma will assess your understanding of the experiment and the science behind it with a series of shorter questions.

For more information on assessment criteria, see the laboratory manual.

List the five <u>known</u> *ee* calibration solutions you made:	
List the sample labels of your five <u>unknown</u> *ee* solutions:	

In the box below, paste both of your calibration curves (one constructed using the integrations of the imine proton peaks, and one using the integrations of the benzylic proton peaks) from either Excel or Sigmaplot. Ensure that the equation ($y = mx + C$) and correlation coefficient (R^2) of the best-fit line is included on each graph.

10 marks

Tabulate the determined *ee* of the unknown amine samples your group used below using the integrations of both the imine and benzylic proton signals.

Unknown amine sample number	Determined *ee* of amine using benzylic proton	Determined *ee* of amine using imine proton
1		
2		
3		
4		
5		
6		
7		

10 marks

In an ideal example what would you expect the line equation for the calibration curves to be?

1 mark

What does the R^2 value of the best fit line tell you?

2 marks

How could you improve the quality and reliability of your calibration curves?

2 marks

Consider both of your calibration curves and discuss their accuracy and precision, ensuring that you address the following points:

1. Quality of each calibration curve, judging by the R^2 values.
2. Anomalous points and reasoning for why they have occurred.
3. Accuracy of the individual points in the calibration curve – for example, did the sample intended to be 25% *ee* actually appear to be 25% *ee* in the NMR spectrum? If not, why not?
4. Which curve you feel will give the more accurate *ee* value.
5. Discuss the difference between accuracy and precision

7 marks

Consider the NMR data provided in the lab manual and also the spectra you produced:

1. Compare the NMR spectra of the starting materials (BINOL, MBA, FPBA) with the host complex produced. Are there any potential overlapping signals?

2. Are there any by-products or impurities present in the host complex NMR spectrum you recorded?

3. What effect will your answers to 1 and 2 have on the analysis of the NMR data?

4. Based on the NMR data, which signal do you think will give more accurate integrations and why?

7 marks

What is the function of molecular sieves and why are they added to this experiment? (State your literature reference)

3 marks

What sources of error were present in this experiment? How could these be overcome?

4 marks

Consider using this experimental procedure for the determination of the *ee* of other amines. Which NMR signals are general to this methodology (i.e. observed in any amine investigated) and which are specific to the use of α-methylbenzylamine?

2 marks

What are the limitations of the amines that can be studied in this experiment?

2 marks