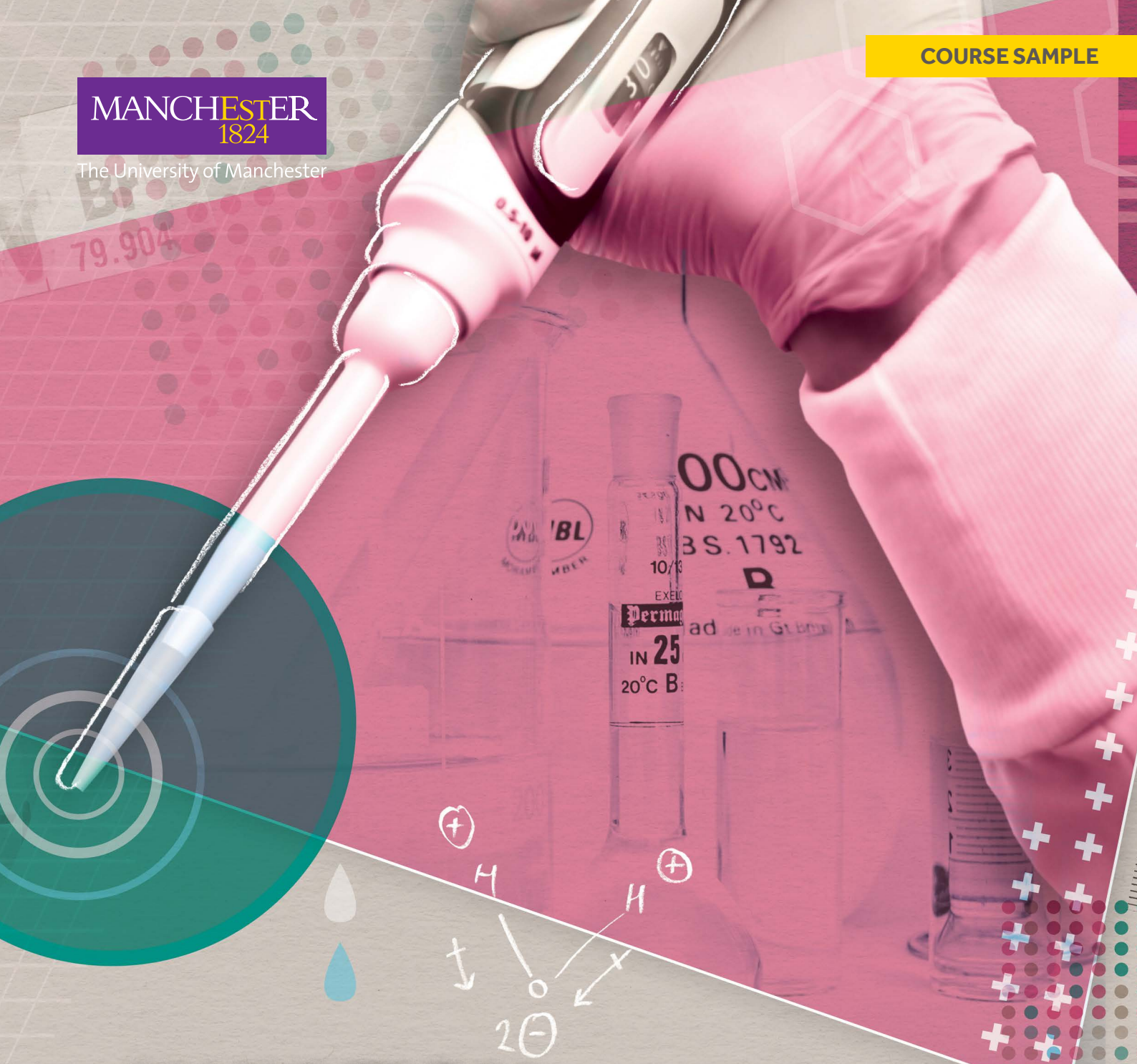
MANCHESTER 1824
The University of Manchester

COURSE SAMPLE



ANAYLTICAL CHEMISTRY AND MEASUREMENT SCIENCE

ONLINE, PART-TIME

www.manchester.ac.uk/analyticalchemistry

COURSE SAMPLE

ANALYTICAL CHEMISTRY AND MEASUREMENT SCIENCE

Online, part-time

1. Fundamentals of Analytical Science
1. CALIBRATION

2. Separation Science
1. VIRTUAL HLPC LAB

3. Mass Spectrometry
1. TANDEM MASS SPECTROMETRY PRESENTATION

Fundamentals of Analytical Science

CALIBRATION

SECTION INTRODUCTION

Welcome. This section will look at the different ways of calibrating individual analytical techniques. As calibration looks at converting instrument signal into calibration, these techniques can be used with any analytical technique. Calibration is a critical part of any Quantitative Analysis, and we will cover some of the basics of statistics while looking into the different techniques. As this is the last section of the Fundamentals of Analytical Science, it also hosts the final assessments for this unit - a report where you're asked to validate the data from one of the practicals and a reflective essay utilising the journal entries you've been keeping throughout this unit. Information on the assignments and details on deadlines can be found here and in the Assessment area on Blackboard. Let's get started!

LEARNING OBJECTIVES

Select the appropriate techniques for trace or bulk analysis

Identify unknowns through IR and UV/Vis spectroscopy and quantify using calibrations

Validate your data using an appropriate regulatory body

COURSE SAMPLE

Fundamentals of Analytical Science

CALIBRATION

CONTENTS

EXTERNAL CALIBRATION

Reading

Before we get started, it will help you to read the following:

Chapter 5, **Calibration methods in instrumental analysis: regression and correlation**, of Statistics and Chemometrics for Analytical Chemistry by James N. Miller and Jane C. Miller.

*Return to Blackboard and access **Reading List** from the link on the left-hand menu.*

External Calibration

External calibration is the most popular form of calibration for analytical techniques and is based on having a series of different concentration standards from which instrumental responses are measured. If this is used to plot an external calibration graph, then an unknown sample can be measured, and the signal obtained can be used to obtain a concentration when the signal is compared with the external calibration graph.

Knowledge check - Worksheet

Below you will find a worksheet with three exercises that accompany the learning materials for this section. You're highly encouraged to solve these after you've read the recommended reading and watched the relevant video lectures. Two of the execrises can be found further down as discussion points.

It's entirely up to you when and how you want to engage with them, but it'll be beneficial to post your answers on the discussion forum and discuss them with the other students before you submit your assignments. The correct answers will be revealed by the tutors later this week.

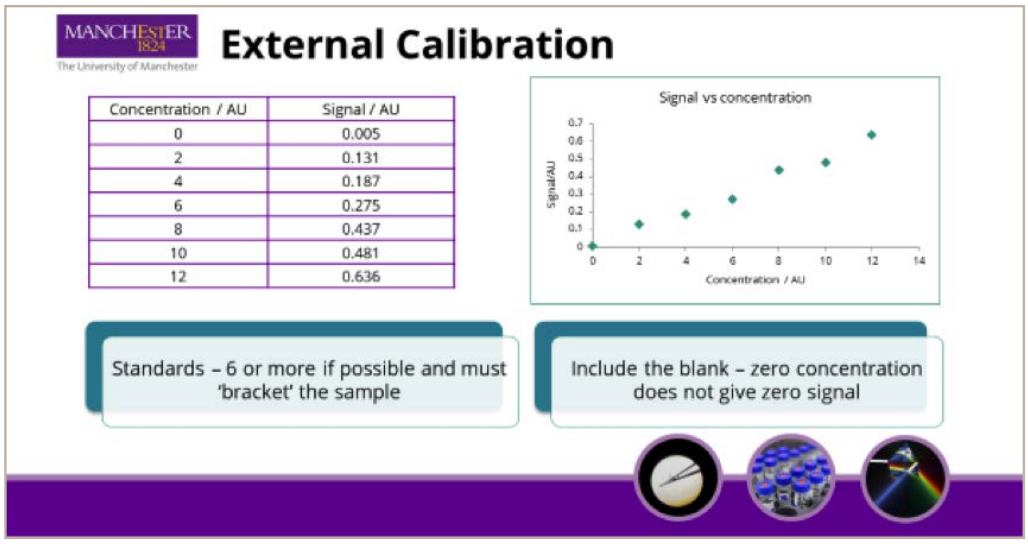
Try Calibration practice worksheet.

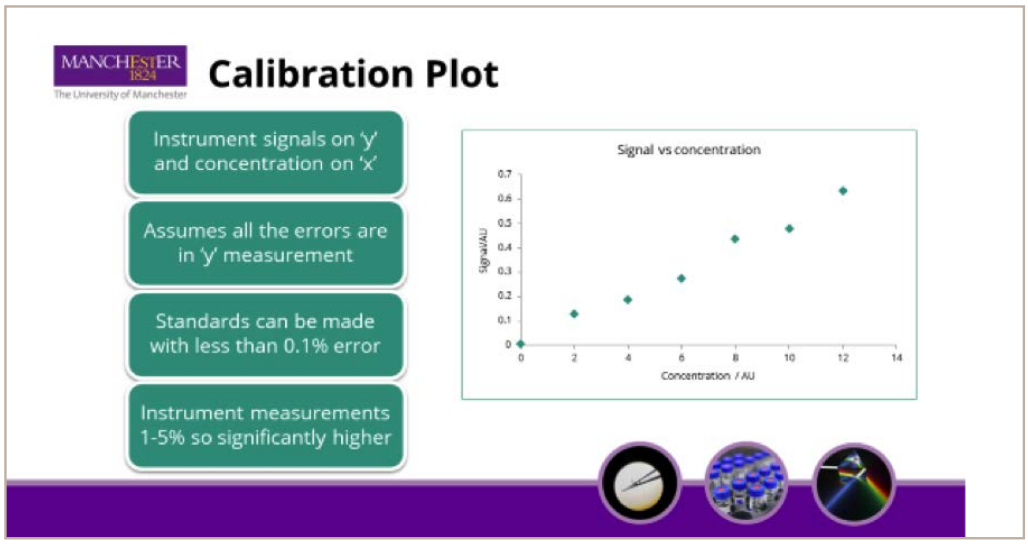
"The course content and overall experience were above my expectations."

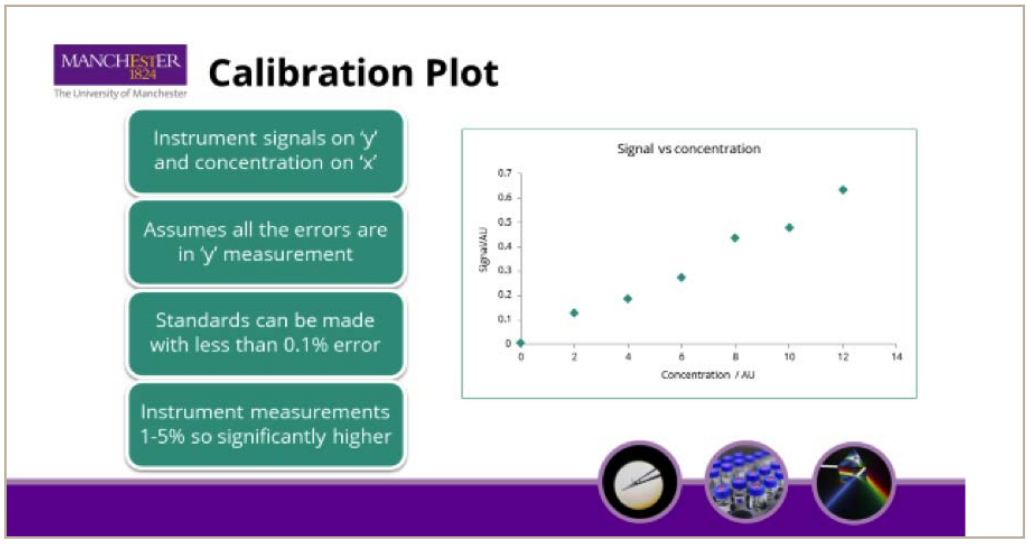
COURSE SAMPLE

EXTERNAL CALIBRATION

Instructional videos







COURSE SAMPLE

EXTERNAL CALIBRATION

Discuss

Considering what we've covered so far, have a go at solving this exercise and discuss the conclusions you've drawn from your results on the discussion forum.

A blood sample is analysed for ethanol level by internal standardization using propan-2-ol as the internal standard. Using the data below calculate the level of ethanol in blood by construction of an appropriate graph. Comment on the level found if you assume the legal limit is 80 mg/100 ml. A level of 81 mg/100 ml was found by external calibration. What are your thoughts on any differences between the two results and what may have caused the differences?

Conc mg/100ml	Ethanol	Propan-2-ol
40	12465	23567
80	26435	25789
120	34786	22564
160	51908	24534
200	61516	23415

Sample	
Ethanol	Propan-2-ol
25641	34125

Discuss your thoughts in the dicsussion forum.

*Return to Blackboard and access **Discussions** from the link on the left hand menu.*

Discuss

In this discussion, we look at validation. The best approach would be to consider what we need for a valid analysis by looking at a set of regulations. So how would we check for accuracy and precision? How linear is the technique? How can we test robustness - what about interferences?

Use the discussion to look at some important points when guaranteeing the results obtained. Consider these questions when writing your assignment report for this section. To discuss your views on the above questions, go to the *Discussion Forum*.

*Return to Blackboard and access **Discussions** from the link on the left hand menu.*

Check your answers for the calibration practice worksheet: Calibration Q&A Chapter 10.

Assessment

What would you need to do to the UV-Vis practical to validate the method to accept the data? OR

How would you validate a method that you are currently using at your place of work? OR

How have you validated a method at work?

Please write a report, not in IMRaD format but considering the headings provided in the Validation Assessment Guidelines, of approximately four pages to fully account for your approach. Please include visual elements to enhance your report (if possible).

*Return to Blackboard and access **Assessments** from the link on the left hand menu.*

Assessment - Reflective essay

For the final journal entry you are requested to think about your progress on this unit and the skills you have developed through the learning materials, personal reading, and assignments and write a critically reflective essay. You should write and submit this essay through the Assessments area on Blackboard and **not as a new journal entry**.

*Return to Blackboard and access **Assessments** from the link on the left hand menu.*

"This course has helped me improve as an analyst, furthering my expertise in analytical testing as not only do I understand the chemistry, I have more knowledge on the instrumentation and how everything links together in the technique."

Separation Science

VIRTUAL HLPC LAB

SECTION INTRODUCTION

An introduction here as to what this section is about, rera eate odis aut inci dolora quunt que ditat voluptatio quo te mod maximus eost, coribus, offic totat hariore mpeliqui officidemo te recatiasima volum ressequis ea nonsequasint fuga. Ore offictus eium nullit officaborem et pero to essi consequam, que eat esequam dolese nim faccum faccumq uisitio nsequam, ullectota doluptat unditatempos mod quiasi ratem aut abor aut pro del int fuga. Nam quunt quo tet quatibus.

LEARNING OBJECTIVES

Use chromatographic data to perform calculations on chromatographic parameters

Select and justify appropriate experimental conditions for HPLC separation of a given system

Predict how column conditions and choice of stationary phase affect chromatographic separation performance

COURSE SAMPLE

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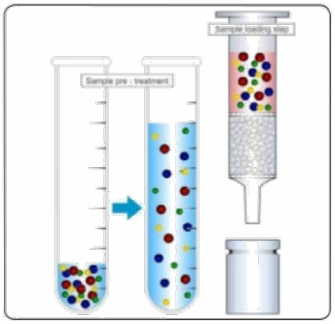
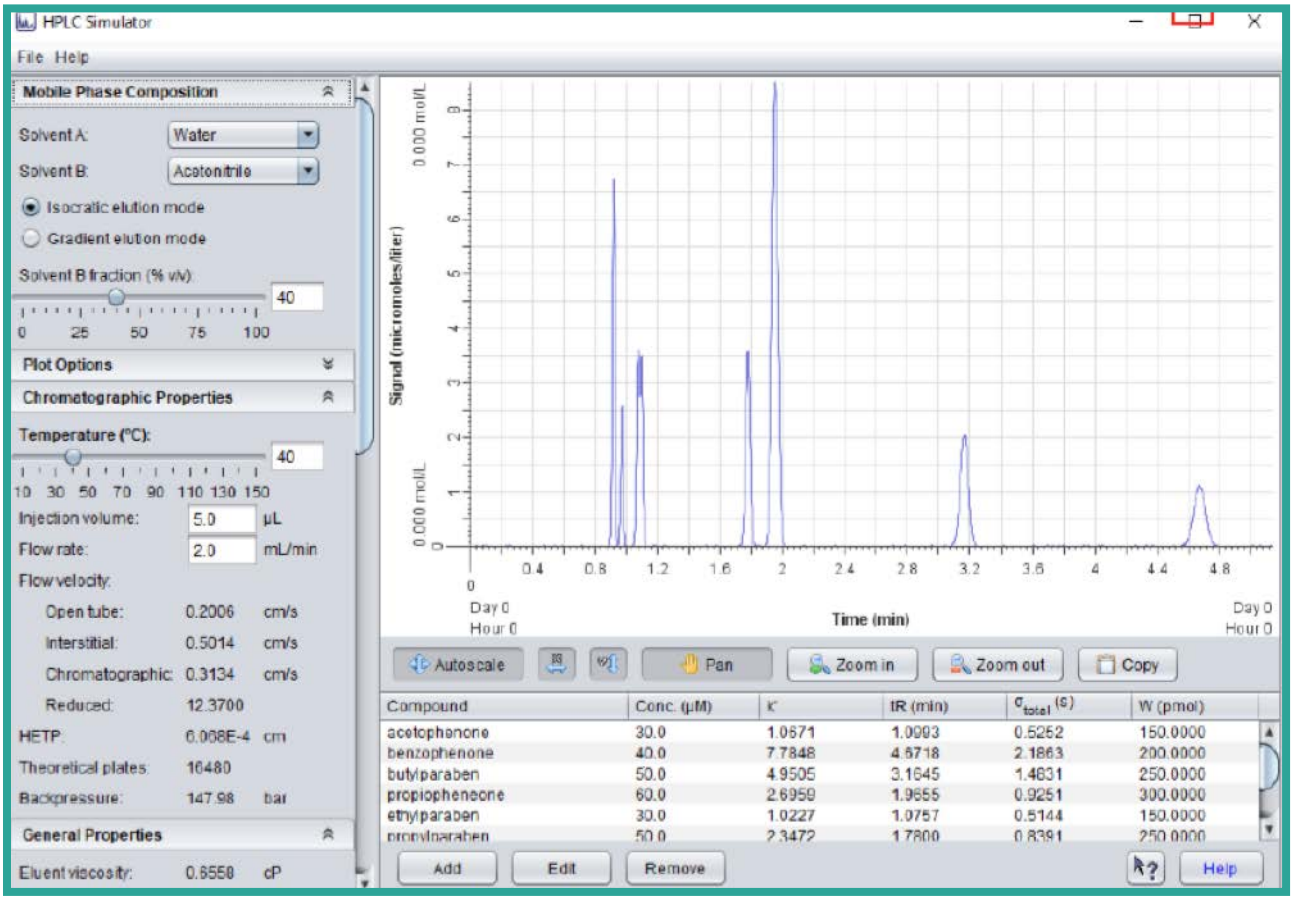
powered by crawford scientific

USING HPLC SIMULATION SOFTWARE



PROCEDURE

Record the result of the following procedure in electronic form, using Microsoft Excel and Word software to tabulate and process your results in suitable formats and compile an electronic lab report for submission.



- Run the HPLCSimulator software. Add 30 µM of acetophenone, ethylparaben, propylparaben, ketoprofen, 3-nitrophenol.
- Chromatographic parameters: i) isocratic elution mode: solvent A- water, solvent B- acetonitrile (AcN), column temperature 40 oC, injection volume 20 µL, flow rate 2.0 mL/min, HPLC column length 100 mm, inner diameter 4.6 mm, particle size 3 µm, stationary phase Waters Acquity BEH C18.
- Predict what will happen to the retention time (tR) as %AcN is increased. Change the mobile phase composition (ϕ) from 10% to 70% AcN (ϕ = 0.10 to 0.70) in 10% steps. Record tR for each compound at each value of ϕ and note which mobile phases give baseline separation of all five compounds.
- Select a compound. Use the simulator void time (tm) to manually calculate the capacity (retention) factor (k') for each ϕ value. Compare to the simulator values.

COURSE SAMPLE

USING HPLC SIMULATION SOFTWARE

- Plot graphs of the capacity (k') of each compound as the mobile phase composition is changed in the forms given by the following equations:

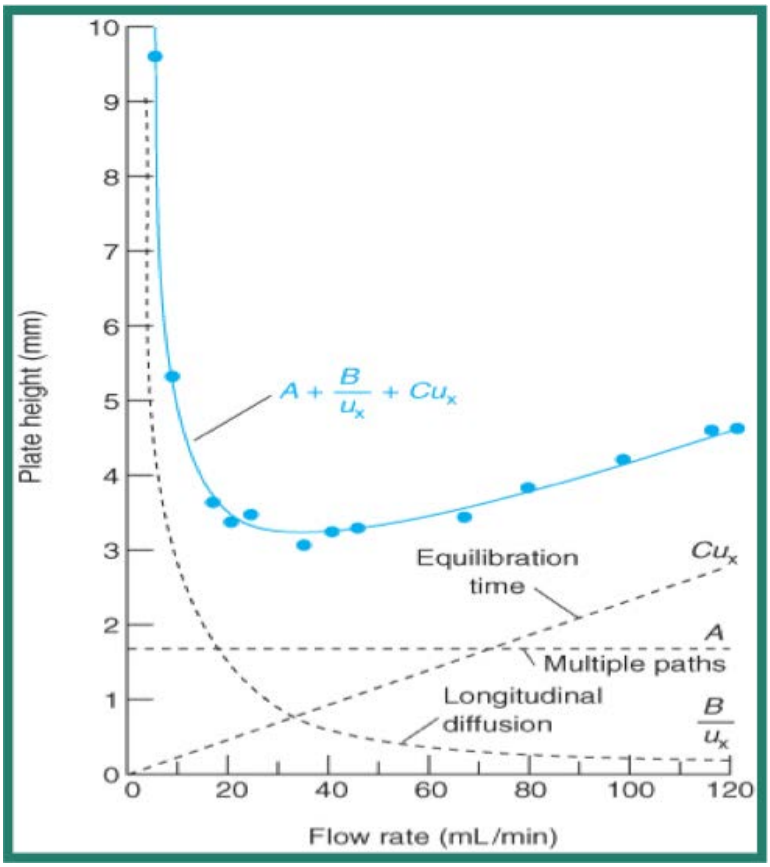
$$K' = a + b\,\phi \quad \ln k' = a + b\,\phi$$

- Use the linear regression tool to decide which of the above formula best represent the mathematical relationship between k' and ϕ. We now have a method to predict the capacity factor for any water/AcN solvent composition.
- With acetophenone, use the equation for the best-fit line of your simulated data to predict the k' value using a mobile phase of 25% AcN in water. Record your prediction and compare it to the simulated data.
- Use the best-fit line equations for each of the five compounds to calculate k'calc over the range ϕ = 0.10 to 0.50 at intervals of 0.02. What are the advantages of using the simulator approach to do this?
- Using the predicted k'calc values, note the two components that are least well resolved at each ϕ value. At each ϕ value, from the two least resolved peaks (with k'calc=k'1 and k'2), calculate the natural logarithm of the selectivity (α).

$$\ln \alpha = \ln k'1 - \ln k'2$$

- This gives optimum ϕ values for separation under these column conditions.
- Adjust the column temperature 30-60 oC for baseline separation of all compounds with minimum run-time. Note the last-eluting compound's optimum temperature and retention time tR(max). Check if small changes to ϕ improve the result.

Note the value of the number of theoretical plates (column efficiency) N given by the simulator. Compare this value with N(max) given in theory for a column of this type. Simulate the theoretical plate height (HETP) as a function of mobile phase velocity (0.1-10 mL/min) to generate a van Deemter plot. Record the back pressure required for each flow rate. What is the optimum value predicted by the simulation for N?



USING HPLC SIMULATION SOFTWARE

- Switch to a typical UHPLC column: stationary phase particle size 1.5 µm, column length 50 mm, column internal diameter 2.1 mm, injection volume 1.4 µL. Adjust the flow rate to backpressure <2000 bar. Note the effect on peak separation and tR(max). Adjust the column length 40-100 mm to resolve all peaks to baseline with minimum run-time. Compare the column efficiency NUHPLC with the HPLC column.

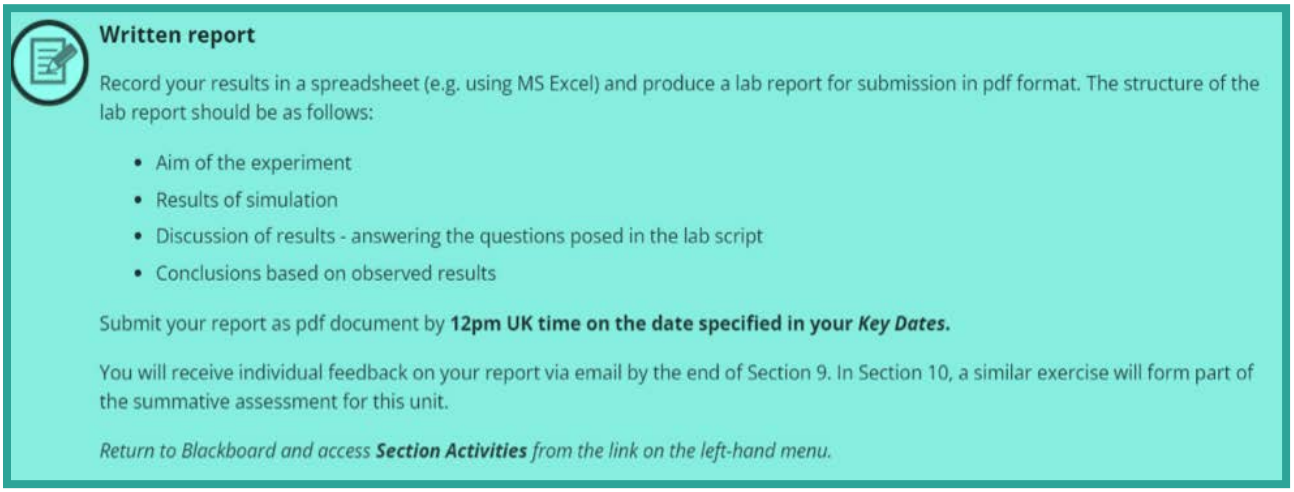
- Predict the composition (ϕ) of a mobile phase consisting of methanol and water with the same eluent strength as the optimal water/AcN mixture. Using the simulator with the optimum UHPLC column, determine the methanol/water composition, with baseline separation and minimum run-time. Is water/AcN or water/methanol better?

Discuss

Share your impressions of the HPLC simulator on the discussion board.

What did you learn from it? How could the software be improved in terms of its functionality at performing accurate simulations and its ease-of-use?

*Return to Blackboard and access **Discussions** from the link on the left-hand menu.*



Written report

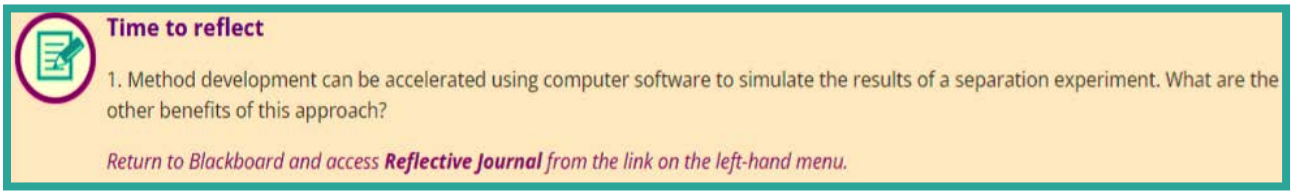
Record your results in a spreadsheet (e.g. using MS Excel) and produce a lab report for submission in pdf format. The structure of the lab report should be as follows:

- Aim of the experiment
- Results of simulation
- Discussion of results - answering the questions posed in the lab script
- Conclusions based on observed results

Submit your report as pdf document by **12pm UK time on the date specified in your *Key Dates*.**

You will receive individual feedback on your report via email by the end of Section 9. In Section 10, a similar exercise will form part of the summative assessment for this unit.

*Return to Blackboard and access **Section Activities** from the link on the left-hand menu.*



Time to reflect

1. Method development can be accelerated using computer software to simulate the results of a separation experiment. What are the other benefits of this approach?

*Return to Blackboard and access **Reflective Journal** from the link on the left-hand menu.*

"Overall, this simulation experiment provided valuable practical experience and enhanced my understanding of chromatography principles and method development. It reinforced the importance of careful planning, data analysis, and interpretation for successful HPLC separations. I feel more confident using HPLC simulation software and applying the knowledge gained to real chromatographic analyses in my professional setting."

Mass Spectrometry

TANDEM MASS SPECTROMETRY PRESENTATION

SECTION INTRODUCTION

In this section, we will formally discuss the method and instrumentation that enables tandem mass spectrometry experiments. In the previous section, we saw how ion traps can perform several rounds of tandem MS experiments – MSN. We will start by considering the different tandem mass spectrometry methods that are commonly employed, then we will consider the construction of mass spectrometers, and finally, we will look at different fragmentation methods.

LEARNING OBJECTIVES

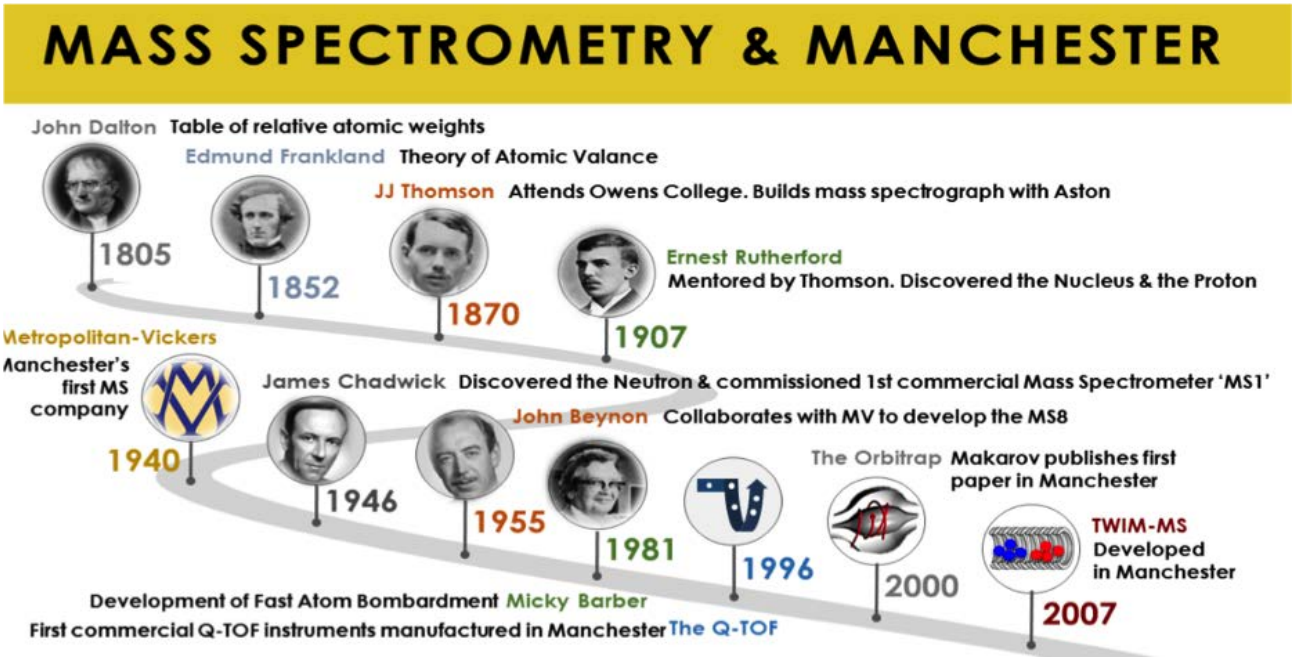
Describe common tandem mass spectrometry experiments

Describe how hybrid mass spectrometers allow tandem mass spectrometry experiments to be performed

Suggest a suitable tandem mass spectrometry workflow for quantitation of unknown compounds from a complex mixture

COURSE SAMPLE

TAMDEM MASS SPECTROMETRY



Presentation

Research commercially available mass spectrometers on the websites of the following manufacturers:

- SCIEX
- WATERS
- BRUKER
- THERMOFISHER
- SHIMADZU
- AGILENT

Prepare a PowerPoint presentation consisting of up to ten slides that you would use in a company/work environment that would support the purchase of a given mass spectrometer. Either use real examples of use from your work environment or from what you have read. Consider what you need it for and what type of analysis you will be doing with it.

Your presentation should cover the performance characteristics of the chosen analyser, speed, sensitivity, mass accuracy and resolving power. It should also give some consideration to the ease of use, the footprint of the instrument and the cost to purchase and to maintain. You should include details of any hyphenated separation capabilities it may have e.g HPLC, ion mobility.

Make a recording of your presentation in PowerPoint and submit it according to the instructions in **Assessment**.

*Return to Blackboard and access **Assessment** from the link on the left-hand menu.*

"As I am dealing with laboratory results daily, I find the applications of mass spectrometry the most interesting part of this course. However, I appreciate that mass spectra for complex biomolecules are difficult to interpret, and considerable experience is required."

COURSE SAMPLE

Online and Blended Learning

ANAYLTICAL CHEMISTRY AND MEASUREMENT SCIENCE

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