Exam Code(s) 3EV2 and 3BS9
Exam(s) Third Science Examination
Module Code(s) CH327
Module(s) Validation and Industrial Chemistry

External Examiner(s) Professor K. Molloy
Internal Examiner(s) Dr. L. F. Jones
  Dr W. Carroll
  Mr Ray McCarthy
  Dr A. G. Ryder
  Dr J. Wurmel

**Instructions:** Answer 2 questions from each section (**4 questions in total**) 
Use separate books for each section

3BS9 Students answer questions from sections A and B only
3EV2 Students answer questions from sections B and C only

All questions will be marked equally

Duration 2 hours
No. of Pages 8
Department(s) Chemistry
Course Co-ordinator(s) Dr. L. F. Jones

**Requirements:**
MCQ
Handout
Statistical Tables
Graph Paper
Log Graph Paper
Other Material
Section A

1. Answer all parts
(i) With respect to Process Analytical Technology (PAT), explain the following terms.

(a) Process Understanding [5 Marks]
(b) Design-of-Experiment [5 Marks]
(c) Chemometrics [5 Marks]

(ii) With respect to pharmaceutical manufacturing, briefly explain the difference between traditional laboratory testing (quality assurance) and using at-/in-/on-line measurements. Give three advantages to using at-/in-/on-line analysis.

[10 Marks]

2. Answer all parts

(i) The following terms are all associated with Validation with respect to the Bio- and Pharmaceutical industries. Define each term in a clear and concise manner.

(a) Good Manufacturing Practice [5 Marks]
(b) Process Validation [5 Marks]
(c) Validation Master Plan [5 Marks]

(ii) Explain the aims and objectives of the Validation Master Plan and discuss its contents.

[10 Marks]

3. Answer all parts

Professor William S. Hankley is about to embark on a University-Industry collaborative study to be carried out in his laboratory at his University workplace. He will need to act on the following topics regarding Good Laboratory Practice including the production of a Study Plan.

(i) Explain what is meant by the term Study Plan and discuss its aims and specific contents.

[13 Marks]
(ii) Professor Hankley is the Study Director for this project. Give four responsibilities associated with his role.

[12 Marks]

4. Answer all parts

(i) In your own words, describe the term medical device? In your answer give examples of 3 different medical devices and explain how they fit this description.

[10 Marks]

(ii) How does the classification of a medical device impact its approval process within the US? In your answer describe why a manufacturer may require a pre-market approval (PMA) for certain device classes and a pre-market notification (510K) for others?

[15 marks]

5. Answer all parts

(i) Describe a medical device that may be used in the treatment of damaged heart valves? In your answer give the classification of the device and explain the reasons for this classification?

[10 Marks]

(ii) Give a brief description of one heart condition that can be treated with a cardiac rhythm management (CRM) medical device? In your answer describe how the device is used to treat the disease.

[10 marks]

(iii) Why might an investigational device exemption (IDE) be required prior to obtaining US approval for either of the examples given in part (i) and (ii) of this question?

[5 marks]
Section B

6. Answer all parts

(i) Aluminium occurs as bauxite, which contains aluminium oxide with impurities mainly of iron oxide and silica. Outline how bauxite is processed to give pure aluminium oxide, showing clearly the chemical basis for the method.

[7 marks]

(ii) Aluminium is produced by electrolysis of purified aluminium oxide. State briefly the necessary conditions used and sketch and label the cell used. Give the reaction at each electrode.

[8 marks]

(iii) Describe the advantages and disadvantages of homogeneous and heterogeneous catalysis and the reactor types that suit each type of catalyst.

[10 marks]

7. Answer all parts

(i) Outline the Monsanto process for the production of acetic acid.

[8 marks]

(ii) What is an alkene (olefin) metathesis reaction?

[4 marks]

(iii) Sketch the mechanism of an alkene metathesis reaction.

[4 marks]

(iv) Which are typical metals used in metathesis catalysts

[4 marks]

(v) Draw the chemical structure of the Grubbs 1st. and 2nd. Generation metathesis catalysts.

[5 marks]

8. Answer all parts

Ammonia is manufactured from hydrogen and nitrogen in the Haber Process: \( \text{N}_2(\text{g}) + 3\text{H}_2(\text{g}) \rightarrow 2\text{NH}_3(\text{g}) \ \Delta H = -92 \text{kJ mol}^{-1}. \)

(i) State the conditions employed industrially in the manufacture of ammonia, and justify them on physico-chemical grounds.

[13 Marks]
(ii) In the production of sodium hydroxide, brine is the raw material used. It is electrolysed in a diaphragm cell using a titanium anode and a steel cathode, separated by a porous asbestos diaphragm. Outline the reasons for the use of the following in the production of sodium hydroxide by this method:

(a) brine, rather than sea water  
(b) a titanium anode rather than a steel one

Suggest why the brine must be purified to remove calcium and magnesium ions.

[12 marks]
Section C

9. Answer all parts

(i) Explain the purpose and fundamental theory of chromatography using the identification of amino acids with thin layer chromatography as an example. In your answer use and define the following terms: eluent, eluate, mobile phase, stationary phase, retention time, polarity, adsorption chromatography, partition chromatography, polarity, affinity, ninhydrin, theoretical plates and band spreading.

[10 marks]

(ii) The retention time of a solute Z is 232s. The peak width at half-height is 5.23s and the column length is 10m. Calculate the number of plates and the plate heights.

[10 marks]

(iii) Outline the advantages and disadvantages of GC and LC.

[5 marks]

10. Answer all parts

(i) Briefly explain, using diagrams, the following three main components of a mass spectrometer:

   (a) electron impact ionization,
   (b) magnetic sector mass analyser, and
   (c) scintillation counter.

In outlining the overall spectrometer follow the species under examination from injection to detection.

[10 marks]

(ii) The mass spectrum of molecule M contains a molecular ion peak at m/z =134 and major fragmentation peaks at m/z = 105 and 77. Identify the species responsible for the peaks at m/z = 105 and 77. Write an equation for the formation, from the molecular ion of the species responsible for the peak at m/z=105.

[10 marks]
(iii) The mass spectrum of butanoic acid is shown below. Indicate which line in the spectrum shows the substance has molecular ion peak of 88. Explain your choice.

[5 marks]

11. Answer all parts

The details of a HPLC analysis of a number of penicillins are provided below.
(i) Provide a schematic diagram of the HPLC system that would be required to carry out the analysis above. [5 marks]

(ii) Identify the type of column that is used in this analysis and justify your choice. List all three analytes in order of polarity starting with the most polar one. [5 marks]

(iii) The detector used in this analysis was a variable wavelength UV/Visible detector. Draw a simple diagram showing how this type of detector works. [5 marks]

(iv) Explain the difference between the detector used here and a fluorescence detector. Provide a diagram of the latter and outline its main advantage. [5 marks]

(v) In HPLC sample injection is achieved using an injection valve. Draw a schematic of an injection valve and explain how an injection valve works in principle. Explain why the sample cannot be injected using a syringe through a rubber septum. [5 marks]

12. Answer all parts

(i) Describe, with the aid of diagrams the principles of X-Ray fluorescence? [12 marks]

(ii) Describe, with the aid of a labelled diagram the process of auger electron emission. What is the consequence of this process for XRF? [7 marks]

(iii) Outline the construction and mode of operation of the following energy dispersive X-ray fluorescence spectrometers: a secondary target and a total reflection based system, and a radioisotope sourced system. [6 marks]