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

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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
Please use separate answer books for each section

Section A

1. With respect to manufacturers and regulators within the pharmaceutical industry, describe the term validation and discuss its origins, aims and objectives. [25 marks]

2. Answer all parts

(i) Briefly discuss the concept of Process Analytical Technology (PAT). [9 marks]

(ii) Give three advantages and three disadvantages in implementing Process Analytical Technology with respect to pharmaceutical manufacturers. Explain your answers. [12 marks]

(iii) Give two advantages in implementing PAT with respect to the regulatory bodies. [4 marks]

3. Answer all parts

(i) With respect to Cleaning Validation, briefly explain the following concepts:

(a) Test-Until-Clean
(b) Clean-In-Place
(c) Bracketing
(d) Rinse Sampling
(e) Swab Sampling

[20 marks]

(ii) Briefly explain the difference between Limit of Detection and Limit of Quantification. [5 marks]

4. Development of an IVIVC model for drug eluting stents is recommended by the American Food and Drug Administration (FDA). What is an IVIVC model, how is one developed, and what is it used for? [25 marks]
5. Answer all parts

Congenital heart failure (CHF) can be caused by a damaged aortic valve. Answer the following questions relating to the disease:

(i) Describe an active medical device that can be used to treat CHF, and provide details on how it functions.

[13 marks]

(ii) TAVI is a valve treatment that is recently gaining in popularity. What is TAVI, and explain some of its advantages and disadvantages as a clinical treatment?

[12 marks]
Section B

6. Answer all parts

(i) Describe the large scale catalytic synthesis of ammonia from elemental hydrogen and nitrogen, commenting on the effect of reaction conditions on yield of product.  

[12 marks]

(ii) Aluminium metal is manufactured by a process in which purified bauxite, dissolved in molten cryolite is electrolysed at 800°C. Graphite electrodes and a high current are used.

(a) Give the ionic equations for the reactions taking place at each electrode.
(b) Explain why the anodes need to be replaced frequently.
(c) Explain why an electrolyte of pure molten bauxite is not used.
(d) What are the main impurities in bauxite ore?

[13 marks]

7. Answer all parts

(i) Describe the Frasch Process for the production of molten sulphur.

[10 marks]

(ii) Describe the Ziegler process for the polymerisation of alkenes and compare the conditions of the process with those of the free radical process for alkene polymerisation.

What are the advantages offered by the recently developed metallocene catalysts?

[15 marks]

8. Answer all parts

(i) Catalytic cracking, reforming and isomerisation reactions are important in refinery processes. Why is this so?

[10 marks]

(ii) The introduction of dimensionally stable anodes into the cell systems used in the chlor-alkali industry has led to very substantial cost savings. Discuss.

[10 marks]

(iii) Give some of the main uses of the two main products produced in the chlor-alkali industry.

[5 marks]
Section C

9. Answer all parts

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

(a) electron impact ionization
(b) TOF mass analyser
(c) scintillation counter

In outlining the overall spectrometer it may be helpful to follow the species under examination from injection to detection.

[10 marks]

(ii) The electron impact ionisation spectrum of propanoic acid (CH$_3$CH$_2$COOH) is given below. Account for the major four peaks (as indicated) in the spectrum. Explain a plausible fragmentation mechanism that will lead to the formation of the respective ions.

[10 marks]

(iii) The atomic masses of some elements are shown below:

- Hydrogen 1.0078
- Carbon 12.0000
- Nitrogen 14.0031
- Oxygen 15.9949
Using the data above, explain why the molecules C₆H₁₂, C₅H₈O and C₄H₈N₂ can be distinguished only using high resolution mass spectrometry. Calculate the resolution factor that would be required.

[5 marks]

10. Answer all parts

(i) Explain the purpose and fundamental theory of chromatography using column chromatography as an example. In your answer use and define the following terms: adsorption chromatography, partition chromatography, mobile phase, stationary phase, retention time, polarity, affinity, theoretical plates, plate heights, Gaussian distribution and band spreading.

[10 marks]

(ii) Explain what is meant by the term ‘theoretical plates’ and explain what information it provides about a chromatographic experiment. The retention time of a solute A is 218s. The peak width at half-height is 2.65s and the column length is 12m. Calculate the number of plates and the plate heights.

[9 marks]

(iii) List 3 major limitations of each of GC and LC.

[6 marks]
11. Answer all parts

The details of a HPLC analysis of a number of barbiturates are provided below

Barbiturates (HPLC)

Column: Discovery C8, 15cm x 4.6mm ID, 5μm particles
Cat. No.: 59353-U
Mobile Phase: 55:45 water:MeOH
Flow Rate: 1.0mL/min
Det.: UV at 214nm
Temp.: ambient
Inj.: 5μL

(i) Provide a schematic diagram of the HPLC system that would be required to carry out the analysis below.  

(ii) The column stationary phase used is of C8 type. Explain what is meant by this term and why knowing that the column used is of this type tells which analyte is the most polar. List all seven analytes in order of polarity starting with the most polar one.  

(iii) What type of detector is used in the analysis? Draw a simple diagram showing how this type of detector works.
(iv) Explain the difference between the detector used here and a diode array detector. Provide a diagram of the latter and outline its main advantage.

[5 marks]

(v) The stationary phase used in the analysis above is C8 (reverse phase). Explain what that means providing a diagram and list two other types of stationary phases that can be used in reverse phase chromatography.

[5 marks]

12. Answer all parts

(i) Describe with the aid of diagrams how GPC and scintillation detectors used in XRF spectroscopy work.

[15 marks]

(ii) The Kα, Kβ and Lα lines of the x-ray fluorescent spectrum of rhodium occur with energies (keV), 22.721, 2.694 and 20.214. Assign the correct energy to each of the lines and explain why you make the specific assignments.

[3 marks]

(iii) Sketch a typical absorption edge diagram and explain the features that are present.

[7 marks]