

**Ollscoil na hÉireann, Gaillimh**  
***National University of Ireland, Galway***

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**Autumn Examinations, 2001-2002**

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**Third University B.Sc. Examination in Science**  
**(Including denominated degrees)**

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**CH304 - Analytical and Industrial Chemistry**

Professor J. Evans  
Professor R. N. Butler  
Professor M. J. Hynes  
Professor P. McArdle  
and Internal Examiners

Time allowed: **Two hours**

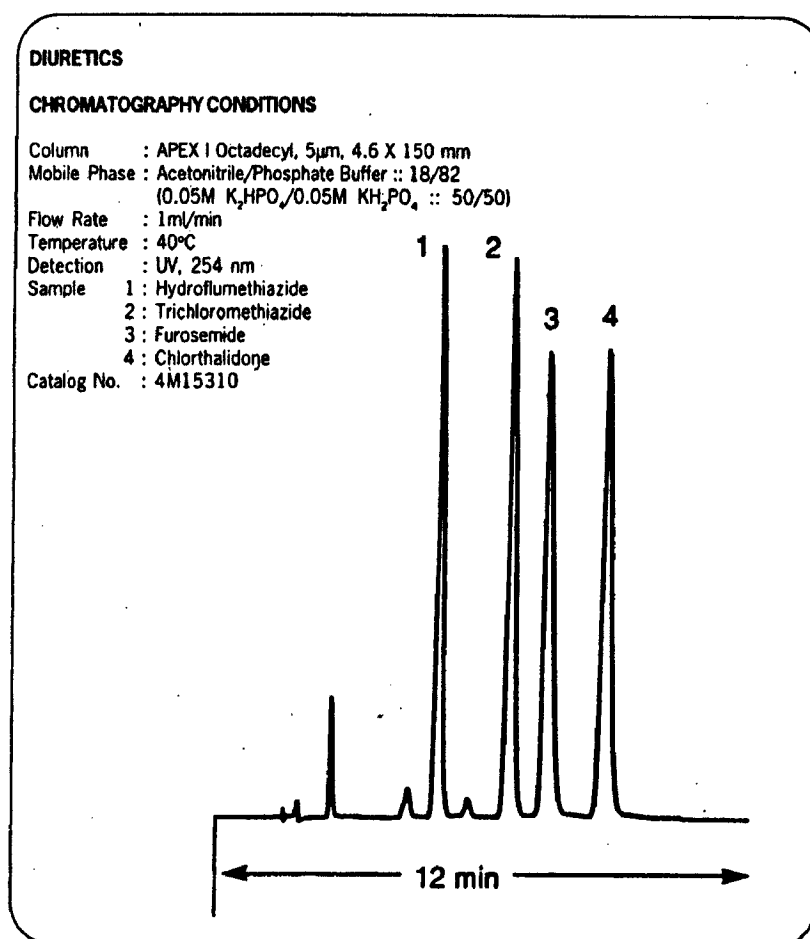
**Answer four questions**

**All questions carry equal marks. Leave the first page of the Answer Book blank and list on it clearly the numbers of the questions attempted.**

1. Answer (a), (b) and (c)
  - (a) Describe the type of useful chemical information that you might obtain from a commercial chemical catalogue ( e.g. Aldrich, Lancaster, Fluka etc.). [9 marks]
  - (b) Write a brief note on the Merck Index and the information contained therein. [ 8 marks ]
  - (c) Discuss concisely from chemists' perspective the CRC Handbook of Chemistry and Physics i.e. the so-called 'Rubber' Handbook. [ 8 marks ]
2. Answer (a), (b) and (c)
  - (a) Write a note on the basic construction and mode of operation of wavelength and energy dispersive x-ray fluorescence spectrometers. [8 marks]
  - (b) Briefly outline the problems associated with sample preparation for quantitative analytical applications of x-ray fluorescence spectroscopy. [9 marks]
  - (c) Outline the advantages of x-ray fluorescence spectroscopy as an analytical technique. [8 marks]

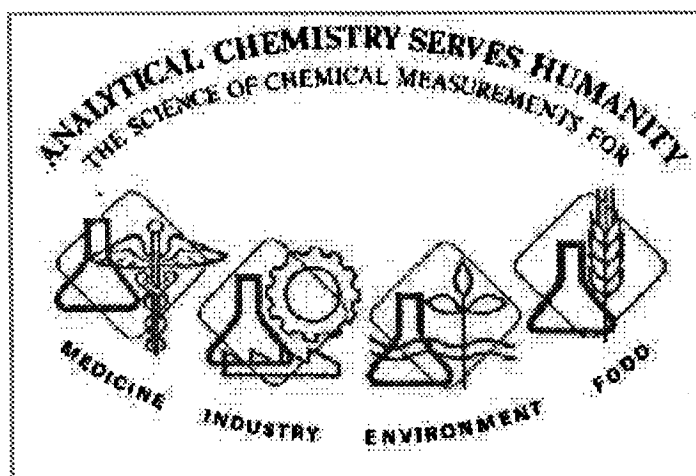
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3. The details of a HPLC analysis of a series of pharmacologically active molecules which function as diuretics are provided below. The mobile phase used is a mixture of acetonitrile and an aqueous phosphate buffer.
- (a) Provide a schematic diagram of the HPLC system that would be required to carry out such an analysis. [5 marks]
  - (b) Draw a diagram showing a section of the surface of the stationary phase particles. [4 marks]
  - (c) Identify the general type of column used and, explaining your answer, indicate whether you consider the choice of mobile phase appropriate for such a column. [4 marks]
  - (d) Which is the least polar of the compounds analysed? Explain your answer. [4 marks]
  - (e) In order to increase the throughput of samples, it is suggested that using the same column you develop chromatographic conditions which would result in a shorter analysis time. What kind of changes would you make? Explain your answer. [4 marks]
  - (f) The HPLC analysis of a urine sample from a patient gives a peak with a retention time which is identical to that of furosemide. Assuming you have access to any appropriate instrumentation, suggest two methods by which you could confirm that that the peak is in fact due to this material? [4 marks]



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4. The following is the logo of the analytical section of the American Chemical Society. Write an essay on whether or not you think this is a true reflection of the role of analytical chemistry today. [25 marks]



5. Of all the thermal analysis techniques available, Differential Scanning Calorimetry (DSC) has proved to be the most useful in the pharmaceutical industry. Outline some of the applications of this technique in the pharmaceutical industry, highlighting its advantages and disadvantages over other analytical techniques with which you are familiar. [25 marks]
6. Answer (a) **and** (b).  
(a) Briefly describe the history of the chlor alkali industry. [12 marks]  
(b) Describe the three most important electrolytic methods used for the production of NaOH and Cl<sub>2</sub>. [13 marks]
7. Answer **each** of the following  
(a) Outline the principle differences between Laboratory and Industrial Procedures. [12 marks]  
(b) Describe the Monsanto Acetic Acid Process and the Wacker process. [13 marks]
8. Answer (a), (b), (c) **and** (d).  
(a) Describe the detectors used in gas chromatography. [6 marks]  
(b) With reference to gas chromatography write brief notes on each of the following:  
i) packed columns [3 marks]  
ii) theoretical plates [3 marks]  
iii) flow rate [3 marks]  
iv) resolution [3 marks]  
(c) A packed column is 2250 mm in length and has a height equivalent of a theoretical plate of 1.5 mm. Calculate the width of chromatographic peaks with retention times (tr) of 2.5 min and 3.2 min. Calculate the resolution between these peaks. [7 marks]