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MAIN EXAMINATION

JANUARY – APRIL 2014 TRIMESTER

FACULTY OF SCIENCE

DEPARTMENT OF NATURAL SCIENCE

REGULAR PROGRAMME

CHEM 100: INTRODUCTION TO LABORATORY TECHNIQUES

Date: APRIL 2014	**Duration: 2 Hours**
INSTRUCTIONS: Answer Question ONE and ANY OTHER TWO Questions. Start each question on a new page.	

Q1. a) i) Burettes and pipettes can be stored either vertically or horizontally. Which way is better and why? **(2 marks)**

ii) The most common cause of the breakage of measuring cylinders is their being knocked over becoming cracked at the top. Suggest a way of preventing this damage, without impairing the efficient use of them. **(1 mark)**

iii) You want to use a metal clamp to hold flask and you notice that the jaws of the clamp are bare metal. What should you do to ensure a firm hold on the flask. **(1 mark)**

iv) Brown rubber tubing soon perishes here in East Africa. It becomes hard and cracks and loses its flexibility and elasticity. Why is this and what should you do to reduce this tendency and extend its useful lifetime? **(2 marks)**

b) i) You come across two pieces of Quickfit apparatus that are stuck together at the ground glass joint. How would you attempt to separate them without breaking them? **(2 marks)**

ii) You have just finished an inorganic chemistry practical and you have to clean up some dirty pieces of apparatus. Some are test-tubes that have residues form the thermal decomposition of metallic salts: iron oxide from heating iron sulphate, lead oxide from heating lead nitrate, etc. Some are evaporating dishes that are covered with soot on the outside. One is a beaker that has become etched by the sodium hydroxide – aluminium powder reactions to test for a nitrate. Suggest how you would clean these apparati. **(6 marks)**

iii) In countries or municipalities where waste disposal regulations are strictly adhered to, there are protocols for the disposal of chemical waste from the laboratory. What are the **FOUR** categories of chemical waste and one important non-chemical waste? **(5 marks)**

c) Answer these questions about heating methods:

i) What is the advantage of an oil bath over a hot water bath? **(1 mark)**

ii) What precautions must be taken with an oil bath? **(3 marks)**

iii) A flask is boiling too vigorously on a heating mantle. What is the simplest way to reduce the boiling rate? **(1 mark)**

iv) You are doing a microscale refluxing operation by heating a test – tube in a sand bath. What is the simplest way to reduce the rate of heating without reducing the setting on the heater control? **(1 mark)**

d) i) Give the reason for the precaution to be taken with an aspirator when you arrive at the end of a procedure, e.g. vacuum filtration on a Buchner funnel, or use of rotary evaporator. **(2 marks)**

ii) What is the most important precaution to take when determining the melting point of a substance? **(1 mark)**

iii) Outline the procedure to provide and maintain an inert atmosphere in a reaction flask. **(2 marks)**

Q2. Sodium sulphate can have varying amounts of water of crystallization due to the length of time and conditions under which it has been stored. So its sulphate content is uncertain. A sample containing sodium sulphate is dried, weighed and dissolved in dilute HCl, Barium Chloride solution is added in excess to precipitate barium sulphate, and the precipitate is digested in the hot solution. The precipitate is filtered through a paper filter which is then ignited and completely ashed. From the weight of the sample and weight of the precipitate, the percentage of sulphate in the sample is calculated. The precipitation reaction is the following:

$$BaCl_2(aq) + Na_2SO4(aq) \rightarrow BaSO_4(s) + 2NaCl(aq)$$

Three samples weighing in the vicinity of 0.35g but known to a precision of ± 0.0001 g are transferred to three 400mL beakers. The method of weighing by difference is used.

a) i) When the precipitation has occurred what is the major unwanted impurity in the mixture? (**1 mark)**

ii) Why are **THREE** samples used? **(1 mark)**

iii) If the samples of sodium sulphate are pure, what mass of barium sulphate precipitate can be expected if none is lost during the procedure? (Data: Relative atomic masses Na=23.0, Ba = 137.3, S = 32.1, O = 16.0). **(2 marks)**

b) i) What are the THREE important steps that must be taken to prepare the weighing bottles before they are ready to receive the sulphate samples? **(3 marks)**

ii) Describe briefly the method of "weighing by difference" by which the samples are transferred from the weighing bottles to the beakers. **(4 marks)**

c) Each sample is dissolved in about 200mL of distilled water and 5 mL of 6M HCl. It is heated to 90^0C and kept at that temperature on a hot plate. 5% barium chloride solution is added drop wise from burette which is mounted above the beaker. After 15-20 mL have been added, the process is interrupted to allow the precipitate to settle, and the completeness of precipitation is tested for by adding a few more drops of barium chloride. When the precipitation is

complete the beaker is covered with a watchglass and left to stand on the hot plate for 1 hour at 90^0C.

i) Why is the mixture never allowed to boil? **(1 mark)**

ii) How could you tell that the precipitation is not complete? **(1 mark)**

iii) Why is the mixture kept at 90^0C for 1 hour after precipitation? **(1 mark)**

d) The filtration s carried out using glass or plastic funnels fitted with ashless filter paper. It is convenient to filter the barium sulphate from a hot solution. Care must be exercised not to lose any precipitate while transferring the filtrate (liquid) and precipitate (solid) to your filter paper. After all the precipitate has been transferred wash the material in the funnel with three 5 mL portions of hot distilled water. Collect each washing separately in a small, clean beaker and then add two drops of $AgNO_3$ solution.

i) What is the purpose of ashless filter paper? **(1 mark)**

ii) Describe the steps taken to transfer ALL the precipitate from the beaker into the filter paper. **(3 marks)**

iii) Why is the material in the funnel washed with hot distilled water and why are drops of $AgNO_3$ added to the washings? **(2 marks)**

Q3. a) Below is a phase diagram of the typical vapour – liquid relation for a two component system (A + B).

i) Explain the significance of a horizontal line connecting a point on the lower curve with a point on the upper curve (such as line xy). **(2 marks)**

ii) Determine the boiling point of a liquid having a molar composition of 50% A and 50% B, and the molar composition of the vapour in equilibrium with it. **(2 marks)**

iii) A sample of vapour has the composition 50% A and 50% B. What is the composition of the boiling liquid that produced this vapour? **(1 mark)**

iv) If liquid of composition 10% A and 90B were to be distilled by fractional distillation, how many theoretical plates would be required to obtain a distillate of 90%A and 10%B? **(1 mark)**

b) i) There are three methods of distillation: simple, fractional and vacuum. What are the criteria for choosing simple distillation in preference to the others? **(2 marks)**

ii) Give a certain volume of liquid to be distilled, what volume of distilling flask should be used? **(1 mark)**

iii) What should be the direction of flow of water in the condenser and why? **(2 marks)**

iv) Why are the first drops of condensate which come over usually discarded? **(1 mark)**

v) Make a sketch showing the correct position of the thermometer bulb in the distillation head. **(1 mark)**

vi) What is a good flow rate of condensate in drops per minute? **(1 mark)**

vii) The material left behind in the distilling flask may become increasingly dark in colour during distillation. Why is this? **(1 mark)**

c) 50mL of a mixture of methanol and 1 – butanol was distilled. The B.P of 1 – butanol is 117.2^0C. the temperature of the thermometer was noted for every mL of distillate collected with the following results:

i) Estimate the boiling point of methanol from the graph. **(1 mark)**

ii) What could be the cause of the continuous rise in temperature as the methanol is collected? **(1 mark)**

iii) Estimate the volume of methanol in the mixture. **(1 mark)**

iv) Why does the temperature drop rapidly before 15mL? **(2 marks)**

Q4. a) i) Thin layer chromatography (TLC) has several uses. Give **FOUR** of them. **(4 marks)**

ii) Give a brief explanation of how TLC works. **(3 marks)**

b) State briefly the main steps and precautions that should be taken in:

i) The setting up of the development tank. **(2 marks)**
ii) The spotting of the solution on the TLC plate. **(3 marks)**
iii) The development of the chromatogram. **(3 marks)**

c) i) Arrange the following four solvents in order of increasing R_f value on a TLC plate: acetaldehyde, acetic acid, 1-butanol, decane. **(3 marks)**

ii) A student spots an unknown sample on a TLC plate and develops it in hexane solvent. Only one sport, for which R_f value is 0.05, is observed.
I) Is the unknown material a pure compound? **(1 mark)**
II) What can be done to verity the purity of the sample using thin-layer chromatography? **(1 mark)**

Q5. a) i) What are the main advantages of volumetric analysis over gravimetric analysis? **(1 mark)**

ii) Give **TWO** requirements for a reaction to be useful in volumetric analysis. **(2 marks)**

iii) Give **THREE** requirements for a substance to be good PRIMARY STANDARD. **(3 marks)**

b) i) State TWO mishaps in the use of the burette and the steps you would take to fix them. **(2 marks)**

ii) Describe in detail how you would prepare 250mL of 0.100M sodium carbonate solution as a primary standard. (RFM = 106). Give the **FIVE** essential steps and the mass of substance needed. **(5 marks)**

iii) You have just completed the first (rough) titration. Describe in detail how you would carry out the next (first accurate) titration. **(4 marks)**

c) i) Why is sodium hydroxide not a good primary standard? **(1 mark)**

ii) 29.5mL of 0.200 hydrochloric acid was titrated against 25.0mL of sodium hydroxide solution of unknown concentration using pheonolthalein indicator. Calculate the molar concentration of the alkali. **(2 marks)**

END