2.3.2

ICT ENABLED TEACHING SUPPORTIVE DOCUMENT

LAB MANUAL

DEPT OF CHEMISTRY (UG)

PRACTICAL MANUAL

B.Sc - II Semester

2019-20

Name:...

Seat No:..

Department of Chemistry
SDM College
Ujire

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OBSERVATIONS AND CALCULATIONS

Laboratory temperature $= \quad$ °C

Density of water at laboratory temperature $=$ gcm^{-3}

Mass of empty beaker $= W_1 =$ g

Mass of beaker + water $= W_2 =$ g

Mass of water $= (W_2 - W_1) g$

$=$

$= \quad$ g

Volume of water pipette out to the beaker $= \dfrac{(W_2 - W_1)}{Density}$ cm^3

$=$

$= \quad cm^3$

Calibration of pipette

Aim: To calibrate the pipette

Procedure:

The pipette provided is cleaned using chromic acid. This is done by keeping the pipette in chromic acid for about an hour. The pipette is then thoroughly washed with water and rinsed with distilled water.

Then the pipette is filled with distilled water which is at room temperature. Water is run out until the lower meniscus is exactly on the mark. The drop adhering to the tip of the pipette is removed by the surface of water contained in a beaker in contact with the tip.

The pipette is then allowed to discharge into a clean and dry beaker which is previously weighed .The tip of the pipette should be in contact with the side of the beaker. The pipette is allowed to drain for 15 seconds after the outflow has stopped. Now the pipette is removed from the beaker. Care is taken that no drop of water is adhering to the outside of the pipette.

The beaker containing the water collected from the pipette is weighed again. The room temperature is noted.

Result:

The calibrated volume of the pipette = cm^3

OBSERVATIONS AND CALCULATIONS

(a) Preparation of standard sodium carbonate solution

Mass of weighing bottle + sodium carbonate = w_1 =   g

Mass of weighing bottle after transferring = w_2 =   g

Mass of anhydrous sodium carbonate transferred = $(w_1 - w_2)$ g

$$= $$

$$= \qquad g$$

$$\text{Strength of sodium carbonate solution} = \frac{(w_1 - w_2) \times 4}{53}$$

$$=$$

$$= \qquad N$$

(b) Standardization of hydrochloric acid solution

Titration of $25 cm^3$ of standard sodium carbonate solution against hydrochloric acid solution

Indicator : Methyl orange

End point : Yellow to orange

Trial no. Burette reading	I	II	III.
Final reading			
Initial reading			
Volume of HCl added in cm^3			

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PRACTICAL MANUAL

B.Sc - IV Semester

2019-20

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Observations and Tabulation:

Time in min	Burette Readings		Volume of NaOH in cm^3	$k = \dfrac{2.303}{t} \log \dfrac{V\infty - V0}{V\infty - Vt} \, min^{-1}$	
	Final	Initial			
0					
10					
20					
30					
40					
∞					

Mean value of k = ... min^{-1}

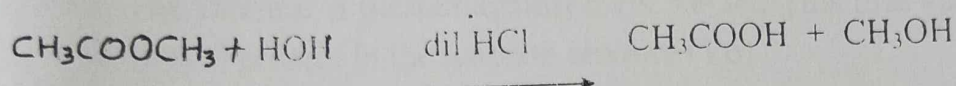Hence, the rate constant of the reaction is = ... min^{-1}

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DETERMINATION OF VELOCITY CONSTANT OF ACID
HYDROLYSIS OF METHYL ACETATE

Aim: To determine the rate constant of acid hydrolysis of methyl acetate

Principle: Methyl acetate is an ester. It is readily hydrolysed by dilute hydrochloric acid into methyl alcohol and acetic acid.

$$CH_3COOCH_3 + HOH \xrightarrow{\text{dil HCl}} CH_3COOH + CH_3OH$$

Two molecules of reactants are involved in the reaction. Since the quantity of ester is large, its concentration practically remains constant. Hence, the above reaction is of pseudo first order. Its rate constant can be calculated using the expression.

$$k = \frac{2.303}{t} \log \frac{a}{a-x}$$

where a is the initial concentration and x is the decrease in concentration of the ester in a time t. The progress of the reaction is followed by titrating a known volume of the solution against standard sodium hydroxide solution at regular intervals of time. Let V_0, V_t and V_α represent the titre values in the beginning, after a time t and at the end of the reaction respectively.

$V_t - V_0$ α Amount of acetic acid formed in a time t α (x)

$V_\alpha - V_0$ α Amount of acetic acid formed at the end (It is the same as the amount of ester taken in the beginning) α (a)

$(V_\alpha - V_0) - V_t - V_0$ α a $-$ x

$V_\alpha - V_t$) α a - x

The above first order equation becomes,

$$k = \frac{2.303}{t} \log \frac{V\infty - V0}{V\infty - Vt} \quad \text{min}^{-1}$$

Chemicals Required:
100 cm³ of 0.5 N HCl , 15 cm³ of methyl acetate and 0.1 N NaOH

Procedure: 100 cm^3 of 0.5N HCl is taken in a dry 250 cm^3 stopppered bottle. This bottle is kept in a water bath which acts as a thermostat. 15cm^3 of methyl acetate taken in another stoppered bottle is also kept in the water bath

A burette is filled with 0.1N NaOH. When the solution in the bottles have attained constant temperature, 5cm^3 of the ester is pipetted out into the bottle containing the hydrochloric acid and shaken well.

5cm^3 of the reaction mixture is immediately pipetted out into a conical flask containing ice cold water (to arrest further reaction) and a drop of phenolphthalein is added. When the pipette is half empty a stop clock is started. The solution is titrated against 0.1N NaOH. This titre value gives the amount of HCl present in the reaction mixture (Vo).

The titrations are repeated with the same volume of the mixture after 10, 20 30 and 40 minutes, in the same manner to get V_t.

After the first titration, a portion of (about 25 cm^3) the reaction mixture is taken in a stoppered bottle and kept in a water bath at 60^0 C for 1 hour to complete the reaction. It is cooled to the original temperature. 5cm^3 of the solution is titrated against the same NaOH and cooled to the original temperature. 5 cm^3 of the solution is titrated against the same NaOH solution to get V∞

Result:

The value of k is found to be a constant. Therefore the reaction is of first order.

The velocity constant of acid hydrolysis of methyl acetate =...........min^{-1}

Observations and Tabulation

(a) Hydrolysis of methyl acetate using 0.5N HCl

Time in min	Burette Readings		Volume of NaOH in cm^3	$k_1 = \dfrac{2.303}{t} \log \dfrac{V\infty - V0}{V\infty - Vt}$ min^{-1}
	Final	Initial		
0				
10				
20				
30				
40				
∞				

Mean value of k_1 =................   min^{-1}

Hence, the rate constant of the reaction using 0.5N HCl is

=...................... min^{-1}

PRACTICAL MANUAL

B.Sc- VI Semester

2019-20

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Department of Chemistry

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Aniline + (acetic anhydride) → Acetanilide + CH₃COOH

Acetanilide from Aniline

Aim: To prepare acetanilide from aniline.

**Principle:** Aniline is acetylated with acetic anhydride to get acetanilide.

Procedure: $2.5cm^3$ Aniline is taken in a clean and dry 100ml conical flask. Add about 8ml of pure acetic anhydride into it in a bulk with stirring.

The contents of the flask were swirled for about 15-20 min. The contents of the flask or the semi liquid of reaction mixture is poured into about 150ml of the ice water taken in a 400 ml beaker with constant stirring. The crude sample of acetanilide is filtered and washed with ice cold water until the washings are free from acetic acid. The crude product is dried and weighed.

A portion of the sample is re crystallized from hot water. The sample is dried between the folds of the filter paper and melting point is determined.

Result: Yield of the product = ...g

   Melting point   =......... ^0C
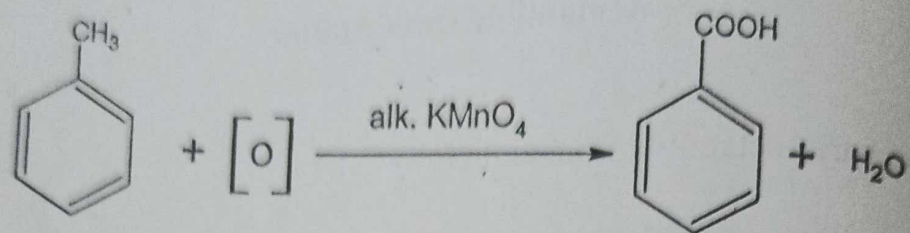
Toluene   Benzoic acid

Expt.No:
Date:

Benzoic acid from toluene

Aim: To prepare benzoic acid from toluene

Principle: Toluene is refluxed with alkaline potassium permanganate solution and the sodium salt obtained is acidified with dilute hydrochloric acid to get benzoic acid.

Procedure: $2.5cm^3$ of toluene is taken in a round bottomed flask fitted with water cooled reflux condenser. 5cm3 of alkaline potassium permanganate (1g of sodium carbonate + 2g of potassium permanganate is dissolved in 50cc of water) is added to the flask. $5cm^3$ of above solution is added to the round bottomed flask at definite intervals of time, till the $50cm^3$ of solution is exhausted.

During this time, till the contents of round bottomed flask is refluxed. When the addition of alkaline potassium permanganate is completed, it is found that the oily layer in the flask is disappeared. Now the flask is cooled and the reflux condenser is disconnected. The contents of the flask are filtered to remove the brown manganous dioxide. The filtrate is now acidified by adding $5cm^3$ of conc. Hydrochloric acid. A little of sodium sulphite is added with stirring (if any brown colour is found in the solution), until the colour completely disappears. Now the white crystals of benzoic acid are separated from the solution by filtration. It is then washed with cold water and dried between the folds of filter paper. Yield of the crude product is determined.

A portion of crude product is recrystallised from hot water. The recrystallised sample is dried and the melting point is determined.

Result: Yield of the product = ...g

Melting point   = ...^{0}C

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NO_2 (Nitrobenzene) $+ HO-NO_2 \xrightarrow{Conc. H_2SO_4}$ m-dinitro benzene $+ H_2O$