PRACTICAL PROBLEMS

PROBLEM 1 (Practical)

Preparation of 2-Iodobenzoic Acid

This laboratory task involves preparation of 2-iodobenzoic acid from 2-aminobenzoic acid. The procedure consists of diazotization of 2-aminobenzoic acid followed by reaction with KI (in H_2SO_4).

Procedure

1) Quantitatively transfer the given sample of solid 2-aminobenzoic acid into a 100 cm^3 beaker placed in the ice-bath. Add 7.2 cm^3 of H_2SO_4 (2.6 M) (labelled H_2SO_4) and mix the contents thoroughly for 1 minute with the help of a glass rod. Cool the solution for 5 minutes.
2) Using a measuring cylinder, measure out 4.4 cm^3 of supplied cooled $NaNO_2$ solution from the vial placed in the ice-bath.
3) With the help of a dropper, slowly add the cooled $NaNO_2$ solution to the acid solution with constant gentle stirring using a glass rod to obtain an almost clear solution (3 – 5 minutes).
4) Remove the beaker from the ice bath and then slowly add 9.4 cm^3 of KI solution from the stoppered tube, with stirring.
5) Get hot water from the laboratory expert. Keep the beaker in hot water for 5 minutes.
6) Filter the crude product and wash it thoroughly with distilled water (10 cm^3). Collect the washings along with the main filtrate.
7) Neutralize the combined filtrate by gradually adding the given solid Na_2CO_3 until effervescence ceases. Dispose of the filtrate in the appropriate plastic bucket.

Purification of the crude product

Place the funnel containing the precipitate on a 100 cm^3 conical flask. Pour about 15 to 20 cm^3 of the supplied $NaHCO_3$ solution (using test tube) over the filter paper so as to dissolve the precipitate completely.

8) Add the supplied charcoal powder to the filtrate and mix it thoroughly. Filter the solution to remove charcoal.

9) Add dilute H_2SO_4 gradually to the filtrate till effervescence ceases. Filter the purified product. Use 10 -15 cm^3 distilled water to wash the precipitate. Keep the filter paper with the product on a watch glass.

10) Cover the product with the same funnel and hand over the product to the laboratory expert for drying (for a minimum of one hour).

Towards the end of the practical session have the product weighed by the laboratory expert and record the same.

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SOLUTION

The following values were required to be written on the Answer Sheet :

- Mass of the product.
- The calculated theoretical yield (based on 2 aminobenzoic acid) in g.
- The yield obtained as a percentage of the theoretical yield.
- Colour of the product obtained.

Tasks:

Write down the balanced chemical equations for:

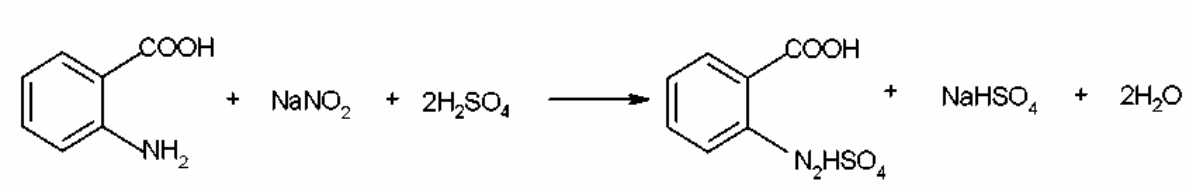
(a) diazotization of 2-aminobenzoic acid using $NaNO_2$ and H_2SO_4.

(b) the reaction of K**I** with the diazotized product.

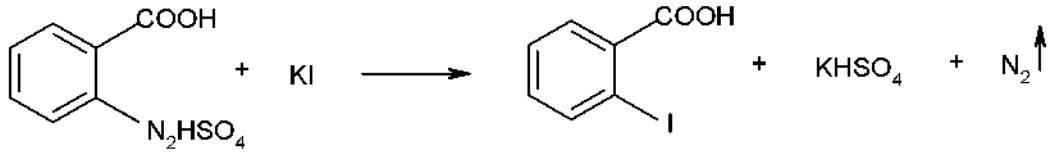
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Solutions of the tasks:

a)



b)



PROBLEM 2 (Practical)

Estimation of Mn(II) and Mg(II) Present in the Given Sample

In this experiment, estimation of the amounts of Mn (II) and Mg (II) present in the given sample is carried out by complexometric titration using standard Na_2EDTA solution. Total metal ion content is obtained from the first titration. At this stage, by using adequate solid NaF, selective and quantitative release of EDTA present in Mg-EDTA complex is achieved. The EDTA thus released is bound again by the addition of a known excess of standard Mn (II) solution. The unused Mn (II) is estimated by a back titration using the same standard Na_2EDTA solution. From these two different titre values, individual amounts of metal ions present can be obtained. Both the titrations are performed using a buffer (*pH* = 10) and Erichrome black T indicator.

The sample in duplicate is given in two 250 cm^3 conical flasks (labelled as Trial I and Trial II). Perform the titrations for both and record your readings on the answer sheet.

Procedure

Two burettes (25 cm^3) are supplied to you. Fill one with the given standard Na_2EDTA solution and the other with the given standard Mn (II) solution.

Titration 1

To the sample solution (supplied in the 250 cm^3 conical flask), add all of the solid hydroxylamine hydrochloride given in one vial followed by 50 cm^3 of distilled water. With the help of a measuring cylinder, add 10 cm^3 buffer solution (*pH* = 10) and one metal spatula full of the solid indicator. Shake the contents of the flask thoroughly and titrate the solution against the standard Na_2EDTA solution until the colour changes from wine red to blue. Record your burette reading (**A** cm^3). Ensure that you shake the contents of the flask thoroughly throughout the titration.

Titration 2

To the same flask, add all of the solid NaF given in one vial and shake the contents well for a minute. To this add 20 cm^3 of the given standard Mn (II) solution from the other burette. The addition of the Mn (II) solution should be done in small increments (2 – 3 cm^3) with thorough shaking. After addition of the total Mn (II) solution, shake the contents for two to three minutes. The colour of the solution will change from blue to wine red. Titrate

the excess of Mn (II) in the solution against the standard Na_2EDTA solution till the colour changes from wine red to blue. Record your burette reading (**B** cm^3).

Repeat the same procedure for Trial II.

SOLUTION

The results of the titration (volumes) were required to be written on the Answer Sheet.

The other tasks to be solved:

2.2 Give the balanced chemical equation for the reactions of Mg(ll) and Mn(ll) with Na_2EDTA . (Use the symbol Na_2HY for Na_2EDTA.)

Answer: $Mg^{2+} + H_2Y^{2-} \rightarrow MgY^{2-} + 2\ H^+$

$Mn^{2+} + H_2Y^{2-} \rightarrow MnY^{2-} + 2\ H^+$

2.3 Give the equation for the release of EDTA by the addition of NaF to the MgEDTA complex.

Answer: $MgY^{2-} + 2\ F^- \rightarrow MgF_2 + Y^{4-}$

2.4 Calculate the amount of Mg (II) and Mn (II) in gram for any one of the two trials. (Show the main steps in your calculation.)

2.5 The colour change at the end point (wine red to blue) in Titration 1 is due to

a) the formation of metal-indicator complex, ❑

b) the release of free indicator from metal-indicator complex, ❑

c) the formation of metal-EDTA complex. ❑

[Mark X in the correct box.]

Correct answer is (b).

PROBLEM 3 (Practical)

Determination of the Rate Constant for the Redox Reaction between Ethanol and Chromium (VI)

The oxidation of alcohols by chromium (VI) forms the basis for analysis of breath samples for measuring alcohol content. A dilute solution of $K_2Cr_2O_7$ in the presence of a strong acid (3.6 M HCI here) is a source of $HCrO_4^-$ which is the oxidant involved in the reaction.

In this experiment, the rate of the reaction between $HCrO_4^-$ and CH_3CH_2OH is determined titrimetrically. Under the given experimental conditions, the rate law reduces to

$$\text{rate} = k[HCrO_4^-]^x$$

where **x** is the order of the reaction.

At any given time, $[HCrO_4^-]$ is obtained by iodometric titration.

Procedure

You are given 100 cm^3 of standard $K_2Cr_2O_7$ solution in HCI in a bottle. Transfer all the absolute ethanol given in a vial into this bottle and stopper it. Mix the contents thoroughly, start the stopwatch immediately and regard this as time t = 0. Fill the burette with this solution.

After every 10 minutes, start to draw 10 cm^3 of this solution to a clean conical flask containing 4 cm^3 of the given KI solution. The solution will turn brown. Titrate this solution with the given standard $Na_2S_2O_3$ solution until the colour changes to pale greenish yellow. Add 2 cm^3 of starch indicator and continue the titration until the colour changes from blue to pale green. Record the burette reading in the answer sheet. Repeat this procedure at 10 minutes intervals to obtain four readings.

SOLUTION

The results of the titration (volumes) were required to be written on the Answer Sheet.

The other tasks to be solved:

3.1 Write down the possible oxidation products in the reaction of $HCrO_4^-$ and CH_3CH_2OH.

Solution: Acetic acid, CH_3COOH

Acetaldehyde, CH_3CHO

3.2 Write the chemical equation for the reaction between $HCrO_4^-$ and KI:

Solution: $2\ HCrO_4^- + 6\ I^- + 14\ H^+ \rightarrow 3\ I_2 + 2\ Cr^{3+} + 8\ H_2O$

3.3 Write down the balanced chemical equation involved in the titration.

Solution: $I_2 + 2\ S_2O_3^{2-} \rightarrow 2\ I^- + S_4O_6^{2-}$

3.4 Give the main steps for the calculation of $HCrO_4^-$ concentration (mol dm^{-3}) for any one titration reading.

3.5 Give **c**oncentrations of $HCrO_4^-$ at different times (in a table attached).

3.6 Plot the graph of log [$HCrO_4^-$] *vs.* time.

3.7 From the nature of the graph determine the order (**x**) of the reaction with respect to $HCrO_4^-$

3.8 Determine the rate constant for the reaction.