Estimation of Acetone by Iodometry

Aim

To determine the amount of acetone present in given unknown acetone solution using iodometry method.

Theory

Acetone reacts with iodine in the presence of sodium hydroxide solution to yield iodoform and sodium acetate.

$$CH_3COCH_3 + 3\ I_2 + 4\ NaOH \rightarrow CHI_3 + CH_3COONa + 3\ NaI + 3\ H_2O$$

Elementary Reactions Involved

$$3\ I_2 + 6\ NaOH \rightarrow 3\ NaOI + 3\ NaI + 3\ H_2O$$

$$3\ NaOI + CH_3COCH_3 \rightarrow CHI_3 + CH_3COONa + 2\ NaOH$$

A dilute aqueous solution of the sample is added to a known volume of 1N sodium hydroxide solution, followed by an excess of standard 0.1N iodine solution. After acidification, unreacted iodine is determined by titration with standard 0.1N sodium thiosulphate solution. This procedure is termed **Messinger's method**. Aldehydes, compounds which contain an acetal group, or a group oxidisable by hypoiodite to an acetal group, interfere; compounds containing a –CH=CH-C=O group will consume iodine and therefore interfere. Methyl and ethyl alcohols should also be absent.

When the iodoform reaction is complete, the solution is acidified with sulfuric acid, and the liberated iodine is titrated with standard sodium thiosulphate solution. This is the excess of iodine not utilised in the oxidation.

$$NaOI + NaI + H_2SO_4 \rightarrow NaSO_4 + I_2 + 2\ H_2O$$

Apparatus and chemicals required

1. Beaker(100 mL and 500 mL), standard volumetric flask(100 mL, 250 mL and 500 mL), funnel, Erlenmeyer's flask(250 mL), burette(50 mL), burette stand, pipette(10 mL and 20 mL), porcelain tile and measuring cylinder(10 mL).
2. $K_2Cr_2O_7$, H_2SO_4, $Na_2S_2O_3$, KI, acetone solution and starch.

Procedure

Preparation of std **0.1 N $K_2Cr_2O_7$** solution

- Weighed accurately 0.4903 g of $K_2Cr_2O_7$ and transferred into 100 mL volumetric flask through funnel.
- Added minimal amount of water into the flask through funnel to dissolve the solid $K_2Cr_2O_7$.
- The solution was made up to the mark and mixed well to get homogeneous std 0.1 N $K_2Cr_2O_7$ solution.

Preparation of **~0.1 N I_2** solution

- Weighed accurately 10 g of KI and transferred into 250 mL volumetric flask through funnel.
- Added 50 mL of water into the flask through funnel to dissolve KI completely.
- Weighed 3.175 g of powdered I_2 and transferred into the same volumetric flask using funnel.
- Added minimal amount of water into the flask through funnel to dissolve the solid I_2.

- The solution was made up to the mark and mixed well to get homogeneous ~0.1N I_2 solution.

Standardisatoin of **0.1 N $Na_2S_2O_3$** solution using std **0.1 N $K_2Cr_2O_7$** solution

- Rinsed the burette with distilled water and $Na_2S_2O_3$ solution respectively.
- Filled the burette with $Na_2S_2O_3$ solution and made up to zero mark without air bubbles.
- Pipetted out 20 mL of $K_2Cr_2O_7$(0.1N) solution in to 250 mL clean conical flask and added 10 mL of 4N H_2SO_4 into the flask.
- Added 10 mL of 10% KI solution to the conical flask. The solution colour became reddish brown because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the change of colour was green. Noted the burette reading.
- Repeated the titration to get concordant titre value.

Standardisation of **~0.1N I_2** solution using standardised **0.1 N $Na_2S_2O_3$** solution

- Rinsed the burette(another) with distilled water and iodine solution respectively.
- Filled the burette with iodine solution and made up to zero mark without air bubbles.
- Transferred 20 mL of I_2(~0.1N) solution in to 250 mL clean Iodine flask and added 10 mL of 1N NaOH solution into it. The solution became pale yellow in colour.
- The flask was kept in dark for 10 minutes.
- Added 10 mL of 1N H_2SO_4 into the flask and the solution became reddish brown, because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the solution became colourless. Noted the burette reading.
- Repeated the titration to get concordant titre value.

Estimation of acetone with standardised **0.1 N $Na_2S_2O_3$** solution

- Pipetted out 10 mL of acetone solution into 250 mL clean Iodine flask.
- Transferred 50 mL of I_2(~0.1N) solution into the Iodine flask and added 25 mL of 1N NaOH solution. Pale yellow was appeared.
- The flask was kept in dark for 10 minutes with occasional mixing.
- Added 25 mL of 1N H_2SO_4 into the flask and the solution became reddish brown, because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the solution became colourless. Noted the burette reading.

Calculation

Preparation of std **0.1 N $K_2Cr_2O_7$** solution

Weight of $K_2Cr_2O_7$ taken in 250 mL std volumetric flask, W_1 = --------- g

Strength of $K_2Cr_2O_7$ solution, N_1 = (W_1/Equivalent weight) X (1000/250)

= _____ N

Standardisatoin of **0.1 N $Na_2S_2O_3$** solution using std **0.1 N $K_2Cr_2O_7$** solution

Table 1

S. No.	Volume of $K_2Cr_2O_7$ solution pipetted out (mL) V_1	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL)	Concordant value (mL) V_2
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

Strength of $K_2Cr_2O_7$ solution, N_1 =_____ N

Volume of $K_2Cr_2O_7$ solution taken, V_1 = 20 mL

Strength of $Na_2S_2O_3$ solution, N_2 = N_2

Volume of $Na_2S_2O_3$ solution consumed, V_2 =_____ mL

$$N_1V_1 = N_2V_2$$

Strength of $Na_2S_2O_3$ solution, N_2 = (N_1V_1)/V_2

= _____ N

Standardisation of **~0.1N I_2** solution using standardised **0.1 N $Na_2S_2O_3$** solution

Table 2

S. No.	Volume of I_2 solution taken in Iodine flask (mL) V_3	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL)	Concordant value (mL) V_4
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

Strength of $Na_2S_2O_3$ solution, N_2 = N_2 N

Volume of $Na_2S_2O_3$ solution consumed, V_3 =_____ mL

Strength of I_2 solution, N_4 =_____ N

Volume of I_2 solution taken, V_4 =_____ mL

$$N_2V_3 = N_4V_4$$

Strength of I_2 solution, N_4 = (N_2V_3)/V_4

= _____ N

Estimation of acetone with standardised **0.1 N $Na_2S_2O_3$** solution

Table 3

S. No.	Volume of acetone solution pipetted out (mL)	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL) V_5
		Initial	Final	
1	10	0		
2	10	0		

Initially, volume of iodine solution transferred to iodine flask, V_6 = 50 mL

Volume of $Na_2S_2O_3$ solution consumed (mL), V_5 = _____ mL
Strength of $Na_2S_2O_3$ solution, N_2 = N_2 N
Strength of I_2 solution, N_4 = N_4 N
Volume of I_2 solution unreacted, V_7 =_____ mL

$$N_4V_7 = N_2V_5$$

Volume of I_2 solution unreacted, $V_7 = (N_3V_6)/N_4$

Volume of I_2 solution reacted, $V_8 = (V_6-V_7)$ mL

1 mL of 0.1 N Iodine solution ≡ 0.968 mg of acetone

V_8 mL of N_4 N iodine solution = $(V_8\ N_4)/0.1$ * mg of acetone

Result

The amount of acetone present in given 10 mL of acetone solution is _____ mg.

Estimation of formaldehyde by Iodometry

Aim

To determine the amount of formaldehyde present in given unknown acetone solution using iodometry method.

Theory

Formaldehyde is oxidised quantitatively to formic acid by excess of iodine in alkaline solution. The effective oxidising agent is probably sodium hypoiodite, and the formic acid formed is neutralised by the alkali present.

$$HCHO + I_2 + 3\ NaOH \rightarrow HCOONa + 2\ NaI + 2\ H_2O$$

Elementary Reactions Involved

$$I_2 + 2\ NaOH \rightarrow NaOI + NaI + H_2O$$

$$NaOI + HCHO + NaOH \rightarrow HCOONa + NaI + H_2O$$

When the oxidation is complete, the solution is acidified with hydrochloric acid, and the liberated iodine is titrated with standard sodium thiosulphate solution. This is the excess of iodine not utilised in the oxidation.

$$NaOI + NaI + 2\ HCl \rightarrow 2\ NaCl + I_2 + 2\ H_2O$$

Apparatus and chemicals required

1. Beaker(100 mL and 500 mL), standard volumetric flask(100 mL, 250 mL and 500 mL), funnel, Erlenmeyer's flask(250 mL), burette(50 mL), burette stand, pipette(10 mL and 20 mL), porcelain tile, glass rod, dropper and measuring cylinder(10 mL).
2. $K_2Cr_2O_7$, H_2SO_4, HCl, $Na_2S_2O_3$, KI, formaldehyde solution and starch.

Procedure

Preparation of std **0.1 N $K_2Cr_2O_7$** solution

- Weighed accurately 0.4903 g of $K_2Cr_2O_7$ and transferred into 100 mL volumetric flask through funnel.
- Added minimal amount of water into the flask through funnel to dissolve the solid $K_2Cr_2O_7$.
- The solution was made up to the mark and mixed well to get homogeneous std 0.1 N $K_2Cr_2O_7$ solution.

Preparation of **~0.1 N I_2** solution

- Weighed accurately 10 g of KI and transferred into 250 mL volumetric flask through funnel.
- Added 50 mL of water into the flask through funnel to dissolve KI completely.
- Weighed 3.175 g of powdered I_2 and transferred into the same volumetric flask using funnel.
- Added minimal amount of water into the flask through funnel to dissolve the solid I_2.
- The solution was made up to the mark and mixed well to get homogeneous ~0.1N I_2 solution.

Standardisatoin of **0.1 N $Na_2S_2O_3$** solution using std **0.1 N $K_2Cr_2O_7$** solution

- Rinsed the burette with distilled water and $Na_2S_2O_3$ solution respectively.
- Filled the burette with $Na_2S_2O_3$ solution and made up to zero mark without air bubbles.

- Pipetted out 20 mL of $K_2Cr_2O_7$(0.1N) solution in to 250 mL clean conical flask and added 10 mL of 4N H_2SO_4 into the flask.
- Added 10 mL of 10% KI solution to the conical flask. The solution colour became reddish brown because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the change of colour was green. Noted the burette reading.
- Repeated the titration to get concordant titre value.

Standardisation of **~0.1N I_2** solution using standardised **0.1 N $Na_2S_2O_3$** solution

- Rinsed the burette(another) with distilled water and iodine solution respectively.
- Filled the burette with iodine solution and made up to zero mark without air bubbles.
- Transferred 20 mL of I_2(~0.1N) solution in to 250 mL clean Iodine flask and added 10 mL of 1N NaOH solution into it. The solution became pale yellow in colour.
- The flask was kept in dark for 10 minutes.
- Added 10 mL of 1N HCl into the flask and the solution became reddish brown, because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the solution became colourless. Noted the burette reading.
- Repeated the titration to get concordant titre value.

Estimation of acetone with standardised **0.1 N $Na_2S_2O_3$** solution

- Pipetted out 10 mL of formaldehyde solution into 250 mL clean Iodine flask.
- Transferred 50 mL of I_2(~0.1N) solution into the Iodine flask and added 25 mL of 1N NaOH solution. Pale yellow was appeared.
- The flask was kept in dark for 10 minutes with occasional mixing.
- Added 25 mL of 1N HCl into the flask and the solution became reddish brown, because of liberated iodine.
- Titrated the liberated iodine against $Na_2S_2O_3$ solution in burette.
- When the solution colour turned pale yellow, 5-10 drops of starch solution was added as indicator. Now the solution colour became blue.
- At the end point the solution became colourless. Noted the burette reading.

Calculation

Preparation of std **0.1 N $K_2Cr_2O_7$** solution

Weight of $K_2Cr_2O_7$ taken in 250 mL std volumetric flask, W_1 = --------- g

Strength of $K_2Cr_2O_7$ solution, N_1 = (W_1/Equivalent weight) X (1000/250)

= _____ N

Standardisatoin of **0.1 N $Na_2S_2O_3$** solution using std **0.1 N $K_2Cr_2O_7$** solution

Table 1

S. No.	Volume of $K_2Cr_2O_7$ solution pipetted out (mL) V_1	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL)	Concordant value (mL) V_2
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

Strength of $K_2Cr_2O_7$ solution, N_1 =_____ N
Volume of $K_2Cr_2O_7$ solution taken, V_1 = 20 mL
Strength of $Na_2S_2O_3$ solution, N_2 = N_2
Volume of $Na_2S_2O_3$ solution consumed, V_2 =_____ mL

$$N_1V_1 = N_2V_2$$

Strength of $Na_2S_2O_3$ solution, $N_2 = (N_1V_1)/V_2$

= _____ N

Standardisation of **~0.1N I_2** solution using standardised **0.1 N $Na_2S_2O_3$** solution

Table 2

S. No.	Volume of I_2 solution taken in Iodine flask (mL) V_3	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL)	Concordant value (mL) V_4
		Initial	Final		
1	20	0			
2	20	0			
3	20	0			

Strength of $Na_2S_2O_3$ solution, N_2 = N_2 N
Volume of $Na_2S_2O_3$ solution consumed, V_3 =_____ mL
Strength of I_2 solution, N_4 =_____ N
Volume of I_2 solution taken, V_4 =_____ mL

$$N_2V_3 = N_4V_4$$

Strength of I_2 solution, $N_4 = (N_2V_3)/V_4$

= _____ N

Estimation of formadehyde with standardised **0.1 N $Na_2S_2O_3$** solution

Table 3

S. No.	Volume of formaldehyde solution pipetted out (mL)	Burette reading (mL)		Volume of $Na_2S_2O_3$ solution consumed (mL) V_5
		Initial	Final	
1	10	0		
2	10	0		

Initially, volume of iodine solution transferred to iodine flask, V_6 = 50 mL

Volume of $Na_2S_2O_3$ solution consumed (mL), V_5 = _____ mL
Strength of $Na_2S_2O_3$ solution, $N_2 = N_2$ N
Strength of I_2 solution, $N_4 = N_4$ N
Volume of I_2 solution unreacted, V_7 =_____ mL

$$N_4V_7 = N_2V_5$$

Volume of I_2 solution unreacted, $V_7 = (N_3V_6)/N_4$

Volume of I_2 solution reacted, $V_8 = (V_6-V_7)$ mL

1 mL of 0.1 N Iodine solution ≡ 1.5013 mg of formaldehyde

V_8 mL of N_4 N iodine solution = $(V_8\ N_4)/0.1$ * mg of acetone

Result

The amount of acetone present in given 10 mL of formaldehyde solution is _____ mg.

Note

- Formaldehyde is usually encountered as an aqueous solution (" formalin ") containing about 33 to 37 per cent, by weight of formaldehyde.
- Stronger solutions polymerise spontaneously to paraformaldehyde.
- The above procedure is applicable only to dilute solutions of formaldehyde (concentration < 1 per cent.). Other aldehydes and most ketones must be absent.

Synthesis of Aspirin from Salicylic acid

Aim

To prepare aspirin from salicylic acid by acetylation.

Apparatus and chemical required

1. Beaker, flask, water bath, filter paper, measuring cylinder, glass rod, dropper and funnel.
2. Salicylic acid, acetic anhydride

Theory

Aspirin is the common name for the compound acetylsalicylic acid, widely used as a fever reducer and as a pain killer. To prepare aspirin, salicylic acid is reacted with an excess of acetic anhydride. A small amount of a strong acid is used as a catalyst which speeds up the reaction. In this experiment, phosphoric acid will be used as the catalyst. The excess acetic acid will be quenched with the addition of water. The aspirin product is not very soluble in water so the aspirin product will precipitate when water is added. Since acetic acid is very soluble in water, it is easily separated from the aspirin product. The aspirin isolated in this step is the "crude product". A "purified product" can be obtained through recrystallization of the crude product in hot ethanol. In this experiment, the crude product will be the desired product. The percent yield of the crude product will be determined for this reaction. The purity of the product will also be analyzed. The product will be analyzed by three different methods: melting point, titration, and spectroscopic assay.

Procedure

1. Weighed accurately 2 g of salicylic acid and transferred into a 250 mL conical flask.
2. Added 5.0 mL of distilled acetic anhydride and 5 drops of phosphoric acid (catalyst) into the flask.
3. Kept the flask in the hot water bath for 45 minutes.
4. Occasionally the flak was swirled for the mixing of reagents
5. Added 2 mL of distilled water to the flask when the reaction mixture is warm.
6. Removed the flask from the water bath and the reaction mixture was brought to room temperature.
7. Added 20 mL of chilled distilled water to the flask.
8. Crystals of aspirin separated out from the reaction mixture.
9. The mother liquor was decanted.
10. Isolated the solid by filtration.
11. Transferred the solid to watch glass and dried the crystal.
12. Weighed the amount of solid
13. Calculated the crude yield.
14. Recrystallized the crude aspirin in ethanol.

Calculation

The crude yield of aspirin was calculated as follows

$$\text{Percentage yield} = \frac{\text{Amount of aspirin obtained}}{\text{Theoritical yield of aspirin}} \times 100$$

Report

The percentage yield of crude aspirin _____.

Caffine Extraction

Aim

To extract the caffeine from tea powder using polar and non-polar solvents extraction technique.

Apparatus and chemicals required

1. Beaker, glass rod, conical flask, funnel, separating funnel, measuring cylinder and water bath.
2. Tea powder, dichloromethane, calcium carbonate and anhydrous sodium sulphate.

Theory

A method to separate compounds based on their relative solubilities in two different immiscible liquids, usually water and organic solvent. This is called solvent extraction. In this process selectively dissolve one or more of the mixture compounds into suitable solvents. The solution of dissolved compound is referred to as the extract. Here the organic solvent dicholoromethane is to extract caffeine from an aqueous solution extract of tea powder because caffeine is more soluble in dichloromethane than water. Tannins are slightly soluble in dichloromethane can be eliminated by adding calcium carbonate because tannins are phenolic compound of high molecular weight and being acidic in nature can be converted to salt by deprotonation.

Procedure

- Weighed accurately 10g of tea powder and transferred into a 500 mL beaker.
- Added 200mL of distilled water into it.
- Digested on Bunsen flame for about 15 mins.
- Filtered and added 5gm of $CaCO_3$ to the filterate.
- Digested on water bath for about 15 mins.
- Filtered and transferred the filterate to 500 mL separating funnel.
- Added 25mL of dichloromethane to the separating funnel and shaked well.

- Two layers appeared. (The lower layer was organic layer and the upper layer was aqueous layer) Collected the organic layer in a clean 100 mL dry beaker.
- Repeated the extraction process two more times.
- Washed the organic layer with distilled water
- Dried the organic layer with anhydrous sodium sulphate
- Evaporated the organic layer to dryness
- Pale yellow solid of caffeine was obtained

Calculation

The percentage of caffeine present in given tea powder was calculated as follows

$$\text{Percentage caffeine} = \frac{\text{Amount of caffeine isolated}}{\text{Amount of tea powder taken}} \times 100$$

Report

The percentage of caffeine present in given tea powder is _____.

Citric acid Isolation

Aim

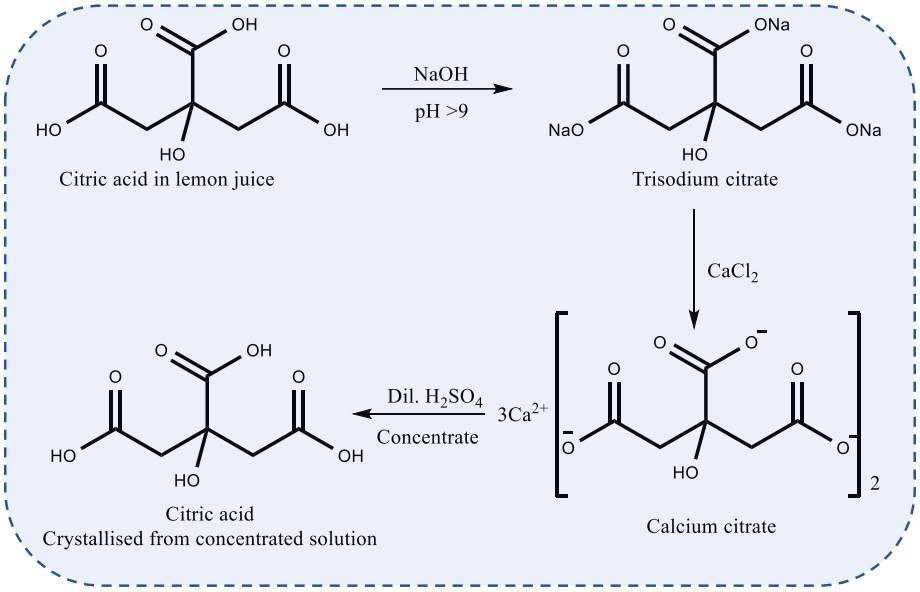
To isolate the citric acid from lemon juice using acid-base and precipitation reactions.

Apparatus and chemicals required

1. Beaker, glass rod, funnel, filter paper, measuring cylinder, buchner funnel, buchner flask, vacuum pump and water bath.
2. Lemon juice, sodium hydroxide, calcium chloride dihydrate, pH paper and sulphuric acid.

Theory

Citric acid exists in greater amounts in citrus fruits. Lemons and limes have particularly high concentrations of the acid. It can constitute as much as 8% of the dry weight of these fruits. In olden days, industrial-scale citric acid is produced by treating the citrus fruit juice with calcium hydroxide to precipitate calcium citrate. Calcium citrate was isolated and converted back to the citric acid using dilute sulfuric acid.



Citric acid can exist either in an anhydrous form or as a monohydrate. The anhydrous form crystallizes from hot water, while the monohydrate forms when citric acid is crystallized from cold water. The monohydrate can be converted to the anhydrous form at about 78 °C. It decomposes with loss of carbon dioxide above about 175 °C.

Citric acid is normally considered to be a tribasic acid, with pK_a values, extrapolated to zero ionic strength, of 2.92, 4.28, and 5.21 at 25 °C. The pKa of the hydroxyl group has been found, by means of ^{13}C NMR spectroscopy, to be 14.4. The solution of citric acid and its salts can act as buffer solutions between about pH 2-8. In biological systems around pH 7, the two species present are the citrate ion and mono-hydrogen citrate ion.

The citrate ion forms complexes with metallic cations. The stability constants for the formation of these complexes are quite large because of the chelate effect. Consequently, it forms complexes even with alkali metal cations. However, when a chelate complex is formed using all three carboxylate groups, the chelate rings have 7 and 8 members, which are generally less stable thermodynamically than smaller chelate rings. In consequence, the hydroxyl group can be deprotonated, forming part of a more stable 5-membered ring, as in ammonium ferric citrate.

Citrate is an intermediate in the TCA cycle (TriCarboxylic Acid cycle, or Krebs cycle), a central metabolic pathway for animals, plants, and bacteria. Citrate synthase catalyzes the condensation of oxaloacetate with acetyl CoA to form citrate. Citrate then acts as the substrate for aconitase and is converted into aconitic acid. The cycle ends with regeneration of oxaloacetate. This series of chemical reactions is the source of two-thirds of the food-derived energy in higher organisms.

Procedure

1. Lemon was cut into pieces and squeezed to get the juice.
2. Measured the amount of lemon juice using a measuring cylinder
3. Transferred the 150 mL of lemon juice into a clean 250 mL beaker and tested its pH (2 or 3)
4. Added 10% sodium hydroxide solution till the pH becomes >9. The solution became cloudy and the colour changed to a deep orange colour.
5. Filtered the solution to a new clean 500 beaker under vacuum.
6. Added 30 % calcium chloride solution to the beaker solution. Mixed the solution thoroughly.
7. Boiled the solution for 45 minutes in a water bath to precipitate the insoluble calcium citrate.
8. Filtered the precipitate of white calcium citrate under vacuum. Dried it.
9. Weighed the amount of isolated calcium citrate.

10. Calculated the required amount of sulfuric acid to neutralise the calcium citrate. Diluted the sulfuric acid with distilled water.
11. Suspended the calcium citrate in a minimum amount of water.
12. Slowly, added the dilute sulfuric acid to the suspended the calcium citrate.
13. Filtered the solution into a clean 250 mL beaker and concentrated to a small volume in a hot plate.
14. Kept the solution with cover for about 1 to 2 weeks to crystallise the dissolved citric acid.
15. Isolated the crystals of citric acid by filtration.
16. Weighed the isolated citric acid.

Report

The amount of citric acid isolated from 150 mL of lemon juice was _____g.