KARPAGAM ACADEMY OF HIGHER EDUCATION
(Deemed to be University)
(Established Under Section of UGC Act 1956)
Coimbatore-641 021
For the Candidates admitted from 2016 Onwards
DEPARTMENT OF CHEMISTRY

SUBJECT :INORGANIC MATERIALS OF INDUSTRIAL IMPORTANCE

PRACTICAL
SEMESTER :V
SUBJECT CODE :16CHU513A CLASS : III B.Sc Chemistry

Course Objectives
The lab course enables the students to

1. Analyse various fertilizers.
2. Determine the composition of dolomite and cement.
3. Analysis of metals in alloys.

Course Outcome
The lab course enabled the students to

1. Determine the free acidity, calcium and phosphoric acid in fertilizers
2. Determine the composition of dolomite.
3. Analysis of metals in alloys.

Experiments
1. Determination of free acidity in ammonium sulphate fertilizer.
2. Estimation of Calcium in Calcium ammonium nitrate fertilizer.
3. Estimation of phosphoric acid in superphosphate fertilizer.
4. Electroless metallic coatings on ceramic and plastic material.
5. Determination of composition of dolomite (by complexometric titration).
6. Analysis of (Cu, Ni); (Cu, Zn) in alloy or synthetic samples.
7. Analysis of Cement.
8. Preparation of pigment (zinc oxide).

Suggested Readings:
Text Books:

1. Stocchi, E. (1990). *Industrial Chemistry*, Vol I. Ellis Horwood Ltd., UK.

2. Felder, R. M. & Rousseau, R.W. (2005). *Elementary Principles of Chemical Processes*. Wiley Publishers, New Delhi.
3. Kingery, W. D., Bowen H. K. & Uhlmann, D. R. (1976). *Introduction to Ceramics.* Wiley Publishers, New Delhi.

Reference Books:

1. Jain, P. C. and Jain, M. (2015). *Engineering Chemistry.* Dhanpat Rai & Sons, New Delhi.
2. Gopalan, R., Venkappayya, D. & Nagarajan, S. (2010). *Engineering Chemistr* (I Edition). Vikas Publications, New Delhi.
3. Sharma, B. K. (2012). *Engineering Chemistry* (VI Edition). Goel Publishing House, Meerut.

WEBSITES

Ec.europa eu
Indanfertiliser.com
https://www.sharathplating.com
Pubs.acs.org
www.dot.ca.gov
Lakshlaksh.weeebly.com

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SEMESTER :V
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S.No.	Lecture Hour	Topics to be Covered	Support Material/Page Nos
1	4	Writing the experimental procedure.	
2	4	Determination of free acidity in ammonium sulphate fertilizer	W1
3	4	Estimation of Calcium in Calcium ammonium nitrate fertilizer.	W2
4	4	Estimation of phosphoric acid in superphosphate fertilizer.	W1
5	4	Electroless metallic coatings on ceramic	W3
6	4	Electroless metallic coatings on plastic	W3
7	4	Determination of composition of dolomite (by complexometric titration).	W4
8	4	Analysis of (Cu, Ni) in alloy or synthetic samples	W4
9	4	Analysis of (Cu, Zn) in alloy or synthetic samples	W4
10	4	Analysis of Cement.	W5

11	4	Preparation of pigment (zinc oxide).	W6
12	4	Model Practical Examination.	
	Total No. of Hours Planned For Practical's =48		

Suggested Readings

WEBSITES

W1:Ec.europaeu

W2: Indanfertiliser.com

W3:https://www.sharathplating.com

W4:Pubs.acs.org

W5:www.dot.ca.gov

W6:Lakshlaksh.weebly.com

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

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INORGANIC MATERIALS OF INDUSTRIAL IMPORTANCE PRACTICAL

LAB MANUAL

FOR

III B.Sc., CHEMISTRY STUDENTS

DEPARTMENT OF CHEMISTRY

KARPAGAM ACADEMY OF HIGHER EDUCATION

(Deemed to be University established Under section 3 of UGC Act, 1956)

Eachanari Post, Pollachi main road,

Coimbatore-641021

Tamilnadu, India

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

CONTENTS

EXPERIMENTS

1.Determination of free acidity in ammonium sulphate fertilizer

2.Estimation of calcium in calcium ammonium nitrate fertilizer

3. Estimation of phosphoric acid in superphosphate fertilizer

4.Electroless metallic coatings on ceramic and plastic material

5. Analysis of (Cu, Ni); (Cu,Zn) alloy or synthetic samples

6.Preparation of pigment

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

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Experiment No 1

Determination of free acidity in ammonium sulphate fertilizer

Aim

To determine free acidity in ammonium sulphate fertilizer.

(a) Reagents

(1) Standard sodium hydroxide solution - 0.02 N.

(2) Methyl red indicator-Dissolve 0.15 g of water soluble methyl red in 500 ml water.

(3) Methyl red - Methylene blue mixed indicator solution - prepared by mixed equal volumes of 0.2 per cent solution in rectified spirit of methyl red and 0.1 percent solution in rectified spirit of methylene blue.

(b) Procedure

(1) Dissolve about 20 g of prepared sample, accurately weighed in about 50 ml cold natural water.

(2) Filter and make up the volume to about 200 ml.

(3) Titrate with standard sodium hydroxide solution, using one or two drops of methyl red as indicator.

(4) If satisfactory end point with methyl red is not obtained, methyl red - methylene blue mixed indicator may be used.

(5) Use preferably a micro biuret for this titration. The filtering medium shall be neutral and shall not contain any alkaline material which would neutralise free acid.

Calculations

Free acidity as H_2S04 per cent by weight = 4.904 AN / W

A = Volume of ml of standard NaOH solution.
N = Normality of standard NaOH solution.
W = Weight in gm of prepared sample taken for the test.

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Result:

Free acidity in ammonium sulphate fertilizer =

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Experiment No 2

Estimation of calcium in calcium ammonium nitrate fertilizer

Aim

To determine calcium in calcium ammonium nitrate fertilizer

(a) Reagents

(1) N-Amyl alcohol.

(2) Dilute hydrochloric acid - approximately 4N.

(3) Standard calcium solution - Weigh 1.0 g of calcium carbonate dried at 120^0 C and dissolve in the minimum quantity of dilute hydrochloric acid. Dilute the solution to 1 litre in a graduated flask.

(4) Ammonium chloride - Ammonium hydroxide buffer solution. Dissolve 67.5 g ammonium chloride in a mixture of 570 ml of ammonium hydroxide (sp. gr. 0.92) and 250 ml water. Also dissolve separately a mixture of 0.931 gm of disodium ethylene diamine tetra-acetate dihydrate and 0.616 g of magnesium sulphate ($Mg\ SO_4.7H_2O$) in about 50 ml of water. Mix the two solutions and dilute to 1 litre.

(5) Standard disodium ethylene diamine tetra-acetate (EDTA) solution - Weigh 3.72 g of disodium ethylene diamine tetraacetate dihydrate in water, and dilute in a graduated flask to 1 litre. The solution shall be standardised frequently against standard calcium solution following the procedure given below.

(6) Eriochrome black-T indicator solution - Dissolve 0.1 g in 20 ml of rectified spirit. The solution shall be used for not more than a week.

(b) Procedure

(1) Grind quickly about 5 g of the material, accurately weighed, with about 50 ml of amyl alcohol in a pestle and mortar and transfer the contents to a conical flask.

(2) Wash the pestle and mortar with a few ml of amyl alcohol and add the washings to the flask.

(3) Shake the contents of the flask manually or in a mechanical shaker for about half an hour and then filter.

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

(4) Transfer the filtrate to a separating funnel and extract calcium nitrate completely with water in five to six installments.

(5) A few drops of dilute hydrochloric acid may be added during the extraction with water to avoid formation of an emulsion of amyl alcohol with water.

(6) Concentrate the water extract at low temperature to nearly half its volume.

(7) Transfer the concentrated solution to a conical flask, add 5 ml of ammonium chloride-ammonium hydroxide buffer solution, 5 drops of eriochrome black-T indicator solution and titrate against standard EDTA solution to a pure blue end point.

Calculations

Calcium nitrate per cent by weight = 8.2 NV / W

Where
N = Normality of standard EDTA solution
V = Volume in ml of standard EDTA solution used in the titration, and
W = Weight in g of the material taken for test.

Result:

Calcium in calcium ammonium nitrate fertilizer =

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Experiment No 3

Estimation of phosphoric acid in superphosphate fertilizer

Aim

To estimate phosphoric acid in superphosphate fertilizer

(a) Reagent

1. Acetone

2. Standard sodium hydroxide solution - 0.1 N

3. Bromocresol green indicator solution - Dissolve 0.1 g bromocresol green in 100 ml of rectified spirit.

(b) Procedure

1. Weigh accurately about 2.5 g of the prepared sample in a 250 ml Erlenmeyer flask.

2. Add 100 ml neutral acetone. Fix to a wrist action shaker. Shake for one hour.

3. Filter rapidly through whatman filter paper No. 1 in to 250 ml erlenmeyer flask, wash with four time, 10 ml portion of acetone.

4. Evaporate acetone as far as possible.

5. Add about 50 ml water and drops of bromocresol green indicator.

6. Titrate with standard NaOH solution, until the colour changes from yellow to blue.

Calculations

Free phosphoric acid (as P_2O_5) per cent by weight = 7.1 x N x V / W

Where :

N=Normality

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

V=Volume .

W =Weight in g of sample taken for the test.

Result

Phosphoric acid in superphosphate fertilizer=

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Experiment No 4

Electroless metallic coatings on ceramic and plastic material

Aim

To determine electroless metallic coatings on ceramic and plastic material.

Procedure:

In this study, the electroless nickel plating on ABS plastic were investigated. Experimental study consisted of four parts: Preparation of materials, etching, coating and analysis; and this study was carried out at room temperature in the fume hood. The effects of two different bath compositions on plating were investigated.

1. ABS plastic is grounded with 400, 800 and 1500 grit sandpapers.
2. Bath 1 and Bath 2 are prepared with different concentrations as listed in Table 1.

Table 1.Bath compositions for electroless nickel deposition (Schlesinger M, 2010)

	Alkaline Baths	
Bath Constituents (g/L)	**Bath 1**	**Bath 2**
Nickel Chloride	20	20
Sodium Hypophosphate	20	20
Sodium Citrate	10	10
Ammonium Chloride	35	35
pH	9-10	9-10
Temperature (°C)	85	85

3. Bath solutions are heated to plating temperatures: Bath 1 to 95°C, Bath 2 to 85°C.
4. According to the two different baths and for three sandpapers, 18 samples of ABS plastic are prepared.
5. NaOH is gradually added during the process to make sure that the pH of the plating solution is between 8- 10 and 9-10 for Bath 1 and Bath 2, respectively.
6. 3 sets of ABS plastic specimen are prepared and plated for 20 minute intervals. First 3 samples are taken out of the baths at the end of 20 minutes, the other 3 are taken out of the baths at the end of 40 minutes and the final 3 samples are taken out of the baths at the end of 60 minutes.
7. All samples are left for drying at room temperature for 24 hours.

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Result

Electroless metallic coatings on ceramic and plastic material=

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

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Experiment No 5

Analysis of (Cu.Ni); (Cu, Zn) in alloy or synthetic samples

Aim

To analyse (Cu.Ni); (Cu, Zn) in alloy or synthetic samples

Reagents :

1. Brass powder
2. conc. HNO_3
3. 10 N HNO_3
4. dil. HNO_3
5. conc. H_2SO_4
6. 1% H_2SO_4
7. conc. HCl
8. dil. HCl
9. 10% ammonium thiocyanate
10. NH_3
11. NH_4Cl
12. 10% ammonium hydrogen phosphate
13. Distilled water

Procedure:

Estimation of Tin

1. Weigh accurately about 1.0000 g of Brass powder
2. Transfer brass into 250 mL beaker
3. To that add 15 mL of 10 N HNO_3 using measuring jar.
4. The precipitate (stannic acid) will form, and keep it for settling. After settling the precipitate ensure the complete precipitation of all stannic acid by adding two drops of dil. HNO_3 along the side of the beaker) and it was evaporated on a water bath nearly to dryness
5. Taken out from the water bath.
6. Dilute with 50 mL water, stirred using glass rod and heat for 25 min
7. Add some paper pulp (1 or 2) and stirred using glass rod
8. Taken out from the burner and allow to cool
9. Filter through whatmann filter paper and wash the precipitate with dil. HNO_3

10. Take silica crucible from desiccator and weighed
11. Transfer the precipitate with filter paper into weighed silica crucible
12. Ignite the precipitate at 900-1000 ^{o}C up to getting constant weight of SnO_2 (**Tin dioxide**)
13. Taken out from the electric burner and cool it and keep it in desiccator (5 min) using tongs.
14. Taken out from the desiccator and weighed

Estimation of Lead

1. Concentrate the above filtrate to 50 mL
2. Cool and add conc. H_2SO_4 and evaporate the solution until fumes evolved
3. Cool it and dilute to 100 mL using distilled water and again heat the solution
4. Filter through a previously weighed sintered crucible
5. Washed the above obtained precipitate with 1% H_2SO_4
6. Dried at 130 ^{o}C and weighed it as $PbSO_4$ (**Lead(II) sulfate**)

Estimation of Copper

1. Evaporate the filtrate (obtained above after complete separation of Lead) to 50 mL
2. Add 5 mL dil. HCl and diluted to 100 mL using water
3. Add 3 g sodium thiosulfate to the solution
4. Heated to boiling and added 10 mL 10% ammonium thiocyanate
5. Boiled and filtered through a previously weighed sintered crucible
6. Washed with water and dried and weighed as CuSCN (**copper(I)thiocyanate**)

Estimation of Iron

1. To the filtrate obtained after the complete separation of copper add 35 mL conc. HNO_3 and 15 mL conc. HCl
2. Evaporate to dryness and the add 3-5 mL of conc. HCl
3. Warm it and dilute to 50-75 mL water and heated to boiling.
4. To the boiling solution, add ammonium hydroxide solution carefully with constant stirring untill a faint,but distinct, smell of ammonia persists over the solution.
5. Boil further for 2-3 minutes.
6. Precipitate iron as $Fe(OH)_3$ (**iron(III) hydroxide**)
7. Filter it through Whatmann filter paper and ignite on previously weighed silica crucible
8. Cool and weighed

Estimation of Zinc

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

1. To the final filtrate add 10 mL conc. HCl
2. Heat the filtrate
3. Taken out and pass H_2S using Kipp's apparatus
4. Filter and wash with water)
5. Collect the filtrate (discard the precipitate)and boil off H_2S
6. Taken out and add 3 g NH_4Cl and conc. HNO_3 boil and add NH_3
7. Again boil and filter using filter paper
8. Collect the filtrate
9. Add methyl red and 5 mL conc. HCl
10. Neutralize with 5 mL NH_3
11. Add 15 mL 10% ammonium hydrogen phosphate
12. Filter, wash with water ignite on previously weighed silica crucible and weighed as $Zn_2P_2O_7$ (**Zinc Pyrophosphate**)

Result

Electroless metallic coatings on ceramic and plastic material is estimated.

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

Experiment No 6

Preparation of Pigment

Aim

To prepare pigment Prussian blue and Chrome yellow.

Preparation of Prussian Blue

Make a solution of hydrated iron (III) chloride by dissolving 5 grams of the salt in 50 mL of water. Stir the solution briskly, using a spatula, until the salt particles are properly dissolved in the water.

2. Make a solution of potassium ferrocyanide by dissolving 10 grams of the salt in 75 mL of water. Stir the solution briskly, using a spatula, until the salt particles are properly dissolved in the water.

3. Add iron chloride solution, slowly, into potassium ferrocyanide solution while stirring briskly.

4. Leave the dark blue (prussian blue) mixture, so formed, undisturbed for 15 minutes.

5. Prepare a gravity filter by setting a folded filter paper in the form of a cone, which is stuck to the inner edge of a funnel, which is then set on a conical flask

6. Pour the mixture, slowly, over the gravity filter, and allow the powder to precipitate.

7. Once the filter paper is dried up, carefully remove the filter paper and pour the powder in a china dish.

8. Scrape the excess powder that is stuck on the filter paper, by using a spatula.

9. The powder obtained, is the paint pigment of the shade prussian blue.

10. Weigh the powder formed on an electronic weighing scale, and calculate the efficiency of formation.

Reaction Involved:

$3K_4[Fe(CN)_6] + 4FeCl_3 \rightarrow Fe_4[Fe(CN)_6]_3 + 12KCl$

Preparation of Chrome Yellow

1. Make a solution of potassium chromate by dissolving 7 grams of the salt in 50 mL of water. Stir the solution briskly, using a spatula, until the salt particles are properly dissolved in the water.

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019

2. Make a solution of lead nitrate by dissolving 10 grams of the salt in 100 mL of water. Stir the solution briskly, using a spatula, until the salt particles are properly dissolved in the water.
3. Add potassium chromate solution, slowly, into lead nitrate solution while stirring briskly.
4. Leave the yellow (chrome yellow) mixture, so formed, undisturbed for 15 minutes.
5. Prepare a gravity filter by setting a folded filter paper in the form of a cone, which is stuck to the inner edge of a funnel, which is then set on a conical flask
6. Pour the mixture, slowly, over the gravity filter, and allow the powder to precipitate.
7. Once the filter paper is dried up, carefully remove the filter paper and pour the powder in a china dish.
8. Scrape the excess powder that is stuck on the filter paper, by using a spatula.
9. The powder obtained, is the paint pigment of the shade chrome yellow.
10. Weigh the powder formed on an electronic weighing scale, and calculate the efficiency of formation.

Reaction Involved:

$K_2CrO_4 + Pb(CH_3COO)_2 \rightarrow PbCrO_4 + 2\ CH_3COOK$

Result

Weight of Prussian blue= ----------gm

Weight of chrome yellow= ----------gm

Class: III B.Sc Chemistry Course Name:Inorganic Materials of Industrial Importance Practical

Course Code:16CHU513A Batch:2016-2019