

17CHU513B	NOVEL INORGANIC SOLIDS - PRACTICAL	Semester-V
		4H 2C
Instruction Hours/week: L:0 T:0 P:04		Marks: Internal: 40 External: 60 Total:100

Scope

The lab course involves practical methods regarding the testing of novel inorganic solids

Objective

The course helps the student to

1. Understand the cation exchange method and coprecipitation methods of novel inorganic solids
2. Understand the method for the preparation of nanoparticles
- 3.

Methodology

Preparation of nano particles, coprecipitation method

1. Determination of cation exchange method
2. Determination of total difference of solids.
3. Synthesis of hydrogel by co-precipitation method.
4. Synthesis of metal nanoparticles.

Suggested Reading:

1. Fahlman, B.D. (2004). *Materials Chemistry*, Springer.

List of Experiments



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(Deemed to be University)
(Established Under Section 3 of UGC Act 1956)
COIMBATORE-21
DEPARTMENT OF CHEMISTRY

List of Experiments

Name of the Staff : **R Kumar**
Department : **Chemistry**
Subject : **Novel Inorganic Solids-Practical**
Subject Code : **17CHU513B**
Class : **III B.S Chemistry**
Year and Semester : **III / V**

S.No	NAME OF THE EXPERIMENT
1	Determine the amount of suspended solids in the given sample of water
2	Determination the amount of total dissolved solids in the given sample of water.
3	Determination the amount of Alkalinity in water sample
4	Determination of free Carbon dioxide water sample
5	Estimation of dissolved oxygen of boiled feed water
6	Estimation of Salinity of water sample
7	Estimate the type and amount of anionic solid present in the given solution



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EXPERIMENT: 1

Aim: To determine the amount of suspended solids in the given sample of water.

Requirements: Water sample 300 ml, Distilled water; watmann filter paper No. 30 or 31;

Funnel, Glass rod, Pipettes 25 ml, 10 ml, & Beaker 500 ml.

Process: Weight one filter – paper exactly. Take 100 ml. (Exactly) of the sample water & filter through the filter paper. Wash the solids with small portions of hot distilled water. Dry and weight filter – paper containing the solids. The difference in the weight gives the amount of suspended solids in 100 ml. of the sample water. Calculate the amount in ppm. Tabulate your observations & present your calculations.

Calculation: ppm suspended solids = $\frac{10^6}{100} * W$

Results: Total Suspended solids = _____ppm.



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EXPERIMENT: 2

Aim: To determine the amount of total dissolved solids in the given sample of water.

Requirement: Porcelain dish, Pipette 25 ml, Sample water 300 ml, Gas burner, Water bath.

Process: Weight a dry clean 250-ml. capacity porcelain evaporating dish. Take 100 ml. (Exactly) sample water in to it. Heat the dish on a sand bath till all the water gets completely evaporated leaving behind dry solids. Dry the dish, cool in a desiccator and weight. The difference in the weight gives the amount of total dissolved solids. Tabulate your observation and express the result in ppm.

Calculation: Total dissolved solids = $\frac{10^6}{100}$ * Difference in weight of the porcelain dish.

Results: Total Dissolved solids = _____ ppm.



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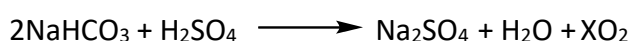
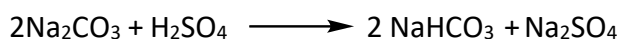
Batch: 2017 -2020

EXPERIMENT: 3

Aim: To determination the amount of carbonate and bicarbonates.

Method: Titration of definite quantity of water against a standard acid using phenolphthalein & Methyl orange as indicators. Firstly phenolphthalein is added as an indicator. On adding the slandered acid drop – wise, the pink colour disappears when all the carbonate in the sample water are converted to bicarbonates. At this stage methyl orange is added as an indicator. Now the colour of the sample water is yellow. Titrate further against the same slandered acid. Colour changes to orange when the end – point is reached. Comparison of the stage wise reading indicates thefollowing.

- (i) If the first stage reading (phenolphthalein indicator) is half the total reading, we may say the alkalinity was due to carbonates along and hence the amount of carbonates can be calculated directly.
- (i) If the first stage reading is zero, the reading shows that the alkalinity is due to bicarbonates along and hence the amount can be calculated directly.
- (ii) If the second stage reading is zero, the alkalinity is due to hydroxides alone and hence can be calculated directly,
- (iv) If the first stage reading is greater than half the total reading the alkalinity is due to both carbonates and hydroxides.
- (v) If the first stage reading is less than half the total reading, the alkalinity is due to both carbonates & bicarbonates. The equations of the reactions in this case are.



Requirements: 0.05M H₂SO₄, 100 ml, Sample water, Conical flasks, Burette, Pipette 25 ml, Phenolphthalein & Methyl orange indicator.

Process: Pipette out 25 ml, of sample water into a clean dry flask. Add 5 drops of phenolphthalein. The solution turns pink showing the presence of

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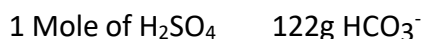
carbonates. Add the acid from the burette drop wise till the solution becomes colour less. Note the reading; now to the same bulk of solution add 3 drops of methyl orange. The solution turns yellow. Titrate further adding the acid from the burette drop wise till the colour change to orange. Note the reading. This procedure should be repeated a number of times with fresh quantity of sample water each time till constant reading are obtained. Calculate carbonates and bicarbonates from readings.

Calculation: If x ml. of 0.05 M H_2SO_4 is required to convert the quantity of carbonates present in 25 ml. sample water to bicarbonate (phenolphthalein reading) then 2 X ml. of 0.05 M H_2SO_4 will be required to neutralize the total amount of carbonates.

If Y ml. of 0.05 M H_2SO_4 are required to neutralize bicarbonates (Methyl orange reading) then Y-X ml. of 0.05 M H_2SO_4 will be required to neutralize the bicarbonates present in 25 ml of the sample water because X ml. are required to neutralize the bicarbonates obtained from carbonates. (Refer to equation)



$$\therefore 2X * \frac{0.006 * 10^6}{25} = \text{ppm Carbonates.}$$



$$\therefore (y-x) * \frac{0.0061 * 10^6}{25} = \text{ppm bicarbonates.}$$

Results: Carbonates in the sample water = _____ ppm.

Bicarbonates in the sample water = _____ ppm.



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EXPERIMENT: 4

Aim: To determination the amount of chloride in the given sample of water.

Method: Soluble chlorides can be determined by titrating then against silver nitrate solution using potassium chromate as an indicator. The reactions taking place are.



Thus as soon as the chlorides gets precipitated as AgCl the next drop of AgNO₃ will react with K₂CrO₄ to give red precipitates which marks the end point of the titration.

Requirement: 100 ml. sample water, 0.01 M. AgNO₃ solution, 5% K₂CrO₄ solution as an indicator, Burette, Pipette, Flasks etc. (To prepare the indicator dissolve 5 gm of K₂CrO₄ in 50 ml. distilled water, add AgNO₃ solution drop wise till it becomes slightly red. Filter & dilute to 100 ml.)

Process: Pipette out 25 ml. of the sample water in a dry clean conical flask. Neutralize CO₃ & HCO₃ by just sufficient quantity of H₂SO₄ and add about 10 ml. of distilled water to it. Now add 2 drops of the indicator and titrate against the given AgNO₃ solution from the burette. Stir continuously while titrating. Add the AgNO₃ solution drop wise till the permanent chocolate red colour is formed in the solution (Sample water). Repeat the procedure a number of times with fresh 25-ml. of sample water each time till a constant reading is obtained Tabulate your observations.

Calculation: 1 Mole AgNO₃ 35.453 gm/mole of Cl⁻

∴ 1 ml of 0.01 M AgNO₃ 0.00035453 gm/mole of Cl⁻

∴ $\frac{\text{Constant reading} \times 0.00035453 \times 10^6}{25} = \text{ppm Cl}^-$

25

Result: To determine the total acidity of the given sample of water _____ppm.



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Class: III BSc (Chemistry)

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Batch: 2017 -2020

EXPERIMENT: 5

Aim: To determination the amount of total acidity in the given sample of water.

Requirement: Burette, Pipette, 0.02 M NaOH solution 500 ml of the sample water & Phenolphthalein indicator.

Process: Pipette out 50 ml. of the sample water in a clean dry conical flask. Add 1 – 2 drops of the indicator. Titrate rapidly against the 0.02 M NaOH solution from the burette, stirring gently till a faint permanent pink colour appears. Repeat the process several times with 50 ml. of sample water each time till a constant burette reading is obtained. Tabulate your observations.

Calculation: The total acidity is expressed in terms of a calcium carbonate.

Constant reading * 0.002 * 10⁶ * 0.05 (Factor)

50ml

= _____ ppm.

Result: The total acidity expressed as Calcium Carbonate is = _____ ppm.



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Batch: 2017 -2020

EXPERIMENT: 6

Aim: To determination the total hardness (Permanent & Temporary) of the given sample of water.

Requirement: 500 ml. of water, burette, Pipette, Flask, buffer solution pH = 10, 0.01 M EDTA & Eriochrome black T indicator.

Process: Pipette out 50 ml. of sample water in a dry clean conical flask. Add 5 ml. of the buffer solution and five drops of indicator to it. Then titrate against the given EDTA solution from the burette till the red color changes to the permanent purple blue. See that no reddish tinge remains in the solution.

Hints: To get a sharp end point add a few drops of dilute HCl boil, cool and neutralize with just sufficient dilute NaOH before adding the buffer solution.

Calculation: 1 ml. 0.01 M EDTA 1 mg. Of CaCO_3 0.001 gm of CaCO_3

$$\therefore \frac{\text{Constant reading} * 10^6 * 0.001}{50\text{ml}} * \text{ppmCaCO}_3$$

The total hardness is to be expressed in terms of CaCO_3 .

Result: Total hardness in terms of CaCO_3 = _____ ppm.



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Class: III BSc (Chemistry)

Course Name: NOVEL INORGANIC SOLIDS PRACTICAL

Course Code: 17CHU514B

Batch: 2017 -2020

EXPERIMENT: 7

Aim: To determine the amount of calcium & magnesium in the given sample of water.

Requirement: 500 ml sample water, calcium precipitating buffer solution, buffer solution pH 10, Eriochrome black T indicator, burette, pipette, flask beaker, 0.01 M EDTA solution, funnels etc.

(To prepare calcium precipitating buffer solution, dissolve 6.0 g. of A.R. ammonium oxalate in 100 ml. distilled water and add 144.0 g. of A.R. grade NH_4Cl and 13-ml. conc. NH_3 – solution to it. Then dilute the bulk to 1.0 liter).

Process: Pipette out 100-ml. of the sample water into dry clean conical flask. Add 30.0 ml of the calcium precipitating buffer solution to it. Stir vigorously Allow it to stand for 1.5 hrs. Then filter this through two filter papers (Whatmann No.42) into dry clean beaker. Now, from the filtrate pipette out 50 ml add 25 ml of distilled water and 50 ml of buffer solution pH 10 add 10 drops of the indicator and titrate again the EDTA solution from the burette till the color change from wine red to blue. Prepare five sets of such titration. Get a constant reading. Tabulate your observation.

Calculation: Constant reading in Exp.6 is the reading for total hardness i.e. due to Ca^{+2} & Mg^{+2} (Say X ml) Constant reading in this Exp.7 is the reading for Mg^{+2} only (Say Y ml)

$\therefore (X-Y)$ ml, 0.01 M EDTA required for Ca^{+2} only

Now 1 ml. of 0.01 M EDTA 0.0004008G. Ca^{+2}

And 1 ml. of 0.01 M EDTA 0.0002432 b. Mg^{+2}

$$\therefore \frac{(X - Y) * 0.0004008 * 10^6}{50} = \text{ppm } \text{Ca}^{+2}$$

$$\text{and } \frac{Y * 0.0002432 * 10^6}{50} = \text{ppm } \text{Mg}^{+2}$$

Result: Amount of Ca^{+2} in the given sample water _____ ppm.

Amount of Mg^{+2} in the given sample water _____ ppm.



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Course Code: 17CHU514B

Batch: 2017 -2020

EXPERIMENT: 8

Aim: To determination the amount of Sulphate in the given sample of water.

Requirement: 250 ml. of sample water, conc. HCl 10% BaCl₂ solution Whatman filter paper No. 40, Funnel, Beaker, Flasks, Pipette, Desiccators, Silica crucible etc.

Process: Pipette out 50 ml. of the sample water into a dry clean beaker. Add 1.0 ml. of the conc. HCl (AR Grad) and boil. Now in another beaker take about 25-ml. of 10% BaCl₂ – solution, add 1 ml of conc. HCl and boil. Then add the hot solution, to the first beaker containing sample water in boiling condition, till precipitation is complete. Digest precipitates for 0.5 hour on a sand bath. Filter and wash the precipitates with hot distilled water till it is free from chloride (Test the filtrate with AgNO₃ solution). Dry the precipitates ignite in a silica crucible previously weighed. Cool the crucible in desiccators and weight. Heat the crucible again for about 15 minutes and weigh to a constant weight. Tabulate your observations.

Calculation: 233.4 g of BaSO₄ 96.06 g of SO₄⁻²
∴ Wt. of the ppts x 96.06 x10⁶
—————
233.4 x50
= _____ppm of SO₄⁻²

Result: The amount of SO₄⁻² in the given sample water ----- ppm.



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SAMPLE WATER ANALYSIS IS SHOWN BELOW

1. Suspended solids	= Nil	
2. Total dissolved solids	= 764	ppm
3. Carbonate	= 85.87	ppm
4. Bicarbonate	= 556.2	ppm
5. Chloride	= 75.9	ppm
6. Total hardness in terms of CaCO_3	= 260	ppm
7. Amount of magnesium	= 32.3	ppm
8. Amount of Calcium	= 51	ppm
9. Amount of sulphate	= 49.4	ppm
10. Total acidity	= Nil	

ANALYSIS OF CEMENT

Introduction: Cement is a technical name of a product which is considered to be a mixed silicate and an aluminosilicate. Thus the constituents for determination will be silica, alumina, ferric oxide, lime and magnesia. We have also to analyse loss on ignition and residue other than silicate.

EXPERIMENT: 1

Aim : To determine the loss on igniting the cement sample.

Requirements: Crucible, Cement sample, burner etc.

Process: Weigh accurately about 1 gm. of the sample in dry clean, previously weighed crucible. Heat the crucible on the burner for about 15 min. cool in desiccators and weigh. Repeat till constant weight is obtained. Record your observations.

Calculations:

$$\frac{\text{Loss in wt.} \times 100}{\text{Actual wt. of the sample taken}} = \% \text{ loss on ignition.}$$

Results: The percentage loss on ignition is----- %

EXPERIMENT: 2

Aim : To determination the total insoluble residue in the cement sample.

Requirements: Sample, evaporating dish, Conc. HCl, 1% NaOH solution methyl red indicator, funnels, conical flasks, wash-bottle, 2% NH₄Cl solution, crucible ash less filter paper etc.

Process: Weigh accurately 1 Gm. of the sample in porcelain evaporating dish. Add 10 ml. of distilled water. Add 5 ml. Conc. HCl and heat till no further effervescence is given out. Add 40 ml. distilled water and digest on a steam-bath for 15 min. Filter through an ordinary filter paper and wash it several times with hot water. Dissolve the residue in 100 ml. 10 % NaOH solution. Acidify the solution with Conc. HCl using methyl red as an indicator and add a few drops of the acid in excess. Filter through the ash less filter paper and wash several times with hot 2 % NH₄Cl solution. Dry and ignite the filter-paper containing residue desiccators and weigh. Heat again, cool and weigh to a constant weight. Record your observations.

Calculations: $\frac{\text{wt. of the residue} \times 100}{\text{Actual wt. of the sample}} = \% \text{ total insoluble residue.}$

N. B. Preserve the residue.

Results: Total insoluble residue in the sample in the sample cement is----- %.

EXPERIMENT: 3

Aim: To determine the total silica in the given sample.

Requirement: Residue from the above experiments, solid NH_4Cl , Conc. HCl , distilled water, beakers, funnels, wash bottle, conical flask 250 ml. measuring flask, crucible, whatmann filter paper No. 41, etc.

Process: Transfer the residue quantitatively to a 250 ml. beaker. Add 1 gm. NH_4Cl . Mix thoroughly. Add conc. HCl drop wise with constant stirring. Heat the beaker on boiling water bath for 0.5 hr. then add 50 ml. of hot distilled water. Filter through whatmann filter paper No. 4, wash the precipitate twice with 1: 7 HCl solutions and then with distilled water till it is free from chloride (10 washings). Collect the filtrate in a 150 ml. measuring flask and preserve it for the determination of total oxides, lime and magnesia. Dry and ignite the residue in a weighed crucible. Heat for 0.5 hr. cool in desiccators, weight, heat again for constant weight is obtained. Record your observations.

Calculation:
$$\frac{\text{Wt. of the residue} \times 100}{\text{Actual wt. of the sample}} = \text{X total silica.}$$

Results: Total silica in the sample cement is----- %.

EXPERIMENT: 4

Aim : To determine the total Oxides (Sesquioxides $\text{Fe}_2\text{O}_3 + \text{Al}_2\text{O}_3$) in the given sample of cement.

Requirement: Filtrate from Expt.3, Solid NH_4Cl , methyl red indicator, 50% liquor ammonia solution, Ash less filter paper, Crucible, Desiccators, Funnel, flasks, Pipettes, 1% NH_4NO_3 solution etc.

Process: Pipette out 50 ml. of cement sample in 250 ml. beaker. Add 5 gm NH_4Cl and two drops of methyl red indicator to it heat the constant just to boiling (90°C , cool up 50°C and add the liquor ammonia solution drop wise with constant string till the precipitates with 1% NH_4NO_3 solution collect the filtrate and preserve it for the determination of lime and magnesia. Dry and ignite the precipitates in a previously weighed, dry clean crucible. Heat the crucible for 0.5 hour, cool in desiccators, weight, and heat again, cool and weight till a constant weight is obtained. Record your observations.

Calculation: $\text{Wt. of Sequioxides} \times 5 = \text{Wt. in the sample taken}$
 $\therefore \frac{\text{Wt. of the residue} \times 100}{\text{Actual weight of the sample}}$
 $= \text{_____ \% Sequioxides}$

Results: Total Sequioxides in the sample cement = _____%

EXPERIMENT: 5

Aim: To determine the amount of lime (CaO) in the given sample of cement.

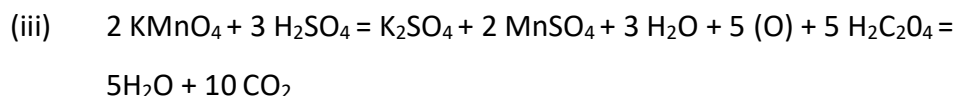
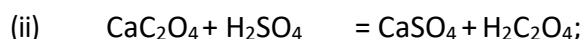
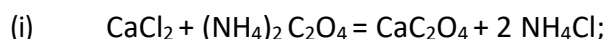
Requirement: Burettes, Pipettes, Beakers, Flasks, Cement sample, 2 M H₂SO₄, 0.01M KMnO₄ solution, Burner, 250 ml.-measuring flask, etc.

Process: Dilute the bulk to 250-ml with distilled water. Shake well. Now pipette out 25-ml. from this solution in a conical flask, add 25 ml. 2 M H₂SO₄, heat to 70⁰ C and titrate against the given 0.01 M KMnO₄ solution from the burette till a permanent pink colour is obtained (KMnO₄ solution is a self indicator in this titration). Repeat the titration each time with 25-ml. of the solution from the 250 ml. measuring flask to get a constant burette reading. Record your observations.

Burette reading: (1)_____ml (2)_____ml (3)_____ml
(4)_____ml

Constant burette reading: _____ml

Calculation: Equations for the reactions:



From the result we see that 2 moles of KMnO₄= 5 Moles of CaC₂O₄.

$$\frac{\text{Con. Burette reading} * 0.01}{1000} = \text{Moles of KMnO}_4.$$

And moles of KMnO₄ * 5/2 = Moles of H₂C₂O₄ = moles of Ca⁺²

Thus moles of Ca⁺² * 10= moles of Ca⁺² in the bulk of 250 ml

∴ Amount of Ca⁺² in the given solution = moles of Ca⁺² in original 250 ml. *
40.08 g/m.

∴ Amount of lime (CaO) present in the given solution = 56.08

Ca⁺² present.

*
A
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Result: Amount of lime (CaO) in the given sample cement = _____ gm.

EXPERIMENT: 6

Aim: To determine the amount of Magnesia (MgO) in the given sample cement.

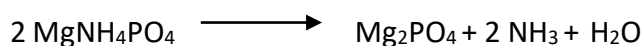
Requirement: Filtrate from Expt. 5, Methyl red indicator, Dilute HCl, 20% Diammonium hydrogen phosphate solution, 50% liquor ammonia solution, Ash less filter paper, Glazed paper, Crucible, Beakers, Flasks, Funnels, Burners etc.

Process: Magnesium content is to be weighed as $Mg_2P_2O_7$. Concentrate the filtrate from expt. 5 to about 250 ml. Add 2 to 3 drops of methyl – red indicator and a few drops of conc. HCl with stirring to make the solution acidic. Cool and add 25 ml. of 20% Diammonium hydrogen phosphate. Now add 50% liquor ammonia solution till the colour of solution turns yellow. Add 5 ml. in excess. Cook the precipitates on a boiling water bath for 1 hr. filtrate through ash less filter paper and wash the precipitates till it is free from chloride and phosphate. (Test for PO_4 is – filtrate + $(NH_4)_2MgO_4$ & heat. Dry the precipitates on a metal conc. with low flame take out the ppts. On a glazed paper and incinerate the filter paper in a previously weighed dry clean crucible. When the all carbons has bunts to ash, transfer the dry precipitates to the crucible with low flame for about 1 hr, cool it in desiccators & weigh. Repeating heating for about 15 min. till a constant weight is obtained. Record your observations.

Calculation: Equation for the reaction taking place are



heat



From the equation we learn that.

1 moles of $Mg_2P_2O_7$ 2 mole of Mg^{+2} 2 moles of $MgCl_2 \cdot 6H_2O$

i.e. 1.0 g of $Mg_2P_2O_7$ 0.2185 g. of Mg^{+2}

so wt. of the $Mg_2P_2O_7$ obtain from the expt. * 0.2185 = gms. Of Mg^{+2}

Gms of $Mg^{+2} \times 5 =$ Amount present original 250 ml. =

present original 250 ml.

% MgO = $\frac{\text{Amount of MgO present} \times 100}{\text{Actual wt. of the sample in expt. 2}}$

24.34

*
M
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2
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Result: The amount of magnesia in the given sample of cement = _____%

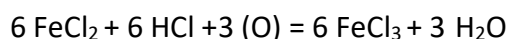
EXPERIMENT: 7

Aim : To determine the amount of Iron as Fe_2O_3 in the given sample of cement.

Requirement: Filtrate from Exp.3, Conc. HCl, 15% SnCl_2 solution, 2% HgCl_2 solution, 2 M H_2SO_4 solution, 2 M Na_2HPO_4 solution, 0.01 M $\text{K}_2\text{Cr}_2\text{O}_7$ solution, Diethyl amine indicator, Pipettes, Beakers, Flasks etc.

Process: Take 25 ml. of the filtrate from expt.3 in a conical flask. Add 1.0 ml. conc. HCl. Heat the content of the flask to boiling. Now with constant stirring of the contents add 15% SnCl_2 solution till yellow colour of the contents disappear. Add a few drops in excess. Cool the flask. Now add 10 ml. of HgCl_2 solution. White colour of the contents is formed. If gray colour is formed then starts with a fresh. To the white colored contents add 25 ml. of 2 M H_2SO_4 and 25 ml. of 2 M Na_2HPO_4 solution successfully and titrate the contents against the standard 0.01 M $\text{K}_2\text{Cr}_2\text{O}_7$ solution from the burettes. Use a few ml. of the indicator. The end – point is reached when the original greenish – blue colour of the contents change to permanent violet blue. Repeat for several times the process with fresh 25 ml. each time till a constant is obtained. Record your observations.

Calculation: Equation of the reactions taking place are:



From the equation we learnt that 1 mole $\text{K}_2\text{Cr}_2\text{O}_7 = 6$ moles FeCl_3 .

1. If X ml of the constant reading; $\frac{X * 0.01}{1000} = \text{moles of } \text{K}_2\text{Cr}_2\text{O}_7$
2. Moles of $\text{K}_2\text{Cr}_2\text{O}_7 * 6 = \text{Moles of } \text{Fe}^{+3}$
Moles of $\text{Fe}^{+3} * 10 = \text{Moles of } \text{Fe}^{+3} \text{ present in 250 ml.}$
3. Amount of $\text{Fe}^{+3} = \text{moles of } \text{Fe}^{+3} \text{ in 250 ml. original solution} * 55.85$
Now, 111.7 gm. of Fe^{+3} 159.7 gm. of Fe_2O_3 .

4. Amount of $\text{Fe}_2\text{O}_3 = \frac{159.7}{111.7} \times \text{amount of Fe}^{+3}$

$\% \text{Fe}_2\text{O}_3 = \text{Amount of Fe}_2\text{O}_3 \times 100 / \text{Actual weight of the sample as in exp.2}$

Result: The amount of iron as Fe_2O_3 in the given sample of cement is _____gms.

Reports of the SAMPLE ANALYSIS OF CEMENT are shown below.

- | | |
|----------------------|-------------------|
| 1. Loss of igniting | = 1.0% by weight |
| 2. Insoluble residue | = 1.2% by weight |
| 3. Total silica | = 7.2 % by weight |
| 4. Sesquioxides | = 28.5% by weight |
| 5. Lime | = 71.5% by weight |
| 6. Magnesia | = 3.83% by weight |
| 7. Ferric oxide | = 2.87% by weight |