

**Green University of Bangladesh**

**Department of CSE**

**CHE-102**

**Inorganic & Physical Chemistry Lab Manual**

|  |  |
| --- | --- |
| Student ID. |  |
| Student Name |  |
| Section |  |
| Name of the Program |  |
| Name of the Department |  |

|  |  |
| --- | --- |
| **CONTENTS** | |
| **Instructions for Laboratory** |  |
| **Laboratory Course Syllabus** |  |
| **Basic Measurement instruments** |  |
| **Experiment -1** | **Standardization of Sodium Hydroxide solution with standard Oxalic Acid solution** |
| **Experiment- 2** | **Standardization of Commercial Hydrochloric Acid solution with standard Sodium Hydroxide solution** |
| **Experiment- 3** | **Standardization of Hydrochloric Acid with Standard Sodium Carbonate Solution** |
| **Experiment- 4** | Standardization of Sodium Thiosulphate solution with standard Potassium Dichromate solution. |
| **Experiment -5** | **Estimation of Copper contained in a supplied solution by Iodometric method.** |
| **Experiment- 6** | Determination of Ferrous Iron by Standard Potassium dichromate solution. |
| **Experiment -7** | Standardization of Potassium Permanganate solution with Standard Sodium Oxalate Solution. |
| **Experiment- 8** | **Determination of Ferrous Iron in a Solution by Standard Potassium Permanganate (KMnO4) Solution.** |

**Instructions for Laboratory**

* The objective of the laboratory is learning. You should understand this laboratory manual clearly and how to use it effectively. It is his duty to learn it carefully. Conduct the experiments with interest and an attitude of learning.
* You need to come well prepared for the experiment
* Work quietly and carefully (the whole purpose of experimentation is to make reliable measurements!) and equally share the work with your partners.
* You must try to understand both the purpose and the principle behind each of the experiment you will do.
* You will organize your time effectively in advance of each experiment. As in the chemistry laboratory a student has to work with various hazardous chemicals, you should wear an apron during your sessional work.
* Do no put inflamed stick of matches here and there. smocking
* All chemicals are toxic to some extent, so you should never eat or drink in the laboratory
* Handle instruments with care. Report any breakage to the Instructor. Return all the equipment you have signed out for the purpose of your experiment.

**GREEN UNIVERSITY OF BANGLADESH (GUB)**

**COURSE SYLLABUS**

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| 1 | | **Faculty** | Faculty of Science & Engineering (FSE) | | | | |
| 2 | | **Department** | Department of CSE | | | | |
| 3 | | **Program** | B.Sc. in CSE | | | | |
| 4 | | **Name of Course** | Chemistry Laboratory | | | | |
| 5 | | **Course Code** | CHE 102 | | | | |
| 6 | | **Trimester and**  **Year** | Fall, 2020 | | | | |
| 7 | | **Pre-requisites** | Chemistry (CHE 101) | | | | |
| 8 | | **Status** | Basic Science Course | | | | |
| 9 | | **Credit Hours** | 1.0 | | | | |
| 10 | | **Section** | 193DA/201EE/202EA | | | | |
| 11 | | **Class Hours** | Wednesday (1.00 – 3.00PM) /Friday (4.30 – 6.00PM) / Friday (7.15 -8.30PM) | | | | |
| 12 | | **Class Location** | ZOOM | | | | |
| 13 | | **Course website** |  | | | | |
| 14 | | **Name (s) of Instructor(s)** | Md. Shakil Ahmed | | | | |
| 15 | | **Contact** | [shakil@tex.green.edu.bd](mailto:shakil@tex.green.edu.bd) or +8801521220396 | | | | |
| 16 | | **Office** | Room # 306, Campus-2 | | | | |
| 17 | | **Counseling Hours** |  | | | | |
| 18 | | **Text Book** | 1. A Text Book of Quantitative Inorganic Analysis by A I Vogel (3rd edition) | | | | |
| 19 | | **Reference** | 1. Vogel's - Textbook of quantitative chemical analysis (5th Edition) | | | | |
| 20 | | **Equipment & Aids** | Bring your notebook, graph papers and calculator. Equipment will be  provided in the laboratory. | | | | |
| 21 | | **Course Rationale** | Chemistry Laboratory is introduction to basic quantitative chemical laboratory techniques. Principles of chemical reactions and physical measurements are illustrated for CSE students. It aims to give students the practical idea about the practical applications of the theory they study at chemistry courses | | | | |
| 22 | **Course Description** | Chemistry is the study of the components of matter and how these components behave and interact with one another. The course will include lectures, daily assignments, and laboratory experiments. students will be able to analyze and discuss the data and potential solutions, using acid/base calculations and appropriate chemical formulas. Students will be expected to work simple mathematical computations and complete outside reading assignments. It is a pre-requisite for many other courses in CSE. | | | |
| 23 | **Course Outcome** | After the end of this course, the students will be able to:  **CO1.** Apply theoretical knowledge in laboratory and realize the insight of the formula and identify different metal  **CO2.**Become efficient in collaboration of various methods of performing experiments  **CO3.**Develop communication skill through simple project presentation and group | | | |
| 24 | **Teaching Methods** | Lecture, Laboratory equipment and experiments | | | |
| 25 | **Topic Outline** | | | | |
|  | **CLASS** | **Topics Or**  **Assignments** | **CLOs** | **Reading Reference** | **Activities** | |
|  | 1 | Introductory overview on the course | 1 |  | Lecture, Question-answer | |
|  | 2 | Standardization of Sodium Hydroxide solution with standard Oxalic Acid solution | 1,2,3 | Lab. Manual, Experiment No. 1 | Laboratory  Experiment | |
|  | 3 | Standardization of Commercial Hydrochloric Acid solution with standard Sodium Hydroxide solution | 1,2,3 | Lab. Manual, Experiment No. 2 | Laboratory  Experiment | |
|  | 4 | Standardization of Hydrochloric Acid with Standard Sodium Carbonate Solution | 1,2,3 | Lab. Manual, Experiment No. 3 | Laboratory  Experiment | |
|  | 5 | Standardization of Sodium Thiosulphate solution with standard Potassium Dichromate | 1,2,3 | Lab. Manual, Experiment No. 4 | Laboratory  Experiment | |
|  | 6 | Estimation of Copper contained in a supplied solution by Iodometric method. | 1,2,3 | Lab. Manual, Experiment No. 5 | Laboratory  Experiment | |
|  | 7 | Determination of Ferrous Ion in a Solution by Standard K2Cr2O7 Solution | 1,2,3 | Lab. Manual, Experiment No. 6 | Laboratory  Experiment | |
|  | 8 | Standardization of Potassium Permanganate solution with Standard Sodium Oxalate Solution. | 1,2,3 | Lab. Manual, Experiment No. 7 | Laboratory  Experiment | |
|  | 9 | Determination of Ferrous Ion in a Solution by Standard Potassium Permanganate (KMnO4) Solution standard sodium oxalate solution | 1,2,3 | Lab. Manual, Experiment No. 8 | Laboratory  Experiment | |
|  | 10 | Practice and review | 2-3 | Lab. Manual, Experiment No.  5-8 | Laboratory  Experiment | |
|  | 11 | Final Term Examination (Quiz, Lab test, Viva, Project Presentation) |  |  | Quiz, Lab test, Viva, Project Presentation | |
| 26 | **Assessment Methods**  **and Marks**  **Distribution:** | Students will be assessed on the basis of their overall performance in all the exams, quizzes, and class participation. Final numeric reward will be the compilation of (tentative):   * Attendance and Performance (AP) (10%) * Lab Project Presentation and viva (LV) (25%) * Continuous Lab Performance (LP) (`25%) * Lap Report (LR) (10%) * Final Exam (FE) (30%) | | | | |
| 27 | **Assessment Methods**  **of COs:** | Assessment methods of COs are given below:   |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | | COs | AP | LV | LR | LP | FE | | **CO1** | **√** | **√** | **√** | **√** | **√** | | **CO2** |  | **√** |  | **√** | **√** | | **CO3** |  | **√** |  |  | **√** | | | | | |
| 28 | **Mapping of COs with PLOs** | Mapping of COs with program learning outcomes (PLOs)are given below:   |  |  |  |  |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | | Program Learning Outcomes (PLOs) | | | | | | | | | | | | | | | COs | PLO1 | PLO2 | PLO3 | PLO4 | PLO5 | PLO6 | PLO7 | PLO8 | PLO9 | PLO10 | PLO  11 | PLO  12 | | **CO1** | **√** |  | **√** | **√** |  |  |  |  |  |  |  |  | | **CO2** |  | **√** |  | **√** |  |  |  |  |  |  |  |  | | **CO3** |  |  |  |  |  |  |  |  |  | **√** |  |  | | | | | |
| 29 | **Grading Policy** | The following chart will be followed for grading. This has been customized from the guideline provided by the School of Engineering and Computer Science.   |  |  |  |  |  |  |  |  |  |  | | --- | --- | --- | --- | --- | --- | --- | --- | --- | --- | | **A+** | **A** | **A-** | **B+** | **B** | **B-** | **C+** | **C** | **D** | **F** | | 80 and above | 75-<80 | 70-<75 | 65-<70 | 60-<65 | 55-<60 | 50-<55 | 45-<50 | 40-<45 | <40 | | | | | |
| 30 | | **Additional Course Policy** | 1. 1. Lab Reports:  Report on previous Experiment must be submitted before the beginning of new experiment. A bonus may be obtained if a student submits a neat, clean and complete lab report.  2. 2. Examination:  There will be a mid-term exam and final exam both of which will be closed book.  3. 3. Unfair means policy:  In case of copying/plagiarism in any of the assessments, the students involved will receive zero marks. Zero Tolerance will be shown in this regard. In case of severe offences, actions will be taken as per university rule.  4. 4. Counseling:  Students are expected to follow the counseling hours posted. In case of emergency/unavoidable situations, students can e-mail me to make an appointment. Students are regularly advised to check the data updates/materials.  5. 5. Policy for Absence in Class/Exam:  If a student is absent in the class for anything other than medical reasons, he/she will not receive attendance. If a student misses a class for genuine medical reasons, he/she must submit an application with the supporting documents (prescription/medical report). He/she will then have to follow the instructions given by the instructor for make-up.  In case of absence in the mid/final exam for medical grounds, the student must also get his/her application forwarded by the head of the department before a make-up exam can be taken.  It is recommended that the students inform the instructor beforehand through mail if they feel that they will miss a class/evaluation due to medical reasons. | | | | |
| 31 | | **Additional Information** | 1. Academic Calendar Fall 2020: <http://www.green.edu.bd/academics/academic-calendar> 2. Academic Information and Policies: <http://www.green.edu.bd/academics/academic-rules-a-regulations> 3. Grading and Performance Evaluation: <http://www.green.edu.bd/academics/academic-rules-a-regulations> 4. Proctorial Rules: <http://www.green.edu.bd/administrator/proctors-office> | | | | |

1. **Standardization of Sodium Hydroxide solution with standard Oxalic Acid solution**

**Objectives:**

1. To study the neutralization reaction
2. To observe the quantity of acid or base needed for neutralization.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength of a base with the help of a standard oxalic acid solution
2. Observe the end point of reaction by color change.

**Theory:—**

In this experiment we shall determine the strength of NaOH solution by a standard solution of Oxalic Acid. This is done by means of “Titration”. The important matters that are related with the experiment are stated below:

**Titration:** —In presence of a suitable indicator, the volumetric analysis in which a standard solution is added in another solution (whose strength is not known) to reach its end point to determine the strength of that solution is called ‘titration’.

**Standard Solution:**—A solution of known concentration is called a ‘standard solution’. **Indicator: —** In our acid-base titration there is an important use of indicator. An ‘indicator’ is a chemical substance that detects the equivalent point (i.e. the end point) of reaction by changing its color.

**Equivalent Point**:— The ‘equivalent point’ is the point in a titration when a stoichiometric amount of reactant has been added.

**Normality**: -The number of gram equivalent weight of a solute per liter of solution is called normality.

**Normality (N)** = gm equivalent of solute /liters per solution.

In this experiment the reaction we shall use is as follows:

HOOC-COOH + 2NaOH ——> NaOOC-COONa + 2H2O

The formula required to determine the strength of NaOH solution is:-

V base × S base = V acid × S acid (For 1:1 mole ratio)

Modified formula for Oxalic acid and Sodium hydroxide will be:

V base × S base = 2V acid × S acid

Macid = Molarity of the acid, Mbase = Molarity of the base

Vacid = Volume of the acid, Vbase = Volume of the base

The volume of Oxalic acid is measured by watching the Equivalent point. The point at which acid-base neutralizes each other is called “Equilibrium point”. This point is determined with the help of an indicator.

Why Phenolphthalein is used as indicator: In this experiment, ‘Phenolphthalein’ is selected as indicator whose working pH range is 8.3-10.0 i.e. it is works when the environment is acid. This indicator gives pink color in basic solution and becomes colorless when the base is neutralized.

|  |  |  |  |
| --- | --- | --- | --- |
| Indicator name | pH range | Colour in Alkaline solution | Colour in Acid solution |
| Phenolphthalein | 8.3-10.0 | Pink | Colourless |

In this experiment we are using NaOH and Oxalic acid. NaOH is a strong base but Oxalic acid is a weak acid. So the solution at equilibrium point consists of a salt whose basic part is strong. As a result there will be more OH- in the solution than H+ as the salt will be dissociated in the aquas solution. So, the solution would be basic which provides phenolphthalein to work properly. So Phenolphthalein becomes the perfect indicator to determine the end point of this reaction.

**Apparatus:**—

1. Conical flask

2. Burette

3. Pipette

4 Volumetric flasks

5. Stand

**Name of the chemicals used:—**

1. NaOH (sodium hydroxide, base)
2. HOOC-COOH (Oxalic-acid)
3. Phenolphthalein (indicator)

**Procedure:-**

Take 10 ml of supplied NaOH solution in a conical flask by means of a pipette and dilute it to about 50 ml. Add one drop of phenolphthalein indicator to the solution. Then add standard oxalic acid solution drop by drop from a burette. Shake the flask frequently while adding the acid solution. Stop the addition of oxalic acid solution as soon as the pink color of the solution as soon as the pink color of the solution just disappears. Note the burette reading. The burette reading should be taken carefully at the lower meniscus of the liquid. Difference of the initial and final burette reading gives the volume of the acid added. The process should be repeated at least twice. These should agree within ±0.1 ml. calculate the normality of the supplied NaOH solution. Using the following relation.

**Data and calculation:-**

Standardization of NaOH solution with standard Oxalic Acid Solution

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume of NaOH  (ml) | Burette  (ml) | reading | Volume  of  Acid  (ml) | Average  Reading  (ml) | Strength  of  Acid (M) |
| Initial  Reading | Final  Reading |
| 1 | 10 | 0 | 10 | 10 |  |  |
| 2 | 10 | 10 | 20.2 | 10.2 |  |  |
| 3 |  |  |  |  |  |  |

**Calculation:—**

from

V base × S base = 2V acid × S acid

we get

2V Oxalic-acid × S Oxalic-acid = V NaOH × S NaOH

Here,

V Oxalic-acid = ml (average)

S Oxalic-acid = M

V NaOH = ml

S NaOH = ?

**Result:**—

Determined strength of NaOH solution is:

S NaOH = N

**Students should know:**

1. What is normality of supllied solution?
2. What are the molecular structure of phenolphthalein and oxalic acid?
3. How does the indicator (phenolphthalein) work?
4. At what pH does the indicator changes it color?
5. Do you know why we see different color of different solutions?

#### Standardization of Commercial Hydrochloric Acid solution with standard Sodium Hydroxide solution

**Objectives:**

1. To study the strength of commercial hydrochloric acid

2. To observe the quantity of acid or base needed for neutralization.

3. To study secondary standard substance for titration

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength of an acid with the help of a standard base.
2. Observe the end point of reaction by color change.

**Theory:—**

The strength determination of both commercial Hydrochloric acid and NaOH is done by means of titration. In presence of a suitable indicator, a chemical substance that detects the end point of a reaction by changing its color, the volumetric analysis in which a standard solution is added in another solution to reach its end point and to determine the strength of that solution is called titration. The reaction between NaOH and Oxalic acid is –

2NaOH + HOOC- COOH ——> NaOOC- COONa + 2H2O

Indicator – Phenolphthalein, and the reaction between HCl and NaOH is -

NaOH + HCl ——> NaCl + H2O

Indicator – Methyl Orange

**Apparatus:—**

1. Conical flask,
2. Burette,
3. Pipette,
4. Volumetric flask,
5. Stand
6. Funnel

**Chemical Reagents**:—

1. Standardized NaOH solution
2. HCl solution
3. Distilled water
4. Oxalic Acid
5. Phenolphthalein
6. Methyl Orange

**Procedure:-**

Take 10 ml standard NaOH solution in a conical flask and dilute it to about 50 ml. Add 2 or 3 drops of methyl orange indicator to the solution. Then add previously prepared (approx. M/10) HCl acid solution drop wise from a burette. Shake the flask frequently during addition of HCl acid. Stop addition of HCl acid solution as soon as the yellow color of the solution as soon as the yellow color of the solution just changes to orange. Note the burette reading. Repeat the process at least two times and these should agree within ± 0.1 ml. Calculate the normality of the dilute HCl and from there calculate the strength of commercial HCl.

**Experimental Data:—**

Standardization of NaOH solution with standard Oxalic Acid solution

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume of NaOH  ( ml) | Burette Reading  (ml) | | Volume  of  Acid  (ml) | Average  Reading  (ml) | Strength  Of  NaOH  (M) |
| Initial  Reading | Final  Reading |
| 1 |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |

Standardization of HCl solution with standard NaOH solution

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume of NaOH  (ml) | Burette | Reading  (ml) | Volume  of  Acid  (ml) | Average  Reading  (ml) | Strength  Of  NaOH  (M) |
| Initial  Reading | Final  Reading |
| 1 |  |  |  |  |  |  |
| 2 |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |

**Calculation: -**

We know that, V acid × S acid = 2V base × S base

Determination of the molarity of NaOH :--

Here,

V acid = ml

S acid = M

V base = ml

S base =?

So, S base = ( × ) / M

= M

Determination of Molarity of HCl :

Here,

V acid = ml

S acid = ?

V ­base = ml

S base = M

So, S HCl (dilute) = ( × ) / = M

We also know, V concentrated × S concentrated = V dilute × S dilute,

Here,

V HCl(concentrated) = ml

S HCl(concentrated) = ?

V HCl(dilute) = ml

S HCl(dilute)  = M

So, S HCl(concentrated) = ( × ) / = M

But in the given HCl solution equal amount of water and hydrochloric acid were present,

i.e. water: hydrochloric acid was 1:1.

So, the actual strength of HCl = ×

= M

**Result:—**

The strength of HCl (dilute) is found: M

The strength of the supplied HCl (concentrated) is: M

**Percentage of error:—**

(Known Value – Observed Value) X 100

Percentage of error =

Known value

= ( ── )× /

= %

So, Percentage of error = %

**Discussion:—**

The following causes can be assumed for the possible cause of error.

1) It is very difficult to determine the end point accurately. So the measured value is deviated from the actual value due to either over titration or under titration. This can also be one of the reasons of the error. This is the main cause of error.

2) Another cause of error may rise from faulty burette reading.

If these causes could be avoided we could have got more accurate result of the strength of Commercial Hydrochloric Acid.

**Student should know**

1. What is the normality of the supplied HCl solution?
2. What is the molecular structure of methyl orange indicator?
3. Can we use phenolphthalein instead of methyl orange here? Justify your answer.
4. At what pH does the indicator changes its color?
5. Is it necessary to use buffer solution here? If not why?

#### Standardization of Hydrochloric Acid with Standard Sodium Carbonate Solution

**Objectives:**

1. To study the strength of hydrochloric acid with standard sodium carbonate.

2. To observe the molar ratio of acid or base needed for neutralization.

3. To study effect of different indicators and end point.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength of an acid with the help of a primary standard base.
2. Observe the end point of reaction by color change.

**Theory:**-

In this experiment we shall determine the strength of commercial Hydrochloric Acid solution by a secondary standard solution of Na2CO3. This is done by means of ‘Titration’. The important matters that are related with the experiment are stated below:

Titration: - In presence of a suitable indicator, the volumetric analysis in which a standard solution is added in another solution (whose strength is not known) to reach its end point to determine the strength of that solution is called ‘titration’.

Standard Solution: A solution of known concentration is called a ‘standard solution’.

Indicator:- In our acid-base titration there is an important use of indicator. An ‘indicator’ is a chemical substance that detects the equivalent point (i.e. the end point) of reaction by changing its color. Indicators have different structures in acidic and in basic solution.

Equivalent Point: - The ‘equivalent point’ is the point in a titration when a stoichiometric amount of reactant has been added.

Normality: The number of gram equivalent weight of a solute per litre of solution is called normality.

Normality (N) = gm equivalent of solute /litres solution.

It is known to us that both alkalimetry and acidimetry are based on neutralization reaction. If an acid-base reaction is such like that,

a ACID + b BASE = PRODUCT

Then we know that

V base × S base = V acid × S acid

so, S acid = ( V base × S base ) / V acid

Here ‘V’ represents the volume and ‘S’ represents the strength of the substance.

Reaction:

Neutralization reaction between Na2CO3 and HCl acid takes place into two steps-

Na2CO3 + 2HCl => NaHCO3 + 2NaCl

NaHCO3+HCl =>NaCl + H2CO3

The ultimate reaction,

Na2CO3 + 2HCl =>2NaCl + H2CO3

In the first step, the solution is basic due to the formation of a salt where the basic part is stronger than the acidic part (NaHCO3).So, in order to determine the equivalent point of this reaction Phenolphthalein is used. As the salt that forms due to the neutralization reaction, produces more OH-, so the solution becomes a basic one and thus it have a pH range above 7. We know that the working environment needed for phenolphthalein is basic; thus phenolphthalein becomes the perfect indicator for determining the end point of the first step of the reaction. In the second reaction, NaCl and Carbonic Acid is formed. Because of the presence of Carbonic Acid in the solution, it becomes acidic. So, ‘Methyl Orange’ (pH range 2.9-4.6) is used as indicator to determine the equivalent point.

|  |  |  |  |
| --- | --- | --- | --- |
| Name of the Indicator | pH Range | Colour in Alkaline solution | Colour in Acid solution |
| Phenolphthalein | 8.3 - 10.0 | Pink | Colourless |
| Methyl Orange | 2.9 - 4.6 | Yellow | Pink |

**Apparatus:-**

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

**Indicator:-**

1) Phenolphthalein

2) Methyl Orange

**Chemical Reagents:-**

1) Standardized Na2CO3 solution

2) HCl solution

3) Distilled water

**Procedure:-**

Take 10 ml of standard Na2CO3 solution in a conical flask and dilute it to about 50 ml. Add one drop of phenolphthalein and titrate against HCl (prepared as expt no. 2) contained in a burette. Now note the burette reading when just one drop of HCl discharges the pink color of the solution. This is the first end point. Then add 3-4 drops of methyl orange inside the same conical flask and continue titration against the same HCl acid. The end point reached when the yellow colour of the solution just changes to faint pink (orange). Note the burette reading. This is the second end point. The difference of the burette reading from initial to second end point will be the volume of the acid required in the titration. Repeat the whole experiment twice and these should agree within ± 0.1 ml initial to second end point. Calculate the strength of dil. HCl and then find out the normality of commercial conc. HCl acid.

**Experimental Data:-**

Standardization of HCl acid with standard Na2CO3 solution

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume of Na2CO3  ( ml) | Burette Reading (ml) | | | Volume  of  Acid  in  ml | Average  Reading  in  ml |
| Initial reading | First end point | Second end point |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |

Weight of Na2CO3 in gram= gm.

**Calculation: -**

Strength of Na2CO3 = ( × )/ ( × )

= M.

We know that, 2V acid × S acid = V base × S base

Determination of the normality of HCl :--

Here,

V base = ml

S base  = M

V acid  = ml

S acid = ?

So, S base = ( × )/ M

= M

**Result:-**

The strength of the supplied HCl is: M

**Discussion:-**

The following causes can be assumed for the possible cause of error:

While the solution of Na2CO3 was prepared, a little amount of extra water might have been added into the volumetric flask, this can be one of the reasons.

If these causes could be avoided we could have get a perfect result of the concentration of Commercial Hydrochloric Acid.

**Students should know:**

1. Is Na2CO3 a primary standard substance?
2. Why do you need to use two indicators here?
3. Can you use methyl orange first instead of phenolphthalein? Justify your answer.
4. Can you calculate the normality or molarity from the half reaction?

#### Standardization of Sodium Thiosulphate solution with standard Potassium Dichromate solution.

**Objectives:**

1. To study the strength of thiosulphate.

2. To study oxidation reduction titration.

3. To study the liberation and its titration.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength of thiosulphate with the standard dichromate solution

& liberated iodine.

1. Observe the end point by color change with starch indicator

**Theory:-**

Standardization is the process by which the strength of a solution is determined with the help of a standard solution. A solution of known concentration is called a standard solution. This experiment is done by means of titration. In presence of a suitable indicator, a chemical substance that detects the end point of reaction by changing its color, the volumetric analysis in which a standard solution is added in another solution (whose strength is unknown) to reach its end point to determine the strength of that solution is called titration.

Titration involving iodine or dealing with liberated iodine in chemical reaction is called iodimetry and iodometry respectively. This reaction is iodometric because iodine is obtained from KI.

The reactions of this experiment are:

6KI+14HCl+ K2Cr2O7 =2CrCl3+3I2 +7H2O+8KCl

(6 I- + 14 H+ + Cr2O7-2 + 6e- = 2 Cr+3 + 3 I2+ 7 H2O)

2Na2S2O3 + I2 = Na2S4O6 + 2NaI

(S2O3 2- - e- = S4O6 2- + I-)

Here K2Cr2O7 is an oxidizing agent and I- is a reducing agent. Again in the second reaction I2 is an oxidizing agent and S2O3-- is a reducing agent.

In the 2nd Step of the reaction a specific indicator is used that is “Starch”- which has a significant effect on iodine.

Starch+I2=Starch-Iodine (blue colour)

Starch-Iodine+6S2O3--=Starch +6I- + 3S4 O6—

If to a solution containing a little iodine, some starch solution is added and Na2S2O3 is run in from the burette, the blue color of the starch-iodine complex will disappear from the solution as soon as all the iodine has been reduced to iodide ion.

**Apparatus:-**

1. Conical flask,

2. Burette,

3. Pipette,

4. Volumetric flask,

5. Stand,

6. Funnel

**Chemicals:-**

1. Na2S2O3,

2. K2Cr2O7  ,

3. KI,

4. NaHCO3

5. HCl (Concentrated),

6. Starch (Indicator)

**Procedure:-**

Take 4 ml of 12% potassium iodide (KI) solution in a conical flask and dilute it to about 50 ml. Add about 1 g. of NaHCO3 and shake the flask until the salt dissolves. Add about 4 ml. conc. HCl acid and then add 10 ml. standard K2Cr2O7 solutions by means of a pipette in the same flask and cover it with a watch glass, allow the solution to stand for about five minutes in the dark (inside the desk). Rinse the watch glass and dilute the solution about 100 ml. titrate the liberated iodine with sodium thiosulphate solution from burette until the brown color fades (light yellow). Add about 1 ml starch solution and continue titration by adding sodium thiosulphate solution from the burette until one drop of sodium thiosulphate solution changes the color of the solution from deep blue to light blue. This is the end point. Calculate the strength of sodium thiosulphate solution using the following equation:

V1×S1= V2×S2

**Data:-**

Standardization of Na2S2O3 solution with standard K2Cr2O7 solution

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| No. of obs. | Volume  of  K2Cr2O7  (ml) | Burette Reading (ml) | | Volume of Na2S2O3  (ml) | Average  (Volume of Na2S2O3)  (ml) |
| Initial | Final |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

**Calculation:-**

We know, Vred × Sred = Vox × Sox

Here,

VK2Cr2O7 = ml , SK2Cr2O7 = N

VNa2S2O4 = ml SNa2S2O4 =?

SNa2S2O4 = ( × )/

**Result:**-

Determined strength of Na2S2O3 solution is = N

**Discussion:-**

As the color change of the titration of Na2S2O3 with K2Cr2O7 is very confusing, the end point of the titration may not have been properly determined. This may be the cause of error.

**Student should know:**

1. How does a redox indicator work?
2. What is the difference between acid-base and redox indicator?
3. Can you identify whether KI, K2Cr2O7, NaHCO3 are primary or secondary substance?
4. Can you use any other chemicals instead of NaHCO3?
5. Why you should cover the conical flask after adding NaHCO3?
6. Why it is necessary to keep your experimental solution in the dark?
7. Why you observe brown color for liberation of iodine instead of deep violet color?
8. How do you say which oxidizing / reducing agent stronger or weaker than other?
9. What is the function of starch and why is the necessary to add starch just before the end point of the titration?
10. **Estimation of Copper contained in a supplied solution by Iodometric method**.

**Objectives:**

1. To study the amount of copper present in copper salt solution.

2. To study oxidation reduction titration.

3. To study the percentage of copper present.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength and amount of copper with the standard dichromate solution

& liberated iodine.

1. Observe the end point by color change with starch and NH4CNS indicator.
2. To get the idea of percentage of purity a substance.

**Theory:**—

In this experiment, the amount of copper in a copper salt solution is determined. The reaction that occurs here is oxidation and reduction reaction as well as iodometric reaction. Titration involving with iodine or dealing with iodine liberated in chemical reaction is called Iodimetric and Iodometric titration respectively. This reaction is iodometric because iodine is got from KI.

The reaction takes place in two steps:

1. Cu2++ KI + H+ = I2 +……

2. Na2S2O3 + I2 = Na2S4O6 + ……

In the 2nd Step of the reaction a specific indicator is used that is “Starch”- which has a significant characteristic on iodine.

Here, for the first part of the experiment, the basis of the volumetric measurement is that one equivalent weight of an oxidizing agent will completely react with one equivalent weight of a reducing agent.

To perform the second part of the experiment, we can follow the process discussed below:-

Since one atom of iodine is set free for each atom of copper 1000 ml (1M) Na2S2O3 solution should be equivalent to one gram molecular weight that is 63.54 gram of copper.

Therefore, 1 ml of (1M) Na2S2O3 ≡ 0.06354 gram of Copper (Cu+2 )

V ml (SM) Na2S2O3 estimated = (VS/1000)\* 63.54 g Cu+2

So, 10 ml supplied solution contained ………………… g Cu+2 ion.

1000 ml supplied solution conatained …………….. = g Cu+2 ion

So, the weight of copper in the supplied solution may then be easily calculated using the above relation.

**Apparatus:**—

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

**Name of the chemicals used**:—

1. Na2S2O3 solution

2. K2Cr2O7 solution

3. KI solution

4. NaHCO3

5. HCl solution (concentrated)

6. Distilled water

7. CuSO4 solution

8. CH3COOH solution

9. NH4CNS solution

10. Starch (Indicator)

**Procedure:**

Standardization of Sodium Thiosulphate solution as experiment no. 04

Pipette out 10 ml of supplied copper salt solution into a cornical flask. Add a few drop of dil. NaOH or NaHCO3 or Na2CO3. A pale greenish precipate should appear. Dissolve the precipate by adding few drop of acetic acid (CH3COOH). Add about 10 ml of 10% potassium iodide (KI) solution and titrate the liberated iodine against the standard thiosulphate solution (standardized previously) until the brown color of iodine changes to light yellow. Add 1 ml of starch solution and continue titration till the blue color begins to fade. Now add few drops of 10% Ammonium thiocyanate solution and continue titration until the blue color is just discharged. Calculate the amount of copper present in one liter of the supplied solution.

**Data:—**

TABLE: 1

(Standardization of Sodium Thiosulphate solution with standard potassium Dichromate Solution)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of observation | Volume of K2Cr2O7  (ml) | Burette | Reading (ml) | Volume of Na2S2O3  (ml) | Average Volume of Na2S2O3  (ml) | Strength of Na2S2O3  (M) |
| Initial Reading | Final Reading |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |

TABLE: 2

(Standardization of Sodium Thiosulphate solution with standard potassium Dichromate Solution)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of observation | Volume of Cu2+ Solution  (ml) | Burette | Reading (ml) | Volume of Na2S2O3  (ml) | Average Volume of Na2S2O3  (ml) | Strength of Cu2+ Solution  (M) |
| InitialReading | Final Reading |
| 1 |  |  |  |  |  |  |
| 2. |  |  |  |  |  |  |
| 3 |  |  |  |  |  |  |

**Calculation**:—

from Vred × Sred = Vox × Sox

Here,

VK2Cr2O7 =

SK2Cr2O7 =

VNa2S2O3 =

SNa2S2O3 =

**Result:—**

The amount of copper (in gm) present in the supplied CuSO4 solution (per litre) is : Wcu = gm.

Known amount of copper = gm

Observed amount of copper = gm

Percentage of error = %

**Discussion:**—

The following may be the possible cause of error:

There might have been a little mistake in determining the end point of titration.

**Students should know:**

1. What is the purpose of adding the Na2CO3 and CH3COOH solution?
2. What is desirable pH for the reaction between Cu2+ and KI?
3. What is the purpose of during NH4SCN solution?
4. Do you know why most of the copper salts have blue color?
5. **Determination of Ferrous Iron (Mohr’s Salt) by Standard Potassium Dichromate Solution**

**Objectives:**

1. To study the amount of Ferrous Iron present in salt solution.

2. To study oxidation reduction titration.

3. To study the percentage of Iron present.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength and amount of Iron with the standard Potassium dichromate solution.
2. Observe the end point by color change with indicator Diphenyl Amine.
3. To get the idea of percentage of purity a substance.

**Theory:—**

In this experiment, the amount of iron in a supplied solution is determined with the help of standard potassium dichromate. The reaction that occurs here is oxidation and reduction reaction. Oxidation-reduction reactions are those in which there is a net change in the oxidation number. Or in other word an oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom). Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom). Reaction involved in this reaction is:

K2Cr2O7 +14H+ + 6Fe2+ ≡ 2K+ + 2Cr3+ + 7 H2O + 6Fe3+

In this reaction, Fe+2 is oxidized into Fe+3. And Cr2O7-2 is reduced to Cr+3.

From the above reaction we get:

1 equivalent weight of K2Cr2O7 ≡ 1 equivalent weight of Fe+2

1000 ml (1 M) K2Cr2O7 solution ≡1000 ml (1M) ferrous iron solution

≡ 1 atomic weight of iron

So, 1000 ml (1M) K2Cr2O7 solution should be equivalent to one gram atomic weight that is 55.855 gram of iron.

Therefore, 1 ml of (1M) K2Cr2O7 ≡ 0.05585 gram of iron

So, the weight of iron in the supplied solution may then be easily calculated using the above relation.

**INDICATOR AND WHY WE USED:**

In this experiment di-phenyl amine is used. A serious limitation to the use of K2Cr2O7 as a titrant is that the end point in the resultant green chromic ion solution is not easily detected. This limitation has been overcome by the use of an internal oxidation reduction indicator and of the potentiometric titration method for detecting the end point. This indicator may be used as internal indicator to determine the end point in titration with dichromate. It is oxidized less readily than ferrous ion. So for proper detection of the end point di-phenyl amine is used here as an indicator.

With this indicator, phosphoric acid must be added in the solution. Because it lowers the reduction potential of the ferrous-ferric system. The Reduction Potential of di-phenyl benzidine is 0.76 volt and the Reduction Potential of ferrous ion is 0.77 volt. Although this potential difference is quite low, but this low difference can cause significant error in determining the weight of ferrous ion. To reduce this potential difference for getting proper result, phosphoric acid is used. So that the two potential can almost coincide. Phosphoric acid lowers the difference between two reduction potential by forming a complex with the ferric ion which is [Fe(HPO3)]+.

**Apparatus:—**

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

**Name of the chemicals used:—**

1. Mohr’s Salt (NH4)2SO4.FeSO4.6H2O

2. H2SO4 solution (5%)

3. H3PO4 solution (concentrated)

4. Diphenyl Amine Indicator (C6H5)2NH

5. K2Cr2O7 solution

6. Distilled water

**Procedure:-**

**Step-1: (Preparation of 100 ml 0.02M K2Cr2O7 solution)**

1000 ml 1M K2Cr2O7 solution contains = 294 gm

Therefore, 100 ml 0.02M solution contains = =0.588gm

Here, weight taken= ---- gm

Weight to be taken = 0.588 gm

So, the strength of Prepared K2Cr2O7 = × 0.02M

= ------ M

**Step-2: Titration of the supplied Iron Solution against K2Cr2O7 Solution:**

1. Take 10 ml of the supplied iron (Mohr’s salt) solution in a conical flask.

2. Add 50 ml 1 M sulfuric acid and 5 ml conc. of phosphoric acid.

3. Then add 4-5 drops of diphenyl amine indicator and titrate slowly against standard potassium dichromate solution dropwise maintaining an interval of few second between each drop until the addition of one drop causes the formation of intense purpule or violet blue coloration which remains permanent.

4. Record the burette reading in the table below.

5. Repeat the steps 1-4 at least twice

6. Calculate the amount of Iron content in the solution.

7. Calculate the amount of iron per litre of solution.

**Data:—**

TABLE: 1

(Determination of Ferrous Iron by Standard Potassium Dichromate Solution)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume Of  Fe2+ Solution  (ml) | Burette | Reading(ml) | Volume of K2Cr2O7  (ml) | Average  Volume of K2Cr2O7  (ml) |
| Initial  Reading | Final  Reading |
| 1. |  |  |  |  |  |
| 2. |  |  |  |  |  |
| 3 |  |  |  |  |  |

**Calculation:—**

Volume of K2Cr2O7 required to titrate 10 ml of Sample= V ml

1 mole K2Cr2O7= 6 mole of Fe(II)

1000 ml of 1 (M) K2Cr2O7 = 1 mole of F (II)

V ml of S (M) K2Cr2O7 = (vs/1000) mole F(II)

= (vs/1000)\*55.84 gm Fe (II)

10 ml of sample contains = -------- gm Fe(II)

------ ml of sample contains = ------ gm Fe(II)

= ------ gm Fe(II)

Again,

1000 ml (1.0M) K2Cr2O7 = 55.85 gm Fe+2(10 ml)

=> ----- ml K2Cr2O7 = ----- gm Fe+2 (10 ml)

Thus the amount of iron present per litre

= ( × 100) gm

= gm

**Result:—**

The amount of iron (in gm) present in the supplied Fe+2 solutions (per litre) is:

WFe = gm.

**Percentage of error:**

(Known Value – Observed Value) X 100

Percentage of error =

Known value

Known amount of iron = gm

Observed amount of iron = gm

Percentage of error = %

**Precautions: —**

At first the burette was cleaned with distilled water carefully; then it was rinsed with rinse solution to avoid the density change of the given solution.

In the same way the pipette was also washed with distilled water and rinsed with the rinse solution.

1. All the apparatus were handled carefully and according to the rules.
2. The pipette was kept on a clean sheet of paper.
3. The key of the burette was operated carefully to make sure that one-drop of. Acid falls into the conical flask at a time.
4. The conical flask was kept on a white paper to trace the color change of the solution.
5. At first the key of the burette was fully opened to let the solution fall free to make the bubbles go out.
6. If any air bubble enters the burette while pouring acid, air bubble should be removed by dropping out the solution forcibly until the air bubble is out of the burette. Or else, the presence of air bubble will hamper the reading of the volume.
7. Reading might be wrong due to poor eye sight and non-vertical readings. The readings must be taken vertically considering the lower meniscus of the concave surface of the liquid.

**Discussion:—**

This experiment is performed in order to determine the ferrous iron by standard potassium dichromate solution. On the basis of the experiment it was found that K2Cr2O7 is a primary standard substance. We observe that there is slight deviation of the obtained result from the original one. The following factors may be mentioned for this deviation:

1) On the basis of the result it was found that the balance is very much sensitive. So for this reason the result can be varied.

2) While measuring the lower meniscus of the burette an error may be happened for the parallax error. So concentration should be needed here.

3) The phosphoric acid and the sulphuric acid should not be mixed in the solution more than it is needed. If this happened then an error can be taken place. The end point of the titration of the experiment will come later.

4) As the color change is very confusing, the end point of titration may not have been properly determined.

5) Concentrated phosphoric acid must be added in order to lower the difference between two substances’ standard reduction potential. If it is not done then it will hamper to determine the weight of ferrous ion correctly.

6) After pouring any substance in the conical flask with the help of the pipette, if there is any liquid at the tip of the pipette, it should be ignored. But by no means should it be blown out.

7) If the solution is not properly mixed with water then error can occur in determining the weight of ferrous ion. This may be attributed as a reason of error in our experiment.

**Students should know:-**

1. Why it is necessary to use both the sulfuric acid as well as phosphoric acid in the experiment?
2. Do you know the equation corresponding to the color changes of diphenyl amine indicator?
3. What causes the intense purple or violet coloration of the experimental solution?
4. Could you use KMnO4 instead of K2Cr2O7 and what advantage does K2Cr2O7 has over KMnO4?
5. Why the solution shows light bottle green color after addition of K2Cr2O7 and also after the end point is achieved?

1. **Standardization** **of Potassium Permanganate solution with Standard Sodium Oxalate Solution.**

**Objectives:**

1. To study the strength f Potassium Permanganate with standard Sodium Oxalate solution.

2. To study oxidation reduction titration.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength Potasium Permanganate with standard Sodium Oxalate Solution.
2. Observe the end point by color change.

**Theory:—**

In this experiment, potassium permanganate solution is standardizing with the help of standard sodium oxalate solution. The reaction that occurs here is oxidation and reduction reaction. An oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species (or increase of oxidation number of an atom). Reduction is the half reaction in which there is a gain of electrons by a species (or decrease of oxidation number of an atom).

Reaction involved in this reaction is:

MnO4- + 8 H + + 5 e- → Mn +2 + 4 H2O

2 KMnO4 + 5 Na2C2O4 + 8 H2SO4 → K2SO4 + 2 MnSO4 + 5 Na2SO4 + 10 CO2 + 8 H2O

In this reaction, MnO4- is reduced to Mn+2 and Na2C2O4 is oxidized to CO2.

The following equation is used to calculate the strength of Potassium Permanganate:

VA×SA = VB × SB

Here,

VA = Volume of Potassium Permanganate

SA = Strength of Potassium Permanganate

VB = Volume of Sodium Oxalate

SB = Strength of Sodium Oxalate

**INDICATOR AND WHY USED:**

The direct reaction is slow as one can see in a titration. The first few drops of permanganate added to the acidified oxalate solution are not decolorized immediately. Mn+2 ions produced in the reaction acts as a catalyst. They react with permanganate to form intermediate oxidation states of manganese. These states, in turn, react rapidly with oxalate to give the products. So, KMnO4 acts as an auto catalyst in this reaction. This is the advantage of KMnO4 is that it serves as its own indicator, the pink color being distinguishable even if the solution is very dilute. Therefore no indicator is used in this reaction.

**Apparatus:—**

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

7. Beaker

8. Wash Bottle

**Name of the chemicals used:—**

1. Sodium Oxalate Solution (Na2C2O4)

2. H2SO4 solution (1M)

3. Potassium Permanganate Solution (KMnO4)

4. Distilled water

**Procedure:**

Pipette out 10 ml M/10 standard oxalate solution in a conical flask. Add about 50 ml 1 M sulfuric acid solutions to it. Heat the solution up to 60-70ºC and titrate against the Potassium Permanganate solution until the color changes to pink. Calculate the the strength of potassium permanganate from the data.

**Data:—**

TABLE: 1

(Standardization of Potassium Permanganate solution with Standard Sodium Oxalate

solution)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Observation | Volume  of  Na2C2O4  (ml) | Burette | Reading (ml) | Volume of KMnO4  (ml) | Average  Volume of KMnO4  (ml) | Strength  Of  KMnO4  (M) |
| Initial  Reading | Final  Reading |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |

**Calculation:**

In this case weight taken Na2C2O4 g = ?

Weight to be taken Na2C2O4 g = ?

So the strength of KMn2O4 = ( × )/ M

= M

From Vred × Sred = Vox × Sox

Here,

VNa2C2O4 = ml

SNa2C2O4 = M

VKMnO4 = 10.7 ml

SKMnO4 = ?

So,

× = × SKMnO4

=> SKMnO4 = M

**Result:—**

The Determined Strength of Potassium Permanganate Solution is:

SKMnO4 = M

**Percentage of error:**

(Known Value – Observed Value) X 100

Percentage of error =

Known value

Known value = M

Observed amount of iron = M

Percentage of error = %

**Precautions: —**

At first the burette was cleaned with distilled water carefully; then it was rinsed with rinse solution to avoid the density change of the given solution. In the same way the pipette was also washed with distilled water and rinsed with the rinse solution.

1. All the apparatus were handled carefully and according to the rules.
2. The pipette was kept on a clean sheet of paper.
3. The key of the burette was operated carefully to make sure that one-drop of

Permanganate falls into the conical flask at a time.

1. The conical flask was kept on a white paper to trace the color change of the solution.
2. At first the key of the burette was fully opened to let the solution fall free to make the bubbles go out.
3. On the basis of the result it was found that the balance is very much sensitive. So for this reason the result can be varied. While measuring the lower meniscus of the burette an error may be happened for the parallax error. So concentration should be needed here.

**Students should know:-**

1. Why do you don’t have to use any indicator in this experiment?
2. Why you have to heat the experiment solution?
3. Do you know about the effect produced by the change in temperature of a reaction mixture?
4. What happens if you do not shake solution properly?
5. **Determination of Ferrous Ion in a Solution by Standard Potassium Permanganate (KMnO4) Solution.**

**Objectives:**

1. To study the amount of Ferrous Iron present in salt solution.

2. To study oxidation reduction titration.

3. To study the percentage of Iron present.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength and amount of Iron with the standard Potassium permanganate solution.
2. Observe the end point by color change without indicator.
3. To get the idea of percentage of purity a substance.

**Theory:**—

In this experiment, the amount of iron in a supplied solution is determined. The reaction that occurs here is oxidation and reduction reaction. An oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. Oxidation is the half reaction in which there is loss of electrons by a species. And reduction is the half reaction in which there is a gain of electrons by a species.

Reaction involved in this reaction is:

KMnO4 + 5Fe2+ ≡ K+ + Mn2+ + 4H2O + 5Fe3+

In this reaction, Fe+2 is oxidized into Fe+3. And MnO4-2 is reduced to Mn+2.

From the above reaction we get:

1equivalent weight of KMnO4 ≡ 1 equivalent weight of Fe+2

1000 ml (N) KMnO4 solution ≡ 1000 ml (N) ferrous iron solution

≡ 1 atomic weight of iron

So, 1000 ml (N) KMnO4 solution should be equivalent to one gram atomic weight that is 55.855 gram of iron. Therefore,

1. ml of (N) KMnO4  ≡ 0.5585 gram of iron

So, the weight of iron in the supplied solution may then be easily calculated using the above relation.

**Apparatus:—**

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

**Name of the chemicals used:—**

1. Sodium Oxalate (Na2C2O4)

2. Mohr’s Salt ((NH4)2SO4.FeSO4.6H2O)

3. H2SO4 solution (2N)

4. KMnO4 solution

5. Distilled water

**Procedure:-**

N.B. standardizes KMnO4 with sodium oxalate solution as Expt. No. 7.

Pipette out 10 ml of iron solution (Mohr’s salt) in a conical flask. Add about 6 ml of 1M sulfuric acid solutions. Dilute it to about 50 ml distill water. Titrate the resulting solution with standard potassium permanganent solution. The end point is indicated by the first appearance of permanent light pink color as in Expt. No.7. Repeat the titration at least thrice. Calculate the amount of iron of the supplied solution.

**Data:—**

TABLE: 1

(Standardization of Potassium Permanganate solution with Standard Sodium Oxalate solution)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| No. of obs. | Volume  of  Na2C2O4  Solution (ml) | Burette | Reading (ml) | Volume of KMnO4  (ml) | Average  Volume of KMnO4  (ml) |
| Initial | Final |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

TABLE: 2

(Determination of Ferrous Iron by Standard Potassium Permanganate Solution)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| No. of obs. | Volume  Of Mohr’s salt (ml) | Burette | Reading (ml) | Volume of KMnO4  (ml) | Average  Volume of KMnO4  (ml) |
| Initial | Final |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

**Calculation:—**

from Vred × Sred = Vox × Sox

Here,

VKMnO4 = ml

SNa2C2O4 = M

VNa2C2O4 = ml

SNa2C2O4 = M

Again,

1000 ml (1.0N) KMnO4 = 55.855 gm Fe+2(10 ml)

=> ml N KMnO4 = gm Fe+2 (10 ml)

Thus the amount of iron present per litre= ( X 100 ) gm

= gm

**Result:—**

The amount of iron (in gm) present in the supplied Fe+2solution (per litre) is:

WFe = gm.

**Percentage of error:**

(Known Value – Observed Value) X 100

Percentage of error =

Known value

Known amount of iron = gm

Observed amount of iron = gm

Percentage of error = ( – )/ ) X 100 %

= %

**Discussion:—**

The following causes may be mentioned for the slight deviation of the result from the original one:

1) As the color change of the titration of KMnO4 with Na2C2O4 is very confusing, the end point of the titration may not have been properly determined.

2) As the color change is very confusing, the end point of the titration may not have been properly determined. During titration one or two extra drop of KMnO4 might have been added to the Na2C2O4 solution after the end point.

**Student should know:**

* 1. What would happen if you use HCl or HNO3 acid instead of H2SO4?
  2. What are the disadvantages of using KMnO4?

1. **Determination of Calcium in a Sample of Calcium Carbonate**

**Objectives:**

1. To study the amount of Calcium present in salt solution.

2. To study oxidation reduction titration.

3. To study the percentage of Calcium present.

**Learning Outcome:** After completing this experiment the students will be able to:

1. Determine the strength and amount of Calcium the standard Potassium dichromate solution.
2. Observe the end point by color change with indicator Diphenyl Amine.
3. To get the idea of percentage of purity a substance.

**Theory:—**

The amount of calcium in a supplied sample of calcium carbonate is determined in this experiment. The reaction that occurs here is oxidation and reduction reaction.

An oxidation-reduction reaction (redox) is a reaction in which electrons are transferred between species or in which atoms change oxidation numbers. ‘Oxidation’ is the half reaction in which there is loss of electrons by a species. And ‘Reduction’ is the half reaction in which there is a gain of electrons by a species (either an atom or an ion).

The reactions involved in this experiment are:

CaCO3 + 2HCl → CaCl2 + H2O + CO2

CaCl2 + (NH4)2C2O4 → CaC2O4 + 2NH4Cl

CaC2O4 +H2SO4 → H2C2O4 + CaSO4

5H2C2O4 + 2KMnO4 +3H2SO4 →K2SO4 + 2MnSO4 + 10CO2 + 8H2O

In this experiment, KMnO4 is standardized with the help of standard sodium oxalate solution. The strength of KMnO4 is determined by the following equation:

Vacid × Sacid = Vbase × Sbase

Where, Vacid = volume of acid

Sacid = strength of acid

Vbase = volume of base

Sbase = strength of base

Calcium carbonate is dissolved in acid and then it is precipitated as calcium oxalate. In order to determine the weight of Calcium, the following equation is used:

1 equivalent weight of KMnO4 ≡ 1 equivalent weight of Oxalic Acid

≡ 1 equivalent weight of CaC2O4

≡ 1 equivalent weight of Ca2+

1000 ml (N) KMnO4 solution ≡ 1000 ml (N) calcium

≡ 1 equivalent weight of calcium

≡ 20 gm of Ca+2

So, 1000 ml (N) KMnO4 solution should be equivalent to one gram equivalent weight that is 20 gram of calcium. Therefore,

1 ml of (N) KMnO4  ≡ 0.02 gram of calcium

So, the weight of calcium in the supplied calcium carbonate may then be easily calculated using the above relation.

**Apparatus:—**

1. Conical flask

2. Burette

3. Pipette

4. Volumetric flask

5. Stand

6. Funnel

**Name of the chemicals used:—**

1. Calcium Carbonate (CaCO3)

2. Ammonium Oxalate ((NH4)2C2O4)

3. Sodium Oxalate (Na2C2O4)

4. H2SO4 solution

5. HCl solution

6. KMnO4 solution

7. NH3 solution (1:1)

8. Distilled water

**Procedure:-**

Take about 0.1 g calcium salt in a 250 beaker. Add about 10 ml distilled water and 4-10 ml of 6 M HCl acid slowly by means of a measuring cylinder to prepare a clear solution. Warm the solution to drive off CO2. Wash the watch glass and walls of the beaker with distilled water and dilute to about 15 ml. Heat to 60-80 0C and add 6% 50 ml of warm solution of ammonium oxalate. If ammonium oxalate solution is not clear, filter before using it. Add 3-4 drops of methyl orange indicator. Then introduce ammonium solution (1:1) dropwise till the color changes from red to yellow. Allow the solution to stand for about 10-15 minutes to settle the precipitate. Filter the solution through an ordinary filter paper, wash the beaker and precipitated with distilled water and check chloride with help of silver nitrate. When it comes free from chloride then dissolved the precipitate in 50 ml of 3 M H2SO4 in a beaker. Heat it to 80-90 0C if necessary. Transfer to cool solution to a 250 ml volumetric flask and make the volume up to the mark by adding distilled water. Standardize supplied KMnO4 solution with standard 0.0M N sodium oxalate solution as in Expt. No. 7. Take 20 ml of calcium solution and titrate it with standard KMnO4 solution. Heat the solution up to 60-80 0C. Take the first pink colors that persist for 1-20 second as an end point.

**Data:—**

TABLE: 1

(Standardization of Potassium Permanganate solution with Standard Sodium Oxalate solution)

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Number of  Obs. | Volume of  Na2C2O4  (ml) | Burette reading (ml) | | Volume of KMnO4  (ml) | Average  Volume of KMnO4  (ml) | Strength  Of  KMnO4  (M) |
| Initial  Reading | Final  Reading |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |  |
|  |  |  |  |  |  |

TABLE: 2

(Determination of Calcium Ion by Standard Potassium Permanganate Solution)

|  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- |
| Number of  Obs. | Volume Of  Fe2+  Solution  (ml) | Burette reading (ml) | | Volume of KMnO4  (ml) | Average  Volume of KMnO4 (ml) |
| Initial  Reading | Final  Reading |
|  |  |  |  |  |  |
|  |  |  |  |  |  |
|  |  |  |  |  |  |

**Calculation:—**

from Vred × Sred = V­ox × Sox

Here, VKMnO4 = ml

SNa2C2O4 = M

VNa2C2O4 = ml

SK2Cr2O7 = M

Again,

1000 ml (1.0N) KMnO4 = 20 gm Ca+2(20 ml)

=> ml N KMnO4 = gm Ca+2 (20 ml)

Thus the amount of iron present in 250 ml= (( × 250) / 20) gm

= gm

100 gm CaCO3 contains 40 gm Ca+2

So gm CaCO3 contains gm Ca+2

**Result:—**

The amount of calcium (in gm) present in the supplied calcium carbonate solution (per 250 ml) is:

WCa = gm.

**Percentage of error:**

(Known Value – Observed Value) X 100

Percentage of error =

Known value

Known amount of calcium = gm

Observed amount of iron = gm

Percentage of error = (( – )/ )× 100 %

= %

**Discussion:—**

The following causes may be mentioned as the causes for errors:

1. As the calcium solution was not given enough time to precipitate, all the Calcium might not have been precipitated as Calcium Oxalate.
2. May be it was not possible to separate all the calcium precipitate from the filter paper.
3. As the color change was confusing, the end point of titration of KMnO4 with Na2C2O4, might not have been properly determined.

**Students should know:-**

* 1. What is the function of ammonium oxalate?
  2. Could you use phenolphthalein here instead of methyl orange indicator?
  3. What would happen if you do not use 1:1 ammonium solution?
  4. Why the solution is need to be heated to 60-80 0C?