Volumetric Analysis

General Procedures

1.Preparation of standard solution

*  Place a clean and dry weighing bottle on the electronic balance and tare it.
*  Weigh out 1.00 g of NaOH.
*  Transfer the weighed substance into a small clean beaker.
*  Rinse the weighing bottle with distilled water and pour the washings into the beaker. Do this step at  least twice.
*  Stir gently to dissolve as much of the substance as possible, and pour the concentrated solution into  a clean 250 cm3 graduated flask using a funnel
*  Rinse the beaker a few times and add the washings to the graduated flask.
*  Remove the funnel and make up to the mark with distilled water **using a dropper for the last few**  **drops**.
*  Mix the solution thoroughly by shaking and inverting the flask.

Mass used for preparation of standard solution

* preparing a 250 cm3 standard solution of 0.100 mol dm‐3 NaOH
* n(NaOH) required = 0.100 X(250 / 1000) = 0.0250 mol
* m(NaOH) required = 0.0250  (23.0 + 16.0 + 1.0) = 1.00 g

2.If given a solution but too concentrated, you only dilute it tenfolded

  Using a burette/pipette, add 25.00/25.0 cm3 of FA1 into a 250 cm3 volumetric flask.

* Make up to the mark with distilled water using a dropper for the last few drops

  Stopper the flask and shake thoroughly.

3.If given stock solution:

* from a burette, add an appropriate volume of the stock solution into a graduated flask.(cv=cv)
* Make up to the mark with distilled water using a dropper for the last few drops
* Mix the solution thoroughly by shaking and inverting the flask.

Titration

*  Pipette 25.0 cm3 of ethanedioic acid solution and place in a conical flask.
*  Add 1 to 3 drops of phenolphthalein to the conical flask.
* From a burette, gradually add the standard NaOH solution, swirling the conical flask continuously to  ensure mixing.
* The end‐point is indicated by a sharp and permanent pale pink colour.
*  Repeat the titration as many times necessary to achieve two consistent readings e.g. both readings  within 0.10 cm3.
* Double indicator titration
* 1. Procedure
*   Using a pipette, transfer 25.0 cm3 of **F1** into a conical flask.
*   Add two drops of phenolphthalein into the conical flask.
*   From a burette, gradually add HCl into the conical flask until the pink colour is discharged.
*   Record the 1st end‐point volume in the table below.
*   Add two drops of methyl orange into the conical flask.
*   From the same burette, continue adding HCl into the conical flask until the solution turns orange.
*   Record the 2nd end‐point volume in the table below.
*   Repeat the titration as many times as necessary to achieve two consistent readings e.g. both  readings within 0.10 cm3.

|  |  |  |  |
| --- | --- | --- | --- |
|  | Rough | 1st | 2nd |
| Final burette reading for 1st end point / cm3 |  | B |  |
| Final burette reading for 2nd end point / cm3 |  | C |  |
| Initial burette reading / cm3 |  | A |  |
| Volume of titrant added for 1st end point / cm3 |  | B– A = X |  |
| Volume of titrant added for 2nd end point / cm3 |  | C– A = Y |  |

2.Show how you can use the experimental results to obtain the percentage of sodium carbonate present.

1 NaOH + HCl 🡪 NaCl + H2O

* 2a  Na2CO3 + HC*l* 🡪 NaHCO3 + NaC*l*
* 2b  NaHCO3 +HC*l* 🡪 NaC*l*+H2O+CO2

First end point (1+2a)

Second end point (2b)

Let volume of HCl for first end‐point be **X** cm3

Let volume of HCl for second end‐point be **Y** cm3

Volume of HCl reacted with NaHCO3 in step 2b = (**Y – X**) cm3

n(NaHCO3) = 0.100 (**Y – X**) mol = n(Na2CO3)

m(Na2CO3) present in 25.0 cm3 = n(Na2CO3)x (23.0 x2 + 12.0 + 16.0x 3) = **W** g

m(Na2CO3) present in 1 dm3 = (1000/25.0)  **W** = **Z** g

%Na2CO3 = 100 x(**Z** / 5)

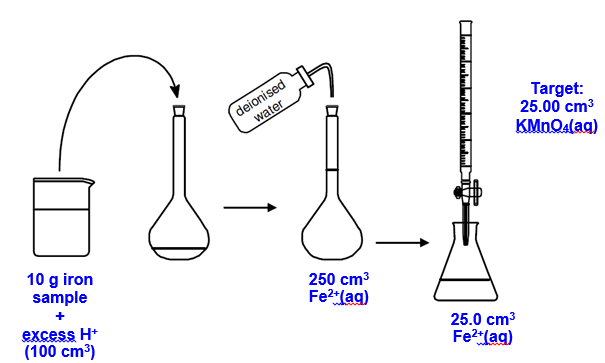
* 3.Suggest an alternative method to accurately measure the end point volumes.Sketch
* an appropriate diagram to illustrate how to obtain the end point volumes.
* Use a calibrated pH meter connected to a data logger and plot the pH changes throughout the titration.

Redox titrations

* 1. Titration procedure
* Pipette 25.0 cm3 of the solution into a 250 cm3 conical flask.
* Using a 10 cm3 measuring cylinder, add 10 cm3 of H2SO4 into the conical flask. (The acid here acts as  an acidic medium for the redox reaction. The volume quoted here is in excess. You may wish to calculate out the minimum volume required but it is not necessary)
* Titrate the contents of the conical flask with KMnO4 from a burette until the solution turns orange.
* Repeat the titration as many times necessary to achieve two consistent readings e.g. both readings  within 0.10 cm3.

2. Explain why the mixture should be filtered before titration against the oxidising agent.

* The filtration step is to ensure that impurities like Cu (which can be oxidised by KMnO4) are removed before titration.
* 3.TEST PURITY OF IRON(CLAIMED 98PERCENT)

Standard solution always made for conical flask thingy(iron)

Need to make sure conical flask thing and burette thing all volume is like 25 cm3( if info about conc. of burette thing not given , need to ….)

Concentration of KMnO4 solution used in burette Assumption:

* 250 cm3 volumetric flask is used for preparing the iron/H2SO4 solution (alternatively 100 cm3 flask may be used)
* Analyte volume used is 25.0 cm3
* Target endpoint volu b me is 25.00 cm3
* Calculation
* n(Fe2+) in 250 cm3 of solution = 0.179 mol (from above calculations)
* n(Fe2+) in 25.0 cm3 of analyte = 0.179 / 10 = 0.0179 mol
* 5Fe2+ + MnO4 + 8H+ 🡪 Mn2+ + 4H2O + 5Fe3+
* n(MnO4) = 0.0179 / 5 = 0.00358 mol
* c(MnO4) = 0.0358 / 0.025 = 0.143 mol dm‐3
* Appropriate concentration of KMnO4 is 0.140 mol dm‐3 such that the endpoint volume is around 25.00 cm3.

4. Show how to use your titration results to calculate the % of iron in the sample.

Let endpoint volume be **VOA**

Amount of KMnO4, **n(KMnO4)** = 0.140  **VOA** mol

 Amount of Fe present in 25.0 cm3 sample = 5 **n(KMnO4)** mol

Amount of Fe present in 250 cm3, **n(Fe)** = (250/25.0)  5 **n(KMnO4)** mol

Mass of Fe present, **m(Fe)** = 55.8  **n(Fe)** g

Let mass of iron wire be **mwire**

Percentage purity = 100 **m(Fe)** / **mwire**

**Precaution**

**Read yourself**

Iodometric titration

Na2S2O3 2-: burette thing

Starch : indicator

I2 + 2S2O32🡪 2I + S4O62

How I2 is obtained? I- oxidized by oxidizing agent

Write here all the eqn

1.What is observed when excess potassium iodide is added to **FA1**? Write an equation to support your answer.

* 2Cu2+ + 4I 🡪Cu2I2 + I2
* A white precipitate in yellow brown solution is observed.
* 2. Suggest a reason for the dilution of **FA1** in the first step.
* Too concentrated. Volume of titrant may exceed 50 cm3 which is the maximum capacity of a standard burette.
* How you dilute tenfold:
*   Using a burette/pipette, add 25.00/25.0 cm3 of FA1 into a 250 cm3 volumetric flask.
*   Make up the contents of the volumetric flask with distilled water.
*   Stopper the flask and shake thoroughly.
* 3.Procedure
*   Using a burette/pipette, add 25.00/25.0 cm3 of FA1 into a 250 cm3 volumetric flask.
*   Make up the contents of the volumetric flask with distilled water.
*   Stopper the flask and shake thoroughly.
*   Pipette 25.0 cm3 of the diluted solution and place in a conical flask.
*   Using a measuring cylinder, add 50 cm3 of excess potassium iodide to the conical flask. (volume is an estimate)
*   From a burette, gradually added sodium thiosulfate to the conical flask, swirling continuously, until a pale yellow colour is observed.
*   Add ten drops of starch solution to the conical flask.
*   Continue titrating until the dark blue colour is discharged.
*   Repeat the titration as many times necessary to achieve two consistent readings e.g. both  readings within 0.10 cm3.
* 4.Titration procedure
* Pipette 25.0 cm3 of the diluted solution and place in a conical flask.
*   Using a measuring cylinder, add 50 cm3 of excess potassium iodide to the conical flask. (volume is an estimate)
*   From a burette, gradually added sodium thiosulfate to the conical flask, swirling continuously, until a pale yellow colour is observed.
*   Add ten drops of starch solution to the conical flask.
*   Continue titrating until the dark blue colour is discharged.
*   Repeat the titration as many times necessary to achieve two consistent readings e.g. both  readings within 0.10 cm3.

5. Find percentage by mass of water of crystallization of salt

Let volume of Na2S2O3 used be **VRA** dm3

2Cu2+ + 4I 🡪Cu2I2 + I2

I2 + 2S2O32🡪 2I + S4O62

n(Cu2+) : n(S2O32)

1:1

**n(Cu2+)** = 0.100 x**VRA** mol

**c(salt)** = **n(Cu2+)** / 0.0250 mol dm‐3

**Mr (salt)** = 200 / **c(salt)**

**Mr of water of crystallization** = **Mr (salt)**  159.6

% = 100 (**Mr of water of crystallization** / **Mr (salt)**

**precaution read yourself**

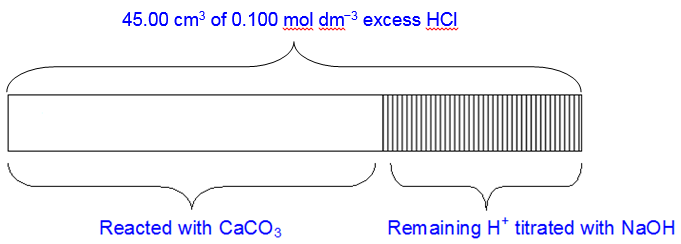
Back titration

1.principle

**As CaCO3 is in solid form, it cannot be titrated directly against HCl. A back titration is necessary**

CaCO3 + 2HCl 🡪 CaCl2 + CO2 + H2O

HCl + NaOH 🡪 NaCl + H2O



Assumption:

*   Analyte vol is 25.0 cm3, target endpt vol is 25.00 cm3  (就是为了差不多，实验好做一点，没什么意思)
*   250 cm3 volumetric flask used to prepare limestone + HCl soln
*   Use 1.00 g of limestone (You may choose to use any amount of mass except 5.00 g. Choose any mass  less than 5.00 g)

2.Calculation to prepare:

* Since [NaOH] is 0.100 mol dm‐3,
* for endpoint to be 25.00 cm3,
* Conc of remaining HCl = 0.100 mol dm‐3
* n(HCl) remaining in 25.0 cm3 =0.100 × 0.0250 = 0.00250 mol
* n(HCl) remaining in 250 cm3 = 0.00250 × 10 = 0.0250 mol
* n(CaCO3) present in 1.00 g = 1.00 / 100.1 = 0.00999 mol
* n(HCl) used to react with CaCO3 = 0.00999 × 2 = 0.01998 mol
* Total n(HCl) present in 250 cm3 = 0.01998 + 0.0250 = 0.04498 mol
* V(HCl) needed = 0.04498 / 1.00 = 0.04498 dm3 = 45.0 cm3

Hence 45.00 cm3 of HCl should be added to 1.00 g of limestone so as to achieve end‐point of approximately 25.00 cm3.

* 称出来想查的procedure（竟然不是冲weighing bottle,不知道为啥）
* Using a clean and dry weighing bottle, measure accurately 1.00 g of limestone on an electronic balance. Do not TARE.
* Tip the contents of the weighing bottle into a 250 cm3 beaker.
* Re‐weigh the empty weighing bottle (with residue).

|  |  |
| --- | --- |
| Mass of weighing bottle + contents / g | A |
| Mass of empty weighing bottle /g | C |
| Mass of weighing bottle with residue / g | B |
| Mass of contents / g | A–B |

3.Titration procedure:

  With a burette, add 45.00 cm3 of HCl into the beaker.

  Stir the beaker with a glass rod to ensure that the limestone has completely reacted.

  Using a funnel, transfer the contents into a 250 cm3 graduated flask (using the glass rod to guide the  flow of the solution).

  Rinse the beaker with distilled water and pour the washings into the flask. Repeat this at least twice.

  Remove the funnel and make up to the mark with distilled water.

  Mix the solution thoroughly by shaking and inverting the flask.

  Pipette 25.0 cm3 of the solution into a 250 cm3 conical flask.

  Add 2 – 3 drops of methyl orange to the conical flask.

  Titrate the contents of the conical flask with NaOH from a burette until the solution turns orange.

  Repeat the titration as many times necessary to achieve two consistent readings e.g. both readings  within 0.10 cm3.

4.Calculation for the purity of limestone CaCO3

Let volume of NaOH be **VNaOH**

Amount of NaOH, **n(NaOH)** = **VNaOH** x0.100 mol

Amount of unreacted HCl in 25.0 cm 3 = **n(NaOH)** mol

 Amount of unreacted HCl in 250 cm 3, **n(HCl unreacted)** = (250 / 25.0) x **n(NaOH)** mol

Original amount of HCl = (45.00/1000)(1.00) = 0.0450 mol

Amount of HCl reacted with carbonate, **n(HCl reacted)** = 0.450 – **n(HCl unreacted)** mol

Amount of CaCO3 present, **n(CaCO3)** = 1⁄2 **n(HCl reacted)** mol

Mass of CaCO3 present, **m(CaCO3)** = **n(CaCO3)x** 100.1 g

Let mass of limestone be **mlimestone** Percentage purity = 100 **m(CaCO3)** / **mlimestone**

Inaccuracies

1.What are the inaccuracies that can arise while preparing the standard solution of NaOH?

* NaOH pellets are highly deliquescent and will dissolve rapidly when exposed to water vapour in the air, hence the mass weighed may be lower than expected.
* The sodium hydroxide solution in step 4 is highly concentrated and CO2 from the air is likely to dissolve into the solution, hence concentration of NaOH may be lower than expected.  Improvement  It is necessary to standardize the resulting solution of NaOH with a standard solution of KHP or HCl  before titrating with ethanedioic acid. Or
* Weigh the pellets quickly and capping the weighing bottle at all times.
* 2.Name two inaccuracies that can arise during the titration.
* (double indicator titration)
*   End point change of phenolphthalein is not distinct enough

  Any errors made in the titration of the first end‐point affects the accuracy of the 2nd end‐point.

Precautions

1. certain acids, bases or oxidizing agents may be corrosive. Wear gloves while handling such chemicals.
2. Fill up the burette below eye level so that chemicals do not accidentally spill and enter the eye.
3. In iodometric titrations, the iodine vapour liberated is irritating to the eyes. Pour away the chemicals immediately after the experiment.

Accuracy

1. apparatus

Measuring cylinder: 0 dp of cm3

Pipette: 1 dp of cm3

Burette: 2 dp of cm3( nearest 0.05cm3)

1. Mr 1dp
2. Percentage error:error/experimental data x 100%

Pipette:0.03cm3

Burette:0.05x2cm3

Measuring cylinder:0.1cm3

4. example on percentage error qn

Summary Of Titration Procedure

Redox Double indicator Iodometric Back Titration Acid-base

1. Pipette 25.0 cm3 of the solution into a 250 cm3 conical flask.
2. Add whatever

3.From a burette, gradually add \_\_\_ to the conical flask, swirling continuously, until a \_\_\_ colour is observed.

1. Repeat the titration as many times necessary to achieve two consistent readings e.g. both readings  within 0.10 cm3.

Redox:

Step 2:

Using a 10 cm3 measuring cylinder, add 10 cm3 of H2SO4 into the conical flask. (The acid here acts as  an acidic medium for the redox reaction. The volume quoted here is in excess. You may wish to calculate out the minimum volume required but it is not necessary)

Acid base

Step 2:

*   Add two drops of phenolphthalein into the conical flask.

Double indicator

From step 4 and onwards:

*  Record the 1st end‐point volume in the table below.

 Add two drops of methyl orange into the conical flask.

 From the same burette, continue adding HCl into the conical flask until the solution turns orange.

*  Record the 2nd end‐point volume in the table below.
*  Repeat the titration as many times as necessary to achieve two consistent readings e.g. both  readings within 0.10 cm3.

Iodometric

Step3 to 3.5

* From a burette, gradually add sodium thiosulfate to the conical flask, swirling continuously, until a pale yellow colour is observed.
* Add ten drops of starch solution to the conical flask.
* Continue titrating until the dark blue colour is discharged.
* Back titration
* Before step1
*   With a burette, add 45.00 cm3 of HCl into the beaker.
*   Stir the beaker with a glass rod to ensure that the limestone has completely reacted.
*   Using a funnel, transfer the contents into a 250 cm3 graduated flask
*   Rinse the beaker with distilled water and pour the washings into the flask. Repeat this at least twice.
*   Remove the funnel and make up to the mark with distilled water.
*   Mix the solution thoroughly by shaking and inverting the flask.

1. When analysing the organic layer, a fixed volume of water (about twice the

volume) is added to the pipetted volume of cyclohexane solution in the conical

flask and shaken vigorously, prior to the titration. Explain the purpose of this

action.

[2]

 NaOH(aq) will not be able to neutralise the ethanoic acid in the organic layer

 The ethanoic acid must be first extracted into the water for complete reaction

with the alkali during neutralization

1. Pb(NO3)2(aq) + 2NaCl(aq) 🡪 PbCl2(s) + 2NaNO3(aq)

