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ACID – BASE TITRATION

Aim: Determination of Alkalinity of water

Theory:

The alkalinity of water is due to presence of hydroxide ion (OH^-), carbonate ion (CO_3^{2-}) and bicarbonate ion (HCO_3^-). Therefore, we have to find out the concentration in ppm of these ions.

In the first step, the water sample is titrated with acid using phenolphthalein as indicator. The color change (pink to colorless) indicates neutralization of hydroxide ion (OH-) to water and / or half neutralization of carbonate ion (CO_3^{2-}) to bicarbonate ion (HCO_3^-).

In the second step, further titration of the above water sample using methyl orange as indicator is carried out. The color change (yellowish to pink) indicates complete neutralization of bicarbonate ion (HCO_3^-) which is formed from (CO_3^{2-}) and / or present in the water sample originally. The concentration of both ions may be calculated from the sets of titration.

<u>1st step</u>

$OH^- + HCl = Cl^- + H_2O$

] Phenolphthalein

$CO_3^{2-} + HCl = HCO_3^- + Cl^-$

<u>2nd step</u>:

$HCO_3^- + HCl = Cl^- + H_2CO_3$] Methyl Orange

The possibility combinations of ions causing alkalinity in water are:

(i) OH^- only, CO_3^{2-} only, HCO_3^- only or
(ii) OH- and CO_3^{2-} , CO_3^{2-} and HCO_3^- together
(iii) The possibility of OH^- and HCO_3^- ions together is very remote since they combine together to form CO_3^{2-} ions.

$$OH^- + HCO_3^- = CO_3^{2-} + H_2O$$

Procedure:

A) Record the room temperature.

B) Preparation of 100 ml (N/50) Na_2CO_3 solution :

Molecular weight of Na_2CO_3 is 106.

Therefore, equivalent weight of Na_2CO_3 is 53.

1000 cc. 1(N) Na_2CO_3 will contain 53 gm Na_2CO_3

100 cc. 1(N/50) Na_2CO_3 will contain 0.106 gm Na_2CO_3

So, to prepare 100 ml (N/50) Na_2CO_3 solution, 0.106 gm Na_2CO_3

C) Standardization of HCl solution:

Pipette out 10 ml sod. Carbonate solution to two conical flask each , add methyl orange indicator and titrate till the color is pink. Now clean the pipette with distilled water.

D) Estimation of HCO_3^- in sample water 1(tap water):

. Collect little **water sample1** supplied and add phenolphthalein, if no pink color is obtained then alkalinity is due to HCO_3^- only. In that case pipette out 10 ml of water sample to two conical flask each, add two drops of methyl orange indicator (the color is yellowish) and titrate till the color is pink.

E) Estimation of CO_3^-, and OH^- in sample water 2:

Collect little **water sample 2** supplied and add phenolphthalein , if pink color is obtained then alkalinity is due to OH^- and CO_3^{2-} (as verified here). In that case pipette out 10 ml of water sample to two conical flask each , add one drop of phenolphthalein indicator and titrate till the color is faint pink or just colorless. Record the volume. Now to this solution add two drops of methyl orange indicator (the color becomes yellowish) and titrate

Calculation on Estimation of Alkalinity of Water

A. Recording of temperature:

TABLE I:

Initial temperature (^{0}c)	Final temperature (^{0}c)	Mean temperature (^{0}c)

B. Preparation of 100 ml (N/50) standard Na_2CO_3 solution:

TABLE II:

weight taken(gm)	Weight to be taken (gm)	Strength (N)
	0.106	

C.Standardization of HCl solution with Na_2CO_3 solution:

TABLE III:

Sl. No.	Na_2CO_3 solution taken	Titration of HCl			
	Volume (ml)	Initial burette reading	Final burette reading	Volume required (ml)	Mean volume (ml)
1	10				
2	10				

D.Determination of alkalinity of water :

TABLE IV: For water sample 1.

Sl. No.	Water sample.	First titration using phenolphthalein				Second titration using methyl orange			
	Vol. (ml)	Initial burette reading	Final burette reading	Vol. required (ml)	Mean volume (ml)	Initial burette reading	Final burette reading	Vol. required (ml)	Mean volume (ml)
1	10	-	-	-	-				
2	10	-	-	-	-				

TABLE V:For water sample 2.

Sl. No.	Water sample	First titration using phenolphthalein				Second titration using methyl orange			
	Vol. (ml)	Initial burette reading	Final burette reading	Vol. required (ml)	Mean volume (ml) = m	Initial burette reading	Final burette reading	Vol. required (ml)	Mean volume (ml) =n
1	10								
2	10								

Calculations:

Strength of Na_2CO_3 = …………(N)

Standardization of HCl solution:

We know,

(V_{HCl} X S_{HCl}) = (V_{Na2CO3} X S_{Na2CO3})

or, S_{HCl} =(V_{Na2CO3} X S_{Na2CO3}) / V_{HCl}

=…………………(N)

Therefore, Strength of HCl =……………..(N)

***ppm** = one litre of water contains how much mg of $CaCO_3$ equivalent.

****Calculation of alkalinity interms of ppm :**

If A ml of sample required B ml of HCl solution of strength W(N) , then

A ml of water sample = B ml of W(N) HCl solution

(1000 ml of 1(N) HCl solution = 1000 ml of 1(N) $CaCO_3$ solution = 1000 ml of one equivalent (50 gm) of $CaCO_3$ solution . Hence ,

1 ml of 1(N) HCl solution = 50/1000 gm of $CaCO_3$ equiv = 50 X 1000/ 1000 = 50 mg of $CaCO_3$ equiv) .

1 ml of 1(N) HCl solution = 50 mg $CaCO_3$ equivalent .

B ml of W(N) HCl solution = B (x)W (x) 50 mg $CaCO_3$ equiv per.

Hence ,

A ml of water sample = B (x)W (x) 50 mg $CaCO_3$ equiv

1 ml of water sample = B (x)W (x) 50 / A mg $CaCO_3$ equiv

1000 ml of water sample = 1000 (x) B (x)W (x) 50 / A mg $CaCO_3$ equiv

Thus alkalinity interms of ppm = 1000 (x) B (x)W (x) 50 / A ppm. = R ppm

In a generalized way

ppm=1000(x)volume of HCl solution (x) strength of HCl solution in normality (x) 50/volumeofwatersample.

The concentration of ions in mg/l = **1000(x)volume of HCl solution (x) strength of HCl solution in normality (x) 50/volumeofwatersample (x) molar mass of ion/ 50.**

i.e concentration of ions in mg/l = R (X) molar mass of ion/ 50.

(50 is the equivalent mass of $CaCO_3$).

Case 1. When phenolphthalein added to water sample the color is not pink that indicates that OH^- and CO_3^{2-} are absent. If addition of methyl orange the color is yellow that is titrated till the color is orange. This indicates HCO_3^- alkalinity.

HCO_3^- alkalinity = …………………. ppm.

Concentration of HCO_3^- = R (x) 61/50 mg/l. =………………….mg/ lit.

Case 2. When phenolphthalein added the color is pink indicates presence of OH^- separately or OH^- and CO_3^{2-} both together. Now after addition of methyl orange if the color is yellow titrate till it to be orange. If volume of HCl solution upto phenolphthalein titration is more than half of titration by use of methyl orange alone (or here half the volume of HCl upto phenolphthalein + Additional volume of methyl orange) will indicate the neutralization of HCO_3^- originated from CO_3^{2-} with reaction of H^+ of added acid. The alkalinity will be due to both OH^- and CO_3^{2-}.

- 1st Titration (phenolphthalein) =Full OH^- +1/2 CO_3^{2-}
- 2nd Titration (methyl orange) =1/2 CO_3^{2-} and if HCO_3^- present in the sample.

From the 2nd titration

Volume of HCl required for **full neutralization** of **HCO_3^-** = n ml =……………ml

Therefore, volume of HCl required for **½ neutralization of CO_3^{2-}** = n ml =……………ml

Thus total volume of HCl required for **complete neutralization ofCO_3^{2-}** = (n+n) ml=2n ml = S ml =…………… ml.

Therefore, volume of HCl required for **total neutralization of OH^-** = (m-n) ml = T ml =………. ml.

(1) **OH^- alkalinity** = ppm

Concentration of OH^- = R (x) 17/50 mg/l. = **mg/ lit.**

(2) **CO_3^{2-} alkalinity** = ppm

Concentration of CO_3^{2-} = R (x) 60/50 mg/l. = **mg/ lit.**

Case 3. For water sample containing other combinations of ions the calculation similarly can be made.

DISCUSSIONS:

Neutralization of CO_3^{2-} proceeds in two steps. In the first step conversion of CO_3^{2-} to HCO_3^- makes the solution alkaline and thus we have used phenolphthalein whose Pk_{in} is 8.6 showing colour change in the P^H region of 9.6 to 10.6 and in the second step there is total neutralization of HCO_3^- leading to formation of H_2CO_3 and the solution becoming acidic and thus methyl orange having Pk_{in} 3.7 shows color change in the p^H region of 2.7 to 4.7.

Thus in order to standardize HCl solution by Na_2CO_3 we have to use methyl orange and not phenolphthalein. Use of phenolphthalein will only gives value for half neutralization of Na_2CO_3 and thus neutralization cannot be done accurately.

For estimation of CO_3^{2-} and OH^- in the mixture, we must avoid using excess phenolphthalein. If we add excess phenolphthalein we may have to use slight excess of HCl solution, this will make the solution slightly acidic. Even if we do not use excess phenolphthalein but do not note the color change carefully, slight excess of HCl solution will also make the solution acidic. Thus our burette reading will show slight excess volume of HCl solution and calculation will be erratic. It is also to be noted that when the solution becomes slightly acidic, addition of methyl orange will immediately show pink coloration and further titration becomes unnecessary and estimation of CO_3^{2-} becomes impossible. Thus titration should be done carefully and methyl orange should be added just when the color is discharged.

For estimation of HCO_3^- one must not use excess methyl orange neither the readings will not be very correct and estimation will not be accurate.

The apparatus should be well cleaned with distilled water prior to the experiment. If it is not done then it may so happen that impurities present in the unclean container will alter the Ph and characteristics of indicator.

CONCLUSION:

The **HCO_3^-** alkalinity of **water sample 1** is …….. ppm and concentration is ……….mg/l.

The **OH^-** alkalinity of **water sample 2** is ……….ppm and concentration ……..mg/l.

The **CO_3^{2-}** alkalinity of **water sample 2** is ………ppm and concentration ……. mg/l.

measured at the temperature of ……….. ^0C.

How to proceed in the laboratory

1. Record the temperature before and after the experiment.
2. Prepare standard sodium carbonate solution.
3. Titrate by supplied HCl solution to know its exact strength ;
Pipette out 10 ml sod. Carbonate solution to two conical flask each , add methyl orange indicator and titrate till the color is pink. Now clean the pipette with distilled water.

4. Collect little **water sample1** supplied and add phenolphthalein , if no pink color is obtained then alkalinity is due to HCO_3^- only. In that case pipette out 10 ml of water sample to two conical flask each , add two drops of methyl orange indicator(the color is yellowish) and titrate till the color is pink.

5. Collect little **water sample 2** supplied and add phenolphthalein , if pink color is obtained then alkalinity is due to OH^- and CO_3^{2-} (as verified here). In that case pipette out 10 ml of water sample to two conical flask each , add one drop of phenolphthalein indicator and titrate till the color is faint pink or just colorless. Record the volume. Now to this solution add two drops of methyl orange indicator (the color becomes yellowish) and titrate till the color become pink. Note down the volume . Repeat with other conical flask containing this 10 ml water sample 2.