Exp. 1

Identification of Group I cations

Group 1

Ag^+, Pb^{2+},Hg_2^{2+}

The reagent of the group is dil. HCl, the ions precipitate as chlorides. The chlorides of other groups are soluble.

Silver Ag^+

Dil. HCl : white ppt. turns to black in the presence of light. The ppt. dissolves in NH_4OH forming the complex $[Ag(NH_3)_2]Cl$.

$AgNO_3 + HCl \rightarrow AgCl\downarrow + HNO_3$

$AgCl + 2NH_4OH \rightarrow [Ag(NH_3)_2]Cl + 2H_2O$

H_2S : passed if gas or added if solution, black ppt., dissolves in hot dil. HNO_3.

$2AgNO_3 + H_2S \rightarrow Ag_2S\downarrow + 2HNO_3$

KI : yellow ppt. doesn't dissolve in acids or ammonia solution.

$AgNO_3 + 2KI \rightarrow AgI\downarrow + KNO_3$

K_2CrO_4 : red ppt., dissolves in dil. HNO_3 and NH_4OH.

$2AgNO_3 + K_2CrO_4 \rightarrow Ag_2CrO_4\downarrow + 2KNO_3$

NaOH : brown ppt., dissolves in dil. HNO_3 and NH_4OH, doesn't dissolve in excess of reagent.

$2AgNO_3 + 2NaOH \rightarrow Ag_2O\downarrow + 2NaNO_3 + H_2O$

NH_4OH : white ppt. of silver hydroxide, turns to black due to the formation of silver oxide. The ppt. dissolves in excess of reagent.

$2Ag^+ + 2NH_3 + H_2O \rightarrow Ag_2O\downarrow + 2NH_4^+$

Lead Pb^{2+}

Dil. Hcl : white ppt., dissolves in hot water, and appears again on cooling.

$Pb(NO_3)_2 + 2HCl \rightarrow PbCl_2\downarrow + 2HNO_3$

H_2S : passed if gas or added if solution, black ppt., dissolves in hot dil. HNO_3. on addition of H_2O_2 white ppt. formed due to oxidation of sulphide.

$Pb(NO_3)_2 + H_2S \rightarrow PbS\downarrow + 2HNO_3$

$PbS + 4H_2O_2\ Pb(NO_3)_2 + PbSO_4\downarrow + 4H_2O$

KI : yellow ppt., dissolves in hot water to a colourless solution, on cooling, the yellow ppt. appears again.

$Pb(NO_3)_2 + 2KI \rightarrow PbI_2\downarrow + 2KNO_3$

K_2CrO_4 : yellow ppt., dissolves in HNO_3 and NaOH.

$Pb(NO_3)_2 + K_2CrO_4 \rightarrow PbCrO_4\downarrow + 2KNO_3$

NaOH : white ppt., dissolves in excess of reagent.

$Pb(NO_3)_2 + 2NaOH \rightarrow Pb(OH)_2\downarrow + 2NaNO_3$

NH_4OH : white ppt. doesn't dissolve in excess of reagent.

$Pb(NO_3)_2 + 2NH_4OH \rightarrow Pb(OH)_2\downarrow + 2NH_4NO_3$

Mercurous Hg_2^{2+}

Dil. Hcl : white ppt. doesn't dissolve in dil. acids or hot water.

$Hg_2(NO_3)_2 + 2HCl \rightarrow Hg_2Cl_2\downarrow + 2HNO_3$

H_2S : passed if gas or added if solution, black ppt. of HgS and Hg.

$Hg_2(NO_3)_2 + H_2S \rightarrow HgS\downarrow + Hg\downarrow + 2HNO_3$

KI : green-yellow ppt., dissolves in excess of reagent forming black ppt. of Hg.

$Hg_2(NO_3)_2 + 2KI \rightarrow Hg_2I_2\downarrow + 2KNO_3$

$Hg_2I_2 + 2KI \rightarrow K_2(HgI_4) + Hg\downarrow$

K_2CrO_4 : brown ppt., turns to red crystals on heating.

$Hg_2(NO_3)_2 + K_2CrO_4 \rightarrow Hg_2CrO_4\downarrow + 2KNO_3$

NaOH : black ppt. doesn't dissolve in excess of reagent, dissolves in dil. HNO_3.

$Hg_2(NO_3)_2 + 2NaOH \rightarrow Hg_2O\downarrow + 2NaNO_3 + H_2O$

NH_4OH : black ppt. of Hg and $HgNH_2Cl$.

$Hg_2Cl_2 + 2NH_4OH \rightarrow HgNH_2Cl\downarrow + Hg\downarrow + NH_4Cl + 2H_2O$

Exp. 2

Preparation and Standardization of HCl Acid

Hydrochloric acid is the most commonly used standard acid for volumetric analysis. Dilute solutions of the reagent are indefinitely stable and can be used in the presence of most cations without complicating precipitation reactions. It is reported that 0.1 N solutions can be boiled for as long as 1 hr without loss of acid.

Solutions of perchloric acid and sulfuric acid are also stable and can serve as standard reagents in titrations where the presence of chloride ion would cause precipitation difficulties. Standard solutions of nitric acid are seldom used because of their oxidizing properties.

A standard acid solution is ordinarily prepared by diluting an appropriate volume of the concentrated reagent and standardizing against a primary standard base. The composition of the concentrated acid is established through careful density measurements, following which a weighed quantity is diluted to an exact volume (tables relating density of reagents to composition are found in most chemistry or chemical engineering handbooks).

Procedure

To prepare 0.1N of an acid like hydrochloric acid (HCl), add about 8 ml of concentrated HCl to approximately 1 liter of distilled water. Mix thoroughly; store in a glass-stoppered bottle.

Sodium carbonate is a frequently used standard for acid solutions. Primary-standard grade sodium carbonate is available commercially.

Dry a quantity of primary-standard sodium carbonate for 2 hr at 110°C, and cool in a desiccator. Prepare 0.1N from sodium carbonate solution, and transfer by a pipette 10 ml in 250 ml conical flask, and introduce 2 drops of methyl orange indicator.

Full a buret with the acid and titrate against the sodium carbonate until the solution just changes from orange to red. Write down the volume of the acid.

The equation of the reaction is:

$$2HCl + Na_2CO_3 \longrightarrow 2NaCl + H_2CO_3$$

no. of the acid milliequivalents = no. of the base milliequivalents

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times \text{(from buret)} = 0.1 \times 10$$

N_1 **=** normality of acid

The strength of HCl solution g/L = $N_1 \times$ equivalent weight (molecular wt./1)

* * *

Exp. 3

Determination of the Percentage of Acetic Acid

The experiment involves the titration of weak acid with a standard solution of a strong base. Thus, an indicator with a basic transition range is required.

The equation of the reaction is:

$$CH_3COOH + NaOH \longrightarrow CH_3COONa + H_2O$$

Procedure

Prepare 0.1N from sodium hydroxide, and full the buret with it. Transfer by a pipette 10 ml from the acid in 250 ml conical flask and introduce 3 drops of ph. Ph indicator.

Titrate until the solution just changes from colorless to pink. Write down the volume of the base.

no. of the acid milliequivalents = no. of the base milliequivalents

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times 10 = 0.1 \times \text{(from buret)}$$

The total acidity of acetic acid in grams per liter = N × equivalent weight (molecular wt./1)

Exp. 4

Analysis of Sodium Carbonate and Sodium Hydroxide Mixture

The analysis of bases mixture is analogous to that for acids. A standard acid solution is used as titrant. The analysis of such mixtures requires two titrations with standard acid. An indicator with a transition in the vicinity of pH 8 to 9 is used for one; an acid-range indicator is used for the other. The composition of the solution can be deduced from the relative volume of acid needed to titrate equal volumes of the sample. Once the composition has been established, the volume data can be used to establish the concentration of each component in the sample.

Sodium carbonate reacts with HCl according to the equation:

$$Na_2CO_3 + 2HCl \longrightarrow 2NaCl + CO_2 + H_2O$$

So to neutralize all the carbonate we need two equivalents from the acid. But when we add one equivalent from the acid to the sodium carbonate, converts to bicarbonate according to the equation:

$$Na_2CO_3 + HCl \longrightarrow NaHCO_3 + NaCl$$

The pH of the solution changes from 11.5 to 8.3, so if we use the ph.ph indicator, its red color changes to colorless by the end of this step, because the change in the pH during this step lies in the same range that the color of the indicator changes (8.3 – 10).

If we use methyl orange indicator, the carbonate reacts totally and needs two equivalents from the acid. The bicarbonate converts to sodium chloride according to the equation:

$$NaHCO_3 + HCl \longrightarrow NaCl + CO_2 + H_2O$$

The pH of the solution changes from (8.3 – 3.8), which is nearly the same range of methyl orange range.

Sodium hydroxide reacts with the HCl according to the equation:

$$NaOH + HCl \longrightarrow NaCl + H_2O$$

That is mean one equivalent from the acid is needed to neutralize the hydroxide.

Procedure

Prepare 0.1N from HCl, and full the buret with it. Transfer by a pipette 10 ml from the bases mixture in 250 ml conical flask and introduce 2 drops of ph. Ph indicator. Titrate until the solution just changes from pink to colorless, and write down the volume of the acid. The volume of the acid here (v_1) is equivalent to the all hydroxide and 1/2 the carbonate.

$$Na_2CO_3 + HCl \xrightarrow{Ph.ph} NaHCO_3 + NaCl$$

Add 2 drops of the methyl orange indicator and continue the titration until the solution just change from orange to red. (It is possible to empty the conical flask and take 10 ml of the mixture and 2 drops of M.O. and start from the beginning). The volume of the acid here (v_2) (which is equal to v_1 and the added volume of the acid), is equivalent to the all hydroxide and all the carbonate.

$$NaOH + HCl \xrightarrow{M.O.} NaCl + H_2O$$

$$Na_2CO_3 + 2HCl \xrightarrow{M.O.} 2NaCl + H_2CO_3$$

$v_2 - v_1 = v_3$ is the volume of the acid equivalent to 1/2 carbonate

$v_3 \times 2$ is the volume of the acid equivalent to all carbonate

$v_2 - 2v_3 = v_4$ is the volume of the acid equivalent to all hydroxide

no. of the acid milliequivalents = no. of the carbonate milliequivalents

$$N_1 \times V_1 = N_2 \times V_2$$

$$0.1 \times 2v_3 = N_2 \times 10$$

N_2 = normality of carbonate

The strength of carbonate solution g/L = N_2 × equivalent weight (molecular wt./2)

no. of the acid milliequivalents = no. of the hydroxide milliequivalents

$$N_1 \times V_1 = N_2 \times V_2$$

$$0.1 \times v_4 = N_2 \times 10$$

N_2 = normality of hydroxide

The strength of hydroxide solution g/L = N_2 × equivalent weight (molecular wt./1)

Exp. 5

Determination of Chloride by the Mohr Method

The formation of a second precipitate of distinctive color is the basis for end-point detection with the *Mohr method*. The procedure has been widely applied to the titration of chloride ion and bromide ion with standard silver nitrate. Chromate ion is the indicator, the point being signaled by the appearance of brick-red silver chromate, Ag_2CrO_4.

The formal solubility of silver chromate is substantially greater than that for the silver halides. In a Mohr titration, then, no silver chromate will be produced until essentially all of the halide has been precipitated. Through control of the chromate ion concentration, it is possible to retard the formation of silver chromate until the silver ion concentration acquires a value that corresponds to the theoretical equivalence-point region for the halide titration.

The reaction between the chloride ion and silver nitrate is accomplished according to the equation:

$$NaCl + AgNO_3 \longrightarrow \underset{\text{white ppt.}}{AgCl\downarrow} + NaNO_3$$

When all of the chloride has been precipitated, the first excess drop from the $AgNO_3$ reacts with the indicator forming silver chromate as a brick-red precipitate, indicating the end point of the reaction.

$$2AgNO_3 + K_2CrO_4 \longrightarrow \underset{\text{brick-red ppt.}}{Ag_2CrO_4\downarrow} + 2KNO_3$$

Procedure

Prepare 0.1N solution from $AgNO_3$ (16.98 g in 1 L), and full the buret with it. Transfer by a pipette 10 ml from the chloride solution in 250 ml conical flask and introduce 10 drops of K_2CrO_4 indicator. Titrate until the solution just changes from yellow to brick-red, and write down the volume of the $AgNO_3$ consumed.

$$\text{no. of the } AgNO_3 \text{ milliequivalents} = \text{no. of the } Cl^- \text{ milliequivalents}$$

$$N_1 \times V_1 = N_2 \times V_2$$

$$0.1 \times (\text{from buret}) = N_2 \times 10$$

$$N_2 = \text{normality of } Cl^-$$

The strength of Cl^- solution $= N_2 \times$ equivalent weight

$= N_2 \times 35.5$

If all NaCl is to be calculated $= N_2 \times 58.5$

Exp. 6

Determination of Chloride by the Volhard Method

The most important application of the Volhard method is for the indirect determination of chloride. A measured excess silver nitrate solution is added to the chloride sample, and the excess silver ion is determined by back-titration with a standard thiocyanate solution. The requirement of a strongly acid environment represents a distinct advantage for the Volhard titration over other methods for chloride because such ions as carbonate, oxalate, and arsenate (which form slightly soluble silver salts in neutral media) do not interfere.

Silver chloride, in contrast to the other silver halides, is more soluble than silver thiocyanate. As a consequence, the reaction:

$$AgCl(s) + SCN^- \rightleftharpoons AgSCN(s) + Cl^-$$

cause the end point in the Volhard determination of chloride to fade; an overconsumption of thiocyanate ion and a negative error for the analysis may result. The magnitude of this error is dependent upon the indicator concentration.

Two general methods are employed to avoid the error resulting from the reaction between thiocyanate and silver chloride. The first involves the use of the maximum allowable indicator concentration [about 0.2M iron(III) ion]. The more popular way involves isolation of the precipitated silver chloride before back-titration with the thiocyanate. Filtration, followed by titration of an aliquot of the filtrate, yields excellent results provided the precipitated silver chloride is first briefly digested. The time required for filtration is, of course, a disadvantage. Probably the most widely employed modification is that of Caldwell and Moyer, which consists of coating the silver chloride with nitrobenzene, thereby substantially removing it from contact with the solution. The coating is accomplished by shaking the titration mixture with a few milliliters of the organic liquid like nitrobenzene prior to back-titration.

Silver nitrate reacts with the chloride according to the equation:

$$AgNO_3 + Cl^- \longrightarrow AgCl\downarrow + NO_3^-$$

The excess silver nitrate (doesn't react) then determined by back-titration with standard potassium thiocyanate solution, and using Fe(III) ions as an indicator.

$$AgNO_3 + KSCN \longrightarrow AgSCN\downarrow + KNO_3$$

When all silver ions is reacted, the first excess drop from the thiocyanate reacts with Fe(III) ions forming red complex indicating the end point of the reaction.

$$Fe^{3+} + SCN^- \longrightarrow [Fe(SCN)]^{2+}$$

Procedure

Weight about 1.5 g from the chloride sample and dissolve it in 250 ml water. Transfer by a pipette 10 ml from the sample solution into a conical flask and add 20 ml silver nitrate 0.1N; silver chloride is precipitate. Add 5-6 drops of HNO_3 6N and 2 ml nitrobenzene. Close the end of the conical flask and shake thoroughly for few minutes, then leave the mixture to settle down. Add about 2 ml of iron(III) ammonium sulfate ($Fe(SO_4)_2NH_4.12H_2O$) indicator. Titrate with 0.1N KCSN solution from a buret until the red-brown color of $FeSCN^{2+}$ is permanent, which indicates the end point. Write down the volume of KSCN consumed to find the volume the excess $AgNO_3$.

$$\text{no. of the KSCN milliequivalents} = \text{no. of the } AgNO_3 \text{ milliequivalents}$$

$$N_1 \times V_1 = N_2 \times V_2$$

$$0.1 \times (\text{from buret}) = 0.1 \times V_2$$

The volume of the $AgNO_3$ needed to precipitate the chloride in the sample v_3 = the volume of added $AgNO_3$ v_1 (which is 20 ml) – V_2.

$$N = \frac{\text{weight of sample in g} \times 1000}{\text{equivalent weight} \times \text{volume of solution in ml}}$$

$$\text{weight of sample} = N \times \text{volume} \times \frac{\text{equivalent weigh}}{1000}$$

$$\text{weight} = N \times \text{volume} \times \text{milliequivalent} \ldots\ldots\ldots\ldots.1)$$

$$\% \, Cl^- = \frac{N \times V \times \text{milliequivalent}}{\text{weight of sample}} \times 100 \ldots\ldots2)$$

$$\% \, Cl^- = \frac{250/10 \times N(AgNO_3) \times V\,(v_3\,(AgNO_3)) \times 0.0354}{\text{weight of sample (1.5 g)}} \times 100$$

equation 1 represent the numerator in the equation $\% = \frac{\text{weight of fraction}}{\text{weight of sample}} \times ١٠٠$

If NaCl is to be calculated we use 0.05845 instead of 0.0354.

Exp. 7

Preparation and Standardization of 0.1 N $KMnO_4$

Potassium permanganate, a powerful oxidant, is perhaps the most widely used of all standard oxidizing agents. The color of a permanganate solution is so intense that an indicator is not ordinarily required. The reagent is readily available at modest cost. On the other hand, the tendency of permanganate to oxidize chloride ion is a disadvantage because hydrochloric acid is such a useful solvent.

One of the most obvious properties of potassium permanganate is its intense purple color, which commonly serves as the indicator for titrations.

The permanganate end point is not permanent and gradually fades. Decolorization results from the reaction of the excess permanganate with the relatively large concentration of manganese (II) ion that is present at the end point.

$$2MnO_4^- + 3Mn^{2+} + 2H_2O \rightleftharpoons 5MnO_2(s) + 4H^+$$

Solid potassium permanganate, which is ordinarily used for the preparation of permanganate solutions, is inevitably contaminated with manganese dioxide to some extent; as a result, preparation of standard permanganate solutions directly by weight is not possible.

A permanganate solution possessing reasonable stability can be obtained provided a number of precautions are observed. Perhaps the most important variable affecting stability is the catalytic influence of manganese dioxide. This contaminant occurs in the starting material and is also produced when permanganate oxidizes organic matter in the water used to prepare the solution. Removal of manganese dioxide by filtration markedly enhances the stability of standard permanganate solutions. Sufficient time must be allowed for complete oxidation of contaminants in the water before filtration; the solution may be boiled to hasten the oxidation. Paper cannot be used for the filtration since it reacts with the permanganate to form the undesirable dioxide.

Standardized solutions should be stored in the dark. If any solid is detected in the solution, filtration and restandardization are necessary. In any event, restandardization every one to two weeks is a good precautionary measure.

In acidic solution, permanganate oxidizes oxalic acid to carbon dioxide and water.

The reaction of permanganate (oxidizing agent) with oxalic acid (reducing agent) is oxidation-reduction reaction, involves electron transfer.

The permanganate solution is added from the buret to the solution of oxalic acid in the conical flask until the color is changed to a pink in the end point.

$$2(MnO_4^- + 8H^+ + 5e^- \rightleftharpoons Mn^{2+} + 4H_2O) \qquad +7 \longrightarrow +2$$

$$5(C_2O_4^{2-} \rightleftharpoons 2CO_2 + 2e^-) \qquad +3 \longrightarrow +4$$

$$2MnO_4^- + 16H^+ + 5C_2O_4^{2-} \rightleftharpoons 2Mn^{2+} + 10CO_2 + 8H_2O$$

The complete chemical equation is:

$$2KMnO_4 + 3H_2SO_4 + 5H_2C_2O_4 \rightleftharpoons K_2SO_4 + 2MnSO_4 + 10CO_2 + 8H_2O$$

Procedure

Preparation of 0.1 N $KMnO_4$

Dissolve 3.2 g of $KMnO_4$ in about 1 liter of distilled water. Heat to boiling, and keep hot for about 1 hr. Cover and let stand overnight. Filter the solution through a fine-porosity sintered glass crucible or through a Gooch crucible with an asbestos mat. Store the solution in a clean, glass-stoppered bottle, and keep in the dark when not in use.

Standardization against oxalic acid

Full the buret with $KMnO_4$ solution. Transfer by a pipette 10 ml from the 0.1N oxalic acid ($H_2C_2O_4$) solution to a conical flask, and add 10 ml 4N H_2SO_4 solution. Heat the mixture to 60-90^{o}C for few minutes, and titrate by adding the permanganate solution, stirring vigorously with a thermometer. The reagent should be introduced slowly so that the pink color is discharged before further additions are made. If the solution temperature drops below 60^{o}C heat. The end point is the first persistent pink color.

If a brown precipitate is formed during titration, it is attributed to one of the followings:

1- The temperature degree of the solution below 60^{o}C.
2- The addition of permanganate was fast.
3- The amount of H_2SO_4 is not sufficient.
4- The vigorous heating lead to dissociation of oxalic acid.

$$\text{no. of the } KMnO_4 \text{ milliequivalents} = \text{no. of the } H_2C_2O_4 \text{ milliequivalents}$$

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times (\text{from buret}) = 0.1 \times 10$$

The strength of permanganate solution = N_1 × equivalent weight (molecular wt./5)
= $N_1 \times 158/5$

Exp. 8

Determination of $K_2Cr_2O_7$ Normality By Titration with Mohr's Salt

It is possible to determine the normality of potassium dichromate $K_2Cr_2O_7$ as oxidizing agent by titration against ferrous ammonium sulfate $Fe(NH_2)_2(SO_4)_2.6H_2O$ (Mohr's salt) as reducing agent.

The dichromate solution is added from the buret to a conical flask containing Mohr's salt and diphenyl ammine as an indicator and sulphuric acid, until the color becomes pink, which indicates the end point.

The reaction proceed according to the equations:

$$Cr_2O_7^{2-} + 14H^+ + 6e^- \rightleftharpoons 2Cr^{3+} + 7H_2O) \quad +6 \longrightarrow +3$$

$$6(Fe^{2+} \rightleftharpoons Fe^{3+} + 1e^-) \quad +2 \longrightarrow +3$$

$$Cr_2O_7^{2-} + 14H^+ + 6Fe^{2+} \rightleftharpoons 2Cr^{3+} + 6Fe^{3+} + 7H_2O$$

The complete chemical equation is:

$$K_2Cr_2O_7 + 7H_2SO_4 + 6FeSO_4 \rightleftharpoons K_2SO_4 + Cr_2(SO_4)_3 + 3Fe_2(SO_4)_3 + 7H_2O$$

Procedure

Full the buret with $K_2Cr_2O_7$ solution. Transfer by a pipette 10 ml from the Mohr's salt 0.1N solution to a conical flask, and add 10 ml 2N H_2SO_4 solution (or mixture of H_2SO_4 and H_3PO_4), and 2-3 drops diphenyl ammine as indicator. Titrate until the appearance of a pink color.

no. of the $K_2Cr_2O_7$ milliequivalents = no. of the Mohr's salt milliequivalents

$$N_1 \times V_1 = N_2 \times V_2$$

$$N_1 \times \text{(from buret)} = 0.1 \times 10$$

The strength of K_2CrO_4 solution = N_1 × equivalent weight (molecular wt./the change in oxidation number × no. of atoms)

$$= N_1 \times \text{M. wt.}/2\times3$$

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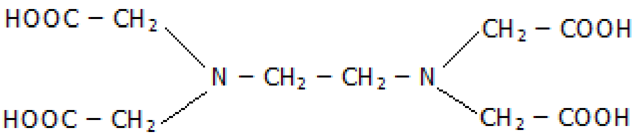
Exp. 9

Determination of Total Hardness in Tab Water

The hardness of water is due to the presence of Mg^{2+} and Ca^{2+} ions. It can be calculated as calcium carbonate. The hardness of water is expressed in mg/L or ppm.

The calcium ions Ca^{2+} can be calculated by titration with Ethylenediaminetetraacetic acid (EDTA).



or its sodium salt disodium dihydrogen ethylene diammine tetraacetate solution.

The end point of the reaction is appeared when EDTA solution reacts with the metal ion exists in the colored complex formed from the metal ion and the indicator Eriochrome black T (EBT).

$$\underset{\text{Metal ion}}{M^{2+}} + \underset{\text{EDTA ion}}{H_2Y^{2-}} \rightleftharpoons \underset{\text{EDTA-metal complex}}{MY^{2-}} + 2H^+$$

If Ca^{2+} is the ion to be reacted the reaction proceeds as follow:

$$Ca^{2+} + H_2Y^{2-} \longrightarrow CaY^{2-} + 2H^+$$

Procedure

Full the buret with EDTA 0.01M solution. Transfer by a pipette 10 ml from the tap water to a conical flask, and add 5 ml of ammonia solution, and 2-4 drops from

EBT solution as an indicator. Titrate until the color of the solution in the conical flask change from red to blue, which indicates the end point.

$$\text{no. of EDTA millimoles} = \text{no. of } Ca^{2+} \text{ions millimoles}$$

$$\text{ml from EDTA solution} \times \text{EDTA molarity} = \text{ml from the sample solution} \times \text{sample molarity}$$

$$\text{sample molarirt } M_1 = \frac{\text{ml from EDTA solution}}{\text{ml from the sample solution}} \times \text{EDTA molarity}$$

$$\text{Mass of calcium g/L} = \frac{\text{ml from EDTA solution}}{\text{ml from the sample solution}} \times \text{EDTA molarity} \times 40.08 \text{ g/mole}$$

$$\text{Mass of calcium mg/L} = \frac{\text{ml from EDTA solution}}{\text{ml from the sample solution}} \times \text{EDTA molarity} \times 40080 \text{ mg/mole}$$

The result = calcium hardness.

$$\begin{array}{l}\text{The water hardness as} \\ \text{calcium carbonate } CaCO_3\end{array} = \frac{\text{ml. EDTA solution}}{\text{ml. sample solution}} \times \text{EDTA molarity} \times 100.08 \times 10^3 \text{ mg/L (ppm)}$$

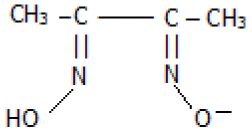
* * *

Exp. 10

Gravimetric Determination of Nickel

Preparation of Bis(dimethylglyoximato)nickel(II) complex and Determination of nickel Percentage

Nickel can forms complex with ligands like dimethylglyoxime, and the percentage of nickel can be calculated.



$$Ni^{2+} + (C_4H_8O_2N_2)_2 \longrightarrow Ni(C_4H_7O_2N_2)_2 + 2H^+$$

Procedure

Take 0.2234 g from the nickel compound and put it in a 200 ml beaker, and add 50 ml distilled water and 5 ml dil. HCl. Dilute the solution to be 200 ml by adding distilled water to the mark.

Boil the solution to 70-80°C, and add about 5 ml from the reagent solution, then add NH_4OH solution, and when the solution becomes basic, a red precipitate formed (nickel with reagent forms red complex in basic media). Stir and add extra quantities from NH_4OH solution and the reagent to be sure that all the nickel is precipitated as dimethylglyoxime complex.

Continue boiling for 30-40 minutes, then leave the precipitate to settle down for about an hour.

Filter the solution in a porous glass crucible, and wash the precipitate with distilled water several times, and dry in a furnace at 110°C for 1 hour. Let the crucible to cool down and weight it with the precipitate. Let the crucible to cool down again and weight it with the precipitate. Repeat the last step until two similar weights is reached.

The difference between the weight of the crucible with the precipitate and empty is the weight of the complex.

1 mole precipitate ≡ 1 mole Ni

288 (molecular wt.) precipitate ≡ 58 g Ni

weight of ppt. ≡ x gm

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x = weight of Ni

$$\text{sample percentage} = \frac{\text{weight of Ni}}{\text{weight of sample (0.2234)}} \times 100$$