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List of Experiments

Experiment No. ……

AIM:

To determine the turbidity of the given sample.

APPARATUS:

Nephelo – Turbidity meter with sample cell

REAGENTS FOR CALIBRATION OF THE INSTRUMENT:

Solution 1:

Dissolve 1 gm Hydrazine Sulphate (NH2)2H2So4 (carcinogen) in distilled water and dilute to 100 ml in a volumetric flask

Solution 2:

Dissolve 10 gms Hexamine LR grade (CH2)2N4 in distilled water and dilute to 100 ml in a volumetric flask Take 12.5 ml of solution 1 and 12.5 ml of solution 2 in a 100 ml volumetric flask and dilute to 100 ml, allow to stand for 24 hours at 250C. the turbidity of this suspension is 1000 Nephelometric Turbidity Unit (NTU).

THEORY:

Turbidity is an expression of optical property that uses light scattering properties of suspension in the sample. Turbidity in water is caused by suspended matter such as clay, silt, finely divided organic and inorganic matter soluble, colored organic compounds, plankton and other microscopic organisms.

Turbidity is measured by shining light through a sample and measuring the degree of scattering as measured by a light detector placed at right angles to the original light path. Above measuring technique is known as Nephelometry.

Turbidity can also be measured by shining light through a sample and measuring the degree of light penetration as measured by a light detector placed in line to the original light path. This measuring technique is known as Turbidimetry.

PRINCIPLE

Nephelo-Turbidity meter operates on the principle that light passing through a substance is scattered by matter suspended in the substance. In this instrument, a strong light beam is passed upward through a tube containing the sample. As the beam passes through the sample, the light is scattered in proportion to suspended particles. At 900 to the light path, this scattered light is sensed by the phototube to give the turbidity reading. The unit of measurement is NTU.

CALIBRATION OF THE INSRTRUMENT

1. Switch on the instrument and keep it ON for some time.
2. Select appropriate range depending upon the expected turbidity of the sample.
3. Set zero of the instrument with turbidity free water using blank solution and adjust 000 with the ‘set zero’ knob. The CAL control should be moved by 5 turns clockwise from 0 position.

4. Now in another test tube, take standard suspension that already prepared. For 0-200 NTU range use 100 NTU solution and for higher range use 400 NTU solution as standard.

PROCEDURE

1. Take the test tube containing distilled water in the test tube holder and close the test tube holder lid.
2. Select the required range for measurement.
3. Adjust the display to 000 by adjusting 'set zero' knob.
4. Remove the test tube containing distilled water & insert another test tube containing standard solution (say 400 NTU). Place it in the test tube holder.
5. Take the measurement of the solution suspension& adjust the 'calibrate' knob so that the display reads the selected standard solution value.
6. Again check the display zero with the test tube containing distilled water.
7. Now the instrument is ready to take measurement of any unknown suspension.

RESULTS

Turbidity of the given sample of water = NTU

INTERFERENCE

1. Turbidity can be determined for any water sample that is free of debris and rapidly settling coarse sediments.
2. Dirty glass ware or the presence of air bubbles disturb the surface visibility of the sample & will give false results.
3. Water color due to dissolved substances that absorb light causes measured turbidities to be lowest.

Precautions

1. Sample test tube must be thoroughlycleaned both inside and outside. In case the test tube gets scratched, discard it.

2. Do not touch the test tube where the light strikes i.e.at the sides of the test tube. So hold the test tube only at the top end.

3. Fill the test tube with samples or standards which have been thoroughly agitated. Allow sufficient time for the air bubbles to escape otherwise the reading will slowly come down. When the readings are taken in the agitated state with air bubbles, the readings will be higher.

Experiment No. ……

AIM: To determine the specific conductivity of the given sample.

APPARATUS

Digital conductivity meter

REAGENTS

Standard KCl Solution: Dissolve 0.7456 gms of Potassium Chloride(dried at 1800C for 1 hour) in distilled water and dilute to 1000 ml. The specific conductance of this solution S/Cm.μat 250C is 1408 μS/Cm.

THEORY

The conductivity of the water is its capacity to carry an electrical current and varies both with number and types of ions the solution contains, which in turn is related to the concentration of ionized substances in the water. Most inorganic substances in water are in the ionized form and hence contribute to conductance. The unit of specific S/Cm).μconductivity is micro Siemens per Cm (μS/Cm).

PRINCIPLE

The specific conductivity is measured by employing the wheat-stone bridge principle. The cell and temperature probes of the instruments are dipped into the given sample to find the specific conductivity of the given sample. The specific conductivity multiplied by a conversion factor gives the total dissolved solids.

CELLCONSTANT VERIFICATION

Checking the cell constant as per the following procedure is recommended before any measurement.

1. Set the 'Function' switch to check position & adjust the display to 1.000 with CAL control.
2. Dip the Conductivity cell in a solution of known value.
3. Adjust the temperature control to the temperature of the solution. (not required when measuring in ATC mode)
4. Move the Function switch to Cond. Position and Range switch to appropriate range.
5. Adjust the cell constant knob so that the display reads the known value of the solution.
6. Bring the Function switch to Cell Const. position.
7. The display shows the cell constant of the conductivity cell.

PROCEDURE

1. Rinse the conductivity cell with solution whose conductivity is to be measured.

2. Dip the conductivity cell in the solution under test

3. Set the function switch to check position
4. Display must read 1.000. If it doesn't , set it with CAL control provided at the back panel.
5. Put function switch to cell constant position and set the cell constant control to the cell constant value of the conductivity cell
6. Move the function switch to the COND. Position and switch to appropriate range

7. Connect the conductivity cell at the rear of the instrument
8. Set the temperature control to the temperature of the solution
9. Bring the range switch at a position where maximum resolution is obtained
10. Read the display
11. This would be the exact conductance of sample at 250C.

RESULT

The specific conductivity of the given sample S/Cmμ=

PRECAUTIONS

1. Rinse the cell in demineralized water.
2. Do not finger the cell electrodes.
3. Check the cell constant as frequently as possible because deposition of film on the plates affects the cell constant.

Experiment No. ……

Object

Determination of pH of a solution using a pH meter and titration of such a solution pHmetrically.

Reagents/Chemicals

Standard NaOH solution……, unknown acid solution, distilled water, buffer solution of pH 4 and 9.

Apparatus

pH meter, glass electrode, reference electrode, beaker (400 ml), burette, stirrer.

Theory

$$pH = -\log [H^+]$$

When an alkali is added to an acid, the pH of the solution increases slowly, but at the vicinity of the equivalence point, the rate of change of pH of the solution is very fast. The equivalence point can be observed from sharp break in curve. From the equivalence point, the strength of the solution can be calculated by normality equation.

Method

First the pH meter was standardized against the buffer of pH 4 and pH 9. Then the glass electrode was washed with distilled water and dried. Then 100 ml of HCl solution was taken in a beaker and the electrode was dipped in HCl solution and pH of the pure acid solution was noted down. Standard NaOH solution (1ml) from burette was added into the beaker, stirred well and noted the pH of solution. Similarly 9-10 ml of NaOH was added into acid solution gradually and noted the pH after each addition of NaOH solution. At equivalence point there was a sharp change in the pH. Now alkali solution was added in fractions (0.2 ml). A graph was plotted which gave the actual volume of NaOH at neutralization.

Observations

Volume of alkali added (ml)																
pH																

Result : The pH of given solution is _____

: The strength of the given acid solution is _____ g/l.

Precautions

1. The electrode is washed thoroughly and dried before dipping into the solution.
2. After each addition of alkali, the solution is stirred thoroughly.
3. The knob of pH meter is on standby position at the time of adding alkali in the acid solution.

Calculations

Volume of alkali used at the end point	=	V_2 ml
N_1V_1	=	N_2V_2
(Acid)		(Alkali)

$N_1 \times 50$ =

N_1 =

Experiment No. …….

Object
Determination of constituents and amount of Alkalinity of the supplied water sample.

Reagents/Chemicals
Water sample, Standard HCl....., Phenolphthalein and Methyl Orange Indicator

Apparatus
Burette, pipette, conical flask, beaker, measuring flask

Theory
Alkalinity of water is mainly due to the presence of the following: (i) Hydroxides only (ii) Carbonates only (iii) Bicarbonates only (iv) Hydroxides and Carbonates (v) Carbonates and Bicarbonates, since OH^- and HCO_3^- ion cannot coexist because both combine together to form carbonates

$$OH^- + HCO_3^- \longrightarrow CO_3^{-2} + H_2O$$

The extent of alkalinity present in a water sample is determined by titrating the water sample with a standard acid using phenolphthalein end point (P). At this point complete neutralization of hydroxide and conversion carbonate to bicarbonate takes place

$$OH^- + H^+ \longrightarrow H_2O$$
$$CO_3^{-2} + H^+ \longrightarrow HCO_3^-$$

Now, methyl orange is added in the solution and further continue the titration till the colour of the solution changes from yellow to brick red. It is methyl orange end point (M)

$$HCO_3^- + H^+ \longrightarrow H_2O + CO_2$$

We are able to calculate the magnitude of various forms of alkalinity by knowing the values of (P) ml and (M) ml

(i) <u>When only OH^- are present</u> – In this case only phenolphthalein indicator is used, therefore volume of HCl used is (P) ml

(ii) <u>When only HCO_3^- are present</u> – In this case only methyl orange indicator is used therefore volume of acid used is (M) ml

(iii) <u>When only CO_3^{-2} are present</u> - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl used is 2M ml.

(iv) <u>Both OH^- and CO_3^{-2} are present</u> - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl for OH^- is (P-M) ml and for CO_3^{-2} is 2M ml

(v) <u>Both CO_3^{-2} and HCO_3^- are present</u> - In this case both phenolphthalein and methyl orange indicator are used. The volume of HCl for CO_3^{-2} ions is 2P ml and for HCO_3^- is (M-P) ml

Method

First the burette was rinsed with standard HCl solution and then filled with standard HCl solution. Then ……. ml of water sample was taken out in a conical flask and 2-3 drops of phenolphthalein indicator was added which may give pink colour. Now the solution was titrated with standard solution of HCl till the pink colour changes to colourless. Disappearance of pink colour indicates the end point. It gives the value of phenolphthalein end point (P). Now 2-3 drops of methyl orange was added in the same solution which may give yellow colour and the solution was further titrated till the colour changes to brick red colour. This gives the value of methyl orange end point (M). The process was repeated till two concordant readings were obtained.

Observations

S. No	**Volume of Water Sample (ml)**	**Volume of HCl used (ml)**		
		Initial Reading	**Phenolphthalein end point (P)**	**Methyl Orange end point (M)**
1.				
2.				
3.				

Result : The alkalinity due to CO_3^{-2} is_____ ppm

The alkalinity due to HCO_3^- is_____ ppm

The alkalinity due to OH^- is_____ ppm

Precautions

1. Phenolphthalein indicator was added first and then methyl orange
2. The volume of indicator was same in all the titrations
3. The reaction mixture was shaken properly

Experiment No. …….

Object

Determination of temporary, permanent and total hardness of water by complexometric titration method.

Reagents/Chemicals

Standard EDTA solution....., water sample, standard hard water, buffer solution, Eriochrome black-T as an internal indicator

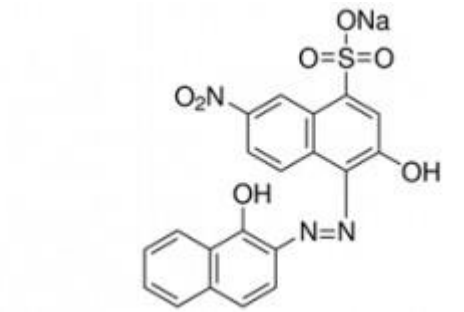
Apparatus

Burette, pipette, measuring flask, conical flask.

Theory

The hardness of water can be determined by complexometric titration. EDTA is used as complexing reagent. The Ca^{++} and Mg^{++} present in water are titrated with EDTA using Eriochrome black-T as an indicator.

When Eriochrome black –T indicator is added to hard water solution at pH 9 to 10, it gives wine red coloured unstable complex with Ca^{++} and Mg^{++} ions of the water sample. As this solution (wine red colour complex) is titrated against EDTA the free Ca^{++} and Mg^{++} ions in water form stable metal ion EDTA complex with the result , the indicator is set free, which gives blue colour to the solution.



Structure of Eriochrome black-T

Method

First the burette was rinsed and filled with standard EDTA solution. Then……ml of hard water sample was taken out in a conical flask, and 2 ml of buffer solution and 2 drops of Eriochrome black-T indicator were added to it. The solution was titrated against standard EDTA solution till the wine red colour changes into blue colour. That was the end point. The process was repeated till two concordant readings were obtained.

Thereafter hard water sample…… ml was taken out in a beaker and boiled gently for about half an hour and then filtered. The filtrate was titrated in the same manner as described above.

The volume of EDTA used corresponds to the permanent hardness. Now the same procedure was repeated for the standard hard water sample.

Calculations

1 ml of standard solution (EDTA) = 1 mg of $CaCO_3$

V_1 ml of EDTA = V ml of standard solution

1 ml of EDTA = $\frac{V}{V_1}$ mg of CaCO3

V ml of hard water solution = V_2 ml of EDTA

= $V_2 \times \frac{V}{V_1}$ mg of $CaCO_3$

1 ml of hard water solution = $\frac{V_2}{V_1} \times \frac{V}{V}$ mg of $CaCO_3$

1000 ml of hard water solution = $\frac{V_2}{V_1} \times 1000$ mg of $CaCO_3$

Total hardness = $\frac{V_2}{V_1} \times 1000$ ppm

V ml of boiled water = V_3 ml of EDTA

Permanent hardness = $\frac{V_3}{V_1} \times 1000$ mg of $CaCO_3$

= $\frac{V_3}{V_1} \times 1000$ ppm

Temporary hardness = Total hardness – Permanent hardness

Observations

Reading with standard hard water

S. No	Volume of water sample V (ml)	Burette Reading (ml)		Volume of EDTA used (ml) (V_1)
		Initial	Final	
1.				
2.				
3.				

Reading with hard water sample

S. No	Volume of water sample V (ml)	Burette Reading (ml)		Volume of EDTA used (ml) (V_2)
		Initial	Final	
1.				
2.				
3.				

Reading with boiled Water

S. No	Volume of water sample V (ml)	Burette Reading (ml)		Volume of EDTA used (ml) (V_3)
		Initial	Final	
1.				
2.				
3.				

Result : The temporary hardness of the given water sample is _____ ppm

: The permanent hardness of the given water sample is _____ ppm

: The total hardness of the given water sample is _____ ppm

Precautions

1. The amount of indicator was same in all the titrations.
2. The pH of the solution was maintained carefully to precipitate the complex.
3. The apparatus was rinsed with distilled water.

Experiment No. ……

Object

Determination of the percentage of available chlorine in a given sample of Bleaching Powder (Iodometrically).

Reagents/Chemicals

Bleaching powder or solution, potassium iodide, dilute acetic acid (~5%) sodium thiosulphate solution…….....distilled water and freshly prepared starch solution as indicator.

Apparatus

Burette, pipette, measuring flask, conical flask, funnel, pastel and mortar.

Theory

The amount of chlorine liberated by the action of dilute acids on hypochlorite is termed as "the available chlorine". It is generally expressed as the percentage by weight of bleaching powder. In practice the available chlorine is determined by treating it with sodium thiosulphate solution in presence of acetic acid, the liberated chlorine is immediately treated with potassium iodide (KI) to give free iodine, which actually reacts with standard sodium thiosulphate solution, hence termed as Iodometric titration.

$CaOCl_2 + 2CH_3COOH \longrightarrow (CH_3COO)_2Ca + H_2O + Cl_2 \downarrow$
(Bleaching Powder) (available chlorine)

$2KI + Cl_2 \longrightarrow 2KCl + I_2$

$2Na_2S_2O_3 + I_2 \longrightarrow 2NaI + Na_2S_4O_6$
(sodium tetrathionate)

$CaOCl_2 = 2Cl =$ $Na_2S_4O_6$
(sodium tetrathionate)

$Na_2S_4O_6 = Cl = I$
(1 gm equivalent) (1 gm equivalent) (1 gm equivalent)

Method

……gm sample of bleaching powder was weighed out accurately and made a paste with little distilled water in a mortar. This mixture was transferred to a 250 ml measuring flask. The solution was made up to 250 ml with distilled water to obtain a homogeneous suspension.

…….. ml of this suspension was taken in an appropriate conical flask and 1-2 gm of potassium iodide and 10-20ml of dilute acetic acid was added to this solution. The liberated iodine was immediately titrated with standard sodium thiosulphate solution till colour turns light yellow. A few drops of freshly prepared starch solution were added to the light yellow solution, which makes it dark. It was further titrated with thiosulphate solution till the colour disappeared and the burette reading noted. The same titration was repeated until at least two concordant readings were obtained.

Observations

Weight of empty weighing tube = ……. (a) gm

Weight of weighing tube + bleaching powder = ……..(b) gm

Weight of bleaching powder = ……. (b-a) gm

Normality of hypo solution ($Na_2S_2O_3$) = …….

S. No	**Volume of bleaching powder**	**Burette Reading (ml)**		**Volume of $Na_2S_2O_3$ (ml)**
		Initial	**Final**	
1.				
2.				
3.				

Result : The percentage of available chlorine present in given sample of bleaching powder is_____%

Precautions

1. All the apparatus were cleaned thoroughly.
2. A uniform paste of bleaching powder was prepared.
3. The amount of starch solution and acetic acid remained constant in all the titration.

Calculations

N_1V_1 = N_2V_2

(Bleaching Powder) (Sodium Thiosulphate)

Normality of available chlorine (N_1) = $\frac{N_2 \times V_2}{V_1}$

Amount of chlorine per litre of solution = $\frac{N_1 \times 35.5}{1000}$

% of available Chlorine = $\frac{N_1 \times 35.5 \times 250}{1000\ (b-a)} \times 100$

Experiment No. …..

Object

Determination of the Chloride Content in the supplied water sample using Mohr's Method

Reagents/Chemicals

Water Sample, Standard $AgNO_3$ solution……, K_2CrO_4 indicator

Apparatus

Burette, pipette, conical flasks, beakers, measuring flask

Theory

A slightly alkaline solution (pH 7-8) of the water sample is titrated against $AgNO_3$ solution using K_2CrO_4 as an internal indicator. The chloride ions present in the water sample reacts with silver ions forming insoluble white precipitate of silver chloride (AgCl). When all the chloride ions forms silver chloride then the extra drop of $AgNO_3$ reacts with chromate ions (CrO_4^{-2}) forming reddish brown precipitate of silver chromate thus indicating the end point

$$AgNO_3 + NaCl \longrightarrow AgCl \downarrow + NaNO_3$$

$$2Ag^+ + CrO_4^{-2} \longrightarrow Ag_2CrO_4$$

$$Ag_2CrO_4 + 2\ Cl^- \longrightarrow AgCl \downarrow + CrO_4^{-2}$$

$$2AgNO_3 + K_2CrO_4 \longrightarrow Ag_2CrO_4 + KNO_3$$

(Reddish Brown)

Method

The burette was rinsed with standard $AgNO_3$ solution and filled it with $AgNO_3$ solution. Now……. ml of water sample was taken in a conical flask and 2-3 drops of K_2CrO_4 indicator were added. The solution was titrated with the standard solution of $AgNO_3$ taken in the burette till reddish brown colour persisted. That was the end point. The process was repeated till two concordant readings were obtained. Now the same procedure was also followed with distilled water to obtain the blank correction. The blank correction reading was deducted from the titre value.

Observations

Reading with Water Sample

S. No	Volume of Water Sample (ml)	Burette Reading (ml)		Volume of $AgNO_3$ used
		Initial	Final	
1.				
2.				
3.				

Reading with Distilled Water

S. No	Volume of Distilled water (ml)	Burette Reading (ml)		Volume of $AgNO_3$ used
		Initial	Final	
1.				
2.				
3.				

Result : The chloride ion concentration in the given water sample is _____ ppm

Precautions

1. The same volume of water sample and distilled water for blank correction was taken.
2. The same drops of indicator were taken in all experiments.
3. The pH of solution was maintained between 7-8.

Calculations

Actual Volume of $AgNO_3$ used = Volume of $AgNO_3$ – Blank Correction

N_1V_1 = N_2V_2

($AgNO_3$) (Water Sample)

$$N_2 = \frac{N_1V_1}{V_2}$$

Strength of Cl^- ion = N_2 x 35.5 x 1000 ppm

EXPERIMENT No –
TOTAL SOLIDS

AIM: To determine the total solids present in water sample.

GENERAL: The term solid refers to the matter either filterable or in filterable that remains as residue after evaporation and subsequent drying at a defined temperature. Different forms of solids are defined on the basis of method applied for their determination.

Residue after the evaporation and subsequent drying in oven at specific temperature, 103-105ºC of a known volume of a sample are total solids. The loss in weight on ignition of the same sample at 550°C (in which organic matter is converted to CO_2 and H_2 O) gives organic solids present in the sample.

PROCEDURE:

1. Take empty weight of beaker. (W_I)
2. Take a known volume of a well mixed sample in the above beaker.
3. Evaporate the sample to dryness at 103°C for 24 hours.
4. Cool and weigh and record the reading (W_2)
5. Keep the beaker for 15-20 min. in a muffle furnace maintained at 550 $\pm$ 50°C.
6. Cool the beaker and record the final weight (W_3).

CALCULATIONS: Total solids mg/L= $(W_2 - \overline{W_1})$ x 1000ml of sample Organic solids mg/L. = $(W_2 - W_3)$ x 1000ml sample

OBSERVATIONS:

Weight of empty beaker	W_1= ----------mg
Weight of beaker after evaporation at 103°C	W_2 =----------mg
Weight of beaker after evaporation in muffle furnace	W_3 = ----------mg

RESULT:

Total Solids = mg/l

EXPERIMENT-

CHEMICAL OXYGEN DEMAND

AIM: To determine the chemical oxygen demand of given sample.

THEORY: Chemical oxygen demand (COD) test determines the oxygen required for chemical oxidation of organic matter with the help of strong chemical oxidant. The limitation of COD test is that it cannot differentiate between the biologically oxidizable and biologically inert material. COD determination has an advantage over BOD determination in that the result can be obtained in about 3-4 hours as compared to 5 days required for BOD test.

PRINCIPLE: The organic matter gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce $CO_2 + H_2O$. The excess $K_2Cr_2O_7$ remaining after the reaction is titrated with Fe $(NH_4)_2$ $(SO_4)_2$. The dichromate consumed gives the O_2 required for oxidation of organic matter.

APPARATUS:

1. Reflux apparatus consisting of a flat bottom 250 to 500 m1 capacity flask
2. Burner or hot plate with temperature regulator.

REAGENTS:

1. **Standard potassium dichromate 0.250 N**: Dissolve 12.259g by $K_2Cr_2O_7$ dried at 103°C for 24 hours in distilled water and dilute to 1000 ml. Add about 120 mg sulphamic Acid to take care of 6 mg/L NO_2-N.
2. **Sulphuric Acid reagent:** Add 10 g of Ag_2SO_4 to 1000 ml cone. H_2SO_4 and keep over night for dissolution.
3. **Standard ferrous ammonium sulphate 0.1 N**: Dissolve 39 g $Fe(NH_4.)_2$ $(SO_4)_26H_2O$ in about 400 ml distilled water. Add 20 ml cone. H2SO4 and dilute to 1000 ml.
4. **Ferroin indicator**: Dissolve 1.485 g of 1, 10 phenanthroline monohydrate and 695 mg $FeSO_4$, $7H_2O$ and dilute to 100 ml with distilled water.
5. **Mercuric Sulphate**: $HgS0_4$ crystals, analytical grade.

PROCEDURE:

1. Place 0.4 g $HgSO_4$ in a reflux flask.
2. Add 20 ml sample and mix well.
3. Add pumice stone or glass beads followed by 10 ml of standard $K_2Cr_2O_7$.
4. Add slowly 30 ml H_2SO_4 containing Ag_2SO_4 mixing thoroughly. This slow addition along with swirling prevents fatty acids to escape out due to high temperature.
5. Connect the flask to condenser: Mix the contents before heating.
6. Reflux for a minimum of 2 hours cool and then wash down the condenser with distilled water.

7. Dilute for minimum of 150 ml, cool and titrate excess $K_2Cr_2O_7$.with 0.1 N Ferry. Amm sulphate using ferroin indicator. Sharp colour change from blue green to wine red indicates end-point.
8. Reflux blank: in the same manner using distilled water instead of sample.
9. Calculate COD from the following formula:

COD mg/L = (a-b)x^2N8000 ml of sample

Where a = ml of Ferr. Amm. Sulphate for Blank, b= ml of Ferr. Amm. Sulphate for sample, N= normality of Ferr. Amm. Sulphate.

OBSERVATIONS:

S. No.	Burette Reading of Ferr. Amm. Sulphate			Remarks
	Initial	Final	Difference	
				.

RESULT:

COD of the given sample = ----------mg/l.