JORHAT ENGINEERING COLLEGE

ENVIRONMENTAL ENGINEERING

LABORATORY MANUAL

DEPARTMENT OF CIVILENGINEERING

JORHAT ENGINEERING COLLEGE

JORHAT, ASSAM-785007

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DEPARTMENT OF CIVIL ENGINEERING, JORHAT ENGINEERING COLLEGE

PUBLIC HEALTH ENGINEERING LABORATORY

DETERMINE THE PH OF GIVEN SAMPLE USING PH PAPER AND DIGITAL PH METER.

THEORY:

pH refers to the hydrogen ion activity. It is expressed as the negative logarithm of the reciprocal of the hydrogen ion activity in moles per litre. It can be measured by pH paper or electrometrically by measuring of hydrogen ion by potentiometric measurement using a standard hydrogen electrode and a reference electrode.

APPARATUS REQUIRED:

i. pH meter along with electrodes
ii. Buffer solution
iii. Thermometer
iv. pH paper

REAGENTS REQUIRED:

Standard buffer solution: Standard buffer solution can be prepared freshly by dissolving the standard buffer tablets or powders (pH 4 and 7.2).

PROCEDURE:

Using pH meters:

Take the liquid sample whose the pH is to be determined in a glass beaker.

Note the sample temperature. Rinse the electrode thoroughly with distilled water and carefully wipe with a tissue paper. Dip the electrode in to the sample solution

Using pH paper:

Dip the pH paper strip in to the solution. Compare the colour given on the wrapper of the PH paper book. Note down the PH of the sample along with temperature.

OBSERVATIONS:

RESULT:

pH value of sample using pH paper =

pH value of sample using pH meter =

CONCLUSION:

REMARK:

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MEASURE MINERAL AND PHENOLPHTHALEIN ACIDITY

THEORY:

Acids contribute to corrosiveness and influence chemical reaction rates, chemical speciation and biological processes. Acidity of water is its quantitative capacity to react with a strong base to a designated pH. The measured value may vary significantly with the end point pH used in the determination. When the chemical composition of the sample is known study mineral acids, weak acids such as carbonic and acetic and hydrolyzing salts such as iron or aluminum sulphate may contribute to the measured acidity according to the method of determination.

Mineral acidity: It is measured by titration to a pH of about 3.5, the methyl orange end point (also known as methyl orange acidity).

Total acidity: Titration of a sample to the phenolphthalein end point of pH 8.3 measures mineral acidity plus acidity due to weak acids, thus this is called as total acidity (or phenolphthalein acidity). In water analysis, this test does not bear significant importance because methyl orange acidity invariably remains absent in the raw water and even phenolphthalein acidity (that too principally due to the excessive-prevalence of dissolved carbon dioxide and carbonic acids) normally does not exist to a significant extent in the raw water. Importance: As for as water analysis is concerned, acidity test does not bear significant importance because methyl orange acidity invariably remains absent in the raw water and even phenolphthalein acidity (that too principally due to the excessive-prevalence of dissolved carbon dioxide and carbonic acids) normally does not exist to a significant extent in the raw water.

APPARATUS REQUIRED:

1) pH meter

REAGENTS:

i. Sodium hydroxide titrant (0.02 N)
ii. Phenolphthalein Indicator
iii. Methyl Orange Indicator

PROCEDURE:

Steps:

1. Take 50 ml sample in a conical flask and add 2-3 drops of methyl orange indicator solution.
2. Fill the burette with 0.02 N NaOH solutions and titrate till the colour of solution just changes to faint orange colour, indicating the end point. Record the volume of titrant consumed as V1 in ml. Calculate the methyl orange acidity using Eq (1a):

3. Methyl orange acidity (or Mineral Acidity) = (V1×1000)/(Sample volume) (1a)
   When the 0.02 N NaOH solution, used in titration is not standardized, mineral acidity is calculated using following Eq (1b):
   Methyl orange acidity= (V1×N×50×1000)/ (Sample vol.) (1b)
   For phenolphthalein acidity test, add 2-3 drops of phenolphthalein indicator solution to water sample from step 2 and continue the titration till the faint pink colour develops in the solution (i.e., the end point of titration). Record the volume of titration consumed as V2 (mL) and calculate total acidity or phenolphthalein acidity using Eq. (2):
   Total acidity (or Phenolphthalein Acidity) = (V2×N×50×1000)/ (Sample vol.) (2)

CONCLUSION:

REMARKS:

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DETERMINE SULPHATE ION CONCENTRATION IN A WATER SAMPLE USING METHOD: 4500-SO4 2- E. TURBID METRIC METHOD.

THEORY:

Sulphate are found in appreciable quantity in all natural waters, particularly high in arid and semi arid regions where natural waters in general have high salt content. Sulphate salts are mostly soluble in water and impart hardness. Water with high concentrations has a bitter test. Sulphate may cause intestinal disorders. These ions can produce hydrogen sulphides as per following equation (1):

SO_4^{2-} + organic matter − >S^{2-} + H_2O + CO_2 (1a) (in the presence of anaerobic bacteria)

$S^{2+} + H^{+} \rightarrow HS^{-}$ (1b)

$HS^{2+} + H^{+} \rightarrow H2S$ (1c)

The sulphate data is used in determining applicability of different water types for their public and industrial applications. It indirectly indicates extent of problems that can arise due to reduction of sulphates to hydrogen sulphides. In addition, sulphate content of organic matter fed to anaerobic digester is important information as it gives idea of generation of hydrogen sulphides, which needs to be removed.

The turbidimetric method depends on the fact that barium sulphate formed following barium chloride addition to a sample (Eq 2) tends to precipitate in a colloidal form and this tendency is enhanced in the presence of an acidic buffer (consists of magnesium chloride, potassium nitrate, sodium acetate, and acetic acid). These precipitates need to be separated through filtration (using a filter) before sample is analyzed for sulphate concentration. This is a very rapid method and can be used for samples with sulphate concentration greater than 10 mg/L (samples can be diluted and then it can be analyzed).

Ba2+ + $SO4^{2-}$−−>BaSO4 (precipitate; poorly soluble) (2)

APPARATUS REQUIRED:

Whatman No. 1 filter paper; Spectrophotometer; Magnetic stirrer

REAGENTS:

1. Buffer Solution A: Dissolve 30 g magnesium chloride (MgCl2.6H2O), 5 g sodium acetate (CH3COONa.3H2O), 1.0 g potassium nitrate (KNO3), and 20 mL acetic acid (CH3COOH; 99%) in 500 mL distilled water and make up to 1000 mL.

2. Buffer Solution B(required when the sample SO_4^{2-} <10 mg/L): Dissolve 30 g magnesium chloride, 5 g sodium acetate, 0.111 g sodium sulphate, and 20 mL acetic acid (99%) in 500 mL distilled water and make up to 1000 mL.

3. Dry Barium Chloride ($BaCl_2$) crystals 4. Standard Sulphate Solution: Dissolve 0.1479 g of anhydrous sodium sulphate in distilled water to make the volume 1 L. This solution contains 100 mg sulphate/L (i.e., 1 mL=100µg SO_4^{2-}). Prepare standards of various strengths (preferably from 0.0 to 40.0mg/L at the intervals of 5 mg/L by diluting this stock solution). Above 40 mg/L accuracy decreases and $BaSO_4$ suspensions lose stability.

PROCEDURE:

1. Filter the sample though filter paper (Whatman No. 1) and take 50 mL of filtrate in an Erlenmeyer flask.

2. Add 20 mL buffer solution and mix in stirring apparatus. While stirring, add 0.15 g of barium chloride to the sample and stir the sample with the help of magnetic stirrer for about an hour.

3. Measure the absorbance against a distilled water blank (DO NOT ADD BARIUM CHLORIDE TO IT.) at 420 nm using spectrophotometer. Absorbance for the blank sample is taken to correct for sample color and turbidity.

Sample Name	Turbidity (NTU)	Sample Name	Turbidity (NTU)
Distilled water blank		Standard 1 (5ppm)	30
Sample 1		10ppm	60
Sample 2		15	89
		20	101
		25	129
		30	157
		35	191
		40	205

4. Process the standard solution of different strengths in similar way and record the absorbance for each solution. Plot a standard sulphate calibration curve on a graph paper from these absorbance values putting strengths (mg/L) on X-axis and absorbance @ 420 nm on Y-axis. Fit a best-fit linear model to the data. Express equation as:

Absorbance value= A+B× Sulphate concentration (in mg/L) (3)

5. Using the standard sulphate calibration curve (a linear-model; Equation 3), find out sulphate concentration in the given unknown sample in mg/L. Sulphate concentration (mg SO_4^{2-}/L) = (1000×mg SO_4^{2-})/(mL sample) (4)

CONCLUSION:

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DETERMINE DO CONTENT OF A GIVEN SAMPLE

THEORY:

Dissolved oxygen (DO) levels in environmental water depend on the physiochemical and biochemical activities in water body and it is an important useful in pollution and waste treatment process control. Two methods are commonly used to determine DO concentration:

(1) The iodometric method which is a titration-based method and depends on oxidizing property of DO.

(2) The membrane electrode procedure, which works based on the rate of diffusion of molecular oxygen across a membrane.

In the Iodometric method, divalent manganese solution is added to the solution, followed by addition of strong alkali in a glass-stopper bottle. DO rapidly oxidize an equivalent amount of the dispersed divalent manganese hydroxide precipitates to hydroxides of higher valence states. In the presence of iodide ions in an acidic solution, the oxidized manganese reverts to the divalent state, with the liberation of iodine equivalent of the original DO content. The iodine is then titrated with a stranded solution of thiosulphate. The titration end point can be detected visually with a starch indicator. Some oxidizing and reducing agents present in solution can interfere with the iodometric method. Oxidizing agents liberate iodine from iodides (positive interference) and some reducing agents reduce iodine to iodide (negative interference). Also, organic matter present in solution can be oxidized partially in the presence of oxidized manganese precipitate, thus causing negative errors. Thus some modification of procedure is required.

Standardization of thiosulphate solution

$2Na2S2O3.5H2O+I2 \rightarrow Na2S4O6 +2NaI+10H2O$ (1a)

$2S2O3^{2-}+I2 \rightarrow S4O6^{2-}+2I^{-}$ (1b)

$Cr2O7^{2-}+6I^{-}+14H^{+} \rightarrow 2Cr3^{+}+3I2+7H2O$ (1c)

$2IO3^{-}+10I^{-}+12H^{+} \rightarrow 6I2+6H2O$ (1d)

The Winkler Method for DO Determination

If no oxygen is present, a pure white precipitate is formed when MnSO4 and alkali-iodide reagent (NaOH+KI) are added to the sample.

$Mn2+ + 2OH- \rightarrow Mn(OH)2$ (white precipitate) (2a)

If sample has some oxygen, Mn2+ is oxidized to Mn4+ and precipitates brown hydrated oxide.

$Mn2+ + 2OH- +0.5O2 \rightarrow MnO2$ (brown hydrated precipitate) + $H2O$ (2b)

The oxidation of Mn2+ to MnO2 is called fixation of the oxygen, occurs slowly at low temperature.

$Mn(OH)2 +0.5O2 \rightarrow MnO2 +H2O$ (2c)

After shaking the sample for a time sufficient to allow all oxygen to react, the floc is allowed to settle so to leave 5 cm of clear liquid below the stopper; then sulphuric acid is added. Under the low pH

conditions, MnO_2 oxidizes to produce I_2. I_2 is insoluble in water and forms complex is excess iodide ion is present in solution, thus preventing escape of iodine ions from solution.

$MnO_2 + 2I^- + 4H^+ \rightarrow Mn^{2+} + I_2 + 2H_2O$ (2d)

$I_2 + I^- \rightarrow I_3^-$ (2e)

Now the sample is ready for titration with thiosuphate solution.

The Azide Modification with the Winkler Method for DO Determination

This modification is used because of presence of nitrite ions. This occurs in effluents from wastewater treatment plants employing biological processes, in river water and in incubated BOD samples. It does not oxidize Mn^{2+} but doses oxidize I^- to I_2 under acidic conditions. When the reduced form of nitrite (N_2O_2) is oxidized by oxygen, it is converted to NO_2^- again, establishing the cycle again that can result in erroneous results, far in excess of amounts that would be expected.

$2NO_2^- + 2I^- + 4H^+ \rightarrow I_2 + N_2O_2 + 2H_2O$ (3a)

$N_2O_2 + 0.5O_2 + H_2O \rightarrow 2NO_2^- + 2H^+$ (3b)

When interference from nitrites is present, it is impossible to obtain a permanent end point. As soon as the blue color of the starch indicator has been discharged, the nitrites formed by the reaction (3b) reacts with more iodide ions to produce I_2 and the blue color of the starch indicator will return. The nitrite interference is easily overcome with use of sodium azide (NaN_3), which is incorporated in the alkali-KI reagent. When sulphuric acid is added, following reactions happen:

$NaN_3 + H^+ \rightarrow HN_3 + Na^+$ (3c)

$HN_3 + NO_2^- + H^+ \rightarrow N_2 + N_2O + H_2O$ (3d)

Method: The Azide Modification (For nitrite-N < 0.05 mg/L and Ferrous iron<1 mg/L)

The azide modification is used to minimize the effect of interfering materials. It removes interference caused by nitrite which is most commonly found interference in biologically treated effluents and in incubated BOD samples.

Collection of Samples for DO Determination

Samplers are designed to ensure that air cannot enter into the sample. Most samplers are designed to retain 3-4 times the volume of samples which is required for analysis purposes. As oxygen values change with time due to any biological activity, it is important to fix it in field immediately after collection. This is done using reagents using in DO test and then the titration is done in laboratory. This method gives low results for samples with high iodine demand and in this case it is better to preserve sample using 0.7 mL concentrated sulphuric acid and 0.02 g sodium azide. When this is doe it is necessary to add 3 mL of alkali-iodide reagent rather than the usual 2 mL because of the extra acid the sample contains. Better results are also obtained if the sample is fixed and stored in the dark and on the ice until the analyses are conducted. The final titration should not be delayed more than 6 hours.

APPARATUS REQUIRED:

Incubation bottle 300mL volume; Air compressor

REAGENTS:

1) Manganese sulphate solution: Dissolve 480 g $MnSO_4.4H_2O$, 400 g $MnSO_4.2H_2O$ or 364g $MnSO_4.H_2O$ in distilled water, filter, and dilute to 1L. The $MnSO_4$ solution should not give a color with starch when added to an acidified potassium iodide (KI) solution.
2) Alkali-iodide-azide reagent
3) Sulphuric acid: One mL is equivalent to ~ 3mL alkali-iodide-azide reagent.
4) Starch solution: Dissolve 2 g laboratory-grade soluble starch and 0.2 g salicyclic acid as preservative in 100 mL hot distilled water.
5) Standard sodium thiosulphate titrant: Dissolve 6.205 g $Na_2S_2O_3 .5H_2O$ in distiller water and add 1.5 mL 6N NaOH or 0.4 g solid NaOH and dilute to 1000 mL. Standardize with bi-iodate solution.
6) Standard potassium bi-iodate solution (0.0021M): Dissolve 812.4 mg $KH(IO_3)$ in distilled water and dilute to 1000 mL.
7) Standardization: Dissolve e ~ 2 g KI, free from iodate in an Erlenmeyer flask with 100 to 150 mL distilled water; add 1 mL 6N H_2SO_4 or a few drops of conc. H_2SO_4 and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate librated iodine with thiosulphate titrant, adding starch toward end of titration, when a pale straw color is reached. When the solution is of equal, 20.00 mL 0.025M $Na_2S_2O_3$ should be required. If not, adjust the $Na_2S_2O_3$ solution to 0.025M.

PROCEDURE:

1. Make dilution water by adding 2mL/L of following reagents in distilled water:
    a. Phosphate buffer solution
    b. Magnesium sulphate solution
    c. Calcium chloride solution
    d. Ferric chloride solution
    e. Sodium Sulphite solution
2. 2) Take 300 mL sample in BOD bottle. Prepare two sets of this sample. Keep one set for DO analysis for day 0 (i.e., Sample0Day) and another sample in BOD incubator for 5 days at 20°C (Sample5Day) (this is how 5-day BOD experiment is done). Here you will prepare duplicate samples and analyze at Day 0 (i.e., Sample0Day_A and Sample0Day_B).
3. 3) For a given sample bottle, add 1 mL of alkali azide and then 1 mL manganeous sulphate solution. Shake well the bottle and keep it open for 5 minutes to settle the precipitate. Add 2 mL concentrated H_2SO_4 and place the cap on the bottle. Shake well the bottle till all the precipitate is dissolved.
4. 4) Take 203 mL of sample in conical flask and titrate with standard sodium thiosulphate solution (0.025N) till the colour changes from dark yellow to light yellow. Then add few drops of starch indicator and continue to titrate till the color of the solution becomes either colorless or changes to its original sample colour. Note down volume of 0.025N sodium thiosulphate consumed.
5. 5) Calculate DO value of the sample.

Remember that in 200 mL sample, 1 mL of sodium thiosulphate of 0.025N equals to 1 mg/L dissolved oxygen:

=>Dissolved oxygen (DO) (in mg/L) = mL of sodium thiosulphate (0.025N) consumed.
Notes: Dilution of Sample
1. 0.1, 0.5, and 1% for strong waste water
2. 1.0, 2.5, and 5% for raw and settled sewage
3. 5.0, 12.5 and 25% for oxidized effluent
4. 25, 50 and 100% for polluted river water

OBSERVATION:

CONCLUSION:

REMARKS

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TO DETERMINE THE AMOUNT OF TOTAL RESIDUAL CHLORINE PRESENT IN THE GIVEN SAMPLE OF CHLORINATED WATER BY STARCH IODIDE METHOD

THEORY:

Chlorine will liberate free Iodine from Potassium Iodide solution at PH 8.0 or less. The liberated Iodine is titrated against standard sodium thiosulphate with starch as indicator

APPARATUS REQUIRED:

1. Burette

2. Pipette

3. Erlenmeyer flask

REAGENTS:

1. Concentrated Acetic acid

2. Potassium Iodide

3. Sodium Thiosulphate (0.025N)

4. Starch solution

5. Iodine solution (0.025N)

PROCEDURE:

1. Take 25 ml of sample in an Erlenmeyer flask

2. Add 5ml of Acetic acid to bring PH 3.0 to 4.0

3. Add 1gm of potassium iodide and mix thoroughly. Yellow colour is obtained

4. Titrate against standard sodium thiosulphate solution in the burette until a pale yellow colour is obtained

5. At these stages add 1ml of starch indicator and continue the titration till the blue colour disappears. Note down the volume (vV1)

RESULT:

Turbidity of sample =

CONCLUSION:

REMARKS:

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MEASURE (1) TOTAL HARDNESS AND (2) CALCIUM HARDNESS USING DYE INDICATORS

THEORY:

Hard waters are generally considered to be those waters that require considerable amounts of soap to produce foam and that also produce scale in water pipes, heaters, boilers and other units in which the temperature of water is increased. Hard water are appropriate for human consumption similar to that as soft waters, however it produces adverse actions with soap and thus their use for cleaning purposes is unsatisfactory and thus their removal from water is required. Hardness of waters varies from place to place. In general, surface waters are softer than ground waters. Waters are commonly classified based on degree of hardness (Table 1):

Table 1. Classification of hardness types

Hardness(mg/L)	Degree of hardness
0-75	Soft
75-100	Moderately hard
150-300	Hard
>300	Very hard

Hardness:

Hardness is caused by polyvalent metallic cations, though the divalent cations, such as calcium and magnesium cations are usually the predominant cause of hardness. In addition, hardness is also caused by Fe2+ and Mn2+ ions. For example, when hard water is heated, Ca2+ ions react with bicarbonate (HCO3-) ions to form insoluble calcium carbonate (CaCO3) (Eq. 1). This precipitate, known as scale, coats the vessels in which the water is heated, producing the mineral deposits on your cooking dishes. Equation 2 presents magnesium hardness.

$$Ca^{+2}_{(aq)} + 2HCO^{-}_{3(aq)} \rightarrow CaCO_{3\,(s)} + H_20 + CO_2 \qquad (1a)$$

$$Mg^{+2}_{(aq)} + 2OH^{-}_{(aq)} \rightarrow MgOH_{2(s)} \qquad (2a)$$

Total hardness is defined as the sum of the calcium and magnesium concentrations, both expressed as calcium carbonate in mg/L. When hardness (numerically) is greater than the sum of carbonate and bicarbonate alkalinity, amount of hardness equivalent to the total alkalinity is called “Carbonate hardness”.

Carbonate hardness (mg/L) = Alkalinity (2a)

When alkalinity > Total hardness:

Carbonate hardness (mg/L) = Total hardness (2b)

The amount of hardness in excess of this is called "Non-carbonate hardness (NCH)". These are associated with sulphate chloride, and nitrate ions. It is calculated using Eq (2c):

NCH (mg/L) = Total hardness-Carbonate hardness (2c)

Determination of Hardness:

Hardness is expressed as mg/L CaCO3. The first method is calculation based method and the second method is titration method using EDTA.

(i) Calculation method

For this method, concentration of cations should be known and then all concentrations are expressed in terms of CaCO3 using Eq. 3:

Hardness (in mg/L as CaCO3) = [M2+ (in mg/L) × 50]/ (E.Wt. of M2+) (3)

Where: M2+ = mass of divalent ions (mg/L) and E.Wt. = Equivalent weight of divalent ions (g/mole)

PROCEDURE:

Reagents: Buffer solution; EDTA Titrant; EBT

1. Measure Ca-Hardness and Total Hardness by titration as described below. Use a different sample for each measurement.

2. Total Hardness: Take 100 ml of the sample and add 2 ml buffer solution in it and add 2-3 drops of Black T. Titrate it with standard EDTA solution (with continuous stirring) until the last reddish colour disappears. At the end point the solution turns blue. Note down the volume used.

Calculate Hardness as follows:

Hardness (in mg/L as CaCO3) = (V× N × 50 ×1000) / (SV) (5)

Where: V = volume of titrant (mL); N = normality of EDTA; 50 = equivalent weight of CaCO3; SV = sample volume (mL)

3. Ca-Hardness: Take 50 ml of the sample and add 1 ml Sodium Hydroxide solution (8%) in it and add pinch of Mercurex Powder. Titrate with standard EDTA solution until the light pink colour of solution converts into light blue colour.

Answer these questions also: 1. Among finished drinking water, raw wastewater and de-ionized water, which water is expected to have the highest carbonate hardness and why?

2. A sample has 50mg/L Ca2+,150mg/L Mg2+ 50 mg/L Na+, 20 mg/L Cl- and 100 mg/L glucose. Calculate its total hardness, carbonate and non-carbonate hardness?

CONCLUSION:

RESULTS:

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DETERMINE THE BOD VALUE FOR DETERMINING BIODEGRADABILITY OF SOLUTION

THEORY:

The most widely used test indicating organic pollution of both wastewater and surface water is the 5-day BOD (BOD5). This determination involves the measurement of the dissolved oxygen used by microorganisms in the biochemical oxidation of organic matter. BOD5 is the total amount of oxygen consumed by microorganisms during the first five days of biodegradation. Oxygen demand is associated with the biodegradation of the carbonaceous portion of wastes and oxidation of nitrogen compounds such as ammonia. The following equations simplify the process of biodegradation:

Organic matter + O2 + microorganisms CO2 + H2O + new microbial cells

Ammonia + O2 + microorganisms NO3 + H2O + new microbial cells:

APPARATUS REQUIRED:

Incubation bottle 300mL volume; Air compressor, 20°C incubator

REAGENTS REQUIRED:

1. Manganese sulphate solution: Dissolve 480 g MnSO4.4H2O, 400 g MnSO4.2H2O or 364 g MnSO4.H2O in distilled water, filter, and dilute to 1L. The MnSO4 solution should not give a colour with starch when added to an acidified potassium iodide (KI) solution.

2. Alkali-iodide-azide reagent

3. Sulphuric acid: One mL is equivalent to ~ 3mL alkali-iodide-azide reagent.

4. Starch solution: Dissolve 2 g laboratory-grade soluble starch and 0.2 g salicylic acid as preservative in 100 mL hot distilled water.

5. Standard sodium thiosulphate titrant: Dissolve 6.205 g Na2S2O3 .5H2O in distiller water and add1.5 mL 6N NaOH or 0.4 g solid NaOH and dilute to 1000 mL. Standardize with bi-iodatesolution.

6. Standard potassium bi-iodate solution (0.0021M): Dissolve 812.4 mg KH(IO3) in distilled water and dilute to 1000 mL.

7. Standardization: Dissolve e ~ 2 g KI, free from iodate in an Erlenmeyer flask with 100 to 150mL distilled water; add 1 mL 6N H2SO4 or a few drops of conc. H2SO4 and 20.00 mL standard bi-iodate solution. Dilute to 200 mL and titrate librated iodine with thiosulphate titrant, adding starch toward end of titration, when a pale straw colour is reached. When the solution is of equal, 20.00 mL 0.025M Na2S2O3 should be required. If not, adjust the Na2S2O3 solution to 0.025M.

PROCEDURE:

DO measurement:

1. Make dilution water by adding 2mL/L of following reagents in distilled water:

a. Phosphate buffer solution

b. Magnesium sulphate solution

c. Calcium chloride solution

d. Ferric chloride solution

e. Sodium Sulphite solution

2. For a given sample bottle, add 1 mL of alkaliazide and then 1 mL manganous sulphate solution. Shake well the bottle and keep it open for 5 minutes to settle the precipitate. Add 2 mL concentrated $H_2SO_4$and place the cap on the bottle. Shake well the bottle till all the precipitate is dissolved.

3. Take 203 mL of sample in conical flask and titrate with standard sodium thiosulphate solution (0.025N) till the colour changes from dark yellow to light yellow. Then add few drops of starch indicator and continue to titrate till the colour of the solution becomes either colourless or changes to its original sample colour. Note down volume of 0.025N sodium thiosulphate consumed.

4. Calculate DO value of the sample. Remember that in 200 mL sample, 1 mL of sodium thiosulphate of0.025N equals to 1 mg/L dissolved oxygen:

=>Dissolved oxygen (DO) (in mg/L) = mL of sodium thiosulphate (0.025N) consumed.

BOD:

1. Prepare BOD dilutions. Use dilution water (it contains nutrients, the exact O_{sample} in 300 mL BOD bottle, fill up with dilution water;15 mL sample in 300 mL BOD bottle, fill up with dilutionwater;20 mL sample in 300 mL BOD bottle, fill up with dilution water

2. Take 300 mL sample in BOD bottle. Prepare two sets of this sample. Keep one set for DO analysis for day 0 (i.e., Sample0Day) and another sample in BOD incubator for 5 days at 20°C (Sample5Day).

3. Measure DO in different samples at t=0.

4. Incubate samples in 20oC for 5 days.

5. Come back in the lab after 5 days and record dissolved oxygen.

6. Record data in following manner

Bottle no.	Wastewater sample (mL)	Initial DO (mg/L) (DO_O)	DO at 5-day (mL) (DO_5)
1			
2			
3			
4			

Calculate 5-day BOD value of the sample at 20°C:

t-day BOD= [DOt-DO0]/(P) (1)

where P= Dilution factor = 300mL/(sample volume in mL)

CONCLUSION:

REMARKS:

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DETERMINE COD VALUE FOR DETERMINING ORGANIC STRENGTH OF SOLUTION (CLOSED REFLUX METHOD)

THEORY:

Chemical oxygen demand (COD) is termed as the amount of a specific oxidizing agent that reacts with sample under controlled conditions and it is expressed as oxygen equivalence. This parameter indicates the extent of organic matter contamination of water and is always higher than the biochemical oxygen demand (BOD). It is used to indicate organic matter contamination and it helps in knowing overall organic load to the receiving body.

Selection of Method

There are two methods for COD determination. The first method: open reflux method is suitable for a wide range of wastes where large volume of sample is required (for samples with COD=50 mg O_2/L). In the second method: closed reflux methods, small quantities of metallic salt reagents are required and small quantities of hazardous waste is produced (for samples with COD= 5 to 50 mgO_2/L). In the closed reflux method, ampules and culture tubes with premeasured reagents are used and then samples is placed in the tube and COD is determined.

In this experiment, closed reflux method is used and samples with COD < 50 mg O_2/L are tested.

Reaction with dichromate solution of sample:

Potassium dichromate is a strong oxidizing agent and it can be used to prepare solution of exact normality.

$C_nH_aO_bN_c + d\ Cr_2O_7^{2-} + (8d+c)\ H^+ \Rightarrow nCO_2 + [(a+8d-3c)/2]H_2O + c\ NH_4^+ + 2dCr^{3+}$ (1)

Here d= (2n/3) + (a/6)-(b/3)-(c/2)

During experiment, excess dichromate concentration is determined by titrating it with ferrous ammonium sulphate (FAS). The reaction is given by:

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

Here d= (2n/3) + (a/6)-(b/3)-(c/2)

Ferroin (ferrous 1, 10-phenanthroline sulphate): It is used to indicate change in oxidation-reduction potential of the solution and it indicates the condition when all dichromate has been reduced by ferrous ion. It gives a very sharp brown colour change which can be seen in spite of blue colour generated by the Cr^{3+} ions formed on reduction of the dichromate.

APPARATUS REQUIRED:

1. Digestion vessels;
2. block heater;
3. microburette; ampule sealer.
4. Borosilicate culture tubes (16mm*100 mm or 20 mm*150mm) with TFE lined-screw caps are used.

The block heater is required to operate at 150±2°C with holes to accommodated digestion vessels. Do not use an oven because of the possibility of leaking samples generating corrosive and explosive atmosphere.

REAGENTS:

a. Standard potassium dichromate digestion solution, 0.01667M: Add to about 500 mL distilled water 4.903 g $K_2Cr_2O_7$, primary standard grade, previously dried at 150°C for 2 h, 167 mL conc. H_2SO_4 , and 33.3 g $HgSO_4$. Dissolve, cool to room temperature, and dilute to 1000 mL.
b. Sulfuric acid reagent:
c. Ferroin indicator solution: Dilute it by a factor of 5 as required. This indicator is used to indicate change in oxidation-reduction potential of the solution.
d. Standard ferrous ammonium sulfate titrant (FAS), approximately 0.10M: Dissolve 39.2 g $Fe(NH_4)_2(SO_4)_2.6H_2O$ in distilled water. Add 20 mL conc. H_2SO_4, cool, and dilute to 1000 mL. Standardize solution daily against standard $K_2Cr_2O_7$ digestion solution as follows: Pipet 5.00 mL digestion solution into a small beaker. Add 10 mL reagent water to substitute for sample. Cool to room temperature. Add 1 to 2 drops diluted ferroin indicator and titrate with FAS titrant.

   Molarity of FAS solution = $[V_{K2Cr2O7} \times 0.1] / (V_{FAS})$ (3)

   Where**:** $\mathbf{V_{K2Cr2O7}}$= volume of $K_2Cr_2O_7$ (mL); $\mathbf{V_{FAS}}$ = volume of FAS (mL)

e. Sulphamic acid:

f. Potassium hydrogen phthalate standard:

PROCEDURE:

1. Wash culture tubes and caps with 20% H2SO4 before using to prevent contamination.

2. Place sample in culture tube or ampule and add digestion solution. Carefully run sulphuric acid reagent down inside of vessel so an acid layer is formed under the sample-digestion solution layer and tightly cap tubes or seal ampules, and invert each several times to mix completely.

3. Place tubes or ampules in block digester preheated to 150°C and reflux for 2h behind a protective shield. CAUTION: These sealed vessels may be under pressure from gases generated during digestion. Wear face and hand protection when handling and dangerous pressures will be generated at 150°C.

4. Cool to room temperature and place vessels in test tube rack. Some mercuric sulphate may precipitate out but this will not affect the analysis.

5. Remove culture tube caps and add small TFE-covered magnetic stirring bar. If ampules are used, transfer contents to a larger container for titrating.

6. Add 0.05 to 0.10 mL (1 to 2 drops) ferroin indicator and stir rapidly on magnetic stirrer while titrating with standardized 0.10M FAS. The end point is a sharp colour change from blue-green to reddish brown, although the blue green may reappear within minutes. In the same manner reflux and titrate a blank containing the reagents and a volume of distilled water equal to that of the sample.

7. COD is given by

COD as mg/L O_2/L = [(A-B) × M ×8000) / (V_{sample}) (4)

Where: A = volume of FAS used for blank (mL);

B= volume of FAS used for sample (mL);

M=molarity of FAS; 8000= miliquivalent weight of oxygen ×1000 mL/L.

CONCLUSION:

REMARKS:

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DETERMINE THE TURBIDITY OF THE GIVEN SAMPLE USING NEPHELOMETER IN N.T.U.

THEORY:

Turbidity can be measured either by its effects on the transmission of light which is termed as turbidimetry or its effects on the scattering of light which termed as Nephelometry. Turbidimetry can be used for sample with moderate turbidity and Nephelometer for sample with low turbidity. Higher the intensity of scattered light higher the turbidity.

APPARATUS REQUIRED:

1) Nephelometric turbidimeter
2) Cuvettes; it take the samples for measurements

REAGENTS:

• Solution (1) Dissolve 1 g hydrazine sulphate in distilled water and dilute to 100 ml in volumetric flask
• Solution (2) dissolve 10g hexamine LR grade in distilled water and dilute to 100ml in volumetric flask
• In 100ml volumetric flask, mix 12.5 ml solution (1) and 12.5 ml
solution (2) let them stand for 24 hours at 250 dilute to mark and mix the turbidity of the suspension is 1000 NTU.

PROCEDURE:

CALIBRATION:

• Switch on the instrument and keep it on for some time
• Select appropriate range depending upon the expected turbidity of the sample.
• Set zero of the instrument with turbidity free water using a blank solution and adjust 000 with set zero knob.
• Now in another test tube take standard suspension just prepared as above for 0 – 200 NTU solution as standard.
• Take its measurements and set the display to the value of the standard suspension with the calibrate knob.

MEASUREMENTS:

To determine the turbidity of water sample place the sample in the cuvette and note the displayed reading .if water has high turbidity it can be suitably diluted and must be shaken before determination.

CALCULATION:

Turbidity = A (B + C) / C
A = NTU found in diluted sample
B = volume of dilution water
C = sample of volume taken for dilution

RESULT: Turbidity of sample =

CONCLUSION:

REMARKS:

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DETERMINE THE ALKALINITY OF GIVEN SAMPLE OF WATER IN MG/L.

THEORY:

Alkalinity is determined by titrating the sample with a standard solution of a strong mineral acid to bicarbonate and carbonic acid equivalence point. Alkalinity is expressed in terms of CaCO3 equivalent. For samples whose PH is above 8.3, titration is done in two steps. In the first step the PH is lowered to 8.3, which is indicated by phenolphthalein indicator losing the pink colour and becoming colourless. In the second phase of titration the PH is lowered to about 4.5, which is indicated by methyl orange indicator changing colour from yellow to orange red.

APPARATUS REQUIRED:

1. Burette
2. Pipette
3. Erlenmeyer flask

REAGENTS:

1. Sulphuric acid 0.02N
2. Sodium thiosulphate 0.1

PROCEDURE:

1. Take 20 ml of the given sample in Erlenmeyer flask (v)
2. Add 1 drop of 0.1N sodium thiosulphate solution to remove the free chlorine if present
3. Add 2 drops of phenolphthalein indicator. The sample turns pink if the PH is above 8.3
4. Run down 0.02N standard sulphuric acid till the solution turn to colourless
5. Note down the volume of H2SO4added (v1)
6. Add 2 drops of methyl orange indicator the sample turns yellow
7. Repeat titration till the colour of the solution turns to orange
8. Note down the total volume of H2SO4 added (v2)

OBSERVATIONS AND CALCULATIONS:

Sl no.	Sample no.	Volume of sample (v)	Initial burette reading(ml)	Final burette reading(ml)	Volume of H2SO4 (ml)	Alkalinity (mg/l)

Phenolphthalein alkalinity expressed as mg/l (CaCO3)

P= (V1X50X1000X0.02N)/vol.of sample used (ml)

Methyl orange alkalinity expressed as mg/l ($CaCO_3$)

M= (V2X50X1000X0.02N)/vol.of sample used (ml)

Total alkalinity expressed as mg/l ($CaCO_3$)

T= (V3X50X1000X0.02N)/vol.of sample used (ml)

RESULT:

Phenolphthalein alkalinity expressed as mg/l ($CaCO_3$) =
Methyl orange alkalinity expressed as mg/l ($CaCO_3$) =
Total alkalinity expressed as mg/l ($CaCO_3$) =

CONCLUSION:

REMARKS: