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LAB MANUAL

Regulation	: 2013
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CE 6611 - ENVIRONMENTAL ENGINEERING LABORATORY

ANNA UNIVERSITY CHENNAI

REGULATION 2013
CE6611 – ENVIRONMENTAL ENGINEERING LABORATORY

LIST OF EXPERIMENTS:

1. Determination of Ammonia Nitrogen in Wastewater.

2. Coagulation and Precipitation Process for Treating Wastewater

3. Determination of Suspended, Volatile, Fixed and Settleable Solids in Wastewater.

4. B.O.D. Test

5. C.O.D. Test

6. Nitrate in Wastewater.

7. Phosphate in Wastewater.

8. Determination of Calcium, Potassium and Sodium.

9. Heavy metals determination - Chromium, Lead and Zinc   .
   (Demonstration only)

Total Period= 45

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2.		Coagulation and Precipitation Process for Treating Waste Water		
3.		Determination of Total Suspended, Volatile, Fixed and Dissolved Solids in Wastewater		
4.		Determination of Bio-Chemical Oxygen Demand (B.O.D Test)		
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SAMPLING

OBJECTIVE:

The objective of sampling is to collect representative sample. Representative sample by means a sample in which relative proportions or concentration of all pertinent components will be the same as in the material being sampled. Moreover, the same sample will be handled in such a way that no significant changes in composition occur before the tests are made. The sample volume shall optimal small enough that it can be transported and large enough for analytical purposes.

Because of the increasing placed on verifying the accuracy and representatives of data, greater emphasis is placed on proper sample collection, tracking, and preservation techniques. Often laboratory personnel help in planning a sampling program, in consultation with the user of the test results. Such consultation is essential to ensure selecting samples and analytical methods that provide a sound and valid basis for answering the questions that prompted the sampling and that will meet regulatory and/or project-specific requirements.

TYPES OF SAMPLE:

1. GRAB SAMPLES:

Grab samples are single collected at a specific spot at a site over a short period of time (typically seconds or minutes). Thus, they represent a "snapshot" in both space and time of a sampling area. Discrete grab samples are taken at a selected location, depth, and time. Depth-integrated grab samples are collected over a predetermined part of the entire depth of a water column, at a selected location and time.

2. COMPOSITE SAMPLE:

The term composite refers to a mixture of grab samples collected at the sample sampling point at different times. Composite samples cannot be used for determination of components or characteristics subject to significant and unavoidable changes on storage. A composite sample of 24 hrs period considered standard for most determinations it provides more meaningful data than the grab samples

3. INTEGRATED SAMPLE:

The mixture of grab sample collected from different points simultaneously or as nearly as possible is called integrated sample. The preparation of integrated sample require special equipment to collect samples from a known depth without contamination by overlying waste water.

SAMPLING FREQUENCY:

The waste water samples should be collected at intervals so that no change in quality could pass un noticed. It depends on the type of data required, purpose of monitoring, availabilities of funds and personnel's. Number of samples and how often samples should be collected are calculated by statistical consideration.

SAMPLE COLLECTION:

Whenever possible the container should be rinsed out two or three times with the sample to be examined before finally filled. The stopper should not be laid down, but kept in hand before the reinserted. Sample preferably at 20cm depth in a shallow channel. Depth greater than 20cm collect 2 samples at 20% and 80% below the surface.

FIELD MEASURED PARAMETERS:

A number of parameters including **pH**, conductivity, dissolved oxygen, ammonia, carbon di-oxide, temperature, turbidity and heavy metals should be measured.

PRESERVATION OF SAMPLES:

Each sampling bottle must be provided with a identification label with necessary information. once the sample is collected, some physical changes, chemical changes and biological and biochemical reactions may takes place in the sample container which will change the quality of the sample. It is necessary to preserve the sample before transporting to minimize their changes. This can be done by keeping the samples in dark, adding chemical preservatives, lowering the temperature to retard reactions by freezing or by a combination of these methods.

TRANSPORTATION OF SAMPLES:

Sample containing bottle should be placed in a box for transportation to the laboratory. It should be insulated wooden or plastic boxes which will protect samples from sunlight prevent the breakage of bottles and should allow a temperature of **4** degree attained and maintained during transport.

SHORT PROCEDURE:(DETERMINATION OF AMMONIA NITROGEN IN WASTEWATER)

S.NO.	CONTENT	REAGENT
1.	Burette solution	Sulphuric acid
2.	Pipette solution	20 *ml* of Waste water sample
3.	Additional solutions	2 *ml* of phosphate buffer solution and 10 *ml* of boric acid
4.	Indicator	Methyl orange indicator.
5.	End point	Change of colour to yellow to orange.

TABULATION:

S.No.	Volume of given sample *ml*	Burette readings *ml*		Concurrent burette reading *ml*	Volume of Sulphuric acid *ml*
		Initial	Final		

CALCULATION:

$$\text{Ammonia Nitrogen in mg/litre} = \frac{Volume\ of\ H_2SO_4 consumed \times 0.28 \times 1000}{Volume\ of\ sample\ taken}$$

Exp. No: 1

Date:

DETERMINATION OF AMMONIA NITROGEN IN WASTEWATER

AIM:

To determine the amount of Ammonia Nitrogen present in the given sample.

PRINCIPLE:

Ammonium ion reacts with Nesslers reagent **(K$_2$HgI$_4$)** to form a brown colour substance, and can be determined colorimentically. Most of the natural waters and wastewaters have interfering substances, therefore, the steam distillation of ammonia becomes essential.

APPARATUS REQUIRED:

1. Measuring jar
2. Conical flask
3. Burette
4. Pipette

REAGENTS:

1. Phosphate buffer solution
2. Boric acid
3. Methyl orange indicator
4. Sulphuric acid **0.02N** (1 ml contains 0.28 mg of nitrogen)

PROCEDURE:

1. Take 20ml of the sample in a conical flask.
2. Add 2ml of Phosphate buffer solution and 10 ml of Boric acid solution.
3. Add 3 -5 drops of Methyl orange indicator.
4. Titrate against 0.02N of Sulphuric acid till the end point is changes from yellow to orange.

RESULT:

Amount of Ammonia Nitrogen present in the given sample is _____*mg/l.*

OBSERVATION:[COAGULATION AND PRECIPITATION PROCESS FOR TREATING WASTE WATER]

Trial No.	Initial turbidity NTU	Ferric chloride dosage *ml*	Final turbidity NTU	Efficiency %

CALCULATION:

Efficiency of the Ferric chloride dosage $= \dfrac{Initial\ turbidity - Final\ turbidity}{Initial\ turbidity} \times 100$

Exp. No: 2

Date:

COAGULATION AND PRECIPITATION PROCESS FOR
TREATING WASTE WATER

GENERAL:

Chemical coagulation is an important process applied extensively in water treatment practice, particularly, where surface supplies are involved chemical coagulation is performed to remove turbidity, true and apparent colour, harmful bacteria and other pathogens algal and other plankton organisms and taste and odour producing substances.

The laboratory studies of chemical coagulation are often required to determine the best chemical or combination of chemicals and amounts needed to get a desired objective in water, sewage and industrial waste treatment practice. The result obtained serve as the basis of design and operation of treatment facilities.

AIM:

To determine the optimum dosage of Alum required for turbidity.

APPARATUS:

1. Jar test apparatus
2. Glass beakers
3. Pipette
4. Nephelometer
5. **pH** meter

REAGENTS:

Ferric chloride solution

PROCEDURE:

1. Measure the initial **pH** and turbidity for whole sample.

2. Measure **500 ml** quantities of above wastewater sample into each of **6** beakers in series.

3. Keep each beaker below each paddle and lower the paddles, such that each one is about 1cm above the bottom.

4. Pipette **1, 1.5, 2, 2.5, 3, 3.5 ml** of the Ferric chloride solution into the test samples.

5. Immediately run the paddles at 100 rpm for 1 minute.

6. Reduce the speed to 30-40 rpm and run at this rate for 10 minutes.

7. Stop the machine, lift out the paddles and allow for 2 minutes.

8. Carefully take a small amount (about 100 ml) of settled waste water from top of each beaker without disturbing the settled sediments.

9. Find the residual turbidity of the supernatant using Nephelometer.

10. Plot a graph with Ferric chloride dosage along x-axis and turbidity along y-axis.

11. The dosage of alum, which represents least turbidity, gives optimum coagulant dosage.

12. Repeat steps 1-10 with higher dose of alum, if necessary.

RESULT:

        Optimum coagulant dosage _____

        Efficiency _____

OBSERVATION:[DETERMINATION OF TOTAL SUSPENDED, VOLATILE, FIXED AND DISSOLVED SOLIDS IN WASTEWATER]

SL. NO.	ITEM		SAMPLES
1.	Volume of sample taken	*ml*	
2.	Wt. of empty evaporating dish = W1 (For total dissolved solids)	*mg*	
3.	Wt. of dish + total solids = W2	*mg*	
4.	Total solids = (W2 – W1)	*mg*	
5.	Wt. of dish + fixed solids = W3	*mg*	
6.	Fixed solids in mg = (W3 – W1)	*mg*	
7.	Wt. of empty evaporating dish = W4 (For total dissolved solids)	*mg*	
8.	Wt. of dish + total dissolved solids = W5	*mg*	
9.	Total dissolved solids = (W5 – W4)	*mg*	
10.	Total solids	*mg/l*	
11.	Total fixed solids	*mg/l*	
12.	Total dissolved solids	*mg/l*	
13.	Total suspended solids	*mg/l*	
14.	Total volatile solids	*mg/l*	

CALCULATION:

1. *mg/l* of total solids= $\dfrac{mg\ of\ total\ solids \times 1000}{ml\ of\ sample}$

2. *mg/l* of total fixed solids= $\dfrac{mg\ of\ total\ fixed\ solids \times 1000}{ml\ of\ sample}$

3. *mg/l* of total dissolved solids= $\dfrac{mg\ of\ total\ dissolved\ solids \times 1000}{ml\ of\ sample}$

4. *mg/l* of total suspended solids = *mg/l* of total solids – *mg/l* of total dissolved solids

5. *mg/l* total volatile solids = *mg/l* of total solids – *mg/l* of total fixed solids

Exp. No:3

Date:

DETERMINATION OF TOTAL SUSPENDED, VOLATILE, FIXED AND DISSOLVED SOLIDS IN WASTEWATER

AIM:

The aim of the experiments is to determination of suspended, volatile, fixed and settleable solids in wastewater.

APPARATUS:

1. Porcelain evaporating dishes of **150–200 ml** capacity
2. Steam bath
3. Drying oven
4. Desiccators
5. Analytical balance or monopan balance
6. Filter paper (preferably of glass fibre)
7. Electric muffle furnace
8. Imhoff cone

PRINCIPLE:

Total solids is the term applied to the material left in the vessel after evaporation of a sample of water/waste water and its subsequent drying in an oven at a definite temperature. Fixed solids is the residue remaining after ignition for 1 hour at **550°C**. The solid portion that is volatilised during ignition is called volatile solids. It will be mostly organic matter. Waters that are low in organic matter and total mineral content and are intended for human consumption may be examined under **103–105°C** or **179–181°C**. But water containing considerable organic matter or those with pH over 9.0 should be dried at **179–181°C**. In any case, the report should indicate the drying temperature. The sample is filtered and the filtrate evaporate in a weighed dish on a steam bath, the residue left after evaporation is dried to constant weight in an oven at either *103–105°C* or **179–181°C**. The increase in weight over that of the empty dish represents total dissolved solids and includes all materials, liquid or solid, in solution or otherwise, which passes through the filter and not volatilised during the drying process. The difference between the total solids and the total dissolved solids will give the total suspended solids. The dishes with the residue retained after completion of the tests

for total solids and total dissolved solids are subjected to heat for **1 hour** in a muffle furnace held at **550°C**. The increase in weight over that of the ignited empty vessel represents fixed solids in each instance.

The difference between the total dissolved/total suspended solids and the corresponding fixed solids will give volatile solids in each instance. All the quantities should be expressed in mg/l. Settleable matter in surface and saline waters as well as domestic and industrial wastes may be determined and reported on a volume basis as ml per litre.

PROCEDURE:

Total Solids:

1. Ignite the clean evaporating dishes in the muffle furnace for **30 minutes** at **550°C** and cool in a desiccator.
2. Note down the empty weight of the dish **(W1)**.
3. Pour a measured portion **(10 ml)** of the well-mixed sample into the dish and evaporate the contents by placing the dish on a steam bath.
4. Transfer the dish to an oven maintained at either **103–105°C** or **179–181°C** and dry it for **1 hour**.
5. Allow the dish to cool briefly in air before placing it, while still warm in a desiccator to complete cooling in a dry atmosphere.
6. Weigh the dish as soon as it has completely cooled **(W2)**.
7. Weight of residue = **(W2 – W1)**.

Total fixed solids:

1. Keep the same dish used for determining total residue in a muffle furnace for **15minutes** at **550°C**.
2. Allow the dish to partially cool in air until most of the heat has dissipated, then transfer to a desiccators for final cooling in a dry atmosphere.
3. Weigh the dish as soon as it has cooled **(W3).**
4. Weight of total fixed residue = **(W3 – W1)**.

Total dissolved solids:

1. Filter a measured portion of the mixed sample (**10 ml**) through a filter paper and collect the filtrate in a previously prepared and weighed evaporating dish.

2. Repeat the steps 3 to 6 outlined in total solids procedure.

3. Weight of dissolved solids = (**W5 – W4**).

   W4 = Weight of empty evaporating dish in mg.

   W5 =Weight of empty evaporating dish in mg + Residue left after evaporating the filtrate in mg.

Total suspended solids:

Total solids – Total dissolved solids.

Total volatile solids:

Total solids – Total fixed solids.

RESULT:

1. Total solids   =_____*mg/l*

2. Fixed solids   =_____*mg/l*

3. Dissolved solids   =_____*mg/l*

4. Suspended solids   =_____*mg/l*

5. Volatile solids   =_____*mg/l*

SHORT PRECEDURE: [DETERMINATION OF BIO-CHEMICAL OXYGEN DEMAND (B.O.D TEST)]

S.NO	CONTENT	REAGENT
1.	Burette solution	Sodium thio sulphate
2.	Pipette solution	50ml of prepared waste water sample
3.	Additional solutions	1 ml of Manganese Sulphate solution is added, followed by 1 *ml* of Alkali-Iodide. After 5 minutes add slowly 1 *ml* of Conc.Sulphuric Acid.
4.	Indicator	starch indicator.
5.	End point	Disappearance of blue colour.

CALCULATION:

DO in (mg/l) = $(V_2 \times N \times 8 \times 1000)/V_1$

V_1 = Volume of water sample in *ml*.

V_2 = Volume of Sodium Thiosulphate consumed in *ml*.

N = Normality of sodium Thiosulphate

Final D.O.:

S.No	Volume of given sample ml	Burette readings ml		Concurrent burette reading ml	Volume of Sodium Thiosulphate ml
		Initial	Final		
1.					
2.					

Exp. No:4

Date:

DETERMINATION OF BIO-CHEMICAL OXYGEN DEMAND
(B.O.D TEST)

AIM:

To determine the BOD in the given wastewater sample.

PRINCIPLE:

If sufficient oxygen is available in wastewater, the useful aerobic bacteria will flourish and cause the aerobic biological decomposition of wastewater which will continue until oxidation is completed.

The amount of oxygen consumed in this process is the BOD. Polluted waters will continue to absorb oxygen for many months, and it is not practically feasible to determine this ultimate oxygen demand.

APPARATUS REQUIRED:

1. BOD incubator
2. BOD bottle (300*ml*)
3. Conical flask
4. Burette
5. Measuring jar

REAGENTS REQUIRED:

1. Sodium thio-suiphateb(**0.01N**)

2. Manganous Sulphate

3. Alkai- iodide

4. Conc.**H_2SO_4**

5. Starch

PROCEDURE:

1. Distilled water is aerated for 4 hours to attain saturated Dissolved Oxygen (DO) level. In distilled water **1 *ml*** of each nutrients (Phosphate buffer, Magnesium Sulphate, Calcium Chloride and Ferric Chloride) and 1 *ml* of pre acclimatized seed per 1 litre of distilled water is added.

CALCULATION: [DETERMINATION OF BIO-CHEMICAL OXYGEN DEMAND (B.O.D TEST)]

BOD$_5$ (*mg/l*) = [(Initial DO – Final DO) x dilution factor]

Where,

$$\text{Dilution factor} = \frac{Volume\ of\ diluted\ sample}{Volume\ of\ wastewater\ sample\ added}$$

2. Two BOD bottles are taken. The wastewater sample 5 *ml* is taken in the BOD bottles and then 245*ml* of aerated water is filled. DO test is conducted for the one BOD bottle sample by the following steps and initial DO is noted.

3. 1 ml of Manganese Sulphate solution is added, followed by 1 ml of Alkali – Iodide – Azide reagent. Then the bottle is mixed twice and allowed to precipitate settle for 5 minutes.

4. 1 *ml* of Conc.Sulphuric Acid is added slowly and mixed twice. 200 *ml* of sample is taken and titrated against Sodium thio Sulphate solution with starch indicator.

5. Disappearance of blue colour is taken as end point. Volume of Sodium thio Sulphate consumed is noted.

6. Another bottle is placed in incubator at **20°C**. After 5 days DO test is conducted and final DO is noted.

RESULT:

BOD$_5$ of given sample at 20^oC in *mg/l* _____

SHORT PRECOEDURE: [DETERMINATION OF CHEMICAL OXYGEN DEMAND (C.O.D TEST)]

S.No.	CONTENTS	REAGENTS
1.	Burette solution	Std.Ferrous Ammonium sulphate
2.	Conical flask solution	150ml of prepared waste water sample & 150ml of prepared distilled water sample(blank)
3.	Salt	0.4g of Mercuric Sulphate crystal
3.	Additional solutions	10ml of more concentrated dichromate solution with glass beeds and then add slowly 30ml of H_2SO_4 containing Ag_2SO_4
4.	Indicator	Ferroin indicator.
5.	End point	Blue green to wine red.

OBSERVATIONS:

S.No	Volume of Sample ml	Burette Reading ml		Volume of FAS consumed ml	
		Initial	Final	Sample	Blank
1.					
2.					

Exp. No:5

Date:

DETERMINATION OF CHEMICAL OXYGEN DEMAND

(C.O.D TEST)

AIM:

To determine the Chemical Oxygen Demand(C.O.D) in the wastewater sample.

PRINCIPLE:

The organic matter present in sample gets oxidized completely by $K_2Cr_2O_7$ in the presence of H_2SO_4 to produce CO_2 and 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 to oxidation of the organic matter.

APPARATUS REQUIRED:

1. COD Reactor
2. Burette with stand & Pipette
3. Measuring jar
4. Reflux apparatus
5. Beakers
6. Conical flask
7. Hot plate

CHEMICALS REQUIRED:

1. Std. Potassium Dichrornate
2. Conc. Sulphuric Acid
3. Ferroin Indicator Solution
4. Std. Ferrous Ammonium Sulphate Solution
5. Mercuric Sulphate

PROCEDURE:

1. Place 0.4g of $HgSO_4$ in the reflux flask.
2. Add 20*ml* of sample (or an aliquot diluted to 20*ml*)
3. 10ml of more concentrated dichromate solution are placed into flask together with glass beeds.
4. Add slowly 30*ml* of H_2SO_4 containing Ag_2SO_4 and mix thoroughly.
5. Connect the flask to condenser. Mix the contents thoroughly before heating.

CALCULATION: [DETERMINATION OF CHEMICAL OXYGEN DEMAND (C.O.D TEST)]

$$\text{Total Chemical Oxygen Demand} = \frac{(A-B) \times N \times 8000}{V} mg/l$$

Where,

A = Volume of Ferrous Ammonium Sulphate used for blank solution

B = Volume of Ferrous Ammonium Sulphate used for sample titration

N = Normality of Ferrous Ammonium Sulphate solution

V = Volume of wastewater sample taken

6. Improper mixing results in bumping and the sample may be blown out.

7. Reflux for a minimum period of 2 hours. Cool and wash down the condenser with distilled water.

8. Dilute the sample to make up 150*ml* and cool.

9. Titrate excess **K₂Cr₂O₇** with 0.1N **Fe(NH₄)₂SO₄** using ferroin indicator.

10. Sharp colour change from blue green to wine red indicates the end point.

11. Reflux the blank in the same manner using distilled water instead of sample.

RESULT:

The COD of the given sample in *mg/l* _____

PREPARATION OF STANDARD CURVE:

[DETERMINATION OF NITRATE IN THE GIVEN WASTEWATE SAMPLE]

Sl.No	Quantity from 100ppm stockSolution *ml*	Distilled water *ml*	Concentration of new solution *ppm*	Display Readings
1	10	90	10	
2	20	80	20	
3	30	70	30	
4	40	60	40	
5	50	50	50	

Sample Measurements:

Sample 1 _____

Sample 2 _____

Exp. No:6

Date:

DETERMINATION OF NITRATE IN THE GIVEN

WASTEWATE SAMPLE

AIM:

To determine the Nitrate in the given wastewater sample.

PRINCIPLE:

Nitrates react with Phenol Disulphonic Acid and produce a Nitrate Derivative, which in Alkaline solution develops yellow colour due to rearrangement of its structure. The colour produced is directly proportional to the concentration of nitrates present in the sample.

APPARATUS REQUIRED:

1. Spectrophotometer
2. Nessler's tube
3. Pipettes
4. Beakers
5. Cuvettes
6. Measuring jar
7. Hot water bath.

REAGENTS:

1. Phenol Disulphonic Acid
2. Sodium Hydroxide
3. Stock Nitrate Solution
4. Standard Nitrate Solution

REAGENTS PREPARATION:

1. **Phenol Disulphonic acid:**23.4 *ml* of phenol is dissolved in 150 *ml* of concentrated sulphuric acid, to which 75*ml* of sulphuric acid is further added and heated for about 2 hours on a water bath and stored in dark bottles upon cooling.
2. **Sodium Hydroxide:** About 50*g* of sodium hydroxide is dissolved in 150-200 *ml* of water and cooled.

3. **Stock Nitrate solution:** 5*g* of Potassium Nitrate **(KNO₃)** was dried at 103-105°C for one hour and stored in a desiccators until it was cool. 3.6107 *g* of Potassium Nitrate **(KNO₃)** as weighed and washed over into a 500*ml* flask with distilled water (1000 ppm standard). It was closed, shaken vigorously and then labelled. 50*ml* was pipette into a 500 *ml* volumetric flask. The sample was then diluted to 500*ml* distilled water (100 ppm stock standard) and again it was closed, shaken vigorously and then labelled.

4. **Standard nitrate solution:**10*ml* was pipette from the 100ppm stock solution into 100ml volumetric flask and it was made upto mark (10ppm standard). It was closed, shaken vigorously and then labelled.

PROCEDURE:

1. Take 12.5*ml* of standards (10,20,30,40,50ppm) and filtered sample in a separate crucibles and evaporated to dryness on a hot water bath.

2. Cool and dissolve the residue in 0.5*ml* of Phenol Disulphonic acid and dilute to 12.5 *ml* with distilled water.

3. Add 2.5*ml* of Sodium Hydroxide or liquid Ammonia to develop the yellow colour.

4. Switch on the spectrophotometer and allow 20 minutes for warm up and set 410 nm by adjusting the wavelength knob.

5. For calibration of the instrument take distilled water in the cuvette holder and place in the compartment on the light path and close the compartment.

6. Press the mode button to set transmittance mode and set 100 % T.

7. Again press the mode button to set it for absorbance to read 0.00.

8. Fill the prepared standard solutions in the cuvette holders and place in the compartment.

9. Read the colour developed on the display and note down the peak value.

10. The standard graph is plotted by taking concentration along X-axis and the spectrophotometric readings (absorbance) along Y-axis.

11. The value of nitrate is found by comparing absorbance of sample with the standard curve and expressed in *mg/l* or ppm.

RESULT:

The amount of Nitrate present in the given sample is _____ppm

PREPARATION OF STANDARD CURVE:

[DETERMINATION OF PHOSPHATE IN THE GIVEN WASTEWATER SAMPLE]

Standard PO$_4$ solution ml	Distilled water ml	Ammonium Molybdate ml	Stannous Chloride drops	Concentration of new solution mg	Display Readings
0	25	1	2	0	
5	20	1	2	0.05	
10	15	1	2	0.10	
15	10	1	2	0.15	
20	5	1	2	0.20	
25	0	1	2	0.25	

CALCULATION:

Sample 1 = _____

Sample 2 = _____

$$\text{Phosphates (mg/l)} = \frac{mg\ PO_4 \times 1000}{volume\ of\ sample}$$

Exp. No:7

Date:

DETERMINATION OF PHOSPHATE IN THE GIVEN
WASTEWATER SAMPLE

AIM :

To determination of Phosphates by spectrophotometer.

PRINCIPLE:

A phosphate in acidic condition reacts with ammonium molybdate to form molybdophosphoric acid which is then reduced to molybdenum blue by adding stannous chloride. The intensity of the blue colored complex is measured spectrophotometrically,which is directly proportional to the concentration of phosphate present in the sample.

APPARATUS:

Spectrophotometer, pipettes, measuring cylinder, glass-rod, beakers, dropper.

REAGENTS:

1. **Ammonium Molybdate solution :**

In 175 *ml* of distilled water, add 25 *g* of ammonium molybdate. Now add 280 *ml* of conc.sulphuric acid in 400 *ml* of distilled water and cool it. Make the volume upto 1 litres with distilled water.

2. **Stannous chloride solution:**

Dissolve 2.5 *g* of stannous chloride in 100 *ml* glycerol by heating in a water bath.

3. **Standard Phosphate Solution:**

Dissolve 4.388 *g* of anhydrous potassium hydrogen phosphate in 1000 *ml* distilled water. Dilute this solution to 100 times (10ml-1000ml).This solution contains 10 mg P/l (1ml=0.01 mg P) which is used as standard phosphate solution.

PROCEDURE:

1. Switch on the spectrophotometer and allow 20 minutes for warm up and set 410 nm by adjusting the wavelength knob.

2. For calibration of the instrument take distilled water in the cuvette holder and place in the compartment on the light path and close the compartment.

3. Press the mode button to set transmittance mode and set 100 % T.

4. Again press the mode button to set it for absorbance to read 0.00.

5. Fill the prepared standard solutions in the cuvette holders and place in the compartment.

6. Read the colour developed on the display and note down the peak value.

7. Take 25 *ml* of filtered and clear sample.

8. Add 1 ml of ammonium molybdate solution and 2 drops of stannous chloride

9. Measure the blue color developed at 690 nm on a spectrophotometer using a distilled water blank with the same chemicals.

10. Note down the readings of spectrophotometer after 5 minutes but before 12 minutes of the addition of the last reagent.

11. Find out the concentration of the phosphate with the help of the standard curve.

RESULT:

    The amount of Phosphates determined from the given sample is _____*mg/l.*

SHORT PREOCEDURE:[DETERMINATION OF CALCIUM IN THE GIVEN WASTEWATER SAMPLE]

S.NO	CONTENTS	REAGENTS
1.	Burette solution	Standard EDTA
2.	Conical flask solution	20*ml* of waste water solution
3.	Additional solutions	1ml of Sodium hydroxide
4.	Indicator	Murexide indicator.
5.	End point	pink colour turns to purple

OBSERVATION:

EDTA Vs Wastewater sample:

S.No	Volume of sample *ml*	Burette Reading *ml*		Concurrent burette reading *ml*	Volume of EDTA *ml*
		Initial	Final		
1.					
2.					

Exp. **No:8**

Date:

DETERMINATION OF CALCIUM IN THE GIVEN
WASTEWATER SAMPLE

AIM :

To determine the calcium in the given waste water sample.

PRINCIPLE:

When EDTA (Ethylene-diamine tetra acetic acid) is added to the water containing calcium and magnesium, it combines first with calcium. Calcium can be determined directly with EDTA when **pH** is made sufficiently high such that the magnesium is largely precipitated as hydroxyl compound (by adding **NaOH** and iso-propyl alcohol). When murexide indicator is added to the solution containing calcium, all the calcium gets complexed by the **EDTA** at **pH** 12-13. The end point is indicated from a colour change from pink to purple.

APPARATUS REQUIRED:

1. Burettes,
2. Pipette,
3. Conical Flask,
4. Beakers,
5. Droppers.

REAGENTS:

1. **Sodium hydroxide (8%):**8g of sodium hydroxide is dissolved in 100ml of distilled water.
2. **Murexide indicator (ammonium purpurate):**0.2 g of murexide is ground well with 100g of sodium chloride thoroughly.
3. **Standard EDTA titrant, 0.01 N:**3.723 g of **EDTA** (disodium salt) is dissolved in distilled water and made up to 1000 ml with the same.

CALCULATION:

Equivalent weight of CaCO3   =   50

Molecular weight of Calcium   =   40.078

Molecular weight of Calcium carbonate   =   100

$$\text{Hardness as CaCO}_3 \text{ equivalent mg/l} = \frac{Volume\ of\ EDTA \times N \times 50 \times 1000}{Volume\ of\ sample\ taken}$$

$$\text{Calcium, Ca mg/l} = \frac{Ca\ hardness\ in\ \frac{mg}{l}\ as\ CaCO3 \times molecular\ weight\ of\ Ca}{Molecular\ weight\ of\ Calcium\ Carbonate}$$

PROCEDURE:

1. Wash the burette with distilled water and fill it with **EDTA** solution upto the zero mark.

2. 20ml of the sample is pipetted into a clean conical flask

3. Add 1ml of sodium hydroxide to rise the **pH**.

4. A pinch of murexide indicator is added to this mixture.

5. Then titrated against **EDTA** until the pink color turns to purple.

RESULT:

The amount of Calcium present in the given sample is _____ *mg/l*

PREPARATION OF STANDARD CURVE:

[DETERMINATION OF POTASSIUM IN THE GIVEN WASTEWATER SAMPLE]

Standard Solution ml	Distilled Water ml	Concentration of New Solution mg	Display Readings
10	90	1	
20	80	2	
30	70	3	
40	60	4	
50	50	5	
60	40	6	
70	30	7	
80	20	8	
90	10	9	
100	0	10	

CALCULATION:

Sample 1_____

Sample 2_____

$$\text{Potassium } mg/l \quad = \quad \frac{(mg\ K \times 1000)}{ml\ sample}$$

Exp. No:9

Date:

DETERMINATION OF POTASSIUM IN THE GIVEN
WASTEWATER SAMPLE

AIM:

To determine the amount of Potassium present in the given wastewater sample.

PRINCIPLE:

Trace amount of potassium can be determined by direct reading of flame photometer at a specific wavelength of 766.5nm by spraying the sample into the flame. The desired spectral lines are then isolated by the use of interference filters or suitable slit arrangements. The intensity of light is measured by the phototube.

Working principle of Flame photometer: The emission of characteristic radiations by alkali and alkaline earth metals and the correlation of the emission intensity with the concentration of the element form the basis of flame photometry. The principle of the flame photometer depends on the "Emission Spectroscopy" in which the electrons of the metals after absorbing energy get excited from ground state to higher energy level and return back to the ground state with emission of light. The sample under test is introduced into flame in solution by means of atomizer. The radiation from the flame enters a dispersing device and isolates it (radiation) from the flame to the desired region of the spectrum. The phototube measures the intensity of isolated radiation, which is proportional to the concentration of the element present in the sample.

APPARATUS REQUIRED:

1. Flame Photometer,
2. Lab Glassware,
3. Whattman Filter Paper.

REAGENTS AND STANDARDS:

To minimize potassium pickup, store all solutions in plastic bottles. Shake each container thoroughly to dissolve accumulated salts from walls before pouring.

     i.   **Reagent water double distilled water**

    ii.   **Stock potassium solution**: Dissolve 1.907g KCl dried at 110ºC and dilute to 1000mL with water; 1ml = 1mg K.

    iii.   **Working Potassium solution:** Working standards of suitable strengths are prepared from the stock solution.

PROCEDURE:

PRE – OPERATIONS:

1. AIR OUT of the compressor unit and AIR IN of the flame photometer are connected properly with connecting tube fitted with plastic nozzle. Fit the capillary tube on the capillary nozzle.

2. Make sure that all the connections are proper and there is no leakage of either air or gas.

3. Switch on the flame photometer, allow it to stabilize for 2 minutes and switch on the compressor unit.

4. Turn on the air control and adjust the air pressure to read initially between 0.4 to 0.6 kg/cm^2 on the air pressure gauge.

5. The filter disk is set at 766.5 nm (marked for potassium, K).

6. The sensitivity is to be selected depending upon the element under test. Initially set the sensitivity switch to position 1.

7. Turn on the gas supply from gas control knob. Press auto ignition switch to light the flame or use manual gas igniter.

8. Adjust gas and air controls to get silent flame with sharp blue cone close to the burner, irrespective of air pressure needle position shown on the compressor pressure gauge.

9. Allow the flame to stabilize for about 10 minutes.

CALIBRATION:

1. Clean the capillary by double distilled water by dipping the loose end of the capillary tube in the double distilled water sample holder. It will start getting aspirated into the nebulizer and thus clean it.

2. Set zero on the display with set zero knob.

3. Replace distilled water beaker with standard solution of higher pm value in a beaker.

4. Set the ppm value of standard solution on the display of flame photometer by rotating the calknob. If the display does not reach the value of the standard solution, increase the sensitivity to the next position.

5. Replace the standard with blank (double distilled water) to clean the capillary tube.

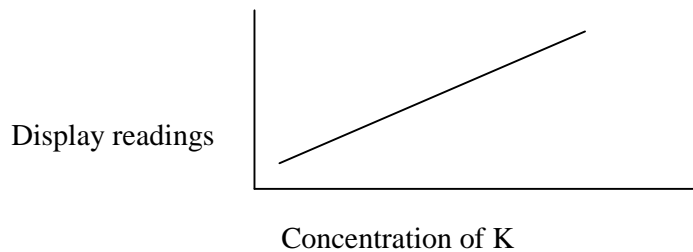6. Now the instrument is ready for taking the readings of any unkown sample solutions.

NOTE:

After every 20- 30 minutes of operation, the instrument should be re-calibrated by repeating the operation of calibration.

Preparation of standard/ calibration curve:

Use stock potassium solution to prepare calibration curve in potassium. Clean the capillary tube with double distilled water immediately every standard solutions.

Calibration curve:

Display readings

Concentration of K

Sample measurement:

1. The sample is filtered through a filter paper and takes in the sample holder.

2. Dip the capillary into the sample, the sample will be aspirated and colour of the flame will be changed.

3. Display will read the exact value of the sample by attain a peak value.

4. Clean the capillary with blank before taking the next sample.

RESULT:

The amount of Potassium present in the given sample is_____*mg/l*

PREPARATION OF STANDARD CURVE:

[DETERMINATION OF SODIUM IN THE GIVEN WASTEWATER SAMPLE]

Standard solution ml	Distilled water ml	Concentration of new solution mg	Display readings
10	90	1	
20	80	2	
30	70	3	
40	60	4	
50	50	5	
60	40	6	
70	30	7	
80	20	8	
90	10	9	
100	0	10	

CALCULATION:

Sample 1 = _____

Sample 2 = _____

$$\text{Sodium } mg/l = \frac{mg\ Na \times 1000}{volume\ of\ sample}$$

Exp. No:10

Date:

DETERMINATION OF SODIUM IN THE GIVEN
WASTEWATER SAMPLE

AIM:

To determine the amount of sodium present in the given wastewater sample.

APPARATUS REQUIRED:

1. Flame Photometer,
2. Lab Glassware
3. Whattman Filter Paper.

REAGENTS REQUIRED:

1. **Reagent water:**

   double distilled water

2. **Stock sodium solution:**

   Dissolve 2.542 g NaCl dried at 140°C and dilute to 1000ml with water, 1 ml = 1 mg Na.

3. **Working Potassium solution:**

   Working standards of suitable strengths were prepared from the stock solution

PROCEDURE:

PRE – OPERATIONS:

1. AIR OUT of the compressor unit and AIR IN of the flame photometer are connected properly with connecting tube fitted with plastic nozzle. Fit the capillary tube on the capillary nozzle.

2. Make sure that all the connections are proper and there is no leakage of either air or gas.

3. Switch on the flame photometer, allow it to stabilize for 2 minutes and switch on the compressor unit.

4. Turn on the air control and adjust the air pressure to read initially between 0.4 to 0.6 kg/cm^2 on the air pressure gauge.

5. The filter disk is set at 581 nm (marked for potassium, Na).

6. The sensitivity is to be selected depending upon the element under test. Initially set the sensitivity switch to position 1.

7. Turn on the gas supply from gas control knob. Press auto ignition switch to light the flame or use manual gas igniter.

8. Adjust gas and air controls to get silent flame with sharp blue cone close to the burner, irrespective of air pressure needle position shown on the compressor pressure gauge.

9. Allow the flame to stabilize for about 10 minutes.

CALIBRATION:

1. Clean the capillary by double distilled water by dipping the loose end of the capillary tube in the double distilled water sample holder. It will start getting aspirated into the nebulizer and thus clean it.

2. Set zero on the display with set zero knob.

3. Replace distilled water beaker with standard solution of higher ppm value in a beaker.

4. Set the ppm value of standard solution on the display of flame photometer by rotating the cal knob. If the display does not reach the value of the standard solution, increase the sensitivity to the next position.

5. Replace the standard with blank (double distilled water) to clean the capillary tube.

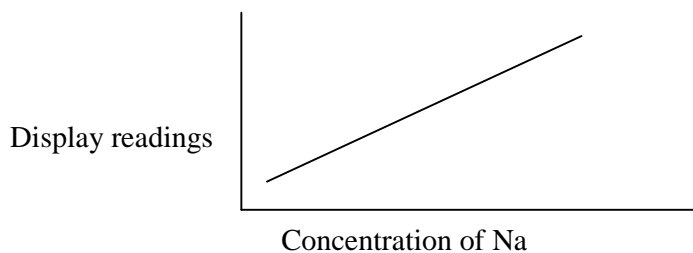6. Now the instrument is ready for taking the readings of any unkown sample solutions.

NOTE:

After every 20-30 minutes of operation, the instrument should be re-calibrated by repeating the operation of calibration.

Preparation of standard/ calibration curve:

Use stock sodium solution to prepare calibration curve in potassium Clean the capillary tube with double distilled water immediately every standard solutions.

Calibration curve:

Display readings

Concentration of Na

Sample measurement:

1. The sample is filtered through a filter paper and takes in the sample holder.

2. Dip the capillary into the sample, the sample will be aspirated and colour of the flame will be changed.

3. Display will read the exact value of the sample by attain a peak value.

4. Clean the capillary with blank before taking the next sample.

RESULT:

The amount of Sodium present in the given sample is_____ *mg/l*

TABULATION: [HEAVY METALS DETERMINATION - CHROMIUM, LEAD AND ZINC. (DEMONSTRATION ONLY)]

[Pb] ppm	Volume Stock Solution ml	Absorbance Trial 1	Absorbance Trial 2	Absorbance Trial 3	Average Absorbance
2					
4					
8					
12					
20					
40					
60					
100					
Unknown					

Section A: Obtaining a Calibration Curve for the LOD Calculation:

Water Sample Unknown Number = _____

Lead stock solution concentration =_____ppm

Calibration Curve Standards Calculations (show only one)

Exp. No:11

Date:

HEAVY METALS DETERMINATION - CHROMIUM, LEAD

AND ZINC. (DEMONSTRATION ONLY)

GENERAL:

Lead, Zinc and Chromium are found as a pollutant in the atmosphere, the soil, and in water. Fortunately the concentration of lead in the atmosphere has decreased since the introduction of unleaded gasolines in automobiles. But these heavy metals can also enter the atmosphere from the combustion of solid waste, coal, and from lead smelter emissions. It may occur naturally in soils in mining areas, and can be found at high concentrations in natural waters associated with mining. For many cities, in Colorado for example, these mine waters can ultimately enter the water supply. The source of lead in drinking water is, however, mainly from solder, pipes, and fixtures (1). The current safe drinking water limit established by the Environmental Protection Agency (EPA) is 50 parts per billion (ppb); the agency has further concerns with this limit and set an "action level" of 15 ppb.

APPLICATION:

Sample is aspirated and atomized in a flame. A light beam from a hollow cathode lamp is directed through the flame into a monochromator and onto a detector. Since wavelength of light beam are specific for Lead, Zinc and Chromium. Light energy absorbed by detector is measure of lead.
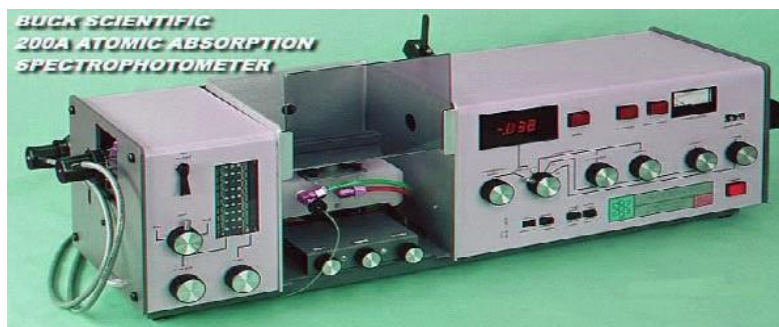
INTERFERENCES:

The most troublesome type is chemical, caused by lack of absorption of atoms bound in molecular combination in the flame. High dissolved solids in sample may result in nonatomic absorbance interference. Background correction is required.

INSTRUMENTATION:

Atomic absorption spectrometer. Lead hollow cathode lamp (283.3 nm) RANGE: 1-20 mg/L (ppm) Minimum Detection Limit: 0.1 mg/l (100 ppb) PRECISION: standard deviation = 128 ppb @ 367 ppb (true value) 74 labs ACCURACY: as bias = +2.9% @ 367 ppb (true value) 74 labs.

Chromium (Cr) Pure Single-Element Standard, 1,000 µg/ml, 2% HNO3, 500 ml

SAMPLING METHOD:

Use glass or plastic containers. Collect 200 g of solids and 600 ml of liquid samples. STABILITY: Cool solid samples to 4°C and analyze as soon as possible. Add nitric acid to liquid samples to pH < 2.

OBTAINING A CALIBRATION CURVE:

NOTE: Refer to Figure 1 to locate the instrument controls listed in parentheses in the following steps.

1.   Your instructor will assign you an unknown that has a large "matrix" or background (pond water). Enter the unknown number in your data sheet.

2.   First you are going to mix up standard solutions of lead(II) nitrate and use them to plot a calibration curve. Use the stock solution of 1000 ppm and the dilution equation to prepare solutions with these concentrations in ppm: 2, 4, 8, 12, 20, 40, 60, and 100 ppm. Use the data sheet for your calculations, and check your results with me before proceeding further. Prepare all solutions using 1% (v/v) HNO3 (helps prevent absorption of the lead into the glass, and therefore producing incorrect calibration curves. Conversely it will leach out any lead in the glass into the solutions, but the amount is low compared to our concentrations).

3.   Turn on POWER SWITCH

4.   Make sure the FUEL TOGGLE SWITCH (A) is off and the OXIDANT SELECTOR VALVE (D) is off.

5.   Turn on the air supply valve located on the analytical bench behind you. It will have clear tubing attached to it.

6.   Turn the FUNCTION SWITCH (8) to the L1 (lamp 1) position.

7.   Adjust the lamp 1 current to 10 mA by using the LAMP 1 dial

8.   Now turn the FUNCTION SWITCH to PMT VOLTS. Adjust the voltage to -350 V using the ABSORBANCE ZERO dial

9.   Set the WAVELENGTH dial to approximately 287 nm. Now rotate the dial UPWARDS until the ENERGY METER is at a maximum (e.g. maximum deflection to the right).

10. Have the instructor turn on the acetylene fuel and ignite the burner.

11. Adjust the HORIZONTAL LAMP positioning dial until a maximum deflection is obtained on the ENERGY METER. Do the same with the VERTICAL LAMP dial

12. Move the FUNCTION SWITCH back to ABSORBANCE ZERO. Press the AUTO ZERO switch. Aspirate the blank using the beaker labeled "blank". Now momentarily depress the ZERO buttons. In about seven seconds the absorbance will be automatically zeroed. If not, press the ZERO button again.

13. Now aspirate the solution labeled 1000 ppm. You should get an absorbance of around 1.50. Maximize this absorbance by adjusting the burner HORZ (H), VERT (V), and ROT (R) knobs. Be careful with the vertical control since it is easy to block the hollow cathode lamp and get a large absorbance reading!

14. Now maximize the absorbance by varying the FUEL FLOW CONTROL VALVE (F).

15. Maximize the absorbance by rotating the knob on the NEBULIZER & OXIDANT FITTING (N).

16. Aspirate the blank and zero the absorbance. Now you are ready to take data for the calibration curve.

17. Fill the beakers labeled 2.0 - 100.0 ppm with about 10 ml of the appropriate sample. There are 8 samples altogether.

18. Aspirate the 2.0 ppm sample and record the absorbance in the data sheet. Then reaspirate the blank, zeroing if necessary. Continue this process with the rest of the samples.

19. Pour out no more than 50 ml of your unknown into a beaker. Aspirate and measure the unknown absorbance three times, re-zeroing in between each time. Enter the absorbances in the data sheet.

20. Have the instructor turn off the burner.

RESULT:

Amount of Chromium, Lead and Zinc ion in the given sample is_____*mg/l.*