

9.0 EXPERIMENT ON DETERMINATION OF SULPHATES

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9.0 EXPERIMENT ON DETERMINATION OF SULPHATES

PREAMBLE:

“How to determine Sulphates in Water and Wastewater”.

Test procedure is in accordance to IS: 3025 (Part 24) - Reaffirmed 2003.

In addition to our Indian Standard, we also discuss in brief regarding the procedure stated in

- (1) APHA Standard Methods for the Examination of Water and Wastewater - 20th Edition. Method 4500 E.
- (2) Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, USEPA, Method 375.4.

9.1 AIM

To determine the sulphates in the given water sample with the stipulations as per **IS: 3025 (Part 24) - Reaffirmed 2003.**

9.2 INTRODUCTION

Sulphates is widely distributed in nature and may be present in natural waters in concentration ranging from few hundred to several thousand mg/L. Sulphates occur naturally in numerous minerals, including barite, epsomite and gypsum. These dissolved minerals contribute to the mineral content of drinking-waters.

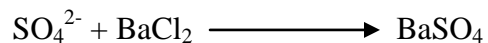
Acid Mine Drainage (AMD) may contribute large amounts of sulphates through pyrite oxidation. Sulfate is the second most abundant anion in seawater. Its high concentration owes to the high to moderate solubility of the salts that it forms with the major cations in seawater, namely, Na, Mg²⁺, and Ca²⁺.

9.2.1 ENVIRONMENTAL SIGNIFICANCE

- Sulphates are of considerable concern because they are indirectly responsible for two serious problems often associated with the handling and treatment of wastewater. They are odour and sewer corrosion problem result from the reduction of sulphates to hydrogen sulphide under anaerobic conditions.
- The amount of sulphates in wastewater is a factor of concern in determining the magnitude of problems that can arise from reduction of sulphates to hydrogen sulphide. For example knowledge of the sulphates content of the sludge or waste fed to digestion units provides a means of estimating the hydrogen sulphide content of the gas produced. From this information, the design engineer can determine whether scrubbing facilities will be needed to remove hydrogen sulphide and size of the units required.

9.3 PRINCIPLE

The turbidimetric method of measuring sulphates is based upon the fact that barium sulphates tends to precipitate in a colloidal form of uniform size and that this tendency is enhanced in presence of a sodium chloride, hydrochloric acid and glycerol.



The absorbance of the barium sulphates formed is measured by a spectrophotometer at 420 nm and the sulphates ion concentration is determined by comparison of the reading with a standard curve.

9.4 MATERIALS REQUIRED

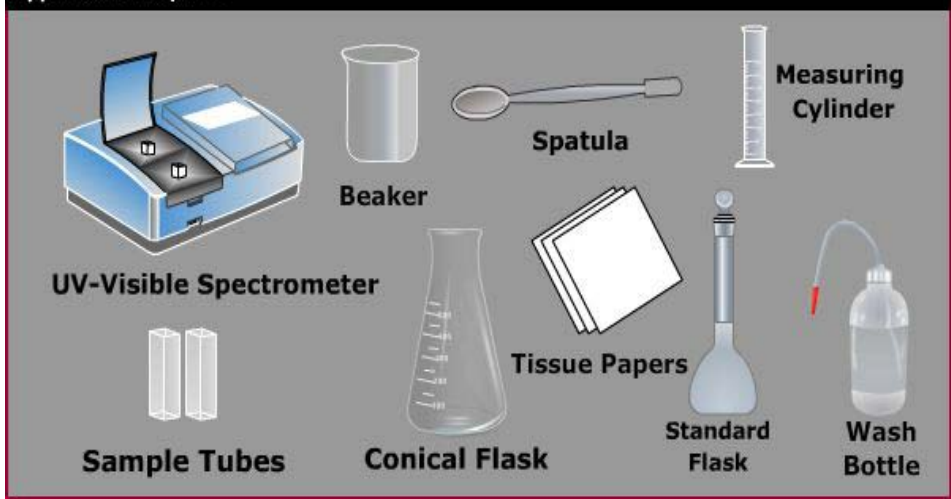
9.4.1 APPARATUS REQUIRED

1. UV-Visible Spectrometer
2. Sample tubes
3. Standard flask
4. Beaker
5. Spatula
6. Measuring Cylinder
7. Wash Bottle
8. Tissue Paper

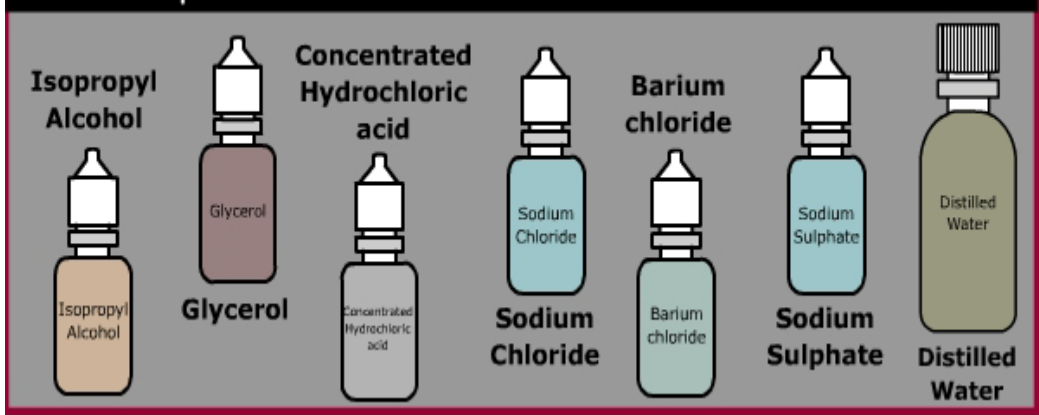
9.4.2 CHEMICALS REQUIRED

1. Isopropyl Alcohol
2. Glycerol
3. Concentrated Hydrochloric acid
4. Sodium Chloride
5. Barium chloride
6. Sodium sulphate
7. Distilled water

Apparatus Required

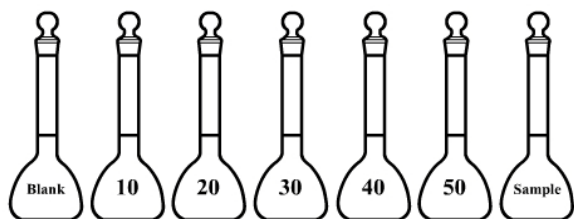


Chemicals Required

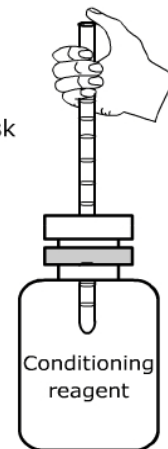


PROCEDURE CHART

Prepare a series of standards
10, 20, 30, 40, 50, a blank
(distilled water)
and known volume of sample



Add 5mL of
Conditioning
reagent to all the flask



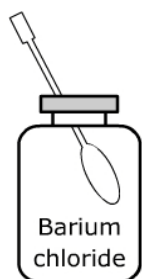
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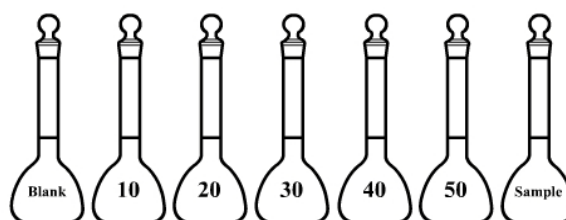
2



Add a pinch of
Barium chloride
to all the flask



Make of the volume to 100mL
using distilled water



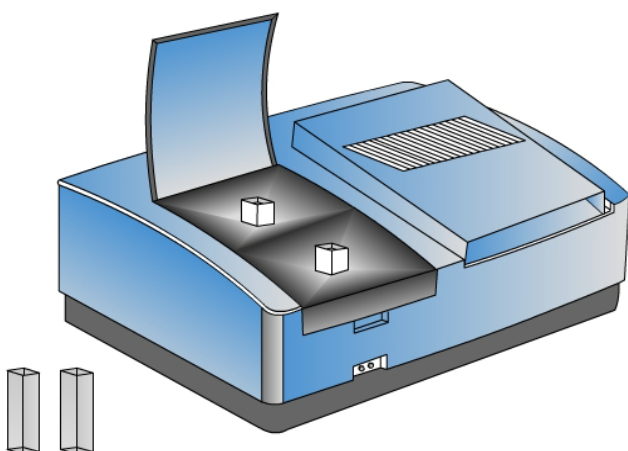
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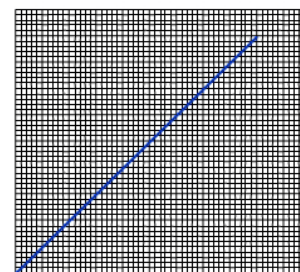


Immediately measure
the turbidity
using UV-Visible
spectrometer



Using the values
draw standard graph

5



6



Using the equation from the
graph calculate the
concentration of sulphate

9.5 SAMPLE HANDLING AND PRESERVATION

- Preservation of sample is not practical. Because biological activity will continue after a sample has been taken, changes may occur during handling and storage.
- During storage of samples, some organic matter present in it may reduce sulphate SO_4^{2-} , to sulphide SO_3^{2-} , to avoid this, store sample at temperature of about 4°C. Do not allow the samples to freeze.
- Analysis should begin as soon as possible.

9.5.1 PRECAUTIONS

- i. If the total cation concentration is more than 250 mg/L or if the heavy metal ion concentration is more than 10 mg/L, the sample is passed through an ion exchange column, to remove these ions.
- ii. Colour and turbidity due to the sample matrix can cause positive interferences which must be accounted. The sample used for the analysis should be free of turbidity. If the sample is
- iii. turbid filtered it through 0.45µm filter paper.
- iv. Silica in concentrations over 25 mg/L will also interfere and should be treated properly.
- v. If the sample containing large quantities of organic matter, then the sample cannot precipitate barium sulphate satisfactorily.

9.6 PROCEDURE

9.6.1 PREPARATION OF REAGENTS

Conditioning reagent

- Measure exactly 25 ml glycerol and pour it to a dry clean beaker.
- Then, measure 15 mL of concentrated hydrochloric acid and add it to the same beaker.
- To the same beaker, add exactly 50 mL of 95 % isopropyl alcohol and mix well.
- Accurate weigh 37.5 g sodium chloride and dissolve it in distilled water.
- Then mix all the contents and make up the final volume to 250 mL using distilled water.

Standard sulphate solution

- Weigh accurately 1.479 g anhydrous sodium sulphate and dissolve it in distilled water.
- Take 1000 mL standard measuring flask and place a funnel over it.
- Transfer it to the 1000 mL standard flask and make up to 1000 mL using distilled water.
- (1 mL = 1.0 mg SO_4^{2-})

Preparation of Blank, Standards and sample for Testing

- Take six 50 mL glass stoppered standard flask (four for standards, one for the sample and one for the blank).
- Add 10 mL of the standard sulphate solution to the first standard flask, 20 mL to the second, 30 mL to the third and 40 mL to the fourth.
- To the fifth standard flask add 20 mL of the sample water.
- The sixth standard flask is for the blank, to this standard flask add distilled water alone.
- Add 5 mL of conditioning reagent to all the standard flasks.
- Then make up the volume to the 100 mL mark using distilled water.

Introduction to UV - spectrometer and spectra manager - software

The UV visible spectrometer is used to measure the Sulphate content of the given sample. This spectrometer is connected to the computer system, loaded with the software spectra manager. Spectra manager is the software which receives input from UV - visible spectrometer; it manipulates the data and displays the absorbance reading of the solution placed in the chambers of the spectrometer. To open the software double click the icon spectra manager. Different choices of measurement will appear namely:

1. Quantitative analysis
2. Spectrum analysis
3. Time course measurement
4. Fixed wavelength measurement
5. Absorbance transmittance meter
6. Environment

To measure the sulphate content select the fixed wavelength measurement method. Double click fixed wavelength measurements, the system information are transferred and then the following window will appear. Click parameter option and select absorbance, fast response and enter starting value of the wave length in nanometers. For this experiment load 420 nm as the starting value. Click 'add' and enter the sample number, here it is 100. Then load the number of cycles the analysis has to be made. Enter 1 for this case. Click ok. The details of the entries will be displayed as shown. Note that the absorbance is not zero so to reset the reading to zero, take distilled water in the two sample tubes and place them in

the chambers of the spectrometer. Now click the button 'auto zero' the instrument resets and shows 0.0000 as reading.

9.6.2 TESTING OF SAMPLE

Transfer blank to the sample tubes and place it in the chamber. Now click 'Blank', the value of absorbance for blank is displayed as 0.0185. Then take standard 1 in the sample tube, place it in the chamber. Click 'start' and observe the reading is 0.0902. Similarly for standard 2 the absorbance is 0.2377 for standard 3 the reading is 0.4604, for standard 4 the reading is 0.6177, finally for standard 5 the value of the reading is 0.8024. Now transfer the given sample from standard flask to sample tube and place it in the chamber. Click 'start' the absorbance reading obtained is 0.7824.

9.7 CALCULATION

9.7.1 TABLE

Sample No.	Volume of Sample/ Standard (ml)	Absorbance

- For the blank the volume is 0 mL and the absorbance is 0.0185.
- For standard 1 volume of the sample is 10 mL and the absorbance is 0.0902.
- For Standard 2 the volume of the sample taken is 20 mL and the absorbance is 0.2377.
- In Standard 3 the volume of the sample is 30 mL and the absorbance is 0.4604.
- Standard 4 the volume of the sample is 40 mL and the absorbance is 0.6177.

- Standard 5 the volume of the sample taken is 50 mL and the absorbance is 0.8024.
- For trial 1 the volume of sample is 25 mL and the absorbance is 0.7824.
- For trial 2 the volume of sample is 25 mL and the absorbance is 0.7301.
- For trial 3 the volume of sample is 25 mL and the absorbance is 0.1742.

9.7.2 DATA SHEET

DETERMINATION OF SULPHATES DATA SHEET

Date Tested : August 30, 2010
Tested By : CEM Class, Group A
Project Name : CEM, NITTTR Lab
Sample Number : BH1
Sample Location : Perungudi (Lat 12' 57" 31.74 & Long 80'14" 8.82)
Sample Description : Surface water

Sample No.	Volume of Sample/ Standard (ml)	Absorbance
Blank	0	0.0185
Std 1	10	0.0902
Std 2	20	0.2377
Std 3	30	0.4604
Std 4	40	0.6177
Std 5	50	0.8024
1	25	0.7824
2	25	0.7301
3	25	0.1742

Model Calculation:

From the calibration graph,

$$Y = mX + c \quad \text{Whereas,}$$

Y = Absorbance of the sample

m = Slope of the straight line

X = Concentration of sulphate in mg

$$0.7824 = 0.15X + 0$$

$$\begin{aligned} \text{Therefore, } X &= 0.7824 / 0.15 \\ &= 5.216 \end{aligned}$$

$$\text{Concentration of Sulphate in mg/L} = \frac{X * 1000}{\text{mL of sample taken}}$$

To convert the sample size from mL to L, multiply the result by 1,000 mL/L

$$\begin{aligned} \text{Concentration of Sulphate in mg/L} &= 5.216 \times 1000/25 \\ &= 208.64 \text{ mg/L} \end{aligned}$$

9.8 INTERPRETATION OF RESULTS

The sulphates concentration in the given sample of water = **208.64 mg/L**.

9.9 INFERENCE

Water containing high levels of sulphates, particularly magnesium sulphate and sodium sulphate may have a laxative effect on persons using the water for the first time. These effects vary with the persons and appear to last only until the person becomes accustomed to using the water. High sulphates content also affects the taste of water and will form a hard scale in boilers and heat exchangers. For these reasons the upper recommended limit for sulphates in water is 250 mg/L.

9.10 EVALUATION

1. In this method, the sulphates present are estimated in the form of
 - a) Barium Sulphate
 - b) Calcium Sulphate
 - c) Sodium Sulphate
 - d) Magnesium Sulphate
2. The turbidity is measured using UV-visible spectrometer at
 - a) 410 nm
 - b) 420 nm
 - c) 430 nm
 - d) 440 nm
3. For plotting a standard graph, the minimum requirement is
 - a) 3 standards
 - b) 2 standards
 - c) 1 standards
 - d) 4 standards
4. The conditioning reagent, contains _____
 - a) Glycerol
 - b) Phenol
 - c) Sodium sulphate
 - d) Sodium Hydroxide

5. The minimum detectable concentration by turbidimetric method is
- a) 10 ppm
 - b) 5 ppm
 - c) 1 ppm
 - d) No limitations
6. A calibration curve is the plot of
- a) absorbance against concentration of solutions
 - b) absorbance of solutions against time
 - c) time against concentration of solutions
 - d) concentration of solutions against time
7. As per Beer - Lambert's Law the amount of light absorbed is _____ the concentration of the solution.
- a) inversely proportional
 - b) directly proportional
 - c) greater than
 - d) smaller than
8. Transmittance is the ratio of the intensity of transmitted light to that of incident light.
- a) True
 - b) False
9. The optical density is _____ to concentration of the substance.
10. Blank solution is used in colorimetric estimation to
- a) nullify the absorbance caused due to the colouring impurities present in the reagents
 - b) check the instrument
 - c) nullify the error caused by the instrument
 - d) nullify the error caused by colour

KEY TO ITEMS:

1) a

2) b

3) b

4) a

5) c

6) a

7) b

8) True

9) directly proportional

10) a