EXPERIMENT NO. 1

OBJECT:

To determine total dissolved and suspended solids in the given water sample with the stipulations as per IS : 3025 (Part 16 and Part 17.)

THEORY:

The term total dissolved solids refer to materials that are completely dissolved in water. These solids are filterable in nature. It is defined as residue upon evaporation of filterable sample. The term total suspended solids can be referred to materials which are not dissolved in water and are non-filterable in nature. It is defined as residue upon evaporation of non-filterable sample on a filter paper.

APPARATUS REQUIRED:

1. Evaporating dish
2. Drying oven
3. Filter paper
4. Desciccators
5. Analytical balance
6. Graduated cylinders
7. Dish tongs
8. Gooch crucibles
9. Wash bottle
10. Vacuum pumps
11. Filter membrane
12. Forceps, smooth-tipped

Fig. 7.1

Procedure Chart

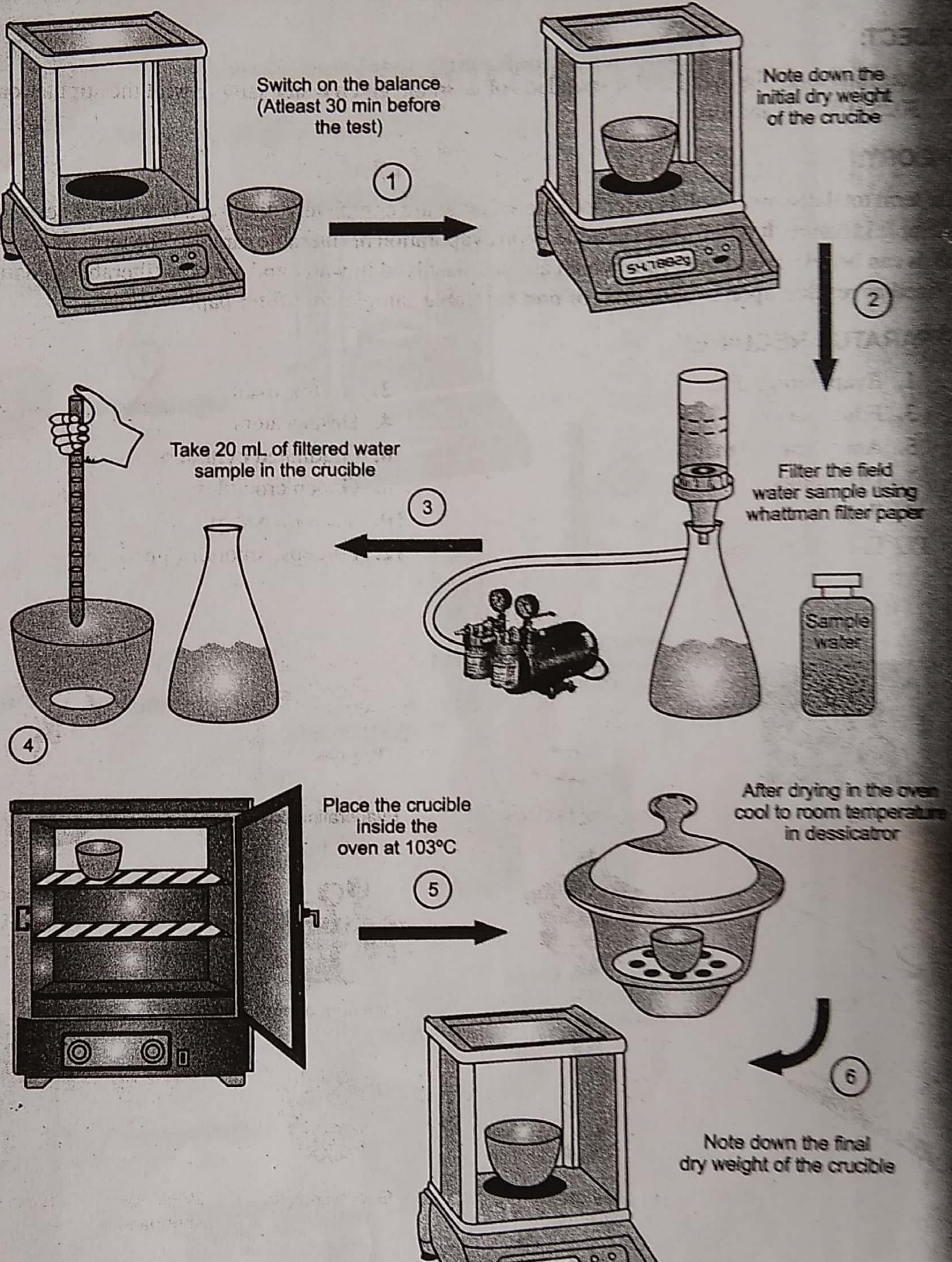

Fig. 7.2

Procedure Chart

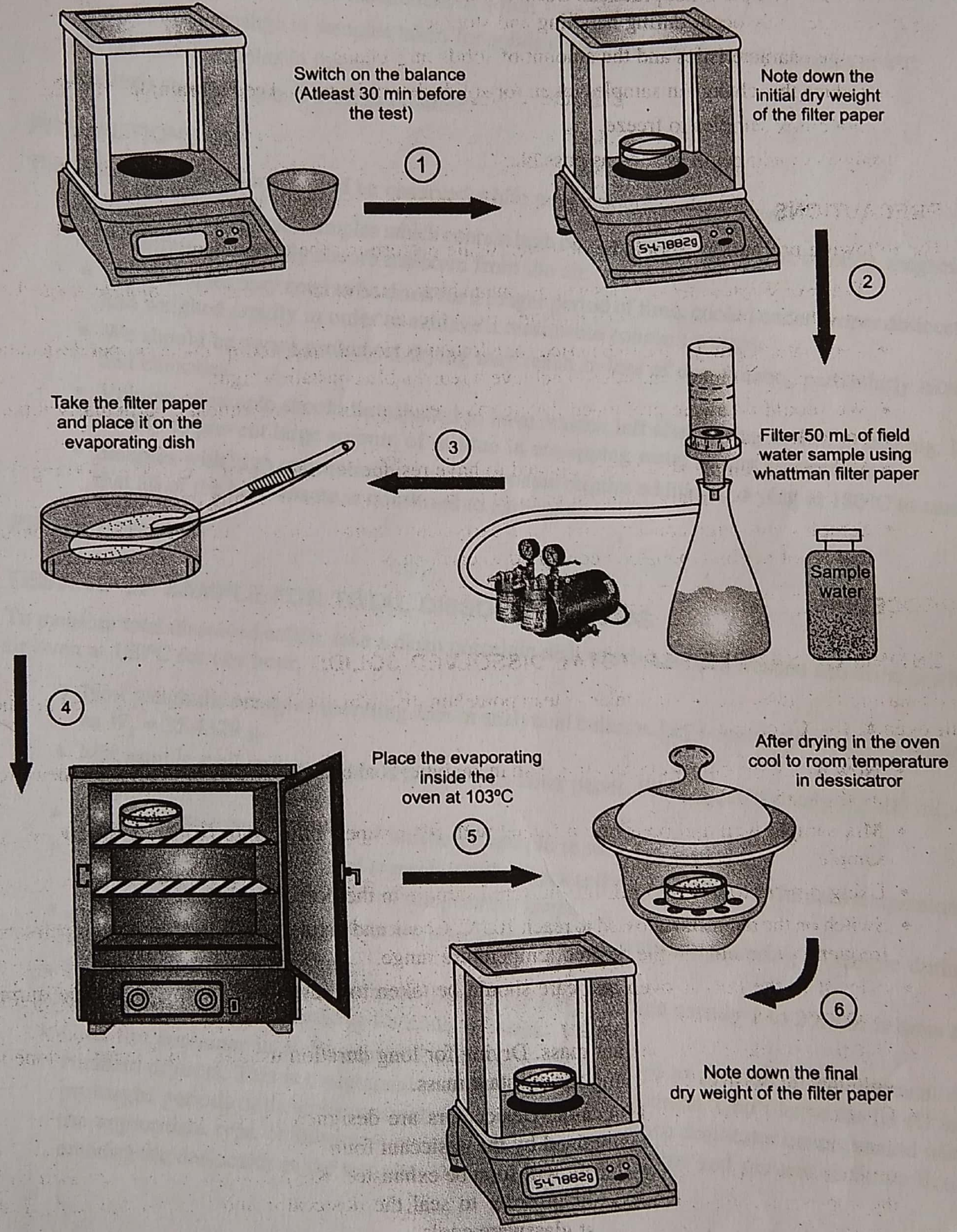

Fig. 7.3

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.

Both the characteristics and the amount of solids may change.

To reduce this change in samples taken for solids determinations, keep all samples at 4°C.

Do not allow samples to freeze.

Analysis should begin as soon as possible.

PRECAUTIONS

The following precautions should be observed while performing the experiment:

- Water or Wastewater samples which contain high concentrations of calcium, chloride, magnesium or sulfate can rapidly absorb moisture from the air.
- Such samples may need to be dried for a longer period of time, cooled under proper desiccation and weighed rapidly in order to achieve a reasonable constant weight.
- We should be aware prolonged drying may result in loss of constituents, particularly nitrates and chlorides.
- Volume of sample should be adjusted to have residue left after drying as 100 to 200 mg. It is mainly to prevent large amount of residue in entrapping water during evaporation.
- Samples with high concentrations or bicarbonate require additional drying at 180°C to ensure that all of the bicarbonate is converted to carbonate.

PROCEDURE

TESTING OF SAMPLE FOR TOTAL DISSOLVED SOLIDS

To measure total dissolved solids, take a clean porcelain dish which has been washed and dried in a hot air oven at 180°C for one hour,

- Now weigh the empty evaporating dish in analytical balance. Let's denote the weight measured as $W_1 = 35.4329$ g.
- Mix sample well and pour into a funnel with filter paper. Filter approximately 80-100 mL of sample.
- Using pipette transfer 75 mL of unfiltered sample in the porcelain dish.
- Switch on the oven and allowed to reach 105°C. Check and regulate oven and furnace temperatures frequently to maintain the desired temperature range.
- Place it in the hot air oven and care should be taken to prevent splattering of sample during evaporation or boiling.
- Dry the sample to get constant mass. Drying for long duration usually 1 to 2 hours is done to eliminate necessity of checking for constant mass.
- Cool the container in a desiccator. Desiccators are designed to provide an environment of standard dryness. This is maintained by the desiccant found inside. Don't leave the lid off for prolonged periods or the desiccant will soon be exhausted. Keep desiccator cover greased with the appropriate type of lubricant in order to seal the desiccator and prevent moisture from entering the desiccator as the test glassware cools.

- We should weigh the dish as soon as it has cooled to avoid absorption of moisture due to its hygroscopic nature. Samples need to be measured accurately, weighed carefully, and dried and cooled completely.
- Note the weight with residue as $W_2 = 35.4498$ g.

TESTING OF SAMPLE FOR TOTAL SUSPENDED SOLIDS

- Place filtration apparatus with weighed filter in filter flask.
- Mix sample well and pour into a graduated cylinder to the selected volume.
- Apply suction to filter flask and seat filter with a small amount of distilled water.
- Pour selected volume into filtration apparatus.
- Draw sample through filter into filter flask.
- Rinse graduated cylinder into filtration apparatus with three successive 10 ml portions of distilled water, allowing complete drainage between each rinsing.
- Continue suction for three minutes after filtration of final rinse is completed.
- Dry filter in an oven at 103-105°C for at least 1 hour.
- Cool filter in desiccator to room temperature.
- When cool, weigh the filter and support.

CALCULATION TABLE

Total Dissolved Solids

Description		Weight (g)
Weight of the clean porcelain evaporating dish (g)	W_1	
Weight of the dish and the residue (g)	W_2	
Weight of residue (g)	W	
Volume of the Sample (mL)	V	
Total Dissolved Solids (mg/L)	**TDS**	

Tabulation for Total Dissolved Solids (TDS):

Weight of the clean porcelain evaporating dish (g)	$W_1 = 35.4329$
Weight of the dish and the residue (g)	$W_2 = 35.4498$
Weight of residue (g)	$W = 0.0169$
The volume of the sample (mL)	$V = 50.0$

TOTAL SUSPENDED SOLIDS

Description		Weight (g)
Weight of the clean filter paper (g)	W_1	
Weight of the filter paper and the residue (g)	W_2	
Weight of residue (g)	W	
Volume of the sample (mL)	V	
Total Suspended Solids (mg/L)	TSS	

Tabulation for Total Suspended Solids (TSS)

Weight of the clean filter paper (g)	$W_1 = 1.6329$
Weight of the clean filter paper and the residue (g)	$W_2 = 1.6531$
Weight of residue (g)	$W = 0.0202$
Volume of the sample (mL)	$V = 100.0$

EXPERIMENT NO. 2

OBJECT:

Determination of turbidity.

THEORY:

The term turbid is applied to waters containing suspended matter that interferes with the passage of light through the water. The turbidity may be caused by a wide variety of suspended material, which range in size from colloidal to coarse dispersions, depending upon the degree of turbulence. In rivers under flood conditions, turbidity will be due to relatively coarse dispersions.

Turbidity is an important consideration in public water supplies. Turbid waters are objected to by consumers because of their appearance and because of their possible association with sewage pollution and health hazard occassioned by it. Filteration of water is rendered more difficult and costly when turbidity increases. Turbidity may also affect the efficiency of disinfection.

An arbitrary standard has been adopted to express turbidity. The standard is 1 mg of SiO_2/litre = 1 unit of turbidity and the silica used must meet certain specifications.

The U.S.P.H. Service has placed a limit of 10 units of turbidity as the maximum amount allowable in public water supplies.

INSTRUMENTS AND PROCEDURES:

Paterson turbidity meter and Hellige turbidity testing apparatus are described below:

1. Paterson Turbidity Meter:

The Paterson turbidity meter is an optical instrument designed to enable the clarify of water or other liquids with ease and rapidity and at the same time to ensure that such determination is accurate and comparable to recognised standard.

Briefly described, the apparatus consists of a vertical ebonite body which has been specially designed to hold one litre of water (although the quantity is not important provided the mirrors are completely covered). On the back and front faces of the body, protected by aluminium back plates, are mounted two mirrors (the reflecting surfaces of which face each other at a slight predetermined angle). In each mirror an aperture is formed, one of these permitting the passage of a light ray from a lamp of standard strength, whilst the other allows the opposing mirror to be viewed. The ebonite head contains the mechanism by which a slider can be raised or lowered, and is removable for filling and/or cleaning purposes. This slider is actuated by the ebonite knob on the right hand side of the head.

On filling the instrument and switching on the attached electric light, multiple reflections are obtained of the original light spot. These reflections being visible as a row of light spots. Visibility is being a measure of the turbidity of the fluid under examination.

2. Hellige Apparatus:

For turbidity measurement, it consists of a graduated and marked tape at the end of which is attached a graduated aluminium strip, and a platinum needle.

Following procedure is used to determine turbidity.

1. Insert the screw-eye into the aluminium strip so that the platinum needle projects from the side with the scale.
2. Insert the end of a 3 ft long stick into the opening of the screw-eye. Hold the tape taut, immerse the aluminium strip in the water, and observe the needle from the point marked "PLACE EYE HERE" at the upper end of the tape.
3. When the needle just disappears read the depth of immersion from the scale on the tape. The number indicates the turbidity directly in parts per million (ppm) of suspended silica.
4. If the water is so turbid that the Needle disappears when the aluminium strip is immersed, take the reading from the scale of the latter.

PRECAUTIONS:

1. Measurement must always be made in the open, preferably during the middle of the day. If the sun is shining, the observer should take such a position that this shadow covers the water around the stick and needle.
2. If the water is agitated by waves or currents, if the depth of the water is not sufficient to immerse the strip until the needle disappears, or if for any other reason the measurement cannot be made properly, a pail or tub may be filled with the water and the reading should be taken immediately before any particles have settled.
3. For highest accuracy the diameter of the vessel must be at least twice the depth to which the needle is immersed.
4. Dry all parts of the apparatus before they are replaced in the case.

EXPERIMENT NO. 3

OBJECT:

Determination of the pH value of water.

THEORY:

pH is the logarithm of the reciprocal of the hydrogen ion concentration — more precisely, of hydrogen ion achieving — in moles per litre. The practical pH scale extends from 0 (very acidic), to 14 (very alkaline), with middle value corresponding to exact neutrality at 25°C. Whereas, alkalinity and acidity express the total reserve or buffering capacity of a sample, the pH value represents the instantaneous hydrogen ion activity.

pH enters into calculation of carbonate, bicarbonate, and carbon dioxide, as well as of the corrosion or stability index and into the control of water and waste water treatment processes.

The pH of most natural waters falls within the range of 4 to 9. The majority of waters are slightly basic due to the presence of carbonate and bicarbonate. A departure from the normal for a given water could be caused by the entry of strongly acidic or basic industrial wastes. A relatively common practice is the pH adjustment of treatment plant effluent for the purpose of controlling corrosion in the distribution system.

METHODS OF pH MEASUREMENT:

pH can be measured by two methods, colorimetrically and electrometrically. The colorimetric methods suffer from severe interference contributed by colour, turbidity, high saline content, celloidal matter, free chlorine, and various oxidants and reductants. The indicators are subjected to deterioration, as are and colour standards with which they are compared. Moreover no single indicator encompasses the pH range of interest in water. For these reasons, the colorimetric method is suitable only for rough estimation and is not considered good for the detailed determination of pH value. The electrometric method also known as Glass Electrode Method is considered standard.

Glass Electrode Method:

This method involves use of an equipment called pH meter, with a pair of electrodes. Several types of electrodes have been suggested for electrometric determination of pH. Although the hydrogen gas electrode is recognized as the primary standard, the glass electrode ion combination with the reference potential provided by a saturated calomel electrode is most generally used.

When a pair of electrodes namely, pH sensitive glass electrode and a reference electrode are dipped in an aqueous solution, they generate e.m.f. which is proportional to the pH of the solution (change of 1 pH unit produces an electrical charge of 59.1 mV at 25°C. The pH meter essentially measures this electrical change and gives direct reading of pH over a calibrated scale. Necessary feedbacks are incorporated to achieve good stability and freedom from aging compensation for temperature and asymmetry potential effect is also incorporated in the instrument.

In our laboratory we have SYSTRONICS pH meter Type 322. For specifications and other details and manual available in the laboratory may be consulted. The procedure described below for determination of pH value is as prescribed by the manufacturer.

PROCEDURE:

1. Keep the CHECK/ZERO/READ switch in ZERO position. Switch on the instrument. After one minute, adjust the zero by 'SET ZERO' control. Then allow ten minutes for warming and to stabilize. Wash the electrodes by a jet of distilled water and wipe them with a soft tissue paper or filter paper.
2. Immerse the electrodes in the beaker containing standard buffer solution. Set 'RANGE' switch in appropriate pH range. Set the zero (7 pH) by 'SET ZERO' control, accurately making use of the mirror on the scale. Measure temperature in degrees centigrade of the solution by a thermometer and set this value on the 'TEMPCOM' control. Then set the CHECK/ZERO/READ switch in the READ position. Now set the buffer pH value on the meter by means of 'CALIBRATE' control. Put back the ZERO/READ switch in ZERO position. Now the meter is standardized. Take out the electrodes. Wash them with distilled water and wipe them with filter paper.
3. To measure the pH of unknown solution, immerse the electrodes in that solution, select the proper range in the 'RANGE' switch. Set the ZERO/READ switch in 'READ' position and read the pH indicated by the meter on the scale to which 'RANGE' is set. The scale reads pH 7-14 from left to right and pH 0-7 from right to left.
4. When a series of measurements are being taken, check the zero and correct it, if necessary, by putting ZERO/READ switch in 'ZERO' position and then adjusting 'SET ZERO' control.

RESULT:

The pH value of water =
Standard (recommended value) =
The recommended value of pH =

EXPERIMENT NO. 6

OBJECT:

To determine the hardness.

THEORY:

Water is universal solvent and dissolves varying amounts of different mineral substances. Those producing hardness do not affect the sanitary quality but are of importance in the domestic use of water, particularly for laundry and boiler purposes. Calcium and magnesium salts, the principal mineral constituents, consume soap and precipitate as insoluble compounds or soap curds until all the calcium and magnesium is precipitated, no lather or washing action is obtained from the soap; the soap consuming power of water is, therefore, a measure of its hardness. Calcium and magnesium generally are dissolved as soluble bicarbonate but may change, owing to heating, to the less soluble carbonate which precipitates and is one source of scale in distributing systems and hot-water heaters. Hard waters are usually less corrosive than soft waters.

PRINCIPLE (EDTA METHOD):

Ethylene diaminetetra-acetic acid and its sodium salts (abbreviated EDTA) form a chelated soluble complex when added to a solution of certain metal cations. If a small amount of a dye such as chrome black T is added to an aqueous solution containing calcium and magnesium ions at a pH of 10.0 + 0.1 the solution will become wine red. If EDTA is then added as titrant, the calcium and magnesium will be complexed. After sufficient EDTA has been added to complex, the solution will turn from wine red to blue. This is the end point of the titration. The sharpness of the end point increases with increasing pH, however, cannot be increased indefinitely because of the danger of precipitating $CaCO_3$ or $Mg(OH)_2$, and because the dye changes colour at high pH values. The pH value of 10.0 + 0.1 (recommended in this procedure) is a satisfactory compromise. A limit (5 min) is set for the duration of the titration in order to minimise the tendency towards $CaCO_3$ precipitation.

Some metal ions interfere with this procedure by causing fading or indistinct end points. This interference is reduced by the addition of certain inhibitors to the water sample prior to titration with EDTA.

APPARATUS:

Burette.

Reagents:

Buffer solution, chrome black *T* indicator, standard EDTA titrant, 0.01 N (1 ml = 1.000 mg $CaCO_3$)

PROCEDURE:

1. Dilute 25 ml of sample to about 50 ml with distilled water in suitable vessel.
2. Add 1-2 ml of buffer solution to give a pH value of 10.0 to 10.1.
3. Add 1-2 drops of indicator solution.
4. Add the standard EDTA slowly with continuous stirring, until the last reddish tinge disappears from the solution, adding the last few drops at 3-5 seconds intervals. The colour of the solution at the end point is blue under normal conditions.

CALCULATIONS:

$$\text{Hardness (EDTA) as mg/1 CaCO}_3 = \frac{\text{ml EDTA titrant} \times 1000 \times f}{\text{ml sample}}$$

where $f = \dfrac{\text{mg CaCO}_3}{\text{ml EDTA titrant}}$

EXPERIMENT NO. 7

OBJECT:

To determine the chlorides in water.

THEORY:

The test for the content of chloride in water may be used for various purposes:

1. Salt is present in sewage from urine in the range of about 50-200 mg/1 and higher yet in countries with low water consumption per capita, so the content of chlorides in polluted water is a rough measure of the degree of solution. This is especially the case with well waters, in which seepage from cess-pools in the groundwater tributary of a well may be disclosed. This, however, required that the normal chloride content of the unpolluted surface and groundwaters be known for comparison purposes. For instance, the normal content in groundwater in a given neighbourhood may be 0.5 mg/1, so a content of, say, 2.2 mg/1 in a well water in the vicinity would disclose significant seepage.
2. Water has salty taste to some people when the contents of chlorides exceeds about 150 mg/1 and 200 mg/1 is the maximum desirable contents. A high chloride content also exerts a deleterious effect on metallic pipes and structures, as well as on agricultural plants.
3. The chloride content of groundwaters is used in the study of salt-water intrusion along the sea coast, which destroys the usefulness of wells so affected. Local deposits of rock salt naturally lead to high chloride content in ground waters — that is salty water not related to pollution or sea water intrusion — and hence without sanitary signification.
4. The test for chloride is used in the control of the regeneration of ion-exchange software with solutions of salt.

Principle:

In a neutral or slightly alkaline solution, potassium chromate can be used to indicate the end point of the silver nitrate titration of chloride. Silver chloride is quantitatively precipitated before red silver chromate is formed.

Substances in amounts normally found in potable waters will not interfere. However, the sample may have to be pretreated for eliminating interference.

Equations:

$$NaCl + AgNO_3 = \underset{\text{(white precipitate)}}{AgCl} + NaNO_3$$

$$2AgNO_3 + K_2CrO_4 = Ag_2CrO_4 + 2KNO_3$$

APPARATUS:

Burette, 1 ml pipette.

Reagents:

Potassium chromate indicator solution, standard silver nitrate titrant (0.0141 N).

PROCEDURE:

To the sample as collected in a 250-300 ml bottle, add 2 ml $MnSO_4$ solution followed by 2 ml alkali-iodide-azide reagent, well below the surface of the liquid stopper with care to exclude air bubbles and mix by inverting the bottle several times. When the precipitate settles, leaving a clear supernatent above the manganese hydroxide floc, shake again. When setting has produced at least 100 ml clear supernatent carefully remove the stopper and immediately add 2.0 ml cone. H_2SO_4 by allowing the acid to run down the neck of the bottle, and mix by gentle inversion until dissolution is complete. The iodine should be uniformly distributed throughout the bottle before decanting the amount needed for titration. This should correspond to 200 ml of the original sample after correction for the loss corresponds to 200 ml of the original sample after correction for the loss of sample by displacement with the reagents has been made. Thus, when a total of 4 ml (2 ml each) of the manganous sulfate and alkali-iodide-azide reagents is added to a 300-ml bottle, the volume taken for titration should be

$$200 \times \frac{300}{300-4} = 203 \text{ ml.}$$

Titrate with 0.025 N thiosulfate to a pale straw colour.

Add 1-2 ml freshly prepared starch solution and continue the titration the first disappearance of the blue colour. Subsequent recolorations due to the catalytic effect of nitrite or to traces of ferric salts which have not been complexed with fluoride should be disregarded.

CALCULATIONS:

Since 1 ml 0.02 N $Na_2S_2O_3$ is equivalent to 0.2 mg DO, each milliliter of sodium thiosulfate used is equivalent to 1 mg/1 DO if a volume equal to 200 ml of original sample is titrated.

If the results are desired in milliliters of oxygen gas per liter at 0°C and 760 mm pressure, multiply mg/1 DO by 0.698.

To express the results as per cent saturation at 760 mm atmospheric pressure, the solubility data may be used. Formulae are available for correcting the solubilities to barometric pressures other than mean sea level.

The solubility of DO in distilled water at any barometric pressure, P (mm Hg), temperature, t (degree C), and saturated vapor pressure, u (mm Hg), for the given t, may be calculated between the temperature of 0 and 30°C by Eq. 1, and between 30 and 50°C by Eq. 2:

$$\text{ml/l DO} = \frac{(P-u)\times 0.678}{35+t} \qquad \ldots(1)$$

$$\text{ml/l DO} = \frac{(P-u)\times 0.827}{49+t} \qquad \ldots(2)$$