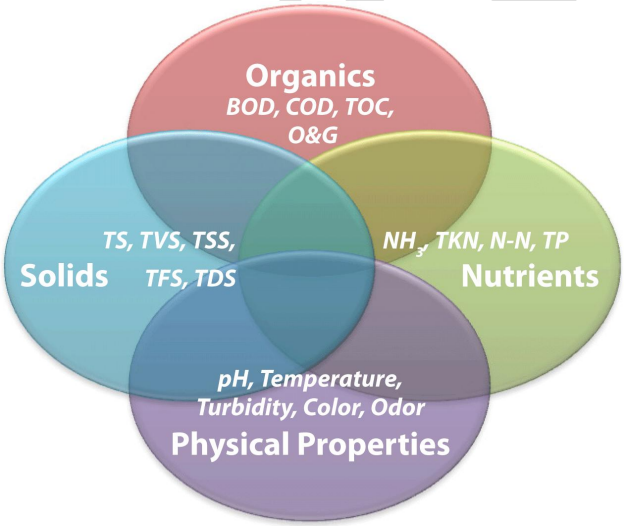
[11] CHEMICAL OXYGEN DEMAND COD

Chemical Oxygen Demand (COD) of domestic and industrial wastewater is a measurement of the amount of oxygen required to oxidize (decrease) the organic matter content of a sample that is susceptible to oxidation by strong oxidant (such as potassium dichromate). Most organic compound will oxidize under the influence of oxidizing agents in acid conditions-oxidation of most organic compounds is 95-100% of the theoretical value.

BOD values are often lower than COD values as some materials (such as cellulose) will react with the dichromate present in COD tests, but not the oxygen present under biological conditions.



COD and BOD values are directly comparable, and are often used side-by-side to allow for differentiation between biologically oxidiazable matter and biologically inert matter. If the COD value of water sample is much larger than the BOD value, it suggests that the sample contains large amounts of organic substances that are not easily biodegradable. COD tests have the advantage of being relatively quick to perform, with results available in about three hours, although they are subject to some interferences and errors.

The basis for the COD test is that nearly all organic compounds can be fully oxidized to carbon dioxide with a strong oxidizing agent under acidic conditions. The amount of oxygen required to oxidize an organic compound to carbon dioxide, ammonia, and water is given by:

$$C_nH_oO_pN_c + \left(n + \frac{a}{4} - \frac{b}{2} - \frac{3}{4}c\right)O_2 \rightarrow nCO_2 + \left(\frac{a}{2} - \frac{3}{2}c\right) + H_2O + cNH_3$$

The COD determination is based on the principal that organic material in water can be oxidized by potassium dichromate in acidic solution in the presence of a silver catalyst.

The dichromate reflux method is preferred over procedures using other oxidants because of its superior oxidizing ability, applicability to a wide range of samples and ease of manipulation. Under these experimental conditions, some of the dichromate is reduced by the organic matter in the water sample and the remainder is titrated with ferrous ammonium sulfate using a ferroin (1,10-phenophthaline) indicator to indicate the end point of the reaction. The ferroin forms an intense red color with ferrous (Fe^{2+}) ions but no color with ferric (Fe^{3+}) ions. During the reaction, most of the organic matter in the sample is oxidized to carbondioxide and water, while the dichromateis reduced to trivalent chromium.

$$\mathbf{Cr_2O_7^{2-} + 6Fe^{2+} + 14H+ \rightarrow 2Cr^{3+} + 6Fe^{3+} + 7H_2O}$$

RED **NO COLOR**

The more organic matter present in the sample, then the more dichromate is reduced. The same test procedure is used for a blank sample of double-distilled, deionized water so that any errors arising from the presence of extraneous organic matter in the reagents can be accounted for.

Time Required

Three hours, depending upon the number of samples determined (assuming all reagents are made up in advance)

Equipment:

1. 1.5 and 10 ml pipette and pipette filler
2. 20 ml measuring cylinder
3. Digestion vessels
4. Block heater or oven, to operate at 150 ± 2°C. Ampoules sealer: use only a mechanical sealer to insure strong, consistence seals.
5. 50 ml burette
6. 100 ml conical flasks
7. Boss, clamp , retort stand and white tile

Reagents:

1. Standard potassium dichromate digestion solution ($K_2Cr_2O_7$) that contains $HgSO_4$.
2. Sulfuric acid reagent. Ag_2SO_4 with concentrated H_2SO_4
3. Standard Ferrous Ammonium Sulfate (FAS) $(NH_4)_2SO_4.FeSO_4.6(H_2O)$
4. Ferroin indicator solution.
5. Sulphric acid; *required only if the interference of nitrites to be eliminated.*

6. Potassium hydrogen phathalate (KHP) standard.

Lightly crush and then dry potassium hydrogen phthalate ($HOOCC_6H_4COOK$) to constant weight at 120 °C. dissolve 425 mg in distilled water and dilute to 1000 ml. KHP has a theoretical COD of 1.176 mg O2/mg and this solution has a theoretical COD of 500 µgO2/ml and this solution is stable when refrigerated up to three months in the absence of visible biological growth.

Significant experimental hazards

- Student should be aware of hazards associated with the use of all glassware (cuts), electrical equipment (shock, burns, and fire), and tongs to move hot objects (burns and dropping hazards) and fume cupboards. This method requires the handling and boiling a strong solution of sulfuric acid potassium dichromate and so great care is necessary.

CAUTION !
Wear face shield and protect hands from heat produced when contents of vessels are mixed. Mix thoroughly before applying heat to prevent local heating of vessel bottom and possible explosive reaction

- Sewage and final effluent samples contain harmful bacteria and may contain toxic organic substances and metals (harmful if ingested)
- Sulfuric acid is harmful by ingestion and may cause severe burns to eyes and skin.
- Ferroin indicator maybe harmful if ingested in quantity and may irritate eyes and skin. It may also stain clothing and skin.
- Ferrous ammonium sulfate may be harmful if ingested in quantity and may irritate eyes and skin.
- Potassium dichromate is harmful by ingestion, inhalation and skin contact. There is also danger of combustion with organic matter.
- Mercuric sulfate is toxic by ingestion and may irritate and burn eyes and skin.
- Silver sulfate is toxic by ingestion and may irritate eyes and skin.

Procedure:

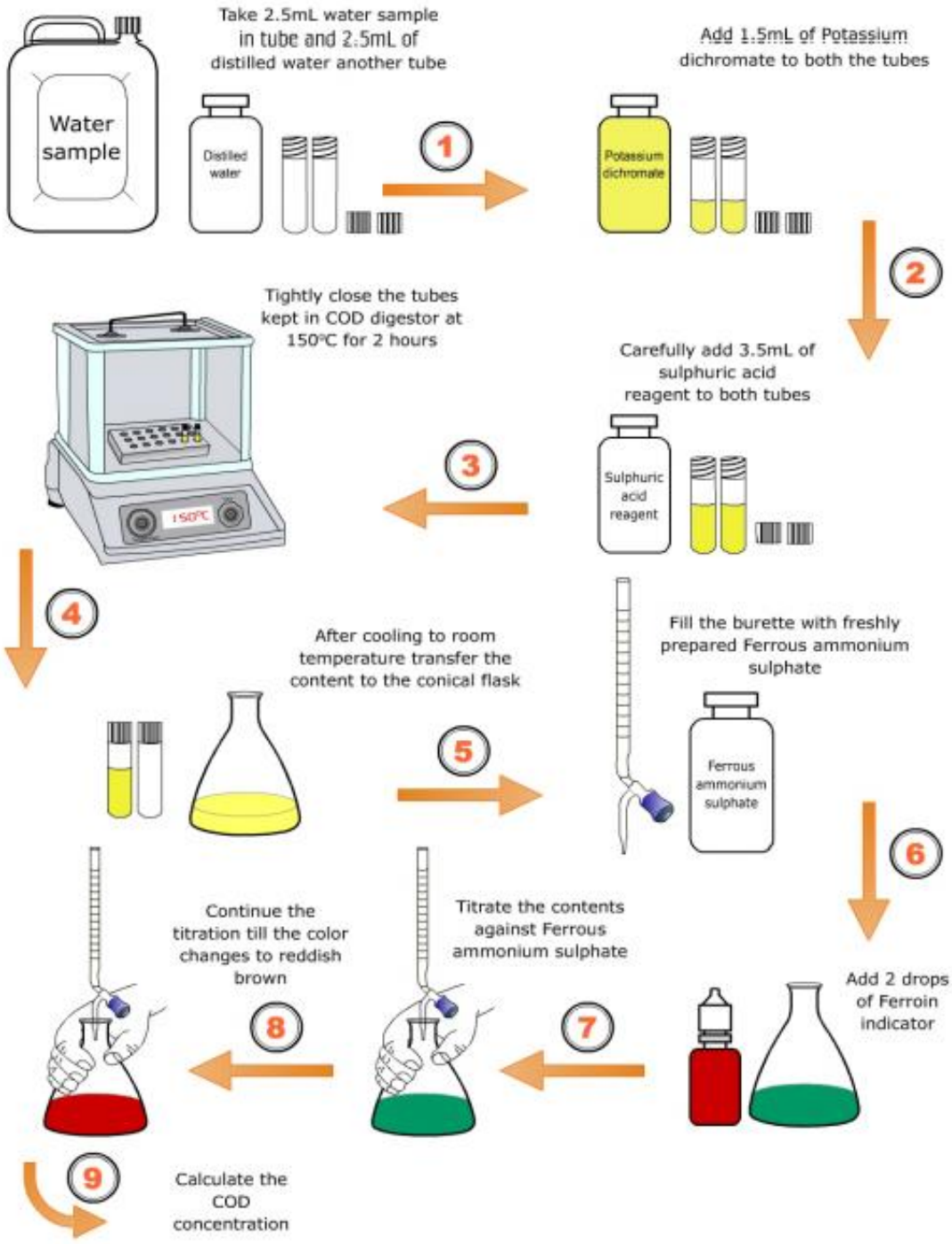
(1) Wash culture tubes and caps with 20% H_2SO_4 before first use to prevent contamination
(2) Use the table below for proper sample and reagent volumes

Digestion Vessel	Sample *ml*	Digestion Solution *ml*	Sulfuric Acid Reagent *ml*	Total Final Volume *ml*
Standard 10-ml ampules	**2.5**	**1.5**	**3.5**	**7.5**

(3) Place 2.5 ml of sample in a labeled ampoules and add the digestion solution.
(4) Carefully rundown sulfuric acid reagent down inside of vessel so an acid layer is formed under the sample digestion solution layer.
(5) Cap tubes or seal the ampoules tightly, swirl carefully to ensure through mixing.
(6)
(7) Place the tubes in block digester or oven preheated to 150 °C and reflux for 2 hr.
(If vapor escapes from the top or side of tubes stop the experiment and be careful of the vapor touching your skin)
(8) Leave the tubes to cool to room temperature for about 15 min and then place them in test tube rack.
(9) Remove culture tube caps and transfer the content to a larger flask for titrating.
(10) Add 1-2 drops of ferroin indicator and stir rapidly either by your hand or magnetic stirrer while titrating with FAS.
(11) The END point is sharp color change from **BLUE-GREEN** to **REDDISH BROWN**, although the blue green may reappear within minutes.
(12) Repeat the steps 7-9 for the blank containing only the reagents and a volume of distilled water equal to that of sample

NOTE
The above procedure is for the determination of one sample. It is desirable to perform the determination in duplicate if possible.

PROCEDURE CHART



Calculation

$$COD\ as\ mg\ O_2/l = \frac{(A-B)\times M\times 8000}{Vol\ of\ sample\ (ml)}$$

Where,
A= Volume of FAS used for the blank (ml)
B = Volume of FAS used for the sample (ml)
M = Morality of FAS

From the equation for the titration, we can see that 1 mole of $Cr_2O_7^{2-}$ reacts with 6 moles of Fe^{2+}

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

Consequently:

$$1 \text{ moles } O_2 \equiv 3/2 \text{ mole } Cr_2O_7^{2-}$$

And hence:

$$1 \text{ mole } O_2 \equiv 3/2 \text{ mole } Cr_2O_7^{2-} \equiv 6 \text{ moles } Fe^{2+}$$
$$1 \text{ mole } O_2 \equiv 6 \text{ moles } Fe^{2+}$$
$$\mathbf{1/6 \text{ mole } O_2 \equiv 1 \text{ mole } Fe^{2+}}$$

During the refluxing process, the organic carbon will be oxidized to carbon dioxide (CO_2); this will require the release of four electrons (i,e. $C^0 \rightarrow C^{4+}$). However, six electrons are required to reduce the dichromate (in which Cr has an oxidation state of +6) to $2Cr^{3+}$.

Issues to consider for your practical report

- What are the potential sources of error and interferences in this analytical determination? How could they be overcome?
- Identify the advantage and disadvantages of this COD test.
- Are there alternative methods for determining the COD of wastewater sample? If so, how do they compare to this method?
- What is the source of the water sample?
- What are typical COD values for domestic and industrial wastewater? How do your data compare with these values?
- Why is the digestion temperature of 150°C used in this determination?

Experiment (11): Chemical Oxygen Demand Experimental Results	
Name	**Date**
ID No.	**Group**

ID	Volume of sample, ml	Initial Reading of Burette	Final Reading of Burette	Volume of FAS , ml	COD (mg/l)	Notes

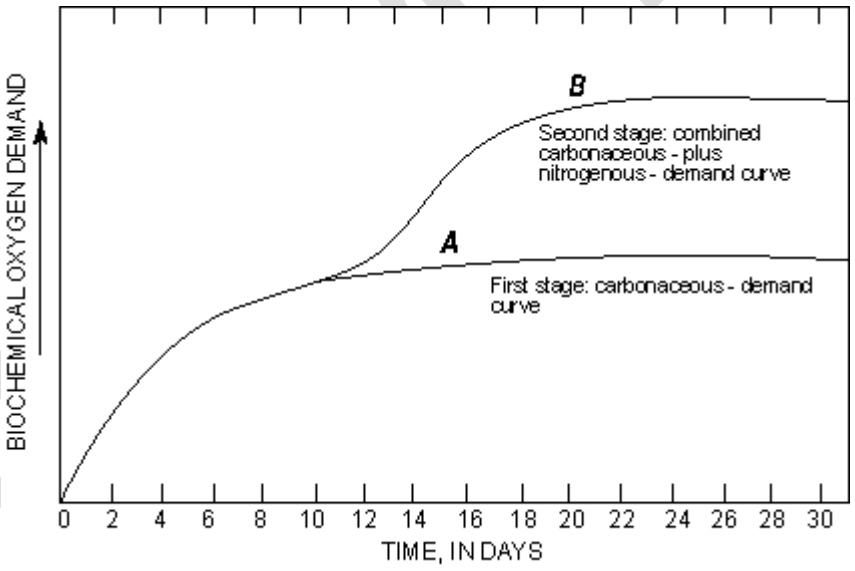
Observations & Calculations:

[12] BIOCHEMICAL OXYGEN BEMAND

Biochemical oxygen demand determination is a chemical procedure for determining the amount of dissolved oxygen needed by aerobic organisms in a water body to break the organic materials present in the given water sample at certain temperature over a specific period of time.

BOD of water or polluted water is the amount of oxygen required for the biological decomposition of dissolved organic matter to occur under standard condition at a standardized time and temperature. Usually, the time is taken as 5 days and the temperature is 20°C.

The test measures the molecular oxygen utilized during a specified incubation period for the biochemical degradation of organic material (carbonaceous demand) and the oxygen used to oxidize inorganic material such as sulfides and ferrous ion. It also may measure the amount of oxygen used to oxidize reduced forms of nitrogen (nitrogenous demand).



Biochemical oxygen demand curves: (*A*) typical carbonaceous-demand curve showing the oxidation of organic matter, and (*B*) typical carbonaceous- plus nitrogeneous-demand curve showing the oxidation of ammonia and nitrite. (Modified from Sawyer and McCarty, 1978.)

BOD is the principle test to give an idea of the biodegradability of any sample and strength of the waste. Hence the amount of pollution can be easily measured by it. Efficiency of any treatment plant can be judged by considering influent BOD and the effluent BOD and so also the organic loading on the unit.

Application of the test to organic waste discharges allows calculation of the effect of the discharges on the oxygen resources of the receiving water. Data from BOD tests are used for the development of engineering criteria for the design of wastewater treatment plants.
Ordinary domestic sewage may have a BOD of 200 mg/L. Any effluent to be discharged into natural bodies of water should have BOD less than 30 mg/L.
This is important parameter to assess the pollution of surface waters and ground waters where contamination occurred due to disposal of domestic and industrial effluents.
Drinking water usually has a BOD of less than 1 mg/L. But, when BOD value reaches 5 mg/L, the water is doubtful in purity.
The determination of BOD is used in studies to measure the self-purification capacity of streams and serves regulatory authorities as a means of checking on the quality of effluents discharged to stream waters.
The determination of the BOD of wastes is useful in the design of treatment facilities.

Principle

The sample is filled in an airtight bottle and incubated at specific temperature for 5 days.
The dissolved oxygen (DO) content of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between initial and final DO.
The sample is filled in an airtight bottle and incubated at specific temperature for 5 days.
The dissolved oxygen (DO) content of the sample is determined before and after five days of incubation at 20°C and the BOD is calculated from the difference between initial and final DO.
The initial DO is determined shortly after the dilution is made; all oxygen uptake occurring after this measurement is included in the BOD measurement.

Equipment:

- BOD Incubator
- Burette & Burette stand
- 300 ml glass stopper BOD bottles
- 250 ml conical flask
- Pipettes with elongated tips
- Pipette bulb
- 100 ml graduated cylinders
- Washing bottle
- Sodium azide Mixture solution
- Concentrated sulfuric acid
- Starch indicator
- Sodium thiosulphate
- Distilled or deionized
- Dilution water

PRECAUTIONS

The following precautions should be observed while performing the experiment:

1. Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L. Discard dilution water if there is any sign of biological growth.

2. The sample should be adjusted to a pH between 6.5 and 7.5, using sulfuric acid for samples with pH in the alkaline side i.e., greater than 7.5 or sodium hydroxide for samples with pH in the acidic side i.e., less than 6.5.

3. Add sodium sulfite (Na_2SO_3) to remove residual chlorine, if necessary. Samples containing toxic metals, arsenic, or cyanide often require special study and pretreatment.

4. While still letting sample water flow down the tube, slowly pull the tube from the bottom of the bottle and fill the bottle to its brim. Check for bubbles. Carefully stopper the BOD bottle as described above.

Significant experimental hazards

- Student should be aware of hazards associated with the use of all glassware (cuts), electrical equipment (shock, burns) and fume cupboards.
- Sulfuric acid is harmful by ingestion and may cause severe burns to eyes and skin.
- Sodium thiosulfate maybe harmful if ingested in quantity and may irritate eyes and skin
- Alkaline iodide-azide solution causes severe burns and may be harmful if ingested.
- Manganese sulfate solution is be harmful by ingestion and may irritate eyes and skin.

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.
- If Analysis is to be carried out within two hours of collection, cool storage is not necessary. If analysis cannot be started with in the two hours of sample collection to reduce the change in sample, keep all samples at 4° C.
- Do not allow samples to freeze.
- Do not open sample bottle before analysis.
- Begin analysis within six hours of sample collection.

Dilution Water

High quality organic free water must be used for dilution purposes.

The required volume of water (five litres of organic free distilled water) is aerated with a supply of clean compressed air for at least 12 hours. Allow it to stabilize by incubating it at 20ºC for at least 4 hours.

For the test we have taken five litres of organic free aerated distilled water, hence add 5mL each of the nutrients.

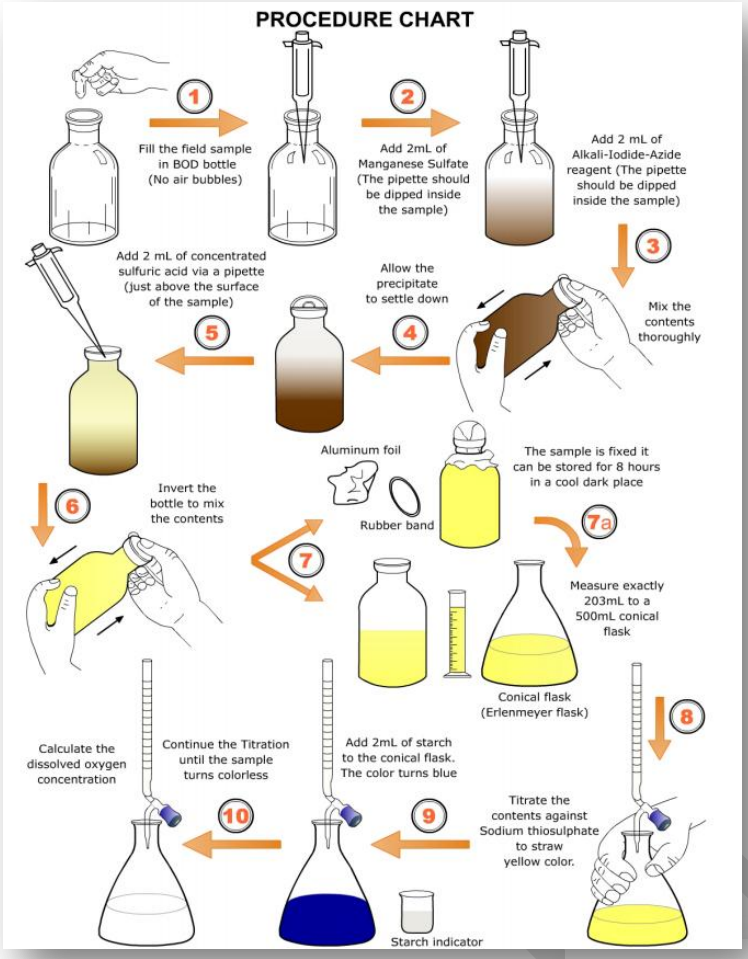
- Add 5mL calcium chloride solution
- Add 5mL magnesium sulphate solution
- Add 5mL ferric chloride solution
- Add 5mL phosphate buffer solution

This is the standard dilution water. Prepare dilution water 3 to 5 days before initiating BOD test to ensure that the BOD of the dilution water is less than 0.2 mg/L.

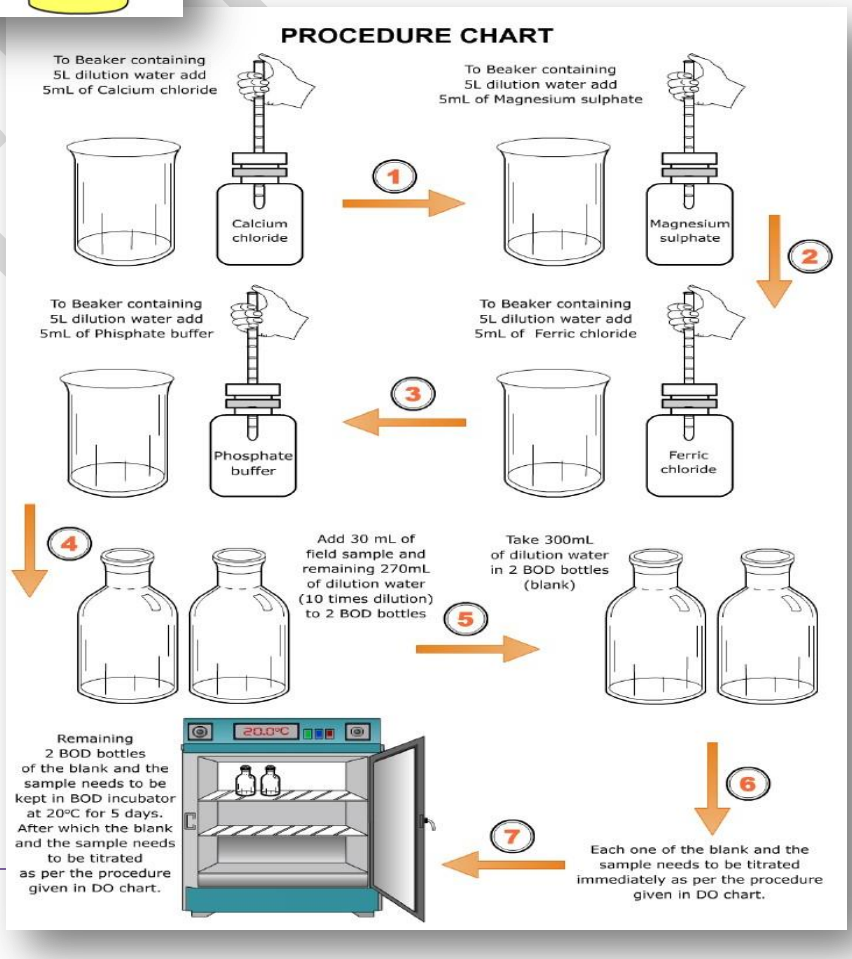
Procedure

1. Take Three 300 ml glass BOD bottles (two for the sample and one for the blank).
2. Add 10 ml of the sample to each of the two BOD bottles and the fill the remaining quantity with the dilution water. i.e., we have diluted the sample 30 times.
3. The remaining BOD bottle is for blank, to these bottles add dilution water alone.
4. After the addition immediately place the glass stopper over the BOD bottles and note down the numbers of the bottle for identification.
5. Now preserve one blank solution bottle and one sample solution bottle in a BOD incubator at 20ºC for five days.
6. The other two bottles (one blank and one sample) needs to be analyzed immediately.
7. Avoid any kind of bubbling and trapping of air bubbles.
8. Add 2mL of manganese sulfate to the BOD bottle by inserting the calibrated pipette just below the surface of the liquid.
9. Add 2 ml of alkali-iodide-azide reagent in the same manner. (The pipette should be dipped inside the sample while adding the above two reagents. If the reagent is added above the sample surface, you will introduce oxygen into the sample.)
10. Allow it to settle for sufficient time in order to react completely with oxygen. When this floc has settled to the bottom, shake the contents thoroughly by turning it upside down.
11. Add 2 mL of concentrated sulfuric acid via a pipette held just above the surface of the sample.
12. Carefully stopper and invert several times to dissolve the floc.
13. Titration needs to be started immediately after the transfer of the contents to Erlenmeyer flask.
14. Measure out 203 ml of the solution from the bottle and transfer to an Erlenmeyer flask.
15. Titrate the solution with standard sodium thiosulphate solution until the yellow color of liberated Iodine is almost faded out. (Pale yellow color)
16. Add 1 ml of starch solution and continue the titration until the blue color disappears to colorless.

Remember
NO bubbles!



17. Note down the volume of sodium thiosulphate solution added, which gives the D.O. in mg/L.
18. Repeat the titration for concordant values.
19. After five days, take out the bottles from the BOD incubator and analyze the sample and the blank for DO.
20. Repeat the titration for concordant values.



Calculations

$$Dissoved\ Oxygen = \frac{Vol\ of\ Thiosulphat \times 0.2 \times 1000}{Volume\ of\ sample}$$

$$BOD_T, \frac{mg}{l} = \frac{(S_{init} - S_{Fin}) - (B_{init} - B_{Fin})f}{P}$$

$$f = \frac{Volume\ of\ seed\ in\ diluted\ sample}{Volume\ of\ seed\ in\ seed\ control}$$

$$P = \frac{Vol\ of\ sample}{Volume\ of\ bottle}$$

Where,

$\mathbf{BOD_T}$: BOD at T Time

$\mathbf{S_{init}}$: DO of diluted sample immediately after preparation, mg/l

$\mathbf{S_{Fin}}$: DO of diluted sample after T d incubation at 20oC, mg/l

$\mathbf{B_{init}}$: DO of seed control (Blank) before incubation, mg/l

$\mathbf{B_{Fin}}$: DO of seed control after (Blank) incubation mg/l

$\boldsymbol{f}$: Ratio of seed in diluted sample to seed in seed control = (% seed in diluted sample)/ (% seed in seed control).

$\boldsymbol{P}$: Decimal volumetric fraction of sample used

Issues to consider for your practical report

- What are the potential sources of error and interferences in this analytical determination? How could they be overcome?
- Identify the advantage and disadvantages of this BOD test.
- Are there alternative methods for determining the BOD of wastewater sample? If so, how do they compare to this method?
- What is the source of the water sample?
- What are typical BOD values for domestic and industrial wastewater? How do your data compare with these values?

Experiment (12): Biochemical Oxygen Demand Experimental Results	
Name	**Date**
ID No.	**Group**

Blank Sample (*B*):

Trial No.	Sample ID	Day	Volume of sample (ml)	Initial Burette Reading (ml)	Final Burette Reading (ml)	Volume of Titrant (ml)	Dissolved Oxygen (mg/l)	*f*	*P*
1.	Blank								
2.									
3.									
4.									
5.									
6.									

Samples (*S*):

Trial No.	Sample ID	Day	Volume of sample (ml)	Initial Burette Reading (ml)	Final Burette Reading (ml)	Volume of Titrant (ml)	Dissolved Oxygen (mg/l)
1.							
2.							
3.							
4.							
5.							
6.							
7.							

BOD:

Trial No.	Sample ID	BOD mg/l
1.		
2.		
3.		
4.		

Trial No.	Sample ID	BOD mg/l
5.		
6.		
7.		
8.		

Observations & Calculations:

[13] SOULIBILITY PRODUCT

Calcium hydroxide is a soft white caustic powder used in making mortar, cements, calcium salts, paints, and petrochemicals. It is also used in saltwater aquaria to make up kalkwasser/limewater solutions for reef tanks, and is used as a pH regulating agent. Notice that calcium hydroxide is divalent and thus has twice the neutralizing power as molecules like NaOH that are monovalent.

A Calcium Hydroxide Molecule:

Calcium hydroxide is manufactured industrially by adding water to calcium oxide (quicklime) in a strongly exothermic reaction:

$$\mathbf{CaO(s) + H_2O(l) \rightarrow Ca(OH)_2(s)}$$

Calcium hydroxide, $Ca(OH)_2$, is an ionic solid that is only slightly soluble in water.

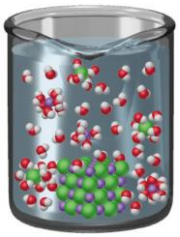
Solid Calcium Hydroxide:

A calcium hydroxide solution is also referred to as limewater. A saturated solution of calcium hydroxide has the solid in equilibrium with its ions as shown below:

$$\mathbf{Ca(OH)_2(s) \leftrightarrow Ca^{2+}(aq) + 2OH^-(aq)}$$

A saturated solution is a solution that contains the maximum amount of dissolved solute possible at a given temperature. (The solution contains undissolved solute in equilibrium with the solution.)Since calcium hydroxide is only slightly soluble in water, it is a difficult base to classify. It is often assumed that since calcium hydroxide has a low solubility that it is a weak base. But it contains hydroxides ions, which automatically makes it a strong base! In fact, the pH of a saturated calcium hydroxide solution is about **12.4**. Thus we can classify a saturated solution of calcium hydroxide as a dilute solution of a strong base.

A molecular view of a saturated solution of calcium hydroxide would look similar to the following, with clumps of undissolved calcium hydroxide at the bottom of the beaker in equilibrium with dissolved Ca^{2+} and OH^- ions (although there would be twice as many OH^- ions than Ca^{2+} ions):



Note that the rate of dissolving is equal to the rate of precipitation in a saturated solution equilibrium.

In this type of equilibrium, the equilibrium constant is called the solubility product, and is represented by the symbol Ksp. Whenever you see the symbol Ksp it refers to a solubility equation, written with the solid to the left of the equilibrium sign, and the dissolved products to the right. The Ksp for this reaction will be:

$$\mathbf{Ksp = [Ca^{2+}][OH^-]^2}$$

(The solid state is not included in a Ksp expression since it is a pure substance and cannot be expressed as a concentration).

Every substance that forms a saturated solution will have a Ksp. However, for very soluble substances like NaCl, the value is so large that the concept is rarely used. In low solubility substances, the value of Ksp is a useful quantity that lets us predict and calculate solubilities of substances in solution. In this experiment you will collect the data that allows you to calculate the solubility of $Ca(OH)_2$, and its Ksp value.

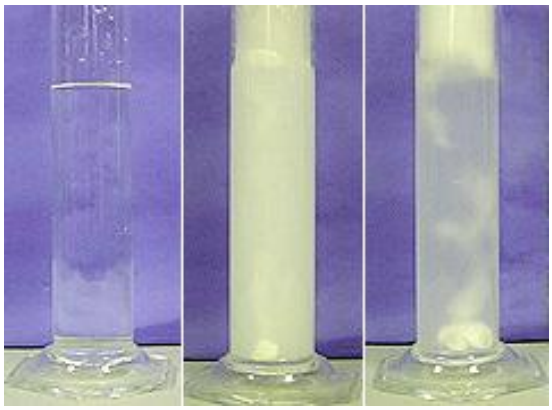
Note that substances that have a large Ksp value have a higher solubility (more dissolved ions); and substances with a small Ksp value have a lower solubility (few dissolved ions).

The concentration of hydroxide ions formed when $Ca(OH)_2$ dissolves can be measured using the titration technique. An acid-base titration is a process in which a measured volume of an acid or base is added to a reaction mixture until the acid-base indicator changes color. In the procedure used in this lab, a dilute solution of HCl is titrated with a saturated solution of $Ca(OH)_2$ to the endpoint of phenolphthalein.

A saturated solution of calcium hydroxide must be made fresh on the day it is to be used as any carbon dioxide that enters the solution will cause it to react to form a calcium carbonate precipitate (chalk), as shown in the first two pictures below:

$$\mathbf{Ca(OH)_2(s) + CO_2(g) \rightarrow CaCO_3(s) + H_2O(l)}$$

Interestingly though, the precipitate will redissolve if the concentration of carbon dioxide is high enough .



In this experiment, you will measure the volume of saturated calcium hydroxide solution required to neutralize a solution of known concentration of hydrochloric acid. The indicator used will be phenolphthalein. From the volume of saturated calcium hydroxide used, you will be able to determine its concentration and thus its Ksp value.

Time required:

Two hour or less depending on the number of samples determined.

Equipment and Reagents:

- Approximately 100 ml saturated calcium hydroxide, ($Ca(OH)_2$) solution*
- Approximately 100 ml 0.050 mol/L HCl solution
- 2-50 ml Burettes
- Filter paper
- Funnel
- 250 ml Erlenmeyer flask
- 2-250 ml beakers
- Phenolphthalein indicator solution
- Universal Stand
- Titration Clamp
- 25 ml graduated cylinder

Note that this is the reverse of a normal titration procedure. Usually, the solution of known concentration in the burette is added to the unknown solution in the Erlenmeyer flask. When using phenolphthalein as indicator, however, it is easier to see the color change from colorless to pink than pink to colorless and thus more accurate results may be obtained by using this particular method, where we are determining information about the substance we are titrating with, rather than the substance we are titrating

Significant Experimental Hazards

- Student should be aware of hazards associated with the use of all glassware (cuts).
- Phenolphthalein indicator is Hazardous in case of skin contact (irritant), of eye contact (irritant), of ingestion. It may also stain clothing and skin.
- Hydrochloric acid is harmful by digestion and may cause severe burns to eye and skin.

Procedure:

1. Measure the temperature of the stock calcium hydroxide (limewater).
2. Set up a funnel, filter paper, and a clean beaker and filter about 100 ml of saturated $Ca(OH)_2$ solution into a clean 250 ml beaker. *(Filter your solution as close as possible to the time it will be used, and do not let it stand around in the air for long periods of time to avoid any CO_2 reacting with the $Ca(OH)_2$ and forming chalk, $CaCO^3$)).*

3. Rinse the burette a couple of times with a few ml of the filtered $Ca(OH)_2$ solution. Then fill the burette and record the initial volume of $Ca(OH)_2$.

4. Rinse a second burette a couple of times with the HCl solution. Then fill the burette and record the initial volume of HCl in the burette.

5. Measure 15 to 20 mL of HCl into a clean Erlenmeyer flask. Record precisely the amount of acid used to the nearest 0.05 ml.

6. Add one or two drops of phenolphthalein solution.

7. Add 25 ml of distilled water to this solution.

8. Record the initial colors of both the $Ca(OH)_2$ and HCl solutions.

9. Gradually dispense some of the $Ca(OH)_2$ solution drop-by-drop from the burette into

If $Ca(OH)_2$ is not available, simply add enough Ca metal to distilled water and react to make a saturated solution. Smaller pieces of Ca will result in a faster reaction rate. The reaction is:

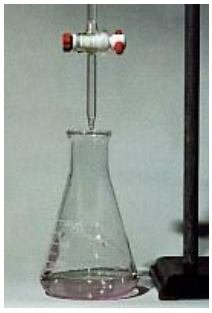
$$Ca(s) + 2H_2O(l) \rightarrow Ca(OH)_2(s) + H_2(g)$$

the solution in the Erlenmeyer flask. Swirl the flask constantly as the drops are added. Note any color changes observed, and do so constantly as $Ca(OH)_2$ is added to the HCl solution.

10. As the equivalence point is approached, a pinkish color will appear and dissipate more slowly as the titration proceeds until the endpoint of the titration is reached (this is the point at which a very light pink color is obtained after 20 seconds of swirling the flask).

11. Record the volume of $Ca(OH)_2$ required to reach the endpoint of the titration.

Phenolphthalein indicator should be titrated to a very light pink endpoint.



12. Repeat this rough titration at least twice more, or until two concordant results are obtained. (SD should be less than 10%)

13. Prepare a data table of your results, including the initial, final and total volumes of $Ca(OH)_2$ used for each titration.

14. Write a balanced chemical equation for the reaction of HCl with $Ca(OH)_2$. Also write the net ionic equation and indicate which two species are undergoing a chemical reaction. Which species are spectator ions?

Calculations:

1. Calculate the moles of H+ ions in the HCl solution:
2. Calculate the moles of OH- ions neutralized by the H+ ions in the titration:
3. calculate the concentration of the OH- ions:
4. Using the concentration of OH- calculated in #3 above, calculate the Ca^{2+} concentration and the $Ca(OH)_2$ concentration .
5. Substitute the concentrations of Ca^{2+} and OH^- calculated above into the Ksp expression, and calculate the value of Ksp.
6. Compare your Ksp result with the actual Ksp value obtained from literature (and be sure to note the temperature given for the particular value of Ksp stated).

Issues to consider for your practical report

- What are the potential sources of error in this experiment? How could they be overcome?
- Are there alternative methods for determining Ksp in water? If so, how do they compare to this method?
- Consult the literature for the solubilities of $Ca(OH)_2$ in water as temperature varies. Explain (in terms of energy) the reasons for this trend.

Experiment (13): The Solubility Product Experimental Results	
Name	**Date**
ID No.	**Group**

HCl Concentration:
$Ca(OH)_2$ Concentration:
Temperature:

Sample ID	Volume of HCl (ml)	Burette Reading $Ca(OH)_2$		Volume of $Ca(OH)_2$(ml)	Observation (color change)	Standard Deviation
		Initial	Final			

Observations & Calculations:

THE END