



كلية الهندسة - جامعة المنصورة

قسم الرياضيات و الفيزياء الهندسية

مجمع الكيمياء الهندسية

Engineering Chemistry Laboratory Book

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1) LABORATORY BASIC INFORMATION:

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مساحة المعمل	٢٠٠ متر مربع

2) SAFETY PRACTICES IN LABORATORY

2.1 The general safety precautions in the laboratory

- 1) Wear always safety glasses and white protective coats.
- 2) Drinking, eating or smoking in the laboratory is strictly forbidden.
- 3) Do not use mouth suction to fill pipettes.
- 4) Do not perform unauthorized experiments.
- 5) Never work in the laboratory alone.
- 6) Handle every chemical with care.
- 7) Do not taste chemicals.
- 8) Use care in smelling chemicals.
- 9) Avoid the inhalation of organic vapors.
- 10) Avoid contact of chemicals with skin and clothes.
- 11) Clean spills immediately on the bench, near the balance and everywhere
- 12) Replace caps on bottles as soon as possible.
- 13) Keep away the opening of your test tube or chemical bottles from your colleague's face
- 14) Limit the use of loose clothes while working.
- 15) Learn the location of safety shower.
- 16) Learn the location and contents of first aid kit.
- 17) Contact lenses should never be worn in the laboratory.
- 18) Check the area near you for flames if you are about to use organic solvents.
- 19) Check over your equipment joined together before work.
- 20) Do not leave the equipment joined together for long period of time.
- 21) Remember that glassware and the laboratory equipment are expensive.
- 22) Do not heat organic solvents on the open flame.
- 23) Write your experimental notes and results in your notebook directly when working.
- 24) Do not hesitate to ask your instructor when you feel doubtful.
- 25) In case of accident or if you feel unwell, seek medical advice immediately.

2.2 In case of accidents

In case of accident each student must notify the laboratory instructor immediately.

A) Chemicals after contact with the skin:

- 1) In case of acids, wash the skin with plenty of water, then saturated sodium bicarbonate solution and then again with great amount of water and finally get prompt medical attention.
- 2) In case of alkaline as hydroxides wash the skin with plenty of water, then 1% acetic acid solution and then again great amount of water and finally get prompt medical attention.
- 3) In case of organic material, wash with ethyl alcohol then with soap and then with hot water and seek medical advice.

B) Chemicals in the eye:

- 1) Flush the eye with large amounts of water for 15 min. using a special bottle and placing the person face up on the floor.
- 2) Hold the eye open to wash behind the eyelids.
- 3) Prompt medical attention must be taken regardless the severity of the injury.

C) Cuts:

- 1) Wash the cut.
- 2) Remove any pieces of glass.
- 3) Apply pressure to stop bleeding.
- 4) Get immediate medical attention.

D) Burns:

- 1) Flush the burned area with cold water for at least 15 min.
- 2) Wash of chemicals with 1% sodium bicarbonate solution then mild detergent and water.
- 3) Remove contaminated clothes.
- 4) Get prompt medical attention.

E) Burning clothes:

- 1) Prevent the person from running and fanning the flames.
- 2) Roll the person on the floor to help extinguishing the flame.

- 3) Hold the person under the shower until flames are extinguished and chemicals washed away.
- 4) Remove contaminated clothes.
- 5) Wrap the person in a blanket to avoid shock.
- 6) Get quick medical attention.
- 7) Don't use toxic fire extinguisher.

F) Burning reagent:

- 1) Extinguish all nearby burners.
- 2) Remove combustible materials and solvents.
- 3) Cover the fires in beakers with asbestos-wire gauze square or a watch glass.
- 4) Use dry chemical fire extinguisher directed at the base of the flame.
- 5) Do not use water.

2.3 Contents of first aid kit

- 1) 1% aqueous solution of acetic acid.
- 2) 1% aqueous solution of boric acid.
- 3) Saturated solution of sodium bicarbonate.
- 4) 1% aqueous solution of sodium bicarbonate.
- 5) Glycerin.
- 6) Pure ethyl alcohol.
- 7) Cotton.
- 8) Disinfectant (dettol).
- 9) Mercurochrome.
- 10) Creams for burns.
- 11) Bandages.
- 12) Adhesive plaster.
- 13) Picrate ointment.
- 14) Glass dropper.

3) Laboratory Instruments:

م	اسم الجهاز	تاريخ التصنيع	عدد	حالة الجهاز
١	pH meter model G 104) Austria (النمسا) 220 V	١٩٨٣	١	يعمل
٢	pH meter Ion model 5986-50 220 V 50-60 Hz 0-40 A	١٩٩١	١	يعمل
٣	T D S tester USA HACH [10-1990]	١٩٩٥	١	يعمل
٤	T D S digital model HI 8734 10 mg / 19.990 / L	١٩٩٦	١	يعمل
٥	Sodium tester	١٩٩٣	١	يعمل
٦	Salinity tester	١٩٩٣	١	يعمل
٧	Conductivity meter model pid 18 + cell S.N. 6837	١٩٨٨	١	يعمل
٨	Conductivity meter model ysi 35 + cell S.N. D9002758	١٩٨٩	١	يعمل
٩	Turbidity meter model D R T 15 C E	١٩٩٧	١	يعمل
١٠	Microtonal ultrasonic model 50 watt. 23 K h z 22KHz	١٩٩٤	١	يعمل
١١	Electrophoresis + D C power Model No. SE 600	١٩٩٤	١	يعمل
١٢	Table Top	١٩٩٥	١	يعمل
١٣	Sound level meter cot no. seg 761 – 9Model 407740	١٩٩٩	١	يعمل
١٤	C O D reactor Model 2951620 700Watt 50-60Hz	١٩٩٥	١	يعمل
١٥	C O D reactor model E C O 6)S.N. 1022546 700Watt 220V +4.4% 50-60Hz COD 10.0120	٢٠٠٢	١	يعمل
١٦	Rotary vaporatorS.N.21002 Model BM-42 115V AMPS:5	١٩٩٣	١	يعمل
١٧	خزان معالجة من الصاج المجلفن	٢٠٠٣	١	يعمل
١٨	U V spectrophotometer model S 2100 U VModel 52180 S.N. BP0505034	٢٠٠٦	١	يعمل
١٩	Turbidity meter model U I G 3703 C HANNA	٢٠٠٦	١	يعمل
٢٠	ظلمية رفع مياه صغيرة بالتنقيط	١٩٨٩	١	يعمل

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يعمل	١	١٩٨٦	مضخة هواء أسباني ٢٢٠ فولت موديل ٧٧٧	٢٢
يعمل	١	٢٠٠٢	Pressure veccum pump pv – 142 N	٢٣
يعمل	١	١٩٩٢	كمبروسور هواء ٨ بار ٢٥ لتر قدرة ١,٥ حصان إيطالي	٢٤
يعمل	١	١٩٩٣	جهاز ترشيح	٢٥
يعمل	١	١٩٨٤	فرن تجفيف ألماني ٢٢٠ فولت	٢٦
يعمل	١	١٩٩٦	(A - sso) (Temp. range 1100° C)فرن إحتراق موديل)	٢٧
يعمل	١	١٩٩٤	جهاز تقطير زجاجي ٥ لتر	٢٨
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4) Laboratory Experimental List:

م	التجربة	الغرض منها
١	Titration of acids and bases	
٢	Oxidation reduction titrations	
٣	Chlorides	
٤	Dissolved oxygen	

4:Laboratory Community Beneficiaries:

رابعا : الخدمات المجتمعية داخل المعمل

- المستفيدين من المعمل طلاب الفرقة الاعدادية وطلاب المستوى الصفري بالبرامج الجديدة إضافة إلى طلاب الدراسات العليا وطلاب مشاريع التخرج بالكلية.
- الجهات التي تتعاون مع المعمل : لا يوجد
- الدخل السنوي للمعمل : لا يوجد.
- الجهات الممولة لأنشطة المعمل : لا يوجد.
- المشاريع التنافسية التي يشارك بها المعمل : لا يوجد

5:Laboratory Student Beneficiaries:

خامسا : الخدمات الطلابية التي يؤديها المعمل

جميع اقسام كلية الهندسة	الأقسام العلمية المستفيدة من المعمل
المستفيدين من المعمل طلاب الفرقة الاعدادية وطلاب المستوى الصفري بالبرامج الجديدة إضافة إلى طلاب الدراسات العليا وطلاب مشاريع التخرج بالكلية.	الفرق الدراسية المستفيدة من المعمل
كيمياء هندسية	المقررات الدراسية التي تستفيد من المعمل
لا يوجد	الأنشطة الطلابية داخل المعمل
٧ طلاب ضمن ماجستير و دكتوراه هندسة البيئة	عدد طلاب الدراسات العليا المستفيدين من المعمل
١٥ رسالة ماجستير و ١٠ رسائل دكتوراه	عدد الرسائل العلمية التي تمت في المعمل
لا يوجد	عدد الدورات العلمية التي تمت داخل المعمل
لا يوجد	المسابقات العلمية التي شارك بها الطلاب المستفيدين من المعمل

6:Laboratory Experiments:

EXPERIMENT (1)

TITRATION OF ACIDS AND BASES

Objectives

The aim of this experiment is to demonstrate the use of titration as an analytical tool. As an example of its uses we shall employ this technique to determine the concentration of unknown acid solution.

Titration is a process of mixing a standard solution (known concentration) with reacting solution of unknown concentration.

Equipment

- Two 50-ml burettes,
- Erlenmeyer flask,
- Burette Clamp,
- Wash bottle,
- One 400-ml and one 1000 ml bottle,
- One 500 ml graduated cylinder,
- ml Volumetric Flask,
- Analytical balance.

Chemicals

- Stopcock grease,
- 6N NaOH,
- Phenolphthalein solution,
- Standard 0.1N HCl,
- Oxalic acid $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$.

Procedure

Make a solution of NaOH and standardize it. This Can be done by titrating with a standard acid solution or titrating with a weighted amount of a primary standard.

A. Standardization of NaOH Solution with Standard acid Solution:

1. Place 5 ml of the 6N NaOH from the shelf in a clean dry bottle. Add enough deionized water to make a solution which is approximately 0.06N.
2. Mix thoroughly and stop with a rubber stopper. Label your solution with the name of the substance, approximate concentration and date. The concentration of NaOH solution is now known only approximately.
3. The chemical storeroom will supply two clean burettes. If their stopcock are not properly greased they will not turn freely and probably will leak or be clogged. Clean and re-greased the stopcocks if necessary. Rinse one of the burettes with about 5 ml of your 0.06N NaOH solution. Next, fill the burette to the 0.00ml mark with this solution. Make sure the tip of the burette is filled. The level of the liquid in the burettes is read to the bottom of the meniscus.
4. Rinse the second burette twice with about 5ml of the standard 0.1N HCl solution. You will find this solution in the reagent shelf. Be sure the tip of the burette is filled. Run 10.00ml of the standard 0.1N HCl solution into a clean 250ml Erlenmeyer flask.
5. Add 2 or 3 drops of phenolphthalein indicator solution. Titrate this acid solution with your NaOH solution until a faint pink color persists for over 15 sec. Swirl the solution in the flask as you titrate. If you overshoot the end point, you may back-titrate by adding know amount of acid until the solution is color and then repeating the base addition until the solution is pink. After the final drops are added, touch the tip of each burette to the side of the flask and wash these small drops into the solution with distilled water.
6. Repeat the above titration using 20.00ml of 0.1N HCl solution. The normality of the base as calculated from the data of the two titration should agree to within 0.001N. If it dose not, repeat titration with 20.00ml of 0.1N HCl.

B. Standardization of NaOH Solution with primary standard:

1. Oxalic acid $\text{H}_2\text{C}_2\text{O}_4 \cdot 2\text{H}_2\text{O}$ is considered to be a primary standard reagent, which means that it is fairly stable under ordinary conditions and can easily be brought to a high state of purity.

2. Weight the calculated amount of the acid (to 0.001g) in clean beaker. Quantitatively transfer the acid into a clean 250ml volumetric flask. To ensure a quantitative transfer, dissolve in distilled water any acid remaining in the beaker or on the stirring rod. Add this to the volumetric flask.
3. Fill the flask about one-half full with distilled water and swirl it until dissolution is complete. Add water carefully until the meniscus comes to graduation mark, adding the last bit with a medicine dropper. Stopper the flask and mix thoroughly for several minutes.
4. Transfer the standard solution to a clean, dry bottle and stopper it. Label the solution with the name of the substance, exact concentration and date.
5. The storeroom will supply two clean burettes. Assure yourself that the burettes are working properly (see part A). Rinse each burette with 5ml of the base solution. Fill each burette with solution and drain enough liquid so that the entire tip of the burette is free of the air bubbles. Adjust the upper level of the solutions so that the meniscus rests on the zero mark. Touch each tip to waste beaker to remove excess liquid.
6. Run about 20 ml (measured to 0.01ml) of the acid solution into a clean 250 ml Erlenmeyer flask. Add 2 drops of phenolphthalein indicator. Now add enough base (at first quickly and later drop by drop) until the final drop leaves the solution slightly pink for at least 15 sec.
7. Calculate the normality of NaOH. Record the average normality obtained on the label of the bottle.

C. Determination of the normality of an unknown using titrimetric technique:

1. Obtain an Unknown acid sample from the storeroom.
2. Rinse out the burette which has filled with acid solution with three 5-ml portions of distilled water and then with three 5-portions of your unknown acid solution. Now fill the burette to the 0.00 mark with your unknown acid.
3. Run about 20.00 ml of this solution into a clean Erlenmeyer flask. Add 2-3 drops of phenolphthalein indicator solution.

4. Titrate with the standard NaOH solution until faint pink color persists for over 15 sec. while the solution is being swirled.
5. Repeat with another sample of your unknown, until the normality of your unknown, as calculated from above data, agrees to within 0.05N.

Results and calculations

If the unknown base has been titrated against a standard acid solution record the volume of acid necessary for neutralization of the unknown base and calculate the normality from the following relation:

$$V_a * N_a = V_b * N_b$$

Where:

V_a = Volume of acid (ml).

N_a = normality of acid

= number of gram equivalent of acid /lit.

= weight of acid/equivalent weight of acid/liter.

V_b = volume of unknown base (ml)

N_b = normality of unknown base.

E_a = equivalent weight of acid = molecular weight of acid /valency

Table 1.1

Trial	Start Point	End Point	Volume consumed	Mean Volume
1				
2				
3				

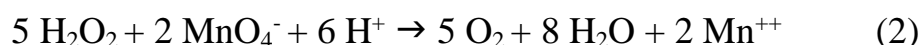
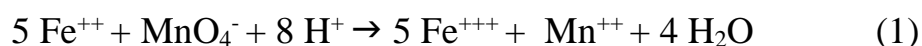
EXPERIMENT (2)

OXIDATION REDUCTION TITRATIONS

Introduction

Volumetric methods can be extended easily to include the analyses of substances which undergo rapid, quantitative oxidation or reduction reactions. We shall study two of these reactions, the oxidation of a ferrous compound and the oxidation of H_2O_2 . Permanganate ion will be the oxidizing agent.

The ionic equations for these reactions are:



From equation (1), you should be able to deduce that the equivalent weight of KMnO_4 is equal to one-fifth its formula weight, or $158.03/5=31.6\text{g}$.

The ferrous salt used in this experiment is a hydrate of ferrous ammonium sulfate (FAS), $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$. Its formula weight is 392.16g .

Equipment

- Two burettes and
- Erlenmeyer flask.

Chemicals

- Reagent grade, $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$,
- Solid KMnO_4 or 0.1N KMnO_4 ,
- $18\text{N H}_2\text{SO}_4$ and
- dil (1:1) H_3PO_4 .

Procedure:

You are to prepare an aqueous KMnO_4 solution and find its normality by titrating it versus weighted samples of $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$. This standard solution will be used to analyze H_2O_2 solution whose concentration is known.

If the KMnO_4 solution has been prepared, you must standardize it with $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$. since KMnO_4 does not meet the requirement of a primary standard, weighed samples of KMnO_4 can not be used to prepare standard solution. However, the approximate concentration of KMnO_4 can be estimated from weighing.

If the KMnO_4 solution has not been prepared, calculate the weight of KMnO_4 necessary to make 500 ml of 0.1N KMnO_4 (Grams $\text{KMnO}_4 = \text{-----}$)

Weight out this KMnO_4 to 0.01g and dissolve in 500 ml of deionized water (measure with a graduate cylinder). Allow this solution to stand for a few days before attempting to standardize it.

To standardize the KMnO_4 , weigh a clean dry Erlenmeyer flask to 0.01g. Then add about 1.5g of $\text{FeSO}_4 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 6 \text{H}_2\text{O}$ to the Erlenmeyer flask and reweigh to 0.01g. record the result in table (2.1)

Fill the burette with KMnO_4 solution and record the initial reading. Add 100 ml of deionized water, 10 ml of 18N H_2SO_4 , flask. Mix the contents well. Titrate immediately.

The end point of the reaction is determined by color change from colorless to faint pink. The permanganate ion imparts a deep purple color to the solution. As it reacts with ferrous ion (reaction (1)), it is converted to Mn^{++} , which is almost colorless. When all the ferrous ion has been oxidized to the ferric state, the permanganate will persist.

The permanganate color will take more time to disappear as the end point of the reaction is approached. Titrate slowly as you approach the end point and gently swirl the flask after each small addition of KMnO_4 solution. If too much KMnO_4 is added, the sample must be discarded. Record the final burette reading.

Repeat the standardization and record the results in table (2.1).

Calculate the normality of KMnO_4 solution from the two sets of data. The results should agree to within 1%. If they do not, do a third titration. Consult with your instructor before proceeding further in this experiment.

Obtain an unknown aqueous solution of H_2O_2 . With a clean burette, free of KMnO_4 and detergent, measure exactly 25.00 ml of this solution into an Erlenmeyer flask. Then add 75 ml of deionized water and 10 ml of 18N H_2SO_4 .

Titrate with the standard KMnO_4 solution until a faint Pink color persists. Repeat this analysis until successive titrations agree to within 0.10 ml. record the results.

Results and Calculations:

$$V_a * N_a = V_b * N_b$$

Where: V_a = volume KMnO_4 (ml).

N_a = normality of KMnO_4 .

V_b = volume of unknown FAS. (ml).

N_b = normality of unknown FAS.

Table 2.1

Trial	Start Point	End Point	Volume consumed	Mean Volume
1				
2				
3				

EXPERIMENT (3)

CHLORIDES

Objective

The aim of this experiment is the determination of chlorides present in waters and wastewaters in order to design a suitable method and equipment for its elimination and to prevent corrosion of pipes and handling systems.

General Considerations

Chlorides, in the form of Cl^- ion, is one of the major inorganic anions in water and wastewater. In Potable water, the salty taste Produced by chloride concentrations is variable and dependent on the chemical composition of the water. Some water containing 250 mg/lit. chloride may have a detectable salty taste if the cation is sodium. On the other hand, the typical salty taste may be absent in waters containing as much as 1000 mg/lit. when the predominant cations are calcium and magnesium.

The chloride concentration is higher in wastewater than in raw water because sodium chloride is a common article of diet and passes unchanged through the digestive system.

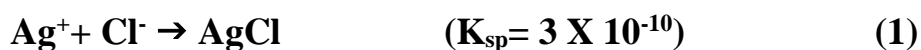
Along the sea coast, chloride may be present in high concentrations because of leakage of salt water into sewerage system. It may also be increased by industrial processes.

A high chloride content harms metallic pipes and structures, as well as agricultural plants.

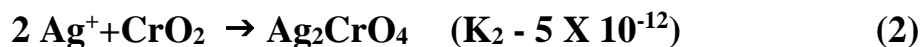
Method of Determination

Chlorides may be readily measured by means of volumetric procedures employing internal indicators. For most Purposes the **Mohr method employing silver nitrate (AgNO_3)** as the titrant and potassium chromate as the indicator is satisfactory. The principles of this method will be presented as follows:

This method recommends the use of a solution of AgNO_3 0.0141 N. The silver nitrate solution can be standardized against sodium chloride. In the titration the chloride ion is precipitated as white silver chloride.



The end point cannot be detected by eye unless an indicator capable of demonstrating the presence of excess Ag^+ is present. The indicator normally used is potassium chromate which supplies chromate ions. As the concentration of chloride ions approaches extinction, the silver ion concentration increases to a level at which the solubility product of silver chromate is exceeded and it begins to form a reddish-brown precipitate.



This is taken as an evidence that all the chloride has been precipitated. Since an excess of Ag^+ is needed to produce a visible amount of Ag_2CrO_4 , the indicator error or blank must be determined and subtracted from all titrations.

Chemical reagents

You will be provided with the following reagents:

Reagent No. 1 : Potassium chromate indicator solution: Which composed of a solution of 50g K_2CrO_4 in one liter of distilled water.

Reagent No. 2 : Standard silver nitrate titrant (0.0141 N): Which composed of 2.395g AgNO_3 in one liter of distilled water.

Reagent No. 3 : Standard sodium chloride (0.0141 N): Which composed of 824.1mg NaCl (dried at 104C) in one liter of distilled water.

Procedure

1. Use 100ml sample or suitable portion diluted to 100ml. Add 1ml H_2CrO_4 indicator solution (reagent No. 1). Titrate with standard nitrate titrant to a pinkish yellow end point.
2. Standardize the silver nitrate titrant (reagent No. 2) with NaCl solution (reagent No. 3) to find the exact normality of AgNO_3 solution using 10ml of NaCl solution completed with distilled water to 100ml. use 1ml of the indicator.
3. Establish the reagent blank value by the titration method outlined in step 1. Above. A blank of 0.2 to 0.3ml is usual for the method.

Results and calculations

$$\text{Cl}^- (\text{mg/lit}) = \frac{(A - B) \cdot N \cdot 35450}{\text{volume} \cdot \text{of} \cdot \text{Cl}^-}$$

Where:

A= ml titration for sample.

B= ml titration for blank.

C= normality of AgNO_3 .

Table 3.1

Mean Volume	Volume consumed	End Point	Start Point	Trial
				1
				2
				3

EXPERIMENT (4)

DISSOLVED OXYGEN

Objective

The aim of this experiment is the determination of dissolved oxygen in waters and to be able to study the waste management techniques necessary for water treatment.

General Considerations

All living organisms are dependent upon oxygen in one form or another to maintain the metabolic processes that produce energy for growth and reproduction. Aerobic processes are the subject of greatest interest because of their need for free oxygen.

All the gases of the atmosphere are soluble in water to some degree. Both nitrogen and oxygen are classified as poorly soluble gases, their solubility is directly proportional to their partial pressures. Hence Henry's law may be used to calculate the amounts of gas present at saturation at any given temperature.

Environmental Significance Of Dissolved Oxygen

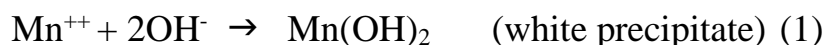
1. In liquid wastes, dissolved oxygen is the factor that determine whether the biological changed are brought about by aerobic or by anaerobic processes. The former use free oxygen for oxidation of organic and inorganic matter and produce harmless end produce, whereas the latter bring about such transformation through the reduction of certain inorganic salts such as sulfates and the end products are often very obnoxious. Dissolved oxygen measurement are vital for maintaining aerobic conditions in natural waters that receive pollution matter and in aerobic treatment processes intended to purify domestic and industrial wastewaters.
2. Dissolved oxygen determinations are used for the control of stream pollution. The presence of dissolved oxygen support the desired aquatic life included in the self purification cycle.
3. Determination of dissolved oxygen serves as the basis of the BOD test, thus they are the foundation of the most important determination used to evaluate the pollution strength of domestic and industrial wastes.

4. Oxygen is a significant factor in the corrosion of iron and steel, particularly in water distribution system and in steam boilers. Removal of oxygen from boiler-feed waters by physical and chemical means is common practice in the power industry. The dissolved oxygen test serves as the means of control.

Methods of Determining Dissolved Oxygen

1. Originally, measurements of dissolved oxygen was made **by heating samples to derive out the dissolved gases** and analyzing the collected gases for oxygen by analytical methods require large samples and are very cumbersome and time consuming.
2. The use of **dissolved oxygen electrodes** allow in situ measurements to be made has increased significantly since their development.
3. **The Winkler or iodometric** method and its modifications are the standard procedures for determining dissolved oxygen at the present time and it will be presented here. The test depends upon the fact that oxygen oxidizes Mn^{++} to a higher state of valency under alkaline conditions and that manganese in higher state of valency is capable of oxidizing I^- to free I_2 under acid conditions. Thus the amount of free iodine released is equivalent to the dissolved oxygen originally present. The iodine is measured with standard sodium thiosulphate solution and interpreted in terms of dissolved oxygen.

The reactions involved in the winkler procedure are as follows:



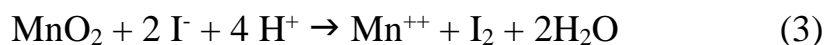
If no oxygen is present, a pure white precipitate of $Mn(OH)_2$ forms when $MnSO_4$ and the alkali-iodide reagent ($NaOH + NaI$) are added to the sample. If oxygen is present in the sample, then some of Mn^{++} is oxidized to Mn^{+4} and precipitates as brown hydrate oxide

The reaction is usually represented as follows:



The oxidation of Mn^{++} to MnO_2 sometimes called fixation of oxygen, occurs slowly, particularly at low temperatures. Furthermore, it is necessary to move the flocculates material through the solution to enable all the oxygen to react. Vigorous shaking of samples for at least 20 seconds is needed.

After shaking the samples for a time sufficient to allow all oxygen to react, the floc is allowed to settle so as leave at least 5cm of clear liquid below the stopper: then sulfuric acid is added. Under the low pH conditions that result, the MnO_2 oxidizes I^- to produce free I_2 .



The sample should be stoppered and taken for at least 10 seconds to allow the reaction to go to completion and distribute the iodine uniformly through the sample.

The sample is now ready for titration with (N/10) sodium thiosulphate. The use of (N/40) thiosulphate is based on the premise that a 200-ml sample will be used for titration. The execution of this method will be explained as follows:

Chemical Reagents

You will be provided with the following reagents as numbered:

Reagent No. 1: Manganese sulfate solution, which composed of (480g $\text{MnSO}_4 \cdot 4\text{H}_2\text{O}$, 400g $\text{MnSO}_4 \cdot 2\text{H}_2\text{O}$ or 364g of $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ in 1 liter of distilled water).

Reagent No. 2: Alkali-iodide-azide reagent which composed of (500g NaOH or 700g KOH + 135g NaI or 150g KI in 1 liter of distilled water). Add to this solution 10g of sodium azide NaN_3 dissolved in 40ml of distilled water.

Reagent No. 3 : Sulfuric acid, concentrated H_2SO_4 .

Reagent No. 4 : Starch indicator which is composed of 5g soluble starch in 1 liter of distilled water, which is preserved by adding 1.25g salicylic acid per liter by adding few drops of toluene.

Reagent No. 5 : Standard sodium thiosulfate titrant 0.025N.

Procedure

1. To the given sample (250-300ml bottle) add 1-2ml of reagent No. 1, followed by 1-2ml of reagent No. 2, will be lower the surface of liquid. Stopper carefully to exclude air bubbles and mix by inverting the bottle at least 15 times. When the precipitate settles, leaving a clear supernate above the manganese hydroxide floc, shake again.
2. After at least 2min. settling has produced at least 100ml of clear supernate, carefully remove the stopper and immediately add 2ml of concentrated H_2SO_4 (reagent No.3) by

allowing the acid to run down the neck of the bottle, restopper, and mix by gentle inversion until dissolution is complete.

3. Use a volume corresponding to 200ml of the original sample after correction for the loss of sample by displacement with the reagents. Thus for a total of 4ml (2ml each) of reagent No. 1 and reagent No. 2 in a 300ml bottle, titrate: $200 \times 300 / (300 - 4) = 203 \text{ml}$.
4. Titrate with 0.025N thiosulfate solution (reagent No.5) to pale straw color using (1-2ml) starch indicator (reagent No.4).

Results and Calculations:

1. For 200ml of original sample, 1ml of 0.025N sodium thiosulfate = 1mg/lit. dissolved oxygen (DO).
2. To obtain the results in ml of oxygen gas per liter, corrected to 0 °C and 760mm Hg pressure, multiply mg/lit. DO by 0.7.

1 ml of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ = 1 mg/lit. of DO

Volume of $\text{Na}_2\text{S}_2\text{O}_3 \cdot 5\text{H}_2\text{O}$ = ml

DO = mg/lit