

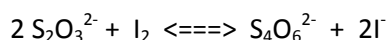
FE 211 ANALYTICAL CHEMISTRY

Experiment No: 4

- A) OXIDATION BY IODINE (IODOMETRY)
- B) DETERMINATION OF OXYGEN IN WATER

OXIDATION BY IODINE (IODOMETRY)

Iodide ion, I^- , is a moderately effective reducing agent that has been used for the analysis of oxidants. The lack of a good method of end point detection makes direct titration of oxidizing agents by solutions of iodide salts impractical. Thus, the indirect procedure is always employed. This involves reduction with a moderate unmeasured excess of potassium iodide, KI. The iodine, I_2 , liberated, equivalent in quantity to the oxidant being determined, is then titrated with a standard solution of a reducing agent. Sodium thiosulfate, $Na_2S_2O_3$, is mostly used for this purpose. The reaction between iodine, I_2 , and ion, $S_2O_3^{2-}$, is as follows.



The end point in the titration is readily established by means of starch solution. It should be emphasized that starch is partially decomposed in the presence of large excess iodine. For this reason the indicator is never added to an iodine solution until the bulk of that substance has been reduced. The change in color of the iodine from a red-brown to a faint yellow signals the proper time for the addition of the indicator.

A) Reagents:

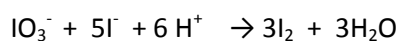
- 0.1 M Sodium Thiosulfate, $Na_2S_2O_3$
- Copper, Cu, solution
- 3 M H_2SO_4
- 1% Starch solution
- Potassium Iodide, (iodate free), KI
- Potassium Thiocyanate, KSCN
- 10% HCl solution
- Potassium Iodate, KIO_3 , solution, (15 g KIO_3 in one liter solution)

B) Procedure:

Standardization of 0.1 M Sodium Thiosulfate

- Transfer 0.6 to 0.7 g of primary standard KIO_3 to a weighing bottle, and dry for 1-2 hours at $160^\circ C$.
- Weigh and transfer the KIO_3 to a 250 mL volumetric flask.
- Dissolve in 100 mL of H_2O , and dilute to the mark.
- With a volumetric pipette, transfer 50 mL aliquots of the KIO_3 solution (**or take 10 mL of prepared KIO_3 sample**) to 250 mL Erlenmeyer flask and dilute to 100 mL with distilled water.
- Add 2 g of reagent grade KI (iodate free) to each sample, and swirl gently until dissolved.
- Carry on each replicate trial one at a time from this point forward.
- Add 10 mL of 10% M HCl.
- Add 5 mL of starch indicator solution.

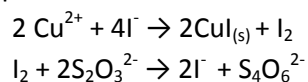
- Titrate with the Na₂S₂O₃ solution.
- The end point signal is the disappearance of the blue starch-iodine complex color.
- From the weight of KIO₃ in each aliquot, calculate the exact molarity of the Na₂S₂O₃ solution.



Determination of Copper by Iodometry

- Take 10.0 mL copper solution and dilute to 50 mL with distilled water.
- Add 10 mL 3.0 M H₂SO₄ and then 4 g of KI.
- Add 2 g of KSCN (Note 1) and 5 mL of starch solution.
- Titrate immediately with 0.1 M Na₂S₂O₃.
- Swirl vigorously for several seconds.
- Continue the titration with vigorous mixing until the blue starch/iodine color is decolorized and does not return for several minutes.

The reactions involved can be expressed as:

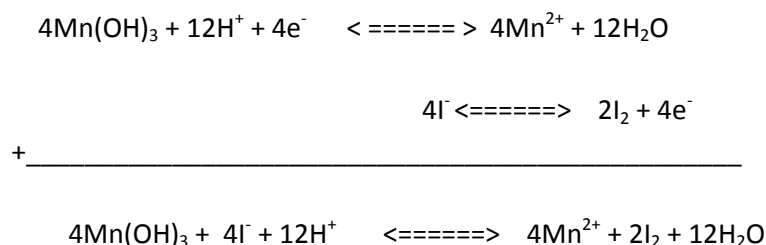


DETERMINATION OF OXYGEN IN WATER (IODOMETRY)

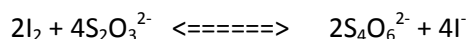
The determination of oxygen in water may be conveniently done by polarographic methods using Dropping Mercury Electrode or simply by using Dissolved Oxygen Meter. When none of those instruments are available, the Winkler method, developed in 1888, may be used. This procedure requires only the usual material present in all analytical chemistry laboratories, but it is not as fast or sensitive as the others. The method is based on the oxidation of Mn²⁺ by oxygen which in turn is reduced by potassium iodide, KI, in an acidic medium. The iodine I₂, formed is then determined by titration with standard sodium thiosulfate, Na₂S₂O₃. In the initial basic medium, Mn(OH)₂ is formed and reacted with dissolved oxygen:



When the medium is acidified with H₂SO₄ and iodide ion (I⁻) added :

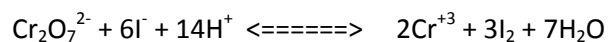


Iodine, I₂, formed is then titrated with standard thiosulfate solution.



The end point in the titration is established by means of a starch solution which gives a blue color in the presence of iodine.

In the preparation of the standard thiosulfate solution potassium dichromate, $K_2Cr_2O_7$, may be used. Dichromate acts with iodine in acidic solution to give iodine;



Iodine, I_2 , liberated in this reaction is titrated with the thiosulfate solution for standardization.

A) Reagents :

- $MnSO_4$ solution:

Dissolve 50 g of $MnSO_4 \cdot 4H_2O$ in water and bring the volume to 100 mL with distilled water.

- Alkaline iodide solution:

Dissolve 50 g of NaOH in water and bring the volume to 100 mL; cool and transfer the solution to a polyethylene bottle, let it stand for a few days to allow Na_2CO_3 to settle down. Dissolve 15 g KI in a small quantity of water. Mix the supernatant of alkaline solution with iodide and dilute to 100 mL.

- 0.01 M $Na_2S_2O_3$ solution:

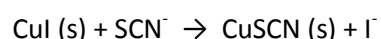
Dissolve about 2.48 g of $Na_2S_2O_3 \cdot 5H_2O$ in water and bring the volume to 1000 mL (use boiled distilled water).

B) Procedure:

Determination of Dissolved Oxygen in Water

- Take a 100 mL tap water sample into a 250 mL Erlenmeyer flask. Take care not to include any air bubbles.
- Add 1 mL of the $MnSO_4$ solution and 1 mL of alkaline KI solution by immersing the pipette below the surface of water (again take care not to include air bubbles). In the absence of oxygen, $Mn(OH)_2$ is produced as a white precipitate. In the presence of dissolved oxygen this is oxidized to the brown $Mn(OH)_3$.
- Allow the precipitate to settle and then mix, repeating this process twice to ensure complete adsorption of oxygen.
- Add 1.5 mL concentrated H_2SO_4 , and mix thoroughly.
- Titrate with sodium thiosulfate, $Na_2S_2O_3$, solution adding starch indicator just before the endpoint.
- Calculate the amount of dissolved oxygen in water.

Note1 : It has been found experimentally that the titration of iodine by thiosulfate in the presence of CuI tends to yield slightly low results because appreciable quantities of iodine are adsorbed on the solid. This difficulty is largely overcome by the addition of thiocyanate (SCN^-) ion, which also forms a sparingly soluble copper (I) salt. Part of the copper iodide is converted to the corresponding thiocyanate at the surface of the solid.



Accompanying this reaction is the release of the adsorbed iodine, thus making it available for titration. Early addition of SCN^- must be avoided, however, because of the tendency for that ion to reduce iodine slowly.

FE 211 ANALYTICAL CHEMISTRY DATA SHEET

Experiment No: 4

A) OXIDATION BY IODINE (IODOMETRY)

1) Standardization of 0.1 M Sodium Thiosulfate

Volume of 0.1 M $\text{Na}_2\text{S}_2\text{O}_3$ used (mL) :

2) Determination of Copper by Iodometry

Volume of 0.1 M $\text{Na}_2\text{S}_2\text{O}_3$ used (mL) :

B) DETERMINATION OF OXYGEN IN WATER

Volume of 0.01 M $\text{Na}_2\text{S}_2\text{O}_3$ used (mL) :

Submitted by:

Submitted to:

Date: