

Redox titration
winkler method for
dissolved oxygen
determination essay
sample



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Redox Titration: Winkler Method for Dissolved Oxygen Determination The three main objectives of the experiment are to (1) determine dissolved oxygen (DO) in water using the classical method (redox titration), (2) discuss the chemistry behind the Winkler method for DO analysis and (3) describe the sampling technique for the analysis of DO in water sample. In the Winkler method, or Iodometry, the dissolved oxygen is made to react with iodide ion to yield Iodine which will be titrated afterwards with thiosulfate. Iodine is a moderately strong oxidizing agent making it useful for titrating strong reducing agents like dissolved oxygen. Iodide ion, on the other hand, is a weak reducing agent, and is the basis of determining the amount of the strong oxidizing agents like the oxygen in the sample pond water. This is done by determining the amount of I_3^- produced, which is equivalent to the dissolved oxygen using titration with thiosulfate.

Thiosulfate may be oxidized by strong oxidation agents or form complexes with some, making it inadvisable to titrate the water sample directly with thiosulfate. By adding iodide ions first before titration, the strong oxidizing agent is "destroyed" and an equivalent amount of Iodine is produced. This iodine will be converted into I_3^- which will react stoichiometrically with the thiosulfate. The thiosulfate to be used for titration was first standardized using KIO_3 as the primary standard. KI crystals and sulfuric acid was added to the KIO_3 solution one after the other. KI crystals should be added first or the solution may produce more O_2 and add to the analyzed dissolved oxygen of the sample:

The resulting solution was titrated right away with $\text{Na}_2\text{S}_2\text{O}_3$ as the titrant and starch as the indicator. The first step of the process resulted to the following reaction:

Like other halogens, iodine reacts with water, yielding the reaction:

To prevent the forward reaction, and produce more of the desired I_2 , the solution was made acidic by adding H_2SO_4 which will cause the equilibrium to shift to the left due to the common-ion effect. This is also the same reason why excess KI was added. Unreacted iodide does not interfere with the reaction but may be oxidized by air if not titrated immediately.

But the iodine has a low solubility in water, so it is first converted to a complex consisting of I_3^- which is very soluble. This is done by dissolving I_2 in a potassium iodide solution:

The I_2 produced in this reaction is then titrated with $\text{Na}_2\text{S}_2\text{O}_3$ which yields the reaction:

From this titration, the molarity of thiosulfate can be calculated using the knowledge that:

The endpoint of the reaction is indicated using starch, which does not dissolve in solutions but exists as colloid and is only suspended in the analyte. The starch indicator should be added just prior to the endpoint or when the yellow iodine color is beginning to fade. Adding starch at the start of titration when the iodine concentration is still high will form an iodine-starch complex, producing a diffuse endpoint and a reddish brown color

which will not be readily discharged at the endpoint because of the excess
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iodine still present due to the slow dissociation of the complex. Also, starch may hydrolyze and interfere with the reaction in acidic solutions, which is the case of the analyte in this experiment. However, if iodine was the titrant, starch can be added at any point since iodine is not in excess until the endpoint. It exhibits a deep blue color when it absorbs iodine. When the iodine is converted into iodide ion, the blue color disappears and the complete disappearance of the color indicates the endpoint.

The presence of strong solutions of electrolytes, organic materials like alcohol and heat may interfere with the absorption of iodine and will not produce a good color, thus these conditions should be avoided. Starch should also not be kept suspended for too long since long exposure to water can make it susceptible to bacterial action, causing it to decompose and become less effective. For the sample analysis, the same concepts were used to determine the amount of dissolved oxygen. The dissolved oxygen was first added with NH_4HCO_3 to form a more reactive oxygen-sensitive carbonate. If NH_4HCO_3 was not added, the dissolved oxygen will not react or will react very slowly with the solution, even though MnSO_4 was already added. Then, a basic KI solution (NaOH with KI crystals) is added to react with the Mn^{2+} ions and to form an OH^- salt with Mn:

The $\text{Mn}(\text{OH})_2$ produced will then react with the oxygen. There are two possible reactions that may occur: which will produce a brownish inorganic oxide floc, the fluffy mass seen suspended in the pond water sample, or which will also produce a brown solution.

The $\text{Mn}(\text{OH})_3$ precipitates formed was then dissolved with phosphoric acid while the $\text{MnO}(\text{OH})$ precipitated will react with the iodide ions from the NaOH solution with KI:

The solution was then closed tightly and kept in a dark locker. This is done to prevent the accelerating effect of light to the decomposition of hypiodous acid:

and the oxidation of iodide ion caused by air:

The I_2 produced in the desired reaction will be equivalent to the amount of dissolved oxygen in the pond water sample:

To be able to determine the exact amount of I_2 , and also the dissolved oxygen, the solution was titrated with thiosulfate, again, with the reaction:

In the experiment, a fresh pond water sample from the Palma Hall was analyzed using the winkler method described above. The sample was analyzed immediately as bacterial action may affect the dissolved oxygen content of the water. As the reagents were added, the tip of the transfer pipettes was kept just below the surface of the water to prevent the penetration of more oxygen from the air into the sample. It was found out that the water contained an average of 8.1 ppm O_2 . This means that the pond water is relatively clean; therefore it can sustain stream fishes, including cold water fishes.

Sources:

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