Protocol
K4 - Oxalic Acid Oxidation

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Contents

1 Objective

In this assignment, the kinetics of chemical reactions are studied through two separate experiments.

In the first experiment, the redox reaction between oxalic acid and potassium permanganate is studied: After determining the absorption maximum of potassium permanganate, a series of solutions is produced and measured as a calibration for concentration of permanganate as a function of absorption. Finally, the change in permanganate-concentration is measured in-situ, both with and without a catalyst, in order to determine the reaction order.

In the second experiment, the acid-base reaction between phenolphthalein and sodium hydroxide is studied. The absorption of the reaction mixture is also measured in-situ and the reaction order regarding phenolphthalein determined.

2 Experiment

2.1 Determination of Absorption Maximum of $KMnO_4$

In order to find the absorption maximum, 79 mg of potassium permanganate were weighed out, dissolved in deionized wate and diluted in a 50 mL volumetric flask, yielding a 0.01 mol/L solution. 5 mL of this solution were taken out and once again diluted in a 50 mL flask, yielding a concentration of 1 mmol/L.

2 mL of the prepared solution were filled into a cuvette and inserted into the photometer. Its Absorption was then measured in wavelength increments of 10 nm between 450 nm and 600 nm. For each wavelength, the photometer was calibrated by setting values of 0% and 100% transmission, using an opaque block and a cuvette filled with deionized water, respectively. Since high absorptions were measured at 530 nm and 550 nm, another series of measurements was taken between 520 nm and 560 nm with increments of 2 nm. Using this process, a maximum at 532 nm was determined.

2.2 Creation of Calibration Curve for $KMnO_4$ Concentration

To create the calibration, the 1 mmol/L potassium permanganate solution, which was prepared in Section 2.1, was further diluted to prepare six further solutions, with concentrations of 0.75, 0.60, 0.50, 0.40, 0.25 and 0.10 mmol/L. After calibrating the photometer as described in Section 2.1, the absorption values of the seven solutions were measured at the previously determined wavelength of 532 nm. To account for inaccuracies, each measurement was taken three times, with the cuvette being filled with fresh solution after every measurement.

2.3 Measurement of the Autocatalyzed Redox Reaction of $KMnO_4$ and Oxalic Acid

By further diluting the 10 mmol/L solution prepared in 2.1, a solution with a concentration of 4 mmol/L was prepared. Additionally, a 40 mmol/L solution of oxalic acid was obtained by dissolving AAAAA g oxalic acid in deionized water in a AAAAA mL volumetric flask. Additionally, a sulfuric acid solution of about 24 w% was prepared by combining three parts water with one part concentrated (96 %) sulfuric acid.

To start the reaction, 10 mL of the oxalic acid solution, 0.4 mL of the sulfuric acid and 4 mL of the potassium permanaganate solution were added to a beaker and mixed thoroughly. A stopwatch was started once the permanganate solution was added. After filling 2 mL of the mixture into a cuvette, absorption measurements were taken every 10 seconds, the measured value reached 0 or stopped changing. This experiment was repeated two more times.

2.4 Measurement of the Mn^{2+}-Catalyzed Redox Reaction of $KMnO_4$ and Oxalic Acid

By dissolving AAAAA g manganese sulfate in deionized water in a AAAAA mL volumetric flask, a 40 mmol/L soluton was prepared.

Three reactions were measured as described in Section 2.3, with the addition of adding 0.2 mL of the prepared manganese sulfate soltion to the reaction mixture before mixing it.

2.5 Measurement of the Deprotonation of Phenolphthalein by Sodium Hydroxide