Experiment 11: Analysis of Drinking Water by Spectrophotometry

Purpose

The goal of this experiment is to calculate the concentration of the permanganate ion, MnO_4^{1-} in solution by means of a spectrophotometer and calibration curve.

Background

Potassium permanganate, $KMnO_4$, is a strong oxidizing material that can be used for water treatment (https://drinking-water.extension.org/drinking-water-treatment-potassium-permanganate/). When applicable, it is used as a point-of-entry water treatment to control the levels of dissolved iron, manganese, and hydrogen sulfide. It can also be used to control iron bacteria growth in wells. Since potassium permanganate is considered toxic and is harmful to skin, there should not be any excess permanganate left in the drinking water. This experiment will have you analyze a sample of drinking water for the presence of permanganate. You will also be analyzing a quality control sample of known concentration to check if your analysis is accurate.

When light of a specific wavelength passes through a solution containing a substance that absorbs the light, the intensity of the light decreases. The absorption of light can be reported as absorbance, which is measured with a spectrophotometer.

Beer's Law states that the absorbance, A, is directly related to the concentration, c, of the substance that absorbs light (the permanganate ion).

$A = abc$ where a is the molar absorptivity constant specific to a substance, and b is the path length through the sample.

Beer's Law has the format $y = mx + b$ where y is the absorbance and x is the concentration of the substance (the b here is the y-intercept, not the path length). This law allows us to prepare a linear calibration curve, so we can calculate the concentration of permanganate ion in an unknown solute when we know the absorbance by the unknown.

Chemicals

Potassium permanganate stock solution, 5.0×10^{-3} M
Potassium permanganate quality control sample, 2.5×10^{-4} M
Drinking water sample of unknown permanganate concentration
Deionized water

Equipment

Volumetric flasks, 50 mL
Test tubes, small 13x100 size
Plastic droppers
Beakers, assorted small sizes
Pipet, 10 mL, and pipettor
Spectrophotometer

The permanganate ion has an absorbance linear range when the following concentrations are used for the standard solutions (Table 1). Calculate the volume of stock solution needed to prepare each standard solution.

Table 1

Standard Solution Number	Concentration, M	Volume of Stock** $KMNO_4$ to pipet
1	1.0×10^{-4}	
2	2.0×10^{-4}	
3	3.0×10^{-4}	
4	4.0×10^{-4}	
5	5.0×10^{-4}	

**Stock solution 5.0×10^{-3} M, volume of volumetric flask is 50.0 mL.

Procedure

Part A: Preparing the Standard Solutions

1) Obtain enough concentrated potassium permanganate solution from the stock bottle in the hood. Enough is determined by how much is needed to prepare all of the standard solutions, plus a little extra to avoid getting air bubbles in the pipet.
2) Rinse each volumetric flask with deionized water before making the solutions.
3) Pipet the appropriate volume of the concentrated solution into each volumetric flask.
4) Dilute the contents of each flask with deionized water. Be sure to get the bottom of the meniscus exactly at the calibration line on the neck of each flask, and mix well.

Part B: Determining the Wavelength for Absorbance

The λ_{max} for the permanganate ion is approximately 530 nm. Test this maximum before finding the absorbance of your solutions.

1) Fill a small test tube with standard number 5, to about 2/3 full.
2) Fill a small test tube with deionized water, to about 2/3 full. This will be the blank.
3) Set the wavelength of the spectrophotometer to 500 nm.
4) Put the blank into the sample compartment of the spectrophotometer. Close the lid. Press the 0 ABS button to set the zero mark for the spectrophotometer.
5) Remove the blank and put the test tube with standard 5 into the sample compartment. Close the lid. Take the absorbance reading.
6) Repeat steps 4 and 5 for the following wavelengths:
   510 nm, 520 nm, 525 nm, 530 nm, and 535 nm
7) Set the spectrophotometer to the λ_{max} just determined.

Part C: Measuring the Absorbance of the Standard Solutions and Samples

1) Use the deionized water blank to re-zero the spectrophotometer.
2) Put standard 1 into a clean, dry test tube, about 2/3 full.
3) Wipe the outside of the test tube, and then place it inside the sample compartment of the spectrophotometer.
4) Record the absorbance displayed on the instrument readout.
5) Repeat steps 2 – 4 for the remaining standard solutions, the quality control-known solution (2.5×10^{-4} M), and the unknown sample solution.

Part D: Prepare the Calibration Curve and Determine the Concentration of the Samples

1) Use Excel or Google Sheets to prepare your calibration curve. This should be a scatter plot, with a linear line. Show the equation of the line on your curve. Also show the R^2 value. A perfect calibration curve is when all data points fall on the line; this would have an R^2 value of 1.

2) Use the standard solution concentrations as the x axis values. Their absorbance values go on the y axis. **DO NOT PLOT** the quality control sample and unknown sample.

3) Use the equation of the line to calculate the concentration of permanganate ion in the quality control sample and in the unknown sample. The absorbances of these samples are known (the y values). Solve for their concentrations (the x values).