

CH 2212

Spectrophotometric Analysis of a Mixture

In this experiment, you will quantitatively determine the concentrations of permanganate (MnO_4^-) and dichromate ($\text{Cr}_2\text{O}_7^{2-}$) ions in a mixture. Because of the overlap of spectra, it is not feasible to generate a calibration curve of one ion and then the other and use a straightforward Beer's Law approach to determine each concentration. The problem is that each ion has significant absorbance at the other's λ_{max} . Dichromate is normally quantified at a wavelength of 440 nm. Permanganate is measured at 545 nm. These wavelengths correspond, more or less, to wavelengths where the absorbance is not changing much over a range of wavelengths. Hence, small deviations in the measurement wavelength do not affect the measured absorbance.

$$A_{440 \text{ nm}} = \epsilon_{440, \text{Cr}_2\text{O}_7^{2-}} (b, \text{cm})(M, \text{Cr}_2\text{O}_7^{2-}) + \epsilon_{440, \text{MnO}_4^-} (b, \text{cm})(M, \text{MnO}_4^-)$$

$$A_{545 \text{ nm}} = \epsilon_{545, \text{Cr}_2\text{O}_7^{2-}} (b, \text{cm})(M, \text{Cr}_2\text{O}_7^{2-}) + \epsilon_{545, \text{MnO}_4^-} (b, \text{cm})(M, \text{MnO}_4^-)$$

You will determine the four absorptivities (one for each ion at each wavelength). From the measured absorbance of the mixture at each wavelength, you will have a system of two equations in two unknowns which can be algebraically solved for the molar concentrations of the two ions.

Procedure

- 1) Give your lab instructor a clean (not necessarily dry) 250-mL volumetric flask labeled with your name. You will receive in this flask a mixture of permanganate and dichromate ions, the concentrations of which are known to the instructor. Dilute this mixture to the mark with distilled water.
- 2) Prepare a safety summary for dichromate. Note that dichromate ion contains hexavalent chromium (Cr^{+6}), which is a known human carcinogen. Your safety summary should contain an MSDS (look up potassium dichromate) and all storage and handling procedures you intend to use in this class. A plan for spillage should be included. We will collect all waste dichromate in a waste container in the hood. **Your lab instructor needs to approve your safety summary before you go on to Step 3 of this experiment.**
- 3) Obtain a small volume (25-50 mL) of each of the two standard solutions. These must be taken in clean, dry vessels to avoid contamination and dilution. The permanganate solution contains 0.0150 mg Mn/mL and the dichromate solution contains 0.150 mg Cr/mL. Note that these concentrations are given in mass of the elements, not the oxyanions.
- 4) Set up your Ocean Optics Spectrometer as you have been instructed. Take three careful measurements of the absorbance of the standard solution of each anion at

each of the analytical wavelengths. Average these measurements. Calculate the molar absorptivity for each anion at each wavelength.

- 5) Measure the absorbance of your mixture from the 250-mL volumetric flask at each of the analytical wavelengths. Repeat three times. Average these measurements.
- 6) From the absorbance data, determine the concentrations of Mn and Cr in your mixed solution in units of mg/mL. Report these values on the CH 2212 Report Sheet available on the web site. Attach the associated notebook pages.