

EXAMINATION IN KJ 2050, ANALYTICAL METHODES I (7.5 sp)

Tuesday 20th December 2010, 9.00 – 13.00.

There are 3 pages in this document. Allowed auxiliary equipment: pocket calculator

All the questions should be answered, however in question 2e select one of the two selectable questions **EITHER** / **OR** and in question 3c select one of the two selectable questions *either* / *or*.

Deadline for examination results: 27th of January 2011 (3 weeks + 10 days).

Contact person during the examination: Øyvind Mikkelsen (928 99 450)

Task 1 (5p + 7p + 2p + 3p + 3p = 20p total)

We have a 50 mL of a 0.005 M strontium solution. This sample is to be titrated with a 0.01 M standard EDTA-solution. The titration is carried out in a buffer solution at pH 12.5 (preventing precipitation) using murexide as indicator.

a. Give the expression for the titration error (T), and calculate the theoretical remaining concentration of Sr^{2+} at equilibrium point for this titration.

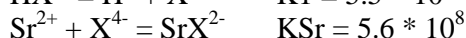
b. Calculate the titration error in percent for this titration if we assume that the actual concentration of remaining strontium is in the range of $1 \cdot 10^{-5}$ to $1 \cdot 10^{-7}$ M at the end of the titration. Give comments to the calculated answer.

c) We now assume that the above sample also could in some cases contain significant amounts of dissolved Li^+ or Cu^{2+} ions. The Formation Constants for the EDTA-metal complexes with Li^+ and Cu^{2+} are $8.9 \cdot 10^2$ and $6.0 \cdot 10^{18}$ respectively. Which of the two metal ions could particularly give problems due to interferences in an EDTA titration carried out under the conditions given in a?

d. There are different ways to determine cation concentration by use of EDTA-titration. A Ca^{2+} solution is to be determined by EDTA titration. Describe how this can be done by using direct titration, back titration and by the displacement method. Point out when it is appropriate to choose each of the individual methods.

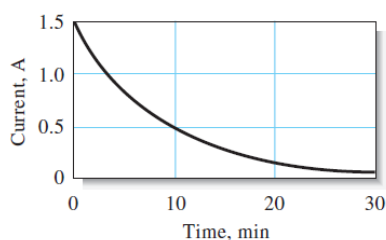
e. Describe briefly an alternative analytical method for Ca^{2+} determination in the concentration range $1 \cdot 10^{-2}$ M to $1 \cdot 10^{-5}$ M, and evaluate this method against the complexometric titration in view of important source of errors and interferences.

DATA ($\text{H}_4\text{X} = \text{EDTA}$)



Task 2 (5p + 3p + 2p + 2 p + 5p = 17p total)

- a. Precipitation gravimetry is a method where the sample solution is added a chemical reagent producing a slightly soluble participate with the analyte. Co-precipitation is a possible source of error in this method. Describe different types of co- precipitation, explain how they can affect the final results, and describe some procedures to improve or avoid such errors from co-precipitation.
- b. Describe briefly the principles for electrogravimetry (without control of the cathode potential), and make a brief evaluation of this method compare to precipitation gravimetry with regard to sources of errors, interferences, and precision/accuracy.
- c. Suggest suitable electrode materials for the cathode and the anode in electrogravimetric determination in individual solutions of copper and zinc respectively.
- d. An electrogravimetric experiment is carried out, study Figure 1 and explain what phenomenon is observed, and then explain why a depolarizer like nitrate is added to such analyses.



(a)

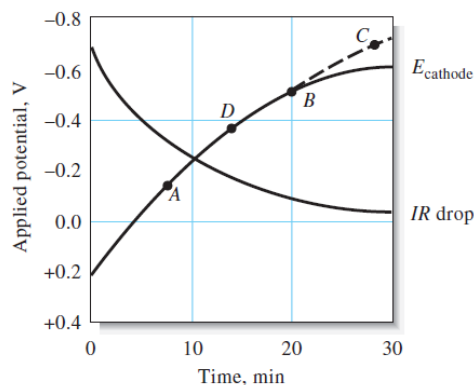


Figure 1

EITHER

- e. Describe the procedures for gravimetric and spectrophotometric determination of iron. Compare the two methods and evaluate important sources of errors and interferences. Describe how the quantification is performed in the two methods.

OR

- e. Describe the procedures for electrogravimetric and iodometric determination of copper. Compare the two methods and evaluate important sources of errors and interferences. Describe how the quantification is performed in the two methods.

Task 3 (2p + 2p + 4p = 8p total)

- a. What requirements are set for a primary standard solution for use in titration? What is the difference between an endpoint and an equivalence point in a titration?
- b. Why does many color indicators have a color change of ± 1 pH unit in acid-base titrations? Mention some other methods in general for determine endpoints in titrations.
- c. Describe in detail a procedure for standardizing *either* a 0,1M HCl solution *or* a 0,1M NaOH solution.

Task 4 (10p + 5p = 15p total)

- a. Describe the principles of Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) and atomic absorption spectroscopy (AAS). Then describe briefly what these methods are used for and their approximately detectable concentration range.
- b. Define “speciation”. Explain important aspects with respect to the choice of analytical methods in connection with speciation studies.

Task 5 (10p)

Indicate by check off if the following statements are correct to wrong

	Correct	Wrong
Iodide could be oxidized to iodine in air.		
Iodine is an important titration reagent because it is a strong oxidizing agent, and therefore might be used for determination of strongly reducing agents in presence of weak ones.		
$\text{OCl}^- + 2\text{I}^- + 2\text{H}^+ = \text{Cl}^- + \text{I}_2 + \text{H}_2\text{O}$		
$\text{I}_2 + \text{OH}^- = \text{IO}^- + \text{I}^- + \text{H}^+$		
Precipitation titration with silver nitrate is used for determination of cations like zinc, magnesium and calcium.		
In brief; the Volhart titration is determination of Fe^{3+} through titration against thiocyanate, and where Ag^+ is used as indicator.		
Potentiometry is based on the measurement of a potential at virtually zero current in the electrochemical circuit.		
Ion selective electrodes have a logarithmic response.		
Voltammetry is an analytical method that is particularly well suited to determine the alkaline and earth alkaline metals.		
Voltammetry is a sensitive technique with detection limits of several important trace and heavy metals in ppt - ppb range.		