

Spring 1996

Tuesday, June 11, 1996

Time: 90 minutes

Prof. Ayssar Nahlé

Chemistry 206

Quantitative Analysis

Lab. Final

Name:

Family

First name

I.D. number: _

Section number:
(Please circle)

1

2

3

Grade

I / 16

II / 18

III / 18

IV / 16

V / 16

VI / 16

total /100

Good luck

I. A 0.500-g sample is analyzed spectrophotometrically for manganese by dissolving it in acid and transferring to a 250-mL volumetric flask and diluting to volume. Three aliquots are analyzed by transferring 50-mL portions with a pipette to 500-mL Erlenmeyer flasks and reacting with an oxidizing agent, potassium peroxydisulfate, to convert manganese to permanganate. After reaction, these are quantitatively transferred to 250-mL volumetric flasks, diluted to volume, and measured spectrophotometrically. By comparison with standards, the average concentration in the final solution is determined to be 1.25×10^{-5} M. What is the percent manganese in the sample?

% Mn in the sample =

II. Nitrate nitrogen in water is determined by reacting with phenoldisulfonic acid to give a yellow color with an absorption maximum at 410 nm. A 100-mL sample that has been stabilized by adding 0.8 mL H_2SO_4 / L is treated with silver sulfate to precipitate chloride ion, which interferes. The precipitate is filtered and washed (washings added to filtered sample). The sample solution is adjusted to pH 7 with dilute NaOH and evaporated just to dryness. The residue is treated with 2.0 mL Phenoldisulfonic acid solution and heated in a hot water bath to aid dissolution. Twenty milliliters distilled water and 6 mL ammonia are added to develop the maximum color, and the clear solution is transferred to a 50-mL volumetric flask and diluted to volume with distilled water. A blank is prepared using the same volume of reagents, starting with the disulfonic acid step. A standard nitrate solution is prepared by dissolving 0.722 g anhydrous KNO_3 and diluting to 1 L. A standard addition calibration is performed by spiking a separate 100-mL portion of sample with 1.00 mL of the standard solution and carrying through the entire procedure. The following absorbance readings were obtained: blank, 0.032; sample, 0.270; sample plus standard, 0.854. What is the concentration of nitrate nitrogen in the sample in parts per million?

Concentration of nitrate nitrogen = ppm

III. Sodium carbonate can coexist with either NaOH or NaHCO₃ but not with both simultaneously, since they would react to form Na₂CO₃. Sodium hydroxide and Na₂CO₃ will titrate together to a phenolphthalein end point (OH⁻ → H₂O; CO₃²⁻ → HCO₃⁻). A mixture of either NaOH and Na₂CO₃ or of Na₂CO₃ and NaHCO₃ is titrated with HCl. The phenolphthalein end point occurs at 15.00 mL and the modified methyl orange end point occurs at 50.0 mL. The HCl was standardized by titrating 0.477 g Na₂CO₃, requiring 30.0 mL to reach the modified methyl orange end point. What mixture is present and how many millimoles of each constituent are present?

The mixture is: NaOH and Na₂CO₃

(tick one)

Na₂CO₃ and NaHCO₃

millimoles of NaOH if any =

millimoles of Na₂CO₃ if any =

millimoles of NaHCO₃ if any =

IV. A 100.0-ml sample of water containing Ca^{2+} and Mg^{2+} is titrated with 22.74 mL of 0.00998 M EDTA solution, at pH 10.0. Another 100.0-mL sample is treated with NaOH to precipitate $\text{Mg}(\text{OH})_2$, and then titrated at pH 13 with 15.86 mL of the same EDTA solution. Calculate:

a) the total water hardness in :

A) French degrees, F^0 (mg CaCO_3 /100 mL):

.....

B) German degrees, D^0 (mg CaO /100 mL):

.....

C) American degrees (mg CaCO_3 /L, i.e. ppm CaCO_3):

.....

b) the ppm of CaCO_3 and MgCO_3 in the sample.

ppm CaCO_3 =

ppm MgCO_3 =

V. A 0.1914-g sample of calcium carbonate is dissolved in hydrochloric acid and the calcium is precipitated as CaC_2O_4 . The precipitate is dissolved in dilute H_2SO_4 , and the resulting solution is titrated with 36.50 mL of KMnO_4 solution, 35.57 mL of which are equivalent to 0.2383 g $\text{Na}_2\text{C}_2\text{O}_4$. A blank determination required 0.08 mL KMnO_4 . Calculate the percent CaO in the sample.

% CaO in the sample =

VI. a) Derive the titration curve of 70 mL of 0.120 M Na_2CO_3 solution when titrated with 0.140 M HCl. Use the HCl volumes shown in the table below, and fill in the corresponding pH values.

($K_{a1} = 4.2 \times 10^{-7}$; $K_{a2} = 4.8 \times 10^{-11}$ for carbonic acid)

<u>$V_{\text{HCl, mL}}$</u>	<u>pH</u>
0.00	
10.00	
30.00	
40.00	
60.00	
70.00	
80.00	
90.00	
120.00	
130.00	
140.00	

b) Plot the derived titration curve on the provided graph paper.