Titration Tango

Points:

13% of total										
Question	Titration 1	Titration 2	2.1	2.2	2.3	2.4	2.5	2.6	2.7	**Total**
Points	30	40	5	4	4	2	1	2	2	**90**
Score										

Introduction

Iron was historically mined and processed in 19 Swiss cantons, meeting local and regional demand. Evidence of this activity remains, particularly in the Swiss Jura. To produce iron and steel efficiently, knowledge of the composition of the iron ore is essential. A versatile method to analyze any metal in solution is the complexometric titration, pioneered by Prof. Gerold Schwarzenbach at ETH in the 1940s.

You are provided with a sample containing only hydrates of $FeCl_3$ and $CaCl_2$, dissolved in aqueous HCl. This simulates an iron ore sample, which has been digested with hydrochloric acid. **Your task is to determine the iron concentration and the overall composition of the sample by complexometric titrations.**

Any aqueous waste of this task is considered to contain heavy metals and should be collected in the beaker labelled "**Waste P2**".

Procedure

Part I. Dilution of Unknown Iron Ore Sample

1. You are given a sample of ca. $1200\ \mathrm{mg}$ of simulated iron ore. The exact mass is written on the label of your vial. **Report** it in your **Answer Sheet**. The sample has already been dissolved in aqueous HCl of pH 1.

2. **Prepare** $100\ \mathrm{mL}$ of sample solution in the $100\ \mathrm{mL}$ volumetric flask using the whole content from the vial labelled "**Sample + [student code]**" and distilled water. You may use a funnel. This solution is called **A**. This solution will be used in part II and IV.

Part II. Direct Titration of Iron Ore Solution

3. **Fill** the burette with $10.0\ \mathrm{mM}$ EDTA solution, labelled as "**EDTA**". You may use a funnel and a beaker.

4. In a $300\ \mathrm{mL}$ Erlenmeyer flask:
   - **Add** $5.00\ \mathrm{mL}$ of solution **A** using a volumetric pipette;
   - **Add** 10 drops of $0.1\ \mathrm{M}$ hydrochloric acid using a glass Pasteur pipette;
   - **Fill up** to the $100\ \mathrm{mL}$ mark of your Erlenmeyer flask with distilled water;
   - **Add** a small amount of variamine blue using a spatula.
5. **Titrate** the content of the Erlenmeyer flask until the solution becomes yellow. **Record** the titration volume $\mathbf{V_1}$ in your **Answer Sheet**.
6. **Discard** the titrated content of the Erlenmeyer flask in the beaker labelled "**Waste P2**".
7. **Repeat** the procedure (steps 3 – 6) as needed.
8. **Report** your final result in the last row on the **Answer Sheet**.

Part III. Titer preparation

9. You are given a sample of ca. $550\ \mathrm{mg}$ of pure calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$). The exact mass is written on the label of your vial. **Report** it in the table in your **Answer Sheet**.
10. **Prepare** $250\ \mathrm{mL}$ of calcium chloride solution in the $250\ \mathrm{mL}$ volumetric flask using the whole sample of solid $CaCl_2 \cdot 2H_2O$ (MW = $147.0\ \mathrm{g/mol}$) and distilled water. You may need a funnel to transfer the solid. This solution is called **B**. It will be used in part IV.

Part IV. Indirect Titration of Iron Ore Solution

11. **Empty** the burette. **Rinse** the burette well with distilled water and then with solution **B**. You may use a beaker. **Discard** the rinse solutions in the beaker labelled "**Waste P2**".
12. **Fill** the burette with solution **B**. You may use a funnel and a beaker.
13. In a $300\ \mathrm{mL}$ Erlenmeyer flask, **add**:
    - $5.00\ \mathrm{mL}$ of solution **A** using a volumetric pipette;
    - $40.0\ \mathrm{mL}$ of $10.0\ \mathrm{mM}$ EDTA solution, labelled as "**EDTA**", using a volumetric pipette;

- 10 drops of buffer solution using a glass Pasteur pipette (**be careful** when opening the buffer solution, as there can be evolution of ammonia);
- $25\ \mathrm{mL}$ of distilled water using a graduated cylinder;
- $30\ \mathrm{mL}$ of ethanol using a graduated cylinder.

Your sample may be turbid.

14. To the $300\ \mathrm{mL}$ Erlenmeyer flask, **add** a small amount of Eriochrome® Black T, from the vial labelled with "**Erio T**". Your sample should now be intense blue-green. ~~Perform~~ **Perform** the titration **immediately** after the addition of the indicator.

    Note: After addition of the indicator, the color will change to red after several minutes, regardless of the progress of the titration. At that point, the titration endpoint is no longer detectable.

15. **Titrate** the content of the Erlenmeyer flask until the solution turns grey. **Record** the titration volume $\mathbf{V_2}$. Your expected volume is below $15\ \mathrm{mL}$.

16. **Discard** the titrated content of the Erlenmeyer flask in the beaker labelled "**Waste P2**".

17. **Repeat** the procedure (steps 12 – 16) as needed.

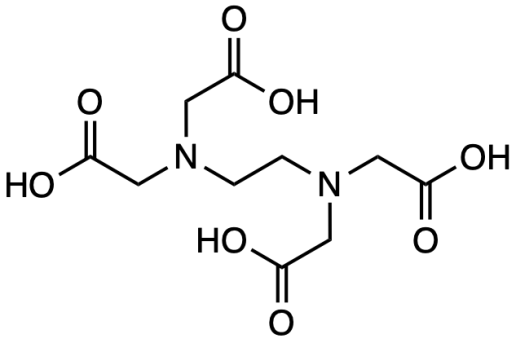
18. **Report** your final result in the last row on the **Answer Sheet**.

Titr.1	30pt

Titr.2	40pt

Questions

2.1	**Provide** the chemical formula of the resulting EDTA complex formed in the direct titration up to the equivalence point. The structure of EDTA is given below. In your chemical formula, abbreviate EDTA as "$\mathbf{H_4Y}$", its conjugate bases as "$\mathbf{H_3Y^-}$", "$\mathbf{H_2Y^{2-}}$" etc. Hint: Under these conditions, one of the metal ions in solution preferentially forms an EDTA complex.	5pt



Structure of EDTA (equivalent to **H_4Y**).

2.2	**Calculate** the mass percentage of iron(III) chloride (without water of crystallization), in $wt.\%$, of the provided sample. The molar mass of $FeCl_3$ is $162.2\ \mathrm{g/mol}$.	4pt
2.3	**Calculate** the mass percentage of calcium chloride (without water of crystallization), in $wt.\%$, of the provided sample. The molar mass of $CaCl_2$ is $111.0\ \mathrm{g/mol}$.	4pt
2.4	**Calculate** the mass percentage of water of crystallization, $wt.\%$, of the provided sample.	2pt
2.5	Why is it necessary to keep the sample solution **A** at pH < 2? **Identify** the correct answer among the four choices below and **fill in** the corresponding checkbox in your **Answer Sheet**.	1pt
2.6	The solution you were given simulates the digestion of iron ore with concentrated HCl. Which of the following mixtures could be analyzed by the same procedure? **Identify** the correct answer among the four choices below and **fill in** the corresponding checkbox in your **Answer Sheet**.	2pt
2.7	Why does the sample for the indirect titration show a color change from blue to red regardless of the progress of the titration? **Identify** the correct answer among the four choices below and **fill in** the corresponding checkbox in your **Answer Sheet**.	2pt