

3rd Practical work: Volumetric acid-basic titration

1/Titration of a solution:

A titration reaction is used to determine the concentration of a species in solution ($C_{A} = ...$?) To do this operation:

A first solution containing the species of **unknown concentration "the titrate solution A**" is brought into contact with a second solution containing a reagent of **known concentration "the titrant solution"**. The **titrant solution** is gradually poured into the titrate solution **drop by drop** until the equivalence point is reached.



At the equivalent point, the two reagents (A and B) are mixed in **stoichiometric proportions**, which allows the unknown concentration to be deduced.



A dosage (or titration) acid-base can be followed by:

pH-metric: we follow the evolution of the pH during the reaction.

Colorimetry: we use a colored indicator, which will be studied in this practical work.

A colored indicator: is a reagent whose color depends on the medium (or pH). It can be used to mark the end of a dosage if equivalence is reached in its turning area (table 1).

Table 1 : Exemples of colored indicators:

Indicator	Acid form	Turning area	Basic form
Helianthine	Red	3,1 - 4,4	Yellow
Methyl red	Red	4,08 - 6	Yellow
Bromothymole bleu	Yellow	6,0 - 7,6	Bleu
Phenol-phtaleine	Incolore	8,2 - 10,0	Pink
Alizarine Yellow	Yellow	10,1 - 12,2	Red



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2/Objectives:

The main objective of this practical work is to: Determine the molar concentration of an acid solution with acid-basic titration.

3/Principal of manipulation :

This titration is based on a colorimetric dosage.

4/Material and products:

-Burette (25-50 mL) -Graduated or volumetric pipettes (10 mL) -Pro-pipette -Volumetric flasks (50 and 100 mL) -Erlenmeyer flask (100 mL) -Beakers -Sodium hydroxide solution (0.1M NaOH) - HCl hydrochloric acid solution -H₂SO₄ sulfuric acid solution -Colored indicator

5/Dosage of a monoacid using a strong base:

The dosage of the hydrochloric acid HCl solution S2 will be carried out with a sodium hydroxide NaOH solution of molar concentration $C_B = 0.1$ mol/L, with some drops of phenolphthalein as a colored indicator.

The global reaction equation is:

HCl + NaOH \longrightarrow (Na⁺_{aq}, Cl⁻_{aq}) + H₂O

5.1/ Preparation of the solution to titrate (HCl hydrochloric acid solution S2):

- 1. Take a clean beaker, and pour a small quantity of HCl solution of unknown concentration S1.
- 2. Rinse the 10 ml pipette with the solution S1 (use the pro-pipette)
- 3. Take a volume of 10 ml of the solution S1 (use the rinsed pipette).
- 4. Introduce this test portion into the 100 ml volumetric flask which contains a small quantity of distilled water
- 5. Complete with distilled water up to the jauge line
- 6. Close the volumetric flask with the stopper and shake .
- 7. You obtain a solution S2 (HCl of unknown concentration).

5.2/ Rapid dosage (equivalent volume supervision):

The rapid dosage is performed before the precis dosage to determine the equivalence point interval (the equivalence point will be included in a volume interval).

Work to be done :

- 1. Check that the burette tap is properly closed.
- 2. Rinse the burette with distilled water, then with the titrant solution (NaOH $C_B = 0.1 \text{ mol/L}$).
- 3. Degas the bottom of the burette by draining the titrant solution into a labeled beaker.
- 4. Fill the burette with NaOH $C_B = 0.1 \text{ mol/L}$.
- 5. Adjust the liquid level to the zero level of the burette by draining the NaOH excess into the labeled beaker 1.
- 6. Pour approximately 40 mL of solution (S2) into a labeled beaker 2
- 7. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
- 8. Introduce this test portion into an erlenmeyer flask add an amount quantity of water



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9. Add 1 to 3 drops of phenolphthalein indicator.

10. Place the erlenmeyer flask under the burette (*figure 1*)

11.Add the titrant solution NaOH mL per mL, and note the color of the solution by completing Table2. 12.Shake manually.

13. The solution change color when you added the equivalent volume of titrant solution (**Veq**). Indicate approximately this volume (by a frame): V 1 mL < Veq < V 2 mL

$\dots V_1 \text{ mL} < \text{Veq} < \dots V_2 \text{ mL}$

Table2 : Volumes equivalents

V _{NaOH titrant} (mL)	1	2	3	4	5	6	7	8	9	10
Color solution										

5.3/ Precise dosage (drop dosage):

The precise dosage (by drop by drop), is carried out after the rapid dosage to determine equivalence points with precision. The titrant solution is poured up to 1.5 mL or 2 mL of the equivalence zone quickly, then by drop by drop until the equivalence. (first drop that changes the color of the solution)

Work to be done:

- 1. Adjust the liquid level to the zero level of the burette by adding the NaOH.
- 2. Pour approximately 40 mL of solution (S2) into a labeled beaker 2
- 3. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
- 4. Introduce this test portion into an erlenmeyer flask add an amount quantity of water
- 5. Add 1 to 3 drops of phenolphthalein indicator.
- 6. Place the erlenmeyer flask under the burette (*figure 1*)
- 7. Quickly at the beginning add the titrant solution NaOH to the solution, until the value of V_1 (determined at the quick dosage)
- 8. Then add the titrant solution **drop by drop**, as the color change approaches (equivalence point). Close the tap of the burette as soon as the first drop changes color, shake.
- 9. Note the equivalent volume V_{eq} and the color of the solution by completing Table 3.
- 10.Repeat the operation two to three times.

Table3 : Equivalents volumes :

	1 st test	2 nd test	3 rd test		
V _{eq} (mL)					
Color solution					

Questions:

- 1. Deduce the average equivalence volume $(V_{eq \cdot ave})$?
- 2. Calculate the concentration of hydrochloric acid solution S_2 ?
- 3. Deduce the concentration of solution S_1



6/ Dosage of a diacid H₂SO₄ using a strong base NaOH:

In this case, the dosage of sulfuric acid $H_2 SO_4$ will be carried out using a solution of sodium hydroxide NaOH $C_B = 0.1 \text{ mol/L}$, with some drops of phenolphthalein as a colored indicator The global reaction equation is:

 $H_2SO_4 + 2NaOH \longrightarrow (2Na^+_{aq}, SO_4^{-2}_{aq}) + 2H_2O$

6.1/ Rapid dosage (equivalent volume supervision):

- 1. Adjust the liquid level to the zero level of the burette by draining the NaOH excess into the labeled beaker 1.
- 2. Pour approximately 40 mL of H₂ SO₄ solution into a labeled beaker 3
- 3. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
- 4. Introduce this test portion into an erlenmeyer flask add an amount quantity of water
- 5. Add 1 to 3 drops of phenolphthalein indicator.
- 6. Place the erlenmeyer flask under the burette (*figure 1*)
- 7. Add the titrant solution NaOH mL per mL, and note the color of the solution by completing Table 4.
- 8. Shake manually.
- 9. The solution change color when you added the equivalent volume of titrant solution (Veq).

10.Indicate approximately this volume (by a frame):

$\dots \dots V_1 \text{ mL} < Veq < \dots \dots V_2 \text{ mL}$

Table 4: Volumes equivalents

V _{NaOH titrant} (mL)	1	2	3	4	5	6	7	8	9	10
Color solution										

6.2/ Precise dosage (drop dosage):

11. Adjust the liquid level to the zero level of the burette by adding the NaOH.

12. Pour approximately 40 mL of solution H_2 SO₄ into a labeled beaker 3

- 13. Take 10 mL of this solution using a clean volumetric pipette fitted with a pro-pipette.
- 14.Introduce this test portion into an erlenmeyer flask add an amount quantity of water
- 15.Add 1 to 3 drops of phenolphthalein indicator.
- 16. Place the erlenmeyer flask under the burette (*figure 1*)
- 17.Quickly at the beginning add the titrant solution NaOH to the solution, until the value of V_1 (determined at the quick dosage)
- 18. Then add the titrant solution **drop by drop**, as the color change approaches (equivalence point).
- 19. Close the tap of the burette as soon as the first drop changes color, shake.
- 20.Note the equivalent volume V_{eq} and the color of the solution by completing Table 5.
- 21.Repeat the operation two to three times.

Table 5 : Equivalents volumes

	1 st test	2 nd test	3 rd test
V _{eq} (mL)			
Color solution			

Questions:

- 1. Deduce the average equivalence volume $(V_{eq \cdot ave})$?
- 2. Calculate the concentration of sulfuric acid H₂ SO₄ ?
- 3. Calculate the normality of the sulfuric acid solution?





Figure 1 : Acid-base titration