TITRATION OF ACTIVE CHLORINE

WITH SODIUM HYPOCHLORITE SOLUTION

TITRATION OF ACTIVE CHLORINE WITH SODIUM THIOSULFATE

EQUIPMENT

1 Measuring scale with ± 10 mg precision
1 Lab hot plate with magnetic stirrer
1 Tripod with three-prong burette clamp and tightening nut
3 Magnetic bars measuring approximately 6x25 mm
1 Burette 25 ml
1 Graduated pipette with 5 ml capacity
1 Graduated pipette with 20 ml capacity
2 Pear pipetting
1 Graduated cylinder 20 or 25 ml
1 Graduated cylinder 50 ml
1 Graduated cylinder 250 ml
3 Erlenmeyer flask 100 ml
1 Volumetric flask 250, 500 or 1000 ml
1 Erlenmeyer Flask 100 or 250 ml
1 Erlenmeyer Flask 250 or 1000 ml (with cork)
1 Thermometer
1 Spatula
2 Funnels (different sizes)

Preparation time for reagents: **approx. 1h30**

INGREDIENTS

NAME	FORMULA	PURITY
Distilled (or deionised) water	H_2O	Resistivity 10-18.2 MΩcm
Sodium thiosulfate pentahydrate (CAS no. : 10102-17-7)	$Na_2S_2O_3 \times 5H_2O$	≥ 99%, extra pure
Potassium iodide (CAS no. : 7681-11-0)	KI	≥ 99.9%
Acetic acid, glacial	CH_3COOH	≥ 98.85%
Potato starch	$(C_6H_{10}O_5)_n$	-

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PREPARING THE SOLUTIONS

- The calculation of the quantities required to prepare the different solutions necessary for the titration of the active chlorine can be found in the table below (page 2).
- Before preparing the reagents, it is important to define the amount you will need.
- As a guideline, consider using 15 ml of solution A and 20 ml of solution C per titration. Allow for at least 3 titrations per sample.
- For solution A, it is recommended to produce an amount corresponding to the volume of one of the volumetric flasks available.
- In the table, the empty cells in yellow should be filled in by the user. The cells highlighted in green correspond to the quantities to be sampled for the preparation of the solutions.

CALCULATING THE QUANTITIES (SEE DYNAMIC FILE ATTACHED)

SOLUTION A : THIOSULFATE 0.1 mol/l			
Volume of solution to prepare:	250	ml	precisely
Thiosulfate pentahydrate:	6.20	g	± 0.01 g
Concentration of the solution $Na_2S_2O_3$:	0.1	mol/l	

SOLUTION B : STARCH 10 g/l			
Volume of distilled water:	50	ml	approx. (± 1 ml)
Quantity of starch:	0.5	g	approx. (pure starch)

SOLUTION C : INDICATOR			
Volume of distilled water:	200	ml	approx. (± 1 ml)
Quantity of acetic acid, glacial (100%):	10.0	ml	approx. (± 0.5 ml)
Quantity of solution B:	10.0	ml	approx. (± 0.5 ml)
Quantity of potassium iodide:	0.8	g	approx. (± 0.1 g)

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METHOD

Note: Given that solution B needs to be brought to boiling point then cooled down, it is advisable to prepare it first (see point 2).

1. SOLUTION A : SOLUTION OF SODIUM THIOSULFATE 0.1 mol/l

- In the volumetric flask:
- Add the sodium thiosulfate pentahydrate (weigh precisely with a margin of ± 0.01 g)
  - Add distilled water to fill up the quantity to the correct volume
  - Close the volumetric flask with its stopper and shake slightly until complete dissolution of sodium thiosulfate.
- Make a note of the solution's concentration (blue cells in the table) on the volumetric flask.

Preparation time: 10 minutes
Storing: maximum 2 weeks in the fridge and sheltered from light (brown glass)

2. SOLUTION B : STARCH SOLUTION 10 g/l

- In 100 ml glass flask, insert :
  - The distilled water (accuracy ± 1 ml)
  - The starch (accuracy ± 0.1 g)
- Insert the magnetic bar and place on the magnetic stirrer (~400 t/m)
- Heat until boiling (approx. 98°C) and until starch has completely dissolved (the solution becomes transparent once completely dissolved)
- Allow to cool down to room temperature

Preparation time: 10 minutes + approximately 1 hour's resting time
Storing: maximum 1 week in the fridge

3. SOLUTION C : SOLUTION INDICATOR

- In a 250 ml Erlenmeyer flask (with cork), add :
  - Distilled water (accuracy ± 1 ml)
  - Acetic acid, glacial (accuracy ± 0.5 ml)
  - Solution B (accuracy ± 0.5 ml)
  - Potassium iodide (accuracy ± 0.1 g)
- Shake the solution well until the potassium iodide dissolves
- Place the solution away from the light.

Preparation time: 10 minutes
Storing: cannot be stored

TITRATION OF ACTIVE CHLORINE

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TITRATION OF THE ACTIVE CHLORINE

METHOD

- Rinse then fill precisely the burette with solution A up to the level corresponding to 0 ml on the graduation
- Extract a 5 ml sample for analysis using the graduated pipette (5ml) and place in a 100 ml Erlenmeyer flask
- Add to this 20 ml of solution C using a graduated pipette (20ml)
- Stir using the magnetic stirrer
- The solution should darken (blue-black)
  - If the solution does not colour, add a second dose of solution C
- Using the burette, add solution A drop by drop until the liquid completely loses its colour and becomes clear
- Wait 2 minutes and make sure that the solution has not turned blue again
  - If the solution is coloured once more, add solution A (sodium thiosulfate) one drop at a time until liquid becomes colourless
- Make a note of the total volume used by solution A in the table below and fill in the other yellow boxes.

CALCULATING THE CONCENTRATION OF ACTIVE CHLORINE (SEE DYNAMIC FILE ATTACHED)

			Concentration of solution A (thiosulfate) :	0.100 mol/l
Titration n°	Volume of sample [ml]	Volume of solution C [ml]	Volume of solution A [ml]	Active chlorine concentration [g/l]
1				
2				
3				
			Average :	n/a

The concentration can be calculated automatically using the following formula:

$$C_{\text{active chlorine}} = \frac{V_{Na_2S_2O_2} \cdot c_{Na_2S_2O_2}}{V_{\text{sample}}} \cdot M_{Cl\ [g/mol]}$$

$V_{Na_2S_2O_2}$: Volume of the sodium thiosulfate solution used for the titration in ml

$c_{Na_2S_2O_2}$: Concentration of thiosulfate: $\frac{m_{Na_2S_2O}}{M_{Na_2S_2O_3} \cdot V_{\text{solution}}} = 0.1$ mol/l

$V_{\text{échant.}}$: Volume of the sample of sodium hypochlorite = 5 ml

M_{Cl} : Molar atomic mass of chlorine = 35.4527 g/mol