

Practical Work n°4: ANALYSIS OF GLASS CONSTITUENTS

1-SODA CALCIUM GLASS ($\text{SiO}_2 + \text{Na}_2\text{O} + \text{CaO}$)

1. OBJECTIVES:

- Identify the main components of glass.
- Perform a qualitative and quantitative analysis of the oxides present (SiO_2 , Na_2O , CaO , etc.).
- Understanding the role of the different components in the structure of glass.

2. MATERIALS AND REAGENTS:

Material :

- Glass samples
- Mortar and pestle.
- Analytical balance.
- Graduated test tubes, beakers, volumetric flasks.
- Hot plates or bain-marie.
- Filter paper, funnels.
- Safety glasses, gloves, lab coat.

Reagents:

- Diluted hydrofluoric acid (HF) (use in a fume hood with strict precautions).
- Sulfuric acid (concentrated H_2SO_4).
- EDTA (solution for complexometric determination of calcium).
- Nitrate silver solution (AgNO_3) to detect Na^+ ions.
- Ammonium molybdate solution for detecting silicates . (**1g to 10ml**)
- Ammonium oxalate solution for detecting calcium oxide. (**0.5–10 ml**)
- Eriochrome Black T indicator (for calcium assay).

3. EXPERIMENTAL PROTOCOL:

Step 1: Sample Preparation

1. Clean the glass sample to remove any surface contamination.
2. Grind the glass into a fine powder using the mortar and pestle.
3. Accurately weigh **1.0 g** of glass powder using the analytical balance.

Step 2: Dissolving the sample

1. Add the powder to a beaker containing 10 mL of distilled water.

2. Add **5 mL of dilute hydrofluoric acid** to dissolve the silicates (SiO_2).
3. Slowly add **2 mL of concentrated sulfuric acid** to complete the chemical attack.
4. Gently heat the mixture ($50\text{-}60\text{ }^\circ\text{C}$) for 10-15 minutes to facilitate dissolution.
5. Dilute the resulting solution to 100 mL with distilled water in a volumetric flask.
6. Filter the resulting solution to remove insoluble particles.

STEP I: QUALITATIVE ANALYSIS

1. SiO_2 detection

Add a few drops of ammonium molybdate to a portion of the filtered solution. A yellow color indicates the presence of silicates.

2. Detection of Na^+ ions

Take a small amount of the solution, add 1 to 2 drops of silver nitrate (AgNO_3). A white precipitate (AgCl) confirms the presence of Na^+ (present as Na_2O in the glass).

3. Detection of Ca^{2+} ions

Add a few drops of ammonium oxalate $(\text{NH}_4)_2\text{C}_2\text{O}_4$. The formation of a white precipitate of CaC_2O_4 confirms the presence of Ca^{2+} .

STEP II: QUANTITATIVE ANALYSIS

1. Calcium (Ca^{2+}) assay :

- Take 10 mL of the filtered solution.
- Add a few drops of Eriochrome Black T indicator.
- Titrate with a standardized EDTA solution (0.01 mol/L) until a color change (blue).

2. Concentration calculation

Use the following formula to determine the concentration of Ca^{2+} :

$$C_{\text{Ca}^{+2}} = V_{\text{EDTA}} \times C_{\text{EDTA}} / V_{\text{CaO}}$$

Or ;

V_{EDTA} is the volume used in mL.

C_{EDTA} : the concentration of EDTA,

$M_{\text{Ca}^{+2}} = 40\text{ g/mol}$.

4. QUESTIONS

1. Name the equipment used in English?
2. What is the role of the following solutions: HF, H₂SO₄ and EDTA in this experiment ?
3. Calculate the mass concentration of calcium?
4. Determine the mass of calcium in grams?
5. What is the role of Na₂O in soda-lime glass?
6. Propose an alternative method for analyzing glass without using HF
7. Define the three types of glass:

1- Soda-calcium , 2- borosilicate, 3- opal

SIMPLE METHOD FOR DETECTING B₂O₃ IN GLASS**2-BOROSILICATE GLASS (SiO₂+B₂O₃+Na₂O)**

Boric ester test (Green flame with methanol)

OBJECTIVE

This is a quick, simple and visual method for qualitatively detecting boron in borosilicate glass.

MATERIALS:

Borosilicate glass sample, Concentrated sulfuric acid (H₂SO₄) , Methanol (CH₃OH)

Beaker or test tube , Match or lighter

EXPERIMENTAL PROTOCOL:**Sample preparation:**

Reduce the borosilicate glass to a fine powder (using a mortar and pestle).

Reaction with sulfuric acid:

Add a few milligrams of glass powder to a test tube.

Add 2-3 drops of concentrated sulfuric acid.

Addition of methanol:

Add approximately 2 mL of methanol to the test tube.

Ignition : Bring a match or lighter close to the opening of the tube.

Observe the color of the flame.

Expected result: If the glass contains B₂O₃ , an intense green flame will appear.

This color is due to the formation of trimethylborate (B(OCH₃)₃) which burns emitting a green flame.

Interpretation:

A green flame confirms the presence of boron in the form of borates in the sample. The absence of color does not indicate the absence of B₂O₃ or a very low concentration.