Everchem

METHOD OF ANALYSIS:

# VOLUMETRIC DETERMINATION OF $\%B_2O_3$, %B

DAEBAK

## 1. PRINCIPLE OF THE METHOD

DAEBAK is a mineral of sodium and calcium double borate; to dissolve the principal component it is necessary a chemical attack with HCl in a system that consists of a Boiling flask and a reflux condenser with refrigeration. Before perform the volumetric determination of B2O3 it is necessary to precipitate iron and aluminum as hydrates.
Titration of a dissolved test portion with a standard volumetric solution of sodium hydroxide in the presence of mannitol or sorbitol, using phenolphthalein as indicator.

## 2. EQUIPMENT

2.1. High sensitivity balance
2.2. pH meter
2.3. Boiling Flask 500mL capacity with ground glass connection
2.4. Reflux condenser with ground glass connection, water cooled
2.5. 500mL volumetric flask
2.6. 100mL volumetric pipet
2.7. 500mL Erlenmeyer flask
2.8. 25mL Buret graduation 1/20

## 3. CHEMICALS

3.1. Distilled water without $CO_2$ or water of equivalent purity
3.2. Concentrated HCl
3.3. HCl 1N solution
3.4. Paranitrophenol ( indicator) solution saturated in water
3.5. NaOH 4N solution
3.6. NaOH 1N solution
3.7. Phenolphthalein (indicator ) solution 10g/l in alcohol
3.8. Mannitol or sorbitol

## 4. PROCEDURE:

4.1. Weigh 10g of the sample (previously milled and mixed) to the nearest 0,001g into the boiling flask **2.3**, add 20mL of concentrated HCl **3.2** and approximately 150mL of water **3.1**, place a magnetic stirring bar in the flask, connect the reflux condenser to the flask and reflux for 30 minutes on the hotplate with constant stirring.
4.2. Remove the hotplate. After the solution has cooled, wash the condenser down with a small amount of water (10mL) and remove from the flask. Transfer the previously obtained solution to a 500mL volumetric flask **2.5** and then bring the volume to the mark.
4.3. Filter the solution **4.2** using filter paper Whatman 41, and transfer 100mL of the filtrated solution into a 500mL Erlenmeyer flask, to add 3 drops of the indicator paranitrophenol and neutralice with NaOH 4N until pH 6,5; heat to 50ºC, wait 5 minutes for the precipitation of iron and aluminum as basic hydrates.

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4.4. Filter these hydrates with Whatman 41 filter paper in a Erlenmeyer flask and wash the filter with hot water, cool down the filtered solution and acidify with HCl 1N until obtain a colorless tonality.

4.5. Add 5 drops of phenolphthalein **3.7** and approximately 12g of mannitol **3.8** and titrate with NaOH 1N until the solution turns violet.

**5. CALCULATION:**

$$\%B_2O_3 = \frac{A \times F \times 3.482}{P}$$

$$\%B = \%B_2O_3 \times 0.3106$$

where:

A : mL of NaOH used in titration
F : Correction factor of normality
P : Mass in grams of the test portion (titrated)

**REFERENCE:** Similar result with A.S.T.M. method: D 4487-90