Method 11.1 – Miscellaneous: available calcium oxide (CaO) in lime

1. Rationale

The method is applicable to limestone and determines the available calcium oxide in the lime in the presence of sugar by an acid-base titration.

2. Principle

A sample of lime is sub-sampled and ground to less than 0.1 mm particle size. The sample is mixed with a sugar solution and titrated against a hydrochloric acid solution.

3. Apparatus

3.1 **Analytical balance** readable to 0.0001 g

3.2 **Pestle and mortar**

3.3 **Tumbler**

3.4 **Volumetric flasks**: 100, 2 × 1 000 and 2 000 cm^3

3.5 **Conical flasks**: 6 × 250 cm^3

3.6 **Measuring cylinders**: 10 and 1 000 cm^3

3.7 **Beaker**: 1 000 cm^3

3.8 **Burettes**: 2 × 50 cm^3

3.9 **Bottles** with screw-on lids: 2 × 900 cm^3

3.10 **Sample bottle** with lid: 100 cm^3

4. Reagents

4.1 **Sodium hydroxide solution** (0.1 M)

Sodium hydroxide (NaOH) is a corrosive base and should only be handled with gloves while wearing safety glasses.

Weigh 4.0000 g sodium hydroxide pellets accurately and dissolve in 150 cm^3 distilled water. Cool the solution, transfer quantitatively to a 1 000 cm^3 volumetric flask and make to the mark with distilled water.

4.2 **Hydrochloric acid solution** (0.1 M)

Hydrochloric acid (HCl) is a corrosive acid and should only be handled with gloves while wearing safety glasses.

Measure 8.9 cm^3 concentrated hydrochloric acid (32%) and add to approximately 700 cm^3 distilled water in a beaker. Always add the acid to the water and not the other way around. This acid dilution is exothermic and the solution will therefore heat. Allow the solution to cool down, transfer to a 1 000 cm^3 volumetric flask and make to the mark.

4.3 Ethanol (absolute alcohol)

Ethanol (CH_3CH_2OH) is a flammable liquid and is toxic. It may cause damage to the eyes and safety glasses must be worn during use.

4.4 Phenolphthalein indicator (1%)

Weigh 1.0 g phenolphthalein and dissolve in ethanol. Transfer quantitatively to a 100 cm^3 volumetric flask and make to the mark with ethanol.

4.5 Sugar solution (10%)

Weigh 200 g of refined sugar (first boiling) into a 1 litre beaker. Add 500 cm^3 distilled water and stir until the sugar is dissolved. Transfer the solution quantitatively to a 2 000 cm^3 volumetric flask, mix and make to the mark with distilled water.

5. Procedure

5.1 Standardisation of the hydrochloric acid solution

Fill the burette with the standard 0.1 M sodium hydroxide solution. Pipette 20 cm^3 of the hydrochloric acid solution into a 250 cm^3 conical flasks. Add 3-4 drops of the phenolphthalein indicator and titrate against the sodium hydroxide solution in the burette until the colourless solution turns red. Do the titration in triplicate. Repeat titres should agree to within 0.1 cm^3. Use the average titre to calculate the exact concentration of the hydrochloric acid solution as indicated in 6.1.

5.2 Sample preparation

Sub-sample the lime to ensure a representative sample. Grind about 50 g of the sub-sample using the pestle and mortar and transfer immediately to the sample bottle and close the lid to prevent the uptake of CO_2 or moisture from the air.

5.3 Calcium oxide determination

Measure 750 cm^3 of the sugar solution in the measuring cylinder and transfer to the one 900 cm^3 bottle. This solution will serve as a blank.

Measure another 750 cm^3 of the sugar solution in the measuring cylinder. Weigh 1.5 g of the finely ground lime sample accurately to 0.0001 g and transfer quantitatively to the second 900 cm^3 bottle by using all of the sugar solution in the measuring cylinder.

Agitate the bottles for 4 hours on the tumbler and stand overnight.

Fill the burette with the standardised hydrochloric acid solution. Pipette 50 cm^3 of the solution from each of the 900 cm^3 bottles into 250 cm^3 conical flasks. Add 3-4 drops of phenolphthalein indicator and titrate immediately against the hydrochloric acid solution in the burette until the red solution turns colourless. Do not shake the flask more than is necessary for mixing.

Repeat the titrations. Duplicate titrations should agree to within 0.1 cm^3. Use the average titres to calculate the calcium oxide content in the sample as indicated in 6.2.

6. Calculations

6.1 Hydrochloric acid standardisation

$$\text{HCl concentration (M)} = \frac{C_{NaOH} \times V_{NaOH}}{V_{HCl}}$$

where C_{NaOH} ≡ Concentration of the NaOH solution (M)
V_{NaOH} ≡ Volume of the NaOH titre (cm^3)
V_{HCl} ≡ Volume of the HCl aliquot (cm^3)

6.2 Calcium oxide determination

$$2\ HCl + CaO \rightleftharpoons CaCl_2 + H_2O$$

From the stoichiometric equation 1 cm^3 of the 0.1 M hydrochloric acid solution will react with 0.5 cm^3 of a 0.1 M CaO solution.

$$\text{Calcium oxide (\%)} = \left[\frac{(V_t - V_b)}{2} \times \frac{C_{HCl}}{1000} \times MM_{(CaO)}\right] \div \left[\frac{\text{mass (g)}}{750 cm^3} \times 50 cm^3\right] \times 100$$

$$= \frac{(V_t - V_b) \times C_{HCl} \times MM_{(CaO)}}{\text{mass lime(g)}} \times 0.75 \qquad \text{(Equation 1)}$$

where V_t ≡ Volume of the titre (cm^3)
V_b ≡ Volume of the titre of the blank (cm^3)
C_{HCl} ≡ Concentration of the HCl solution (M or mole/litre)
$MM_{(CaO)}$ ≡ Molecular mass of CaO (g/mole)

7. Example

7.1 Hydrochloric acid standardisation

Mass of NaOH = 4.0231 g
Molecular weight NaOH = 40.0 g/mole

$$\text{Concentration of NaOH} = \frac{4.0231\ g}{40.00\ g/mole} \div 1\ litre$$

= 0.1006 M (or mole/litre)

Volume NaOH titre (cm^3) = 19.8 cm^3

$$\text{HCl concentration (M)} = \frac{0.1006\ M \times 19.8\ cm^3}{20.0\ cm^3}$$

= 0.0996 M (or mole/litre)

7.2 Calcium oxide determination

Volume HCl titre = 16.2 cm^3
Volume blank titre = 0.1 cm^3
Concentration of HCl = 0.0996 M or mole/litre
Molecular weight of CaO = 56.08 g/mole

Mass of lime sample	=	1.5432 g	
Calcium oxide (%)	=	87.41%	(Equation 1)

Report as 87%

8. References

SASTA (1985). *Laboratory Manual for South African Sugar Factories*. 3rd Edition: 361 - 362.