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**Quantitative Analysis of a Mixture of Carbonates
(Inquiry-Based Approach)**

**Teachers’ Notes**

**Some Possible Methods for the Analysis**

**Method 1 (Measure the mass of insoluble CaCO3 residue after filtration)**

Potassium carbonate is soluble in water, while calcium carbonate is insoluble. Weigh a sample of the unknown mixture accurately and stir the sample with water. Filter the suspension and find out the mass of the **residue** collected. It gives the amount of **insoluble calcium carbonate** present in the mixture.

**Method 2A (Precipitation between BaCl2 and filtrate)**

Barium carbonate is insoluble. The amount of **soluble potassium carbonate** present in the sample can be determined by **precipitation**. Weigh a sample of the unknown mixture accurately and stir the sample with water. Filter the suspension to collect the **filtrate**. Add an excess amount of barium chloride solution to the filtrate to precipitate out the carbonate ions. Find out the mass of barium carbonate collected, and use the data to calculate the amount of potassium carbonate present in the mixture.

**Method 2B (Titration between filtrate and standard strong acid)**

The amount of **soluble potassium carbonate** present in the sample can also be determined by **titration**. Weigh a sample of the unknown mixture accurately and stir the sample with water. Filter the suspension to collect the **filtrate**. Titrate the filtrate against a standard strong acid, use methyl orange as the indicator. Find out the volume of acid used to reach the end-point, and use the data to calculate the amount of potassium carbonate present in the mixture.

**Method 3 (Direct Titration)**

The **total** amount of carbonate ions present in the sample can be determined by **direct titration**. Weigh a sample of the unknown mixture accurately, and stir the mixture with water, and titrate the mixture against a standard strong acid with the use of methyl orange as the indicator. It gives the total amount of carbonate ions present in the mixture. The ratio of potassium carbonate and calcium carbonate in the sample can be determined from the experimental data by carrying out the appropriate calculations.

**Method 4 (Back Titration)**

The **total** amount of carbonate ions can also be determined by **back titration**. Weigh a sample of the unknown mixture accurately and stir the mixture with water. Add a known amount of excess strong standard acid to the mixture, and titrate the resulting mixture against a standard alkali with the use of phenolphthalein as the indicator.

*(The experimental technique of back-titration is not required in DSE Chemistry curriculum.)*

*Remarks:*

1. *Other methods, such as to react sample mixture with acids and then measure the volume of CO2 gas produced or the loss of mass in the sample due to the loss of CO2 gas, have been tested and the results obtained are not satisfactory due to various reasons. Therefore these two methods are not recommended.*
2. *For methods 3, the determination of end-point is difficult due to various reasons, such as the poor solubility of calcium carbonate in water. However, the accuracy of this method is comparable with other methods so it is not excluded.*

**Sample Data and Calculations**

Carbonate sample used in the trials: 60% K2CO3 + 40% CaCO3

**Method 1 (Measure the mass of insoluble CaCO3 residue after filtration)**

Mass of unknown sample used (g) = 2.01

Mass of solid residue collected (CaCO3) (g) = 0.72

Mass of K2CO3 in the sample (g) = 2.01 - 0.72 = 1.29

Mass % of K2CO3 = 64%, mass % of CaCO3 = 36%.

**Method 2A (Precipitation between BaCl2 and filtrate)**

Mass of unknown sample used (g) = 2.01

Solution used for precipitating soluble CO32- = 1.0 M BaCl2(aq)

Volume of excess 1.0 M BaCl2(aq) used (cm3) = 50.0

Mass of residue (BaCO3) collected (g) = 1.44

No. of mole of K2CO3 in the sample (mol) = 0.00730

Mass of K2CO3 in the sample (g) = 1.01

Mass of CaCO3 in the sample (g) = 2.01 – 1.01 = 1.00

Mass % of K2CO3 = 50%

Mass % of CaCO3 = 50%

*Notes to Teachers about Method 1 and 2A:*

*1. For convenience, it is advised to weigh the filter paper before carrying out the filtration, and then weigh the total mass of the filter paper and residue after filtration and drying. The mass of residue can be determined from the difference between the two mass readings. Collect the residue from the filter paper and then weigh the residue collected will induce a large experimental error because a lot of residue would stick firmly on the filter paper.*

*2. To increase the accuracy of the results, it is advised to heat the residue on filter paper in an oven at 110oC for at least 30 minutes before weighing, to ensure the residue and the filter paper are dried completely.*

**Method 2B (Titration between filtrate and standard strong acid)**

Mass of unknown sample used (g) = 0.4682

Concentration of the standard HCl(aq) (mol dm-3) = 0.209

Volume of standard HCl(aq) used to reach end-point (cm3) = 18.00

No. of mole of H+ used (mol) = 0.00376



No. of mole of CO32- in the filtrate (mol) = 0.00376 / 2 = 0.00188

Molar mass of K2CO3 (g mol-1) = 138.205

Molar mass of CaCO3 (g mol-1) = 100.0869

Mass of K2CO3 in the sample (g) = 0.00188 $×$ MW(K2CO3) = 0.260

Mass of CaCO3 in the sample (g) = 0.4682 – 0.260 = 0.208

Mass % of K2CO3 = 55%, mass % of CaCO3 = 45%.

**Method 3 (Direct Titration)**

Mass of unknown sample used (g) = 1.5234

Concentration of the standard HCl(aq) (mol dm-3) = 1.034

Volume of standard HCl(aq) used to reach end-point (cm3) = 24.4

No. of mole of H+ used (mol) = 0.0252



No. of mole of CO32- in the sample (mol) = 0.0252 / 2 = 0.0126

Let no. of mole of K2CO3 in the sample = x

 no. of mole of CaCO3 in the sample = y

Molar mass of K2CO3 (g mol-1) = 138.205

Molar mass of CaCO3 (g mol-1) = 100.0869

 (1) x $×$ MW(K2CO3) + y $×$ MW(CaCO3) = 1.5234

 (2) x + y = 0.0126

Solving (1) and (2), x = 0.00688, y = 0.00572

 Mass of K2CO3 in the sample (g) = 0.951

 Mass of CaCO3 in the sample (g) = 0.572

Mass % of K2CO3 = 62%, mass % of CaCO3 = 38%.

**Method 4 (Back Titration)**

Mass of unknown sample used (g) = 0.4265

Concentration of the standard HCl(aq) used (mol dm-3) = 0.209

Volume of excess HCl(aq) used to react the sample (cm3) = 50.00

Concentration of standard NaOH(aq) used for titration (mol dm-3) = 0.200

Volume of standard NaOH(aq) used to react with excess HCl(aq) (cm3) = 17.00

No. of mole of HCl(aq) used to react with CO32- in the sample (mol)

= (0.209 $×$ 50.00 / 1000) – (0.200 $×$ 17.00 / 1000) = 0.00705



No. of mole of CO32- in the sample (mol) = 0.00705 / 2 = 0.00352

Let no. of mole of K2CO3 in the sample = x

 no. of mole of CaCO3 in the sample = y

Molar mass of K2CO3 (g mol-1) = 138.205

Molar mass of CaCO3 (g mol-1) = 100.0869

 (1) x $×$ MW(K2CO3) + y $×$ MW(CaCO3) = 0.4265

 (2) x + y = 0.00352

Solving (1) and (2), give x = 0.0195, y = 0.00157.

 Mass of K2CO3 in the sample (g) = 0.269

 Mass of CaCO3 in the sample (g) = 0.4265 – 0.269 = 0.158

Mass % of K2CO3 = 63%, mass % of CaCO3 = 37%.

*Notes to Teachers about Methods 3 and 4:*

*1. The accuracy of the results obtained from method 3 (direct titration) and method 4 (back titration) were comparable. However, the determination of end-point in back titration of this experiment was easier in our trials.*

*2. The experimental technique of back titration is not required in DSE Chemistry curriculum.*

**Summary of results**

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| Method | Mass % of K2CO3 | Mass % of CaCO3 |
| 1 | 64 | 36 |
| 2A | 50 | 50 |
| 2B | 55 | 45 |
| 3 (Direct Titration) | 62 | 38 |
| 4 (Back Titration) | 63 | 37 |