SEPARATION OF COMPONENTS FROM A TERNARY MIXTURE BY DISSOLUTION AND FILTRATION (1)

Objective

A mixture of solids can be separated for two reasons. One is to obtain the individual substances for further use. The other is to determine its composition as accurately as possible.
The objective of the practice is to quantitatively determine the composition of a ternary mixture containing salt (sodium chloride, NaCl), limestone (calcium carbonate $CaCO_3$), and sand (silicon dioxide or silica, SiO_2) by the difference in their solubility.

Theoretical background

Limestone and sand are insoluble in water, while salt is highly soluble. Therefore the addition of water to the solid sample will result in the dissolution of salt, while limestone and sand remain solid. From the decrease in the mass of the sample, the salt content can be calculated.

Of the two water insoluble substances sand is chemically very resistant. Limestone, on the other hand, decomposes in relatively weak acids. It becomes soluble even in carbonic acid, which forms from dissolved carbon dioxide from the air according to the following equations:

$$H_2O(l) + CO_2(g) = H_2CO_3(aq) \quad (1)$$

$$CaCO_3(s) + H_2CO_3(aq) = Ca^{2+}(aq) + 2HCO_3^-(aq) \quad (2)$$

The reaction above should be avoided, because if water with dissolved carbon dioxide is used for washing, it will continuously reduce the amount of limestone in the solid residue, hence erroneously leading to an increase in the amount of dissolved salt. Therefore in the practice we have to use water with the smallest amount dissolved carbon dioxide, i.e., we have to use boiling distilled water.

Since limestone is very sensitive to acids, it can be used to determine the amount of chemically resistant sand in the mixture, because the addition of acid solution, e.g. hydrochloric acid, to the solid results in the dissolution of both salt and limestone. The chemical dissolution takes place according to the following reaction:

$$CaCO_3(s) + 2H_3O^+(aq) = Ca^{2+}(aq) + 3H_2O(l) + CO_2(g) \quad (3)$$

From the mass of the remaining solid, the sand content can be calculated. If we know the content of two substances (salt and sand) in a ternary mixture, the third can be calculated.

Materials

- *beaker of 100 cm^3*
- *beaker of 250 cm^3 (2 pieces)*
- *glass funnel (2 pieces)*
- *glass rod (2 pieces)*
- *wide-neck Erlenmeyer flask of 250 cm^3 (2 pieces)*
- *wash bottle*
- *filter paper (2 pieces)*
- *scissors*
- *porcelain dish (2 pieces)*
- *watch glass*
- *metal tripod stand*
- *wire gauze*
- *Bunsen stand*
- *funnel holder (2 pieces)*

Procedure

Preparation of hot water

- Put the wire gauze on the tripod stand.
- Fill the clean beaker of 250 cm^3 with distilled water to about 1/3 of its capacity, then place it on the wire gauze and start to heat it.
- While waiting for the water to boil, continue with the practice.

Safety: Make sure that water is always in the beaker, if necessary add more. Make sure that the cold water is not spilled on the hot glass leading to the breakage of the glassware and spilling of the hot water.

Sample preparation for chemical dissolution by HCl

- Pick up your unknown sample. If the two dissolution is performed in a single practice, split the unknown into two (one sample to the dissolution by HCl, one to that by hot water), otherwise use the whole sample.
- Place the dry and clean beaker of 100 cm^3 volume on a top-loading balance and tare it.
- Put your unknown (sample) into the beaker of 100 cm^3 volume, and measure the mass of the sample and record it in your record sheet (**I.**).

Sample preparation for dissolution by hot water

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- Put your unknown (sample) into the beaker of 250 cm^3 volume, and measure the mass of the sample and record it in your record sheet (**II.**).

Chemical dissolution by HCl

- Place your beaker of 100 cm^3 volume in the fume hood.
- Add the dilute hydrochloric acid slowly, dropwise with a glass rod as shown in Fig. 1, because carbon dioxide forms in the reaction giving rise to strong bubble formation.
- Stir thoroughly after each addition. The reaction is complete when the solution has cleared up and when upon the addition of a further portion of hydrochloric acid no effervescence is observed.

Safety: Special care should be taken when handling hydrochloric acid.

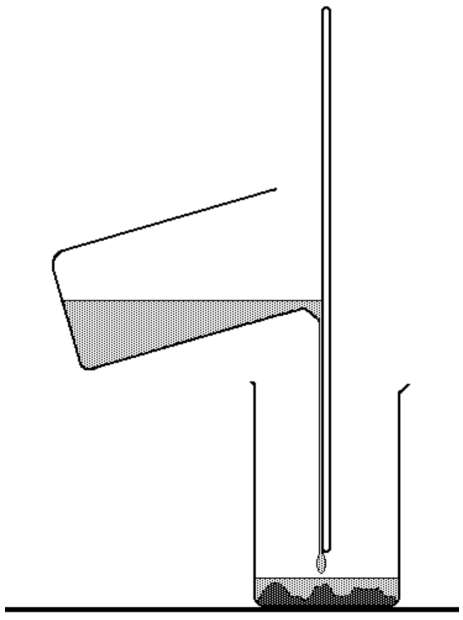

Figure 1: Apparatus to be used in chemical dissolution by HCl.

Dissolution by hot water

- Place your beaker of 250 cm^3 volume on your desk. Add 40-50 cm^3 boiling distilled water using your washcloths carefully and slowly with a glass rod as shown in Fig. 1. Stir thoroughly.
- Wait until the insoluble calcium carbonate sediments. The dissolution of salt is complete if there is no chloride ion in the filtrate which should be tested during decantation (see below).

Safety: Do not touch with your hand the hot glass, use washcloths when handle the hot beaker.

Assembling the filtration apparatus

Two filtration equipments are needed for the two samples, one for each.

Filtration apparatus

Fix the funnel holder on the Bunsen stand in a height that the stem of the funnel when placed in the holder touches the mouth of the Erlenmeyer flask from inside as illustrated in Fig. 2. The latter is used to keep the filtrate. A plain filter paper is used for filtration.

Preparation of plain filter paper

- Pick up a square filter paper from the main desk.
- Fold it into four and cut off a quarter circle.
- Unfold one layer to form a cone (the other side will be three-layered).
- Fit this cone of paper in the dry funnel, push the tip into the neck and make sure the edge of the cone is under the edge of the funnel with ca. 5-10 mm (cut it further if required).
- Measure the mass of the filter paper on a top-loading balance. Record it in your record sheet (for chemical dissolution by HCl (**III.**), whereas for dissolution by hot water (**IV.**)).
- For effective filtering, the paper has to fit to the wall of the funnel. Wet the paper a little with the wash bottle, and gently press to the wall (perhaps adjust the folding a little). *Tips: Make sure do not puncture the filter paper.*

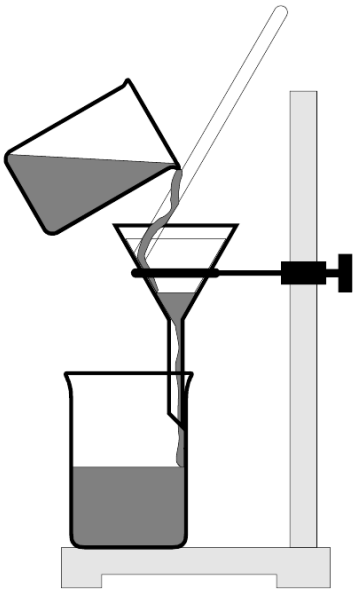

Figure 2: Scheme of the filtration equipment.

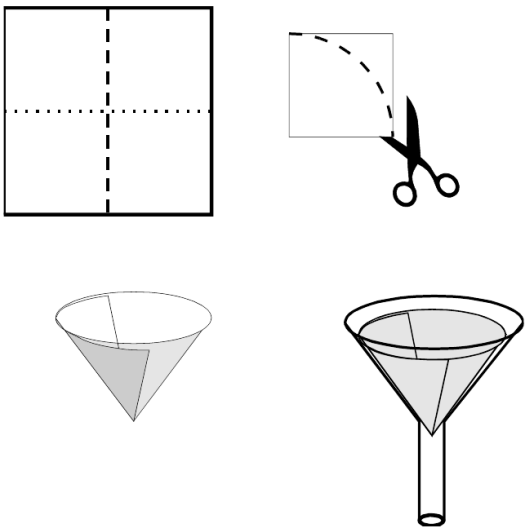

Figure 3: Cutting of the filter paper.

Decantation and filtration of the samples

- Add $\sim$100 cm^3 distilled water from the wash bottle to the beaker of 250 cm^3 volume to dilute the acid (and washing the inner side of the beaker) for the sample used in chemical dissolution by HCl and add only 30-40 cm^3 boiling distilled water for the sample used in dissolution by hot water. Stir the solution. *Tips: The stirring rod should be kept on the top of the beaker to eliminate any solid particle loss by placing it on the desk. Make sure to use the same stirring rod during the entire process for each sample.*

- Let the solid sediment in the beaker.
- Decantate the solution along a glass stick onto the filter paper, that is pour the solution along a glass rod onto the filter while keeping the solid in the beaker. *Tips: Be careful, not to pour over the edge of the filter paper, since it would be a waste from the solid.*
- Test for chloride ions (see below). If the test is positive repeat the procedure above using smaller amount of water than in the first step. If the test is negative the solid material can be filtered (see below). The first test should be done after the third washing. *Safety: Special care should be taken on washing with the boiling distilled water. Tips: Do not forget to add more water from time to time for boiling.*

Chloride test

- Collect few drops of the filtrate in a watch glass as depicted in Fig. 4.
- Add few drops of silver nitrate ($AgNO_3$) solution of 0.1 mol/dm^3 concentration to it. If white precipitate forms, the filtrate contains too much chloride because AgCl precipitate is formed according to

$$Ag^+(aq) + Cl^-(aq) = AgCl(s) \quad . \qquad (4)$$

  If the solution is opalescent, the filtrate is practically chloride free.

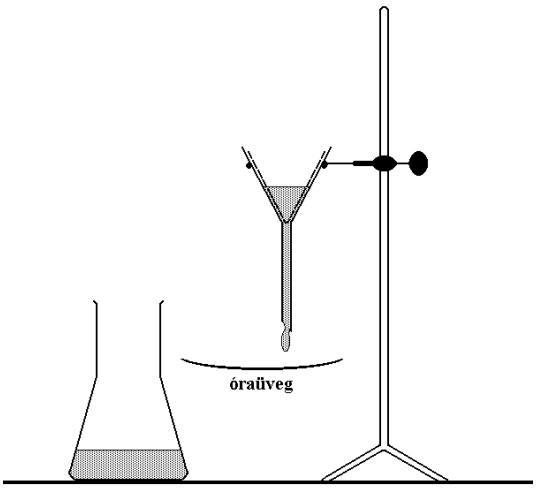

Figure 4: Setup for the chloride test.

Tips: The stem of the funnel should always touch the inner side at the neck of the Erlenmeyer flask because the weight of the liquid column accelerates filtration. More attention should be taken on handling the dissolution sample with hot water as the sedimentation of limestone is a much slower process. The filtrate from the Erlenmeyer flask should be emptied to the basin from time to time because we do not need it.

Filtration

- If the chloride test is negative, i.e., the filtrate is chloride ion free, filter all the solid from the beaker. With the last water portions, transfer all the solid onto the filter using the glass rod. Repeat it until all the solid material from the beaker and from the glass rod is transferred onto the filter paper. *Tips: Be careful, not to pour over the edge of the filter paper, since it would be a waste from the solid.*
- Wait until all the water flows through the funnel.

Drying of the samples

- Take out the filter papers with the solid left on them.
- Put them into separate porcelain dishes marked with your name.
- Let them air-dry in a drying cabinet shown by the instructor. The samples must be dried to a constant weight.
- After an appropriate drying time (1 h), weigh the sample on the top-loading balance. Record the mass of the filter paper and the sample (for chemical dissolution by HCl (**V.**), whereas for dissolution by hot water (**VI.**)).
- Return the sample to the drying cabinet for a further 15 to 20 minutes, or wait until the following week and then re-weigh it at room temperature. If the loss in mass is less than 0.01–0.02 g, the sample is dry.

Calculations and finishing

- When the sample is dry, measure the mass of the dried sample and the filter paper (for chemical dissolution by HCl (**V.**), whereas for dissolution by hot water (**VI.**)).
- Calculate the composition of the ternary mixture according to the sample calculations.
- Fold a paper capsule. On the capsule, write your number, your name, the name of the substance and its chemical formula along with its mass.
- Wrap the solid sample with it, and turn it in.

Sample calculations

Evaluation of chemical dissolution by HCl:

I.	Mass of ternary mixture (sand+limestone+salt):	$m_m = 5.83$ g
III.	Mass of the smaller filter paper:	$m_{fp} = 1.51$ g
V.	Mass of the smaller filter paper + sand:	$m_{fp,s} = 3.60$ g
VII.	Mass of sand:	$m_{fp,s} - m_{fp} = 2.09$ g
	Sand content of the mixture:	$m_s/m_m \cdot 100\ \% = 35.8\%$

Evaluation of dissolution by hot water:

II.	Mass of ternary mixture (sand+limestone+salt):	$m_m = 5.13$ g
IV.	Mass of the bigger filter paper:	$m_{fp} = 2.74$ g
VI.	Mass of the bigger filter paper+sand+limestone:	$m_{fp,s,l} = 5.89$ g
VIII.	Mass of sand and limestone:	$m_{s,l} = m_{fp,s,l} - m_{fp} = 3.15$ g
IX.	Mass of dissolved salt:	$m_{salt} = m_m - m_{s,l} = 1.98$ g
	salt content of the mixture:	$m_{salt}/m_m \cdot 100\ \% = 38.6\ \%$

The limestone content can be calculated from the contents of sand and salt:
100 % - 35.8 % - 38.6 % = 25.6 %