DETERMINATION OF THE PERCENT COMPOSITION OF A MIXTURE

In this experiment you are to determine the composition of a mixture containing unknown proportions of barium chloride dihydrate (BaCl₂•2H₂O) and anhydrous barium chloride (BaCl₂). To do this, you will take the somewhat indirect route of determining the amount of BaCl₂•2H₂O present by measuring the amount of water present and assuming (reasonably enough) that it all comes from the dihydrate. Water content is determined by measuring the weight loss (lost water vapor) when the sample is heated and converted entirely to anhydrous barium chloride. The barium percentage in the sample is determined independently by isolating the barium as solid BaSO₄, weighing the dry solid, and calculating the mass of barium it contains. To precipitate the BaSO₄, you dissolve the sample in water, add an excess of sulfate ion (SO₄²⁻) in the form of sulfuric acid (H₂SO₄) to form insoluble BaSO₄,

Complete equation:

\[
\text{BaCl}_2(\text{aq}) + \text{H}_2\text{SO}_4(\text{aq}) \rightarrow \text{BaSO}_4(\text{s}) + 2 \text{HCl}(\text{aq})
\]

Net ionic equation:

\[
\text{Ba}^{2+}(\text{aq}) + \text{SO}_4^{2-}(\text{aq}) \rightarrow \text{BaSO}_4(\text{s})
\]

which is then filtered off, washed, dried, and weighed.

EXPERIMENTAL PROCEDURE

1. Preparation of the Crucible and Sample Weighing

Thoroughly clean and dry your porcelain crucible and its cover. To ensure dryness, place the crucible and its cover on the triangle mounted on a ring stand (see Figure) and heat thoroughly with a burner. Let this assembly cool completely to room temperature, and then weigh the crucible and cover together on the analytical balance; record this mass on your report form.

2. Obtain an unknown from your instructor and record the unknown number on the report form. To determine the water and barium content of this unknown, you should use

Barium sulfate is used in radiological studies of the digestive tract. For more information, and some World Wide Web URLs, see the last page. If you are reading this online, click here for the MSDS sheet. The URL is http://www.jtbaker.com/msds/b0504.htm

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Your unknown should have an accurately known mass somewhere around 2 grams. For example, your unknown could have a mass of 2.096 g or 1.850 g.

a sample that has a mass of about 2 grams.

Use the following procedure to obtain an accurate mass: Place a piece of weighing paper on the balance and note its approximate mass. Using your spatula, transfer about 2 grams of unknown sample to the paper. Now weigh the sample and paper accurately and record the mass on your report form. Transfer the sample carefully to the crucible, and then weigh the paper, again to the nearest 0.001 g; again, be sure to record the mass on your report form.

2. Determination of Water

a) Place the crucible with the sample in the triangle mounted on a ringstand.
b) Adjust the height of the ring so that the bottom of the crucible is only a few centimeters above the top of the burner barrel. Displace the cover carefully slightly to one side to allow water vapor to escape.
c) Light the burner and, holding the burner so that the flame just brushes the bottom of the crucible, heat the crucible gently for 5 to 10 minutes.
d) After the initial heating, place the burner directly under the crucible and heat until the bottom of the crucible glows a cherry red. Heat in this manner for at least 5-6 minutes, then remove the flame and allow the assembly to cool to room temperature.
e) When the crucible is no longer warm to the touch, put the top on the crucible and determine the mass of crucible, cover, and dehydrated sample.

3. Determination of Barium

a) Carefully wash a 250 mL beaker and rinse it with distilled water. Use distilled water from your wash bottle to rinse off the bottom of the outside of the crucible containing the unknown sample. Do not dry the crucible, but place crucible, cover, and contents in the beaker.
b) Add distilled water until the crucible, lying on its side with the cover off, is just covered. Stir with a clean glass stirring rod until the sample is dissolved.
c) Carefully clean and rinse the tips of your crucible tongs and use the tongs to remove the crucible and cover from the beaker. Be sure to rinse any adhering solution back into the beaker with water from your wash bottle.
d) Add 1 mL of concentrated hydrochloric acid (HCl) and then dilute the solution to a total volume of 150 ml. (If the solution volume is near 150 mL or even over that amount, simply continue without adding any water.)
e) Bring the solution to near boiling.
f) Measure 8 mL of 3 M H$_2$SO$_4$ (sulfuric acid) in your graduated cylinder and then add the acid rapidly, with stirring, to the hot solution. A white precipitate of BaSO$_4$ will form.
g) Cover the solution with a watch glass and simmer for about 10 minutes. After about 10 minutes, remove the burner and allow the precipitate to settle to the bottom.
h) While the precipitate is settling, fold a piece of filter paper (as shown in
the figure on the next page) and weigh the cone to the nearest 0.001 g. (NOTE: If you are going to do more than one barium determination, be sure to label the pieces of filter paper so you know which paper is for the first determination and which is for the second.)

i) Mount the weighed filter cone in the filter funnel and moisten with a small amount of distilled water so that the paper adheres to the wall. Place a clean 600 mL beaker under the funnel (which should be placed in a ring on a ringstand) and begin to filter off the liquid.

j) Add the liquid in small amounts using the pouring technique shown in the figure on the next page. Try not to disturb the solid until about 2/3 of the liquid has been removed. If the filtrate is perfectly clear, discard it and replace the beaker under the funnel. If the liquid is cloudy, place another clean beaker under the funnel and pour the filtrate through the filter setup again. Discard any clear liquid you obtain. Now stir the remaining precipitate-liquid mixture and pour this suspension into the filter paper. Do not let the liquid level rise any more than within 1 cm of the top of the filter paper. If the new filtrate is cloudy, re-filter as described above.

k) Rinse any adhering portions of solution and precipitate from the beaker into the filter paper using distilled from your wash bottle. Be sure to rinse all the precipitate out of the beaker onto the filter. (Any solid left in the beaker will not be weighed later and will introduce an error into your analysis.)

l) After this rinsing is complete, wash the precipitate in the paper three times with distilled water, using about 10 mL portions each time. Follow this washing with five more washes using 10 mL portions of acetone.

m) Carefully remove the damp filter cone from the filter funnel and place it in a dry, clean beaker or in your evaporating dish; store it in your desk to dry until the next laboratory period.

n) When the BaSO₄ precipitate is dry (this usually takes until your next laboratory period), weigh the filter paper containing the BaSO₄ to the nearest 0.001 g. Record the mass of the paper and BaSO₄ on your report form.


The directions above describe the complete analysis for water of hydration and barium of one, 2 g sample of unknown. To ensure the greatest possible accuracy, you should make every attempt to complete the analysis of two samples of your unknown; the final result will then be the average of these two determinations. If you organized your work properly, you should have enough time to do this second analysis.
CALCULATIONS

1. Determination of Water Content.
   The mass lost by the sample on heating is simply the mass of water in the sample. Thus, the mass percent of water is
   
   \[
   \text{Mass percent of water} = \frac{\text{mass of water in the sample (g)}}{\text{mass of unknown sample (g)}} \times 100\%
   \]

2. Determination of Barium Content.
   The mass of barium (Ba) in your unknown sample, and then the mass percent, can be calculated from the mass of BaSO₄ that you isolated. In the example below, let us say you have isolated 1.568 g of BaSO₄ from a 2.016 g sample of unknown.
   
   \[
   1.568 \text{ g BaSO}_4 \cdot \frac{1 \text{ mol BaSO}_4}{233.4 \text{ g}} = 6.718 \times 10^{-3} \text{ mol BaSO}_4
   \]
   
   \[
   6.718 \times 10^{-3} \text{ mol BaSO}_4 \cdot \frac{1 \text{ mol Ba}}{1 \text{ mol BaSO}_4} = 6.718 \times 10^{-3} \text{ mol Ba}
   \]
   
   \[
   6.718 \times 10^{-3} \text{ mol Ba} \cdot \frac{137.4 \text{ g Ba}}{1 \text{ mol Ba}} = 0.9231 \text{ g Ba}
   \]
   
   \[
   \text{Mass percent Ba} = \frac{0.9231 \text{ g Ba}}{2.016 \text{ g unknown sample}} \times 100\% = 45.79\% \text{ Ba}
   \]

NOTE: As done above, your answer should be carried out to 4 significant figures.

Information on Barium Sulfate

1. MSDS sheet: http://www.jtbaker.com/msds/b0504.htm
2. Barite is the mineral form of barium sulfate. For more information on this important mineral go to http://mineral.galleries.com/minerals/sulfates/barite/barite.htm.
3. The homepage for the “largest producer of barium sulfate in the world:” http://www.cimbar.com/index.html
REPORT FOR
PERCENT COMPOSITION OF A MIXTURE

Name _________________________________________Section _______ Grade ________ /10
Unknown Number ________________
Comments _____________________________________________________________________

<table>
<thead>
<tr>
<th>First Determination</th>
<th>Second Determination</th>
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Mass of Unknown

1. Mass of paper + sample
2. Mass of paper
3. Mass of sample (1 - 2)

Determination of Mass Percent of Water

Experimental data

4. Mass of crucible + cover +
   dehydrated sample
5. Mass of crucible + cover
6. Mass of dehydrated sample (4 - 5)

Calculation

7. Mass of water in sample
8. Mass percent of water
9. Average percent of water

Determination of Barium

Experimental data

10. Mass of filter paper + BaSO₄
11. Mass of filter paper
12. Mass of BaSO₄ (10 - 11)

Calculation

13. Moles of BaSO₄
14. Moles of Ba
15. Mass of Ba
16. Mass percent of Ba
17. Average mass percent of Ba

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Composition of the Original Sample
Your unknown sample was a mixture of barium chloride dihydrate (BaCl$_2$•2 H$_2$O) and anhydrous barium chloride (BaCl$_2$). Use the mass of water lost in the first determination above (line 7) to determine the quantity of BaCl$_2$•2 H$_2$O in that sample of your unknown. Outline your calculations below.

Knowing the number of grams of BaCl$_2$•2 H$_2$O that were in your unknown sample, calculate the mass percent of this compound in the unknown. Outline the calculation below.