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Indirect Gravimetric Determination of Waters of Hydration

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Abstract: An alternate gravimetric experiment is described that can be employed in the general chemistry or the quantitative analysis laboratory course. The procedure takes less time than conventional methods and introduces students to waters of hydration and indirect determinations.

Introduction

Gravimetric analysis is taught in quantitative analytical chemistry laboratories, but the time allotted for such experiments is becoming less. Thus, rather than analyzing in triplicate the nickel oxide in a nickel ore by nickel dimethylglyoxime precipitation [1] over two laboratory periods, the analysis of the waters of hydration in aluminum sulfate has been substituted. This experiment is completed in one laboratory period and a microwave oven can be used to speed up the drying process [1,2]. The rate-limiting step in the previous analyses was the filtration through medium porosity crucibles. Now, a higher flow rate is achieved using glass vacuum filters with 0.45 micron filter paper. Precision and accuracy, while desirable, are no longer graded. Instead, stoichiometry and applying percent yield to the subsequent analysis is emphasized. Thus, this experiment could be employed in the General Chemistry laboratory course as well.

The determination of the waters of hydration for aluminum sulfate can be determined by the precipitation of barium sulfate from mixed solutions of aluminum sulfate and barium chloride. Barium sulfate is insoluble and can be collected by filtration. However, the yield may not be 100% and thus the percent yield is first determined when doing the analysis. Also, a sample with a known amount of waters of hydration is employed to determine the percent accuracy. Thus, sodium sulfate anhydrous, aluminum potassium sulfate with 12 waters of hydration and aluminum sulfate with 12 to 18 waters of hydration are analyzed. The aluminum sulfate sample is considered the unknown with respect to the number of waters of hydration.

The three reactions expected are as follows:

$$Na_2SO_4 + BaCl_2 \rightarrow 2NaCl + BaSO_4\ (s) \qquad 1$$

$$AlK(SO_4)_2 \cdot 12H_2O + 2BaCl_2 \rightarrow AlCl_3 + KCl + 2BaSO_4\ (s) + 12H_2O \qquad 2$$

$$Al_2(SO_4)_3 \cdot (12\text{-}18)H_2O + 3BaCl_2 \rightarrow 2AlCl_3 + 3BaSO_4\ (s) + (12\text{-}18)H_2O \qquad 3$$

Following the stoichiometry and with known measured weights of Na_2SO_4 and $AlK(SO_4)_2 \cdot 12H_2O$, the measured weight of the resulting dry $BaSO_4$ (s) can be used to determine the percent yield as well as the percent accuracy of the twelve waters of hydration. The percent yield of $BaSO_4$ and percent accuracy can be used for the third analysis: $Al_2(SO_4)_3 \cdot (12\text{-}18)H_2O$ and thus a predicted value of the waters of hydration can be determined.

Experimental

Students work in pairs and a number of glass vacuum filtration apparatuses are shared among many groups. This is typically the first experiment in the quantitative analytical chemistry laboratory course. This experiment can be completed in a typical 3-4 hour laboratory period. The chemicals needed are as follows: Na_2SO_4, $BaCl_2 \cdot 2H_2O$, $AlK(SO_4)_2 \cdot 12H_2O$, and $Al_2(SO_4)_3 \cdot (12\text{-}18)H_2O$.

Student Procedure. Label four small beakers and four small weigh boats with the four chemical names. Weigh out individually to four decimal places into individual weigh boats between 0.1 and 0.2 g of Na_2SO_4, $AlK(SO_4)_2 \cdot 12H_2O$, and $Al_2(SO_4)_3 \cdot (12\text{-}18)H_2O$ via weighing by difference. Weigh out in the fourth weigh boat between 1.1 and 1.2 g of $BaCl_2 \cdot 2H_2O$. Transfer the solids quantitatively into their respective beakers with deionized water. Swirl continuously until dissolved. Adding water up to the 40 mL mark is acceptable. Ensure that all solids are dissolved.

Transfer the $BaCl_2$ solution equally into the three 10 or 25 mL graduated cylinders (the volume can be more than 10 mL). Thus, there are three graduated cylinders and three remaining beakers. Pour from one graduated cylinder into one beaker and repeat until all three beakers have white precipitate formed. Swirl. Wait five minutes.

Label the three watch glasses (using a marker not tape) with Na_2SO_4, $AlK(SO_4)_2 \cdot 12H_2O$, and $Al_2(SO_4)_3 \cdot (12\text{-}18)H_2O$, (using one name per watch glass). Place a filter on a watch glass and record the weight and repeat for each watch glass respectively using a new filter each time.

After being trained on the use of the filtration apparatus, filter the three solutions in sequence. Using a spatula, carefully remove the filter and place it on its respective watch glass. Place all three watch glasses in the oven to dry for 30 minutes or use a microwave oven in cycles to dry the precipitates. Remove the three watch glasses and allow them to cool to room temperature. Record the weights. Determine the weight of the precipitate. Calculate the % yield of $BaSO_4$ for the Na_2SO_4 sample and the % accuracy for the waters of hydration for the $AlK(SO_4)_2 \cdot 12H_2O$ sample, and the experimentally determined waters of hydration for the third sample. Consider the percent yield and/or accuracy from your first two analyses in determining the waters of hydration for the third sample. Determine any sources of error.

Hazards. Safety glasses and gloves should be worn. All solutions and the isolated precipitates should be discarded according to state and local laws. The chemicals and procedures used in this laboratory are much less hazardous than the previously used gravimetric methods [1].

Results and Discussion

This one experiment introduces the students to indirect determinations, waters of hydration, the calculation of the correct molecular mass and stoichiometry. Their exposure to hazardous and dangerous chemicals is lessened and the time to do the experiment is dramatically shortened. In addition, the students learn to use a new piece of equipment. Losses of the solid barium sulfate do occur on the edges of the glass filtration apparatus as students are unable to transfer it all to the watch glass. Those who do not place their filter paper directly in the center such that it covers all of the fritted glass find their product passing through and these students must repeat the experiment. Some students use the blue paper which separates each filter paper in the container as the filter paper and quickly realize that that paper does not filter at all. Most students do achieve close to 100 % yield. Those with much less than 100% yield, typically did not wait for their solids to dissolve, did not weigh by difference correctly, did not weigh the correct amount, or did not transfer their product quantitatively. The challenge is no longer the time to wait for the filtration but the calculations themselves (see Supporting Material). While the use of the microwave oven can shorten the time to wait, that time can be used to ensure that the students have the calculations worked out before they obtain the data necessary to complete the experiment. The students are not allowed to leave until they have completed their calculations and found the percent yield, accuracy and waters of hydration. They can also be asked to determine if the barium chloride was added in excess. The students are required to use an equation writing program such as Microsoft Equation Editor 3.0 to show all of their calculations in their laboratory report.

Conclusion

This experiment utilizes the calculations of molecular weight, percent yield and stoichiometry in an indirect determination of waters of hydration.

Supporting Materials. The materials needed and sample calculations are provided (http://dx.doi.org/10.1333/s00897092223a).

References and Notes

1. Carmosini, N.; Ghoreshy, S.; Koether, M.C. *J. Chem. Educ.* **1997**, *74*, 986–987.
2. Thompson, R.Q.; Ghadiali, M. *J. Chem. Educ.* **1993**, *70*, 170–171.