Microwave-Assisted Dehydration of Aqua Complexes

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Abstract: A microwave oven is used for the gravimetric analysis of several transition metal complexes, in particular, the dehydration of Fe, Ni, Co, Cu and Zn aqua complexes. This is a simple, convenient, and colorful experiment using transition metal aqua complexes. The procedure is designed to help chemistry students understand the concept of coordinated water.

Introduction

Microwave ovens have been used extensively in a variety of applications [1–5], including the rapid synthesis of organic compounds. The microwave oven not only produced results comparable to those of conventional methods, but also decreased the overall time required [6]. Recently, aspirin and acetanilide were synthesized using a microwave oven in 5 min [7], and Ni ore was dried in a microwave oven at medium power [8]. We reported that the blue complex $CuSO_4•5H_2O$ yielded a white complex $CuSO_4•H_2O$ when dehydrated using a microwave oven [9]; however, few experiments of gravimetric analysis using a microwave oven have been described [8]. Here, we apply a microwave oven for the gravimetric analysis of the transition metal complexes. In particular, this report discusses the dehydration of Fe, Ni, Co, Cu and Zn aqua complexes with a microwave oven. Here, we report simple, convenient, and colorful experiments using transition metal aqua complexes. The procedure is designed to help chemistry students understand the concept of coordinated water.

Experimental

Equipment and Chemicals. This procedure requires the following equipment: microwave oven, balance, beakers, and crucibles. The following chemicals are used: $FeSO_4•7H_2O$, $CoSO_4•7H_2O$, $Co(NO_3)_2•6H_2O$, $CoCl_2•6H_2O$, $NiSO_4•6H_2O$, $Ni(NO_3)_2•6H_2O$, $CuSO_4•5H_2O$, $CuCl_2•2H_2O$, $Cu(NO_3)_2•6H_2O$, and $ZnSO_4•7H_2O$.

Procedure. A typical procedure for $Ni(NO_3)_2•6H_2O$, $NiSO_4•6H_2O$, $CoSO_4•7H_2O$, $CuCl_2•2H_2O$, $ZnSO_4•7H_2O$, and $CoCl_2•6H_2O$ is as follows. Measure the weight of the beaker. Next, measure the weight of the same beaker containing ca. 2.90 g of $Ni(NO_3)_2•6H_2O$. Place the beaker in the microwave oven and irradiate it at medium power for 5 min. Observe the change of $Ni(NO_3)_2•6H_2O$ to its liquid state. Once the beaker has cooled to room temperature, measure the weight of the beaker. Note that the solid has turned light green. Next, pour 10 mL of water onto the solid and observe the change. Repeat the same procedure for all of the above complexes, except $NiSO_4•6H_2O$. Irradiate the $NiSO_4•6H_2O$ complex at high power for 15 min because it is difficult to dehydrate.

The other aqua complexes, $Co(NO_3)_2•6H_2O$ and $Cu(NO_3)_2•3H_2O$ should also be subjected to a procedure similar to that for $Ni(NO_3)_2•6H_2O$. Measure the weight of the crucible. Next, measure the weight of the beaker that contains ca. 2.92 g of $Co(NO_3)_2•6H_2O$. Put the crucible in the microwave oven and irradiate it at medium power for 1 min. Observe the change of the red complex, $Co(NO_3)_2•6H_2O$, to its black form. Once the crucible has cooled to room temperature, measure the weight of the crucible again. Observe that the solid has turned black, and then pour 10 mL of water onto the solid. Repeat the same procedure for the other complexes.

Hazards. The nitrate complexes, except $Ni(NO_3)_2•6H_2O$, change to oxides. The nitrate complexes produce heat; therefore, a crucible should be used. $Cu(NO_3)_2•6H_2O$ produces nitric acid when heated; therefore, adequate ventilation should be used. No more than the amount of the nitrate complexes specified in Table 1 should be dehydrated at any one time.

Results and Discussion

$Ni(NO_3)_2•6H_2O$, when heated, becomes $Ni(NO_3)_2•4H_2O$ at 54 °C and $Ni(NO_3)_2•2H_2O$ at 85 °C. Table 1 shows the relationship between the weight change of the aqua complex and the number of coordinated water molecules that is expected from the calculated values. $Ni(NO_3)_2•6H_2O$ releases 4.2 water molecules when irradiated by the microwave oven for 5 min and becomes a complex with two molecules. As soon as the water is added to the slightly green Ni complex, it returned to its green form (Table 1).

$CuCl_2•2H_2O$ released 2.0 water molecules when irradiated in the microwave oven for 10 min and becomes a brown powder. Once water is added to the powdery-brown Cu complex, its well-known blue solution appears immediately.

$CoCl_2•6H_2O$ also loses 6.0 water molecules after 15 min of irradiation in the microwave oven, and it becomes a light-blue precipitate. Once water is added to the light-blue Co complex, a red solution appears at once. From the weight and color change of the complexes, we can suggest that $CuCl_2•2H_2O$ and $CoCl_2•6H_2O$ has changed to $CuCl_2$ and $CoCl_2$, respectively.

$Co(NO_3)_2•6H_2O$ loses 5.3 water molecules after 1 min of irradiation in the oven and becomes a black precipitate after 5 min of irradiation (Table 1). When water is added to the black precipitate, no red precipitate is obtained. This indicates that $Co(NO_3)_2•6H_2O$ has changed to cobalt mixed oxide.

Conclusion

The water molecules coordinated to the complexes are stable, however, by using microwaves from an electronic oven, the water molecules were heated and activated vigorously, then the temperature of the aqua complexes rose and released water molecules easily. The water associated with the chloride complexes is apt to be lost and the complexes eventually turn

Table 1. Weight in Grams of Aqua Complexes and the Number of Coordinated Water Molecules.[a]

Complex	Before irradiation	1 min	5 min	10 min	15 min	Calculated values and color
$FeSO_4•7H_2O$[b] Light green	3.160 (7.00)	3.150 (6.96)	2.176 (2.20)	1.998 (1.34)	1.960 (1.15)	1.930[c] (1.00) Brown
$CoSO_4•7H_2O$ Orange	2.81 (7.00)	2.80 (7.00)	1.854 (1.27)	1.760 (1.17)	1.730 (1.00)	1.730[c] (1.00) Light red
$Co(NO_3)_2•6H_2O$ Red	2.919 (6.00)	1.963 (0.69)	0.759	0.759	0.759	0.754[d] Black
$CoCl_2•6H_2O$ Red	2.380 (6.00)	1.550 (1.39)	1.418 (0.66)	1.379 (0.44)	1.290 (0.00)	1.300[e] Light Blue
$NiSO_4•6H_2O$[f] Green	2.63 (6.00)	2.61 (5.94)	1.99 (2.50)	1.688 (0.14)	1.688 (0.14)	1.540[d] (0.00) Yellow
$Ni(NO_3)_2•6H_2O$ Green	2.900 (6.00)	2.141 (1.80)	1.688	1.680	1.541	2.180[g] (2.00) Light green
$CuSO_4•5H_2O$[b] Blue	0.624 (5.00)	0.618 (4.87)	0.515 (2.58)	0.451 (1.15)	0.444 (1.00)	0.444[c] (1.00) White
$CuCl_2•2H_2O$ Green	1.710 (2.00)	1.698 (1.93)	1.564 (1.19)	1.346 (0.00)	1.345 (0.00)	1.350[e] (0.00) Brown
$Cu(NO_3)_2•3H_2O$ Blue	1.210 (3.00)	0.469	0.346	0.346	0.346	0.400[d] Black
$ZnSO_4•7H_2O$ White	2.875 (7.00)	2.862 (6.94)	1.647 (1.02)	1.644 (1.02)	1.616 (1.00)	1.615[c] (1.00) White

[a] The figures in parenthesis are the coordinated number of water molecules calculated from the weight change of the complexes. [b] Reference 6. [c] Calculated values for the complex with one water molecule. [d] Calculated values for oxide. [e] Calculated values for anhydride. [f] Irradiated at high power. [g] Calculated values for the complex with two water molecules.

to the anhydrides and nitrate complexes, when heated in a microwave oven, become the metal oxide.

In this experiment, a microwave oven is applied in the analysis of various complexes. Students can try various times in the microwave oven and can add water to the residue to see what changes occur. This encourages students to question the meaning of coordinated water and to consider the various metal–water complexes that can form. Also, this experiment is useful as an introduction to gravimetric analysis of aqua complexes. The experiment described here is appropriate for a high school junior-level laboratory.

References and Notes

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