

Synthesis and Identification of a Cobalt-Complex

Law, Lauren

Synthesis Procedure III

Dr. George Kennedy

Principles of Chemistry II Laboratory (CHEM 1212L; F 8:00 AM)

22 April 2016

Introduction

The purpose for this experiment was to create and identify an unknown cobaltous halide compound. The resulting solid from the formation of synthesis procedure III (using 5.0037g cobalt carbonate, 20 mL of 9M HBr, 2.0003g of charcoal, 25 mL of aqueous ammonia, along with other things), was a beautiful 3.1254 g, orange powder-like substance. In order to analyze this soft orange solid, many titrations, distillations, and filtrations were succeeded. According to all of the experiments performed, the data yielded the resulting compound to be $[\text{Co}(\text{NH}_3)_6]\text{Br}_3$. The initial percent yield was 59.00% Br.

Analysis for % Halide

By producing a mixture of 5.0037g CoCO_3 with 20 mL of 9M HBr and 15 mL of H_2O , then filtrating and adding 2.0003 g of charcoal, 25 mL of ammonia, and 6 mL of 30% H_2O_2 (3-4 drops at a time), I collected a dark solid. I was able to isolate the halide by first purifying the collected solid and then determining the % halide by calculating the mass of the precipitate times the mass of Bromine, divided by the mass of Cobalt sample (for each sample). The higher value I calculated (sample B) was 59% bromide and lined up best with that of $[\text{Co}(\text{NH}_3)_6]\text{Br}_3$. Sample A was roughly 56% Br. By taking the higher amount, I was able to effectively identify the unknown compound.

Analysis for % Ammonia

By preparing three samples of the cobalt compound, the percent ammonia was able to be determined after first adding boric acid to each sample, and then adding NaOH to the cobalt sample. Distillation was effectively carried out by connecting both solutions with a plastic tube in order to allow the ammonia vapor to successfully flow from the Co-NH₃-X sample in NaOH to the boric acid solution. The biggest problem in this part of the experiment is the loss of ammonia through the tube when being delivered between solutions. The boric acid solution traps the NH₃ and decomposes the cobalt sample. Once this was done, five drops of brom-cresol-green indicator was used with .3M HCl to titrate until a yellow-greenish color was present. Upon having two successful trials, I had one outlier that did not match up with the other two trials (24.85%, 24.3456%, and 14.3456%, respectively). This outlier (14.3456% NH₃) was thrown out due to an error in passing the equivalence point while titrating. After all of this, I found there was roughly 24% NH₃ in the Co-compound.

Analysis for % Co

A spectrophotometer was used to analyze the wavelength of the Co-complex in order to ultimately find the % cobalt in the resulting unknown compound. Using .1501g Co-complex (precisely), 4 drops of HBr, and deionized water, two samples were taken and evaluated based on the outcome of molar absorptivity, absorbance reading, and distance the light shined through (using the equation $M=A/bE$). Based on the results gathered from both calculations of sample A

and sample B, sample B of 14.22 % Co was used, and sample A of 4.74 % Co was tossed out, due to it being an outlier.

Conclusion

After the analysis of halide, ammonia, and cobalt, it appears that compound #6 ($[\text{Co}(\text{NH}_3)_6]\text{Br}_3$) is the identity of the unknown product. This determination was made based on all of the calculations done from the completion of each experiment performed throughout the semester. In particular, the three main calculations used for deciphering the species of this unknown was finding the % halide, the % Co, and the % NH_3 . Following these calculations, reference to page 66 of the Georgia State University Chemistry 1212k Lab Manual was needed to effectively evaluate the exact compound produced. By looking at the second chart (because my compound's halide was Br, not Cl), I matched up the numbers that were found (horizontally). Wherever there were more stars checked in a line horizontally, indicated that this was where my product fell in line with the particular compound that yielded those numerical outcomes (percentages). Ultimately, on average, there was 14.22% Co, 24.85% NH_3 , and 59% Br. These numbers varied from the given set of numbers in the chart slightly, due to human and technological error (spills, inaccurate readings, etc.)

Appendix/Data

Determining % Halide in Co-Compound



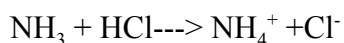
	Mass of Co-sample	Mass of precipitate	% halide in Co-sample
A	.2534 g	.3343 g	56 %
B	.2498 g	.3437 g	59 %

Sample Calculation:

Mass of precipitate * MM Br/ MM AgBr 187.77g / Mass of Co Sample

$$.3437\text{g} * 79.904\text{g} / 187.77\text{g} / .2498 = 59\% \text{ Br}$$

Determining % NH3 in Co-Compound



	Mass of Co-Compound	Vol. of HCl used in titration	%NH3 in Co-Compound
Trial 1	.3542 g	.01900 L	24.8500 %
Trial 2	.3553 g	.01100 L	14.3456 %
Trial 3	.3501 g	.01870 L	24.3456 %

Sample Calculation:

Avg Molarity * Volume of HCl used in titration (in L) * MM HCl 17.03g = g NH₃

$$.2721 * .01900\text{L} = .005169 \text{ mol NH}_3 * 17.03\text{g} = .08802 \text{ g NH}_3$$

Determining % Cobalt

- 1.) Find molarity using $M = A / bE$ (Determine epsilon's value using pg. 31-- it is 58.93 g/mol)
- 2.) Use the molarity you find to determine the grams of Co (found molarity * .100L * 58.93g/mol = grams cobalt)
- 3.) Calculate % Co (Xgrams Co/ 0.15g Co-complex made * 100 = final % Cobalt)

Sample Calculation:

$$M = .21 / (1)(58)$$

$$.00362069 * .100L * 58.93 \text{ g/mol} = \text{Grams of Cobalt} = 0.02134\text{g}$$

$$\% \text{ Co} = 0.02134\text{g} * 100 / .1501\text{g Co} = 14.22\% \text{ Co}$$