

# Gravimetric Analysis Workbook

## Question 1

A sample of magnesium weighing 14.932 g was placed in a crucible weighing 56.064 g. The crucible containing the magnesium was then heated intensely for several minutes. It was allowed to cool and the combined weight of the crucible and its contents was found to be 81.006 g. The crucible and its contents were heated again and allowed to cool and found to weigh 80.825 g. This procedure was repeated and the constant weight of the crucible and contents were established to be 80.825 g.

a) By how much did the mass inside the crucible increase after heating?

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(b) How many moles of magnesium were placed in the crucible?

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(c) What element did the magnesium react with during heating?

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(d) What is the empirical formula of the compound?

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## Question 2

Jeff was doing a school SAC on finding information on the sulfate content within a fertilizer. In this particular case Jeff used approximately 3.340g of fertilizer. After treating it adequately – according to the instructions – Jeff added 10% v/v barium chloride solution. The solution turned cloudy. Jeff then allowed the cloudy matter to settle. After taking a few steps Jeff weighed the dried precipitate. It weighed 1.342g.

a) What is the formula of the precipitate in this experiment?

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b) Jeff added two only drops of extra barium chloride solution. Why did he do this?

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c) When choosing the precipitate for gravimetric analysis the chemist has to take care to follow three criteria. What are the three properties that are required for the precipitate?

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d) Assuming that the sulfate in the precipitate came from the sample, find the % of sulfate within the sample.

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e) Find the % sulfide within the sample.

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f) Jeff found out that there is iron (III) sulfate within this sample. What is the % of it within the sample?

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e) Why is it important that the precipitate settles?

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### Question 3

What is the formula of a hydrate that is 86.7%  $\text{Mo}_2\text{S}_5$  and 13.3%  $\text{H}_2\text{O}$ ?

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### Question 4

During lab, 1.62 g of  $\text{CoCl}_2 \cdot A \text{H}_2\text{O}$  were heated. After heating, only 0.88 g of  $\text{CoCl}_2$  remained. What was the formula of the original hydrate?

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### Question 5

During lab, 1.04 g of  $\text{NiSO}_4 \cdot A \text{H}_2\text{O}$  were heated. After heating, only 0.61 g of  $\text{NiSO}_4$  remained. What was the formula of the original hydrate?

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### Question 6

*Gravimetric Analysis is a type of quantitative analysis in which the amount of a species in a material is determined by converting the species to a product that can be isolated completely and weighed. In this experiment a sulfate salt will react with  $\text{BaCl}_2$  to form  $\text{BaSO}_4(\text{s})$ , which can be analyzed. The amount of sulfate in the original sulfate salt can then be calculated.*

*Materials: Unknown sulfate salt solution 0.20 M  $\text{BaCl}_2$  filter paper*

*Procedure:*

- 1) Measure the mass of a 250 ml beaker and watch glass. (mass = 2.67g)*
- 2) Measure out 10 ml of the unknown sulfate salt solution and place in the 250 ml beaker. (Do not pour back into flask; put extra into waste.)*
- 3) Evaporate to dryness and measure the mass of the sulfate salt solid. (This will be the total mass of the salt.)*

*Start step #4 while it is evaporating. Remember: At first heat without the watch glass, then put it on when the solid begins popping, then take it off. Heat until dry.*

- 4) *Measure out 10 ml of the unknown sulfate salt solution into a 50 ml beaker.*
- 5) *Put around 10 ml of 0.2 M BaCl<sub>2</sub> into the 25 ml graduated cylinder. Then use a pipet to add the 0.2 M BaCl<sub>2</sub> solution drop-wise into the 50 ml beaker, until no more precipitate forms. You may need to let it settle between drops. (Try dropping at different places around the surface of the sulfate solution.)*
- 6) *Measure the mass of 2 filter papers. Filter the solid and allow to dry overnight. Put filtrate solution into waste.*
- 7) *Measure the mass of the BaSO<sub>4</sub>(s). (mass = 1.45g)*

i) Using the mass of BaSO<sub>4</sub>(s), do stoichiometry to find the mass of SO<sub>4</sub><sup>2-</sup> that was in the 10 ml sample of the unknown sulfate salt solution.

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ii) Find the percent of sulfate in the 10 ml sample of the unknown sulfate salt solution.

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iii) What is the percent of the cation part of the unknown sulfate salt solution?

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iv) Assuming this cation is sodium, do an empirical formula problem, using the percent sulfate and percent sodium to find the formula of the sulfate salt.

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v) Does this empirical formula agree with what you know about these two ions?

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**Question 7**

A 0.8310 g sample of waste material from a zinc smelter was treated with concentrated HNO<sub>3</sub> and KClO<sub>3</sub> to convert the sulphur to sulphate. After removing the nitrated and chlorate by repeated fuming with concentrated HCL, the sulphate was precipitated as BaSO<sub>4</sub>. After ignition and cooling BaSO<sub>4</sub> precipitate weighed 0.2997g. What is the %S in the waste material?

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**Question 8**

A 644.0 mg sample containing magnesium sulphate and inert material was dissolved and treated with (NH<sub>4</sub>)<sub>2</sub>HPO<sub>4</sub> to precipitate the magnesium as MgNH<sub>4</sub>PO<sub>4</sub>·6H<sub>2</sub>O. After being filtered and washed the precipitate was ignited at 1050°C to Mg<sub>2</sub>P<sub>2</sub>O<sub>7</sub> that, upon cooling, weighed 293.0mg. Calculate the % Mg in the sample.

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**Question 9**

Calculate the weight of CaCO<sub>3</sub> produced from heating 400mg of CaC<sub>2</sub>O<sub>4</sub>·H<sub>2</sub>O at 600°C.

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**Question 10**

What will 628mg of Zr(HPO<sub>4</sub>)<sub>2</sub> weigh after ignition to ZrP<sub>2</sub>O<sub>7</sub>?

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**Question 11**

A 1.0000g sample of “ultrapure silicon” was treated with an oxidizing agent and the resulting SiO<sub>2</sub>, after proper collection and drying weighed 2.1387g. Calculate the %Si in the sample.

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**Question 12**

A 350mg sample containing KClO<sub>3</sub> was carefully reduced and treated with excess AgNO<sub>3</sub>. The resulting AgCl weighed 185mg. Calculate the percentage of KClO<sub>3</sub> in the sample.

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**Question 13**

A commercial algaecide containing a organocopper compound was treated with concentrated nitric acid and evaporated to dryness. After dissolution of the residue the copper was precipitated with α-benzoin oxime. If the sample weighed 15.443 g and the precipitate of Cu(C<sub>14</sub>H<sub>12</sub>NO<sub>2</sub>)<sub>2</sub> weighed 0.6314g, calculate the %Cu in the algaecide.

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**Question 14**

A 674.3mg sample was determined by a gravimetric method. If the precipitate consisted of 214.4mg of barium sulphate and 9.7mg of Ba<sub>3</sub>(PO<sub>4</sub>)<sub>2</sub> calculate the;  
i) S% in the sample;

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%sulfate in the sample:

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% Barium in the sample;

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**Question 15**

A 0.4890 g sample of a  $\text{CaCO}_3$  is dissolved in an acidic solution. The calcium is precipitated as  $\text{CaC}_2\text{O}_4 \cdot \text{H}_2\text{O}$  and the dry precipitate is found to weigh 0.6188 g. What is the percentage of CaO in the sample?

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ii) What is the % of calcium ions in the sample. Are there any assumptions that you are making to obtain this answer?

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**Question 16**

You are analyzing a steel material that contains some nickel. If you find that 1.1634 grams of steel gave 0.1795 grams of  $\text{Ni}(\text{DMG})_2$  what is the weight percent nickel in the steel? ( $M_r [\text{Ni}(\text{DMG})_2] = 288.91$ ;  $M_r [\text{Ni}] = 58.69$ ).

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**Question 19**

A 0.2356-g sample containing only NaCl (58.44 g/mol) and BaCl<sub>2</sub> (208.25 g/mol) yielded 0.4637 g of dried AgCl(143.32 g/mol). Calculate the percent of each halogen compound in the sample.

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**Question 20**

Twenty dietary iron tablets (containing iron as FeSO<sub>4</sub>·7H<sub>2</sub>O) with a total mass of 22.131 g were ground and mixed thoroughly. Then 2.998 g of the powder was dissolved in HNO<sub>3</sub> and heated to convert all the iron to Fe(III). Addition of NH<sub>4</sub>OH caused quantitative precipitation of Fe<sub>2</sub>O<sub>3</sub>·x H<sub>2</sub>O, which was ignited to give 0.2641 g of pure Fe<sub>2</sub>O<sub>3</sub>. What was the average mass of FeSO<sub>4</sub>·7H<sub>2</sub>O in each tablet?

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