

Lecture – 14

Calculation of Results From Gravimetric Data

The results of a gravimetric analysis are generally computed from two experimental measurements: the mass of sample and the mass of a product of known composition. The examples that follow illustrate how such computations are carried out.

* Tutorial: Calculating Percentage of a Compound in a Sample Using Production of a Different Compound

Example 13-1

The calcium in a 200.0-mL sample of a natural water was determined by precipitating the cation as CaC_2O_4 . The precipitate was filtered, washed, and ignited in a crucible with an empty mass of 26.6002 g. The mass of the crucible plus CaO (56.077 g/mol) was 26.7134 g. Calculate the concentration of Ca (40.078 g/mol) in water in units of grams per 100 mL of the water.

The mass of CaO is

$$26.7134 \text{ g} - 26.6002 \text{ g} = 0.1132 \text{ g}$$

The number of moles Ca in the sample is equal to the number of moles CaO or

$$\begin{aligned} \text{amount of Ca} &= 0.1132 \text{ g CaO} \times \frac{1 \text{ mol CaO}}{56.077 \text{ g CaO}} \times \frac{1 \text{ mol Ca}}{\text{mol CaO}} \\ &= 2.0186 \times 10^{-3} \text{ mol Ca} \end{aligned}$$

$$\begin{aligned} \text{conc. Ca} &= \frac{2.0186 \times 10^{-3} \text{ mol Ca} \times 40.078 \text{ g Ca/mol Ca}}{200 \text{ mL sample}} \times 100 \text{ mL} \\ &= 0.04045 \text{ g/100 mL} \end{aligned}$$

Tutorial : Calculated Gravimetric Factors.

Example 13-2

An iron ore was analyzed by dissolving a 1.1324-g sample in concentrated HCl. The resulting solution was diluted with water, and the iron(III) was precipitated as the hydrous oxide $\text{Fe}_2\text{O}_3 \cdot x\text{H}_2\text{O}$ by the addition of NH_3 . After filtration and washing, the residue was ignited at a high temperature to give 0.5394 g of pure Fe_2O_3 (159.69 g/mol). Calculate (a) the % Fe (55.847 g/mol) and (b) the % Fe_3O_4 (231.54 g/mol) in the sample.

For both parts of this problem, we need to calculate the number of moles of Fe_2O_3 . Thus,

$$\begin{aligned}\text{amount Fe}_2\text{O}_3 &= 0.5394 \text{ g Fe}_2\text{O}_3 \times \frac{1 \text{ mol Fe}_2\text{O}_3}{159.69 \text{ g Fe}_2\text{O}_3} \\ &= 3.3778 \times 10^{-3} \text{ mol Fe}_2\text{O}_3\end{aligned}$$

(a) The number of moles of Fe is twice the number of moles of Fe_2O_3 , and

$$\begin{aligned}\text{mass Fe} &= 3.3778 \times 10^{-3} \text{ mol Fe}_2\text{O}_3 \times \frac{2 \text{ mol Fe}}{\text{mol Fe}_2\text{O}_3} \times 55.847 \frac{\text{g Fe}}{\text{mol Fe}} \\ &= 0.37728 \text{ g Fe} \\ \% \text{ Fe} &= \frac{0.37728 \text{ g Fe}}{1.1324 \text{ g sample}} \times 100\% = 33.32\%\end{aligned}$$

(b) As shown by the following balanced equation, 3 mol of Fe_2O_3 are chemically equivalent to 2 mol of Fe_3O_4 . That is,

$$\begin{aligned}3\text{Fe}_2\text{O}_3 &\rightarrow 2\text{Fe}_3\text{O}_4 + \frac{1}{2}\text{O}_2 \\ \text{mass Fe}_3\text{O}_4 &= 3.3778 \times 10^{-3} \text{ mol Fe}_2\text{O}_3 \times \frac{2 \text{ mol Fe}_3\text{O}_4}{3 \text{ mol Fe}_2\text{O}_3} \times \frac{231.54 \text{ g Fe}_3\text{O}_4}{\text{mol Fe}_3\text{O}_4} \\ &= 0.52140 \text{ g Fe}_3\text{O}_4 \\ \% \text{ Fe}_3\text{O}_4 &= \frac{0.52140 \text{ g Fe}_3\text{O}_4}{1.1324 \text{ g sample}} \times 100\% = 46.04\%\end{aligned}$$

Tutorial: Using Gravimetric Analysis for Determining Percent in a Mixture.

Example 13-3

A 0.2356-g sample containing *only* NaCl (58.44 g/mol) and BaCl₂ (208.23 g/mol) yielded 0.4637 g of dried AgCl (143.32 g/mol). Calculate the percent of each halogen compound in the sample.

If we let x be the mass of NaCl in grams and y be the mass of BaCl₂ in grams, we can write as a first equation

$$x + y = 0.2356 \text{ g sample}$$

To obtain the mass of AgCl from the NaCl, we write an expression for the number of moles of AgCl formed from the NaCl. That is,

$$\begin{aligned} \text{amount AgCl from NaCl} &= x \text{ g NaCl} \times \frac{1 \text{ mol NaCl}}{58.44 \text{ g NaCl}} \times \frac{1 \text{ mol AgCl}}{\text{mol NaCl}} \\ &= 0.017111x \text{ mol AgCl} \end{aligned}$$

The mass of AgCl from this source is

$$\begin{aligned} \text{mass AgCl from NaCl} &= 0.017111x \text{ mol AgCl} \times 143.32 \frac{\text{g AgCl}}{\text{mol AgCl}} \\ &= 2.4524x \text{ g AgCl} \end{aligned}$$

Proceeding in the same way, we can write that the number of moles of AgCl from the BaCl₂ is given by

$$\begin{aligned} \text{amount AgCl from BaCl}_2 &= y \text{ g BaCl}_2 \times \frac{1 \text{ mol BaCl}_2}{208.23 \text{ g BaCl}_2} \times \frac{2 \text{ mol AgCl}}{\text{mol BaCl}_2} \\ &= 9.605 \times 10^{-3}y \text{ mol AgCl} \\ \text{amount AgCl from BaCl}_2 &= 9.605 \times 10^{-3}y \text{ mol AgCl} \times 143.32 \frac{\text{g AgCl}}{\text{mol AgCl}} \\ &= 1.3766y \text{ g AgCl} \end{aligned}$$

Because 0.4637 g of AgCl comes from the two compounds, we can write

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$$2.4524x + 1.3766y = 0.4637$$

The first equation can be rewritten as

$$y = 0.2356 - x$$

Substituting into the previous equation gives

$$2.4524x + 1.3766(0.2356 - x) = 0.4637$$

which rearranges to

$$1.0758x = 0.13942$$

$$x = \text{mass NaCl} = 0.12960 \text{ g NaCl}$$

$$\% \text{ NaCl} = \frac{0.12956 \text{ g NaCl}}{0.2356 \text{ g sample}} \times 100\% = 55.01\%$$

$$\% \text{ BaCl}_2 = 100.00\% - 55.01\% = 44.99\%$$

Example 13-4

- An ore containing magnetite, Fe_3O_4 , was analyzed by dissolving a 1.5419-g sample in concentrated HCl, giving a mixture of Fe^{2+} and Fe^{3+} . After adding HNO_3 to oxidize any Fe^{2+} to Fe^{3+} , the resulting solution was diluted with water and the Fe^{3+} precipitated as $\text{Fe}(\text{OH})_3$ by adding NH_3 . After filtering and rinsing, the residue was ignited, giving 0.8525 g of pure Fe_2O_3 . Calculate the %w/w Fe_3O_4 in the sample.

SOLUTION

This is an example of a direct analysis since the iron in the analyte, Fe_3O_4 , is part of the isolated precipitate, Fe_2O_3 . Applying a conservation of mass to Fe, we write

$$3 \times \text{moles Fe}_3\text{O}_4 = 2 \times \text{moles Fe}_2\text{O}_3$$

Using formula weights, FW, to convert from moles to grams in the preceding equation leaves us with

$$\frac{3 \times \text{g Fe}_3\text{O}_4}{\text{FW Fe}_3\text{O}_4} = \frac{2 \times \text{g Fe}_2\text{O}_3}{\text{FW Fe}_2\text{O}_3}$$

which can be solved for grams of Fe_3O_4 and %w/w Fe_3O_4 in the sample.

$$\frac{2 \times g \text{ Fe}_2\text{O}_3 \times \text{FW Fe}_3\text{O}_4}{3 \times \text{FW Fe}_2\text{O}_3} = \frac{2 \times 0.8525 \text{ g} \times 231.54 \text{ g/mol}}{3 \times 159.69 \text{ g/mol}} = 0.82405 \text{ g Fe}_3\text{O}_4$$

$$\frac{g \text{ Fe}_3\text{O}_4}{g \text{ sample}} \times 100 = \frac{0.82405 \text{ g}}{1.5419 \text{ g}} \times 100 = 53.44\% \text{ w/w Fe}$$

Example 13-5

A 0.6113-g sample of Dow metal, containing aluminum, magnesium, and other metals, was dissolved and treated to prevent interferences by the other metals. The aluminum and magnesium were precipitated with 8-hydroxyquinoline. After filtering and drying, the mixture of $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$ and $\text{Mg}(\text{C}_9\text{H}_6\text{NO})_2$ was found to weigh 7.8154 g. The mixture of dried precipitates was then ignited, converting the precipitate to a mixture of Al_2O_3 and MgO . The weight of this mixed solid was found to be 1.0022 g. Calculate the %w/w Al and %w/w Mg in the alloy.

SOLUTION:

This is an example of a direct analysis in which the two analytes are determined without a prior separation. The weight of the original precipitate and the ignited precipitate are given by the following two equations ;

$$g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3 + g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = 7.8154$$

$$g \text{ Al}_2\text{O}_3 + g \text{ MgO} = 1.0022$$

containing four unknown terms. To solve this pair of equations, we must find two additional equations relating the four unknowns to one another. These additional equations describe the stoichiometric relationships between the two compounds containing aluminum and the two compounds containing magnesium and are based on the conservation of Al and Mg. Thus, for Al we have

$$2 \times \text{moles Al}_2\text{O}_3 = \text{moles Al}(\text{C}_9\text{H}_6\text{NO})_3$$

Converting from moles to grams and solving yields an equation relating the grams of Al_2O_3 to the grams of $\text{Al}(\text{C}_9\text{H}_6\text{NO})_3$

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$$\frac{2 \times g \text{ Al}_2\text{O}_3}{\text{FW Al}_2\text{O}_3} = \frac{g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3}{\text{FW Al}(\text{C}_9\text{H}_6\text{NO})_3}$$

$$g \text{ Al}_2\text{O}_3 = \frac{g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3 \times \text{FW Al}_2\text{O}_3}{2 \times \text{FW Al}(\text{C}_9\text{H}_6\text{NO})_3} = \frac{g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3 \times 101.96 \text{ g/mol}}{2 \times 459.45 \text{ g/mol}}$$
$$= 0.11096 \times g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3$$

For Mg we have

$$\text{Moles MgO} = \text{moles Mg}(\text{C}_9\text{H}_6\text{NO})_2$$

$$\frac{g \text{ MgO}}{\text{FW MgO}} = \frac{g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2}{\text{FW Mg}(\text{C}_9\text{H}_6\text{NO})_2}$$

$$g \text{ MgO} = \frac{g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 \times \text{FW MgO}}{\text{FW Mg}(\text{C}_9\text{H}_6\text{NO})_2} = \frac{g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 \times 40.304 \text{ g/mol}}{312.61 \text{ g/mol}}$$
$$= 0.12893 \times g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2$$

Substituting the equations for g MgO and g Al₂O₃ into the equation for the combined weights of MgO and Al₂O₃ leaves us with two equations and two **unknowns**.

$$g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3 + g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = 7.8154$$

$$0.11096 \times g \text{ Al}(\text{C}_9\text{H}_6\text{NO})_3 + 0.12893 \times g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = 1.0022$$

Multiplying the first equation by 0.11096 and subtracting the second equation Gives

$$-0.01797 \times g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = -0.1350$$

which can be solved for the mass of Mg(C₉H₆NO)₂.

$$g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = 7.5125 \text{ g}$$

The mass of Al(C₉H₆NO)₃ can then be calculated using the known combined mass of the two original precipitates.

$$7.8154 \text{ g} - g \text{ Mg}(\text{C}_9\text{H}_6\text{NO})_2 = 7.8154 \text{ g} - 7.5125 \text{ g} = 0.3029 \text{ g Al}(\text{C}_9\text{H}_6\text{NO})_3$$

Using the conservation of Mg and Al, the % w/w Mg and % w/w Al in the sample can now be determined as in Example 5, where AW is an atomic weight

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$$\text{Moles Mg} = \text{moles Mg}(\text{C}_9\text{H}_6\text{NO})_2$$

$$\frac{\text{g Mg}}{\text{AW Mg}} = \frac{\text{g Mg}(\text{C}_9\text{H}_6\text{NO})_2}{\text{FW Mg}(\text{C}_9\text{H}_6\text{NO})_2}$$

$$\text{g Mg} = \frac{\text{g Mg}(\text{C}_9\text{H}_6\text{NO})_2 \times \text{AW Mg}}{\text{FW Mg}(\text{C}_9\text{H}_6\text{NO})_2}$$

$$= \frac{7.5125 \text{ g} \times 24.305 \text{ g/mol}}{312.61 \text{ g/mol}}$$

$$= 0.5841 \text{ g}$$

$$\% \text{Mg} = \frac{\text{g Mg}}{\text{g sample}} \times 100$$

$$\frac{0.5841 \text{ g}}{0.6113 \text{ g}} \times 100 = 95.55\% \text{ w/w Mg}$$

$$\text{Moles Al} = \text{moles Al}(\text{C}_9\text{H}_6\text{NO})_3$$

$$\frac{\text{g Al}}{\text{AW Al}} = \frac{\text{g Al}(\text{C}_9\text{H}_6\text{NO})_3}{\text{FW Al}(\text{C}_9\text{H}_6\text{NO})_3}$$

$$\text{g Al} = \frac{\text{g Al}(\text{C}_9\text{H}_6\text{NO})_3 \times \text{AW Al}}{\text{FW Al}(\text{C}_9\text{H}_6\text{NO})_3}$$

$$= \frac{0.3029 \text{ g} \times 26.982 \text{ g/mol}}{459.45 \text{ g/mol}}$$

$$= 0.0178 \text{ g}$$

$$\% \text{Al} = \frac{\text{g Al}}{\text{g sample}} \times 100$$

$$\frac{0.0178 \text{ g}}{0.6113 \text{ g}} \times 100 = 2.91\% \text{ w/w Al}$$