

Chemistry 1215 Experiment #7
Quantitative Analysis of Iron Metal in Total Cereal

Objective

The objective of this experiment is to extract the iron metal from a sample of Total Cereal, determine how much iron is present, and then compare the determined amount with the manufacturer's claim of 100% RDA.

Introduction

Many consumers read packaging labels to determine the nutritional content of the foods they buy. People often want to know what has been added to their food and to have an idea of the amounts of fat, protein and carbohydrate as well as vitamins, minerals, dyes, and preservatives added by the manufacturer. All processed foods sold in stores in the United States are required by the FDA to carry a label showing the relevant nutritional information. But is the information accurate?

The label on a package of Total cereal tells us that the cereal contains 40 mg of potassium per serving. Potassium metal is extremely reactive. Potassium atoms will transfer electrons to the hydrogen ions in water generating explosive hydrogen gas. Imagine your surprise when, as a result of pouring milk on your breakfast cereal, it promptly blew up in your face. The information on the cereal package is misleading. Total cereal does not contain elemental potassium; rather it contains potassium cations, K^+ . Potassium ions have very different properties than potassium metal. While elemental potassium is extremely reactive and toxic, potassium ions form soluble ionic compounds and are essential for life. The difference between these two forms of potassium is that potassium ions have one less electron.

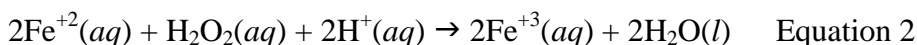
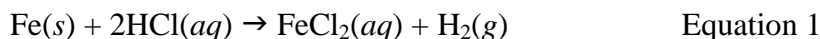
The loss of electrons by an element or ion is called oxidation. The corresponding gain of electrons is called reduction. In the example above the potassium metal is oxidized indicating the loss of an electron. For some individuals the mnemonic "oilrig" is helpful. It stands for "oxidation is loss, reduction is gain," meaning the loss or gain of electrons.

The label on a box of Total cereal also states that it contains 100% of the daily recommended intake of iron. Elemental iron is not as reactive as elemental potassium and is added to Total cereal as the pure element rather than in the form of ions. In fact, any food label that indicates that iron is present in a reduced form or as reduced iron contains metallic iron in the form of iron filings. The term "reduced iron" means that the iron in the sample has as many electrons as it can hold. Since metals lose electrons to form cations, reduced iron is the metallic, un-ionized form of iron. The oxidized, cationic, forms of iron are Fe^{+2} and Fe^{+3} .

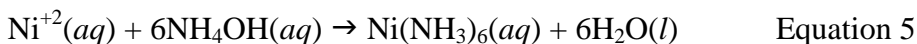
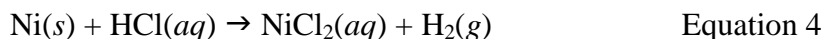
Elemental iron is ferromagnetic meaning that it is attracted to an external magnet. Ferromagnetism comes about when unpaired electron spins on atoms or ions in a solid become permanently aligned. When electrons are unpaired, the electron spin, coupled with its orbital angular momentum, creates a magnetic field. Most electrons around an atom are spin paired and their magnetic fields cancel each other. However, when atoms have unpaired electron spins, they develop a net magnetic moment. When many such atoms are together, their tiny magnetic moments can align in the same direction and create a measurable macroscopic magnetic field. Such is the case with iron. An iron atom

has six electrons spread across five d-orbitals so it can have as many as four unpaired electrons. There is enough iron in a single flake of Total cereal that the flakes can be moved with a strong magnet. In this experiment your task will be to extract the iron from a cereal sample and determine its mass. The amount of iron is small so great care must be taken to avoid loss of even part of the sample.

Because iron is not the only ferromagnetic metal, the extracted metal will be tested to determine its identity. A distinctive chemical test for iron involves first dissolving the metal in hydrochloric acid, HCl. The FeCl_2 that is formed is a yellow, water soluble compound so it stays in solution. The other product, H_2 gas, bubbles out of the solution into the atmosphere. This reaction is an oxidation-reduction reaction that involves transfer of electrons from the iron to hydrogen ions according to Equation 1. Once the iron is ionized it must be further oxidized to iron (III). This is done by adding hydrogen peroxide to the acidic iron (II) solution as shown in equation 2. Once formed, iron (III) will react with potassium thiocyanate to form the blood red complex ion, $\text{Fe}(\text{SCN})_6^{-3}$ (Equation 3). Formation of the red iron (III) thiocyanate complex confirms the presence of iron in the sample.



Nickel metal is weakly ferromagnetic so it is also necessary to test the sample for nickel. Nickel metal is oxidized by hydrochloric acid to nickel (II) chloride at the same time the iron is oxidized (Equation 4). Because the test for nickel (II) requires that the solution be basic, aqueous ammonia, NH_4OH (aq), is added (Equation 5) followed by a small amount of dimethylglyoxime, $(\text{CH}_3)_2\text{C}_2(\text{NOH})_2$, (referred to here as DMG) solution (Equation 6). DMG forms a complex with nickel (II) producing a pink precipitate. The pink precipitate confirms the presence of nickel.



Control experiments with both Fe^{+3} and Ni^{+2} will be conducted alongside the sample tests so that you can observe the indicated color changes first hand. Remember that the test solutions may be more dilute than the control solutions. You are looking for the same color but not necessarily the same intensity of color.

Procedure

To release the iron from the flakes it is necessary to crush the cereal into as fine a powder as possible using a mortar and pestle. Approximately 10 g of cereal is recommended but it is important to measure the mass of the cereal as accurately as

possible after it is ground up. After grinding and weighing the cereal, place it in a beaker and add distilled water to make a thin slurry. Add a magnetic stirring bar (previously weighed, see below) and place the beaker on a stirring plate. Place an iron ring around the beaker (hold it in place with a ring stand) so it can't easily fall off the stirring plate. The stirring bar is a 1-2 cm Teflon coated piece of metal that spins when the rotating magnet inside the stirring plate is turned on. When the stirring plate speed is properly adjusted the stirring bar will spin fast enough to stir the cereal slurry at a moderate speed and collect the bits of iron in the sample. If the slurry is too thick or the stirring speed is too fast, the iron filings will not stick to the stirring bar due to shear forces.

After about 5-10 minutes of stirring you can carefully remove the stirring bar from the slurry with non-magnetic tongs. Carefully rinse off any cereal sticking to the bar by gently squirting it with water from a Pasteur pipet (**note:** do the washing step over the beaker, not over a sink). You should observe a small amount of black metal filings magnetically attached to the bar. If not, return the bar to the slurry and continue the stirring for another five minutes. The sample can be dried quickly by carefully rinsing with the volatile organic solvent, acetone. Allow the acetone to evaporate (about 2 minutes) then weigh the stirring bar on an analytical balance with the metal filings attached. Because the stirring bar is magnetic, it can interfere with the internal workings of the analytical balance. To avoid problems it is necessary to invert a small (50 or 100 mL) beaker on the pan of the balance and place the stirring bar on top of the up-side-down beaker to keep it several centimeters above the balance pan. Make sure that you tare the balance with the beaker in place (but without the stirring bar) or that you use the same beaker each time you weigh the bar. The mass of the metal filings can be obtained by subtracting the mass of the clean bar from the mass of the bar plus filings.

Once the mass of the filings is obtained, the material can be tested for the presence of iron and nickel as described in the introduction. The stirring bar, still coated with the magnetic metal filings, should be placed in a medium-sized test tube. One milliliter of **concentrated** (12 M) hydrochloric acid is added. To speed up the dissolution, gently heat the sample in a fume hood (this can be done by placing the test tube in a boiling water bath). After the filings dissolve, add three milliliters of distilled water.

The tests for iron and nickel are performed in a spot plate, a glass or porcelain plate with small wells where small volume chemical reactions can be observed and colors recorded. Place two drops of the dissolved test solution into each of two wells of the plate. Mark these Fe^{+3} test and Ni^{+2} test with a lab marker, record the colors on your data table. In a third well add one drop of the control iron (III) solution (marked Fe^{+3} control) record the sample color. In a fourth well add one drop of the control nickel (II) solution (marked Ni^{+2} control) record the sample color. Next add two drops of 3% H_2O_2 solution to the test Fe^{+2} sample well. Allow the sample to sit for 1-2 minutes then record the sample color. Next add one drop of KSCN to both the Fe^{+3} test well and the Fe^{+3} control well. Observe and record the sample colors. If iron (III) is present in the sample the two samples should be the same color although the intensity of the color may be different.

To test for nickel, add 6 M NH_4OH solution to both the Ni^{+2} test well and to the Ni^{+2} control well. Continue adding NH_4OH one drop at a time until the solution becomes basic. To test the pH, dip one end of a glass stirring rod into the solution in the well then spot it on a piece of red litmus paper. The solution is basic when the red litmus paper

turns blue. Once the well solutions are basic, record the sample color then add one drop of DMG solution. Again, observe and record the colors.

Chemistry 1215 Experiment #7, Data Table

Mass of the clean stirring bar _____.

Mass of the stirring bar plus the metal filings _____.

Mass of the metal filings _____.

Sample	Initial solution color	Intermediate solution color	Final solution color
Fe ⁺³ test well			
Fe ⁺³ control well			
Ni ⁺² test well			
Ni ⁺² control well			

Discussion: Write a short discussion including a statement explaining your sample data. Compare your results with the manufacturer's claim of 100% daily requirement of iron. Remember that a serving size is 30 grams.

Chemistry 1215 Experiment #7: The Quantitative Analysis of Iron Metal in Total Cereal,

Pre-lab

Name _____

1. What is the daily recommended intake for iron? List your reference source and be as specific as possible.
2. Assuming your sample of cereal is 10.00 grams and a recommended serving of cereal is 30.00 grams, how many mg of iron would you expect to extract from your sample? Use the recommended daily intake of iron in question 1 and the Manufacturer's claims of iron present in Total cereal to justify your answer.
3. In the colorimetric (color producing) assay for iron used in this lab; potassium thiocyanate, KSCN, is added to produce a deep red colored precipitate. The lab outline suggests that H_2O_2 may be added to enhance the color. Write a balanced chemical equation showing the function of the peroxide. In your equation identify the oxidizing agent and the reducing agent.
4. Write a balanced chemical equation that describes the reaction of iron metal with HCl. Identify the oxidizing and reducing agents in your balanced equation.

