Kjeldahl Analysis for Nitrogen in Corn Flakes

Much of the real work of analytical chemistry is involved in sample pretreatment. Most samples are not in the proper form for the chosen analysis when they are received. Subjects as diverse as digestion, atomization and separations (chromatography) fall under the broad heading of sample pretreatment. This lab exercise uses a titration for the final analysis but is mostly about creating an analyzable solution from the sample (corn flakes.)

Look at the nutrition data on a box of corn flakes. A 28.4 g serving contains about 2 g of protein. Proteins are typically about 16% nitrogen. From these data calculate the sample size that will produce about 2 millimoles of nitrogen.

Do the analysis in triplicate if time allows, but at least twice.

CAUTION!

The Kjeldahl analysis uses hot concentrated acid and concentrated sodium hydroxide. It is imperative that you follow safety directions carefully.

PROCEDURE

Crush the corn flakes in a mortar and dry for 2 hours at 105 ºC before weighing. *Your TA may have taken care of the crushing and drying steps in advance.*

You will need to make up a 0.1 M NaOH and a 0.1M HCl solution for the capture and titration part of the experiment.

Weigh out the required amount of cornflakes and transfer quantitatively to the flasks. To do this, roll up a piece of clean paper and insert it into the neck of the Kjeldahl flask. This will keep the neck of the flask clean while you add the corn flakes. Be sure your sample weight is accurate to ±10 ppth.

Add ~5 g potassium sulfate and ~0.1 g copper sulfate to each flask.

For the rest of this experiment – except for the final titrations – we will be working in the fume hoods. (Remember to keep the sash closed as much as possible)

CAUTION!

98% sulfuric acid is dangerous. Wear safety goggles and rubber gloves. If you get any on your skin, wash immediately with copious amounts of cold water, followed by soap and water.

Add 12 – 15 mL concentrated H_2SO_4 to the flask. Allow the cornflake/H_2SO_4 mixture to cool. Clamp the Kjeldahl flask in a ring stand IN THE HOOD, with the neck pointing toward the BACK OF THE HOOD. Heat with a heating mantle and insulate with glass wool.

Reflux until mixture becomes clear. Significant fuming will occur during this process: this is normal. As the amount of fuming decreases, your solution will change from brown to red to yellow to green. (Note: many of the mantles do not have a temperature setting, but just a rheostat. In these cases, turn the heat to high and watch the reflux. Make sure you do not boil off all of the liquid during this process.) Allow the flask to cool in the air and then in an ice bath.

CAUTION !

Recall the rule "Always add acid to water; never add water to acid." Now we have to break that rule, so use caution.

Add 25 mL ice cold water very carefully, a little at a time, and KEEP THE FLASK POINTED TOWARD THE REAR OF THE FUME HOOD.

Set up the distillation apparatus as shown by your TA. Pipette 25.00 mL of ice cold [0.1M] HCl into the receiving flask. The delivery tube must extend below the surface of the HCl solution. (You may need to angle the Erlenmeyer using a clamp) You must have this apparatus all set up and ready to go before the next step. Set the heat source to ~ 100°C.

CAUTION !

25% sodium hydroxide can dissolve skin and cause blindness within seconds. Wear safety goggles and rubber gloves. KEEP THE KJELDAHL FLASK POINTED TOWARD THE REAR OF THE HOOD. If you get any on your skin immediately wash with copious amounts of running water.

As soon as you make the solution basic, ammonia begins to escape. You must do the following operation with some finesse to avoid ammonia loss.

Add ~50 mL of 25% (9.5 M) NaOH. Make sure to add NaOH slowly but constantly. While adding the NaOH, seal the top of the flask with the bottom of the syringe.

Violent reaction can occur, which is minimized by slow addition and having the NaOH and Kjeldahl flask sufficiently cold. After a few mLs are added, the solution may begin to turn dark and cloudy. This is normal, and will not affect the analysis. Continue addition until 50 mL of NaOH have been added, or until the addition of continued amounts of the solution does not result in a vigorous reaction within the vessel, whichever comes later. The absence of vigorous reaction is confirmation that the acid solution has been neutralized. During the NaOH addition process, significant bubbling may occur in the receiver vessel, and the solution may change colors from red/pink to gray, blue or green. This is due the emission of NH_3 by the sample, and is normal. (Make sure not to get any of the NaOH in the distillation arm as it will ruin your results!)

Once the flask is warm enough to do so, place it on heat source. Distill until about 15 mL of water has been carried over. After distillation, rinse the tubing that dips into the HCl, and titrate the unreacted HCl with standardized sodium hydroxide using bromocresol green indicator.

REQUIRED MEASUREMENTS

Present the results for the % nitrogen in the sample to your lab TA before you leave. We will be a bit more generous about the allowable error limits, considering the complexity of the operations performed. You may choose to use the results of only the last two runs (if you complete three) if the first appears to be seriously in error. The Q-test (SWHC p. 167-8) may help in this decision, but the choice will be up to you.