Agri 201

Kjeldahl Part II
Ammonia Distillation
material

4% boric acid w/ bromocresol green/methyl red indicator	100 ppm NH4-N Ammonium PTSA*
(7) 250 ml test tubes	Magnetic stir bar
ammonia still (Tecator)	Magnetic stirrer
(7) 250 ml Erlenmeyer flasks This are the receiver flasks.	Kjeldahl Waste container
100 ml graduated cylinder	50% sodium Hydroxide
waste container	
1 burette, 50 ml	0.05 N sulfuric acid

Safety:
Use goggles not safety glasses, **face shield**, **apron, gloves,**
fume hood, safety eyewash and shower
Another person should be present in the lab while doing this lab.
Hazardous waste container labeled Kjeldahl is in the fume hood.

Method:

Kjeldahl Distillation

Wear: Goggles, face shield, gloves and apron

1. Turn on the Kjeldahl still following the instructions posted by the still.
2. Measure approximately 25 mL of boric acid solution (3 pumps from dispenser) into 250 ml receiver flask and place on still.(right side)
3. Add 40 of sample and 10 ml of DI water to a 250 ml digestion tube. Place digestion tube on still (left side)
4. Slowly pull alkali dispenser handle down. (If you pull the handle too fast the digest will blow out the top of the tube)
5. Pull the steam handle down.
6. Set the timer for 5 minutes or use watch.
7. After 5 minutes push the steam handle up.
8. Take the digestion tube off, place tube in a plastic container, cork it and then move it to the fume hood .
9. Pour the contents of the tube into the container labeled Kjeldahl waste. (The fume hood should be on.)
10. Remove the receiver flask. (You will do the titration on this flask) The solution should be green.

Note:
1.) Before analysis of samples try this on a tube with 50 ml of water.
2.) The distillations can be completed first and the titrations afterwards.
3.) The 100 ppm NH4-N PTSA can be used to practice the distillation. Put 10 ml of the 100 ppm NH4-N PTSA into 250ml digestion tube, add 40

ml of DI water and follow the above directions, steps 1-10. It should take 1.4ml of 0.05N Acid to titrate the ammonia recovered from this solution.

Titration:

1. Titrate the receiver flask with a 0.05 standard acid until the solution is the same color as the original 4% boric acid solution. Once the solution has turned gray or colorless add the acid a drop wise until a proper color is obtained. A flask with the proper endpoint has been provided.

Calculate the Nitrogen % using the following formula:

N% = [(Normality of acid) X (ml of acid) X (14.01) / (sample in mg)] X 100
%protein = %N X 6.25 (This is a constant used for most commodities. However, some commodities have different constants.

1. Discuss the working range, resolution and detection limit of this procedure and how they could be changed.
2. What is the precision of the procedure?
3. What is the accuracy of the procedure?
4. What are some potential sources of error?
5. How would you improve this procedure?
6. What are the components of the measurement system?