

Kjeldahl Digestion Procedure (Hach Digester) Lab 4 (part 1)

Purpose: to learn how to digest samples for total N analysis

Materials:

Kjeldahl digestion procedure (Hach Digester) handout.	concentrated Sulfuric Acid (36N H ₂ SO ₄) dispenser ²
(6) 100 ml Hach digestion flasks ²	ground alfalfa ³
(7) 100 ml plastic bottles with labels ³	weighing paper (by balance in drawer)
50% Hydrogen Peroxide (H ₂ O ₂) ¹	glycine-PTSA ¹
Hach digestahl heater ²	Balance (mettler PM 2500) .001g
Dextrose (reagent grade) ³	Kitchen timer ²
Finger cots	Gloves, Face shield, Apron

1. Analytical room refrigerator 2. Digestion room fume hood 3. Group drawer in Drying and Grinding room

Method:

Goggles, apron or lab coat, and gloves must be worn during this procedure.

Warning! The chemicals (sulfuric acid & Hydrogen peroxide) you are using are extremely reactive and caustic. These chemicals can cause severe burns and eye damage. Goggles and gloves must be worn when working with these chemicals. If you get these chemicals on you, wash at least 5 minutes in cold flowing water. Inform lab tech of any exposure. Never add hydrogen peroxide to organic solvents like alcohols, acetone and ether (There is a possibility of explosion.).

1. Weigh 0.25 g of sample or standard into Hach 100 ml Volumetric flask. note: never use more than .5 grams of sample.
 - a. Ground alfalfa sample. (3 reps) (drawer)
 - b. Glycine-PTSA. (1 rep) (refrigerator)
 - c. Dextrose (1 rep)

**The following steps should be carried out in a fume hood.
(Setup is in the digestion room.)**

2. Add 5 ml of concentrated sulfuric acid (36N H₂SO₄) using a dispensing pump.
3. Turn on the digester. Set the temperature to 440 by pushing the temperature switch to set and turning the adj knob. When finished push the read switch. Note: the temperature is usually already set to 440 C and needs no adjusting.
4. Place the flask weight, followed by the fractionating column with funnel on the flask. Turn on water for aspirator

5. Place the flask on the heater when the temperature reads 440C and boil 4 minutes.

Note: White acid vapors accompanied with a reflux line indicated that the boiling point of sulfuric acid has been reached.

Note: If sample starts to foam up into the neck of the flask, lower temperature to 335 C. Continue heating at lower temperature until all water is evaporated off. Then return to original digestion temperature and continue with instructions in step 4.

Note: Some organic samples may need more than five minutes for complete digestion.

6. Check to see if sulfuric acid is visible in flask. If no sulfuric acid is visible then take the flask off of heater and let cool.

WARNING: Do not proceed if sulfuric acid is not visible in the flask. Hydrogen Peroxide added to a dry flask can cause an explosion.

7. Place safety shield in front of heater.
8. Add 10 ml of 50% Hydrogen Peroxide to the capillary funnel at the top of the fractionating column. (Hydrogen peroxide kept in refrigerator. Return to the refrigerator when finished.)
9. Boil off excess hydrogen peroxide by heating for one more minute after addition of hydrogen peroxide is complete. Do not heat to dryness.
10. Use finger cots to take flask off the heater and place on cooling pad. Allow the flask to cool. Remove the fractionating column from the digestion flask.
11. Dilute the digest with approximately 70 ml of water and swirl to mix. Add DI water to 100 ml mark on flask. Place stopper on flask and invert flask at least 20 times to mix.

Note: Water should be added a few drops at a time and mixed until approximately 20ml has been added. Always point the neck of the flask toward the fume hood when adding and mixing water.

12. Pour contents of flask into a labeled plastic bottle.

NOTE: If the flask should break at any time during the digestion, unplug the heater, post a sign informing of problem and contact the lab technician. Do not clean up chemical spills; contact the technician.