RAPID KJELDAHL
BENCHNOTES

Rapid Kjeldahl Methodology for the Determination of Nitrogen in Paper Products with the Rapid Digestor and the RapidStill II

Principle: *This method covers the determination of organic nitrogen in paper products including those containing glue, adhesives and other collagenous material.*

Apparatus

1. Labconco Rapid Digestor:
   Rapid Digestor 4 (Model 23080)
   Rapid Digestor 25 (Model 23012)

2. Labconco Fume Removal System:
   Fume Removal System-4 place (Model 23540)
   Fume Removal System-25 place (Model 23500-25)

3. Labconco Rapid Distillation Unit:
   RapidStill II (Model 65200)

4. Labconco 250 ml Digestion Tubes:
   Volumetric, Pkg. of 5 (Model 23030-05)
   Volumetric, Pkg. of 25 (Model 23030-25)
   Straight, Pkg. of 5 (Model 23040-05)
   Straight, Pkg. of 25 (Model 23040-25)

5. Two 25 ml Class A burets

6. 500 ml Erlenmeyer flasks

Reagents

(All reagents should be reagent grade and nitrogen-free.)

1. Sodium hydroxide 45% (w/v) pellets or beads for nitrogen determination: Dissolve 450 g sodium hydroxide (NaOH) in deionized water. Cool. Dilute to one liter with deionized water.

2. Methyl red/methylene blue indicator: Dissolve 200 mg (0.2 g) methyl red in 100 ml ethyl alcohol. In a separate beaker, dissolve 200 mg methylene blue in 100 ml ethyl alcohol. Combine two parts methyl red with one part methylene blue solution and mix.

3. Boric acid/indicator solution 2% (w/v): Dissolve 20 g boric acid (H_3BO_3) in 800 ml of deionized water. Add 10 ml methyl red/methylene blue indicator solution and dilute to one liter with deionized water.

4. Sulfuric acid (H_2SO_4) - concentrated

5. Red mercuric oxide (HgO) or cupric sulfate ($CuSO_4$ or $CuSO_4 \cdot 5\ H_2O$)

6. Potassium sulfate (K_2SO_4) suitable for nitrogen determination

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7. 0.2N sulfuric acid (H_2SO_4) solution

8. Alundum boiling stones

Procedure

1. Cut paper samples into pieces (approximately 1" x 1/2"). Weigh samples and place in digestion tubes. A one gram sample works best for paper containing 3% nitrogen.

2. Place weighed sample in 250 ml digestion tube with 0.42 g HgO, 9.0 g K_2SO_4, and 15 ml H_2SO_4.

3. Place digestion tubes in Rapid Digestor which has been preheated to 410° C.

4. Digest for 45 minutes. When digestion is complete, remove tubes. Make sure digestion sample is clear. If necessary, digest longer until sample is clear and then cool for ten minutes.

5. Add 50 ml deionized water to each tube, dissolving all crystallized material. It may be necessary to place tubes back in the Rapid Digestor, to expedite dissolving the crystallized material. Allow to cool. Cooling times depend on the temperature and circulation of air around the tubes and may require adjustment depending on your conditions. Do not allow hard cakes to form.

6. After the sample has cooled, attach digestion tube to the distillation head and twist securely in place.

7. Place delivery tube in 500 ml Erlenmeyer flask which contains 50 ml boric acid/indicator solution. The tip must be below the surface of the boric acid/indicator solution.

8. Add approximately 80 ml sodium hydroxide solution to the digestion tube.

9. Set the RapidStill II for 8 minutes.

10. Start the distillation process and steam vigorously until 125 ml of distillate collects.

11. When distillation is complete, fill a class A buret to the mark with 0.2N sulfuric acid (H_2SO_4) and titrate to the purple endpoint. Correct for reagent blank.

$$\%N = \frac{(\text{ml std. } H_2SO_4 - \text{ml } H_2SO_4 \text{ blank})(\text{N } H_2SO_4)(1.4007)}{\text{gram sample weight}}$$

Example:
blank = 1 ml sample = 1 gram
acid added = 11 ml

$$\%N = \frac{(11 \text{ ml} - 1 \text{ ml})(0.2\text{N } H_2SO_4)(1.400)}{1 \text{ gram}}$$

%N = 2.8%
%Protein = 17.5%

Notes

1. Run a blank sample (reagents only, no sample) and subtract the milliliters of H_2SO_4 consumed by the blank from that consumed by the sample as indicated in the calculation.

2. When titrating, titrate to the purple endpoint. Do not titrate all the way back to red.

3. With careful technique, this equipment will give results with a relative standard deviation of no greater than 1.5%.

4. All testing done at our laboratory was performed with a Kjeldahl digestion mixture containing $K_2SO_4/TiO_2/CuSO_4$ in the ratio 10/0.3/0.3.

Reference

Official Methods of Analysis AOAC. 15th ed. Arlington, VA: (1990) • 7.032, 24.038 - 24.040.

This methodology is for reference only. It is not a citable document. It is based on data we believe to be reliable. It is offered in good faith but without guarantee.

RAPID KJELDAHL
BENCHNOTES

Rapid Kjeldahl Methodology for the Determination of Total Kjeldahl Nitrogen in Water with the Rapid Digestor and the RapidStill II

Principle: *This method covers the determination of free ammonia and organic nitrogen in drinking water, surface and saline water, and domestic and industrial wastes.*

Apparatus

1. Labconco Rapid Digestor:
   Rapid Digestor 4 (Model 23080)
   Rapid Digestor 25 (Model 23000, 23006, 23012)

2. Labconco Fume Removal System:
   Fume Removal System-4 place (Model 23540)
   Fume Removal System-25 place (Model 23500-25)

3. Labconco Rapid Distillation Unit:
   RapidStill II (Model 65200)

4. Labconco 250 ml Digestion Tubes:
   Volumetric, Pkg. of 5 (Model 23030-05)
   Volumetric, Pkg. of 25 (Model 23030-25)
   Straight, Pkg. of 5 (Model 23040-05)
   Straight, Pkg. of 25 (Model 23040-25)

5. 250 ml Erlenmeyer flasks or flat bottomed Florence flasks

6. Spectrophotometer with sensitiviy less than 5 ppm

Reagents

(All solutions must be made with ammonia-free water)

1. Mercuric sulfate solution: Dissolve 8 g mercuric oxide (HgO) in 50 ml 1:4 sulfuric acid (10 ml H_2SO_4: 40 ml deionized water) and dilute to 100 ml with deionized water.

2. Sulfuric acid-mercuric sulfate solution (digestion solution): Dissolve 267 g potassium sulfate (K_2SO_4) in 1300 ml deionized water and 400 ml concentrated H_2SO_4. Add 50 ml mercuric sulfate solution in step 1 and dilute to two liters with deionized water.

3. Sodium hydroxide/potassium sulfide solution: Add 500 g sodium hydroxide (NaOH) and 10 g potassium sulfide (K_2S) to 700 ml water in a one liter container on a magnetic stirrer in a well-ventilated area. Agitate until dissolved, then dilute to one liter with deionized water. Cool before using.

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4. Boric acid/indicator solution: Add 20 g boric acid (H_3BO_3) to 800 ml of water, dissolve and dilute with deionized water to one liter. Keep covered to prevent carbon dioxide (CO_2) contamination. Premix two volumes of .2% methyl red in 95% ethanol with one volume of .2% methylene blue (prepare fresh every 30 days). Add 10 ml of the indicator solution to boric acid solution if performing titrimetrically.

5. 0.02N standard sulfuric acid.

6. Nessler reagent: In deionized water, dissolve 100 g mercuric iodide (HgI) and 70 g potassium iodide (KI). Add this slowly to a cooled solution containing 160 g NaOH diluted in 500 ml of deionized water and dilute with deionized water to one liter.

7. Ammonium chloride (stock solution): 1.0 ml = 1.0 mg NH_3-N. Dissolve 3.819 g NH_4Cl in deionized water and dilute to 1 liter in a volumetric flask.

8. Ammonium chloride (standard solution): 1.0 ml = 0.01 mg NH_3-N. Dilute 10.0 ml of the stock solution in step 7 to one liter with deionized water .

Procedure

1. Place the Rapid Digestor under a fume hood near a water source, aspirator and a cup sink. Preheat to 200° C.

2. Place an identifying label or mark on each 250 ml digestion tube.

3. Place tubes in the digestion rack.

4. To each tube add 4-5 alundum boiling stones and the volume recommended below:

TKN in Sample(mg/l)	Sample(ml)	Volume of Digestion(ml)
0-20	100	20
20-50	50	10
50-500	25	10

5. Place the rack containing the tubes on the preheated digestion block. Attach heat shields. Pre-evaporate at 110° C for 30 minutes to avoid splashing. Turn the temperature control knob to 380° C. Conduct the evaporation phase for 60 minutes (or until evaporation is complete) without using the Fume Removal System. After sulfur trioxide (SO_3) gas evolves, conduct the digestion phase for 30 minutes with the Fume Removal System in place.

6. Make sure digestion sample is clear. If sample is not a clear liquid, digest longer unitl sample is clear.

7. Allow to cool. Cooling times depend on the temperature and circulation of air around the tubes and may require adjustment depending on your conditions. Do not allow hard cakes to form.

8. Add 50 ml of deionized water to each digestion tube and keep covered.

9. Determine the nitrogen content titrimetrically or colorimetrically.

Titrimetric Method

1. Add approximately 50 ml of 2% boric acid/indicator solution to each receiving flask.

2. Twist the distillation tube securely in place on the RapidStill II. Place an Erlenmeyer flask containing the receiving solution (Reagent step 4) at the receiving end with the delivery tube submerged in it.

3. Add 40 ml of NaOH for every 20 ml of digestion solution used. You will be collecting approximately 100-125 ml of distillate.

4. Set the RapidStill II timer for 8 minutes.

5. Start the distillation process. Collect approximately 125 ml of distillate.

6. When the distillation is complete, fill a class A buret to the mark with 0.02N H_2SO_4 and titrate to the purple endpoint.

$$\text{ppm N} = \frac{(\text{ml acid - ml acid of blank})(\text{N } H_2SO_4)(14.007)}{\text{Liters of sample}}$$

$$\text{ppm ammonia} = \frac{(\text{ml acid ml acid blank})(\text{N } H_2SO_4)(17.03)}{\text{Liters of sample}}$$

Colorimetric Method

1. When distillation is complete:
   Prepare Nessler tubes from a standard ammonium solution.
   a. Stock solution: Dissolve 3.819 g anhydrous NH_4Cl in water and dilute to one liter with deionized water.
   b. Standard solution: Dilute 10.0 ml of the stock ammonia solution to one liter with deionized water.

2. From the standard solution, prepare a series of Nessler standards by diluting the volumes given in the following chart to 50 ml in ammonia-free water and adding one ml of Nessler reagent.

ml Sample	mg NH3-N/50 ml
0	0
0.5	0.005
1.0	0.010
2.0	0.020
4.0	0.040
5.0	0.050
8.0	0.080
10.0	0.10

3. Mix thoroughly.

4. After 30 minutes, read the absorbance at 420 nm against a blank.

5. Place a blank in spectrophotometer. Read absobance. Adjust absorbance to read zero. Alternate samples and blanks, readjusting blank to zero each time.

6. Plot a calibration curve of absorbance vs mg NH_3-N.

7. Measure the volume of the distillateand reveiving solution and record it. Pipet 50 ml of the distillate and add one ml of the Nessler reagent to develop the color. Measure the absorbance after 30 minutes, and from the calibration curve, read the mg NH_3-N.

$$\text{TKN, mg/L} = \frac{A \times 1000}{\text{ml sample}} \times \frac{B}{C}$$

where:
A = mg NH_3-N read from curve
B = ml total distillate measured
C = ml of original sample

Notes

1. It is a good practice to regularly check procedural accuracy with standards such as those supplied by the EPA.

2. Twenty ml of digestion reagent solution appears to work well for levels of nitrogen up to about 150 ppm.

3. When titrating, titrate to purple endpoint. Do not titrate all the way back to red.

4. Run a blank sample (reagents only, no sample) and subtract the milliliters of H_2SO_4 consumed by the blank from that consumed by the sample.

5. With careful technique, this equipment will give results with a relative standard deviation of no greater than 1.5%.

6. The Nessler reagent is stable for at least a year if stored in a rubber stoppered brown bottle.

7. Use reagent blank to zero spectrophotometer.

8. If splashing occurs during digestion (Step 5) pre-evaporate for 30 minutes at 100° C.

References

EPA Methods for Chemical Analysis of Water and Wastes. Cincinnati, OH: (1983) • pp 351.3-1 to 351.3-6.

Official Methods of Analysis AOAC. 15th ed. Arlington, VA: (1990) • 7.026, 7.031, 7.032, 24.038-24.040, and 47.023.

Standard Methods for the Examination of Water and Wastewater. 16th ed. Washington, D.C.: (1985) • pp 358-410.

RAPID KJELDAHL
BENCHNOTES

Rapid Kjeldahl Methodology for the Determination of Nitrogen in Meat Products With the Rapid Digestor and the RapidStill II

Principle: *This method covers the determination of organic nitrogen and protein in meats.*

Apparatus

1. Labconco Rapid Digestor:
   Rapid Digestor 4 (Model 23080)
   Rapid Digestor 25 (Model 23012)

2. Labconco Fume Removal System:
   Fume Removal System-4 place (Model 23540)
   Fume Removal System-25 place (Model 23500-25)

3. Labconco Rapid Distillation Unit:
   RapidStill II (Model 65200)

4. Labconco 250 ml Digestion Tubes:
   Volumetric, Pkg. of 5 (Model 23030-05)
   Volumetric, Pkg. of 25 (Model 23030-25)
   Straight, Pkg. of 5 (Model 23040-05)
   Straight, Pkg. of 25 (Model 23040-25)

5. Hand chopper, food processor, or blender

6. 500 ml Erlenmeyer flasks

Reagents

(All reagents should be reagent grade and nitrogen-free.)

1. Red mercuric oxide (HgO) or cupric sulfate ($CuSO_4$ or $CuSO_4 \cdot 5H_2O$)

2. Potassium sulfate (K_2SO_4) suitable for nitrogen determination

3. Sulfuric acid (H_2SO_4) - concentrated

4. Sodium hydroxide/potassium sulfide: Dissolve 450 g solid sodium hydroxide (NaOH) in deionized water. While still warm, dissolve 10 g potassium sulfide (K_2S) in the sodium hydroxide solution.

5. Methyl red/methylene blue indicator: Dissolve 200 mg methyl red in 100 ml ethyl alcohol. In a separate beaker, dissolve 200 mg methylene blue in 100 ml ethyl alcohol. Mix 2 volumes of methyl red with 1 volume methylene blue solution.

6. Boric acid/indicator solution: Dissolve 20 g boric acid (H_3BO_3) in 800 ml of deionized water. Add 10 ml of the indicator solution in step 5 and dilute to one liter with deionized water. NOTE: potassium sulfide is only required with mercury catalyst.

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7. 0.2N Sulfuric acid standard solution (volume depends on % N expected)

8. Alundum boiling stones

Procedure

1. Preheat the Rapid Digestor to 410° C.

2. Place preweighed (up to 2 g), finely ground homogenous samples into digestion tubes.

3. Add 0.42 g HgO, 9.0 g K_2SO_4, 15 ml H_2SO_4 and 10-12 boiling stones to the digestion tubes.

4. Place samples in the Rapid Digestor and digest for 45 minutes. Make sure digestion sample is clear. If sample is not a clear liquid, digest longer until sample is clear.

5. Remove tubes from the Rapid Digestor and cool for 10 minutes. Do not allow hard cakes to form.

6. Add 50 ml deionized water and dissolve all crystallized material.

7. After the sample has cooled, attach digestion tube to the distillation head of the RapidStill II.

8. Place distillation outlet tube in 500 ml Erlenmeyer flask which contains 100 ml boric acid/indicator solution. The tip must be below the surface of the boric acid/indicator solution.

9. Add 60 ml sodium hydroxide solution to the digestion tube.

10. Set the RapidStill II timer for 8 minutes.

11. Start distillation and steam distill vigorously until 125 ml of distillate is collected or a total volume of 225 ml is in receiving flask.

12. When distillation is complete, fill a class A buret to the mark with .2N sulfuric acid (H_2SO_4) standard solution and titrate to the purple endpoint. Correct for reagent blank.

$$\%N = \frac{(\text{ml std. } H_2SO_4 - \text{ml } H_2SO_4 \text{blank})(N \bullet H_2SO_4)(1.4007)}{\text{gram sample weight}}$$

$$\%\text{Protein} = \%N \times 6.25$$

Example:
blank = 1 ml sample weight = 1 gram
acid added = 11 ml

$$\%N = \frac{(11 \text{ ml} - 1 \text{ ml})(0.2N\ H_2SO_4)(1.400)}{1 \text{ gram}}$$

%N = 2.8%
%Protein = 17.5%

Notes

1. Samples with high moisture content may need to be preheated to a lower temperature (200° C) for 15 minutes to boil off the moisture and then taken to 410° C.

2. When titrating, titrate to the purple endpoint. Do not titrate all the way back to red.

Reference

Official Methods of Analysis AOAC. 15th ed. Arlington, VA: (1990).

RAPID KJELDAHL
BENCHNOTES

Rapid Kjeldahl Methodology for the Determination of Nitrogen in Feeds, Foods, Grains, Cereals, and Grasses with the Rapid Digestor and RapidStill II

Principle: *This method covers the determination of organic nitrogen in feeds, foods, grains, cereals, and grasses.*

Apparatus

1. Labconco Rapid Digestor:
   Rapid Digestor 4 (Model 23080)
   Rapid Digestor 25 (Model 23012)

2. Labconco Fume Removal System
   Fume Removal System-4 place (Model 23540)
   Fume Removal System-25 place (Model 23500-25)

3. Labconco Rapid Distillation Unit:
   RapidStill II (Model 65200)

4. Labconco 250 ml Digestion Tubes:
   Volumetric, Pkg. of 5 (Model 23030-05)
   Volumetric, Pkg. of 25 (Model 23030-25)
   Straight, Pkg. of 5 (Model 23040-05)
   Straight, Pkg. of 25 (Model 23040-25)

5. 25 ml Class A buret

6. 500 ml Erlenmeyer flasks

Reagents

(All reagents should be reagent grade and nitrogen-free)

1. Red mercuric oxide (HgO) or cupric sulfate ($CuSO_4$ or $CuSO_4 \cdot 5H_2O$)

2. Potassium sulfate (K_2SO_4) - suitable for nitrogen determination.

3. Sulfuric Acid (H_2SO_4) - concentrated, 95-98%.

4. Sodium hydroxide/potassium sulfide solution: Dissolve 450 g solid sodium hydroxide (NaOH) in deionized water. While still warm, dissolve 10 g potassium sulfide (K_2S) in the sodium hydroxide solution. Cool and dilute with deionized water to one liter. **NOTE: Potassium sulfide is only required with mercury catalyst.**

5. Methyl red/methylene blue indicator: Dissolve 200 mg methyl red in 100 ml ethyl alcohol. In a separate beaker, dissolve 200 mg methylene blue in 100 ml ethyl alcohol. Mix 2 volumes methyl red with 1 volume methylene blue.

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6. Boric acid/indicator solution: Dissolve 20 g boric acid (H_3BO_3) in 800 ml deionized water. Add 10 ml methyl red/methylene blue indicator solution and dilute to one liter with deionized water.

7. 0.2 N sulfuric acid standard solution (volume depends on % N expected).

8. Alundum boiling stones.

Procedure

1. Preheat Rapid Digestor to 410° C.

2. Place weighed (approximately 1 g) finely ground homogeneous samples into digestion tubes.

3. Add 0.42 g HgO, 9.0 g K_2SO_4, 15 ml H_2SO_4 to the digestion tubes.

4. Place samples in Rapid Digestor and digest for 45 minutes or until white smoke comes off.

5. Make sure digestion sample is clear. If necessary, digest longer until sample is a clear liquid.

6. Remove tubes from Rapid Digestor and cool for 10 minutes. Do not allow hard cakes to form. If cakes form, reheat samples until cakes are dissolved.

7. Add 50 ml deionized water and dissolve all crystallized material.

8. After the sample has cooled, attach digestion tube to the distillation head and twist tightly in place.

9. Place delivery tube in 500 ml Erlenmeyer flask which contains 100 ml boric acid/indicator solution. The tip must be below the surface of the boric acid/indicator solution.

10. Add 60 ml sodium hydroxide solution to the digestion tube. (Total volume in the Erlenmeyer flask will be 225 ml.)

11. Set the RapidStill II timer for 8 minutes.

12. Start the distillation process. Distill until 125 ml of distillate collects.

13. When distillation is complete, fill a class A buret to the mark with .2N sulfuric acid standard solution and titrate to the purple endpoint. Correct for reagent blank.

$$\%N = \frac{(\text{ml std. } H_2SO_4 - \text{ml } H_2SO_4 \text{ blank})(\text{N } H_2SO_4)(1.4007)}{\text{gram sample weight}}$$

Example:
blank = 1 ml sample weight = 1 gram
acid added = 11 ml

$$\%N = \frac{(11 \text{ ml} - 1 \text{ ml})(0.2\text{N } H_2SO_4)(1.400)}{1 \text{ gram}}$$

%N = 2.8%
%Protein = 17.5%

Notes

1. Run a blank sample (reagents only, no sample) and subtract the milliliters of H_2SO_4 consumed by the blank from that consumed by the sample.

2. When titrating, titrate to purple endpoint. Do not titrate all the way back to red.

3. With careful technique, this equipment will give results with a relative standard deviation of no greater than 1.5%.

Reference

Official Methods of Analysis AOAC. 15th ed. Arlington, VA: (1990).

RAPID KJELDAHL
BENCHNOTES

Rapid Kjeldahl Methodology for the Determination of Protein Content in Milk with the Rapid Digestor and the RapidStill II

Principle: *This method covers the determination of organic nitrogen in fluid and dry milk.*

Apparatus

1. Labconco Rapid Digestor:
   Rapid Digestor 4 (Model 23080)
   Rapid Digestor 25 (Model 23012)

2. Labconco Fume Removal System:
   Fume Removal System-4 place (Model 23540)
   Fume Removal System-25 place (Model 23500-25)

3. Labconco Rapid Distillation Unit:
   RapidStill II (Model 65200)

4. Labconco 250 ml Digestion Tubes:
   Volumetric, Pkg. of 5 (Model 23030-05)
   Volumetric, Pkg. of 25 (Model 23030-25)
   Straight, Pkg. of 5 (Model 23040-05)
   Straight, Pkg. of 25 (Model 23040-25)

5. One 25 ml class A buret

6. 500 ml Erlenmeyer flasks

Reagents:

(All reagents should be reagent grade and nitrogen-free.)

1. Sulfuric acid (H_2SO_4) - concentrated

2. Red mercuric oxide (HgO) or cupric sulfate($CuSO_4$ or $CuSO_4 \cdot 5H_2O$)

3. Potassium sulfate (K_2SO_4) suitable for nitrogen determination

4. 45% sodium hydroxide: Add 450 g sodium hydroxide (NaOH) to 700 ml deionized water. Add 10 g potassium sulfide (K_2S), dissolve, and dilute to one liter with deionized water. NOTE: Potassium sulfide is only required with mercury catalyst.

5. Methyl red/methylene blue indicator: Dissolve 200 mg (0.2 g) methyl red in 100 ml ethyl alcohol. In a separate beaker, dissolve 200 mg methylene blue in 100 ml ethyl alcohol. Combine two parts methyl red with one part methylene blue solution and mix.

6. Boric acid/indicator solution 1%: Dissolve 10 g boric acid (H_3BO_3) in 800 ml deionized water. Add 10 ml methyl red/methylene blue indicator solution and dilute to one liter with deionized water.

7. 0.1N sulfuric acid (H_2SO_4) solution.

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Procedure

1. Place the Rapid Digestor under a fume hood near a water source, aspirator and a cup sink. Preheat to 410° C.

2. First weigh the weigh boat and then weigh the samples. AOAC recommends 5 ±0.1 ml for liquid milk or approximately 1 g for dry milk. Transfer the sample from the weigh boat into the digestion tube.

3. Place the tubes in the digestion rack of the Rapid Digestor.

4. To each tube add 0.42 g HgO, 9 g K_2SO_4, 15 ml H_2SO_4 and 3-4 alundum boiling stones.

5. When the light on the Rapid Digestor begins blinking, place the rack containing the digestion tubes on the Rapid Digestor block. Attach heat shields. On the top of the tubes, place the Fume Removal System and connect to the water aspirator. Turn on the water to the water aspirator and digest for 45 minutes.

6. After 45 minutes, remove the tubes from the block. Make sure digestion sample is clear. If sample is not a clear liquid, digest longer until sample is clear.

7. With the Fume Removal System still in place, set the tube-filled rack on a hard, dry, heat-resistant surface. Remove the heat shields and cool for ten minutes. Cooling times depend on the temperature and circulation of air around the tubes and may require adjustment depending on conditions. Do not allow hard cakes to form.

8. Add 50 ml deionized water to each tube, dissolving all crystallized material. It may be necessary to place tubes back in the Rapid Digestor, to expedite dissolving the crystallized material. Cover to prevent contamination.

9. After the sample has cooled, attach digestion tube to the RapidStill II and twist securely in place.

10. Place delivery tube in 500 ml Erlenmeyer flask which contains approximately 100 ml boric acid/indicator solution. The tip must be below the surface of the boric acid/indicator solution.

11. Set the RapidStill II timer for 8 minutes.

12. Start the distillation process.

13. Steam distill until approximately 125 ml distillate collects, or a total of 225 ml is collected.

14. When distillation is complete, fill a class A buret to the mark with 0.1N sulfuric acid (H_2SO_4). Titrate drop by drop to the purple endpoint. Correct for reagent blank.

$$\%N = \frac{(\text{ml } H_2SO_4 - \text{ml } H_2SO_4 \text{of blank})(\text{N } H_2SO_4)(1.4007)}{\text{gram sample weight}}$$

Example:
For a 5.0 g sample of milk containing 3.0% protein, 16.78 ml 0.1N H_2SO_4 will be required for titration.

Notes

1. Run a blank sample (reagents only, no sample) and subtract the milliliters of H_2SO_4 consumed by the blank from that consumed by the sample.

2. When titrating, titrate to the purple endpoint. Do not titrate all the way back to red.

3. With careful technique, this equipment will give results with a relative standard deviation of no greater than 1.5%.

Reference

Official Methods of Analysis AOAC. 15th ed. Arlington, VA: (1990) • 16.036, 7.025 - 7.032, 24.038 - 24.040.

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