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# User's Manual

## Two & Six-Place (Macro) Kjeldahl Distillation Systems

### Models

2128501

2127601

2127602

2127603

To receive important product updates,  
complete your product registration card  
online at [register.labconco.com](https://register.labconco.com)

Please read the User's Manual before operating the equipment.

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## **Warranty**

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The warranty for Two and Six-Place Kjeldahl Distillation Systems will expire one year from date of installation or two years from date of shipment from Labconco, whichever is sooner. Warranty is non-transferable and only applies to the owner (organization) of record.

Buyer is exclusively responsible for the set-up, installation, verification, decontamination or calibration of equipment. This limited warranty covers parts and labor, but not transportation and insurance charges. If the failure is determined to be covered under this warranty, the dealer or Labconco Corporation will authorize repair or replacement of all defective parts to restore the unit to operation. Repairs may be completed by 3<sup>rd</sup> party service agents approved by Labconco Corporation. Labconco Corporation reserves the rights to limit this warranty based on a service agent's travel, working hours, the site's entry restrictions and unobstructed access to serviceable components of the product.

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## **Returned or Damaged Goods**

Do not return goods without the prior authorization from Labconco. Unauthorized returns will not be accepted. If your shipment was damaged in transit, you must file a claim directly with the freight carrier. Labconco Corporation and its dealers are not responsible for shipping damages.

The United States Interstate Commerce Commission rules require that claims be filed with the delivery carrier within fifteen (15) days of delivery.

## **Limitation of Liability**

The disposal and/or emission of substances used in connection with this equipment may be governed by various federal, state, or local regulations. All users of this equipment are required to become familiar with any regulations that apply in the user's area concerning the dumping of waste materials in or upon water, land, or air and to comply with such regulations. Labconco Corporation is held harmless with respect to user's compliance with such regulations.

## **Contacting Labconco Corporation**

If you have questions that are not addressed in this manual, or if you need technical assistance, contact Labconco's Customer Service Department or Labconco's Product Service Department at 1-800-821-5525 or 1-816-333-8811, between the hours of 7:30 a.m. and 5:30 p.m., Central Standard Time.

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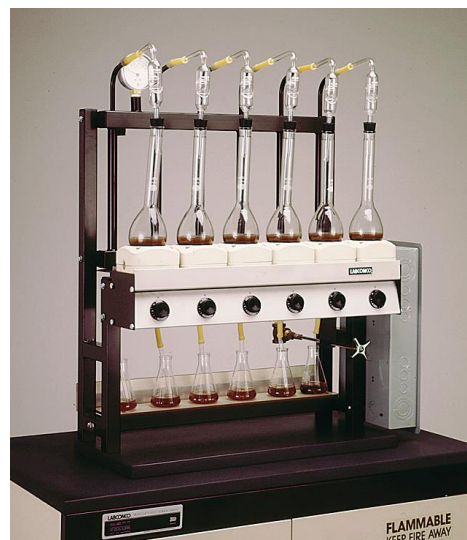
# Chapter 1: Introduction

## Components Shipped

Carefully check the contents of your Two-Place or Six-Place Kjeldahl Distillation System for shipping damage while it is still on the shipping pallet. Do not discard the packaging material until the contents have been checked and the equipment has been approved for installation.

The Two-Place or Six-Place Kjeldahl Nitrogen Distillation System has been shipped in one complete crate to minimize damage that may occur in transit.

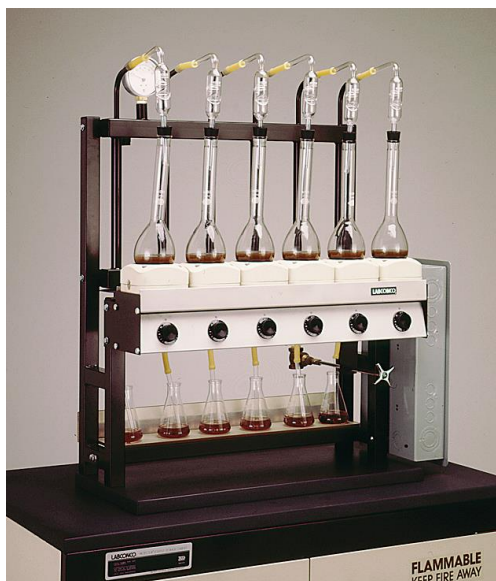
Make sure to inspect the product thoroughly **prior** to installation and report any damage that may have occurred in transit (see Warranty page for instructions).



## General Description

Labconco's Two and Six-Place (**Macro**) Kjeldahl Distillation Systems are designed to facilitate the determination of Total Nitrogen content within materials such as feeds, grains, soils, fertilizers, plant tissue, water, organic wastes, and food products. The expression '**Macro**' identifies the size of sample vessel and the sample size that can be used with this equipment. Other Kjeldahl systems are available, which use smaller sample vessels and require smaller sample sizes. This apparatus can be used for the distillation of all types of nitrogen containing samples. The Two and Six-place Distillation Systems can be used to perform a standardized Macro-Kjeldahl Nitrogen Determination Method.

The Kjeldahl Distillation Systems are not enclosed, excess heat and personnel safety during the distillation reaction must be considered when selecting an installation location. The Kjeldahl Distillation Method is a 'Closed Distillation,' no chemical fumes are released during the procedure. The Distillation procedure does involve boiling concentrated sodium hydroxide at high temperatures. Labconco recommends placing Two and Six-Place Distillation Systems inside a chemical fume hood for safety. Labconco recommends a splash shield be placed in front of the Distillation Systems if they are installed on the open lab bench.



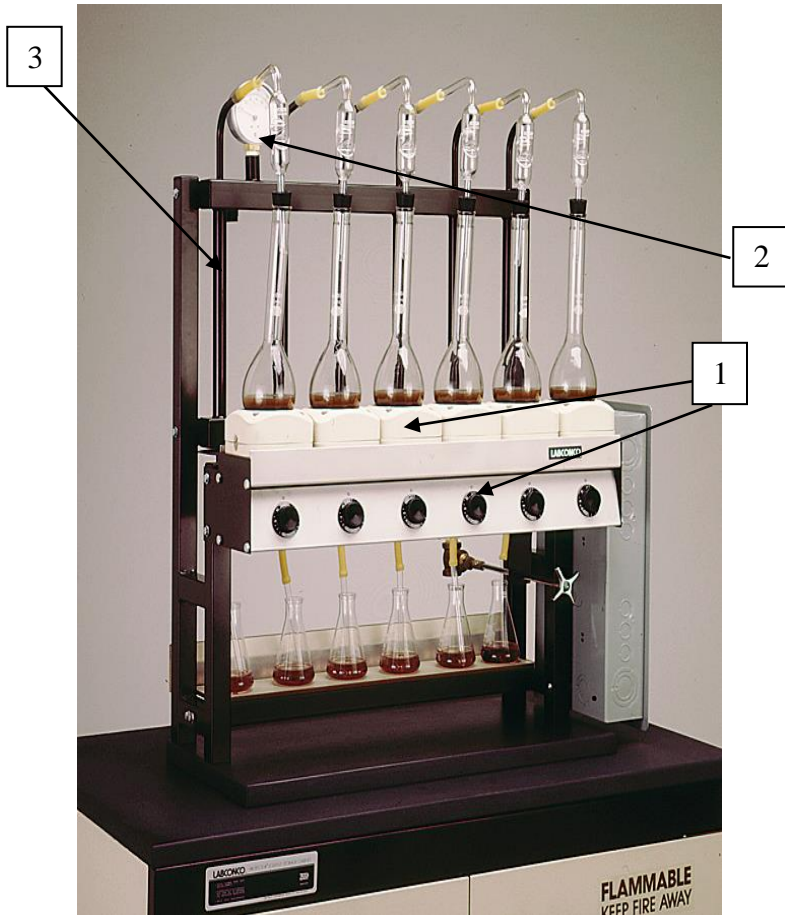
**2128501 & 2127601 Two-Place and Six-Place Kjeldahl Distillation Systems**

## Performance

The Two and Six-Place Kjeldahl Distillation Systems have been designed for use in the determination of nitrogen (or ammonia) concentrations. By calculation, nitrogen can be converted to protein values for products such as plant tissues, meats and other food substances. Nitrogen determinations with the (**Macro**) Distillation Systems can accommodate sample sizes up to 300-400 mls, due to the size of the distillation flasks. Detailed procedures developed by professional laboratory organizations such as American Association of Analytical Chemists (AOAC) and American Association of Cereal Chemists (AACC) should always be consulted for step by step analytical procedures when operating this equipment. For methodology precision and accuracy please consult the specific published method for the type of sample or substance that will be analyzed.

## Component Identification

1. **Electric Heaters.** 600-watt heaters are used in the distillation rack of the units. Infinite control switches regulate each of the heaters. The curved heater elements are provided and allow for the round base of the Kjeldahl flasks, providing faster and more evenly distributed heat. All heaters and temperature controllers are wired to a common subpanel electrical box, for easy electrical connection to a facility electrical circuit.
2. **Temperature Gauge.** The condensation rack behind the distillation heating elements requires cooling water. The temperature of the distillation cooling water on the Six-Place Distillation System can be adjusted to promote condensation and suit individual requirements with the flow control valve handle, located below the distillation heaters. Water temperature is indicated on the thermometer, which is located at the water outlet of the distillation manifold. The Two-Place Distillation Systems does not feature a flow control valve or temperature gauge. The cooling water connections are 3/4" NPT and the supply line should be 1/2" minimum for proper water flow through the unit. For facilities where the ground water temperature exceeds 80°F, a recirculation water chiller system will be required to achieve sufficient condensation results.
3. **Distillation Condensation Manifold.** Epoxy coated steel outer columns with stainless steel inner condensation columns provide long life.



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# Chapter 2:

## Installation

### General Installation Notes

The Two and Six-Place Kjeldahl Distillation Systems are shipped fully assembled. The Two-Place Kjeldahl Distillation System water supply and drain can be connected by the customer to a common laboratory faucet. The Two-Place Kjeldahl Distillation System comes with an 8-foot, 3 wire cord and plug.

The Six-Place Kjeldahl Distillation System will require electrical and water supply/drain connections which should be made by a licensed contractor. The customer will need to purchase the required Kjeldahl flasks, with rubber stoppers and distillate receiving flasks. The glass connecting bulbs and delivery tubes are provided with each Kjeldahl Distillation System.

**Connecting Bulbs and Delivery Tubes are included with Macro Kjeldahl Systems. For installation of the Connecting Bulbs and Delivery Tubes see section on Glassware Installation.**



Use caution when you remove the protective packaging material taped to the heater runways as fragile components are involved. Levelers are shipped installed, as are the distillation cooling water thermometers.

### Location

The Two-Place Kjeldahl Distillation System can be carried by hand to its final location.

The Six-Place Kjeldahl Distillation System is heavy. Special arrangements should be made in advance for moving the system to its final location. Special instruction tags are attached to the apparatus; they must not be removed until the installation has been completed.



The Two and Six-Place Kjeldahl Distillation Systems do not include a surrounding safety enclosure. Labconco advises installing these products in a fume hood, or if installed on the open laboratory bench, a safety shield should be placed in front of the burners. High heat and boiling Sodium Hydroxide present a safety hazard to personnel in close proximity to the Kjeldahl Distillation Systems. Labconco also recommends spot exhaust ventilation for an open bench installation to remove excess heat produced by the heaters.

### Electrical

The Two-Place Kjeldahl Distillation System comes with an 8-foot, 3 wire cord and plug and can be connected to a 115 volt 60Hz 11 Amp circuit.

The Six-Place Distillation Kjeldahl System has been wired at the factory per the product model number ordered by the customer. Labconco model numbers ending in 01 are 115 volts. Model numbers ending in 02 are 230 volts. Models ending in 03 are 230 volt, three phase. Amperage requirements vary depending on the number of heaters per the Distillation rack. The voltage and amperage table below provides the electrical requirements for the Six-Place Distillation Systems. A qualified electrician will need to connect the facility electrical supply to the main breaker box of the Digestion System.

1. Remove circuit breaker box cover panel.
2. Main line lead connection terminals are identified and connections must be made accordingly.
3. Line leads to the apparatus must conform to local electrical codes.
4. Provide an electrical ground to the apparatus per code.
5. Before applying power to apparatus, check the electrical panel and breakers for loose connections.
6. Reinstall box cover panel.
7. Power the breaker box and reset breakers to check circuits in the apparatus.
8. All electrical wiring and connections must conform to local codes and should be performed by qualified electricians. An earth ground must be provided.

#### Six-Place Distillation System Electrical Code

Code #	AC Electrical Codes
01	115 volt, single phase, 50/60 Hz, 32 amps
02	230 volt, single phase, 50/60 Hz, 16 amps
03	208/230 volt, three phase, 50/60 Hz, 9 amps

## Tap Water Supply

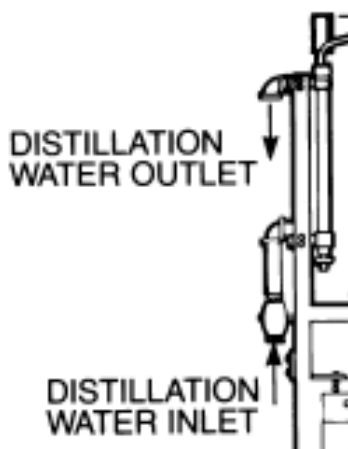
**NOTE: ALL PLUMBING CONNECTIONS AND COMPONENTS MUST BE FREE OF FOREIGN MATERIAL BEFORE FINAL CONNECTIONS ARE MADE.**

## Distillation Manifold

The Kjeldahl Distillation Systems feature an upper distillation rack with individual condensation columns, these are connected together by a common manifold (6 columns per manifold). The tap water inlet and outlet connections are 3/4" NPT and the supply line should be minimum 1/2" ID for proper water flow and heat removal capacity through the condensation columns (6-8 gallons/23-30 liters per minute).

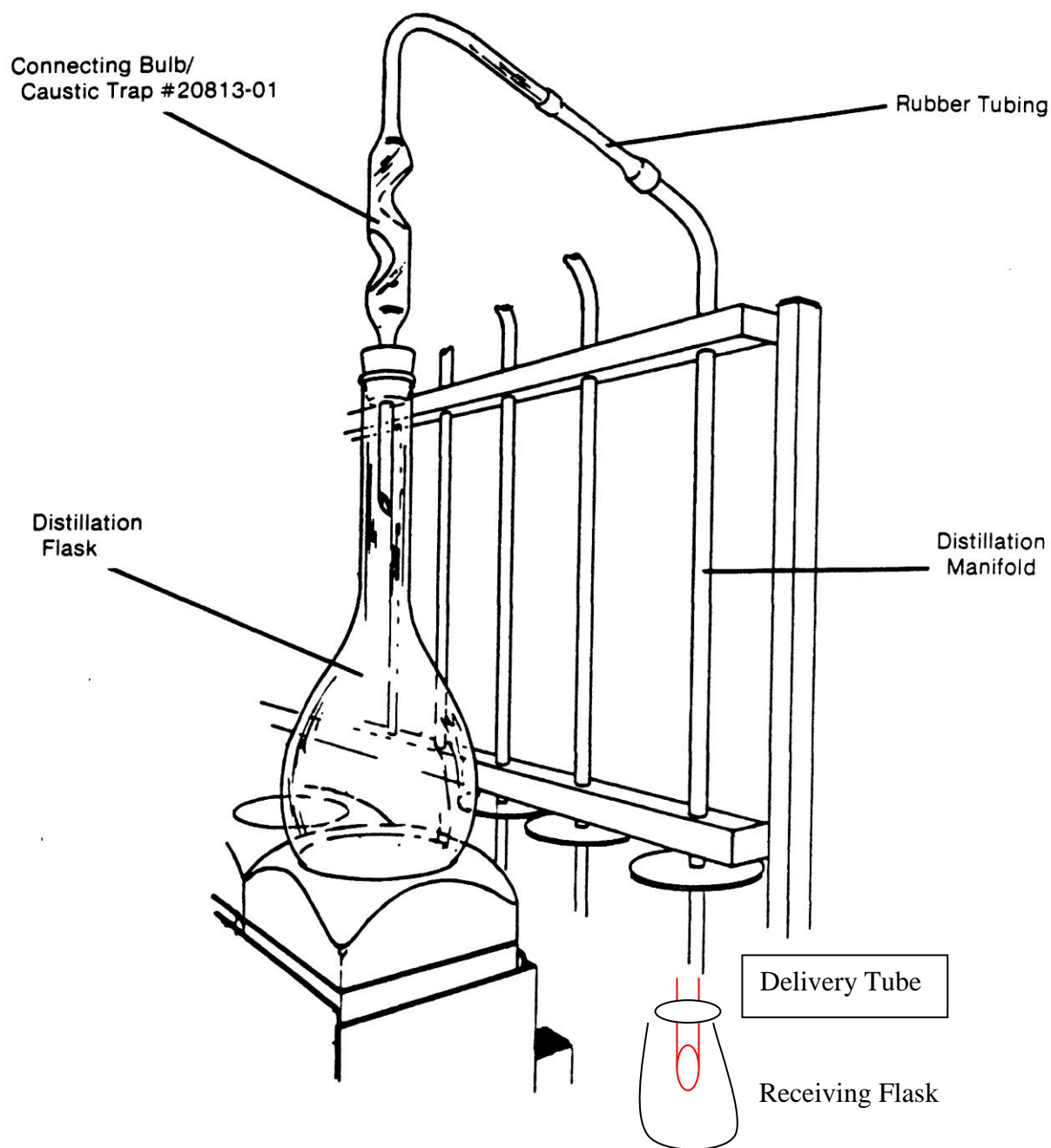
The plumbing line for the outlet of the distillation manifold must be free of back pressure to avoid flow restriction of the cooling water. If the Kjeldahl Distillation System is equipped with the Acid Fume Water Aspirator Exhaust System option, the distillation water drain should not be connected to the aspirator drain, unless provisions are made to handle the larger water volumes without water backing up over the aspirator vent line.

Water discharged from the distillation manifold is **not** acid contaminated and standard drain lines may be used for its removal.



**Distillation Manifold Tap Water Connections**

## Glassware Installation



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## Chapter 3:

# Normal Operation & Routine Maintenance

### Normal Operation Start-Up

1. The KNA apparatus is designed for use with either 500 or 800-ml Kjeldahl Flasks. The distillation heaters are free to move forward or backward on the runway to accommodate either size flask.
2. Turn the heat controllers to the high setting on both the Digester and Distillation racks if both racks will be used simultaneously. Wait approximately 5 to 10 minutes to insure proper preheating.
3. Turn on either the acid fume removal blower or water aspirator (depending upon model number purchased). Place a small piece of tissue paper over the nipple on the Distillation manifold. Suction should be sufficient to hold 2" x 2" tissue paper in place.

A generalized Kjeldahl Total Nitrogen method is present in Appendix C. The method does not detail sample preparation or preservation procedures, which may be required depending on specific types of samples.



### **Clean-Up and Cosmetic Guidelines**

- Keeping the Kjeldahl Distillation System clean will preserve the appearance and improve life of the equipment.
- The equipment can be kept clean by washing with a weak solution of sodium hydroxide and rinsing with clear water.
- Sulphate, a salt of Sulfuric Acid, may build up between the channel support and the fume removal manifold. Neutralize this with a weak solution of sodium hydroxide periodically to keep the unit clean.

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# Appendix A:

## Replacement Parts

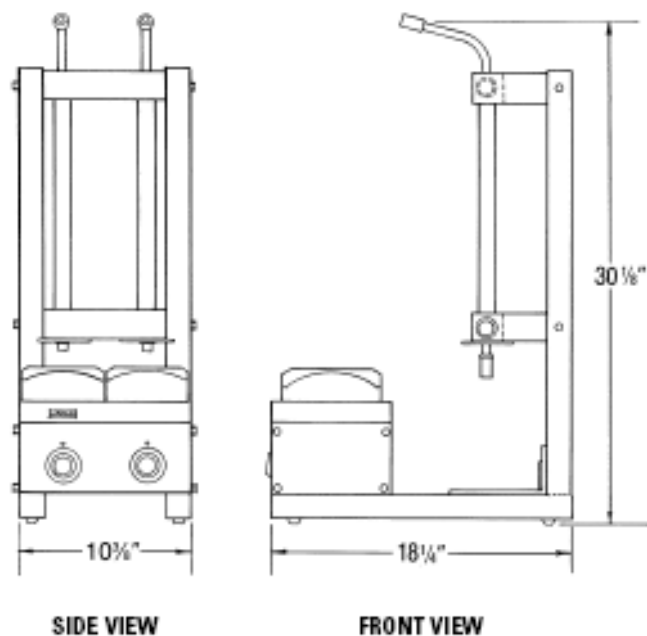
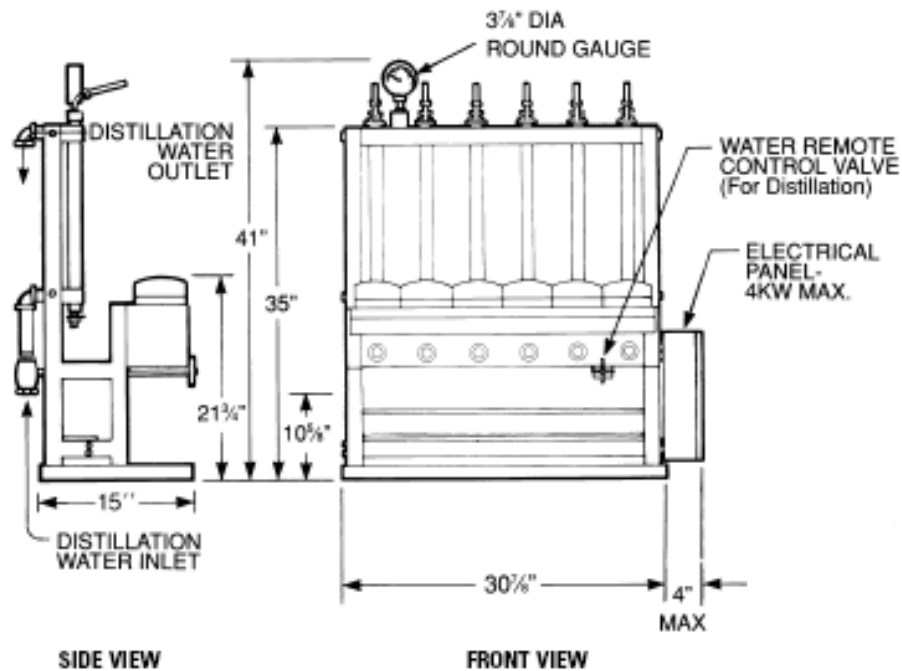
### Electrical Heaters and Motor/Blower Parts

<u>Part Number</u>	<u>Description</u>
1315400	Heater Control, Infinite (115Volts)
1315500	Heater Control, Infinite (230Volts)
2011500	Core Plate Casting, (package of 6)
2023200	Heater Base Casting (electric or gas)
2033100	Heater element, 115V, 600 W, (package of 6)
2033200	Heater element, 230V, 600 W, (package of 6)
2023100	Heater Top ceramic casting (electric or gas)
2031800	Heater lead wire assembly, (package of 6 pairs)
1870200	Knob for Infinite Heater Control
1317100	Blower Motor Switch
1850500	Adjustable sheave (pulley) for belt drive blower only
2053900	Blower housing assembly (plastic housing only)
1200000	Blower motor 1/3 H.P., 115V, 60 Hz
1203200	Blower motor 1/3 H.P., 230V, 60 Hz
1203600	Blower motor 1/3 H.P., 230V, 50 Hz
1851800	Blower shaft bearing (belt drive only)
2054500	Blower bearing assembly
1451300	Blower wheel
2056600	Ceramic nipple
2055400	Cover, blower wheel
2055500	Blower Shaft (belt drive only)
1851100	V-belt (drive only)
2053000	Water ejector nozzle
1967000	Clamp, hose
2144600	Flex sleeving
1662200	Grommet 1/2 ID x 1-1/2 OD
2165800	PTFE nipple
1880128	Plastic screws for PTFE nipple
2164000	Motor shield

## Miscellaneous Replacement Parts

<b><u>Part Number</u></b>	<b><u>Description</u></b>
1620500	Tubing, rubber condenser connecting, per foot
2152407	Distillation manifold – 6 place stainless steel
2038800	Wire, No 14 Black, SEWF-2, 30 ft length
2038900	Wire No. 14 White, SEWF-2, 30 ft length
2031700	Heater terminal assembly, package of 24
2146400	Assembly inlet & outlet manifold (12 unit only)
2081300	Connector bulb/caustic trap, package of 6
2128800	Delivery tube, package of 6
2078800	Kit, replacement fume pipe to blower housing

## Appendix B: Dimensions & Airflow Requirements





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# Appendix C:

## Total Kjeldahl Nitrogen Method

### Titrimetric Determination

Various scientific associations offer approved Kjeldahl methods. These methods are available at their websites. The AOAC International (*Official Methods of Analysis*), Association of American Cereal Chemists (*Approved Methods*), American Oil Chemists Society, Environmental Protection Agency (*EPA Methods for Chemical Analysis of Water and Wastes*), International Standards Organization, The National Forage Testing Association, and United States Department of Agriculture.

*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.*

#### 1. Scope and Application

- 1.1.** This method determines the Total Kjeldahl Nitrogen (TKN) in water, and organic substances. There are three steps within the method: 1. the digestion procedure – which converts organic nitrogen to ammonia, 2. the distillation procedure - transferring ammonia from the digested sample to an ammonia trapping solution 3. sample titration – the quantification of ammonia in the trapping solution.

**NOTE 1:** Some compounds containing nitrogen may not be converted; such as amines, nitro-compounds, hydrazones, oximes, semicarbazones and some tertiary amines.

- 1.2.** This method is described for use with Kjeldahl equipment using Macro-Size glassware (500 - 800 ml flasks).

#### 2. Definitions

- 2.1.** TKN is defined as the sum of free-ammonia and organic nitrogen compounds, converted to ammonium sulfate  $(\text{NH}_4)_2\text{SO}_4$  by acid digestion.

#### 3. Apparatus

- 3.1.** Digestion Apparatus: A Kjeldahl digestion apparatus with multiple 600 watt electric burners, using 500 - 800 ml flasks and a fume removal manifold system.
- 3.2.** Distillation Apparatus: A Kjeldahl distillation apparatus with multiple 600 watt electric burners, using 500 - 800 ml flasks. The distillation system includes connecting bulbs to prevent mechanical carryover of NaOH during distillation, water cooled condensers and

distillate receiving vessels, such as 250 ml Erlenmeyer flasks. The equipment is available as a combined system, featuring both digestion and distillation apparatus. The preheated burners should bring 250 ml of water at 25° C to a rolling boil with in 5 minutes. Condensation columns should cool distillate to 75° F (23.8°C).

**3.3.** Titration Class A Burets, 25-50 ml. for dispensing Standardized Acid solution.

**3.4.** Analytical balance, sensitive to 0.0001grams.

#### **4. Reagents**

**4.1.** Purified water, nitrogen free.

**4.2.** Sulfuric acid,  $\text{H}_2\text{SO}_4$  concentrated, Specific Gravity of 1.84. (95-98%, nitrogen free)

**4.3.** Copper sulfate,  $\text{CuSO}_4$ , anhydrous. Nitrogen free. (catalyst)

**4.4.** Potassium sulfate,  $\text{K}_2\text{SO}_4$ . Nitrogen free. (boiling point elevator)

**4.5.** Sodium hydroxide NaOH, reagent grade Specific Gravity 1.3, nitrogen free. 45% solution, (dissolve pellets, 450g NaOH in distilled water and dilute to 1 liter).

**4.6.** Boiling stones, Antibumping agent, aluminum oxide stones, (Hengar granules).

**4.7.** Mixed color indicator: 0.75 g of Methyl Red and 0.5 g Methylene Blue in 300 ml of 95% ethanol. (Prepare fresh every 30 days)

**4.8.** Boric acid  $\text{H}_3\text{BO}_3$ , saturated solution, (dissolve granules, 40 g boric acid, in purified water and dilute to 1 liter). Add 3 ml of the mixed color indicator solution to the 1 liter of boric acid solution.

**4.9.** Sulfuric acid,  $\text{H}_2\text{SO}_4$ , standardized solution: (0.02 N) Prepare a stock solution of approximately 0.1 N sulfuric acid by diluting 3 ml of conc.  $\text{H}_2\text{SO}_4$  (sp. Gr. 1.84) to 1 liter with  $\text{CO}_2$ -free distilled water. Dilute 200 ml of this solution to 1 liter with  $\text{CO}_2$ -free distilled water.

Alternatively, premade standardized acid and base solutions with a certified specification ranges are commercially available through most Scientific Equipment Dealers. If the Sulfuric Acids standard solutions are prepared, their normality must be determined analytically.

#### **5. Digestion**

**5.1.** The distillation condensation columns should be cleaned before use by distilling a 1:1 mixture of distilled water and sodium hydroxide until the distillate is ammonia-free. Then repeat this cleaning procedure if the apparatus is out of service long enough to accumulate ammonia (> 4 hours).

##### **5.2. Digestion**

**5.2.1.** Place a homogenous measured sample (0.250 – 1.000 gram) into a 500 - 800 ml Kjeldahl flask. Weigh or measure the sample to the nearest 0.1 mg/ml.

**5.2.2.** Add to each flask:  
20 ml sulfuric acid, 0.04 g  $\text{CuSO}_4$ , 15g  $\text{K}_2\text{SO}_4$  potassium sulfate, 8-10 boiling stones. Place flask on the digestion burner in inclined position (promotes acid refluxing), with

the flask neck on the fume removal system. If necessary lower the burner heat setting to prevent sample from foaming up into neck of flask. Once, the sample comes to a smooth rolling boil return heat setting to maximum. When digestion sample clears (no black specs with pale green tint color), continue to boil for an equal length of time as required to reach the clear point, (total time approx. 1 hr). If black specs occur in the flask neck, rotate the flask 180 degrees to allow refluxing acid to rinse the internal flask surface. Allow digestion mixture to cool and **carefully** add 50 ml of distilled water to prevent  $K_2SO_4$  salt solids from forming. All salt crystals must be dissolved before proceeding to the next step. If necessary partially reheat sample and agitate to dissolve crystals.

### 6. Distillation

- 6.1. The distillation burners should be preheated and condenser cooling water turned on. The receiving flask should be in-place with enough boric acid  $H_3BO_3$  plus the mixed indicator solution to submerge the tip of condenser delivery tube well below the level of the boric acid receiving solution in the receiving flask. (Erlenmeyer 250 ml flasks recommended)
- 6.2. In a fume hood, carefully adding of 100 ml of 45% sodium hydroxide solution without mixing to make the digested sample alkaline. Tilt the flask in to the hood while adding the sodium hydroxide to the sulfuric acid digestion solution. Slowly add the heavy sodium hydroxide solution down the neck of the flask. The heavier sodium hydroxide solution will slip under the aqueous sulfuric acid solution without mixing and with out loss of free-ammonia. Do not mix until the flask has been connected to the distillation apparatus.
- 6.3. Immediately connect the flask to a condenser using the rubber stopper on the distillation connecting bulb attached to a condenser column.
- 6.4. Vigorously swirl the Kjeldahl flask to mix contents thoroughly; heat until all  $NH_3$  has been distilled ( $\geq 8$ -10 minutes). During the ammonia transfer the receiving solution with change color from purple to green. Lower receiving flask and let liquid drain from condenser tip. Turn off distillation burners.
- 6.5. For nitrogen concentrations above 1 mg/l, the ammonia can be determined titrimetrically. For concentrations below this level, colorimetric or potentiometric determination methods are recommended.

## 7. Titration

**7.1.** Depending on the samples expected nitrogen level and the sensitivity of the titration desired, select the appropriate standardized H<sub>2</sub>SO<sub>4</sub> solution (0.02 or 0.10 normal). High or low nitrogen concentrations will require standardized H<sub>2</sub>SO<sub>4</sub> solutions with stronger or weaker normalities. Titrations should require at least 15 ml of titrant to be accurate. Fill a class A. buret to the zero line with the standardized H<sub>2</sub>SO<sub>4</sub> solution. Titrate the H<sub>3</sub>BO<sub>3</sub> receiving solution with standardized H<sub>2</sub>SO<sub>4</sub> solution to first trace of the original purple color. A white stirring plate will aid color visualization of end point. Record ml H<sub>2</sub>SO<sub>4</sub> titrated to the at least nearest 0.05 ml. Match the endpoint against a sample blank containing the same volume of distilled water and H<sub>3</sub>BO<sub>3</sub> solution.

## 8. Calculation

### For Dry Samples

% Nitrogen, = (ml H<sub>2</sub>SO<sub>4</sub>, sample - ml H<sub>2</sub>SO<sub>4</sub>, blank ) x Normality H<sub>2</sub>SO<sub>4</sub> x 1.4007/ **weight of sample in grams.** (1.4007 = a single factor that takes into account the molecular weight of Nitrogen, the conversion of milli-equivalent results of V\*N, and the conversion to %)

% Nitrogen, = (ml H<sub>2</sub>SO<sub>4</sub>, sample - ml H<sub>2</sub>SO<sub>4</sub>, blank) x normality H<sub>2</sub>SO<sub>4</sub> x 1400.7 x 100/ **weight of sample in milligrams.**

**For Liquid Samples:** calculate Total Kjeldahl Nitrogen, in mg/1, in the original sample as follows:

**Milligrams Total Nitrogen per Liter = (ml H<sub>2</sub>SO<sub>4</sub>, sample - ml H<sub>2</sub>SO<sub>4</sub>, blank) x normality H<sub>2</sub>SO<sub>4</sub> x 14.007x 1000/ volume of sample in milliliters.**

If desired to determine % protein instead of % nitrogen, the calculated % N is multiplied by a factor, the magnitude of the factor depending on the sample matrix.

Common Protein Factors

5.7 – bread, wheat and wheat flour

6.25 – other grains

6.38 – milk

6.25 – unknown source

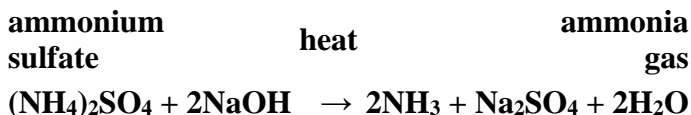
## Total Kjeldahl Nitrogen Method Overview

Digestion is accomplished by boiling a homogeneous sample in concentrated sulfuric acid. The end result is an ammonium sulfate solution. The general equation for the digestion of an organic sample is shown below:

**Organic N + H<sub>2</sub>SO<sub>4</sub> →**

**(NH<sub>4</sub>)SO<sub>4</sub> + H<sub>2</sub>O + CO<sub>2</sub> + other sample matrix byproducts**

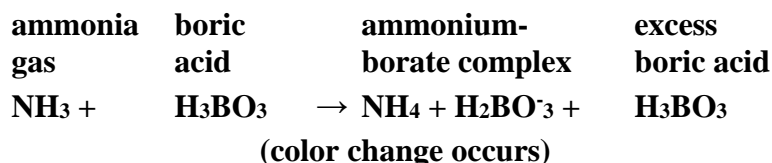
Distillation: Excess base is added to the digestion product to convert  $\text{NH}_4$  to  $\text{NH}_3$  as indicated in the following equation. The  $\text{NH}_3$  is recovered by distilling the reaction product.



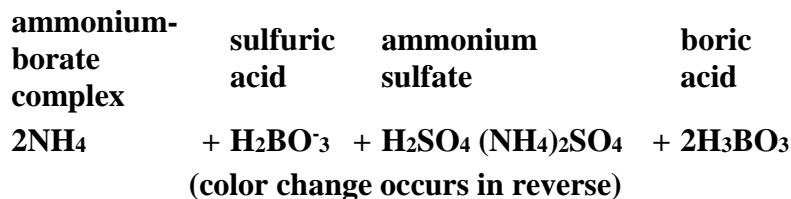
Titration quantifies the amount of ammonia in the receiving solution. The amount of nitrogen in a sample can be calculated from the quantified amount of ammonia ion in the receiving solution.

There are two types of titration—back titration and direct titration. Both methods indicate the ammonia present in the distillate with a color change.

In the direct titration, boric acid is used as the receiving solution instead of a standardized mineral acid, the chemical reaction is:



The boric acid captures the ammonia gas, forming an ammonium-borate complex. As the ammonia collects, the color of the receiving solutions changes.



The boric acid method has the advantages that only one standard solution is necessary for the determination and that the solution has a long shelf life.