Rapid Distillation Apparatus

MODEL 65000

INSTRUCTION MANUAL

6504000 REV. N / ECO H435
Printed in U.S.A.

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General Description

The Labconco Rapid Distillation Apparatus is designed expressly for rapid, semi-automatic steam distillation from sulfuric acid digest prepared from nitrogen-bearing materials such as feeds, grains, soils, plant tissue, water effluent, organic waste food products, etc. Sample digestions are accomplished utilizing either Labconco's Micro Distillation Apparatus or Rapid Digestor units. The distillation apparatus can be used for micro or macro levels of nitrogen determination.

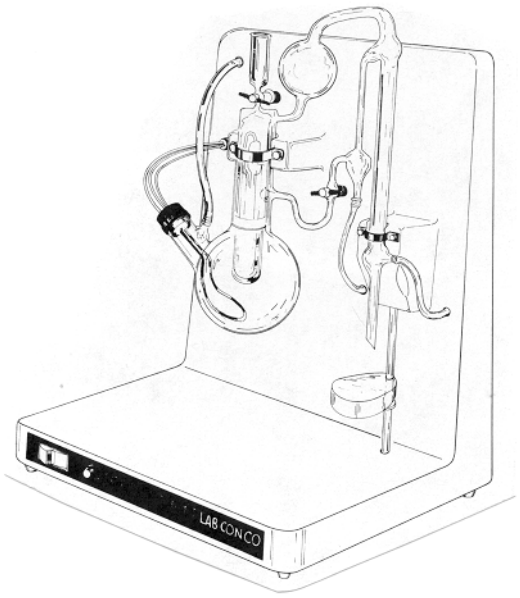

Figure 1

Performance

Your Labconco Rapid Distillation Apparatus has been designed to accept digested samples up to 4 ml (max.) equivalent of concentrated acid, and is capable of giving reproducible results in ranges as low as 10 micrograms (gamma) #1% nitrogen. Ideal nitrogen of 0.15 mgs makes this apparatus an excellent companion for the Labconco Micro and Rapid Digestors

The distillation apparatus many also be utilized for other steam distillations that DO NOT exceed the volume limitations of the units mixing chamber (approx. 50 to 55 ml).

INTRODUCTION

Component Identification

(1) **Console.** A one piece assembly molded out of linear polyethylene to provide both chemical resistance and light weight capabilities for the distillation apparatus.

(2) **Still.** One piece still fabricated from borosilicate glass and featuring chemically inert PTFE stopcocks. Because the still does not incorporate rubber or plastic tubing connections, the possibilities of leakage have been greatly reduced.

(3) **Variable Heat Control.** The electric immersion heater provides fast, efficient heat. Steam generation starts very rapidly from a cold start. Electrical input to the heater is controlled by a built-in variable heat control switch, which allows complete control of the steam.

(4) ***Flow Control Valve.** The unit is supplied with a fine metering valve that is installed between a water source and the water inlet tube connector to the steam reservoir. Adjust valve to keep water level in the steam reservoir approximately 2/3 full during distillation.

(5) **Aspirator.** The unit has a built-in glass aspirator to purge spent samples after determination. The water used for cooling of this condenser is also used for aspirating. A twist of the stopcock draws the sample and rinse water down the drain.

***Not shown**

Component Identification

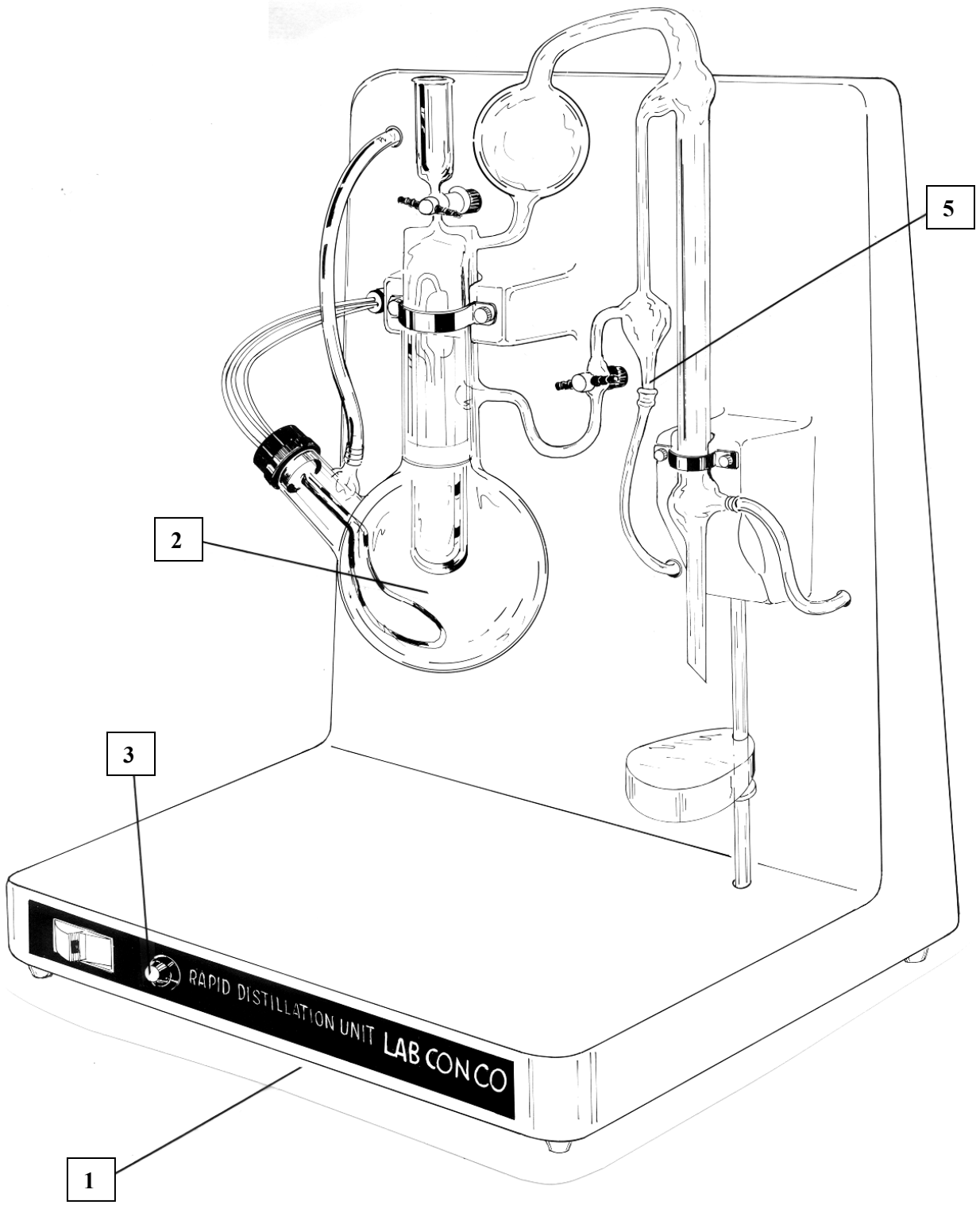

Figure 2

INSTALLATION

Your Rapid Distillation Apparatus has been shipped to you in one carton. In unpacking, remove the packing material carefully, checking all material for two separately packed parts. The carton will contain a heater element assembly and clmaps wrapped in protective paper, and the one-piece glass still packed between foam pads inside of a cardboard carton.

Inspect this material thoroughly prior to installation and report any damage that may have occurred in transit.

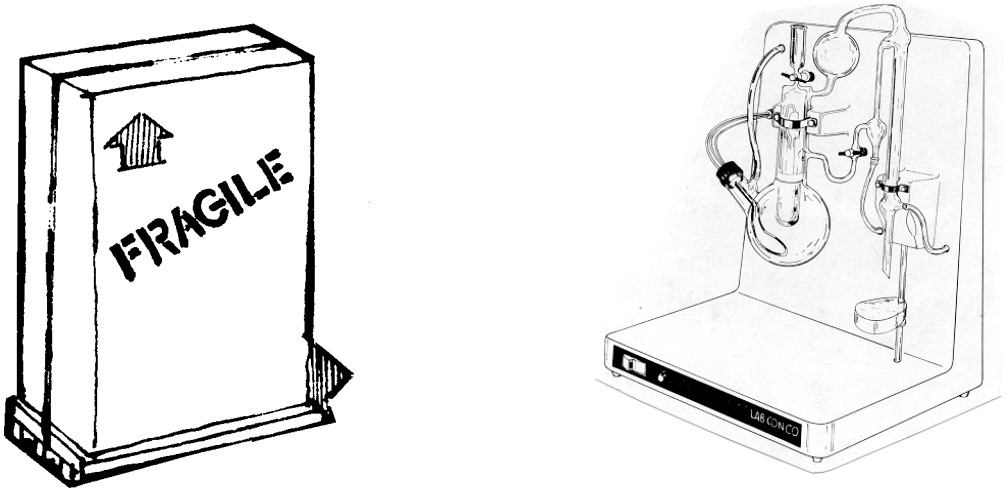

Figure 3

Installation Factors

Assembly

(1) Remove the condenser clamp screws and clamp from the console.

(2) Lay the console unit on its back and position the glass still assembly on the console, lining up the glass trap and condenser sections, as shown in Figure 2.

(3) Replace both the large and small clamps, insert and tighten down the clamp screws until the glassware is firmly attached to the console.

NOTE: Check clearance on the condenser tip with the movable flask support shelf. Adjust the entire glassware unit, by slightly loosening the clamp screws, so that enough room is provided to insert the receiver flask. Retighten clamp screws to secure this position.

(4) Place still in a location having a convenient 115 volt, AC outlet, and accessibility to distilled water, tap water and drain facilities.

(5) Obtain several lengths of Tygon tubing (5/16" bore x 1/16" wall). Tubing IS NOT furnished with the unit and may be obtained from local laboratory supply dealers. Tubing will be used for connecting water lines (refer to steps 6, 11 and 12).

(6) Measure distance from tube connector on steam reservoir through the tubing access port in console (upper left corner) to a water outlet. Cut tubing and connect to tube connector on still and water source. If the flow control valve is to be used, install between water source and tube connector located on top of steam reservoir. The valve is provided with a hose barb fitting on one end and a female 1/8" NPT connector on the other. Install with ridged tubing from water source to 1/8" NPT. Connect 5/16" Tygon tubing to the hose barb fitting. A tee may be installed to allow use of the same water source for the condenser cooling and steam reservoir..

(7) Remove protective wrapper from formed metal heater element and seal stopper assembly.

NOTE: Water tubing clamps are usually placed on heater "lead wires" to prevent loss in shipping. Two clamps are provided – remove them from the heater leads and use when making tubing connections at the tap water inlet and tap water source connection points.

(8) Insert heater assembly in steam reservoir (see labeled diagram in Figure 2).

NOTE: A very tight seal between rubber stopper and neck of the flask must be obtained.

(9) Insert line cord in 115 Volt AC receptacle. Turn off with console "On-Off" switch to prevent possible heater burn-out.

(10) Connect heater lead wire plug to female receptacle provided on the console.

(11) Connect Tygon tubing to the outlet of the condenser water aspirator. Run tubing through access port on console to drain area.

(12) Connect Tygon tubing to condenser inlet. Run tubing through access port provided to the tap water source. Clamp tubing securely at water source and condenser inlet points.

Start-up and Initial Check-out

If all of the assembly steps, as listed previously have been followed, your Rapid Distillation Apparatus will now be ready for its initial start-up.

The start-up process should begin as follows:

(1) Make sure the illuminated "On-Off" switch is in the off position.

CAUTION: Do not energize heater unit unless heater element is properly immersed in water.

(2) Fill steam reservoir flask 2/3 full with ammonia free distilled water. Adjust flow control valve to give approximately 7 ml/minute flow. Proper adjustment of valve will replace the water lost from the distillation. If the flow control valve is to be used for low level nitrogen determinations, it may be necessary to install a deionization cartridge (available from your laboratory supply dealer) between the flow control valve and water source. Usually a blank determination on reagents and water is sufficient for most low level nitrogen determinations.

(3) Check sample addition funnel stopcock. Mare sure it is CLOSED!

(4) Close aspirator stopcock located between condenser and steam reservoir.

(5) Turn on tap water source and fill condenser.

(6) Place a small graduated cylinder on receiving holder at output of condenser to measure distillation rate.

(7) Turn heater control dial to "9." Press On/Off switch to "On."

(8) The water in the steam reservoir will begin boiling in about 5-6 minutes. The heater control setting should be turned down until a distilling rat of 5 ml/min. is obtained. The flow control valve is adjusted to replace water lost during distillation.

(9) Measure temperature of distillate and adjust cooling water input at tap water source to maintain distillate temperature of 25°C or lower.

(10) For low level nitrogen determinations, allow steam distillation to continue until distillate is nitrogen free. Removal of nitrogen entrained in steam reservoir water may require 30 minutes at initial start-up, or if the apparatus has been out of use for sufficient time to allow nitrogen containing fumes to be reabsorbed in steam reservoir liquid. Add approximately 15 ml of ammonia free distilled water to the mixing chamber through the sample-addition funnel. Close sample stopcock after test liquid drains through! Open the aspirator stopcock and siphon off all liquid in the mixing chamber to check out aspirator operation. Water flow to condenser can be adjusted to increase or decrease "siphon" rate. When mixing chamber is cleared of the test liquid, close the aspirator stopcock.

(11) The unit is now ready for samples. Refer to the following appropriate method for the determination of nitrogen content from predigested samples.

Samples Digested Utilizing Micro-Digestor –

Start-up and Operation

The Labconco Rapid Distillation Apparatus is designed to conform with standard Micro Kjeldahl distillation techniques. The preparation of digested samples, chemical treatment of the sample prior to distillation and titration techniques following the distillation are adequately described in A.O.A.C. Methods of Analysis and other technical references, and should be referred to in ALL cases.

(1) Turn on cooling water and adjust to normal rate.

(2) Turn on heater – check reservoir level and adjust to 2/3 full if required. Set heat control at "9" on the dial. Allow steam reservoir water to come to "boiling" and turn down heat control to proper setting. Adjust flow control valve to replace water lost during distillation.

(3) Allow still to reach thermal equilibrium – check for 4-5ml/min. distilling rate, with small graduate (refer to Step 8 of Start-up and Check Out).

(4) Open aspirator stopcock joining still chamber and water aspirator aspirate inner chamber – close the stopcock.

(5) Place a receiver flask, containing a volume of receiving solution on shelf at outlet of condenser.

(6) Check stopcock on sample addition funnel to see that it is closed. Place entire diluted digest sample in addition funnel.

(7) Open "addition" funnel stopcock to transfer digest sample to the mixing chamber. Close stopcock.

(8) Rinse digestion flask with several small amounts of ammonia-free distilled water (total volume of rinses not to exceed 8 to 10 ml). Adding each small rinse to the inner chamber through the addition funnel separately and closing stopcock after each addition.

(9) Rinse addition funnel with 3 to 4 ml ammonia-free distilled water, leaving some water as a liquid seal.

(10) Raise receiver flask shelf, positioning it so that the condensate outlet is slightly below the receiving liquid surface level.

(11) Add strong alkali solution (calculated to produce excess base in mixing chamber when completely added) to addition funnel. Open funnel SLOWLY and allow alkali to slowly flow into the mixing chamber. Stop flow if "neutralizing action" becomes too vigorous or siphon back of receiving solution. Continue intermittent feeding of alkali to mixing

chamber until all of the solution is added. Leave column of caustic in the funnel stem to act as a liquid seal.

NOTE: If reaction is uncontrollable, REDUCE all future acid digest sample volumes and re-calculate required alkali solution strengths so that inner chamber contents will have a normality of 4 to 8. A small amount of caustic should be left in the addition funnel to act as a liquid seal during each distillation. If foaming is a problem during distillation, add 1 or 2 drops of anti-foam reagent.

(12) Allow distillation to proceed long enough to complete the quantitative recovery of ammonia free from the sample. Average time of distilling is 3 to 5 minutes.

(13) Lower receiver flask shelf and allow distillation to continue approximately one minute.

(14) Place receiver flask aside. It is now ready for titration.

(15) Place an extra flask on receiver shelf, open aspirator tube stopcock, to "drain out" the mixing chamber. Open sample addition stopcock, letting liquid seal pass into the mixing chamber. Close addition stopcock. Drain out by means of the aspirating action. Follow with several good washes of ammonia-free distilled water, adding each wash through the addition funnel, closing the addition stopcock and aspirating the washing solution to the drain.

NOTE: Close aspirator stopcock before addition of each wash.

(16) The unit is ready to handle the next sample

NOTE: If long delays are encountered between sample runs, aspirate mixing chamber just prior to sample digest introduction.

Shutdown

(1) Turn off heater with main switch.

(2) Remove receiver flask.

(3) Wash out mixing chamber thoroughly, leaving it about ½ full (10-12 ml – refer to Step 15 of Start-up and Operation).

(4) Turn off cooling water source to condenser and flow control valve.

Samples Digested Utilizing Rapid Digestor –

Start-up and Operation

Same as information under Samples Digested Utilizing Micro-Digester.

Principle

(1) After sample digestion, utilizing the Labconco Rapid Digestor-4, Rapid Digestor-25, or equivalent digestion apparatus, a portion of the sample is transferred to the Rapid Distillation Apparatus. Ammonia is steam distilled into standard acid and back titrated with standard alkali. Complete recovery of nitrogen is accomplished in 3 – 5 minutes distillation time.

Apparatus

(1) Labconco Rapid Distillation Apparatus Model 65000. Available from Labconco Corporation, 8811 Prospect Avenue, Kansas City, MO 64132.

(2) Erlenmeyer Flasks. 125 ml Erlenmeyer flasks for standard acid receiving solution.

(3) Pipets. 20 ml volumetric pipets for sample transfer.

(4) Burets. 15 ml buret for standard acid and standard alkali. Separate burets should be used for the addition of the acid and alkali.

Reagents

(1) Sodium hydroxide/potassium sulfide. Dissolve 45g solid NaOH in water. While still warm, dissolve 10g of K_2S in the NaOH solution, cool and dilute to 1 liter.

(2) Methyl red indicator. Dissolve 1g methyl red in 200 ml ethyl alcohol.

(3) Sodium hydroxide standard solution. 0.01N, prepare as in AOAC Official Methods of Analysis, Thirteenth Edition, p. 877.

(4) Sulfuric acid standard solution. 0.05N, prepare as in AOAC Official Methods of Analysis, Thirteenth Edition, p. 877.

Standardize each standard solution with primary standard and check one against the other. Refer to AOAC Official Methods of Analysis, Thirteenth Edition, Chapter 50. Test all reagents before use by blank determination with 1g sugar.

Determination

(1) Samples are digested utilizing the Labconco Rapid Digestor 5 place of the 25 place units equipped with 250 ml volumetric digestion tubes (refer to digestion methods available upon request from Labconco). After digestion, remove rack of tubes from digestor and allow to cool for 8 – 10 minutes (cooling time depends upon air flow around tubes). Direct rapid spray of water (kitchen sink dish rinsing sprayer works well) to bottom of each tube to dissolve acid digest completely. If precipitate forms, dissolve by manual mixing or by placing tubes in ultrasonic bath. Low recovery of nitrogen will result if precipitate is not completely dissolved. Let sample cool, dilute to the mark and mix thoroughly by inversion 20 times. Using a 25 ml buret, prepare the sulfuric acid receiving solution by adding the calculated amount of 0.05N sulfuric acid standard solution to a 125 ml Erlenmeyer flask. Add 5 – 7 drops of indicator (see calculating section for amount of standard solution to add). Dilute volume with water to approximately 70 – 75 ml.

Pipet ml aliquot of sample solution to sample addition funnel on the Rapid Distillation Apparatus. Distillation rate must be between 4 – 5 ml minute. Adjust water flow control valve to keep water level in the steam reservoir approximately 2/3 full. Introduce sample into sample chamber. Rinse funnel with 3 – 4 ml portions of water. Raise 125 ml Erlenmeyer flask, containing the receiving solution, into position under the condensate outlet tube of the still. Correct position is where the top of the outlet tube is completely submerged below the receiving solution liquid level. Add 10 ml + 1 ml of sodium hydroxide/potassium sulfide to the addition funnel. Slowly add alkali to the sample chamber. (It is very important to add the caustic slowly, while visually monitoring the standard acid solution to see that the solution does not siphon back through the condenser and into the steam reservoir.) Leave column of caustic in the funnel stem to act as a liquid seal. Distill for 3 – 5 minutes, lower receiving flask and allow distillation to continue for approximately one minute. Titrate excess standard acid with 0.01N sodium hydroxide standard solution. Correct for blank determination on reagents.

Standard acid to add to receiving flask (ml)=

$$\left[\frac{(\%\text{ nitrogen expected in sample}) \times (\text{sample aliquot taken} \times \text{g sample weight})}{(\text{normally of standard acid}) \times 1.4007 \times 250}\right] + 2$$

% Nitrogen=

$$\left[\frac{(\text{ml std acid} \times \text{normality} - (\text{ml std base} \times \text{normality})}{\text{effective weight}}\right] \times \frac{1.4007}{}$$

%Protein=

$$\left[\frac{(\text{ml std acid} \times \text{normality} - (\text{ml std base} \times \text{normality})}{\text{effective sample weight}}\right] \times \frac{1.4007 \times \text{factor}}{}$$

where:

$$\text{effective sample weight} = \frac{\text{weight of sample in grams}}{250} \times \text{sample aliquot}$$

factor =	wheat	5.70
	milk	6.38
	rice	5.95
	all other	6.25

NOTE: Labconco recommends the use of this method with the Labconco Rapid Distillation Apparatus. However, alternative methods may be readily adapted to the apparatus.

ROUTINE MAINTENANCE

Console

The console is constructed of plastic and resists staining. To preserve its general appearance, acid and alkali "spills" should be removed as soon as possible by washing and sponging with a damp cloth.

Glass Still (Replacement)

(1) Disconnect line cord from 115 volt AC outlet disconnect heater plug connections.

(2) Remove heater assembly and stopper.

(3) Disconnect ALL tubing connections on glass still.

(4) Remove clamp screws (4).

(5) Remove clamps (2).

(6) Lift still assembly from console and lay carefully aside.

(7) Remount new glass still assembly and reconnect all parts and tubing, by reversing above steps.

Stopcock

The Labconco Rapid Distillation Apparatus is equipped with (2) PTFE stopcocks. The PTFE stopcocks do not require lubricants. The plug and glass barrel may be cleaned with Acetone to wash away any solid particles that might accumulate during long service. If solid particle s become lodged between the plug and glass barrel, or project from the glass surfaces, they can score the PTFE plug around its bore and result in a "leaky" plug.

When alkalies or other liquids corrosive to glass are used in PTFE-glass stopcock valve assemblies, rinse all valve components thoroughly with water after each use. Roughness of the glass barrel due to liquid concentrate formed on evaporation may score the plug. Additional information is provided with this unit and may be referred to for service.

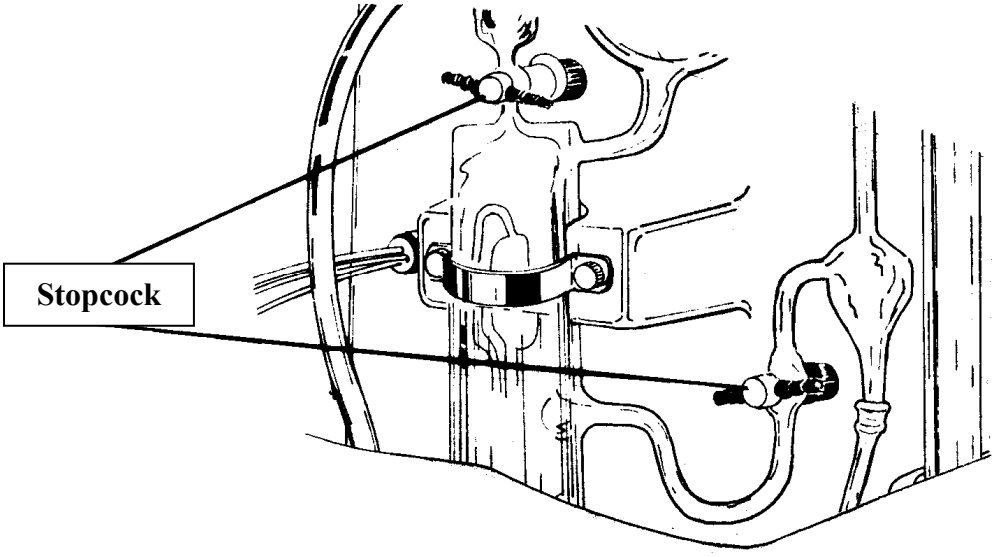

Figure 4

REPLACEMENT PARTS

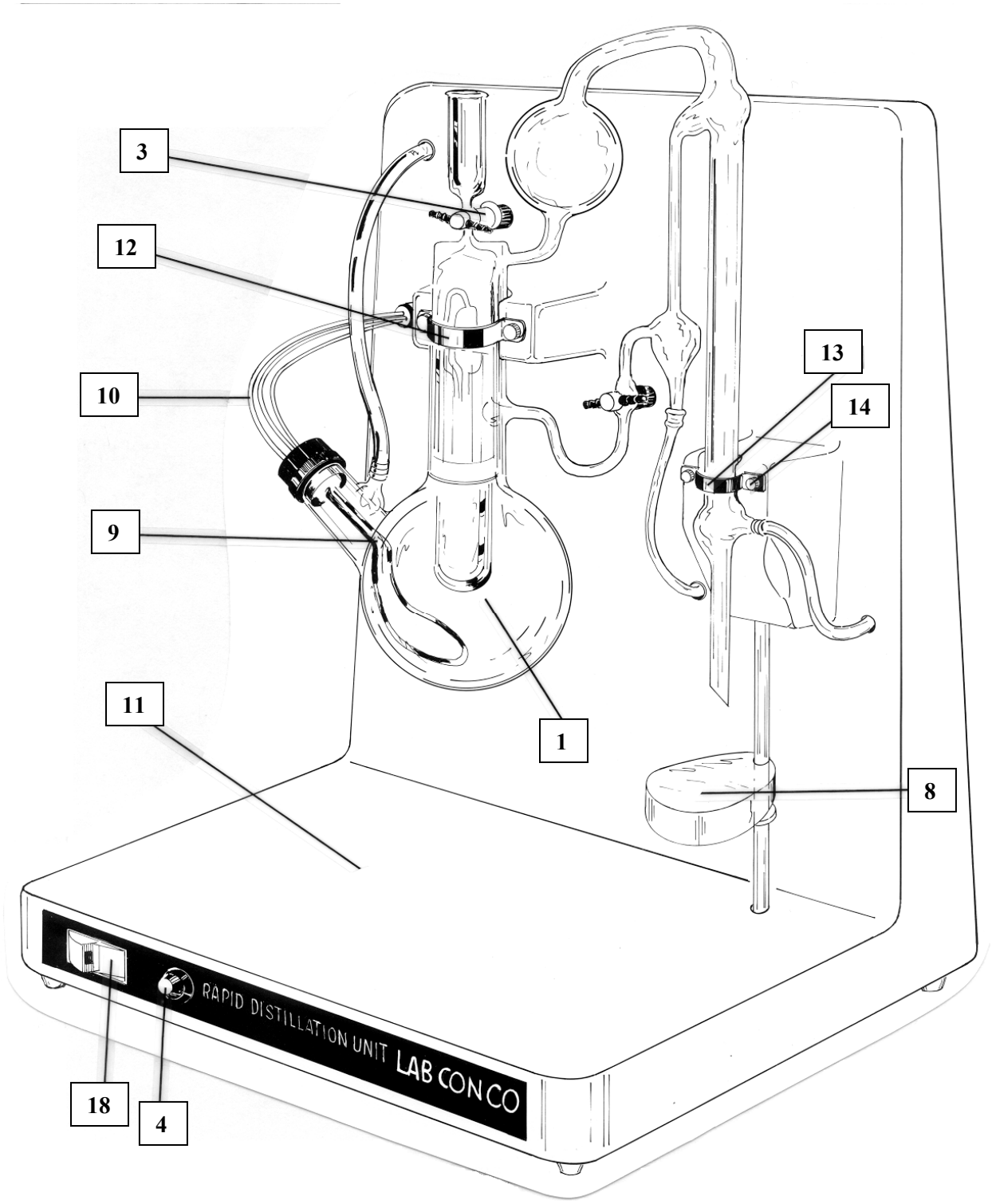

Figure 5

ITEM	PART NUMBER	DESCRICPTION
1	65001	Glass micro still assembly with PTFE stopcocks
2	13525	PTFE plug replacements for PTFE stopcock
*3	13237	Heat controller
4	18765	Knob, for heat controller
*5	19175	Fastener, nylon round head, canoe type
*6	13432	Fuse holder
*7	13410	Fuse only – 115 Volt AC AGC-3
8	65008	Receiver flask platform assembly
9	65042	Heater assembly, complete with stopper and male plug; 115 VAC, 280 Watts
10	12823	Plugs, heater lead jack, only
11	65021	Housing assembly, complete (less all glassware and heater)
12	65056	Saddle clamp, large
13	65058	Saddle clamp, small
14	19030-08	Screws, for large and small clamps
*15	13174	Transformer (230 V to 115 V, 50/60 Hz, 300 Watt)
*16	19093-26	Nut
*17	65045	Flow control valve
18	13270	Switch-lighted rocker

*Items not shown

WIRING DIAGRAM

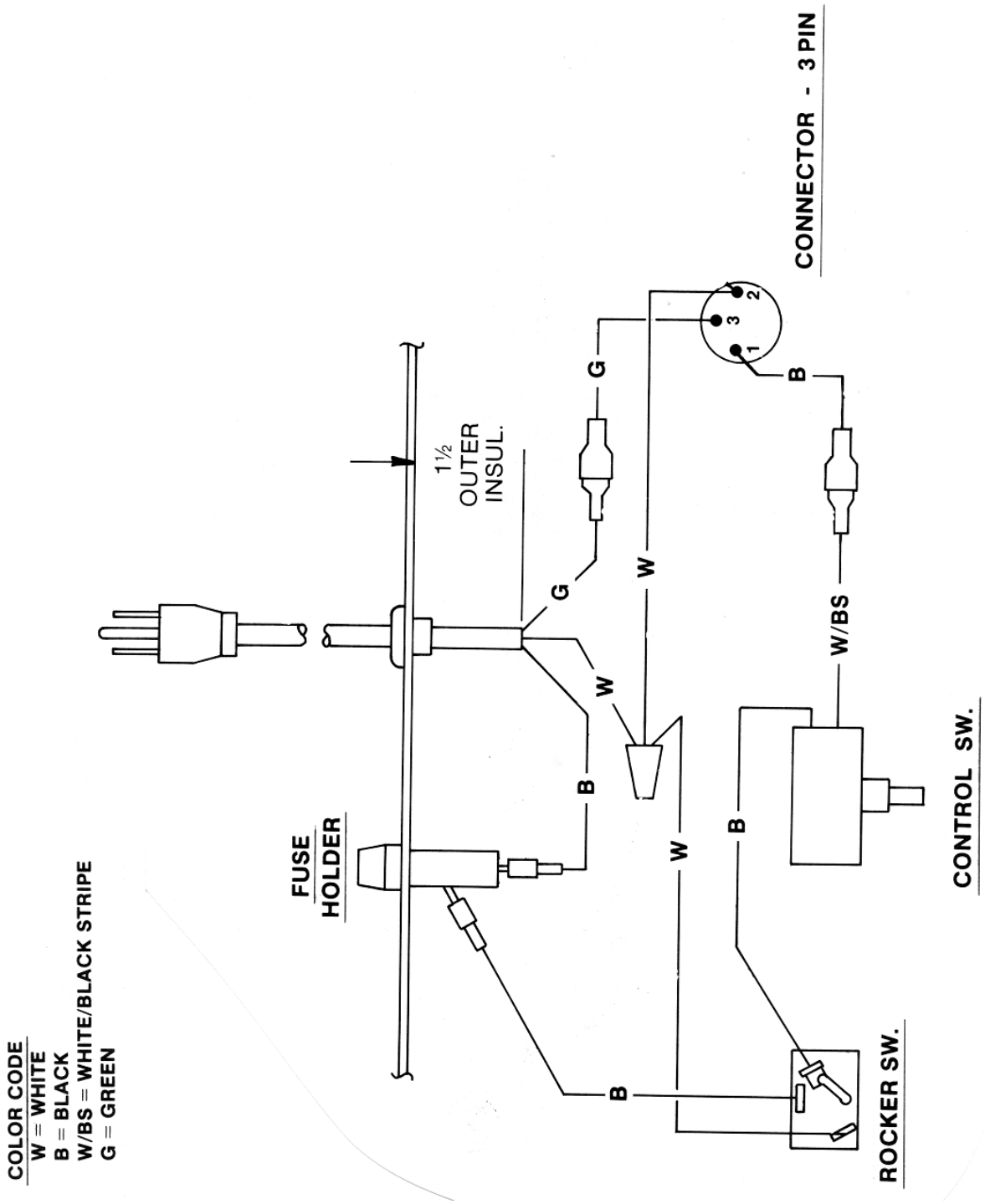

Figure 6

REFERENCES

Many excellent reference texts and booklets are currently available. The following is a brief listing:

Official Methods of Analysis of the Association of Official Analytical Chemists, Thirteenth Edition, Washington, District of Columbia, 1980.

Hambleton, L.G. and Noel, R.J., Journal of the Association of Analytical Chemists, 58:143-145, 1975.

"Digestion Method for the Determination of Protein Nitrogen in Feeds, Foods, Grains, Cereals, and Grasses Utilizing the Labconco 4-Place Rapid Digestor (R/D-4)," Labconco Extracts 2:880, Labconco Corporation, Kansas City, MO.

"Digestion Method for the Determination of Protein Nitrogen in Feeds, Foods, Grains, Cereals, and Grasses Utilizing the Labconco 25-Place Rapid Digestor (R/D-25)," Labconco Extracts 3:880, Labconco Corporation, Kansas City, MO.

WARRANTY

We are committed to providing our customers with quality equipment and service after the sale. Part of this objective involves keeping you informed of changes and new product additions. We therefore request that you take a moment to fill out the product registration card so we may know your location as well as some of the reasons that prompted you to purchase our products

Labconco Corporation warrants products of its manufacture for one year, from receipt of the equipment by they purchaser, against defects in materials and workmanship. This limited warranty covers parts and labor but not transportation and insurance charges. In the event of a warranty claim, contact the dealer who sold you the product. If the cause is determined to be a manufacturing fault, the dealer or Labconco Corporation will repair or replace all defective parts to restore the unit to operation. **Under no circumstance shall Labconco Corporation be liable for indirect, consequential or special damages of any kind.** This statement of warranty may be altered by a specific published amendment. No individual has authorization to alter the provisions of the warranty policy or its amendments. Lamps and expendable items such as filters are not covered by this warranty. Damage due to corrosion or accidental breakage is also not covered.

WARNING: 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 urged 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.

If shipment is received in visibly damaged condition, be certain to make a notation on the delivering carrier's receipt and have his agent confirm the damage on your receipt. Otherwise, the damage claim may be refused.

If concealed damage or pilferage is discovered, notify the carrier immediately and retain the entire shipment intact for inspection. Interstate Commerce Commission rules require that the claim be filed with the carrier within 15 days after delivery.

NOTE: Do not return goods. Goods returned without prior authorization will not be accepted. Labconco Corporation and its dealers are not responsible for shipping damage. Claims must be filed directly with the freight carrier by the recipient. If authorization has been received to return this product, by accepting this approval, the user assumes all responsibility and liability for biological and chemical decontamination and cleansing. Labconco reserves the right to refuse delivery of any products, which do not appear to have been properly cleaned and/or decontaminated prior to return.

CONTACTING LABCONCO

If you have any questions that are not addressed in this manual, or if you need technical assistance, please contact Labconco's Customer Service Department at either (800) 821-5525 of (816) 333-8811, between the hours of 8:00 a.m. and 5:00 p.m., Central Standard Time.

Labconco's mailing address is:

Labconco Corporation
8811 Prospect Avenue
Kansas City, Missouri 64132-2696

Fax # 816-363-0130

Visit Labconco through the Internet at:

http://www.labconco.com

or

email: labconco@labconco.com