LABCONCO CORPORATION
8811 Prospect Avenue
Kansas City, MO 64132
(800) 821-5525, (816) 333-8811
(816) 363-0130 fax
labconco@labconco.com

User's Manual

RapidStill™ I

Models
6500000

To receive important product updates,
complete your product registration card
online at **register.labconco.com**

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

Warranty

Labconco Corporation provides a warranty to the original buyer for the repair or replacement of parts and reasonable labor as a result of normal and proper use of the equipment with compatible chemicals. Broken glassware and maintenance items, such as filters, gaskets, light bulbs, finishes and lubrication are not warranted. Excluded from warranty are products with improper installation, erratic electrical or utility supply, unauthorized repair and products used with incompatible chemicals.

The warranty for the RapidStill I 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 3rd 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.

Under no circumstances shall Labconco Corporation be liable for indirect, consequential, or special damages of any kind. This warranty is exclusive and in lieu of all other warranties whether oral, or implied.

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

Thank you for displaying confidence in us by selecting a Labconco RapidStill I. Our design engineers, assemblers and inspectors have utilized their skills and years of experience to ensure that the Labconco RapidStill I meets our high standards of quality and performance.

This manual should be read carefully by all the end users in order to become familiar with the operation of the RapidStill I. Recommendations are made within the manual to help you obtain maximum performance and life from your product.

We have included sections on initial set up, operation, and maintenance to provide you with all the tools necessary to achieve maximum performance. If you have questions or concerns, do not hesitate to call us at 1-800-821-5525 for assistance.

WARNING: The disposal 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.

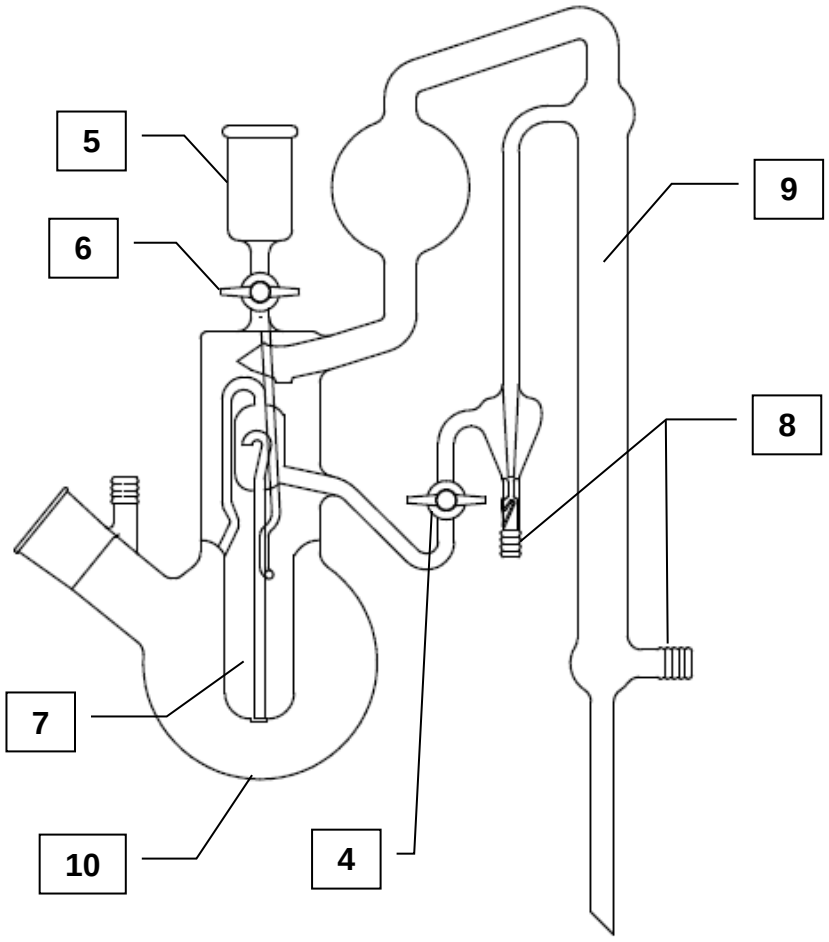
General Description

The Labconco RapidStill I 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 Digestion Apparatus or Rapid Digestor units.

Performance

Your Labconco RapidStill I has been designed to accept digested samples up to 4 ml (maximum) equivalent of concentrated acid, and is capable of giving reproducible results in ranges as low as 10 micrograms (gamma) #1% nitrogen.

The RapidStill I may also be utilized for other steam distillations that DO NOT exceed the volume limitations of the units mixing chamber (approximately 50 to 55 ml).



Component Identification

(1) **Power Switch** Switches power to the Heater. When ideal Heat Control setting is achieved, use this switch to turn Heater ON and OFF.

(2) **Heat Control** The electric immersion heater provides fast, efficient heat. Steam generation starts very rapidly from a cold start. Control input to the heater and steam output by adjusting this knob.

(3) **Fill Valve** The Fill Valve is opened only when filling Boiling Flask. Use distilled water from the Water Bottle to keep water level in the steam reservoir approximately 2/3 full during distillation.

(4) **Aspirator Stopcock** 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 Aspirator Stopcock draws the sample and rinse water down the drain.

(5) **Sample Addition Funnel** Use this funnel to transfer digested sample to the Mixing Chamber.

(6) **Sample Addition Stopcock** The upper PTFE stopcock that allows for the addition of samples or pH controlling solutions.

(7) **Mixing Chamber** The inner bulb in the lower portion of the still where the sample is mixed with appropriate solutions during the distillation.

(8) **Cooling Water Connections** Note the two barbed fittings extending through the back of the housing. Connect the “IN” fitting to a cold water source and the “OUT” fitting to a drain.

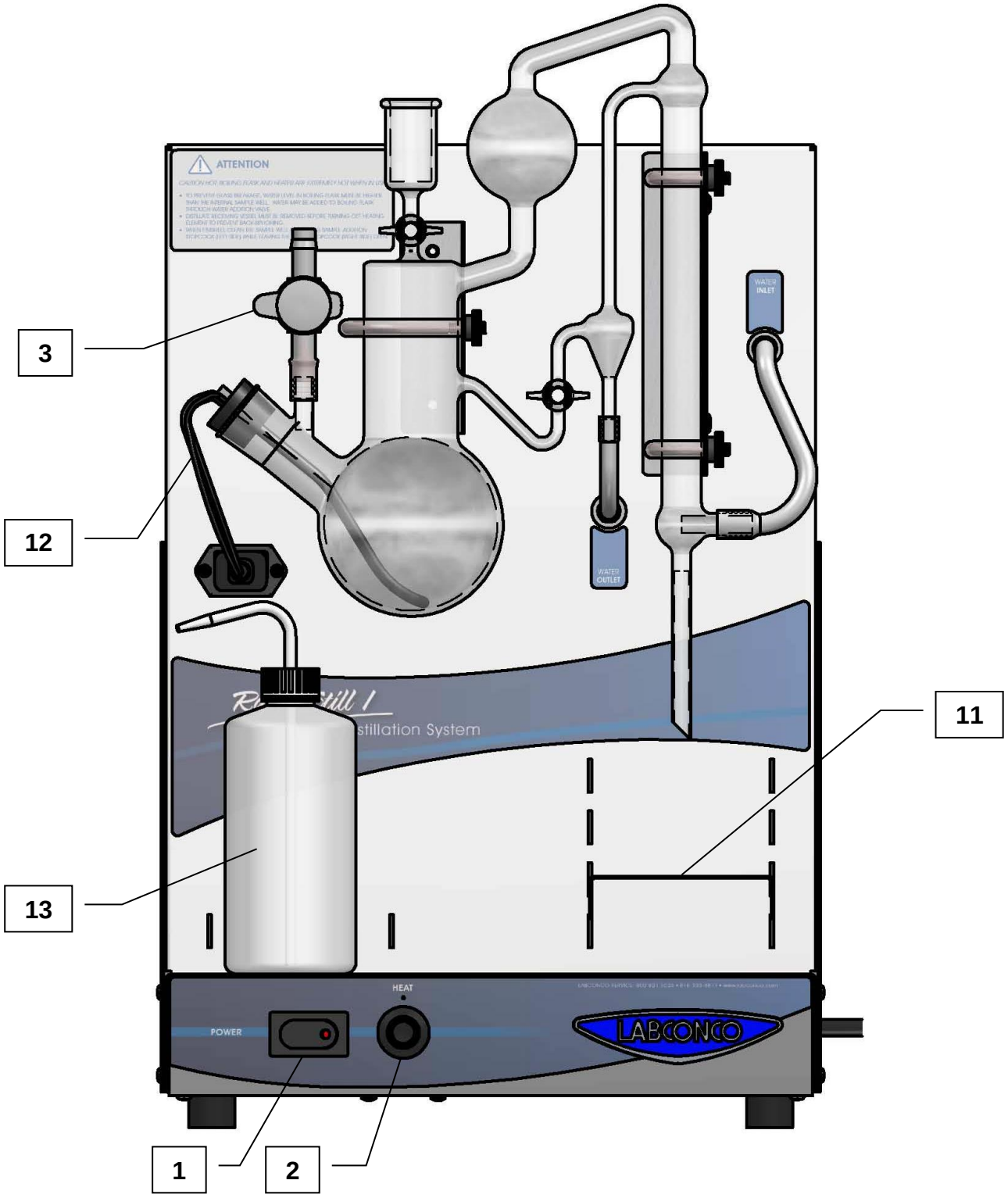
(9) **Condenser** Cold water flows through the outer wall of the tall column to condense steam.

(10) **Boiling Flask** The round flask reservoir which contains distilled water to be heated during the distillation.

(11) **Flask Shelf** Shelf is adjustable in height. Positioning the flask is important to maintain relationship between the condenser outlet and liquid level in the Receiving Flask.

(12) **Heater** Heater is energized when there is sufficient water in the Boiling Flask. Heater may be unplugged from the housing if required.

(13) **Water Bottle** Use distilled water only in the still. Water Bottle is squeezed to introduce water to the Boing Flask via the Fill Valve.


3
12
11
13
1
2
ATTENTION
WATER INLET
WATER OUTLET
HEAT
POWER
LABCONCO

Chapter 2: Installation

Your RapidStill I has been shipped to you in one carton. When unpacking, remove the packing material carefully, checking all material for two separately packed parts. The carton will contain:

Base Housing
Glass Still
Heater
Stainless Steel Flask Shelf
"U" Clamp (large) (1)
"U" Clamp (small) (2)
Clamp Nuts (6)
Fill Valve and Tube
Pinch Clamps (2)
Power Cord

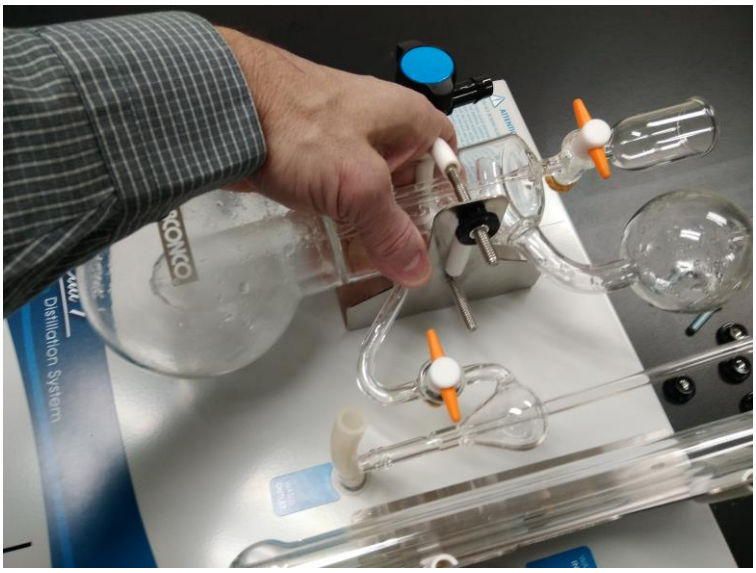
NOTE: Plastic tubing and valve for connection of the condenser to water and drain source is NOT included.

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

Installation Set Up

Assembly

(1) Lay the Base Housing on its back and position the Glass Still assembly on the console, as shown. Do not connect to electrical power source until set up is complete.

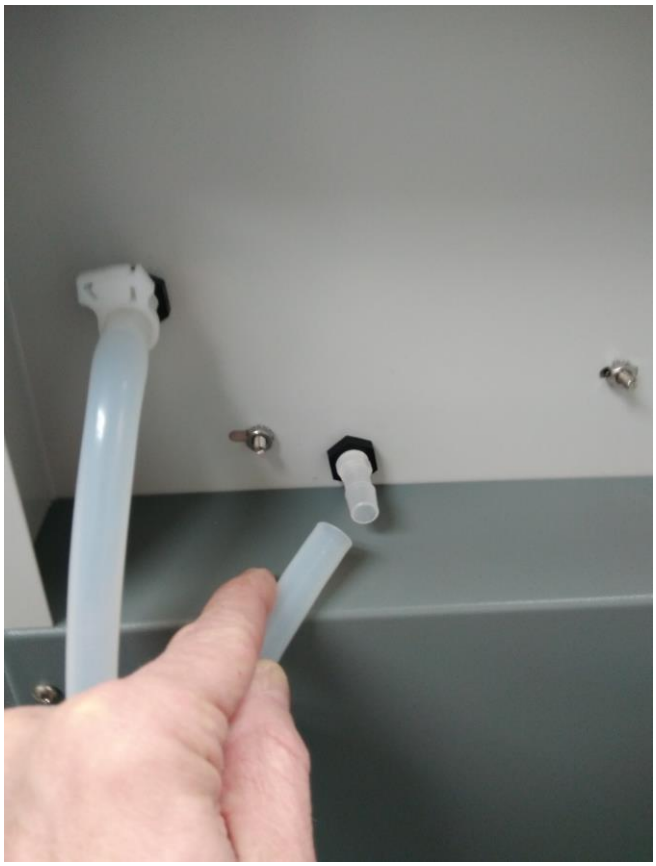


(2) Starting with the large U-clamp, place both the large and small U-clamps around the glassware and through the brackets. Gently tighten the clamp nuts so that the glassware is firmly held to the Base Housing.

(3) Carefully attach the condensing water tubing to the glass barb fittings. Do not add clamps or any lubricant (other than water) to these connections. Turn Base Housing upright.

(4) Locate the RapidStill I where there is convenient 115 volt outlet and accessibility to cold tap water and drain.

(5) Obtain several lengths of flexible plastic or silicone tubing (3/8" ID x 1/16" wall). Tubing IS NOT furnished with the unit and may be obtained from laboratory supply dealers. Connect tubing from the rear of the housing to a drain or sink for effluent. Connect tubing for the inlet from the rear of the housing to a cold water source (<25°C) with fine flow control through a valve or faucet. Two pinch clamps have been provided. Check for leaks.



(6) Keep power OFF. Insert the heater element into the Boiling Flask and press the stopper into the Flask opening firmly. Heater should not be in contact with any of the glass. NOTE: This must be sealed for distillation. Plug the Heater into the Base Housing connector.

(7) NOTE: Make certain the Power Switch is in the OFF position. Connect the Power Cord to the back of the Base Housing. Power Cord may be plugged into outlet at this time.

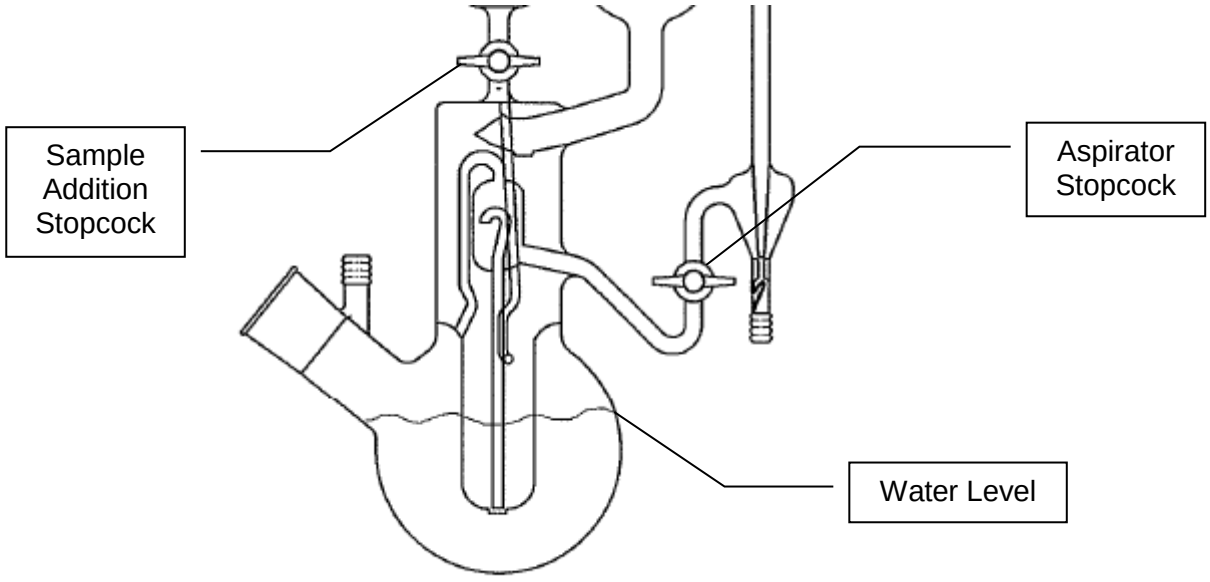
Initial Test

The start-up process should begin as follows:

(1) Make sure the Power Switch is in the OFF position.

CAUTION: Never energize the Heater unless heater element is properly immersed in water.

(2) Fill Boiling Flask 2/3 full with ammonia-free distilled water or deionized water through the Fill Valve. It is important to maintain a sufficient level in the Boiling Flask at all times. The Fill Valve should be closed.



(3) Check Sample Addition Stopcock; it must be closed.

(4) Close Aspirator Stopcock.

(5) Turn on tap water source and fill the Condenser. The rate of water through the Condenser will be adjusted later based on the temperature of the distillate.

(6) Place a small graduated cylinder on Flask Shelf at output of condenser and measure distillation rate.

(7) Turn Heat Control dial to "9." Press Power Switch to ON.

(8) The water in the Boiling Flask will begin boiling in about 5 to 6 minutes. The Heat Control setting should be turned down until a distilling rate of 5 ml/min. is obtained at the output. Replace water lost in the Boiling Flask during distillation.

(9) Measure the 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 the Boiling Flask 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 the Boiling Flask. Add approximately 15 ml of ammonia-free distilled water to the Mixing Chamber through the Sample Addition Funnel. Close Sample Addition 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 RapidStill I is now ready for samples. Refer to the following appropriate method for the determination of nitrogen content from predigested samples.

Chapter 3:
Normal Operation

Samples Digested Utilizing Micro Digestor

Start-up and Operation

The Labconco RapidStill I 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 described in the A.O.A.C. Methods of Analysis and other technical references, and should be referred to in ALL cases.

(1) Turn on cooling water to the Condenser.

(2) Turn the Power Switch ON to energize Heater. Check Boiling Flask level and adjust to 2/3 full if required. Set the Heat Control at "9" on the dial. Allow steam reservoir water to come to a roll.

(3) Reduce the Heater Control setting to a desired 4 to 5 ml/min distilling rate, (refer to Installation Set Up, Initial Test).

(4) Open the Aspirator Stopcock and aspirate the Mixing Chamber. Close the stopcock when complete.

(5) Place the Receiving Flask containing the distillate receiving solution on Flask Shelf at outlet of the Condenser.

(6) Check the Sample Addition Stopcock to ensure it is closed. Place entire 4 ml maximum acid digestion sample in Sample Addition Funnel.

(7) Open the Sample Addition Stopcock slowly to transfer digested sample to the Mixing Chamber. Close the stopcock.

(8) Rinse Sample Addition Funnel with 3 to 4 ml ammonia-free distilled water, leaving some water in the bottom as a liquid seal.

(9) Raise the Flask Shelf; positioning it so that the condensate outlet is slightly below the receiving liquid surface level.

(10) Add strong alkali solution (calculated to produce excess base in mixing chamber when completely added) to Sample Addition Funnel. Carefully open the Sample Addition Stopcock and allow alkali to slowly flow into the Mixing Chamber. Stop flow if "neutralizing action" becomes too vigorous or causes receiving solution to syphon backward. Continue intermittent addition 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 subsequent 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 Sample 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.

(11) Allow distillation to proceed. The average time of distilling is 5-10 minutes.

(12) Lower the Flask Shelf and allow distillation to continue approximately one minute.

(13) Place the Receiving Flask aside. It is now ready for titration.

(14) Place an additional flask on Flask Shelf. Open the Aspirator Stopcock to "drain out" the mixing chamber. Open Sample Addition Stopcock to allow any remaining liquid to pass into the Mixing Chamber. Close the Sample Addition Stopcock. Drain fluids by opening the Aspirator Stopcock. Follow with several good rinses of ammonia-free distilled water. Add rinse water through the Sample Addition Funnel; closing the Sample Addition Stopcock and aspirating the rinse solution to the drain.

NOTE: Close the Aspirator Stopcock before addition of each rinse.

The RapidStill I is ready to process the next sample.

NOTE: If long delays are encountered between sample runs, rinse the Mixing Chamber with distilled or deionized water prior to new sample distillation.

Shutdown

(1) Turn OFF Heater with the Power Switch.

(2) Remove the Receiving Flask.

(3) Rinse the Mixing Chamber thoroughly, leaving it about ½ full (10 to 12 ml – refer to Step 14 of Start-up and Operation).

(4) Turn off cooling water source to the Condenser.

Calculations

Standard acid to add to receiving flask (ml) =

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

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

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

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 RapidStill I. However, alternative methods may be readily adapted to the apparatus.

Chapter 4:
Routine Maintenance

Glass Still (Replacement)

(1) Disconnect the Power Cord from the outlet before performing any service work.

(2) Remove Heater assembly by unplugging it and remove stopper from flask.

(3) Disconnect ALL tubing connections on glass still.

(4) Lay the Base Housing down on its back.

(5) Remove U-clamp nuts and bolts (3).

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

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

Stopcock

The Labconco RapidStill I is equipped with two 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 particles 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 alkalis 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.

Chapter 5:
Replacement Parts

ITEM	PART NUMBER	DESCRICPTION
1	6500100	Glass Still Assembly with PTFE stopcocks
2	1352500	PTFE plug replacements for PTFE stopcock
3	6507700	Heat Controller
4	1876500	Knob, for Heat Controller
5	1327000	Power Switch
6	1331300	Power Cord, 115V
7	1327200	Circuit Breaker, 3 amp
8	6506900	Shelf for Receiving Flask
9	6504201	Heater assembly, complete with stopper and male plug; 115 VAC, 280 Watts
10	6507200	Large U-Clamp bolt (1 required)
11	6507100	Small U-Clamp bolt (2 required)
12	6507500	Nuts, for U-Clamps (6 required)
13	1624400	Tubing for U-Clamps (1/4” ID)
14	1355400	Fill Valve
15	1317400	Transformer (230 V to 115 V, 50/60 Hz, 300 Watt)
16	6507600	Water Bottle
17	1625000	Tubing for water outlet (3/8" ID) (6" required)
18	6523100	Tubing for water inlet (12mm ID) (9" required)

Chapter 6:
Wiring Diagram