DIGITAL CHLORIDOMETER

Model 442-5000 (115V/60Hz)
Model 442-5100 (115V/50Hz)
Model 442-5150 (220V/50Hz)

INSTRUCTION MANUAL

Form 0114630 A/ ECO 9426
Printed in U.S.A

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

IMPORTANT NOTICE

This manual should be read carefully by all the end users in order to become familiar with the operation of the Digital Chloridometer. 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, maintenance and troubleshooting 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.

Components Shipped

Carefully check the contents of the carton for damage that might have occurred in transit. Do not discard the carton or packing material until all components have been checked against the following component list and the equipment has been installed and tested.

As shipped, the carton should contain the following:

Catalog #	Description	Qty
442-5000	Digital Chloridometer, 115V/60Hz, or	1
442-5100	Digital Chloridometer, 115V/50Hz, or	1
442-5150	Digital Chloridometer, 220V/50Hz	1
586-0007	Sample Vials, 20 x 40mm, Box of 100	2
442-5064	Vial, Gelatin Reagent	1
442-5068	Bottle, Chloridometer Acid Reagent, 475mL	1*
442-5069	Bottle, Chloride Standard, 100mEq/L, 120mL	1
442-5080	Vial Rack	1
442-5094	Jar, Silver Polish	1
442-5095	Rinse Bottle, Plastic	1
442-5079	Instrument Cover	1
011-4630	Instruction Manual	1
	Replacement fuses:	
531-0020	1 Amp, 115VAC, Slo Blo, or	1
531-0095	1/2 Amp, 220VAC, Slo Blo	1
442-5198	Electrode Care Tag	1
515-1001	Power Cord, 10A, 120V or	1
515-1030	Power Cord, 10A, 220V	1

***International shipments will receive 3 vials of Catalog No. 442-5064 Gelatin Reagent in lieu of Catalog No. 442-5068 Acid Reagent, as acid reagent requires shipment by ground transportation only.**

General Description

The Labconco Digital Chloridometer is a dedicated coulometric titrator designed to determine the chloride ion concentration of a solution. It displays this concentration directly in millequivalents chloride per liter when 10 microliter or 100 microliter samples are used. Some typical samples are serum, urine, biological extracts, food product extracts, industrial effluents and other aqueous solutions. The combination of silver ions and chloride ions is a quantitative reaction that results in an insoluble precipitate of silver chloride (AgCI). This reaction is carried out at a constant rate by passing a fixed direct current between a pair of silver electrodes immersed in an acid solution. The anode, which is consumed in this reaction, is a continuous spool of silver wire. As the immersed portion of the wire is consumed, fresh wire is drawn from the spool. As the equivalence point of the reaction is reached, an increase in current between a pair of separate indicator electrodes is detected. At a preset indicator current, the instrument automatically stops the incremental counter and the generation of silver ions. Since the generator current is constant, the total titration time is directly proportional to the number of chloride ions that are introduced into the sample vial. The instrument displays this relative time in units of millequivalents chloride per liter.

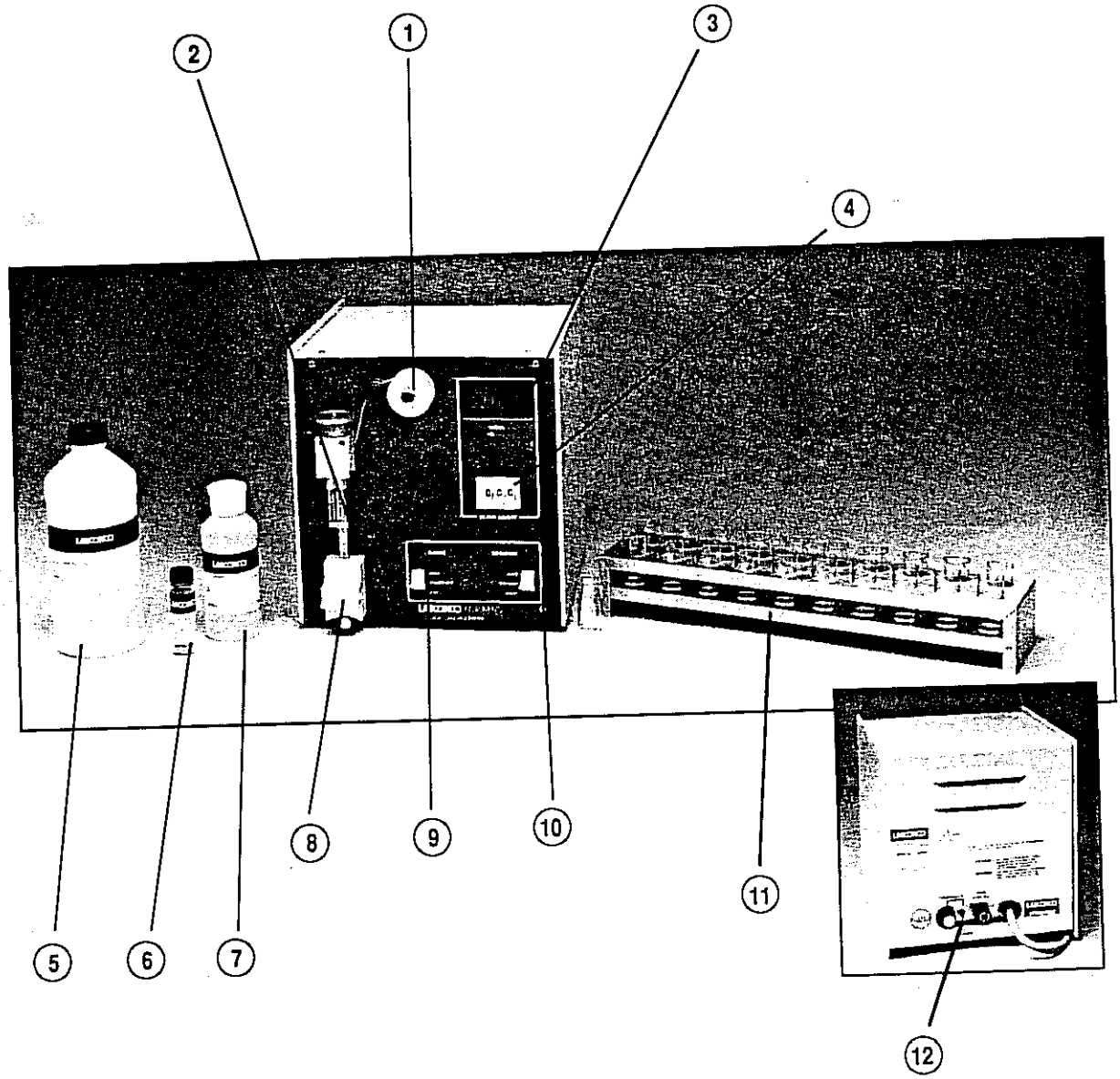
The Digital Chloridometer is a self-contained instrument requiring no special installation procedures. Simply connect the grounded AC line cord to a convenient wall outlet and the instrument is ready for operation. No warm-up time is required.

Specifications

Display Range:	1.0 to 999.9 mEq/L
Reproducibility:	±0.5%*
Accuracy:	±0.5%*
Resolution:	1mEq/L with standard 3-digit readout. 0.1mEq/L with optional 4-digit readout (cover plate removed to expose fourth digit).
Titration Time:	Less than 20 seconds for 100mEq/L concentration.
Sample Size:	10µL on **LOW** range for direct reading 100µL on HIGH range for direct reading
Display:	Digital, 3 or 4 digits, 5/8" high
Dimensions:	21.4cm wide x 22.3cm high x 22.3cm deep (8-3/8" x 8-3/4" x 8-3/4")
Net Weight:	4.5Kg (10 lbs.)
Shipping Weight:	6.8Kg (15 lbs.)

*Generally limited by the sample dispensing system.

DESCRIPTION OF UNIT & ACCESSORIES



1. **SILVER WIRE** - A continuous spool of wire is the anode in the electrode assembly. The wire is consumed in the reaction and fresh wire is drawn from the spool.
2. **ELECTODE ASSEMBLY** - Cathode and anode electronically generate silver ions at a constant rate. A stirrer mixes the sample during titrations.
3. **DIGITAL READOUT** - Directly displays from 1 to 999 mEq/L with an optional fourth digit (0.1mEq/L)
4. **BLANK ADJUST** - 3-digit thumbwheel counter with correction maximum value of 99.9mEq/L. When samples are titrated, one per vial, the background reading of the reagent solution may be automatically subtracted by setting the average blank reading on these thumbwheel switches. During this subtraction process, the display is blank.
5. **ACID REAGENT** - Complete reagent solution to which the sample is added directly for titration. Store at room temperature. Expiration date is printed on the bottle.
6. **GELATIN REAGENT** - Combined with acid solution, this reagent is used as a susbtitute for Chloride Acid Reagent (which can only be shipped via ground transportation).
7. **CHLORIDE STANDARD SOLUTION** - Contains 100mEq/L chloride ions ±1.0%. This solution is used to initially calibrate the unit in conjunction with the **STANDARD/COMPENSATE** switch on the rear of the Chloridometer. Also used to monitor continued calibration of the unit.
8. **VIAL HOLDER** - Raises and lowers the sample vials, introducing the sample to the electrodes and locking the vial in position.

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9. **RANGE** - 3-position switch turns the unit on and selects the appropriate current level for the sample volume. **HIGH** corresponds to 100µL samples and **LOW** corresponds to 10µL samples.

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10. **TITRATION** - 3-position switch for Auto/Standby/Start, in **AUTO** position, the addition of each sample with a concentration greater than 30mEq/L will automatically reset the display and begin a new titration. In **STANDBY** position, the switch must be momentarily depressed to **START** to begin a new titration.
11. **VIALS & RACK** - Included are 200 vials (20 x 40mm) and rack which has capacity to hold 20 vials.
12. **STANDARD/COMPENSATE SWITCH** - A toggle switch on the back of unit used to correct calibration.

NOTE: Suitable care must be exercised in the preparation and handling of acid solutions. See Preparations of Reagents, page 6

Preparation of Reagents

1. **Chloridometer Acid Reagent** - supplied ready to use.

2. **Chloride Standard Solution** - supplied ready to use.

NOTE: The following are not required when using the preformulated Acid Reagent but may be substituted for it.

3. **Acid Solution** - To 900mL of distilled water, add 6.4mL concentrated nitric acid and 100mL of glacial acetic acid. Use reagent-grade chemicals only.

4. **Gelatin Reagent** - Heating one liter of distilled water to boiling, slowly empty the contents of one vial into the water while continuously stirring until the powder is dissolved. The gel solution may be stored under refrigeration for up to six months and may be kept at room temperature for eight hours. For convenience it is suggested that the gelatin solution be stored in small (10mL) vials.

Add 4 drops to acid solution prior to titrating the sample. Once the gelatin is added, the solution will turn violet.

Electrode Cleaning and Conditioning

1. With the instrument off, check that the generator electrode (silver wire spool) is the same length as the other electrodes and is thicker than the shaft of an ordinary pin. If it is not, snip off the thinning segment and draw off enough wire from the spool to even its length with the other electrodes. Tighten the binding post (located to the left of the wire spool) so that it makes good contact with the silver wire. Do not put the wire through the hole in the binding post since this will deform the soft metal.

2. Thoroughly clean all four electrodes with silver polish, rinse with distilled water and buff with tissue. Be certain no residue remains between the indicator electrodes at their common mounting post. Avoid getting skin oils on the electrodes.

3. After cleaning, place a vial filled with 4mL of Chloridometer Acid Reagent or 4mL of acid solution and 4 drops of gelatin reagent in the vial holder. Set the **RANGE** switch to **LOW**, the **TITRATION** switch to **AUTO** and raise the holder so that the electrodes are immersed and the stirrer begins.

4. If a reading does not appear after 30 seconds, re-rinse the electrodes and re-titrate using a fresh vial. Do this until a reading is obtained.

Calibration

1. Titrate several 100µL or 10µL samples of the 100mEq/L solution utilizing the **HIGH** or **LOW** range respectively.

2. If the results are consistently **HIGH** or **LOW**, the direct readout may be corrected to show the actual concentration by using the **STANDARD/ COMPENSATE** switch on the rear of the unit.

3. Switch the toggle from **STANDARD** to **COMPENSATE** and adjust the knob. For each knob revolution, the reading is changed by 2.5% on 115V/60Hz units and 3% on 220V/50Hz units. Perform a new titration using the standard solution in order to determine the magnitude of each adjustment.

4. The toggle switch must remain in the **COMPENSATE** position of all future measurements to show the affect of the adjustment. If the compensation is no longer needed, return the toggle switch to the **STANDARD** position.

Display readings to 0.1 mEq/L

To expose the fourth display digit:

1. Remove the top/back/bottom panel by loosening the screws on one side panel and removing the four screws on the top and bottom panel. The U-shaped housing will now slide off to the rear of the unit.

2. A one inch wide strip of black metal is screwed to the supporting frame just above the front panel display window. Remove the screw and the strip to expose the fourth digit.

NOTE: The Digital Chloridometer has been supplied with a three-digit display for reading to one millequivalent resolution. We have found that in most clinical or quality control applications that this is sufficient resolution based on the limits imposed by dispensing accuracy and clinical standards. A fourth digit (0.1mEq/L) can be easily exposed on this instrument. We do not recommend that this procedure be followed for standard applications of the Chloridometer. It is provided for research applications requiring greater resolution.

Test Procedures

SERIAL TITRATION- Appropriate for multiple samples with a minimum concentration of 30mEq/L. Two methods may be used for serial titration:

A. FIRST METHOD

1. Pipette 4mL of acid reagent or 4mL acid solution and 4 drops of gelatin reagent into the vials supplied with the unit.
2. Place the range switch in the **LOW** or **HIGH** position for 10µL or 100µL samples respectively. When in the **LOW** position, more samples can be titrated per vial. Larger sample size increases accuracy.
3. Place the vial in the vial holder and slide it up until the electrodes are immersed.
4. Place the **TITRATION** switch to the **AUTO** position. Within a few seconds, the display will begin to count. The final reading corresponds to the blank reading of the solution and should be disregarded.
5. Keep the vial in the raised position and add 10µ or 100µL of sample. The addition of a sample with a chloride concentration greater than 30mEq/L will automatically reset the display to zero and begin the titration.
6. After the endpoint is reached and the number is recorded, a new sample may be added.
7. As the solution becomes whiter with precipitated silver chloride, the delay before a titration begins will increase until a titration cannot be initiated.
8. Lower the vial, replace with fresh vial and, after raising the new vial, proceed as described in step 4 above.
9. For each new vial, the titration procedure is the same. The electrodes only need to be cleaned if there are visible deposits. When the instrument is not in use, leave the electrodes immersed in distilled water and set the **TITRATION** switch to **STANDBY** or the **RANGE** switch to **POWER OFF**.

B. SECOND METHOD

1. Fill the sample vials in the same manner as described above.
2. Place the **TITRATION** switch to **STANDBY**.
3. Select the appropriate range (**HIGH** for 100µL samples or **LOW** for 10µL samples).
4. Place the vial in the vial holder and slide it up until the electrodes are immersed.

5. Depress the **TITRATION** switch to start the unit. The final value corresponds to a blank value which is disregarded.

6. Lower the vial holder and add your sample.

7. Raise the vial and press the **TITRATION** switch to start to initiate the titration process.

8. The Digital Chloridometer will now display the value for the sample.

9. Lower the vial, add fresh sample and, after raising the vial, depress the **TITRATION** switch to start the unit.

NOTE: In both methods, the chloride concentration should be 30mEq/L. The start of titration will be delayed by excess AgCI accumulated in the vial. The limitation on the number of determinations done per vial is both the total volume added and the amount of AgCI generated.

INDIVIDUAL TITRATION

Blank Determination

1. Place **TITRATION** switch in **STANDBY**.

2. Select the appropriate range for the sample volume to be used.

3. Fill each of four vials with 4.0mL of Acid Reagent or acid solution plus four drops of gelatin reagent.

4. Place each vial in turn on the vial holder, raise the holder and depress the **TITRATION** switch.

5. Determine the average of the four blank determinations and enter that number on the **BLANK** thumbwheel switch. If the blank is greater than 100, enter the blank value minus 100 on the thumbwheel switch and subtract 100 manually for all determinations.

6. Place 100µL or 10µL of sample in a vial containing 4.0 mL of fresh Acid Reagent or acid solution/gelatin reagent.

7. Place this on the vial holder, raise into position and depress the **TITRATION** switch. The blank value will be automatically subtracted and the reading displayed will be the chloride concentration of the sample.

Measurements of Very High and Low Chloride Concentrations

LOW CHLORIDE CONCENTRATION - For enhanced resolution and accuracy, titrate 100µL samples on the **LOW** range and move the decimal point one place to the left.

HIGH CHLORIDE CONCENTRATION - For greater measurement speed and/or enhanced readout capability, titrate 10µL samples on the **HIGH** range and shift the decimal point one place to the right. The same manipulation may be done with other sample sizes as long as the display reading is corrected. For example, if 200µL is added on the **LOW** range (20 times the volume the instrument expects) the display reading will have to be divided by 20.

NOTE: If the **RANGE** switch is changed during serial titrations, the first reading after the range is changed will be incorrect. this is due to an overtitration time difference (proportional to the amount of silver ions generated before the sense circuitry stops the counter) between the **LOW** and **HIGH** ranges. All readings after this transitional one will be accurate.

VERY LOW CHLORIDE CONCENTRATIONS - For very low concentration samples that read less than 30mEq/L even when 100µL of sample is added on the **LOW** range (a reading of 30 in this case would be equivalent to 3mEq/L), more sample must be added. Since the blank time and overtitration time are related to total volume, the combination of sample and reagent should closely approximate 4mL for a single test.

In this case a 4 times more concentrated (0.4 N HNO_3 and 40% glacial acetic acid) stock solution should be prepared as follows: 250mL distilled water is added to a clean, 500mL volumetric flask. To this, add 12mL concentrated nitric acid and then 200mL glacial acetic acid. Bring the total volume to 500mL with distilled water. The solution will then be diluted to the standard 0.1 N $HNO_3$10%CH_3COOH solution using sample and/or distilled water in the sample vial.

Example: 3mL of an unknown is to be titrated on the **LOW** range (300 times the normal volume).

First obtain an average blank value using 1mL of the 4X concentrated acid solution, 3mL distilled water and 4 drops of gelatin. Enter the blank number in the **BLANK ADJUST** switches. Now, add 1mL of the 4X concentrated acid solution and 4 drops of gelatin to 3mL of the unknown. Titrate this sample in the normal manner and divide the final readout by 300.

Typical conditions for measuring low concentration of chloride over more than a hundredfold range are as follows:

ANTICIPATED CONCENTRATIONS* mEq/L

		0.05-0.9	0.9-5	5-30**
Conditions	Blank	1.8-32	33-177	177-1065ppm
mL sample	0	3.0	0.5	0.1
mL .4N HNO_3				
40%CH_3COOH	1.0	1.0	1.0	1.0
drops gelatin	4	4	4	4
mL distilled H_2O	3	0	2.5	3.0
total volume	4.0	4.0	4.0	4.1
display reading	-	15-270	45-250	50-300
correction factor	--	1/300	1/50	1/10

*Assumes using **LOW** range in all cases.
**Concentrations in this range can use the serial titration procedure and the standard acid solution.

CORRECTION FORMULA FOR CONVERTING DISPLAY UNITS

For solid Samples: mEqCI/L to %NaCI:

$$\%\text{Na CI} = \frac{(\text{reading})(5.85)(\text{final sample volumes, liters})}{\text{original sample weight, grams}}$$

This assumes a known weight of a solid sample has been mixed in a known final volume of solution.

For liquid samples:

$$\%\text{Na CI} = \frac{(\text{reading})(5.85)(\text{final sample volumes, liters})}{(\text{sample volume, liters})(\text{densityg/l})}$$

mEq CI/L to mg NaCI/L: mg NaCI/L = (reading x (58.5)
mEq CI/L to ppm CI: ppm CI = (reading) x (35.5)

The following conditions can contribute to an error in the Chloridometer's readout:

1. An error in sample volume is the most common source of an inaccurate reading. New operators should familiarize themselves with pipetting into the small vial by performing serial titrations with a standard salt solution. The measure of reproducibility obtained will establish confidence in the technique or suggest areas of pipetting improvement.

2. Bromide and iodide will combine with silver in the same manner as chloride and cannot be differentiated by the Chloridometer. The Chloridometer actually reads the total concentration of CI, Br, and I.

3. Be certain that the electrodes and vials are clean and that the generator electrode is of the proper length and is securely connected to the binding post.

4. Very high protein to chloride concentrations can slightly elevate readings.

5. If the toggle switch on the rear of the unit is in the **COMPENSATE** position, the reading may be in error (refer to Routine Calibration Checks).

6. Discard any prepared gelatin solution (if used) that has remained at room temperature for more than 8 hours or that has been refrigerated for longer than 6 months.

Basic Electronic Checkout

In order to determine that the Digital Chloridometer is functioning properly, the following checks should be made:

1. Place the **RANGE** switch in **HIGH** position and the **TITRATION** switch in the **AUTO** position.
2. Set the **BLANK ADJUST** switches to 00.0.
3. Raise the empty vial holder. The stirrer should start rotating and in approximately four seconds the readout should start counting.
4. Short the two sensing electrodes located on the left side of the electrode assembly by using a jumper wire or the blade of a screwdriver. This should cause the readout to stop counting. Removing the short will cause the counter to immediately reset to zero and begin counting again.
5. Set the **BLANK ADJUST** switches to 60.0 for instruments powered by a 60 Hz AC line or 50.0 for units powered by a 50Hz AC line.
6. Place the **STANDARD/COMPENSATE** switch located on the back of the unit, in the **STANDARD** position.
7. Short the sensing electrodes again. Removing the short will now cause the display to go blank for precisely ten seconds and then resume counting.

ROUTINE MAINTENANCE

Do not clean the unit with organic solvents or abrasive cleaners. Instead use mild detergent or soap. Do not allow the plastic portion of the electrode assembly to be immersed in the acid solution during titration. If the electrode assembly becomes damaged or deteriorated past the point of reliable operation, it may be replaced by simply unplugging the old assembly and plugging in a new one. Refer all electrical service problems to qualified service personnel. To quickly screen the instrument for any electronic problems, refer to the Basic Electronic Checkout Procedure. The only maintenance required for the Digital Chloridometer beyond general cleanliness is occasional polishing of the electrodes and immersion of the electrodes in distilled water when the unit is temporarily not in use.

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 product.

Labconco Corporation warrants products of its manufacture for one year, from receipt of the equipment by the 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 this 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.

SHIPPING CLAIMS

If a shipment is received in visibly damaged condition, be certain to make a notation on the delivering carrier's receipt and have their 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.

If you have any questions that are not addressed in this manual, or if you need technical assistance, please contact Labconco's Sales Information Department at 800-821-5525 and Service Information at (800)-522-7658 or (816) 333-8811 between the hours of 7:00 a.m. and 6:00 p.m. Central Standard Time.

Labconco's mailing address is:

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