



TL-2800 AMMONIA/NITRATE ANALYZER

SYSTEM INSTRUCTION MANUAL

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Timberline Model TL-2800 Ammonia/Nitrate Analyzer

System Instruction Manual

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SECTION 1 - INTRODUCTION

PRINCIPLES OF OPERATION

The Timberline Model TL-2800 (Single Channel) and TL-2900 (Dual Channel) Ammonia/Nitrate Analyzers are based on the principle of diffusion across a gas diffusion membrane coupled with electrical conductivity measurement as first described by Carlson¹.

The pump directs the sample, caustic, and absorbing solutions into the Membrane Diffusion Cell. Within the cell, the sample is mixed with a caustic solution. The resulting solution has a pH sufficiently high (pH 11-13) to convert virtually all of the ammonium ion present in the sample to dissolved ammonia gas, as shown in EQUATION 1. The sample/caustic solution flows past one side of a membrane that is permeable to gases but not to liquids or ionic species. The dissolved ammonia gas in the sample/caustic mixture will diffuse across the membrane.



On the other side of the membrane, a buffered solution² absorbs the diffused ammonia gas. The buffered solution then flows into the conductivity detector. The flow passes through a low volume heat exchanger to establish thermal equilibrium of the solution before it reaches the conductivity cell. The conductivity cell measures the change in electrical conductance of the absorbing solution. This change is proportional to the concentration of ammonium in the original sample.

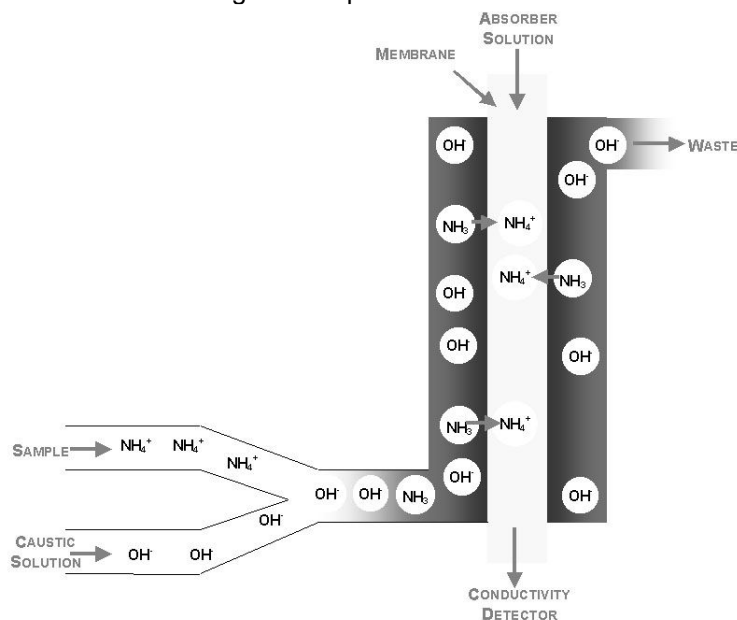


Figure 1-1: Diffusion Membrane Cell Operation Principles

INTRODUCTION TO NITRATE/NITRITE ANALYSIS

Passing the sample/caustic mixture through an optional zinc reduction cartridge reduces nitrate and nitrite ions to ammonium, allowing measurement of the total inorganic nitrogen concentration.³ If ammonium has previously been determined on an unreduced sample, nitrate plus nitrite may be calculated from the difference in values before and after reduction. The nitrate and nitrite in the sample is reduced to ammonia, the metal is oxidized, and hydrogen gas is evolved.



¹ Carlson, R.M.; *Anal. Chem.*, 50 (1978) 1528

² Patent Pending, Hansen-Williams Patent

³ Carlson, R.M.; *Anal. Chem.*, 58(1986) 1590

Small amounts of hydrogen gas are generated at irregular intervals and cause erratic pulses of air to enter the sample channel. These erratic pulses of gas cause an unstable signal due to the increased concentration of ammonia in the air gaps. The introduction of a continuous airflow from air segmentation in the sample/caustic line eliminates these pulses.

Carlson best outlines nitrate determination in his 'Reduction of Nitrate to Ammonia with Zinc' article (Robert M. Carlson, "Continuous Flow Reduction of Nitrate to Ammonia with Granular Zinc," *Analytical Chemistry*, (1985)).

Use of zinc allows for quantitative measurement of ammonia and nitrate. Since many practical samples contain more nitrate than ammonia it is possible to obtain the nitrate values by difference.

$$\text{Total Amount Ammonia + Nitrate} - \text{Amount Ammonia} = \text{Amount of Nitrate} \quad \text{Eq. 1-3}$$

INTERFERENCES

Interferences can be expected from any substance that can transfer across the membrane and dissolve in the absorbing solution to change its conductivity. From the alkaline sample/caustic stream, the likely candidates for interference are volatile amines. Volatile amines such as methyl and ethyl amines will interfere but are not present in most practical samples.

Volatile acids will not cause problems for ammonia determination at the high pH produced by the caustic solution. If the sample were analyzed at a low pH, volatile acids (i.e., carbonic, acetic, etc.) would traverse the membrane and produce a response. The instrument can be used to determine such acids by mixing the sample with a nonvolatile acid (i.e. sulfuric, citric, etc.).

The reduction of ammonia volatility by complex formation with some metals may reduce its availability to transfer across the membrane. Some metallic ions, such as mercury, are known to form very stable complexes with ammonia, but experience to date indicates that the DTPA binds the metal ions sufficiently to release the sample ammonia and thus eliminate this potential interference.

Wetting agents or organic solvents must be avoided. They can cause the membrane to wet out and lose its ability to exclude solution transfer.

WHAT YOU WILL NEED

In addition to the Ammonia/Nitrate Analyzer you will need:

- pH measuring device
- stir bar and plate

REQUIRED REAGENTS

- Ammonia-Free Deionized Water
- Sodium Hydroxide or Potassium Hydroxide, Reagent Grade, ACS or Better
- A High Purity Ammonium Salt for Standards
- Boric Acid, Reagent Grade, ACS or Better (For Absorbing Solution)
- Ammonium Hydroxide, Reagent Grade, ACS or Better (For Neutralizing Boric Acid Solution)
- Diethylenetriaminepentaacetic Acid (DTPA), Reagent Grade, ACS or Better (For samples containing high levels of Ca or Mg)

SYSTEM COMPONENTS

DIFFUSION MEMBRANE CELL AND COMPARTMENT

The Diffusion Membrane Cell directs the liquid flow through the tubular membrane to the conductivity detector. These include the caustic solution, the absorbing solution, the samples, and a recommended air segmentation line. The caustic and sample solutions are mixed in a tee before entering the gas diffusion cell. This cell is housed in a temperature-controlled enclosure. Included in this enclosure are two pre-heating coils used to heat the incoming reagents to a stable temperature. The temperature should be maintained at least 5°C above the highest expected ambient temperature. Temperature control improves baseline stability, reproducibility and detection limits.

With proper care, the diffusion cell should last for 15,000 samples. It is warranted against manufacturing defects for 1 year.

CONDUCTIVITY MEASUREMENT

An ultra sensitive conductivity monitoring system is used to detect the ammonia present in the absorbing solution after it passes through the diffusion membrane. The flow cell is located in a temperature-controlled compartment close to the outlet of the diffusion membrane. The electrical signal obtained from this cell is collected and reported by the conductivity meter. The meter is capable of correcting for the background conductance of the absorbing solution by using a zeroing circuit. The system also has operating ranges to accommodate a wide range of sample concentrations. The output of the meter is displayed in Timberline's TL-2800 data acquisition system.

TOTAL INORGANIC NITROGEN ANALYSIS COMPONENTS

Reduction of nitrate and nitrite to ammonium ion allows for the determination of total inorganic nitrogen. An inline reduction cartridge performs this conversion. With Timberline's data acquisition system, it is possible to automatically switch between running with the reduction cartridge and bypassing the reduction cartridge.

FLUID LINE CONNECTIONS FOR VARIOUS APPLICATIONS

Optional zinc reduction cartridges, filter cartridges, and air tees can be added between the sample/caustic tee and the sample/caustic inlet. FIGURE 1-2: shows the fluid connections for different applications of the system.

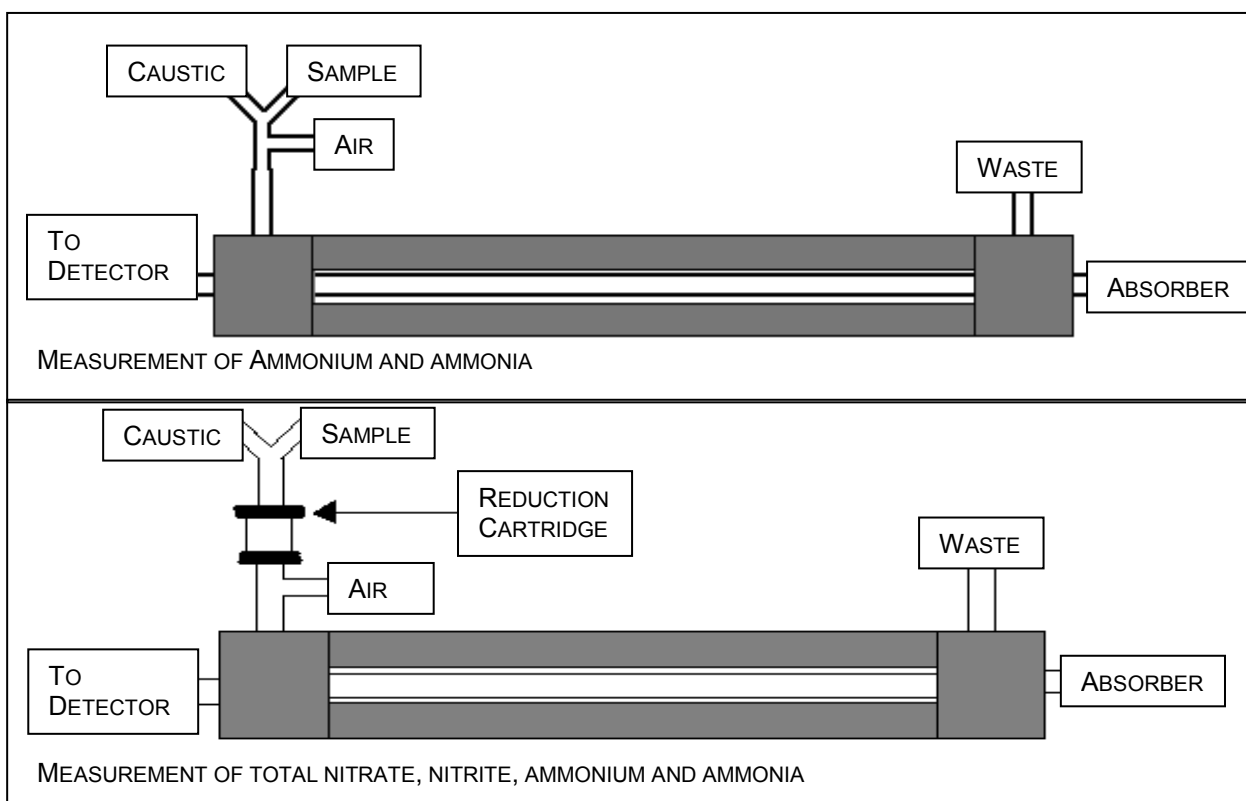


Figure 1-2: Fluid Connections for Various Applications from the Diffusion Membrane Cell

Diffusion Membrane Cell Pressure

The pressures inside the Diffusion Membrane Cell must be maintained to achieve minimum baseline noise and efficient ammonia transfer. All the system pressures should be kept in the range of 5-12 psig. A pressure gauge can be attached to the absorber outlet line using a tee to monitor the pressure. The pressure on the caustic/sample side of the membrane must be lower than the pressure on the absorber side.

Caustic/Sample Pressure

To achieve this, the waste outlet of the Diffusion Membrane Cell must drain to a waste reservoir placed at least 12" below the system. Additionally, the tubing supplied to connect the waste outlet of the Diffusion Membrane Cell to the waste reservoir is of a larger diameter.

Absorber Pressure

A pressure relief valve is used to control the pressure of the absorber line at approximately 6 psig. Alternatively, a restriction tube can be placed in the absorber outlet line (for example, 10 - 12" of 0.01" ID PEEK Tubing).

PERISTALTIC PUMP

A peristaltic pump is used to direct the reagents and samples into the system. The flexible tubing is held against the pump rollers with a channel cartridge. Stops on the tubing secure the tubing to the cartridge. The flow rate is controlled by the ID of the tube. The pump tubing is considered a consumable. With use, the tubing will wear and must be replaced. The actual flow rate can be determined by measuring the volume of water that can be collected in a minute with a graduated cylinder.

SECTION 2 - REAGENT PREPARATION

The ammonia/nitrate analyzer uses a caustic solution to convert the ammonium ion to dissolved ammonia gas and an absorbing buffer solution to absorb the ammonia gas as it diffuses across the membrane.

All solutions should be made from ammonia-free deionized water. Reagent grade chemicals should be used; any un-dissolved salts should be filtered from the solution before use. Generally, the system can be run without filters. However, a filter cartridge can be placed in the sample/caustic line to eliminate any particles from the sample or caustic solutions, or those that may form during the mixture of these, from entering the system. The zinc reduction cartridge is used when total inorganic nitrogen determination is desired.

CAUSTIC SOLUTION

The caustic solution is a dilute sodium hydroxide or potassium hydroxide solution. For most samples, sodium hydroxide works quite well and can be conveniently prepared from commercial 50% NaOH. For samples containing high levels of Ca or Mg, the caustic should be made up with KOH and a complexing agent.

PURPOSE

The caustic solution serves two purposes:

- To neutralize acid in the sample and raise the pH sufficiently high (11-13) to shift the $\text{NH}_3\text{-NH}_4\text{OH}$ equilibrium substantially toward NH_3 gas.
- To complex any metals in the sample and prevent their precipitation at the elevated pH values of the caustic solution.

CHEMICAL PURITY

The highest grade of NaOH, KOH, and DTPA available should be used. Less pure chemicals may contain ammonia, which will raise the baseline, or contain insoluble salts that block the system flow path.

SAMPLES WITH CALCIUM, MAGNESIUM, OR OTHER METALS

For samples containing high levels of Ca or Mg, diethylenetriaminepentaacetic acid (DTPA) can be added to the caustic solution to prevent the formation of CaCO_3 and Mg(OH)_2 precipitates. For these samples, potassium hydroxide is favored over sodium hydroxide because potassium has fewer tendencies than sodium to complex with DTPA, leaving more DTPA available to form a complex with the metals present in the sample. EDTA may be used instead of DTPA when little or no magnesium is present in the sample, but it is not recommended. The MgDTPA complex stability is substantially greater than that of the MgEDTA.

The caustic reagent should have enough diethylenetriaminepentaacetic acid (DTPA) to chelate the maximum concentration of calcium, magnesium or other metals expected in the samples and provide 10% in excess. The potassium hydroxide (KOH) concentration needs to be high enough to convert the DTPA to the penta-potassium salt and neutralize the maximum amount of acid expected in the sample. It must also provide a minimum amount of 0.1M free KOH in the final mixed sample/caustic stream. An excess of DTPA and KOH in the final mixture is recommended although extreme excess should be avoided. Always observe the sample/caustic mixture after changing the sample matrix or caustic solution. Any particles being formed are a result of too little DTPA.

Slight ammonia contamination is generally found in the DTPA and KOH. Excessive amounts may increase the background reading. Since samples and standards are both mixed with the same caustic solution in the same ratio, this slight ammonia contamination is automatically corrected.

In addition to the maximum expected calcium and magnesium concentration and sample acidity, the required caustic solution composition depends on the relative sample and reagent pumping rates. Actual pumping rates for the individual channels are easily estimated by timing the removal of liquid using a small graduated cylinder.

COMPOSITION

The composition of the caustic solution may be varied to accommodate various sample compositions. In general, exact concentrations of reagents are not necessary for successful operation. A slight excess of reagents is always desirable. Check the pH of the final sample/caustic stream mixture (waste) to ensure it is 11-13 (which is necessary to convert the ammonium ion to ammonia gas).

TABLE 1 provides representative caustic concentrations that have been used successfully in particular applications.

Table 1: Commonly Used Caustic Solutions: Compositions of NaOH, KOH, and DTPA in deionized water.

ANALYSIS	NaOH	KOH	DTPA
Water, sub ppm	1%		
Wastewater	5%		
Acetic Acid or KCl Extracts		3.6 %	1%
Kjeldahl Digests	10-15%		

ABSORBER SOLUTION

Boric acid buffered in deionized water, adjusted to pH of 6.5-7 by addition of ammonium hydroxide is recommended as the absorbing solution. The use of pH 6.9 borate buffer substantially enhances baseline stability and provides greater sensitivity.

EXAMPLE CALCULATION FOR PREPARATION OF 100PPM BORIC ACID BUFFER SOLUTION:

Calculation for Grams per Liter:

$$100\text{ppm Boric Acid} = \frac{100\text{mg Boric Acid}}{1\text{L}} \times \frac{1\text{g}}{1000\text{mg}} = \frac{0.1\text{g Boric Acid}}{1\text{L deionized water}} \quad \text{Eq. 2-1}$$

After preparing the boric acid in solution, dilute some aqueous ammonium hydroxide with deionized water, and add drops of this until the pH of the solution reaches approximately 6.9.

CARE OF THE ABSORBER LINE AND SOLUTION

It is recommended to continuously stir the solution using a magnetic stir bar and plate to maintain a homogenous solution. For low level work (sub 50ppb) the headspace of the solution reservoirs should be purged with nitrogen gas to control any changes in the background conductivity of the solutions through dissolution of CO₂ or NH₃ from the atmosphere.

DEGASSING THE ABSORBER SOLUTION

A significant number of bubbles in the absorbing solution may cause baseline disturbances as the bubbles pass through the cell. If such disturbances are observed, the absorbing solution can be degassed by one of the following methods:

- Sparging with helium: Turn the helium tank flow on, but do not exceed 50psi. Let this run for a few minutes and then turn the helium flow off. The solution should be free of bubbles.
- Applying a Vacuum: Pour the deionized water to be used in the buffer solution into a heavy walled vacuum flask, cap the flask with a Teflon or rubber stopper, and attach a water aspirator or vacuum pump to the side arm of the flask. Pull a vacuum on the flask until bubbles stop appearing. Slowly pour the degassed water into the buffer container and continue preparing the buffer solution.
- Filtering: Filter through a 0.2 micron membrane filter under vacuum
- Sonication: Sonication in an ultrasonic bath

There is no need to degas the caustic solution or DI water.

PREPARATION OF STANDARDS

Ammonium standards should be prepared from non-hygroscopic ammonium salts such as ammonium sulfate or ammonium chloride. Nitrate standards can be made from potassium nitrate or sodium nitrate. The standards should be prepared according to the unit of ammonia/nitrate you wish to report, i.e. ammonium ion, ammonia-N, nitrate ion, nitrate-N, etc. A high concentration stock solution can be made from the salt and then be diluted to lower concentrations.

EXAMPLE CALCULATION FOR PREPARATION OF 100PPM N AS NH₄ STANDARD:

Molecular weight of (NH₄)₂SO₄: 132.14

Molecular weight of N₂: 28

$$\frac{28}{132.14} = .2119 \quad 1\text{g (NH}_4\text{)}_2\text{SO}_4 \text{ per liter} = .2119\text{g N per liter} = 211.9 \text{ ppm}$$

Calculation for grams per liter:

$$100 \text{ ppm N as NH}_4 = \frac{.100\text{g N}}{1\text{L}} \times \frac{1\text{g (NH}_4\text{)}_2\text{SO}_4}{.2119\text{g N}} = \frac{.4719\text{g (NH}_4\text{)}_2\text{SO}_4}{1\text{L de-ionized water}} \quad \text{Eq. 2-2}$$

EXAMPLE CALCULATION FOR PREPARATION OF 1PPM STANDARD BY DILUTION:

1 g of 100 ppm standard diluted with 99 g deionized water

$$\frac{1\text{g} \times 100 \text{ ppm}}{1\text{g} + 99 \text{ g de-ionized water}} = 1\text{ppm} \quad \text{Eq. 2-3}$$

SECTION 3 - OPERATION

INITIAL START-UP

1. Turn on the system and wait for it to reach thermal stability.
2. Prepare the caustic solution and place it in the caustic reservoir bottle. Fill the absorbing solution reservoir with prepared buffer. Fill a beaker and the autosampler reservoir with ion-free water.
3. Turn on the computer and the autosampler. Open the TL-2800 Data Acquisition software.
4. Place the caustic and absorber lines in the beaker of water. Start the pump. Observe all connections for leaks.
5. Once you have checked for leaks in all the lines of the system, transfer the ABSORBER and CAUSTIC fluid lines to the respective solutions. Allow the system to equilibrate. Equilibration is noted by the stabilization of the electrode output reading.
6. Zero the electrode output through the software.
7. Inject an ammonia standard that falls within the typical sample range. Observe the peak to ensure that the system has equilibrated (no significant baseline drift or noise compared to the peak). Adjust timing parameters and gain settings accordingly.

This initial set up procedure should be repeated whenever any of the following occur:

- A new batch of caustic or absorbing solution is used.
- The instrument is "cold-started" after the power has been off for more than 24 hours.

ROUTINE OPERATION

WARM-UP TIME

Under normal circumstances, the power to the analyzer should be left on at all times with the pump turned off. If this has been done, essentially no warm-up time is required, and the instrument may be used as soon as the flow has stabilized. The system may still require several minutes to equilibrate. If the instrument has been shut down for a prolonged period, then the Initial Start-up procedures should be followed.

RUNNING SAMPLES

Samples are typically run using an autosampler and the Timberline Data Acquisition system. Sample is introduced through timed injection with the peristaltic pump. Deionized water is introduced between samples to return to baseline. The concentration of ammonia in the sample may then be determined by reference to a calibration curve.

Samples can also be analyzed manually by placing the sample line into a reservoir of sample and waiting approximately 20-30 seconds before returning the sample line to a reservoir of water.

SHUT DOWN PROCEDURE

1. Transfer the caustic and absorber lines to a reservoir of deionized water.
2. Thoroughly rinse the system with water for 5-10 minutes.
3. Shut off pump.
4. Empty the waste reservoirs.

Note: The POWER to the analyzer should be left ON if the instrument will be used in the near future.

USING ZINC REDUCTION CARTRIDGES

The TL-2800 is configured to automatically switch between running samples in ammonia mode, which bypasses the zinc reduction cartridge, and nitrate mode, which includes the zinc reduction cartridge.

Prepare two calibration curves for nitrate and ammonia standards, one with reduction (total inorganic nitrogen concentration), and one without (ammonia concentration). Compare the sample values to the appropriate calibration curves. The concentration of nitrate (plus nitrite) in the sample is determined from the difference between the total inorganic nitrogen concentration and the ammonia concentration.

If there are doubts about the functionality of the reduction cartridge, reduction can be confirmed by comparing the signal from nitrate and ammonium standards of identical nitrogen concentration when both are run in nitrate mode. It is good practice to run these check samples periodically.

In routine operation, the cartridges should be replaced when half of the zinc has been consumed or when check samples indicate that there is no longer a high percent conversion to ammonia.

SECTION 4 - MAINTENANCE

ROUTINE MAINTENANCE

Because of its simple, rugged design, the analyzer requires only minimal maintenance or service on a regular basis.

REPLACEMENT OF THE DIFFUSION MEMBRANE CELL

The tubular membrane should last for an extended amount of time. If the membrane fails, it must be replaced. The entire Diffusion Membrane Cell needs to be removed and returned to Timberline Instruments. You can order Diffusion Membrane Cells from Timberline Instruments, LLC. The Diffusion Membrane Cells are guaranteed against defective workmanship for 1 year.

CONSUMABLES

With use, peristaltic pump tubing will wear and need to be replaced. For those measuring nitrate, the zinc cartridges will need to be replaced when either half the zinc has been consumed or when there is poor recovery on nitrate check samples.

APPENDIX A: APPLICATIONS

Robert M. Carlson, "Automated Separation and Conductimetric Determination of Ammonia and Dissolved Carbon Dioxide," *Analytical Chemistry*, 50 (1978) 1528.

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David R. Hershey and J.L. Paul, "Leaching-Losses of Nitrogen from Pot Chrysanthemums with Controlled-Release or Liquid Fertilization," *Scientia Horticultuae*, 17 (1982) 145.

David R. Hershey and J.L. Paul, "Iron Nutrition of the Broadleaf Evergreen Shrub, *Euonymus Japonica* Thunb.," *Journal of Plant Nutrition*, 7 (1984) 641.

P. R. McCleaf and Edward D. Schroeder, "Denitrification using a membrane-immobilized biofilm" *Journal of the American Water Works Association*, 87 (3) (1995) 77-86.

A.P. Kruzic and Edward D. Schroeder, "Nitrogen removal in the overland flow wastewater treatment process – removal mechanisms", *Journal Water Pollution Control Fed.*, 62 (3) (1990) 867-876.

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Hansen-Williams Patent

APPENDIX B: WARRANTY AND RETURNS

WARRANTY

Timberline warrants its products against defects in workmanship or material under normal use or service for one year. All obligations or liabilities under this warranty are limited to repair or replacement, at Timberline's option, F.O.B. Boulder, CO, of parts that are returned, freight prepaid and which are accepted as being defective upon inspection of Timberline.

Components that are subject to normal wear and/or are scheduled for routine replacement within the warranty period, and/or parts that are subjected to effects of corrosion or deterioration by chemical or other action are excluded from the above warranty. Repair or replacement will not be made under warranty for malfunction because of inadequate facilities, operating conditions or utilities.

Equipment and components must be returned with Timberline's prior approval and contain a Timberline Return Authorization Number. Call Timberline (1-800-777-5996).

Guarantees/Warranties on accessories and equipment included by Timberline from other manufacturers are limited to the guarantees given on such equipment by the respective manufacturers.

Any modifications made to equipment covered by this warranty, without written permission from Timberline will void the warranty. Timberline reserves the right not to honor this warranty if the products are obviously mishandled by the user.

Timberline assumes no responsibility for consequential, economic, incidental damages of any nature, or on-site re-installation costs arising out of future alleged failure of any of its products or their accessories.

This warranty supersedes any and all previous warranties unless otherwise agreed upon at the time of sale, such as for customized equipment.

SHIPMENTS

All shipments are made F.O.B. Boulder, CO. Instruments and major components will be packed and shipped via surface, unless otherwise requested. Supplies and/or replacement parts are packed and shipped via UPS, UPS Blue, air parcel post, or parcel post, unless otherwise requested.

DAMAGED SHIPMENTS

The Interstate Commerce Commission has held that carriers are as responsible for concealed damage as for visible damage in transit. Unpack shipment promptly after receipt, as there may be concealed damage even though no evidence of it is apparent. When concealed damage is discovered, cease further unpacking of the unit involved and request immediate inspection by the local agent or carrier and secure a written report of the findings to support a claim. This request must be made within 15 days of receipt. Otherwise, the carrier will not honor the claim. Do not return damaged goods to the factory without first securing an inspection report and contacting Timberline for a return authorization number.

FILING OF CLAIMS

After a damage inspection report has been secured, Timberline will cooperate in supplying replacements and handling a claim that may be initiated by either party.

RETURNS

No returns may be made without prior notification and authorization. If for any reason, it is necessary to return material to us, please contact our customer service department for a return authorization number and forwarding instructions.

APPENDIX C: SPECIFICATIONS

Figure B-1: Timberline TL-2800 Ammonia/Nitrate Specifications

Principle of Operation:	Membrane Diffusion / Conductivity Detection
Conductivity Range:	10 ppb – 2000 ppm
Detection Limit:	10 ppb
Sample Rate:	Up to 1 per minute
Power:	120V, 60Hz;
Detection:	Alternating Current, Synchronous Detection, 10KHz Frequency
Noise:	Less than 0.0004 μ S (DI Water, 1mL/min)
Drift:	Less than 0.004 μ S/hr (DI Water, 1mL/min, 35°C)
Insulation:	Minimum 1/2" all sides
Heater:	25W DC heating element vulcanized to aluminum block
Preheater:	Tefzel tubing knitted and encased in high thermal mass housing
Fluid Path:	Wetted parts are inert polymers (PEEK, Tefzel) & Stainless Steel
Flow Rates:	1-3 ml/min
Pump motor speed	30 rpm

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