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1.0 Introduction

1.1 Purpose

The Savillex DST-1000 Acid Purification System (DST-1000) is intended to provide a means of purifying acids or water to a level suitable for use in highly sensitive analytical chemistry applications. The DST-1000 is designed and manufactured to be as simple to use and safe to operate as possible.

This manual describes the Savillex DST-1000 and contains product specific information, current as of the date of publication, for the safe use and operation of this equipment. Please read this manual carefully and familiarize yourself with the DST-1000 prior to operating the equipment. Should you have any questions regarding this product, or the contents within this manual, please contact Savillex.

Savillex Corporation

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1.2 Liability Disclaimer

Every effort has been made in the preparation of this publication to provide the user with the most current, correct and clearly expressed information and instructions required for use and operation of the DST-1000. Savillex Corporation assumes no liability or responsibility for damages resulting from errors, omissions and accuracy of the information in this manual. The information included has been compiled from a variety of sources and is subject to change without notification. Savillex Corporation makes no warranties or representations regarding the quality, content, completeness, suitability, adequacy, sequence, accuracy, or timeliness of such information. If you discover any errors or omissions we encourage you to contact Savillex immediately.

We actively request your comments regarding improvements to this manual and the DST-1000 itself. We reserve the right to make technical changes as deemed necessary.

1.3 Sub-Boiling Principle

The Savillex DST-1000 Acid Purification System uses sub-boiling distillation to produce high purity acid or water from a PFA distillation unit (still). Sub-boiling distillation is a process in which a starting material is heated to accelerate vaporization at a temperature well below its boiling point. This heating process gently vaporizes the liquid without bubbling and the associated aerosol formation which can cause contamination of the collected distillate. Sub-boiling distillation greatly eliminates aerosol formation and significantly improves the purification process.

The DST-1000 allows the heated, clean, vaporized liquid to condense upon the cooler surface of the domed section of the still. The pure condensate forms droplets, which collect and flow to the outlet port of the still. The purified liquid distillate is then collected into a suitable container for later use in analyses.

2.0 Safety Information

2.1 Danger Categories and Signal Words

The signal words described below are used in connection with warnings throughout this manual. For your own safety and to avoid property damage, please observe these warnings!

Signal words are highlighted in boldface and often accompanied by symbols denoting the hazard type. Symbols and warnings are taken from the United Nations Globally Harmonized System of Classification and Labeling of Chemicals.

DANGER!	DANGER! Denotes most severe hazards. Failure to follow safety instructions is likely cause severe personal injury, death or severe property damage.	
WARNING!	Denotes less severe hazards. Failure to follow safety instructions may cause severe personal injury, death or severe property damage.	
(!)	This symbol indicates that this is important information regarding the product or refers to a part of the manual that requires particular emphasis.	

2.2 Qualified Users

This unit is intended as an experimental tool for research and development in the laboratory by qualified analytical chemists. Only those individuals that have adequate technical training, knowledge and demonstrated expertise should attempt operation of this unit. Observe all safety procedures and best laboratory practices to assure safe operation. As in all laboratory operations, the user must assure that adequate safety procedures are established to protect all personnel from the potential hazards in the use of this product.

2.3. Intended Use



The DST-1000 is intended **ONLY** for use with the following materials:

Ultra-pure water (H₂O)

≤ 70% nitric acid (HNO₃)

≤ 37% hydrochloric acid (HCI)

≤ 51% hydrofluoric acid (HF)

DANGER! The DST-1000 is not approved for use with any other material. Using the DST-1000 with any other material may result in personal injury or property damage. Please observe all appropriate regulations published by government agencies, professional associations, chemical manufacturers and other regulating bodies when working with chemicals.

2.4 General Safety Information

Purification of acids by sub-boiling distillation requires safety precautions beyond those of general laboratory practices. The following safety information should be read carefully prior to using the DST-1000, and should be observed at all times during the use and operation of the still.

Savillex assumes no liability for injury or damages resulting from improper handling or a failure to comply with this information.







DANGER! CAUSTIC











Electrics may ignite flammable or explosive mixtures. The apparatus is not designed for organic chemicals. Improper use of the DST-1000 with organic material may cause personal injury or property damage.

Causes Severe Skin Burns and Eye Damage

Take appropriate precautions during filling with and dispensing of acid. In case of contact, flush with copious quantities of water. See reagent manufacturer's material safety data sheet (MSDS) for safe handling instructions.

Personal Protective Equipment

Wear recommended safety apparel when handling or working with acids.

Harmful If Inhaled

Avoid inhalation of acid vapors.

Causes Serious Eye Irritation

Wear eve protection.

Ensure the system is in a properly functioning controlled environment (e.g. fume hood). Acid vapor is emitted from the DST-1000 during use.

The Unit Employs High Voltage!

The DST-1000 must be connected to a grounded conductor. Connect the power cord of the control box to a grounded power source.

Chemical Hazard!

Chemical hazards associated with the connections, tubing and containers are identical to those noted above.



No Serviceable Parts! Do not attempt to alter or repair this unit. This instruction manual does not include repair instructions. Contact Savillex or its local agent for service. Do Not Attempt to Open the DST-1000 Reservoir/Condenser Assembly! The DST-1000 is not designed to be opened and has no parts inside the reservoir/condenser assembly. Attempting to separate the reservoir and condenser will destroy the unit.

2.5 Safety Shut-Off

If the system shows signs of damage or fails to function, turn the control knob to the OFF position and unplug the power control box from the electrical outlet. Place an "Out of Order" sign on the still and separate and store the power control unit to prevent accidental use. Indicator signs include:

- Visible signs of damage/leakage
- Loose components (other than tubing and ferrule nuts) are discovered
- The DST-1000 fails to operate

Contact Savillex or its local representative for service. See section 7.2 Troubleshooting.

2.6 Hazards Associated with Connections, Tubing and Containers

DANGER! Fittings, valves, tubing, vent membranes and collection containers associated with distillate or waste collection may contain hazardous chemicals or residue.

Extreme caution should be taken when handling or replacing these items to avoid bodily contact, splashing, or spilling. Appropriate personal protective equipment should be worn to avoid skin and eye contact.

2.7 Electrical Hazards

DANGER! The DST-1000 utilizes high voltage electrical power to control the internal heating device. Repair and servicing of this equipment must only be performed by trained and qualified electrical personnel. Proper safety precautions and procedures must always be observed.



The Unit Employs High Voltage!

The DST-1000 must be connected to a grounded conductor. Connect the power cord of the control box to a grounded power source.



Electrical Hazard!

High voltage is present at the electrical receptacle connector on the control box when energized. Use CAUTION.

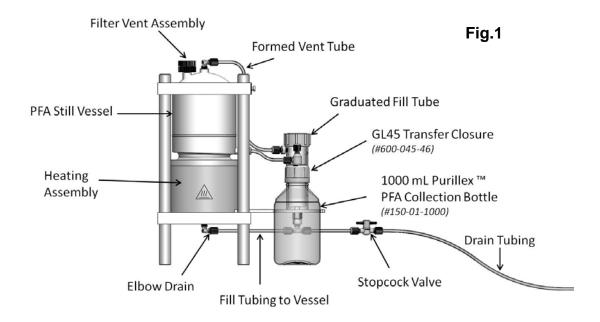
- Always de-energize and remove from the power source before any electrical servicing
- The power cord must only be connected to a wall outlet that is equipped with a separate grounded connection
- Never replace the power cord. In case of damage return to Savillex for replacement
- Should fuse replacement be necessary, use only 1.0 Amp 250V 3AB fast-acting ceramic body style
- Never energize with the control box cover removed
- Never operate in a wet, damp or spray down environment
- Never energize if the power cords have been damaged, frayed, corroded or wires exposed
- Use extreme caution if the heater cable connector is unplugged from the receptacle on the control box. High voltage is present when energized

2.8 Safety Labeling

The DST-1000 is designed with safety as a primary consideration. In addition to safety information in this manual, Savillex has labeled and marked the unit to identify model number, operating voltage, wattage, frequency, serial number and certification authority.

3.0 About the DST-1000 Acid Purification System

3.1 Visual Identification



3.2 Description

The DST-1000 is intended for purification of reagent grade acids or water. The main sections of the still are the reservoir and condenser which are molded from PFA. PFA construction minimizes the contamination potential from extractable compounds and also allows for distillation of hydrofluoric acid. **Note:** THE STILL ASSEMBLY CANNOT BE OPENED.

Acid is added to the unit via a front fill tube which also acts as gauge to show the amount of acid remaining in the still. The large domed PFA condenser operates on the principle of differential temperature between ambient air and the heated solution in the reservoir. There is no need for chilled water baths or re-circulating coolant.

Heat is transferred to the reservoir via a silicone rubber heating jacket with an embedded low wattage resistance heating element. This design provides efficient heat transfer while reducing electrical hazard. The heater is designed so that heating the still at maximum power will not damage the unit even if the still is operated until dry (avoid operating the still in this manner). The heating jacket also has a thermal fuse to disable the heater to further protect against overheating.

The heating jacket is operated by a remote power control unit which should be located away from the DST-1000 and outside of the fume hood. The power control unit is powered by alternating current voltages of 100 VAC, 115 VAC or 230 VAC, specific to local markets and geographic locations. The power cable to the still is sheathed in silicone coated fiberglass to increase the chemical resistance, and the power cable connector is electrically keyed to prevent the mis-application of voltage. The approximate load is 70 watts. The power setting is adjustable, and allows for an operating distillation temperature of 50-90°C. Operating temperatures may vary with the liquid volume in the reservoir, and the temperature of the operating environment.

When the acid is heated to sub-boiling temperatures, high purity acid vapor is produced, which condenses on the inside of the vessel, runs down the side walls into a collection channel, and flows through a tube into the collection bottle.

3.3 Specifications

Power Source (three-wire earth grounded only)

Power 70 Watts (resistance heater)

Shipping Weight 7.25 kilograms/16 pounds

Dimensions

Still with bottle bracket 36.5 cm x 20 cm (L x W)

Height 45.0 cm

Power cord 3 meters (still to control unit)

2 meters (control unit to grounded outlet)

Installed Location Distillation unit within chemical fume hood

Reservoir Capacity 1000 mL

Materials

Still Reservoir and condenser PFA
Purillex 1000 mL PFA collection bottle PFA
Fill Tube Assembly PFA
Tubing and fittings PFA

Heating jacket Silicone rubber
Stand and bottle bracket Polypropylene
Power control ABS housing

Cable to still Silicone rubber sheathed

Ambient Temperature $+21^{\circ}\text{C} \pm 10 \text{ } (70^{\circ}\text{F} \pm 18)$

Distillation Temperature Approximately 50°to 90°C (adjustable)

Warm-up Time 2-3 hours (depending on fill volume)

Over Temperature Protection Provided

Cooling Water Not required

Approx. Distillation Rate (after warm-up) 50 mL/hr @ 90°C for water

30 mL/hr @ 80°C for water 10 mL/hr @ 60°C for water

3.4 Unpacking and Inspection

Open the shipping carton and remove all still components. Check the components against the following packing list and inspect for shipping damage such as cracks, scratches, dents, etc. Should any components be missing or damaged, contact your local representative or Savillex. We recommend retaining all packaging until it is determined that the equipment is functioning properly.

For safety reasons, never operate the unit if it exhibits visible signs of damage.



If the shipping carton and contents are damaged, retain the packaging for inspection by the freight company. Notify your representative or Savillex for assistance in filing an insurance claim.

Packing list for the Savillex DST-1000 Acid Purification System

(1)	Still assembly, PFA reservoir/condenser and stand
(1)	Heating assembly with power cord
(1)	1000 mL Purillex™ PFA collection bottle
(1)	Graduated fill tube and bottle bracket
(1)	GL45 transfer closure with (2) 1/4" tube outlets with vent nut
(4)	Base legs
(1)	PFA distillate tubing, ¼" OD x 22 cm long
(1)	PFA drain tubing, ¼" OD x 90 cm long
(1)	Fill tube connector tubing 3/8"OD x 19.3 cm long
(2)	PFA 1/4" OD ferrule nut
(10)	Replacement 9 mm PTFE filters for bottle vent
(10)	Replacement 25 mm PTFE filters for filter vent
(1)	PFA/PTFE stopcock drain valve
(1)	Power control unit for heating assembly
	(for appropriate local VAC power)
(1)	DST-1000 Acid Purification System user's manual (CD)

Note:

The DST-1000 was shipped with PFA cap plugs on the (1) DISTILLATE port and (1) TEE FITTING DRAIN port. The top vent on the still condenser was also fitted with a 25 mm PTFE filter membrane. Confirm that all of these components are in place.

4.0 Preparation for Use

4.1 Recommended Environment

The DST-1000 should ONLY be operated under an acid fume hood. Although the liquid will remain fully contained within the vessel, small amounts of acidic vapors will be released through the breather vents. It is strongly recommended that DST-1000 be used in a fume hood dedicated to acid purification only. The DST-1000 should not be operated with other sample preparation activities which generate acidic fumes which can damage the DST-1000 heating element. The DST-1000 should not be operated or used with potentially flammable, explosive or contaminating materials. The unit is not rated for use in areas requiring intrinsic safety, explosion proof or any areas that may contain flammable or explosive materials. Please observe all appropriate regulations published by government agencies, professional associations, chemical manufacturers and other regulating bodies.

The DST-1000 power control unit is intended for installation outside the fume hood, with the 3 meter power cable passing through a designated port in the hood to the heating jacket connection.



The following additional conditions are recommended for the environment

Emissions	Operate DST-1000 in fume hood with vent caps and membranes in place.
Ambient Temperature	+21°C ±10 (70°F ±18) Note: The temperature of the environment may affect the rate of distillation and the quality of the distillate.
Relative Humidity	< 85% - High humidity is not good for electrical parts. DO NOT operate the still in wet or wash down conditions.
Space Requirements	The footprint of the DST-1000 stand is 20cm x 20cm x 45cm high. Space should also be allocated for bottle bracket and 1000 mL PFA collection bottle.
Power Source	The power control unit is shipped in a configuration suitable for standard local AC wall outlet power. 100 VAC +/- 5%, 50/60 Hz 115 VAC +/- 5%, 50/60 Hz 230 VAC +/- 5%, 50/60 Hz

Verify that the power source voltage falls within the range given above. If not, contact your local representative or Savillex for advice.

4.2 Installation

DAMGEDI



Explosive; Fire, Blast or Projection Hazard

Electrics may ignite flammable or explosive mixtures.

The apparatus is not designed for organic chemicals. Improper use of the DST-1000 with organic material may cause personal injury or property damage.

Causes Severe Skin Burns and Eye Damage

Take appropriate precautions during filling with and dispensing of acid. In case of contact, flush with copious quantities of water. See reagent manufacturer's material safety data sheet (MSDS) for safe handling instructions.



Personal Protective Equipment

Wear recommended safety apparel when handling or working with acids.

Harmful If Inhaled

Avoid inhalation of acid vapors.

Causes Serious Eye Irritation

Wear eye protection.

Ensure the system is in a properly functioning controlled environment (e.g. fume hood). Acid vapor is emitted from the DST-1000 Acid Purification System during use.



The Unit Employs High Voltage!

The DST-1000 must be connected to a grounded conductor. Connect the power cord of the control box to a grounded power source.



Chemical Hazard!

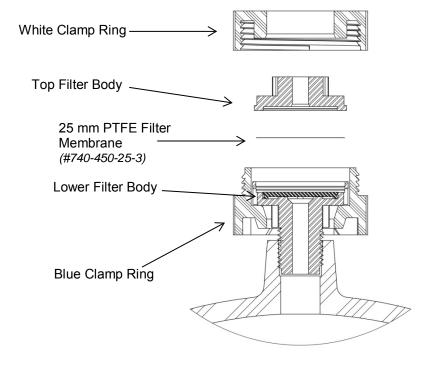
Chemical hazards associated with the connections, tubing and containers are identical to those noted above.

Place the DST-1000 on a solid surface under an acid fume hood. It is strongly recommended that the fume hood be dedicated to acid purification only, and not used for other sample preparation activities which generate acidic fumes. See section 4.1 Recommended Environment.

- a) Lay the DST-1000 unit on its side and screw the (4) base legs onto the bottom plate. Stand the unit upright.
- b) Optional: Place a spill containment tray of suitable material under the still. The tray should have a minimum capacity of 1000 mL. A spill tray is available as an accessory from Savillex (#730-0525).
- c) Run the 3 meter power cord from the distillation unit to outside the fume hood
- d) Connect the power cord from the distillation unit to the power control unit. **Note:** The electrical connector is keyed to the appropriate voltage.
- e) Place the power control unit within 2 meters of an appropriately rated voltage and grounded outlet. Plug the power cord into the outlet but do not turn power ON at this time. See Section 3.3 Specifications.

f) Verify that the top 25 mm filter vent assembly fitting has a PTFE membrane installed and is secure. See Fig. 2 exploded filter vent assembly drawing

Fig. 2



25 mm Filter Membrane Replacement Instructions

- 1. Grasp blue clamp ring while turning white clamp ring counter-clockwise to loosen
- 2. Remove white clamp ring and top filter body
- 3. Replace with new filter membrane being careful that membrane sits flat without wrinkles
- 4. Snap top filter body back in place by first aligning tabs with lower filter body
- 5. Finger-tighten by holding blue clamp ring and threading white clamp ring clockwise
- g) Remove the 1/4" OD ferrule cap from the bottom tee fitting in the fill tube and save for future use
- h) Take one of the 1/4" OD ferrule nuts supplied and slide it on to the 90cm long waste drain tubing
- i) Insert one end of the tubing into the remaining opening in the tee fitting in the bottom of the fill tube assembly (It should be facing away from the DST-1000 unit), slide the ferrule nut up to the fitting and finger tighten. Pull slightly on the tubing to make sure the tubing will not accidentally pull out during use. **Note**: The OD groove on the tubing will catch on the ferrule nut preventing pullout.
- j) Insert the other end of the drain tubing into the stopcock valve provided and finger tighten the ferrule nut. Turn the valve to the CLOSED position. Should you wish to shorten the drain tubing simply cut the tubing off square at the desired length, install the stopcock valve and turn it to the CLOSED position. **Note:** If the tubing is shortened, the section with the OD groove will be lost. Grooving the tubing is not necessary for a leak proof connection, but is a safety measure to prevent accidentally pulling the tube out of the connection. If you wish to groove the tubing contact Savillex and order ¼" groove tool (#730-0004).

- k) If desired, install an additional length of tubing to the other port on the stop cock valve and run it to a waste collection container of suitable material.
- Test the power control unit by turning the control knob on the power control unit to the "HI" temperature setting. The red heater light should illuminate. If heater light does not illuminate, contact Savillex or your local representative.
- m) Turn the control knob back to the OFF position. You are now ready to clean the DST-1000. (Section 4.4)

4.3 Starting Materials

To obtain ultrapure acid with 10 ppt or less elemental impurities, a high quality starting acid is required for distillation. Trace metals grade acid (acid with 1 ppb or less elemental impurities) will typically yield acid that is equivalent to commercial grade ultrapure acid (10 ppt) in a single distillation. Starting acids with higher levels of impurities, such as reagent grade acids, will produce poorer quality distillate and may require multiple distillations to achieve acid that is equivalent to ultrapure acid quality. Even with multiple distillations, poorer quality starting acids may not produce acid that is equivalent to ultrapure acids.

Acid	Commonly Used Concentrations	Azeotropic Concentration with H₂O
HNO₃	67-70%	68%
нсі	33-37%	20.2%
HF	47-51%	37%

Note: Regarding Acid Purification

The final concentration of acid produced by the DST-1000 will likely be different from the concentration of the starting material used. The change in concentration is due to the azeotropic nature of the acids (see table above).

If starting material acid used for distillation has a concentration that is higher than the azeotropic concentration, the concentration of the acid produced will be lower (closer) to the azeotropic concentration after distillation.

The concentration difference is most notable during distillation of HCI which is most often sold at a commercial concentration of 35% and has an azeotrope concentration of 20%. The concentration difference will often produce excess HCI vapor during the distillation process. To minimize the acidic vapor produced, concentrated HCI can be diluted to a concentration closer to the azeotrope prior to distillation.

4.4 Initial Cleaning

The DST-1000 is constructed of the ultimate material for acid purification. All wetted parts within the still reservoir/condenser are made of PFA. PFA is well known to provide the highest level of chemical resistance and lowest level of ionic contamination. To achieve these benefits, an initial cleaning procedure is required to extract any contaminates from the wetted surfaces within the still left after manufacturing. Savillex recommends the following cleaning procedure prior to using the unit for high purity acid distillation. It is also recommended that the DST-1000 be dedicated to a single chemistry to achieve the highest levels of purity.

- a) Check to make sure that the \(\frac{1}{4} \) OD ferrule plug on the distillate output port is finger tight
- b) Verify that the waste drain fittings are secure and that the drain stopcock valve is CLOSED
- c) The unit is now ready for initial filling. It is recommended that the initial starting material for cleaning be the same type and grade of chemical that you are intending to distill and purify. See section 4.3; Starting Materials.
- d) Unscrew and remove the threaded cap from the graduated filling tube
- e) Carefully and slowly pour the chemical into the filling tube. DO NOT FILL TOO QUICKLY as you will notice the chemical drains slowly into the reservoir. The fill tube has graduation marks indicating liquid level in the reservoir.

Note: Should spilling occur, immediately follow spill and safety protocols. See section 7.1 for details.

- f) Continue filling until a liquid level of approximately 500 mL has been reached. You can monitor the liquid level by observing the graduation marks on the fill tube.
- g) Replace the threaded cap on the fill tube and turn clockwise until hand tight
- h) The unit is now ready for cleaning. Turn the control knob on the power control unit to the HI setting.



Explosive; Fire, Blast or Projection Hazard

Electrics may ignite flammable or explosive mixtures. The apparatus is not designed for organic chemicals. Improper use of the DST-1000 with organic material may cause personal injury or property damage.





Take appropriate precautions during filling with and dispensing of acid. In case of contact, flush with copious quantities of water. See reagent manufacturer's material safety data sheet (MSDS) for safe handling instructions.

Personal Protective Equipment

Wear recommended safety apparel when handling or working with acids.



Harmful If Inhaled

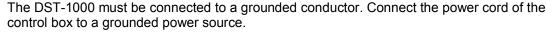
Avoid inhalation of acid vapors.

Causes Serious Eye Irritation



Wear eye protection. Ensure the system is in a properly functioning controlled environment (e.g. fume hood). Acid vapor is emitted from the DST-1000 during use.

The Unit Employs High Voltage!





Chemical Hazard!

Chemical hazards associated with the connections, tubing and containers are identical to those noted above.

- i) The lower section of the PFA vessel will now gently heat to 80°- 90°C. It will take 2-3 hours to reach this temperature.
- j) Allow the still to operate on the HI setting for the next 48 hours. The acid inside the vessel will continually reflux during this time which will clean the entire inside surfaces.
- k) Turn the control knob to the OFF position and allow the DST-1000 to cool
- I) Drain the contents of the unit by opening the stopcock drain valve and directing the contents into an appropriate waste container. Close the drain valve after emptying.
- m) The remaining distillate (approximately 50 mL) in the collection channel inside the vessel will also need to be drained. Carefully tilt the unit backwards so the distillate outlet is angled upwards and the distillate has drained away from the opening.
- n) Remove the ¼" OD ferrule plug from the distillate outlet. Slide the ¼" OD ferrule nut onto the 22 cm long distillate tubing and insert the grooved end into the outlet fitting. Finger-tighten the ferrule nut.
- o) After the tube is attached, level the unit and drain any remaining distillate into a waste container
- p) Remove the 22 cm distillate tubing and set aside for future use. Re-close the port with the ¼" OD ferrule plug.
- q) Repeat the above cleaning procedure using high purity de-ionized water
- r) Completely drain the unit and close all ports. The unit is now ready for use.

4.5 Cleaning of 1000 mL Purillex™ PFA Distillate Collection Bottle and Transfer Closure

Cleaning and preparation of the 1000 mL distillate collection bottle is as important as cleaning and preparation of the DST-1000 acid purification system. Although Purillex bottles are manufactured and bagged inside a cleanroom, for ultratrace (low ppt level) metals use, such as the collection and storage of high purity acids, they should be thoroughly cleaned prior to their first use.

You are welcome to follow the established methods and protocols within your laboratory for cleaning PFA labware products prior to use. In addition, a guideline for cleaning the bottles prior to use is provided in the steps below.

- a) Rinse the bottle thoroughly with Deionized Water (DIW)
- b) Fill with a solution of 2% high purity HNO₃ /1% high purity HF and replace the closure
- c) Store bottle at 50°C for a minimum of 48 hours
- d) Empty the bottle and rinse thoroughly with DIW
- e) Repeat steps b-d
- f) Bottle is now ready for ultratrace use



If HF cannot be used, use HN03 alone.

The accompanying GL45 Transfer closure should also be thoroughly cleaned. It is recommended to soak the transfer closure in a dilute acid solution (2% HNO₃) that is heated to approximately 90°C for a minimum of 48 hours to remove contaminants. After soaking, rinse the closures using DIW.

5.0 Operation

5.1. Standard Operating Instructions



Explosive; Fire, Blast or Projection Hazard

Electrics may ignite flammable or explosive mixtures. The apparatus is not designed for organic chemicals. Improper use of the DST-1000 with organic material may cause personal injury or property damage.



Causes Severe Skin Burns and Eye Damage

Take appropriate precautions during filling with and dispensing of acid. In case of contact, flush with copious quantities of water. See reagent manufacturer's material safety data sheet (MSDS) for safe handling instructions.



Personal Protective Equipment

Wear recommended safety apparel when handling or working with acids.



Harmful If Inhaled

Avoid inhalation of acid vapors.

Causes Serious Eye Irritation



Wear eye protection. Ensure the system is in a properly functioning controlled environment (e.g. fume hood). Acid vapor is emitted from the DST-1000 during use.

The Unit Employs High Voltage!

The DST-1000 must be connected to a grounded conductor. Connect the power cord of the control box to a grounded power source.



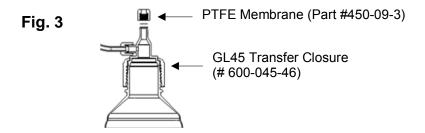
Chemical Hazard!

Chemical hazards associated with the connections, tubing and containers are identical to those noted above.

The DST-1000 will produce a reliable supply of highly purified acid once the cleaning operation has been completed. To achieve the highest level of purity, Savillex recommends using the still for only one type of acid.

DANGER! NEVER MIX CHEMICALS WITHIN THE STILL

- a) Replace the top vent fitting with a fresh 25 mm filter membrane. See Fig. 2
- b) Remove the 1/4" OD ferrule plug from the distillate outlet
- c) Remove the standard threaded closure from the 1000 mL collection bottle and replace with the included GL45 transfer closure
- d) Slide the \(\frac{1}{4} \) OD ferrule on to the 22 cm long distillate tubing and insert the grooved end into the outlet fitting. Finger-tighten the ferrule nut.
- Connect the other end of the 22 cm distillate tubing to the ported closure of the 1000 mL PFA collection bottle. NOTE: The closure must be vented to permit sealed filling. See Fig. 3





Distillate tubing should be no longer than 16 cm to allow proper draining of distillate into collection bottle. Make sure tubing is sloped downward toward collection bottle to allow continuous flow.

Position the distillate collection bottle into the bottle bracket to prevent tipping and spillage during use. See section 4.2 for proper instillation of the bottle bracket.

- f) Verify that the waste drain fittings are secure and that the drain stopcock valve is CLOSED
- g) Remove the threaded cap from the graduated filling tube
- h) Carefully and slowly pour the chemical into the filling tube. DO NOT FILL TOO QUICKLY as you will notice the liquid drains slowly into the reservoir.



Should spilling occur, immediately follow spill and safety protocols. See section 7.1 for details.

- i) Fill to the desired volume, but DO NOT EXCEED 1000 mL of chemical. You can quickly and easily check liquid volume level by observing the graduation marks on the fill tube.
- j) Replace the threaded cap on the fill tube and hand-tighten

5.2 Turn the Power ON

a) The DST-1000 is now ready for distillation. Turn the control knob to the desired setting. Approximate distillation rates for water are listed below:

HI	80°C (176°F) to 90°C (194°F)	50 mL/hr
MID	70°C (158°F) to 80°C (176°F)	30 mL/hr
LO	50°C (122°F) to 60°C (140°F)	10 mL/hr



The HI setting will produce high purity 10 ppt grade acid. The LO and MID settings are useful for reducing the distillation rate during prolonged unattended operation of the DST-1000 to prevent the unit from running to dryness.

- b) The reservoir of the PFA vessel will now begin gently heating to the desired setting. It will take 2-3 hours to reach this temperature.
- c) As the internal chemical temperature rises, evaporation and condensation rates will increase. The purified chemical distillate will collect within the DST-1000 and begin to flow into the distillate collection bottle.
- d) As the purified distillate collects, the contents of the PFA reservoir are being depleted. It is recommended that you never allow the reservoir to run dry. A minimum of 100 mL should always be maintained to prevent the drying of impurities onto the inside surface of the reservoir.



If the DST-1000 runs dry, impurities will be deposited onto the inside surface of the reservoir. Introduction of acid will re-dissolve these impurities, and may reduce the quality of the distillate for a period of time. The DST-1000 will not be damaged.

5.3 Refilling

- a) Monitor acid level in the reservoir during operation using the graduation marks on the fill tube. The DST-1000 may produce up to 50 mL per hour varying with the type of starting material and environmental temperature.
- b) Remove the threaded cap from the graduated filling tube



Remove the threaded cap from graduated filling tube slowly to alleviate any internal pressure that may have occurred during distillation process. The observed liquid level in the filling tube may change as the pressure reaches equilibrium

c) Carefully and slowly pour the chemical into the filling tube. DO NOT FILL TOO QUICKLY as you will notice the liquid drains slowly into the reservoir.



Should spilling occur, immediately follow spill and safety protocols. See section 7.1 for details.

- d) Fill to the desired volume. DO NOT EXCEED 1000 mL OF ACID. You can check liquid volume by observing the graduation marks on the fill tube.
- e) Replace the threaded cap on the graduated fill tube and hand-tighten

5.4 Changing Acid Types

Savillex recommends the DST-1000 be dedicated to a single distillation chemistry to obtain the highest purity acid. If you wish to change the acid distillation from one type of acid to another type of acid (e.g. change from HNO_3 to HCI) you should follow the steps outlined below:

- a) Make sure the collection channel is empty after you finish the distillation. This is accomplished by tipping the DST unit toward the collection vessel so the distillate remaining in the channel is completely removed.
- b) Replace all PTFE vent membranes
- c) Inspect all connections for trace amounts of acid that could be left in the threads or tubing
- d) Check to see that the reservoir is completely drained before adding any new material to the DST-1000



To help clean up the unit faster, do a series of quicker deionized water distillations rather than a single day-long distillation. Fill with deionized water, distill for a few hours (or overnight), empty and discard the water, refill with deionized water and repeat the process several times.

6.0 Optional Operational Configuration

6.1 Use of a Timer

The still may be operated via any commercially available timer control unit used for operation of electrical appliances.

6.2 Overnight Operation

The DST-1000 may safely run overnight. Be aware of the production rate of your specific starting material, and choose the appropriate operating setting (LO, MID, HI) to control the rate of distillation. At 50 mL/hour, an 18 hour distillation will produce 900 mL of distillate. It is recommended that you do not allow the DST-1000 to operate until dry.

7.0 Maintenance, Troubleshooting and Service

7.1 Cleaning and Decontamination



Explosive; Fire, Blast or Projection Hazard

Electrics may ignite flammable or explosive mixtures.

The apparatus is not designed for organic chemicals. Improper use of the DST-1000 with organic material may cause personal injury or property damage.



Causes Severe Skin Burns and Eye Damage

Take appropriate precautions during filling with and dispensing of acid. In case of contact. flush with copious quantities of water. See reagent manufacturer's material safety data sheet (MSDS) for safe handling instructions.



Personal Protective Equipment

Wear recommended safety apparel when handling or working with acids.



Harmful If Inhaled

Avoid inhalation of acid vapors.

Causes Serious Eye Irritation

Wear eve protection.



Ensure the system is in a properly functioning controlled environment (e.g. fume hood). Acid vapor is emitted from the DST-1000 during use.

The Unit Employs High Voltage!

The DST-1000 must be connected to a grounded conductor. Connect the power cord of the control box to a grounded power source.



Chemical Hazard!

Chemical hazards associated with the connections, tubing and containers are identical to those noted above.

The DST-1000 requires little maintenance to provide a reliable means of producing highly purified acid on a regular basis. The few simple tasks listed below will keep the unit maintained and ensure that the DST-1000 is ready for the next distillation.

- Keep the unit clean and free from potential contaminates. The exterior of DST-1000 unit should be cleaned using a damp cloth and a mild cleansing agent suitable for the laboratory environment, such as dilute Micro-90. Gently wipe down the exterior of the unit. DO NOT immerse the DST-1000 unit, power control box, or heating assembly in water or any other cleaning agent. Do not spray the unit with water to clean the exterior. Store the unit in a clean, dry area when not in use, preferably a chemical fume hood.
- Should acid be spilled on the exterior of the DST-1000 unit during filling, or at any other time. immediately follow the proper cleaning and safety protocols in place at your facility for acid spills. These protocols should, at a minimum, include:
 - Containment of the acid spill
 - Neutralization of the acid spill using neutralizing liquids, solids or gels such as "Spill-X"
 - Clean-up and proper disposal of the neutralized spill in accordance with local, state and federal regulations
 - Final rinse and clean of the affected area with an approved cleaning solution



Take special care to avoid spillage while filling the DST-1000. Acid spilled on the DST while filling has the potential to contact the heating element which may damage the heating element resulting in failure of the element.

- After every 40 hours of use, it is recommended to replace all filter membranes to assure optimal operation. See Fig.2 & Fig.3
- For best results, the DST-1000 should be completely drained of waste acid or water after every distillation to remove the highly concentrated contaminants that have accumulated in the reservoir. If scale deposits occur on the interior of the reservoir, a simple cleansing reflux/rinse with hydrochloric acid should remove these scale deposits. Begin by completely draining the unit and follow the initial cleaning steps in Section 4.4 to dissolve any buildup of scale inside the reservoir. Once the scale has been dissolved, drain the HCl rinsate into an appropriate waste container. After this cleaning procedure, the DST-1000 is ready for use.
- Should you wish to decontaminate the interior of the unit, start by:
 - Completely draining all chemical contents as described in Section 4.4
 - o Fill with a maximum of 1000 mL of high purity deionized water
 - o Set the power control unit on "HI" and allow to reflux for a minimum of 48 hours
 - Turn the power control OFF and allow the unit to cool
 - Completely drain the contents
 - Repeat this process two additional times
 - Plug all ports for unit storage or transfer

The wetted parts of the DST-1000 are manufactured of 100% PFA which is highly chemical resistant. The support stand of the still is manufactured of polypropylene, which may stain or become slightly discolored upon contact with acid. Also, extreme or prolonged exposure to liquid acid may damage the polypropylene components of the DST-1000. Should the polypropylene components of the still be exposed to liquid acid for a prolonged period of time, Savillex recommends replacing the affected polypropylene components. Replacement parts are available from Savillex. If the cordage, power control box or heating assembly is damaged by exposure to liquid acid, do not attempt to operate the DST-1000. Contact Savillex for replacement or repair of these items.

7.2 Troubleshooting

Fault	Possible Cause	Corrective Action
Distillation rate is unusually high (or low)	· · · · · · · · · · · · · · · · · · ·	
	Voltage out of specification	Check input voltage to specification.
	Heating element is damaged	Contact Savillex
Poor quality of distillate	Starting material is impure	Use trace metals grade (1 ppb) starting material
	Contaminant concentration in the reservoir is too high	Drain the reservoir and start with new material
No heat to still	Disconnected power cord or cable. CHECK THIS FIRST!	Reconnect cord or cable
	Power control fuse is blown	Replace fuse (1 Amp 250V 3AB fast-acting ceramic body)
	Thermal fuse in heating jacket is blown	Contact Savillex
	Heating element is damaged	Contact Savillex
Warm-up time is slow	Voltage out of specification	Check input voltage to specification
System ran dry	This happens if the output rate is higher than expected or if an attendant is unable to service the still at the appropriate time	Running dry does not damage the still. Simply re-fill when ready to operate again. Consider putting the still on a timer to run for a specific time period and shut-off automatically.
Tube fittings leak	Nuts are loose	Re-tighten

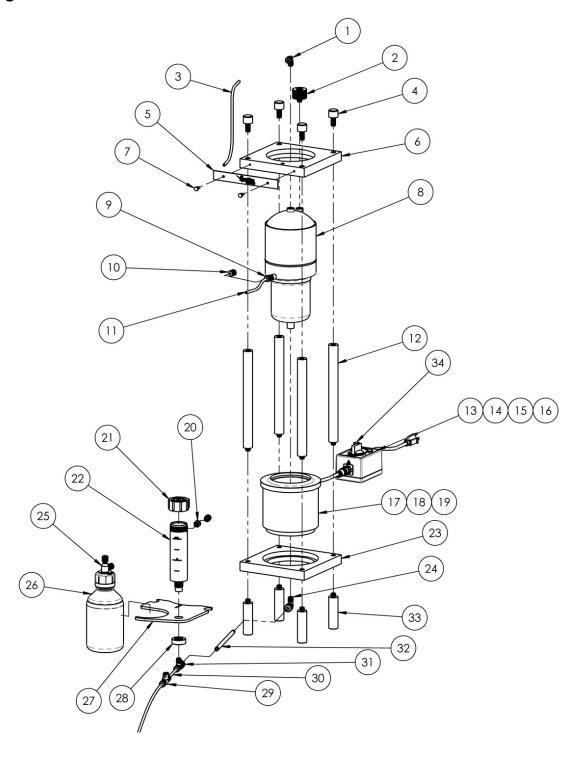
You can obtain technical support from your regional Savillex representative or directly from Savillex. **Phone:** (952) 935-4100 **Fax:** (952) 936-2292 **Email:** info@savillex.com

Please provide your unit's serial number with all repair questions or repair orders. The serial number is located on the back of the still.

7.3 Repairs

The Savillex DST-1000 has no user-serviceable parts, except those that can be readily removed and replaced, i.e. nuts, tubing, stopcock, etc. Specifically, do not attempt to service the heating jacket. Any modification to the still and heating jacket will void the warranty. Replacement parts are available. See Fig. 4.

Fig. 4



Item	Description	Part Number
1	Vent Elbow	750-E4-4N
2	25 mm Vent Assembly	740-100750
3	Vent Tubing (30 cm)	740-200040
4	Assembly Fastener	740-400566
5	Nameplate	740-400603
6	Top Plate	740-400569
7	Nameplate Fastener	740-200157
8	PFA Still Vessel	740-100642
9	1/4" Knurled Ferrule Nut	750-FN4
10	1/4" Knurled Ferrule Nut, Cap	750-FP4
11	Distillate Tubing (22 cm)	740-400602
12	Support Column	740-400570
13	Heating Control Unit - 100VAC	740-100647
14	Heating Control Unit - 115VAC	740-100648
15	Heating Control Unit - 230VAC	740-100649
16	Heating Control Unit - 230VAC, UK	740-100715
17	Heating Assembly - 100VAC	740-100650
18	Heating Assembly - 115VAC	740-100651
19	Heating Assembly - 230VAC	740-100652
20	Vent Connector	740-600669
21	Fill Tube Closure	600-058-04
22	Fill Tube	740-400904
23	Bottom Plate	740-400568
24	Drain Elbow	740-600644
25	GL45 Transfer Closure	600-045-46
26	1000 mL Purillex™ PFA Bottle	150-01-1000
27	Bottle Bracket	740-400905
28	Fill Tube Nut	740-400864
29	Stopcock Valve	740-400599
30	Drain Tubing (90 cm)	740-400601
31	Tee Fitting	740-100740
32	Fill Tube to Vessel 3/8" Tubing	740-400919
33	Base Leg	740-400565
34	Fuse, Type 3AB, Rated 1A, Fast Acting	740-200143

8.0 Appendix

8.1 Warranty Statements

LIMITED WARRANTY

Savillex warrants its DST-1000 Acid Purification System against defects in materials and workmanship for 90 days from the time of shipment to the end-user. The DST-1000 is expressly intended for preparation of high purity reagents through the sub-boiling distillation process. Savillex makes no claim for specific levels of distillate purity, or that the DST-1000 still is suitable for use with all liquids (See *Intended Use*) Warranty coverage excludes product damage which Savillex determines is due to accident, misuse, lack of reasonable care or physical modification of the product. Savillex makes no warranty, express or implied, with respect to any components, products, information or services provided by any third party.

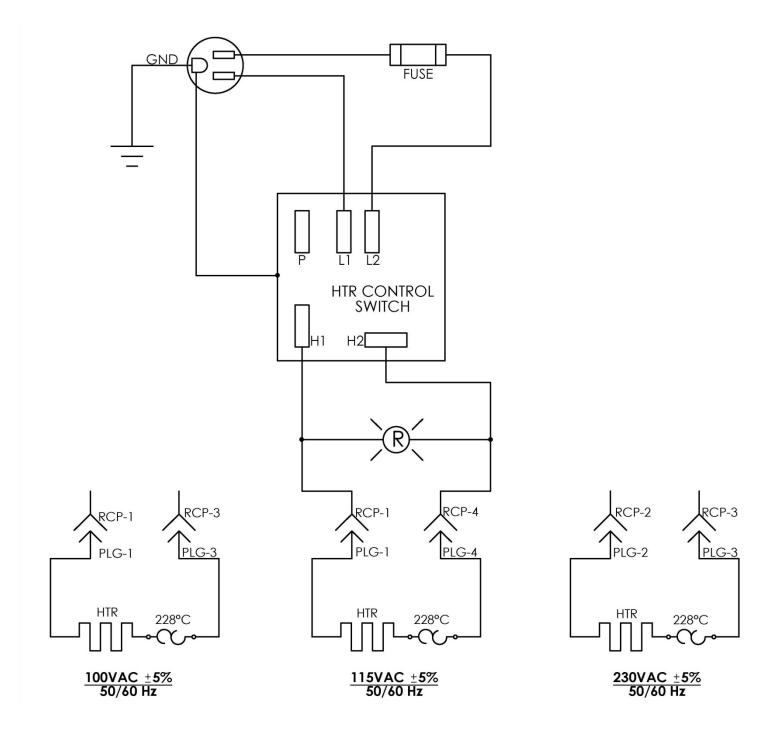
CLAIMS

The end-user's warranty rights are subject to Savillex being promptly notified in writing upon discovery of a warranty claim with a detailed explanation of the defect and verification of the defect by Savillex. Savillex will consider claim submitted during the warranty period and up to 30 days thereafter. Upon confirmation of defects by Savillex, the end-user's EXCLUSIVE REMEDY shall be for Savillex, at its option, to repair or replace the defective product, or to refund the price paid by the Buyer for such product. This remedy is subject to return of the defective product to Savillex or its agent, freight prepaid. **EXCEPT AS STATED IN THIS DOCUMENT, NO OTHER WARRANTY, EXPRESS OR IMPLIED, IS GIVEN OR AUTHORIZED BY SAVILLEX. IMPLIED WARRANTIES OF MERCHANTABILITY AND FITNESS FOR A PARTICULAR PURPOSE ARE EXPRESSLY DISCLAIMED.** ALL REPRESENTATIONS MADE OF SAVILLEX PRODUCTS CONTAINED IN CATALOGS, ADVERTISING, OR REPRESENTATIONS MADE BY REPRESENTATIVES OF THE COMPANY ARE NOT EXPRESSED WARRANTIES AND DO NOT REPRESENT TO THE BUYER THAT A PRODUCT WILL PERFORM TO PARTICULAR SPECIFICATIONS.

LIMITATION OF LIABILITY

SAVILLEX DISCLAIMS AND IS NOT LIABLE FOR ANY SPECIAL, INDIRECT, INCIDENTAL OR CONSEQUENTIAL DAMAGES OF ANY NATURE OR TYPE, WHETHER ARISING OUT OF WARRANTY OR OTHER CONTRACT, NEGLIGENCE OR OTHER TORT, OR OTHERWISE, INCLUDING WITHOUT LIMITATION, FORESEEABLE LOSS, LOST PROFITS AND RELIANCE DAMAGES. IN NO EVENT SHALL SAVILLEX'S LIABILITY UNDER ANY CAUSE OF ACTION RELATING TO A PRODUCT EXCEED THE PURCHASE PRICE OF THE PRODUCT.

8.2 Electrical Schematic



8.3 Declaration of CE Conformity



The Undersigned, representing the manufacturer

Savillex Corporation 10321 West 70th St. Eden Prairie, MN 55344 USA

Herewith declare that the products:

525-1000-100, 525-1000-115,

525-1000-230, 525-1000-230UK

are in conformity with the essential requirements of the following EC Directive(s) when installed in accordance with the installation instructions contained in the product documentation

2001/61010-1/IEC	IEC System for conformity, testing and certification of electrical equipment (IECEE)

and that the standards and/or technical specifications referenced below have been applied:

EN 61010-1: 2001 Safety Requirements for Electrical Equipment for Measurement, Control and

Laboratory Use - Part 1: General Requirements

EN 61010-2-01:2003 Safety Requirements for Electrical Equipment for Measurement, Control and

Laboratory Use - Part 2-010: Particular requirements for laboratory

equipment for the heating of materials

Date of Issue November 24, 2008

Issuer:

Signature

Name: David McKeown

Position: Director of Operations Quality

Date: 01-December-2008



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