Triangle Biomedical Sciences, Inc. Solvent Recovery System

Operation And Service Manual

SR12 Version 1.2

Serial # _____



TRIANGLE BIOMEDICAL SCIENCES, INC.

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FOREWORD

This manual is designed to exhibit the information needed for installing, operating and troubleshooting the TRIANGLE BIOMEDICAL SCIENCES, INC. SR-12 SOLVENT RECOVERY SYSTEM.

The SOLVENT RECOVERY SYSTEM is specifically designed to recycle Alcohol and Xylene. The user may need to manage the concentration of the contaminated solvent to ensure a higher percentage of recycled solvent.

WARRANTY

TRIANGLE BIOMEDICAL SCIENCES, INC. (TBS), for itself and all its subsidiaries, does hereby warrant for a period of one year (12 months) from the date of shipping, under normal and proper usage, all of its products against defects in workmanship and material, and will repair or replace the same without charge when shipped prepaid to TBS's factory in Durham, NC, USA. If the product cannot be so shipped (except for parts and accessories), there will be a charge for all expenses incurred for repairing or replacing the product on the user's premises.

Parts and accessories manufactured or supplied by others are warranted only under the regular warranty of the manufacturer or supplier of such parts in so far as TBS or its subsidiaries are able to transfer the benefit of such warranties to the user.

This warranty supersedes and is given in lieu of all implied warranties, and is void if user does not provide unit with continuous ample electrical power at constant voltage, consistent with the specifications of the product.

User is required to return the warranty card to TBS within ten (10) days after receipt of the product in order to validate the warranty.

EXTENDED WARRANTY

Triangle Biomedical Sciences, Inc. has available an Extended Warranty Agreement which will provide an extension or the warranty for a 12 month period on parts and labor for preventive maintenance and repairs. For details and costs, please contact the TBS office at 1-919-384-9393.

CONDITION OF RETURNED EQUIPMENT

Before returning equipment to TBS, you must contact TBS or your dealer and receive a return good authorization. **All returned units must be cleaned and decontaminated**. The RGA paperwork includes a Certificate of Decontamination for you to sign indicating that you have performed these steps. TBS will not accept the shipment until this signed certificate is received.

You must prepay transportation to the service depot. In addition, TBS must receive unit packed and shipped in compliance with our shipping/crating instructions. Failure to properly crate and ship the unit could result in additional repair fees.

GENERAL INFORMATION

The TBS Solvent Recovery System is designed for recovering solvents used in Histopathology with a boiling point between 50 - 180° C. Using a closed system with fractional distillation design, the Solvent Recovery System can be operated simply and safely.

This Manual - Reading this manual is essential for correct and safe use of this equipment.

Application - The TBS Solvent Recovery System will successfully and economically recover most solvents used for dehydration, extraction of fat and as an inter medium (Xylene, acetone, ethanol, toluene, etc.).

Function - The waste solvents, distilland from tissue processors and/or tissue stainers, are heated inside the distillation vessel of the recycler, converted to vapor and then condensed back into a liquid. A thermostat and a timer will control the process. The vaporized solvent is directed through a collection pipe to an air cooled distilling condenser where the vapor is condensed back into liquid. The distillate is then collected in a closed container on the outside of the unit. The recovery rate of the solvent will depend on the grade of contamination of the waste solvent being recycled.

Separation Module - The TBS Solvent Recovery System utilizes the principle of Simple Distillation, however, it will achieve the results of Fractional Distillation using the Separation Module and Baffle. The Switching Module allows an operator to set a <u>fraction temperature</u> at which the distillate of the different solvents will be separated. The fraction temperature is set relative to the boiling point of the solvent you wish to recover. This fraction temperature is easily set at the beginning of each recovery cycle.

Safety Measures - TBS Solvent Recovery Systems work ONLY with atmospheric pressure. Should the thermal oil temperature exceed the maximum set value of the working thermostat by more than 25°C or if a defective cooling fan allows the distillate to emerge at a temperature higher than 40°C (104°F), the distillation process will be interrupted by a complete and total shut down of the unit. The unit will shutdown if the Thermal Oil temperature exceeds 225°C.

TBS solvent Recovery Systems have been extensively tested and approved by the ETL Testing Laboratories

OPERATIONAL SAFETY

General - TBS Solvent Recovery Systems adhere to ETL guidelines for safety and manufacturing. It is suggested that your laboratory purchase an appropriate fire extinguisher to be placed within ten feet of any electrically heated equipment.

TBS recommends that the Safety Officer for your facility be contacted for proper storage and disposal of any hazardous substance being recycled.

Location - It will be necessary for to contact the Environmental health and Safety Officer and the local fire marshal regarding placement recommendations for your Solvent Recovery System.

TBS Solvent Recovery Systems have been designed and manufactured to be ignition resistant if operated in a highly volatile environment (explosion proof). All units are shipped with a normal 15 amp Hospital Grade, ETLTM & UL approved electrical plug. Be advised that if you intend to operated your unit in a dangerously volatile environment an explosion proof plug must be installed with its special receptacle.

Solvent Containment - TBS offers a stainless steel splash pan, designed for your equipment, to be placed on the floor under your recovery unit. This pan is recommended for use as a safety pan to prevent solvents from spilling onto the floors in case of an over flow due to operator error.

Appropriate 3-gal containers for the collection of recycled solvents and run off waste are available from TBS.

Safe Exposure Temperature - The temperature should be at 50°C or below, according to the top bulb thermometer, before the distilling chamber cover is opened. Opening the cover at a higher temperature will release solvent fumes into the surrounding air and can also cause burn injury to the operator.

NOTE: Opening the cover sooner than one hour after the distillation cycle has finished wil cause the cover gasket to swell.

OPERATING PROCEDURES

Purpose - The TBS Solvent Recovery System was manufactured for the purpose of recovering solvents used within the laboratory setting. Many laboratories now use the TBS equipment for recovering solvents such as xylene and graded alcohols. (See *List of Acceptable Distillation Solvents* in this manual) In the average size laboratory, the recovery of these two solvents have proven to have saved thousands of dollars each year as well as protecting the environment from toxic chemicals being stored and/or transported. In addition, laboratories have decreased their cost of hauling hazardous waste off sight as well as the undesirable, regulatory, responsibilities that go along with that waste removal.

NOTE: When handling chemicals, use gloves, safety glasses with splash guard and an apron.

Procedure for Recovering:

Alcohol:

- 1. Place clean and dry collection containers under each of the lower distillate solvent outlet nozzles left and right.
- 2. Verify that the Thermal oil level is at the correct level.
- 3. Place the disposable plastic bag into the vessel using the expandable metal ring to hold the bag in place. Note: For the 3-gal unit, small hole should be cut near the top edge of the bag to fit over the vapor collection duct.
- 4. Place the contaminated solvent in the distilling chamber, up to 3 gallons.

NOTE: Take special care NOT to allow any contaminated solvent to enter the Vapor Collector Duct Manifold when you are filling the distilling chamber. This may create a build up of wax inside of the condensing coils that can cause blockage and unwanted pressure within the distilling chamber during recovery.

- 5. Close the cover of the chamber by securing the locking knob by tighten clockwise until the <u>knob stops</u>. The spring will act as a release valve if an unlikely blockage of the vapor collector duct occurs.
- 6. Set the distillation temperature with the Working Thermostat 15°C above the boiling point of the contaminated solvent to be recovered.
- 7. Setting of the fractional temperature depends on the solvent you are recycling. You should then set the fractional temperature at approximately 20-30°C higher than your highest boiling point of the contaminates. Alcohol has a boiling point of 78°. Your fractional temperature switch will be set at a minimum of 85°C for the recovery of alcohol.
- 8. Set the timer for distillation. For a full distilling chamber it will take 5-6 hours for complete distillation to be accomplished. This may vary with the solvent. Alcohol, for example, may take up to 6 hours. The final results will not be effected and the unit will not be damaged if you over set the timer. NOTE: When you wish to recycle alcohol (boiling point of 78°C), it is necessary to change the temperature of

the Working Thermostat dial setting. The setting should be reduced to 150°C the same for Alcohol, and you must adjust the Fractional Thermostat temperature down to 85° C when recovering Alcohol.

- 9. The first container under the solvent outlet nozzle will collect the purest alcohol. Your second container, under solvent outlet nozzle will collect the recovered alcohol.
- 10. After a recovery cycle has been completed, let the unit cool to at least 50°C prior to opening the secured top. Premature opening will allow a release of solvent fumes into the surrounding atmosphere and can also cause burn injury to the operator.
- 11. Open the cover, remove expandable ring and disposable bag. Dispose of the waste in the bag according to appropriate state and local guidelines. NOTE: Take special care not to spill any remaining waste material (especially wax) into the vapor collecting duct.
- 12. Be sure that the distilling chamber remains free of liquid and debris, after each cycle.

Approximately 45-90 minutes are necessary before the distillate begins to flow. Be certain that there is sufficient ventilation to prevent dangerous gases and vapors from forming. During the first few distillation cycles there may be a small amount of thermal oil leakage. This is quite normal and is to be expected. Do not tilt or shake the unit once loaded or when operating. This could create a dangerous situation.

Xylene:

- 1. Place clean and dry collection containers under each of the lower distillate solvent outlet nozzles left and right.
- 2. Verify that the Thermal oil level is at the correct level.
- 3. Place the disposable plastic bag into the vessel using the expandable metal ring to hold the bag in place. Note: For the 3-gal unit, small hole should be cut near the top edge of the bag to fit over the vapor collection duct.
- 4. Place the contaminated solvent in the distilling chamber, up to 3 gallons.

<u>NOTE</u>: Take special care NOT to allow any contaminated solvent to enter the Vapor Collector Duct Manifold when you are filling the distilling chamber. This may create a build up of wax inside of the condensing coils that can cause blockage and unwanted pressure within the distilling chamber during recovery.

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IMPORTANT: IF CHLORINATED SOLVENTS ARE TO BE RECOVERED, YOU MUST USE THE WORKING TEMPERATURES GIVEN IN THE ACCEPTABLE DISTILLATION SOLVENTS TABLE. WORKING WITH A HIGHER TEMPERATURE COULD RESULT IN FORMATION OF HYDROCHLORIC ACID. THIS CHEMICAL REACTION COULD CAUSE DAMAGE TO BOTH THE VESSEL AS WELL AS THE CONDENSER, BOTH MADE OUT OF STAINLESS STEEL. THIS WOULD VOID ANY AND ALL WARRANTIES.

ACCEPTABLE DISTILLATION SOLVENTS

Flammable Solvents	Boiling	Ignition
<u>Name</u>	Point (°C)	Point (°C)
Acetone	50	535
Benzol	80	560
2-Butanol	118	366
N. Butyl Acetate	128	370
N. Butyl Alcohol	118	366
Butyl Cellosove	173	239
N. Butyl Ether	143	185
Cellosolve	143	235
Cellosolve Acetate	156	377
Cyclohexane	81	260
Cyclohexanon	155	419
1.2 DiChlorethane	84	412
DiEthyl Cellosolve	121	208
DiEthyl Ketone	104	445
Ethanol	82	400
Ethyl Acetate	79	427
Ethyl Amyl Keytone (EAK)	160	
Ethylene Glycol	198	413
2-Puran Methanol	162	490
N. Hexane	70	240
Kerosene	190-259	210
Isobutyl Acetate	119	420
Isobutyl alcohol	111	430
Isopropanol	83	400
Isopropyl Acetate	89	460
Methyl Ethyl Ketone (M.E.K.)	80	530
Methyl Acetate	58	475
Methyl Amyl Ketone	150	311
Methyl Cellosolve	124	285
Methyl Cellosolve Acetate	156	377
Methyl Isobutyl Ketone (M.I.B.K.)	117	459
Mineral Spirit 1	162-180	250
Mineral Spirit 2	185-215	-
N. Octane	126	220
N. Propanol	98	273
S.B.A. Sec. Butanol	101	-
Shellosol TD-Isopar H-Sepisol TD Helpasol 170-195	170-187	-
Shellsol T-Isopar L-Sepisol T Halpasol 180/185	180-214	-
Shellsol D40-Exol D40-Sepisol D40	162-197	-
Shellsol HD60-Exxol D60 Sepisol HD60	185-219	-

Shellsol D70-Exxol D80-Sepisol KD70	190-257	-
Shellsol A Solvesso 100 Sepisol A	162-180	-
Shellsol AB-Solvesso 150	182-215	-
Shellsol H-Varsol 60-Sepisol H	180-210	-
Toluene Toulol	111	535
Turpentine Gum Spirits	152-270	250
Xylol/Xylene	140	525

IMPORTANT INFORMATION: Never open the recycler prior to the suggested cool of time of 50° C. Always be certain that the cover gasket does not show signs of wear or have any debris attached to it. The green "on" light should be indicated prior to starting each cycle.

WARNING: Never change the thermal oil before allowing the machine to cool for at least 48 hours.

Nonflammable Solvents

	Boiling	Working
<u>Name</u>	Point (°C)	Temp.(°C)
Chloroform-Trichloromethane	661	90
Methilene Chloride-DiChlormethane	40	70
Freon 113-Frigen 113	46	80
Carbon Tetrachloride-Tetrachloromethane	78	110
1.1.2.2 Tetrachloroethane Acetylene Tetrachloride	147	160
Tetrachloroethylene-Pestabil-Vors Perchloroethylene-Per	121	150
Trichloroethane-1.1.1.3*1- Chlorothene-Genklene-Solvetane		
Baltane-Mecloran	74	110
Tricholoethylene-Tri Vorclin-Althene-Triklone	87	120

Warnings:

- 1. When treating nonflammable solvents do not set the working thermostat at a temperature higher than that shown above. Higher temperatures may produce hydrochloric acid, acidify the solvent and consequently damage the recycler.
- 2. Any solvent containing more than 35% water will not recycle successfully.

MAINTENANCE

- **Daily -** A) Clean the distillation vesselremainder of 8.1 from Omega Manual
 - B) it is best to remove paraffin build-up when the unit is warm
- **Weekly** A) Using a soft cloth and mild solvent clean up the exterior of the apparatus.
 - C) Check the thermal oil level when the unit is cold, the level should be at the lower indicator.

NOTE: Because the oil expands when it is heated leakage will occur if the oil reservoir is over filled.

Annual - After 2,000 hours of operation of the 3-gal unit or after one year (which ever is earlier), it will be necessary to have preventive maintenance performed on the unit. This annual maintenance will include a change of the thermal oil and servicing of various other parts along with the extension of the limited warranty for an additional 12 months. This can be accomplished by either, 1) purchasing an Extended Warranty Agreement or 2) for a fee, having a technician from the laboratory trained and certified to perform the maintenance.

SAFETY FEATURES

The red indicator light when illuminated indicates that one of the two Safety thermostats has completely turned off the system at sometime during the recovery cycle. The following is a description of all four thermostat and timer. Only <u>Safety</u> thermostats will activate the red indicator light.

<u>Timer</u> Basic five hour timer. Rotate clockwise to start the recovery cycle. Note that the cooling fan will continue to operate for 30 minutes after the heating cycle has ended.

<u>Working Thermostat</u> Used to control the temperature of the thermal oil that transfers heat to the distilling chamber. Range 40-200° C. Note: The oil temperature is generally 15 - 20° C higher than the temperature of the contaminated solvent within the distilling chamber.

<u>Safety Thermostat</u> Monitors the temperature of the thermal oil. If the Working Thermostat should malfunction Safety Thermostat will STOP the heating if the oil temperature to exceed exceeds 225° C. <u>The</u> red indicator Light will be activated.

<u>Safety Thermostat</u> The cooling fan cools the distillate to a temperature slightly above room temperature. Should the fan malfunction the distillate pouring out of the outlet nozzles will be hot. The Safety Thermostat monitors the temperature of the distillate. If that temperature exceeds 40° C the electrical heating will be stopped and <u>the red indicator light will be activated.</u>

<u>Fractional Thermostat</u> A probe monitors the temperature of the vapor at the vapor collector duct. The Fractional Thermostat allows the operator the ability to control the temperature when the three-way valve will switch the distillate from the left distilled outlet nozzle to the right distilled outlet nozzle.

The red indicator light "out of work" signals that one of the safety thermostats have intervened. Turn off the apparatus and refer to the "Malfunction, Causes and Remedies" portion of this manual.

MALFUNCTIONS, CAUSES AND REMEDIES

Problem:

Unit is heating up to set temps.

Unit will recycle off water, alcohol and xylene into one container, second container, first container remains empty.

Initial troubleshooting revealed that the switching module was okay and did not need replaced.

Possible Solutions:

Middle thermostat, which controls distillation temp may have become loose within well and is not registering temp.

Middle Thermostat may need replaced.

To Check:

Turn unit on heat it without anything in it. Move the middle thermostat back and slowly move to higher temp. Watch to see if the indicator light changes from set to distillation temp. If this happens, thermostat is okay.

Thermostat probe may still be out or loose in distillation chamber. Take front cover off of unit, it comes in on the top right side of the unit.

	t	i
Distillation time lasts much	1. Contaminated solvent	1. Replace the solvent
longer than 4 hours	contains too much water	2. Add oil
	2. Thermal oil level is low	3. Change the oil
	3. Thermal oil is old and	4. Remove and clean with
	dirty	solvent
	4. Heating elements need	
	cleaning	
Heats but does not distill	1. Distilling chamber is dirty	1. Clean chamber
	2. Solvent distillation temp. is	2. Increase the Working
	higher than set on working	Thermostat temp. setting
	thermostat	3. Change solvent
	3. Distillate temperature too high	4. Add oil
	4. Thermal oil level is low	
Red Indicator Light is "ON"	1. Working temperature is too	1. Reduce the working temp
	high	2. Change the Working
	2. The max. temp. Safety	Thermostat
	Thermostat intervened	4. Replace motor
	3. The distillate Safety	4. Clean or replace
	Thermostat intervened	5. Remove the protective cage
	4. Cooling motor not operating	and clean it with a brush or air
	5. Distilling Condenser scaled	pressure
	6. Maximum Temp. Safety	6. Replace Max. Temp. Safety
	Thermostat is defective or out	Thermostat
	of calibration	
	Condensing apparatus has	

TECHNICAL DATA

Maximum Capacity 12 Liters (3.2 gal) 19 Liters (5.0 gal) Type of Distillation Simple Simple

Volume of Thermal Oil 5 Liters (1.5 gal) 14 Liters (4.0 gal)
Voltage 115V/60Hz 115V/60Hz
Current 9 Amps 17.5 Amps

Working Temperature 40 - 200°C. (424°F.) 40 - 200°C. (424°F.)

Cycle Time 5 -6 Hours 5 -6 Hours
Condenser Air Cooled Air Cooled
Size (W x D x H) 59 X 59 X 145 cm 61 X 76 X 173 cm
Weight 74 kg 135 kg

LIST OF PARTS

- 1. Distilling Chamber
- 2. Camber Cover
- 3. Electrical Box Cover (Expl Prf)
- 4. Electric Box Housing (Expl Prf)
- 5. Bushing Carrier (Expl Prf)
- 6. Fraction Switch Cable
- 7. Electrical Heating Element(s)
- 8. Thermal Oil Vessel
- 9. Fan Motor Junction Box (Expl Prf)
- 10. Distilling Condenser
- 11. Cooling Fan Motor (Expl Proof)
- 12. 3-Way Fraction Switch (Expl Prf)
- 13. Heater Cable Sheath Pipe (Expl Prf)
- 14. Solvent Receiver Tanks
- 15. Solvent
- 16. Oil Discharge
- 17. Thermal Oil Level Indicator
- 18. Working Thermostat T-1
- 19. Timer
- 20. Fraction Thermostat T-4
- 21. Indicator Window
- 22. Vapor Temperature Gauge
- 23. Oil Bleeding Valve

- 24. Locking Knob
- 25. Cover Gasket
- 26. Arm for Cover
- 27. Vapor Collector Duct (Manifold)
- 28. Solvent Outlet Nozzle- Left
- 29. Solvent Outlet Nozzle- Right
- 30. Oil Temperature Gauge
- 31. Oil
- 32. Bag Holder/Max Level of Solvent
- 33. Main Power Electric Cable
- 34. Motor Cable Sheath Pipe (Expl Prf)
- 35. Unit Support
- 36. Shell
- 37. Heater On Indicator-Orange
- 38. Main Power Indicator Green
- 39. Alarm Indicator Light Red
- 40. Timer Module
- 41. Working Thermostat T-1
- 42. Safety Thermostat T-2
- 43. Safety Thermostat T-3
- 44. Fraction Thermostat T-4
- 45. Safety Relay
- 46. Heater Power Relay
- 47. Fraction Switch Junction Box

SPARE PARTS

<u>Code</u>	Description	<u> 12 liter</u>	<u> 19 Liter</u>
_	Cover Gasket, Ether	, [_
=	Cover Gasket, Ether	v -	√
239004	Cover Gasket, Xylene	√	v -
239010	Cover Gasket, Xylene	• -	$\sqrt{}$
382000	Condenser, Steel		v -
382001	Condenser, Steel	• -	$\sqrt{}$
384007	Warning Light Socket	$\sqrt{}$	$\sqrt{}$
384008	Safety Thermostat (T3)	$\sqrt{}$	$\sqrt{}$
384009	Working Thermostat (T1)	$\sqrt{}$	$\sqrt{}$
384010	Safety Thermostat (T2)	$\sqrt{}$	$\sqrt{}$
465000	White Warning Light	$\sqrt{}$	$\sqrt{}$
465001	Red Warning Light	$\sqrt{}$	$\sqrt{}$
465002	Green Warning Light	$\sqrt{}$	$\sqrt{}$
475000	Ventilator Motor	$\sqrt{}$	$\sqrt{}$
468402	Heating Element	$\sqrt{}$	$\sqrt{}$
385007	Timer	$\sqrt{}$	$\sqrt{}$
425002	Control Knob	$\sqrt{}$	$\sqrt{}$
350901	Bushing for Spring (Locking Stay)	$\sqrt{}$	_
350911	Bushing for Spring (Locking Stay)	-	$\sqrt{}$
353702	Spring for Stay	$\sqrt{}$	-
428001	Spring for Stay	-	$\sqrt{}$
357400	Stay	$\sqrt{}$	-
357401	Stay	-	$\sqrt{}$
427000	Hand Wheel	$\sqrt{}$	-
427001	Hand Wheel	-	$\sqrt{}$
NG20001	Three way Fraction Valve (Expl. Proof)	$\sqrt{}$	$\sqrt{}$
NG20002	Fraction Outlet Nozzle	$\sqrt{}$	$\sqrt{}$

GLOSSARY

Auto-ignition Point - The temperature at which a solvent will self ignite (no stimulus required).

Baffle - A metal duct used to direct the vapors to the vapor collection manifold. The baffle is filled with packing to impede the flow of vapor causing a reflux. The condensed liquid rectifies vapor.

Boiling Point - The temperature at which the given product starts to transform into its gaseous form.

Cut - A fraction or portion of distilled material taken off at the head (Vapor Collection Duct Manifold) of the still.

Distilland - The mixture of materials placed in the distilling vessel: the material to be distilled.

Distillate - The material that has been boiled through a column: The separated material taken off at the head.

Efficiency - A means of comparing the actual useful separating power of distillation columns.

Flash Point - The temperature at which the solvent will ignite if brought in contact with a spark.

Flooding - Excess liquid forced up the still into the condenser as a result of overfilling the vessel and the high heat input at the pot. Usually detrimental, since the distlland mixture is forced into the area in which only pure material is desired.

Fraction - A portion of the collected separated material recovered from the head of the still.

Fractional Distillation - distillation in which rectification is used to obtain a product as nearly pure as possible. In the operation, part of the vapor is condensed as it passes through packing and the liquid is refluxed down the column; thus producing a high vapor-to-liquid contact. The term is also used to describe any distillation in which the product is collected in a series of fractions or cuts.

Rectification - The enrichment or purification of vapor by contact and interaction with a counter-current stream of liquid condensed from the vapor.

Reflux - the term used to describe the liquid condensed from rising vapor, which is allowed to flow back down the column.

Simple Distillation - Distillation in which no appreciable rectification or reflux occurs: All condensed vapor is removed from the still at the head.

Takeoff - the liquid removed from the still per unit time: The amount of separated material recovered from the still.

Throughput - the rate at which vapor is capable of passing up the column against resistance of the packing: it is a measure of the output capacity of the still